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ANAEROBIC CO-DIGESTION OF MUNICIPAL SOLID WASTE AND SEWAGE SLUDGE

by

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ABSTRACT

As a solution to the problems of municipal solid waste management, anaerobic digestion possesses the optimal combination of volume reduction, probability of success and potential for both energy and resource recovery. An innovative application of anaerobic processes is the co-digestion of sewage sludge and the organic fraction of municipal solid waste (OFMSW). In Europe, anaerobic co-digestion has become a popular alternative to landfilling and is currently breaking into the North American market. The purpose of this study is to evaluate the technical feasibility of this process in the context of typical Canadian solid waste.

Lab-scale experiments were initially conducted using one litre batch bioreactors operated mesophilically (37 °C) and fed a mixture of primary sludge (RAW), thickened waste activated sludge (TWAS) and simulated OFMSW. Based on biological activity tests an optimal region for the OFMSW content was found to be less than 50% OFMSW. Of the experimental runs, a mixture of 25% OFMSW and 75% sewage sludge (60% RAW, 40% TWAS) displayed the highest biogas production and was hence used in ensuing experiments. Based on the rate of biogas production, the most anaerobically biodegradable components were paper and grass. The TWAS and the newspaper were found to be the least biodegradable components.

To facilitate organics solubilization, three pretreatments were evaluated: thermal, alkaline and thermochemical. Using a central composite experimental design, two factors were studied, the total solids content of the feed and its particle size. For all three pretreatments, second order empirical models were developed with respect to common indicators of metabolic activity, namely, biogas production, biogas methane concentration, soluble chemical oxygen demand (COD) removal and total and volatile solids reduction. The particle sizes studied ranged from 0.8 mm to 8.0 mm, the total solids concentrations (TS) from 6.6% to 23.5% and alkaline treatments from 16 meq/L to 184 meq/L of sodium hydroxide.

Both batch and continuous reactors indicated that alkaline pretreatment increased the biodegradability of the sewage sludge/OFMSW mixture the most, as compared to the untreated control mixture, by as much as 46%. Thermal pretreatment also appeared to increase biodegradability, but only slightly (7%). Thermochemical pretreatment indicated an initial increase in biodegradability, however, after approximately 250 hours of digestion, displayed inhibitory effects. This inhibition resulted in performances up to 12% lower than the control. Overall, the models indicated that all three factors evaluated in the empirical models were significant with respect to the response variables studied. In all cases, the optima were at high TS concentrations (22.1%). In the case of particle size, the optima for the control were at small particle sizes (0.85 mm), however for all pretreatments, the optima were at large particle sizes (8.0 mm). The reversal in the trend is most likely due to microbiological instability, where in the pretreated feeds the hydrolytic microorganisms predominate not allowing the methanogens to symbiotically associate as well as at larger particle sizes. For both the alkaline and thermochemical pretreatments optima were observed at low alkaline doses (16 meq/L).

RÉSUMÉ

Comme solution aux problèmes de la gestion des déchets solides municipaux, la digestion anaérobique possède la combinaison optimale de réduction de volume, probabilité de succès et potentiel pour la récuperation d'énergie et ressources. Une application innovative des procédés anaérobiques est la co-digestion des vidanges et la fraction organique des déchets solides municipaux (FODSM). En Europe, la co-digestion anaérobique est devenue une alternative populaire au lieu de l'utilisation des dépotoires et maintenant s'introduise au marché nord américain. Le but de cette étude est l'évaluation de la faisabilité de ce procédé dans le contexte des déchets solides canadiens.

Des expériences d'échelle du laboratoire ont été conduites avec des bioréacteurs d'un litre mésophiliquement (37 °C) et alimenté un mélange de boue primaire (BP), boue épaisse de déchets activés (BEDA) et du FODSM simulé. Basé sur des analyses d'activité biologique une région optimale pour le contenu d'FODSM a été trouvé d'être moins de 50% FODSM. Parmis les expériences faites, un mélange de 25% FODSM et 75% vidanges (60% BP, 40% BEDA) a démontré la plus haute production de biogaz et donc a été utilisé dans les expériences qui s'ensuit. Basé sur le taux de production de biogaz, les composants les plus biodégradables sont le papier et l'herbe. La BEDA et le papier de journal étaient les moins biodégradables.

Pour faciliter la solubilisation des organiques, trois prétraitements ont été étudiés: thermal, alcalin et thermochimique ont été démontrés efficaces avec plusieurs substances. Utilisant un design expérimental de composée centrale, deux facteurs ont été étudiés: la concentration de solides totaux de l'alimentation et la taille des particules. Par conséquent, ces facteurs ont été investigés en utilisant un design expérimental de composée centrale. Pour tous les trois prétraitements, des modèles empiriques d'ordre second en termes d'indicateurs communs, la production de biogaz, la concentrations de méthane dans le biogaz, la réduction de la demande d'oxygène chimique soluble, les solides totaux et volatiles. Les tailles de particules étudiées ont rangées de 0,8 mm à 8,0 mm, les concentrations de solides totaux de 6,6% à 23,5% et les doses d'alcalin de 16 meq/L à 186 meq/L d'hydroxide de sodium.

Les réacteurs de lot et continuels ont indiqué que le prétraitement alcalin a augmenté la biodégradabilité du mélange d'FODSM/vidanges le plus, comparé au contrôle, d'au plus 46%. Le prétraitement thermal a aussi démontré une augmentation en biodégradabilité, mais insignifiant (7%). Le prétraitement thermochimique a indiqué une augmentation initialle, mais, après approximativement 250 heures de digestion, a démontré des effets inhibiteurs. L'inhibition a résulté dans des performances d'au plus 12% moins que le contrôle. En général, les modèles ont indiqué que les trois facteurs évalués dans les modèles ont été trouvé signifiants en termes des variables de réponses étudiés. Dans tous les cas, les optima ont été à hautes concentrations de solides totaux (22.1%). Dans le cas de taille de particule, les optima pour le contrôle étaient à taille petite (0.85 mm), mais pour les prétraitements, les optima étaient à grande taille (8.0 mm). Le renversement dans la tendance est probablement due à d'instabilité microbiologique, où pour l'alimentation prétraitée les microorganismes hydrolytiques prédominaient empêchant les méthanogènes d'associer symbiotiquement comme aux grandes tailles. Pour les prétraitements alcalins et thermochimiques, les optima ont été observés aux petits doses d'alcalin (16 meq/L).

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Dedication

To Nadia Mahmoud Sayed,

whose giant footsteps I aspire to fill.

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LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
BTA	Biotechnische Abfallverwertung GmbH
BEDA	Boue épaisse de déchets activés
BIOS	Biosolids
BP	Boue primaire
C/N	Carbon-to-Nitrogen Ratio
DRANCO	Dry Anaerobic Composting of Organic Wastes
EPA	Environmental Protection Agency
FAS	Ferrous Ammonium Sulphate
FODSM	Fraction organique de déchets solides municipaux
HSAD	High-Solids Anaerobic Digestion
IC&I	Industrial, Commercial and Institutional Waste
KHP	Potassium Hydrogen Phthalate
MSW	Municipal Solid Waste
OFMSW	Organic Fraction of Municipal Solid Waste
RAW	Raw Primary Sludge
RefCOM	Refuse Conversion to Methane
RMOC	Regional Municipality of Ottawa-Carleton
ROPEC	R.O. Pickard Environmental Centre
SEBAC	Sequential Batch Anaerobic Composting
TWAS	Thickened Waste Activated Sludge
WAS	Waste Activated Sludge

NOMENCLATURE

A	Volume of FAS required for blank, mL
ALK_DOSE	Coded alkaline dosage
B	Volume of FAS required for sample, mL
COD	Chemical Oxygen Demand, mg O_2/L
E(Y)	Expected value of Y
f	Degrees of freedom
F_{lof}	Lack-of-Fit test statistic
H_o	Null Hypothesis
HRT	Hydraulic Retention Time, days
k	Number of factors being studied
M	Concentration of FAS, mg/L
m_{aft}	Weight of dried residue plus dish after ignition, mg
m_{bef}	Weight of dried residue plus dish before ignition, mg
m_{dish}	Weight of empty dish, mg
m_{res}	Weight of dried residue plus dish, mg
N	Total number of design points
N_{tip}	Number of tips during timing period
n_a	Number of axial points
n_f	Number of points in factorial position
n_o	Number of centre points
p	Number of coefficients
PAR_SIZE	Coded Particle Size
Q	Air flowrate, mL/s
R^2	Coefficient of Determination
SRT	Solids Retention Time, days
SS	Sum of Squares
t	Time for run, s
TOT_SOL	Coded Total Solids Concentration

TS	Total Solids Concentration, weight %
V	Volume of air per tip of wet tip meter, mL
V_s	Volume of sample, mL
VFA	Volatile Fatty Acids, mg/L
VS	Volatile Solids Content, mg/L
VS_a	Volatile Solids Addition
VS_r	Volatile Solids Removal
X	Coded factor
X_1	Particle size, mm
X_2	Total solids, %
X_3	Alkaline dose, meq/L
y	Observed value

Greek Letters

α	Distance from star point to centre
β	Model parameter
σ	Standard Deviation
σ^2	Variance

CHAPTER 1:
Introduction

With landfill sites quickly approaching capacity and the enforcement of stricter environmental regulations, solid waste management has become an increasing challenge for Canadian municipalities. Included among the many solid waste streams to be treated are municipal solid waste and sewage sludge from the wastewater treatment process. The complex nature of these waste streams complicates the implementation of efficient and effective treatment technologies. This thesis focuses on the evaluation of anaerobic co-digestion as a potential solid waste management strategy for the treatment of municipal solid waste and sewage sludge in the Regional Municipality of Ottawa-Carleton (RMOC).

1.1 Municipal Solid Waste Management in Ottawa-Carleton

Municipal solid waste (MSW) consists of all the solid and semi-solid materials discarded by a community including food scraps, containers, packaging, yard trimmings and miscellaneous inorganic wastes from residential, commercial, institutional and industrial sources (EPA, 1995). In 1995, 174,000 tonnes of MSW were collected by the Regional Municipality of Ottawa-Carleton (RMOC, 1995). Accordingly, cost-effective and environmentally sound management of MSW has been identified as a high priority issue. The concept of integrated solid waste management is being implemented by the RMOC and other municipalities as they plan for the future. Future plans typically include reduced waste production, composting and recovery of waste for recycling.

To facilitate the development of effective solid waste management plans, accurate characterization of MSW was necessary to identify appropriate treatment technologies. The RMOC performed a waste audit (RMOC, 1992) on both residential and industrial, commercial and institutional (IC&I) wastes. A detailed breakdown of these waste streams is given in Appendix A.

Residential waste represented 43% by weight of the total solid waste stream. The classes of materials which made up the largest fractions of residential waste were paper, yard trimmings and other organics (consisting of mainly food waste). Since food waste was found to be the largest single subcategory of material in residential waste, one of the recommendations of the audit was to integrate composting into the RMOC's solid waste management strategy. Yard trimmings were also identified as a potential feed for composting.

The remaining 57% of the total solid waste stream was IC&I waste composed of mainly paper and other organics (food waste). From the study, two main recommendations were proposed. The first was to develop programs to recover food waste from hotels, restaurants, supermarkets and educational establishments. To address the high proportion of paper wastes, the development of a program to recover newspapers from hotels was also suggested.

As indicated in Figure 1, both MSW streams possess high proportions of organic materials rendering them amenable to biological treatment processes. Consequently, the RMOC undertook an investigation of alternative technologies for the biological treatment of the organic fraction of MSW. The key criteria for evaluation of these alternatives were: technical feasibility, impact on the environment and cost effectiveness (RMOC, 1992).

Composting was identified by the RMOC as a sound alternative for the stabilization and ultimate disposal of MSW. In 1992, windrow systems were adopted to treat regional yard waste. The windrows were comprised of rows of MSW four to five metres high and five

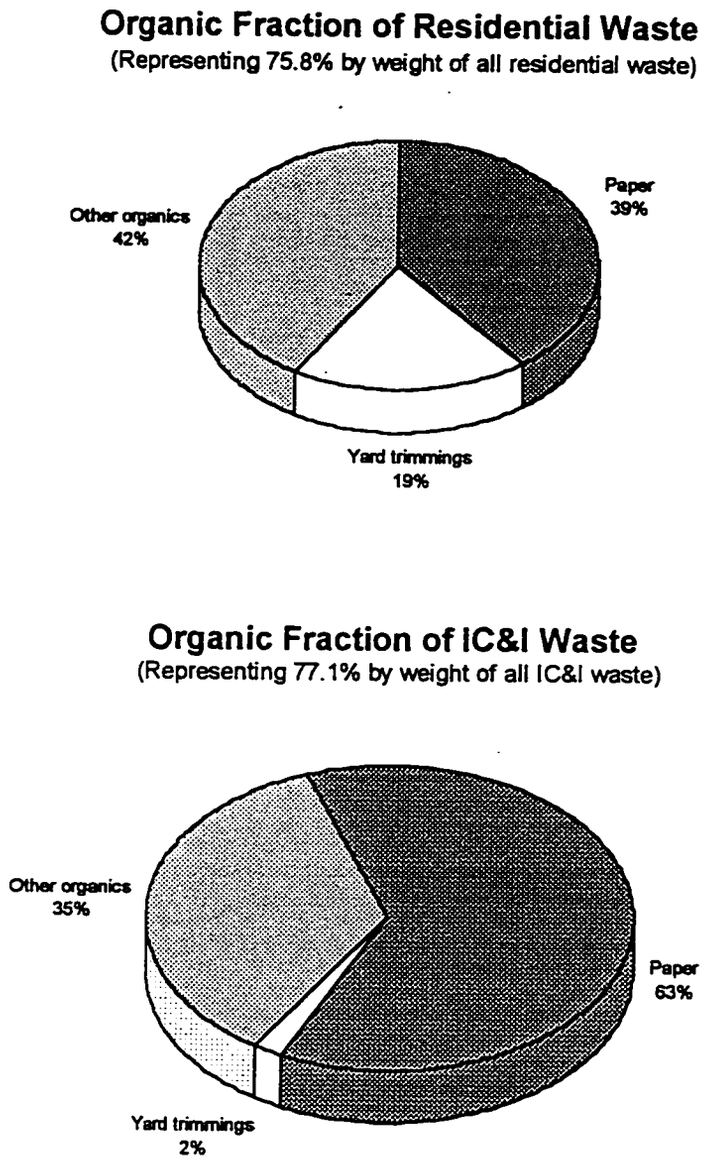


Figure 1: Organic Fraction Distribution of MSW in the RMOC (RMOC, 1992)

to seven metres at the base (RMOC, 1992). Aeration was achieved by periodical turning and mixing of the rows. Within this configuration, resource recovery was achieved through the generation of a quality compost.

An emerging alternative to aerobic composting is high-solids anaerobic digestion (HSAD), a leading technology in Europe. In addition to resource recovery, HSAD processes offer the advantage of energy recovery through generation of methane biogas. Given these advantages, the technical and economic feasibility of HSAD of MSW should be investigated for implementation within the RMOC.

1.2 Sludge Management at the R.O. Pickard Environmental Centre

The R.O. Pickard Environmental Centre (ROPEC) is a secondary wastewater treatment plant treating on average 4.34 million litres of wastewater a day (RMOC, 1995). As indicated in Figure 2, sewage sludge is generated in the process during primary and secondary clarification. The current sludge treatment process consists of thickening, anaerobic digestion and dewatering. After the thickening stage, the sludge is anaerobically digested in mesophilic digesters (37 °C) operated at a solids retention time (SRT) of about 20 days (RMOC, 1992). The digested sludge is then dewatered to produce 30 tonnes of biosolids (BIOS) daily. The biosolids are currently spread on agricultural land as a soil amendment and also as a component in final cover material at the Laidlaw Carp Road Landfill; the remainder is landfilled. The sludge treatment process and the disposal of BIOS are currently ROPEC's most costly operations. Consequently, the RMOC considers its current solids management program to be adequate for the short term, however it is investigating alternatives for long-term solids management. This requires a careful analysis of alternatives for solids treatment at all points in the wastewater treatment process where solids are produced.

The raw primary sludge (RAW), produced after primary settling, is generally comprised of putrescible material and pathogenic organisms (Gore and Storrie Ltd., 1995). Daily, 1.3 million litres of RAW are produced and typically contain three to seven percent total solids. Of these solids, approximately 80% are volatile and hence highly organic in nature. In the activated sludge aeration tanks, biological solids are produced through the consumption of soluble organic matter. These solids (approximately one percent TS),

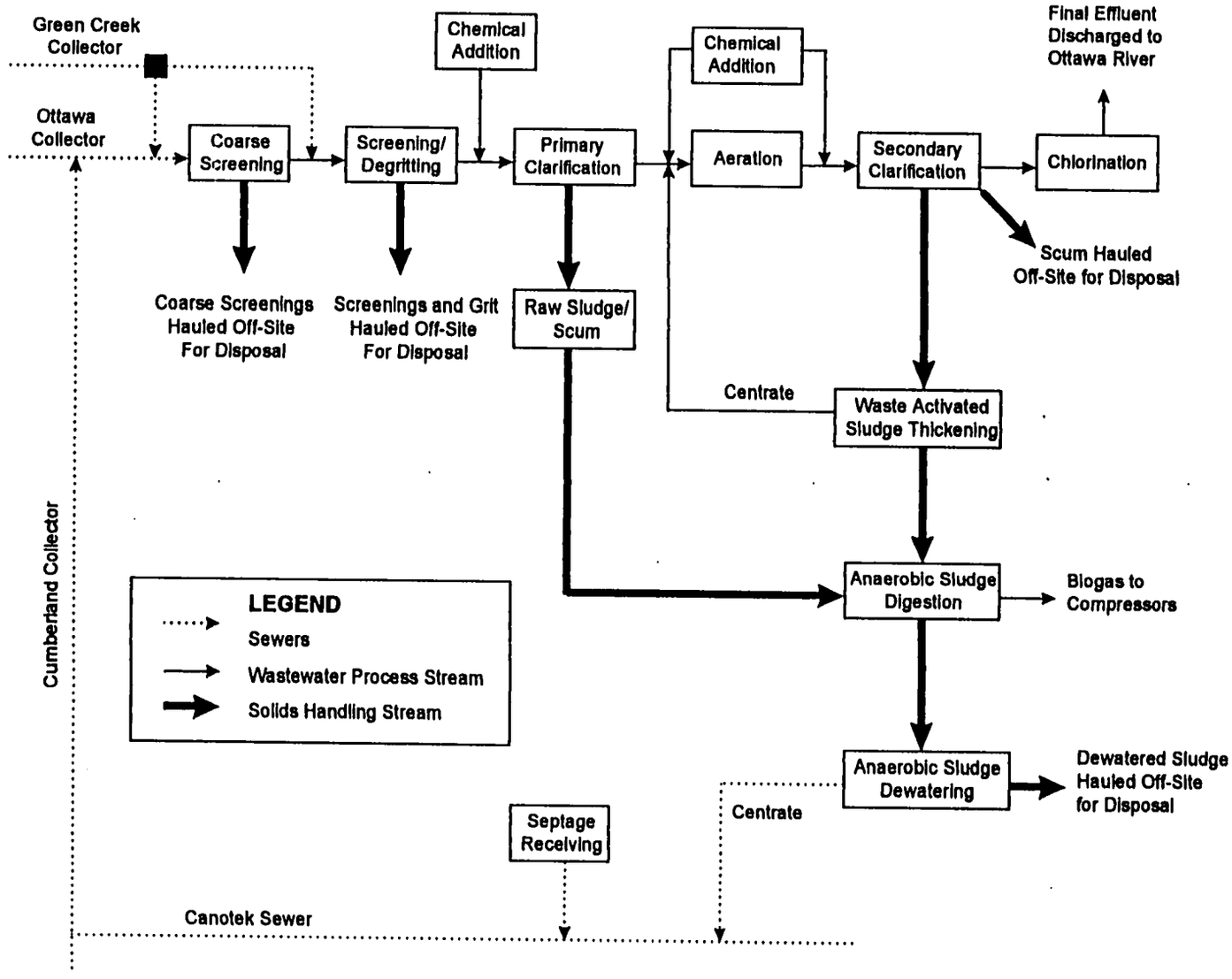


Figure 2: ROPEC Wastewater Treatment Facility Flow Diagram (Gore and Storrie Ltd., 1995)

collected after secondary settling, consist of waste activated sludge (WAS). To reduce sludge volumes, the WAS is centrifugally thickened to achieve a 90% volume reduction thereby producing thickened waste activated sludge (TWAS). The centrate from the WAS thickening process is returned to the aeration tanks. On average, 0.5 million litres of TWAS are produced daily with a total solids concentration of six percent, 70% of which are volatile (Gore and Storrie Ltd., 1995). The TWAS is mixed with RAW in a 60:40 volumetric ratio and then fed to the anaerobic digesters. Following anaerobic digestion, the solids are further treated by mechanical dewatering to reduce the volume of solids and ultimately produce BIOS. Typically the total solids content of the BIOS range from 30% to 35% (Gore and Storrie Ltd., 1995).

The high level of solids destruction in anaerobic digestion, about 60%, produces a very stable BIOS product. Further biodegradation of ROPEC BIOS was studied by Shaw *et al.* (1994) in aerobic composters and was found to be negligible due to the high stability of the BIOS. However, the RAW/TWAS sludge mixture originally fed to the anaerobic digesters in combination with the organic fraction of MSW (OFMSW) collected by the RMOC would be a richer source of organic materials and hence conducive to effective anaerobic biodegradation. Anaerobic co-digestion of a RAW/TWAS sewage sludge mixture and OFMSW would take advantage of the inherent microbial population in the sewage sludge to digest the OFMSW as well as the sludge itself. Ultimately, this would allow for resource recovery through the production of a nutrient-rich compost, and energy recovery through methane biogas production. As such, a feasibility study of such a process as a long term solids management alternative for ROPEC is warranted.

1.3 Objectives and Scope of Research

The overall purpose of this research was to evaluate the technical feasibility of anaerobic co-digestion of OFMSW and the RAW/TWAS sludge mixture currently fed to the anaerobic digesters at ROPEC. Specifically, the goals of the research were:

1. To determine the contributions of each of the individual components to the biodegradability of the mixture;
2. To identify an optimal ratio of OFMSW to RAW/TWAS;
3. To study the effect of alkaline, thermal and thermochemical pretreatments on the solubilization of feed solids (and hence anaerobic biodegradability).

The most commonly used HSAD technologies incorporate either batch or continuous reactor configurations and hence both were used for the scope of the research. The feasibility of the anaerobic co-digestion process was first evaluated using batch reactors. To identify an optimal ratio of OFMSW to sludge, several mixtures were prepared and the performance of the reactors evaluated based on biogas production profiles. Also, given the highly variable composition of OFMSW, a detailed study of the contributions to biodegradability of each of the individual mixture components was performed. To minimize this source of variability and allow evaluation of mixture components, a representative synthetic OFMSW was used in this work.

The next stage of the investigation involved characterization of the effects of key feed properties, namely total solids and particle size, on commonly used indicators of process performance and metabolic activity: biogas production, biogas methane content and removal of soluble chemical oxygen demand (COD), total solids (TS) and volatile solids (VS). Based on a central composite experimental design, these effects were quantified through the development of empirical models for the batch reactors.

The enhancement of solubilization of particulate organics is also of significant engineering consequence. Accordingly, the effects of alkaline, thermal and thermochemical pretreatments were investigated in the final stage of the research. The effectiveness of these pretreatments were studied in both batch and continuous reactors. For the experiments in the batch reactors, empirical models were also built, based on a central composite experimental design, to elucidate the effects of total solids content of the feed,

its particle size and alkaline dosage (where applicable) on the above-mentioned process performance indicators.

1.4 Layout of Thesis

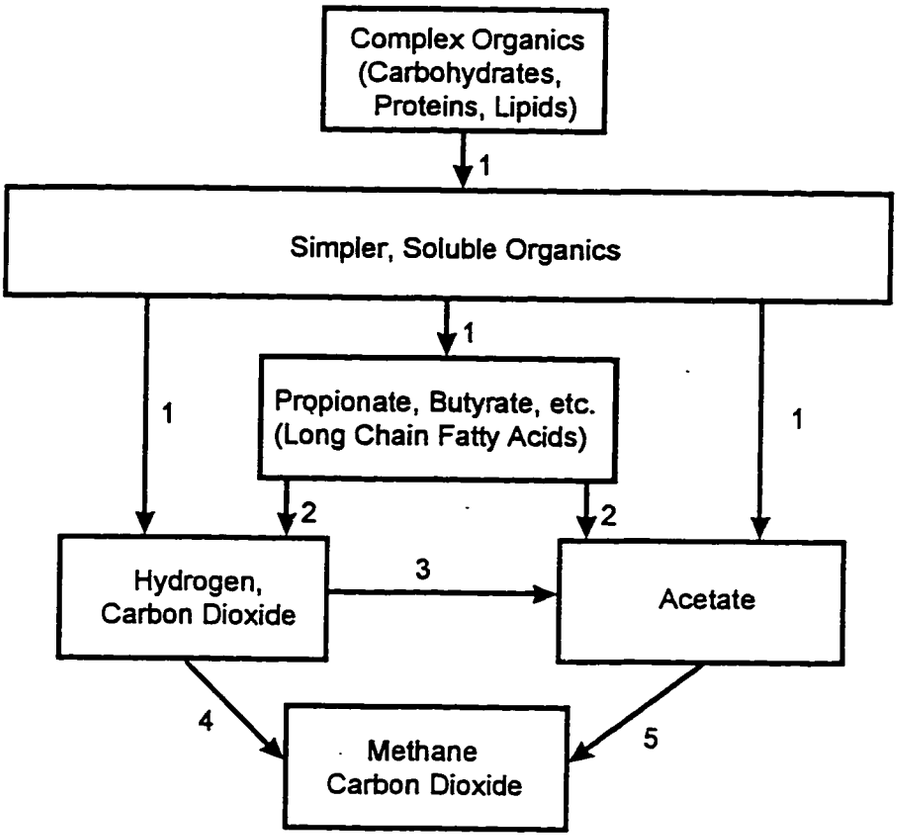
The thesis has been divided into two sections: the assessment of the technical feasibility of anaerobic co-digestion of OFMSW and ROPEC's RAW/TWAS sludge mixture and the study of the effects of the three pretreatments on anaerobic biodegradability. The basis for this approach is presented in Chapter 2. This chapter includes a critical analysis of the development and evolution of HSAD systems. Chapter 3 presents a technical feasibility study into the anaerobic co-digestion of OFMSW and the RAW/TWAS sludge mixture. It includes methodology and results for optimization of the ratio of OFMSW to the RAW/TWAS sludge mixture, an analysis of the contributions to biodegradability of each of the mixture components and the empirical models developed for the characterization of the effects of total solids content of the feed and its particle size. Chapter 4 includes the experimental method and a comprehensive analysis of the effects of the three pretreatments on the anaerobic co-digestion process. Results of both the continuous and batch reactors are included, as well as the empirical models that were developed. A summary of the findings from the entire work and recommendations for further research are presented in Chapter 5. References are provided in Chapter 6.

CHAPTER 2:
High-Solids Anaerobic Digestion: State of the Art

2.1 Anaerobic Digestion

The main purpose of treating solid waste is the realization of a net reduction in the quantity of solids requiring disposal. This reduction can be achieved anaerobically through the activity of a consortium of bacteria which, in the absence of oxygen, ultimately produce methane, carbon dioxide and trace amounts of hydrogen, carbon monoxide and hydrogen sulfide. Anaerobic biodegradation of organic substrates is a complex biological process involving a number of microbial populations linked by their individual substrate and product specificities. As shown in Figure 3, the overall mechanism involves symbiotic associations between these different groups of microorganisms. Based on this mechanism, studying anaerobic digestion processes involves the consideration of the contributions of each of these families of microorganisms.

The first group of microorganisms, referred to as fermentative or hydrolytic bacteria, is responsible for the hydrolysis of polymers such as carbohydrates, fats and proteins. The initial products of polymer hydrolysis are soluble sugars, amino acids, long-chain carboxylic acids and glycerol. The fermentative bacteria then break these intermediates down to short-chain carboxylic acids (volatile fatty acids), carbon dioxide and hydrogen (Parkin and Owen, 1986). A second group of bacteria, hydrogen-producing bacteria, then oxidize the fermentation products of the first group to acetate, carbon dioxide and hydrogen. This conversion is thermodynamically favourable only at very low hydrogen



Bacterial Groups:

1. Fermentative Bacteria
2. Hydrogen-Producing Acetogenic Bacteria
3. Hydrogen-Consuming Acetogenic Bacteria
4. Carbon Dioxide Reducing Methanogens
5. Acetoclastic Methanogens

Figure 3: Methane Formation in Anaerobic Digestion (Parkin *et al.*, 1986)

partial pressures, i.e. less than 10^{-4} atm (Parkin and Owen, 1986). Hence, these bacteria can only function in syntrophic association with a hydrogen scavenger such as hydrogen-consuming acetogens and methanogens. The latter are responsible for the ultimate production of methane.

Based on a review of current data, Pavlostathis and Gossett (1991) found that the type of waste being digested (i.e., soluble versus particulate) has a significant effect on microbial activity (Pavlostathis and Gossett, 1991). This is evident by the fact that different

investigators dealing with different wastes using the same experimental conditions have arrived at quite diverse process performances. To this end, this study focuses on establishing a better understanding of the role of the solids content on the behaviour of high-solids anaerobic digestion processes.

Maintenance of optimal levels of organic carbon and nitrogen is critical for cell synthesis and metabolism in the anaerobic digestion process. For example, a high carbon-to-nitrogen (C/N) ratio in the substrate can lead to potential problems due to nutrient (nitrogen) deficiency. This can be alleviated by removing a portion of the carbonaceous material, or by adding nitrogen. On the other hand, a low C/N could result in problems with ammonia toxicity which can be corrected by diluting the contents of the digester or by reducing the ammonia concentration in the liquid phase, or by adjusting the C/N ratio of the feedstock. A universally accepted ratio for optimal biological conversion of the organic fraction of MSW has not been established. Kayhanian and Tchobanoglous (1992) suggested that the C/N ratio should range from 25 to 30. This is in conflict with the common heuristics for anaerobic digestion processes which state that C/N ratios of 400:7 at high loading rates and as high as 1000:7 for low loading rates are optimal (Pohland, 1992). It is for this reason, that the first stage of this study involves the identification of an optimal feed mixture to be used throughout the research.

2.2 High-Solids Anaerobic Technologies

Anaerobic treatment of high-solids feedstocks, commonly referred to as dry anaerobic fermentation, has been shown to be highly effective in Europe (Cecchi *et al.*, 1988a). In these systems, total solids concentrations of 15% or greater define 'dry' conditions. Some of the advantages of dry anaerobic fermentation of solid wastes (as compared to conventional low-solids anaerobic systems) include smaller reactor volumes, reduced heating requirements to maintain optimal temperature conditions, and minimal chemical additions. HSAD systems range from uncontrolled landfills to large-scale engineered treatment plants.

2.2.1 Landfills

Uncontrolled spontaneous dry anaerobic fermentation occurs in landfills at solids contents ranging from 40% to as high as 70%. By controlling landfill conditions to promote anaerobic microbial activity, enhanced methane production results. The production of methane leads to a concomitant reduction in leachate strength reducing leachate treatment costs and the risk of groundwater contamination. A second advantage is the potential to reduce costs and even profit from biogas control through methane recovery.

While anaerobic digestion does occur in landfills, the rate and extent of degradation tend to be quite low. Low moisture content, mass transport limitations and poor penetration, diffusion and distribution of microorganisms throughout the substrate mass are potential causes of the poor performance associated with dry anaerobic fermentation in landfills (Ghosh and Lall, 1988). This has spawned considerable research directed towards the improvement of dry anaerobic digestion technology in controlled landfills.

Research on the decomposition of solid waste in landfills was first reported by Merz and Stone (1964) and since that time, numerous researchers have tried to enhance MSW methanogenesis by manipulation of the landfill ecosystem (Barlaz *et al.*, 1990). Merz and Stone (1964) showed that high moisture contents (26% to 52%) stimulated methane production in field-scale test cells filled with non-shredded MSW. Their work prompted deeper investigations into the underlying role of total solids content in anaerobic digestion systems.

Extending the work of Merz and Stone, Barlaz *et al.* (1987) concluded that higher moisture contents (55%) in shredded MSW lead to more rapid acid accumulation, hampering methane production. They also reported that methane production was not reliably observed at low moisture contents (35%). This is supported by the work of Halvadakis *et al.* (1988) which concluded that moisture content is one of the most critical variables controlling biogas production in field-scale test cells. Water serves many vital functions in the landfill: it is needed as a reactant in the first step of decomposition

(hydrolysis); it is the transport medium by which the nutrients reach bacteria and the enzymes reach substrates; it modifies the conformational structure of enzymes; it dissolves metabolites; and, it aids in exposing more of the substrate surface area to microbial attack (Noble *et al.*, 1988). In a heterogeneous system, such as a landfill, the limited opportunity for contact between microorganisms, their substrates and other necessary growth factors may limit biodegradation. As the MSW moisture content increases, the opportunity for contact is increased, which should enhance microbial activity. Halvadakis *et al.* (1988) indicated that in most landfills, the hydrolysis rate is slow enough that the rates of acid production and consumption (methane production) are balanced. However, if the hydrolysis rate is too high or if an excess of soluble substrates were available (due to excess moisture), fermentative bacteria would grow at a much faster rate than the methanogens. This would lead to an increased potential for acid accumulation and hence a pH decrease, ultimately inhibiting anaerobic microbial activity.

At higher MSW densities, that is, smaller particle sizes, each particle of MSW is in close contact with adjacent particles. Hence, for a given moisture content, increasing MSW densities would lead to greater water to MSW contact per unit volume of landfill. Since moisture contact stimulates the rate of hydrolysis and too high a rate of hydrolysis is inhibitory, then as the density increases the optimum moisture content would be expected to decrease. Accordingly, by shredding MSW, compaction is enhanced leading to higher densities and lower optimal moisture contents as compared to unprocessed MSW.

De Walle *et al.* (1978) working with shredded MSW at 44% moisture in 0.21 m^3 drums, suggested that a large particle size (25 cm) stimulated methane production relative to drums with MSW particle sizes of 2.5 cm and 12.5 cm. However, the average rate of methane production in the apparently stimulated drum was only 0.001 litres of methane per kg of MSW per day, which is well below the rates reported by others for enhanced methane production [Barlaz *et al.* (1987) and Halvadakis *et al.* (1988)].

Consistent with this work, Buivid and Wise (1981) found that MSW with a 25 cm to 35 cm particle size produced 32% more methane after 90 days than MSW with a 10 cm to 15 cm particle size with biogas production as high as 1.6 litres of methane per kg of MSW per day. Similarly, in a comparison of MSW with particle sizes of 10 cm to 15 cm and 1.25 cm to 2.5 cm, the MSW with the larger particle size produced 16 times more methane after 90 days. The MSW density for all four containers mentioned above was 406 kg/m^3 and the moisture content was 75%. Contrary to the works of both Buivid and Wise and De Walle *et al.*, Ham and Booker (1982) found that the onset of methane production as measured by biogas concentration, was stimulated by shredded MSW (7.5 cm sieve) relative to unshredded MSW in 204 m^3 field cells covered with soil.

The use of MSW with a reduced particle size relative to unprocessed MSW implies some mixing of the MSW associated with the size reduction operation. Well-mixed, shredded MSW permits greater contact between the key MSW constituents required for methane production: moisture, substrate and microorganisms. As discussed earlier, a smaller particle size could result in an increase in the rate of hydrolysis. Under the conditions used in the work of Ham and Booker, the smaller particle size did not stimulate the hydrolysis rate to the point where the MSW soured. While Ham and Booker concluded that a smaller particle size was stimulatory for the overall HSAD process, Buivid and Wise concluded the opposite. This may be explained by the range of particle sizes tested by Buivid and Wise (1 cm to 2 cm, 10 cm to 15 cm and 25 cm to 35 cm), as compared to Ham and Booker who studied processed MSW with a 7.5 cm particle size. Another difference between Ham and Booker. and Buivid and Wise is the higher moisture content and density used by Buivid and Wise relative to Ham and Booker, who relied on rainwater infiltration. The higher density should, by itself, stimulate hydrolysis as previously explained. Thus, further stimulation by a reduced particle size may have caused too rapid a rate of hydrolysis, leading to a build-up of acidic end-products and a lower pH.

Buivid and Wise (1981) also investigated various factors that could increase the rate of biogas production in a landfill. They reported that digested sewage effluent and recycle of leachate stimulated biogas production. They showed that leachate recycle was most stimulating when anaerobic seed sludge and calcium carbonate were initially added to the MSW. The anaerobic sludge served as an inoculum and the calcium carbonate was intended to buffer the system. The calcium carbonate/sludge addition comprised 10% of the MSW dry weight, although the proportion of calcium carbonate was not specified. The MSW had a high shredded paper content (77% as compared to 40% which is typical of Canadian MSW (RMOC, 1992)) which most likely decreased the initial soluble substrate concentration normally associated with food waste. This, in turn, decreased the potential for a rapid accumulation of volatile fatty acids (VFA) from acetogenesis and hence provided more favorable conditions for reactor start-up.

Anaerobically digested sewage sludge can serve as a seed of microorganisms as well as a source of nitrogen, phorphorus and other nutrients. Mata-Alvarez and Martinez-Viturtia (1986) observed rapid MSW decomposition in containers seeded with pig manure. In addition to pig manure, calcium carbonate was added to the MSW and leachate was recycled. Sludge addition without buffer addition did not stimulate methane production for Barlaz *et al.* (1987), rather, an accumulation of VFA was detected. It is difficult to draw a definitive conclusion since the effects of seed, nutrient addition and buffer are confounded in the study by Mata-Alvarez and Martinez-Viturtia (1986). To more clearly address this, the effects of buffer (sodium bicarbonate) addition on the anaerobic digestion of MSW were studied by Kasali *et al.* (1989). They found that the addition of 2.5% sodium bicarbonate (84 mg sodium bicarbonate per g dry weight of MSW) promoted both acidogenesis and methanogenesis. However, with 5% sodium bicarbonate addition, partial suppression of methanogenesis and acidogenesis was reported with cation toxicity the most likely cause.

The observations by Barlaz *et al.* (1987) suggest that fermentative bacteria in the sludge inoculum are better able to adapt to the MSW ecosystem than are the methanogens. The fermentative activity thereby results in an accumulation of VFA, which is not consumed by the methanogens, consequently decreasing the pH. The addition of a buffer holds the pH of the MSW ecosystem near neutral, allowing the methanogens of the sludge the opportunity to acclimate to the new environment.

2.2.2 Anaerobic Fermentors

Building on the findings from controlled/simulated landfill technologies, the dry anaerobic fermentation process can also be applied to anaerobic reactors. Under controlled bioreactor conditions, optimization of the process is facilitated through the acceleration of microbial reactions. However, the challenges of dry anaerobic digestion in controlled fermentors remain in the development of new reactor designs, capable of handling solid substrates under anaerobic conditions at a cost that remains economically justifiable. Pioneering works include that of Ducellier and Isman (1941) who treated manure in silos, and Jewell (1979) who proposed low-cost batch reactors to treat all organic wastes and crop residues on farms. Jewell's research indicated that significant methane production rates could be obtained at TS contents up to 40%. He also found that the rate of digestion was greatly influenced by the TS content of the feed.

In 1975, Waste Management Inc., was selected to develop a proof-of-concept design to provide full-scale production of methane biogas from solid waste at Pompano Beach, Florida. This process is known as Refuse Conversion to Methane (RefCOM). The primary technical objective of the RefCOM facility was to demonstrate long-term biogas production at rates sufficiently high to result in attractive economics. The facility consisted of a front-end which removed metals, glass, grit and plastics coupled with a semi-continuously-fed stirred-tank reactor. The reactor was operated thermophilically (55 °C) at 10% feed solids concentration and a 20-day to 30-day average SRT. Effluent was dewatered and the centrate was used to dilute the incoming MSW/sewage sludge feed

blend. The centrate was also recycled to conserve nutrients and recover energy. The facility experienced a variety of mechanical problems, some common to most resource recovery systems, some unique to anaerobic digestion. The former included failure in the size reduction and separation equipment, sub-optimal performance of air classification equipment, and entanglement of plastics and textiles in impellers. The most significant problems arose in pumping the slurry to the digester and in mixing the sewage sludge and solid waste (Walter, 1982). Although significant improvements were achieved during the experimental program, the process was not found to be economically attractive under the given operating conditions.

The most widely used dry anaerobic digestion process is the VALORGA (Valorization of Organics) process developed by Ducellier in the early eighties for the fermentation of municipal solid wastes (Cecchi *et al.*, 1988a). The process is comprised of four stages as shown in the schematic in Figure 4. The first step involves crushing and sorting where the organic fraction of MSW is separated from metals and then shredded. This step is followed by the mesophilic (35 °C) digestion section where the organic fraction of MSW

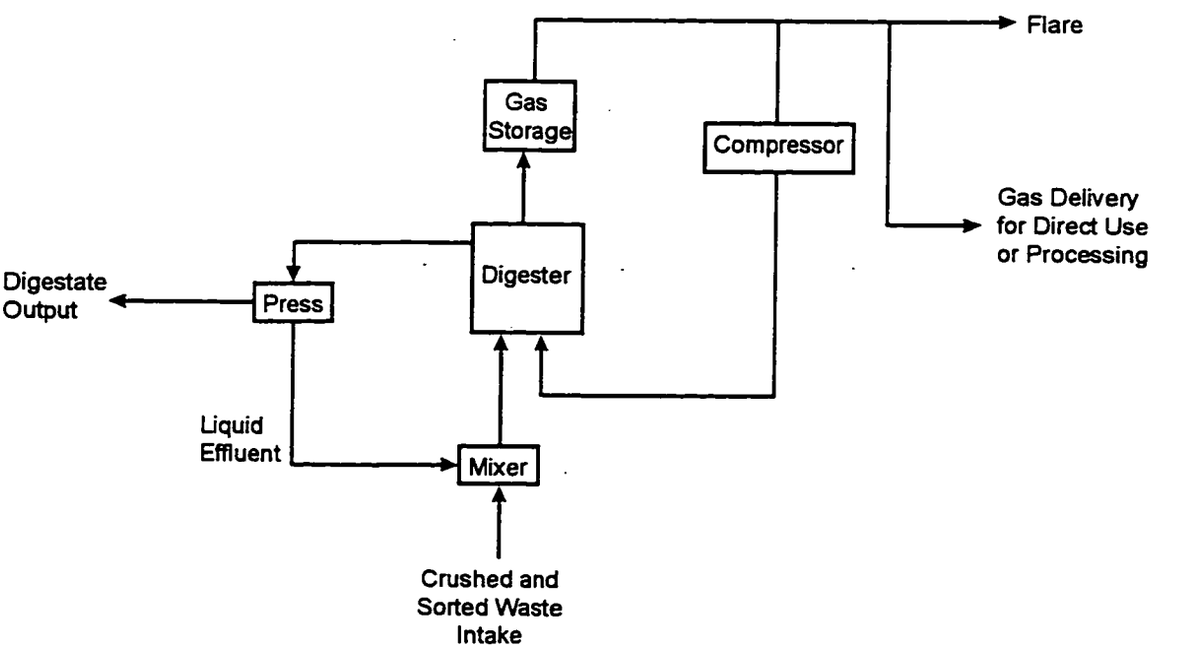


Figure 4: Schematic of the VALORGA Process (Zaoui, 1989)

(30% to 35% TS) is digested; the organic material remains in the digester for more than 15 days. In the following finishing and biogas utilization sections, the digested liquor is dewatered, the solids recovered as humus and the biogas stored and utilized. The digester is fed semi-continuously, such that after the reactor has been fed, the biogas production causes an increase in digester pressure which forces the digested residue through an outlet. Pulse biogas recycling (500 mbar) is used to mix the digester. This novel reactor configuration is a hybrid between a stirred and a plug-flow reactor. One of the main advantages of this process is the rapid stabilization of the refuse with a total treatment time of less than 20 days. The first VALORGA system set up in La Buisse, France was a 500 m^3 digester able to handle 30 tonnes of waste daily. The process has been operated with organic loading rates ranging from 13 to 20 kg of volatile solids per m^3 reactor per day resulting in biogas productions rates of 4 to 8 m^3 per m^3 reactor per day, 55% to 65% of which is methane (Zaoui, 1989). Throughout this thesis, organic loading rates and biogas production are reported per m^3 reactor, that is, per unit of working volume of the respective reactor.

Another widely used European system is the DRANCO (Dry Anaerobic Composting of Organic Wastes) process developed by De Baere *et al.* (1985) for the biodegradable fraction of household refuse. The process is shown in Figure 5 and can be integrated into existing recycling plants, or can be a stand-alone complete treatment system with the addition of the DRANCO front-end removal system for metals, glass, plastic and recoverable paper. The anaerobic treatment takes place over a period of 12 days to 18 days, followed by a post-fermentor (aerobic) with a retention time of 2 days to 3 days. Both steps take place at a thermophilic operating temperature of 55 °C and a TS concentration of 30% to 35%. The feed moves downward through the reactor in a plugflow manner without mechanical mixing. For organic loading rates of 8 to 21 kg VS per m^3 reactor per day, 2 to 5 m^3 of methane are produced per m^3 reactor per day. The biogas produced typically contains 55% to 65% methane. The digested residue is dewatered to a solids content of 45% to 55% TS in order to produce the water needed to adjust the solids content of the raw incoming substrate. The dewatered cake on the other

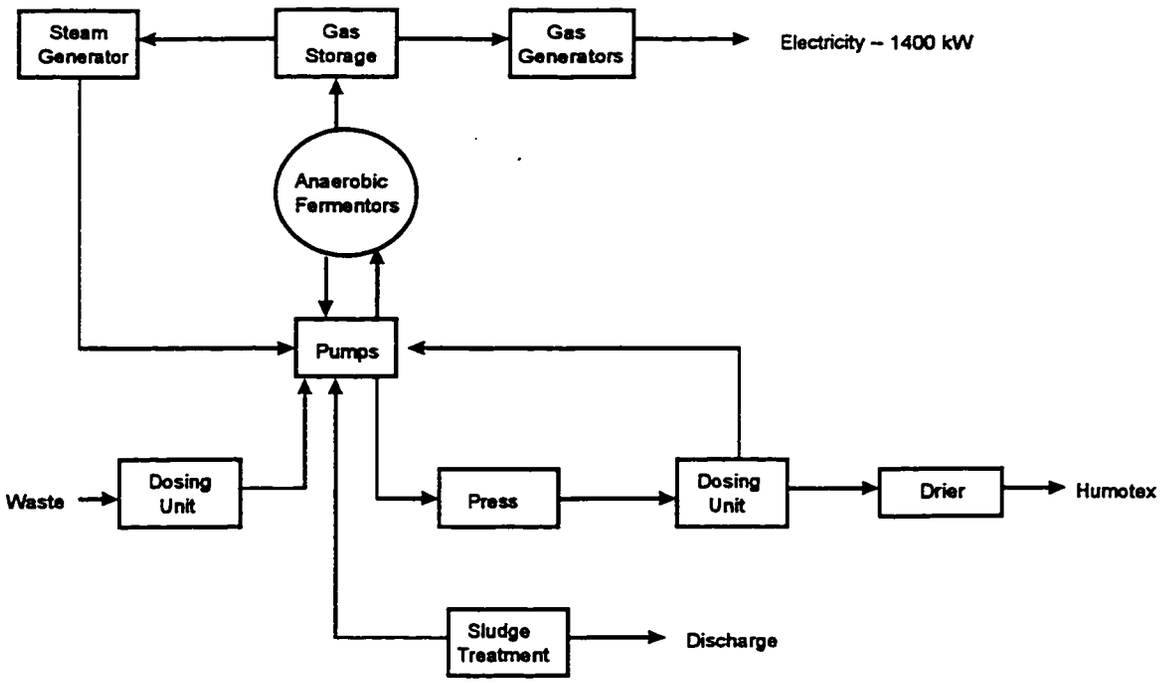


Figure 5: Flowsheet of the DRANCO Process (De Baere *et al.*, 1985)

hand is broken and transported into a drier. The energy needed to dry the cake comes from the exhaust gases from the engines running on biogas. The dry end-product (65% to 70% TS) can be shredded further and sieved resulting in a highly stabilized humus-like product called humotex. Each tonne of feed to the plant results in 350 kg of humotex, 130 kg of biogas (100 m^3), 320 kg of wastewater and 200 kg of other products (100 kg non-recyclable and 100 kg recyclable) (De Baere *et al.*, 1985). This technology has been proven effective on household MSW, thickened aerobic sludges and manures, papermill sludge, restaurant wastes, cannery wastes and agricultural surpluses. The largest plant, in Salzbourg, Austria, has been treating 20,000 tonnes of organic MSW a year since 1994.

Fruit, yard and vegetable wastes release considerable amounts of water during digestion and hence tend to float in downflow systems such as the DRANCO process. To treat these wastes, the KOMPOGAS system shown in Figure 6 was developed in Switzerland by Wellinger *et al.* (1993). It consists of a stirred-tank reactor with an intermittently operated stirrer able to handle feeds ranging from 18% to 40% TS. The process is

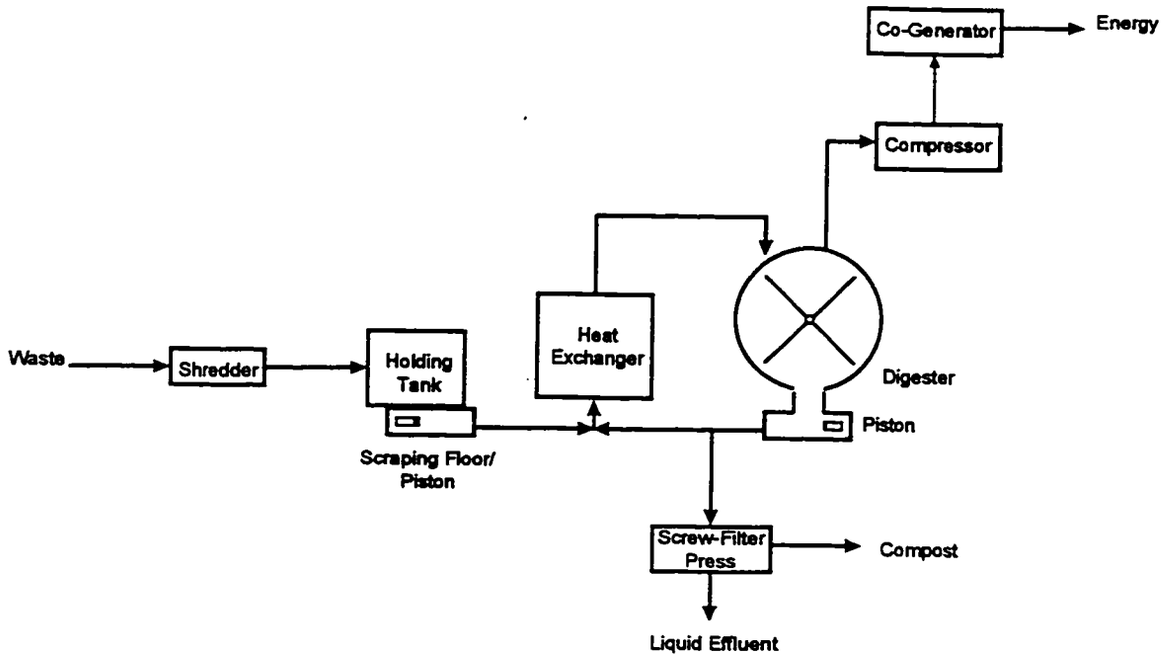


Figure 6: Flowsheet of the KOMPOGAS System

initiated with a shredder to reduce the size of the material to less than 6 cm in diameter. From a holding tank, fresh material is fed to the digester after passing through an external heat exchanger to attain a temperature of 55 °C. The digester is initially seeded with sewage sludge. Digested material is removed by a second piston into a screw filter press where it is separated into humus-like material of about 45% to 50% TS (part of which is recycled as inoculum) and a centrate liquor of about 15% to 22% TS. The reactor operating at a 40-day SRT produces 2.7 m^3 of biogas per m^3 reactor per day and at an SRT of 13 days, 3.7 m^3 of biogas are produced per m^3 reactor per day. The methane content of the biogas ranges from 60% to 65%. A full-scale installation has been in operation since 1992 with a digester volume of 200 m^3. It was designed to treat 3000 tonnes of fruit, yard and vegetable waste a year.

A novel reactor was designed by Klein and Rump (1987) for the treatment of the organic fraction of municipal solid waste as shown in Figure 7. A 1200 litre demonstration plant was constructed using this reactor in Tanusstein, Germany. Called, the KWU-Fresenius

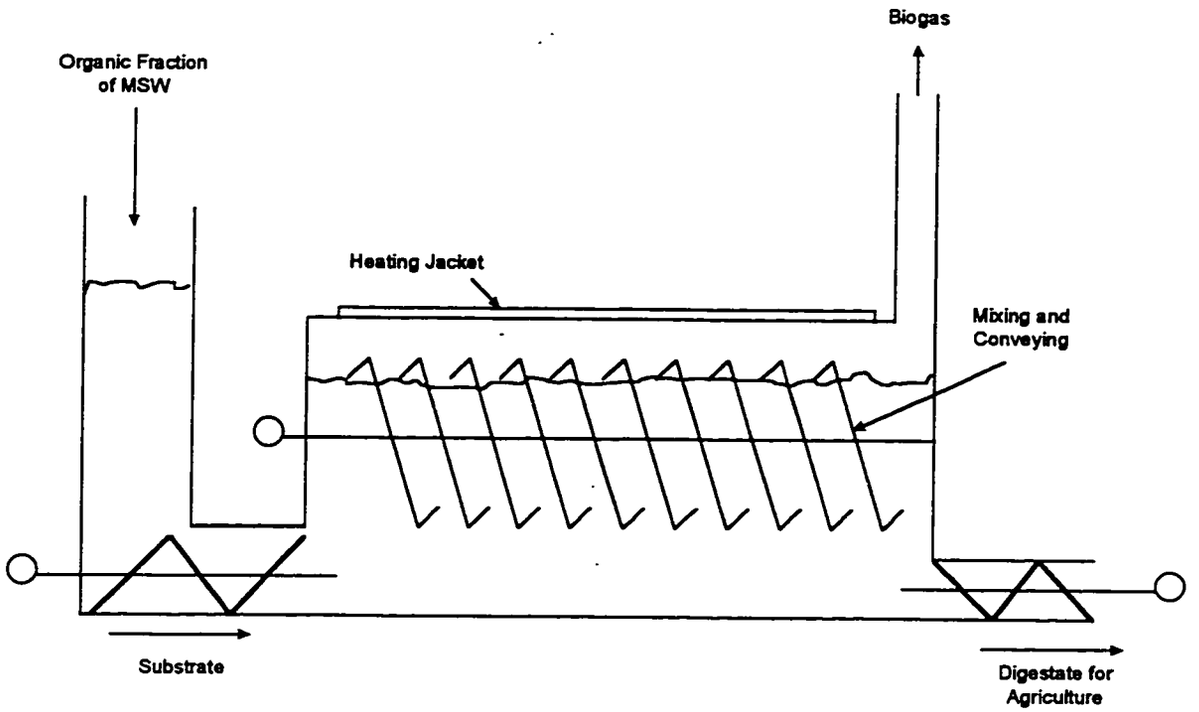


Figure 7: Reactor Configuration for KWU-Fresenius Process (Klein and Rump, 1987)

process, the system generates two to three times the amount of energy (biogas) than required to operate the process. It operates with a feed throughput of 5 tonnes per day at a total solids concentration as high as 40%. At a hydraulic retention time of 10 days, 500 litres of biogas are produced per kilogram of organic dry matter. The methane content of the biogas is 55 to 65% which results in an energy content of 10.8 GJ per 500 m^3 biogas. Assuming that the biogas methane concentration is 55%, that 70% of the TS are VS, and that the density of the MSW is 0.3 kg TS per dm^3, the methane production rate can be estimated to be 5.8 m^3 per m^3 of reactor per day. With this reactor, they have found that no sewage sludge is required to be added because the digestion is sufficiently effective with the natural moisture content of the organic fraction of MSW.

Another process for the dry digestion of the organic fraction of MSW is the BIOCEL process developed in the Netherlands by ten Brummeler *et al.* (1992). Tested at both laboratory and pilot-scale (5 m^3), the BIOCEL process is based on batch-wise digestion of solid organic waste materials in static piles. During the process, a mixture of the MSW

and a methanogenic seed are digested mesophilically (37 °C) at a total solids concentration of 35% and SRT of 30 days. The biogas can easily be extracted for energy production, and the residue is a well-stabilized organic end-product. The advantage of this process is that no mixing is involved, significantly reducing operating costs for the reactor. However, due to the batch-wise operation and the absence of mixing, there is an initial sour period where there is a build-up of VFAs and a concomitant pH drop. Minimization of this sour period or alternatively increasing the SRT will increase the degradation rate of the volatile solids. The maximum loading rate that could be applied was 7 kg of VS per m^3 reactor per day resulting in 70 m^3 of biogas per tonne of organic waste fed to the reactor. This corresponds to a biogas production rate of 1.1 m^3 of methane per m^3 of reactor per day assuming 70% of the TS are VS. In this system, leachate recycle is essential to increase the rate of degradation. Leachate recycling provides organisms, moisture and nutrients required for rapid conversion of MSW and removal of inhibitory fermentation products during start-up.

Chynoweth *et al.* (1992) introduced a novel sequential batch anaerobic composting (SEBAC) process for the treatment of high-solids wastes that also employs leachate management. The design goals of the process were to minimize water addition, minimize solids handling, eliminate mixing, maximize conversion kinetics, and prevent development of instability during start-up and operation. The system of 0.7 m^3 reactors is operated thermophilically (55 °C) at total residence times ranging from 21 days to 42 days. The system is shown in Figure 8. In stage 1, the organic fraction of MSW (mainly paper, yard and food waste) is coarsely shredded (to about 10 cm), placed into the reactor, moistened and inoculated by recycling leachate from stage 3. In stage 2, the fermentation is active and balanced and thus operated in the batch mode. Stage 3 allows for completed conversion of degradable particulates and also serves as an inoculum for start-up of stage 1. Stage 3 also converts acids pumped out of stage 1 via leachate recycle. They found that during the initial part of stage 1, VFAs accumulated to over 3000 mg/L and methane production was minimal. However, methane yields and methane biogas content rapidly increased after 5 days, indicating development of a balanced methane fermentation. Most

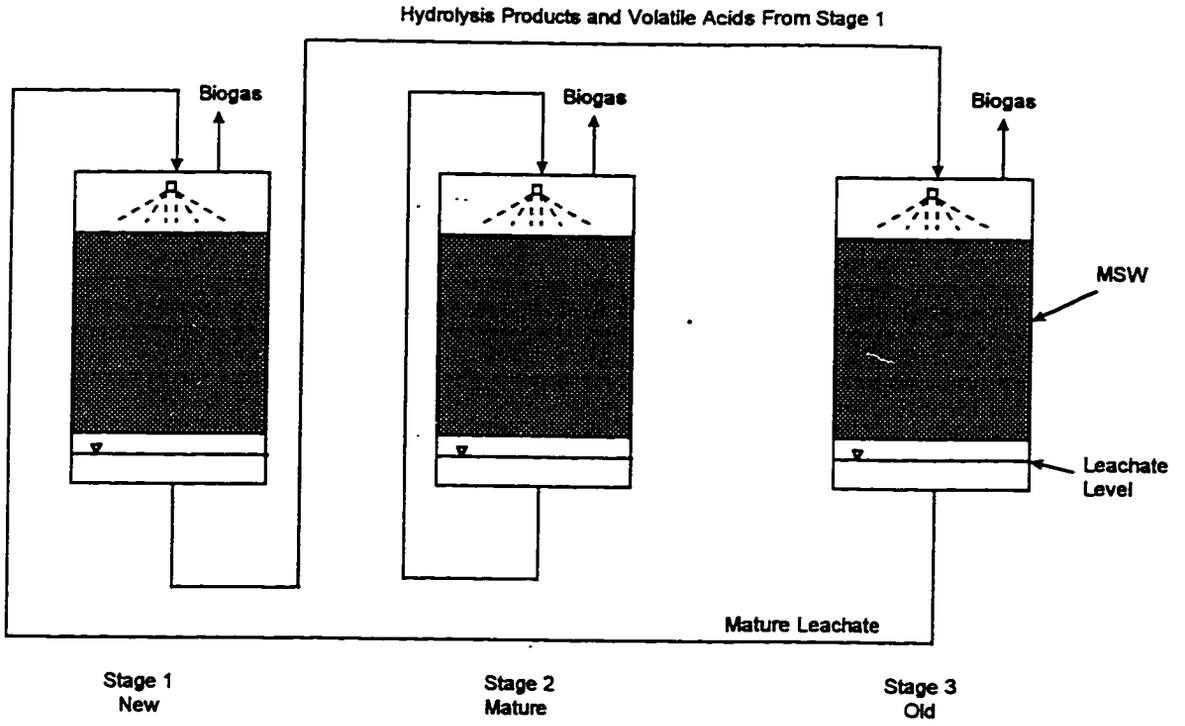


Figure 8: Schematic of Sequential Batch Anaerobic Composting (Chynoweth *et al.*, 1992)

of the methane is produced in stage 1. The rate of methane production levels off in stage 2. Methane in stage 3 is produced in part from residual conversion of MSW and in part from VFAs carried over from another reactor in the process of start-up. Conversion and associated methane yields were found to be slightly lower for the 21-day runs compared to 42-day runs. Methane yields ranged from 0.13 to 0.22 m^3 per kg VS with VS reductions ranging from 20 to 50% corresponding to methane production rates of 1.3 to 2.2 m^3 per m^3 reactor per day.

Two-phase digestion processes were first promoted by Ghosh *et al.* (1975). In this process, the biological liquefaction and acidification of the MSW is accomplished in a first reactor, while methanogenesis takes place in a second reactor. Two-phase processes are advocated for wastewaters containing complex wastes, because an acidification phase prior to the methane reactor allows the complex molecules to be broken down first, in order to prepare an optimal substrate for the methanogens. The work of Ghosh *et al.* (1975) involved two completely mixed reactors in series for the combined digestion of

sewage sludge and MSW. Based on their findings, the conversion of this feedstock to volatile acids, and volatile acids to methane could best be obtained by operating a first mesophilic reactor at a retention time of 3 days and the second mesophilic methanogenic reactor at a retention time of 5 days. Unfortunately, no controls were run so advantages over conventional single phase digestion could not be verified.

Augenstein *et al.* (1977) proposed a packed-bed reactor for the first phase and a non-biological activated carbon system as the second phase. Water was pumped continuously through the solids bed and a sieve separated the solids from the liquid. The organic-rich wastewater was pumped to the activated carbon, where the toxic substances were removed, and recycled to the first phase. When digestion was completed, the undigested material was partially dewatered via a pump. The partially dewatered material was then removed and replaced with fresh substrate. At a retention time of 20 days, experiments were performed with a mixture of newspaper and sewage sludge and local municipal solid waste and sewage sludge. The total solids content in the reactor reached 12% to 15% and a methane production rate of 0.5 m^3 per m^3 reactor per day was observed. Output biogas ranged from 10% to 65% methane. The total methane yield was 0.17 m^3 per kg of substrate added as compared to 0.11 for the same mixture fed to a conventional stirred tank reactor.

Ishida *et al.* (1979) added a thermochemical pretreatment of the mixture of sewage sludge and MSW prior to two-phase thermophilic digestion. The pretreatment of the feed slurry (13% to 15% solids) consisted of elevation of the pH to 9.8 at 60 °C for 3 hours. After 24 hours, the pretreated substrate produced 3 times as much volatile fatty acids per kilogram of volatile solids (VS) as compared to the untreated substrate. After pretreatment, the two-phase digestion consists of a hydrolysis phase at 60 °C and a pH of 6.0 for 36 to 48 hours, and a methanogenesis phase at 60 °C and a pH of 7.5 to 8.2 for a time of 108 to 144 hours. The treatment time for the total process was from 6 to 8 days. Biogas production rates of 3.2 m^3 of methane per m^3 reactor per day at a loading rate of 9.4 kilograms of VS per m^3 reactor per day were obtained for the system consisting of a 1

m^3 pretreatment reactor, 0.5 m^3 liquefaction reactor and a 1.5 m^3 gasification reactor. The feed had volatile solids contents ranging from 8% to 16%.

Kayhanian and Tchobanoglous (1993) coupled high-solids anaerobic digestion with aerobic composting in their two-stage process shown in Figure 9. The feed to the reactor consisted of paper, yard and food waste mixed in proportions to simulate the composition of the organic fraction of MSW in the United States. The process involves 1) the high solids (22% to 30% TS) anaerobic digestion of the organic fraction of MSW to produce a biogas composed principally of methane and carbon dioxide, and 2) the aerobic composting of the anaerobically digested solids to increase the solids content from 25% to 65% TS. Essentially all of the biodegradable solids in the feedstock can be removed, and biogas production levels of up to 6 m^3 per m^3 of reactor per day were achieved. The anaerobic reactor was operated under thermophilic conditions (55 °C) with solids retention times ranging from 15 days to 30 days. With an organic loading rate of 23.8 kg of VS per m^3 of reactor per day, the process stability and biogas production decreased slightly when the retention time was reduced to 15 days. Methane concentrations ranged from 48% to 60%. Tchobanoglous *et al.* (1994) proposed applying this high-solids anaerobic digestion/aerobic composting process to the co-treatment of the organic fraction of MSW and wastewater sludges. Preliminary economic analyses indicate the process to be cost-effective, because most of the physical and chemical processes associated with the current practice of sludge dewatering and management would be eliminated. Of particular

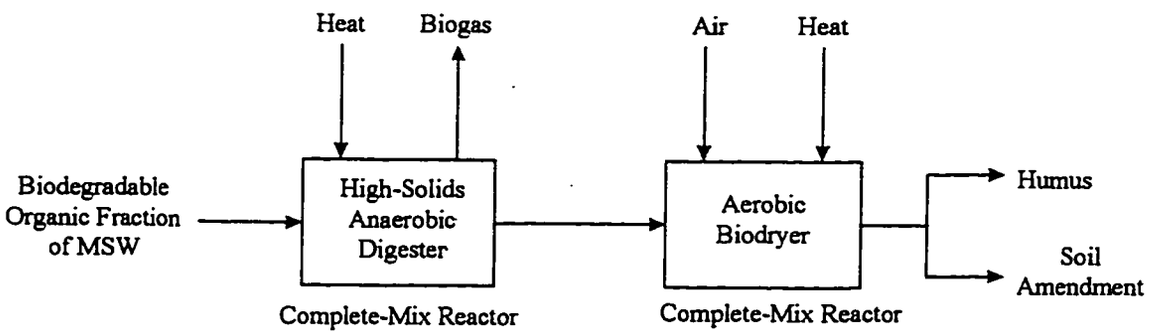


Figure 9: Flow Diagram for the Two-Stage High-Solids Anaerobic Composting Process (Kayhanian and Tchobanoglous, 1993)

interest in their research was the conclusion that particle size of the feed has a direct effect on the performance of the digester - smaller particle sizes increase the rate of substrate utilization. However, the particle sizes studied were not reported.

The Biotechnische Abfallverwertung GmbH company developed a three phase digestion process called the BTA process. Currently there are four BTA plants operating in Germany and Denmark and the first plant in North America is being constructed in Newmarket, Ontario by Canada Composting Inc. (Kübler and Schertler, 1994). The entire processing cycle for incoming waste, as shown in Figure 10, is just over two days. Inorganic elements are removed during a pretreatment phase and the remaining organic suspension is decomposed into compost and biogas and water which is purified on-site. The compost can be applied immediately or may be cured for an additional period of three to eight days, depending on the intended use of the material. The BTA compost is well within the Canadian guidelines for unrestricted usage. The facility in Newmarket will accept 120,000 tonnes of MSW a year and use approximately 30% of the electricity

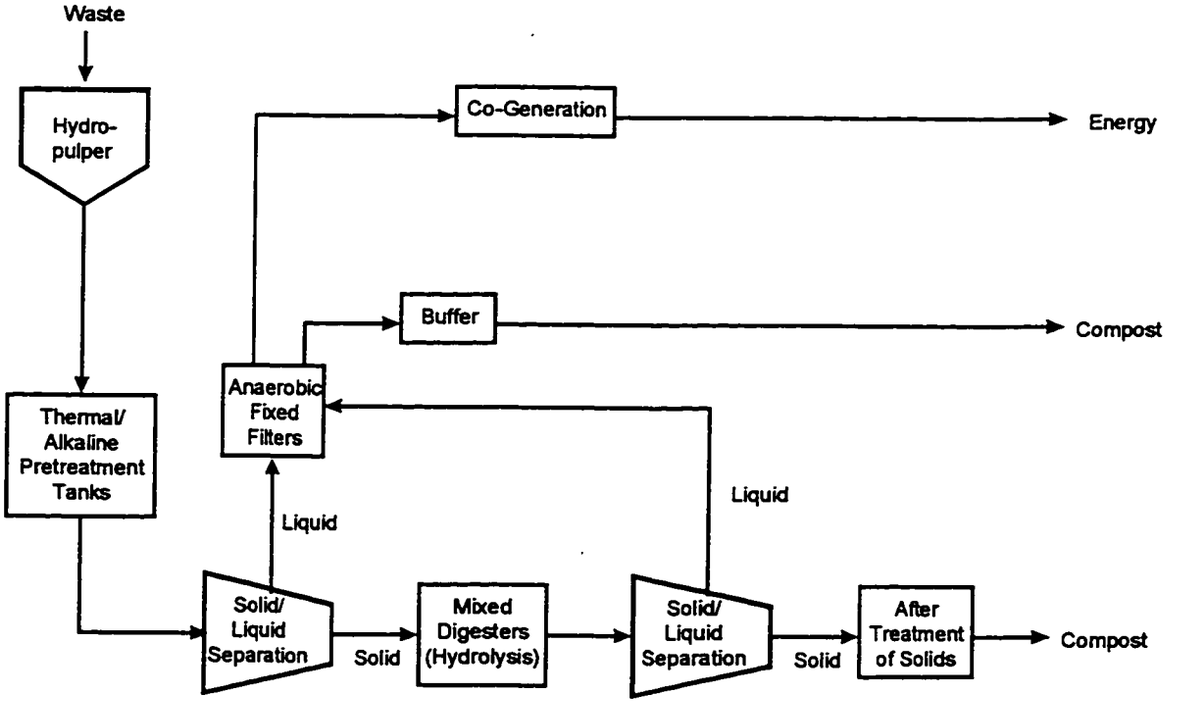


Figure 10: Flowsheet of the BTA Process (Blanchard, 1993)

produced to achieve energy self-sufficiency, with the remainder available for external consumption (Blanchard, 1993). The process is divided into two separate stages; pretreatment of waste stage and biological stage. The concept of the pretreatment stage is to prepare the organic fraction of the input waste stream for the biological decomposition stage. The pretreatment separates the organic from the inorganic material followed by a thermochemical pretreatment of the organic fraction. The dissolved materials are then separated from the solids centrifugally. The liquid is fed to an anaerobic filter operated mesophilically (35 °C) where specific cultures of anaerobes digest the organic-rich water and produce biogas. This biogas has been reported to consist of 75% to 80% methane. The solids move into a mesophilic (35 °C) continuously stirred hydrolysis reactor where another group of anaerobes decompose the organic solids into a compost material. Once the solid material has reached the required time in the hydrolyser (48 hours), the solids are removed and placed in an aerated static pile for approximately three days of final curing. For a loading rate of 20 kg COD per m^3 per day (equivalent to 28.6 kg VS per m^3 per day assuming 70% of COD correlates to VS (Tchobanoglous and Burton, 1991)), methane production was 9.4 m^3 per m^3 per day (Kübler and Schertler, 1994). Since the process is operated batchwise and the flow of pulp produced changes significantly, the pulp is pumped into a buffer tank. The pH inside the hydrolysis reactor is controlled by the recirculation rate of hydrolysis contents to solid/liquid separation. The TS-contents in the hydrolysis reactor is adjusted by the ratio of recycled methane reactor effluent to liquid phase produced by solid/liquid separation. The relatively high reported biogas production from the BTA process is most likely due to particulate solubilization resulting from the aggressive feed pretreatments.

Table 1 summarizes the operating conditions and performance of the main HSAD systems. Overall, findings from both lab- and full-scale installations indicate that further investigation of the anaerobic co-digestion of MSW and sewage sludge is warranted. In particular, research in both landfill and full-scale anaerobic fermentors indicate that the particle size of the feed has a definite effect on anaerobic digestion performance. However, there are several inconsistencies in the literature as to whether particle size

Table 1: Summary of Operating Conditions and Performance of HSAD Technologies

Process	Feed	SRT (days)	Organic Loading Rate (kg VS/m^3 reactor•day)	Methane Production Rate (m^3/m^3 reactor•day)
VALORGA	MSW	15	13 to 20	2.4 to 4.8
DRANCO	MSW	12 to 18	8 to 21	2 to 5
KOMPOGAS	Fruit, Yard & Vegetable Waste	13	11	2.2
		40	7	1.6
KWU-Fresenius	MSW	10	5	5.8[1]
BIOCEL	MSW/Sewage Sludge	30	7	1.1[2]
SEBAC	MSW	21	6.4	1.3[1]
		42	3.2	2.2[1]
Anaerobic Composting	MSW	15 to 30	23.8	6.0
BTA	MSW	2	27	9.4

[1] Calculated assuming 55% methane in the biogas, 70% of TS are VS and the feed has a density of 0.3 kg TS/dm^3

[2] Calculated assuming 70% of TS are VS

reduction is beneficial (Ham and Booker (1982) and Buivid and Wise (1981)). Most of the large-scale facilities reported in the literature are initiated with a size reduction unit and hence an evaluation of its effectiveness should be performed to form the basis for a cost-benefit analysis of these units. Also, the work of Halvadakis *et al.* (1988) highlighted scale studies were operated with TS concentrations ranging from 12 % to 40%, however, the impact of feed TS on the performance of the anaerobic co-digestion process was not clearly identified. This becomes critical in reactor design for the commonly used intermittently-mixed tank reactors. These reactors, as well as batch reactors are most widely used since they bypass many of the problems with mixing introduced in plug-flow and continuous-stirred tank reactors. Hence, to effectively apply the findings to current full-scale technologies, batch and intermittently-mixed tank reactors should be used in further research studies on the effects of TS of the feed and its particle size.

The clearly enhanced methane production rate of the BTA process as reported in Table 1 indicates the potential increase in anaerobic biodegradability through the integration of thermal and alkaline pretreatments. Since limited research has been reported on the effectiveness of each of these pretreatments on OFMSW/sewage sludge mixtures, a detailed study is required to identify optimal operating conditions and the extent of process improvement.

CHAPTER 3:
Technical Feasibility of Anaerobic Co-Digestion of Sewage Sludge and Municipal Solid Waste

Traditionally, the treatment and disposal of MSW and of sewage sludge has involved two distinct waste management strategies. Conventional MSW management has focused primarily on disposal with little or no emphasis on preprocessing or resource recovery alternatives. Sewage sludge management, in contrast, has involved extensive sludge treatment (stabilization, dewatering) and beneficial-use practices (land application, sludge composting) before landfill disposal. Increased waste disposal costs and recent environmental concerns have prompted a re-evaluation of these conventional management approaches. These concerns coupled with a renewed interest in resource recovery have fueled the search for innovative waste management practices.

The stabilization of sewage sludge produced from a typical municipal wastewater treatment plant is normally achieved by conventional low-solids (four to seven percent TS) anaerobic digestion. Major benefits associated with the anaerobic digestion of sludge include: reduction of solids for ultimate disposal, improved sludge dewaterability, generation of methane for in-plant use and reduction of pathogenic organisms (Kayhanian and Rich, 1996). This renders the anaerobic co-digestion of MSW with sewage sludge especially attractive. This results from the large amount of sewage sludge produced in wastewater treatment plants and the large number of existing anaerobic digesters to stabilize them. Use of the existing infrastructure to perform the treatment can significantly reduce capital and operating costs. Other benefits of co-digestion include dilution of

potential toxic compounds coming from the co-substrates, improved balance of nutrients, synergistic effects of microorganisms, increased organic load of biodegradable matter, and better yields per unit of digester volume (Kayhanian and Rich, 1996).

3.1 Literature survey

At present, the anaerobic co-digestion process appears to be a promising technology for sewage sludge and MSW management. Early studies of the co-digestion of MSW and sewage sludge were carried out by Klein (1972), Diaz *et al.* (1974) and Diaz and Trezek (1977). These studies concluded that source-separated MSW could be anaerobically digested without nutrient supplementation up to organic loading rates of 2.6 kg of VS per m^3 of reactor per day. They also reported that, with nutrient addition, biogas production could be enhanced and a more stable process achieved.

The BIOMET process developed by Szikriszt *et al.* (1988) was used to treat a mixture of sorted MSW and sewage sludge pretreated in a mechanical hydropulper. The mixed pretreated material was digested mesophilically (38 °C) at an inlet TS of 8%. At organic loading rates of 1.6 kg of VS per m^3 reactor per day (27-day HRT) and of 2.6 kg of VS per m^3 reactor per day (19-day HRT), the biogas yields were 0.82 m^3 per m^3 reactor per day (57% methane) and 1.12 m^3 per m^3 reactor per day (53% methane) respectively. These experiments confirmed that a considerable amount (as high as 50%) of the organic fraction of MSW could be converted to biogas in anaerobic digesters. They also found that higher conversion could be obtained at a 27-day HRT compared to a 19-day HRT; the VS reduction was 17% higher and the biogas production 26% higher at the 27-day HRT. They concluded that the 15-day to 20-day HRT heuristic for anaerobic digesters should be lengthened to 25 days to 30 days to take full advantage of the anaerobic digestion process.

The co-digestion process was also studied by Cecchi *et al.* (1988b). They varied the ratio of source-separated OFMSW and primary sewage sludge. They operated a 3500 litre pilot plant mesophilically (37 °C) treating a feed of 6% TS at organic loading rates from

1.7 to 3.9 kg VS per m^3 reactor per day resulting in biogas productions of 1.5 to 7.8 m^3 per m^3 reactor per day. They found that biological activity and specific biogas production, increased linearly with the percent of MSW in the feed. Their study included the full range of MSW/sewage sludge mixtures, from 0% to 100% sewage sludge on a percent TS basis. Contrary to the findings of Szikriszt *et al.* (1988) the optimal HRT was found to be 14 days to 15 days.

Based on this work, Cecchi *et al.* (1990) proposed the Step-Diffusional kinetic model. The model proposes that there are three utilization rates for the process, each one linked to the degradation of a specific group of compounds: the first composed of acetate and VFAs with less than three carbon atoms, the second consisting of VFAs with greater than three carbons and the third, complex organic matter. This kinetic model was used to fit the data from their study but has not been validated with other studies or feed substrates.

These co-digestion studies were low-solids processes, with total solids contents ranging from 4% to 8%. Only recently has the HSAD process been utilized for the co-digestion of MSW and sewage sludge (Kayhanian and Tchobanoglous, 1993; Poggi-Varaldo and Oleszkiewicz, 1992). With higher TS levels in the reactors, the organic loading rates to the reactors can be increased by reducing feed dilution rates. Consequently, this leads to reduced reactor volumes ultimately resulting in reduced capital and operating costs. In HSAD studies, the sludge is typically used to provide sufficient nutrients for microbial growth and metabolism.

The Anaerobic Composting Process combines an HSAD process with a second stage aerobic biodrying process to recover energy and a humus-like material from MSW. Studies on this process indicate that stable digestion was achieved using organic loading rates of 23.8 to 29.3 kilograms of VS per m^3 reactor per day, approximately ten times those reported in the low-solids studies of Szikriszt *et al.* (1988) and Cecchi *et al.* (1988a). Under these conditions, methane biogas production rates were as high as 6.0 m^3 per m^3 reactor per day. Through this process, approximately 65% more substrate volume

reduction was achieved compared to a well compacted landfill (Kayhanian and Tchobanoglous, 1993). Consistent with the findings of Cecchi *et al.* (1988b), Kayhanian and Rich (1996) reported an optimal mixture with 80% MSW (VS basis) using the Anaerobic Composting Process. In an earlier study, Kayhanian and Hardy (1994) also found that the rates of biogas and methane production were inversely proportional to the average feedstock particle diameter.

The feasibility of anaerobic co-digestion of WAS and a simulated organic fraction of typical North American MSW was examined by Poggi-Varaldo and Oleszkiewicz (1992). In total, 16 experiments were run at four levels of TS (25% to 40%), two retention times (15 days and 21 days), two temperature regimes (39 °C and 53 °C) and two ratios of OFMSW to sludge (1:1 and 2:1 by weight) using a fractional factorial experimental design ($4 \times 2^{3-1}$ factorial). In this design, the effects of retention time, temperature and ratio of OFMSW to sewage sludge are each confounded with two-factor interactions of the other two variables. For example, a conclusion that the retention time has a significant effect could not be justified since the effect may be the result of the temperature-OFMSW/sewage sludge interaction. This problem with interpretation exists for all three factors.

In their study, Poggi-Varaldo and Oleszkiewicz (1992) only qualitatively reported the effects of each of the experimental factors on the response variables studied as well as the optimal operating conditions. Also, no empirical models (based on the experimental design used) were reported to characterize the behaviour over the entire operating region. They reported that reactors operated at 30% TS and 21 days gave the best organic removal efficiencies (up to 44% and 53% for mesophilic and thermophilic regimes respectively) and highest biogas productivities (up to 6.0 m^3 per m^3 reactor per day). The methane concentration in the biogas ranged between 55% and 60%. Thermophilic reactors generated 15% to 30% more biogas than the corresponding mesophilic digesters. However, instability and failure of some thermophilic reactors at 15 day retention times were observed. Contrary to the findings of both Cecchi *et al.* (1988b) and Kayhanian and

Rich (1996), Poggi-Varaldo and Oleszkiewicz (1992) concluded that decreasing the ratio of OFMSW to WAS (the 1:1 mixture) had a stimulating effect on the process as indicated by improved biogas production and TS removal efficiency. The differences in process performance are most likely due to inherent compositional differences in the sludges and local MSW from the different countries in which these studies were performed.

The experimental design and results presented in this chapter build on the preliminary findings of Poggi-Varaldo and Oleszkiewicz (1992). Their work established a foundation for further research on anaerobic co-digestion of Canadian OFMSW and sewage sludge. To develop a better understanding of the impact of key experimental factors on anaerobic digestion process performance, empirical models need to be built to more quantitatively characterize process behaviour over the typical operating region. Important experimental factors for the anaerobic co-digestion process include feed particle size (Kayhanian and Hardy, 1994) and TS concentration (Poggi-Varaldo and Oleszkiewicz, 1992). In both cases, there are currently no models available.

Given the potential for reduced capital and operating costs for HSAD (as opposed to low solids processes), the process should be further analyzed. Several aspects of the process have yet to be studied. Cecchi *et al.* (1988b) studied the effect of modifying the ratio of OFMSW to sludge over a wide operating range. However, the comparable study on Canadian MSW and sludge (Poggi-Varaldo and Oleszkiewicz, 1992) was performed at only two points and the confounding of the factors within their experimental design limits the interpretation of their results. Further experimentation is required to more accurately identify an optimal Canadian OFMSW/sludge mixture. To better understand differences between European and Canadian wastes, a component-by-component analysis should also be performed.

Consequently, the overall objective of this study was to evaluate the feasibility of the HSAD process for the joint management of ROPEC's wastewater-treatment sludges (RAW and TWAS) and OFMSW. The specific objectives of this thesis were:

1. To evaluate the performance of the high-solids anaerobic co-digestion process using different ratios of OFMSW and sewage sludge (RAW/TWAS);
2. To evaluate the contribution to biodegradability of each of the components of the OFMSW and RAW/TWAS sludge mixture;
3. To quantify the effects of total solids content and particle size of the feed mixture on biogas production, biogas methane concentration, and reduction of soluble chemical oxygen demand, volatile and total solids.

To obtain better control of feed compositions, a simulated OFMSW, based on reported compositions of MSW from the Ottawa-Carleton region, was used throughout the study.

3.2 Materials and Methods

3.2.1 Analytical Methods

Measurements of biogas production, biogas methane concentration, chemical oxygen demand (COD), pH, volatile fatty acid (VFA) concentrations, and total and volatile solids were carried out. All analytical procedures were performed in accordance with *Standard Methods 18th Edition* (Greenberg *et al.*, 1992) with the exception of biogas production. Details of all methods are outlined in Appendix B.

3.2.2 Feedstock Materials

A simulated OFMSW mixture was used throughout all experiments. The composition of the mixture was based on the results of a waste audit performed by the RMOC (RMOC, 1992). The simulated mixture reflected the composition of residential mixed waste which was found to consist of 20% yard waste, 40% paper waste and 40% food waste by volume. Yard waste was simulated by a 50:50 volumetric mixture of grass trimmings and leaves. The grass and leaves were cut in 2 cm pieces. The simulated paper waste consisted of a 50:50 volumetric mixture of shredded newspapers and white paper (0.02 cm wide and 1 cm long). Lettuce and tomatoes in a 50:50 volumetric ratio were used to simulate food waste. The lettuce and tomatoes were blended in a food processor for one

minute before use. The volatile solids concentrations were measured as per section B.7 in Appendix B and are reported in Table 2.

Table 2: Volatile Solids Concentrations of the Simulated OFMSW Components

Component	Volatile Solids (g/L)
Lettuce	27.2
Tomatoes	42.0
Leaves	57.3
Grass	19.6
Paper	21.1
Newspaper	13.3

Sewage sludge (RAW and TWAS) samples were obtained from ROPEC. The samples were stored at 4 °C for no longer than one week prior to use. Throughout all experiments, the RAW and TWAS were mixed in a 60:40 volumetric ratio, consistent with the feed ratio used in the anaerobic digesters at ROPEC. Anaerobic seed sludge was also obtained from the anaerobic digesters at ROPEC and was used as an inoculum for all experimental runs.

3.2.3 Experimental Protocols

3.2.3.1 Study of COD Variability of ROPEC Sewage Sludge

To address the variable nature of the composition of sewage sludge, a components of variance study was performed on both WAS and RAW. Using a nested experimental design, samples were taken on five days of the week, twice a day, at two different sampling ports (for WAS only) as shown in Figure 11. At each sampling port, two samples were taken. The organic content of each sample was measured in duplicate using the COD test.

3.2.3.2 Determination of Optimal OFMSW/Sludge Mixture

Several studies conducted at both lab- and full-scale have been reported which used OFMSW blended with sewage sludge to provide added nutrients, but a rigorous analysis of the degradation rates of the biodegradable components has not been addressed (Rivard

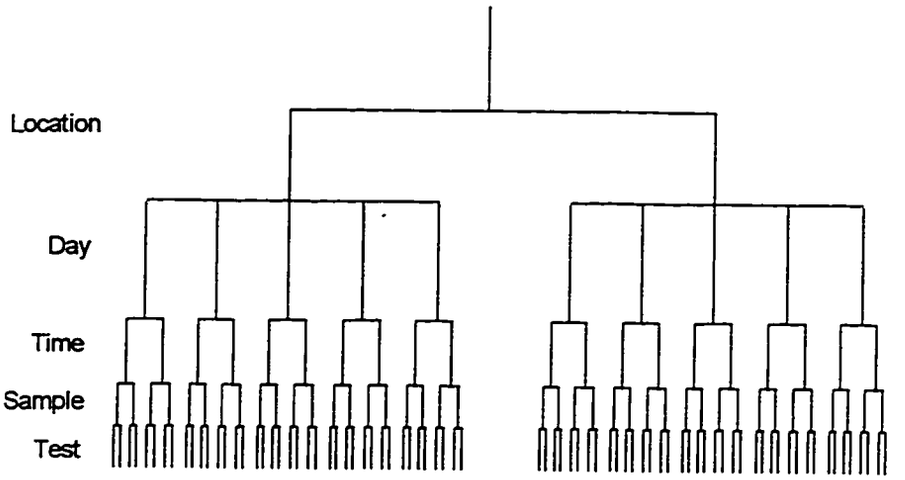


Figure 11: Nested Design for Components of Variance Study for Sewage Sludge

et al., 1990). Consequently, an optimization study was first performed to determine an appropriate ratio of OFMSW to RAW/TWAS. Five 800 mL mixes were prepared in duplicate, 150 mL of which were inoculum (ROPEC anaerobic seed with 2.4% VS), in one litre batch digesters (one litre Wheaton bottles). Supplementary nutrients were not required since the inoculum was found to contain sufficient nutrients for the entire 800 mL mixture. However, sodium bicarbonate (2.4 g) was added to ensure an adequate level of buffering capacity within the batch reactors. All tests were conducted at 37 °C in a heated shaker operated at 100 rpm. Digester performance was evaluated based on the respective cumulative biogas production profiles. Biogas production was monitored since it is widely regarded as the most accurate measure of anaerobic digestion process performance (Switzenbaum *et al.*, 1990). This heuristic is evaluated with respect to this study in section 3.2.3.4.

3.2.3.3 Component Contribution to Biodegradability

Upon identification of an optimal mix ratio, the contribution to biodegradability of each component of the mixture was then evaluated. The volume of each component required in the mix was combined with 150 mL of inoculum and using distilled water, brought up to an 800 mL volume in the one litre digesters. The cumulative biogas production profiles

were again monitored for each component. All tests were conducted in duplicate in heated shakers operated at 37 °C and 100 rpm.

3.2.3.4 Batch Test Study of Particle Size and Total Solids

To study the effects of total solids content and particle size of the feed, a central composite experimental design was used as described in Appendix C.2. Long-term batch tests were run in a random order at five levels of each of the two factors studied: particle sizes ranged from 0.9 to 8.0 mm and total solids contents ranged from 7.0 to 22.1%. The particle sizes studied (0.9 mm, 2.0 mm, 4.0 mm, 6.4 mm, 8.0 mm) were chosen based on available sieving equipment. Also, since there is currently no data available for the performance of the anaerobic co-digestion of Canadian OFMSW and sewage sludge, the TS concentrations chosen (7.9%, 10.0%, 15.0%, 20.0%, 22.1%) include both low-solids and high-solids concentrations to be able to benchmark against European studies. The TS of the feed was initially measured and then adjusted to the required TS concentration by diluting the mixture with water. Particle size of the feed was controlled by sieving through meshes of the corresponding particle sizes. These batch tests were based on the optimal mixture identified in the preceding optimization study, including the 150 mL of ROPEC anaerobic inoculum and 2.4 g of sodium bicarbonate. All tests were conducted in heated shakers operated at 37 °C and 100 rpm. Throughout the experiments, initial and final measurements were recorded for soluble COD, VS, TS and daily measurements of biogas production. The final pH, VFA and biogas methane concentration were also measured. To develop empirical models to describe the effects of feed particle size and TS concentration on the response variables, a least-squares analysis was performed using STATISTICA Release 4.5. The development of the experimental design and model building methods are presented in Appendix C.

3.3 Results and Discussion

3.3.1 Study of COD Variability of ROPEC Sewage Sludge

In the early stages of the experimental strategy, the contributions of the soluble and particulate fractions of both RAW and WAS were investigated. During the course of this initial study, in order to obtain a better understanding of the sources of variability of the organic content of RAW and WAS, a components of variance study was performed. A nested experimental design was used as discussed in the experimental protocol presented in section 3.2.3.1. The raw data from the study are presented in Appendix D.1 and are summarized in Table 3 for the soluble fraction. The data is reported in terms of the soluble COD for both sludges since there is very limited historical plant data available for the soluble fractions of these sludges.

Table 3 indicates that for RAW, the day-to-day variability had the highest contribution to variance, 76.3%, whereas for WAS, the time-of-day sampling was most significant, with a contribution to variance of 86.4%. Table 3 also indicates a negative value for the variance introduced by location of sampling for the WAS samples. In the calculation of the components of variance, the contribution to variance of each component is estimated sequentially starting from the bottom of the hierarchy shown in Figure 11. Within this scheme, the contribution to variance of location was calculated last; hence any errors in estimating the four other variances accumulated, ultimately resulting in a negative variance.

Table 3: Components of Variance for RAW and WAS Soluble COD

Variable	% Variance for RAW	% Variance WAS
Test	0.003	2.196
Sample	2.894	4.515
Time	20.800	86.385
Day	76.304	10.714
Location	---	-3.810
Total	100.0	100.0

Based on the data collected during the experiment, the average soluble COD for RAW was 1663 mg O_2/L and 63 mg O_2/L for WAS. These CODs are very low compared to the COD contribution of the respective particulate fractions and were hence not studied further. Historical data shown in Figure 12 indicate average values of 35,787 and 6,421 mg O_2/L for RAW and WAS particulate fractions respectively. As expected in most wastewater treatment plants, ROPEC's historical data indicated a high degree of variability in the CODs of the particulate fractions of RAW and WAS as shown in Figure 12. Plant operators reported COD measurements once a week. With sewage sludges processed through the entire plant within 2 days, it is very difficult to deduce any conclusions from this data.

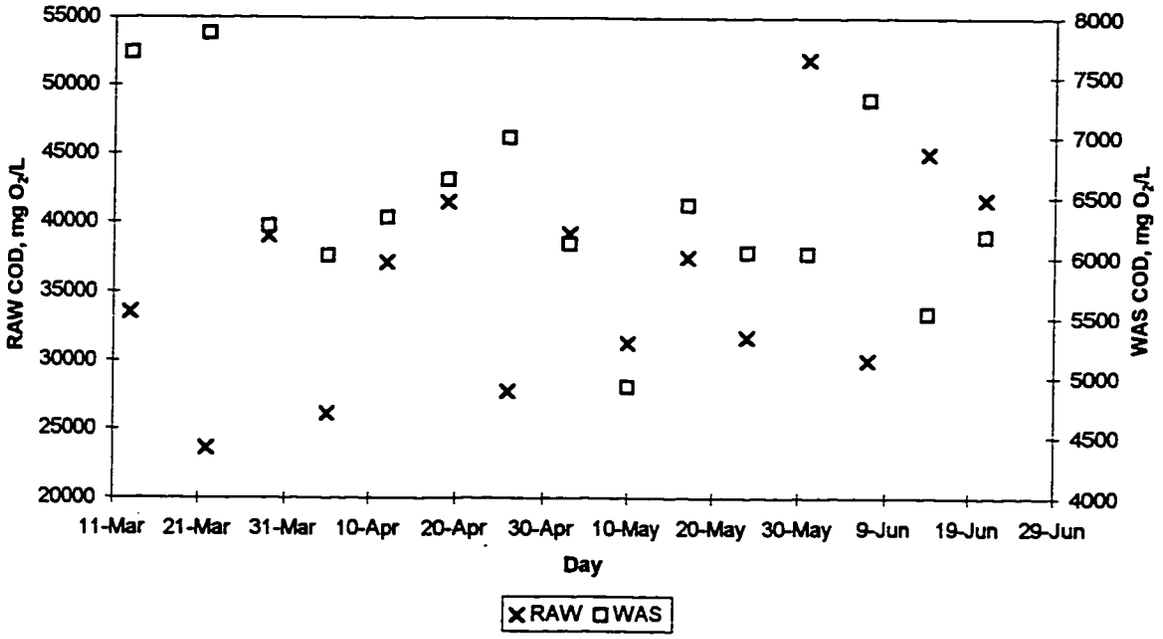


Figure 12: Historical COD Profile of ROPEC RAW and WAS

The results obtained from the soluble fractions should reflect the trends expected in the particulate fractions. Based on the results presented in Table 3, time-of-day and day-to-day differences would have the highest contribution to variance. Since the variability introduced through time-of-day and day-to-day differences cannot be controlled, further studies are required with several sludge samples to determine the robustness of HSAD to

a wide range of sludge strengths. This is beyond the scope of this work and hence a single sludge sample was collected and assumed to represent a typical strength.

Figures 13 and 14 are normal probability plots of RAW and WAS particulate historical CODs. Based on the historical data, the average RAW particulate COD was 35,787 mg

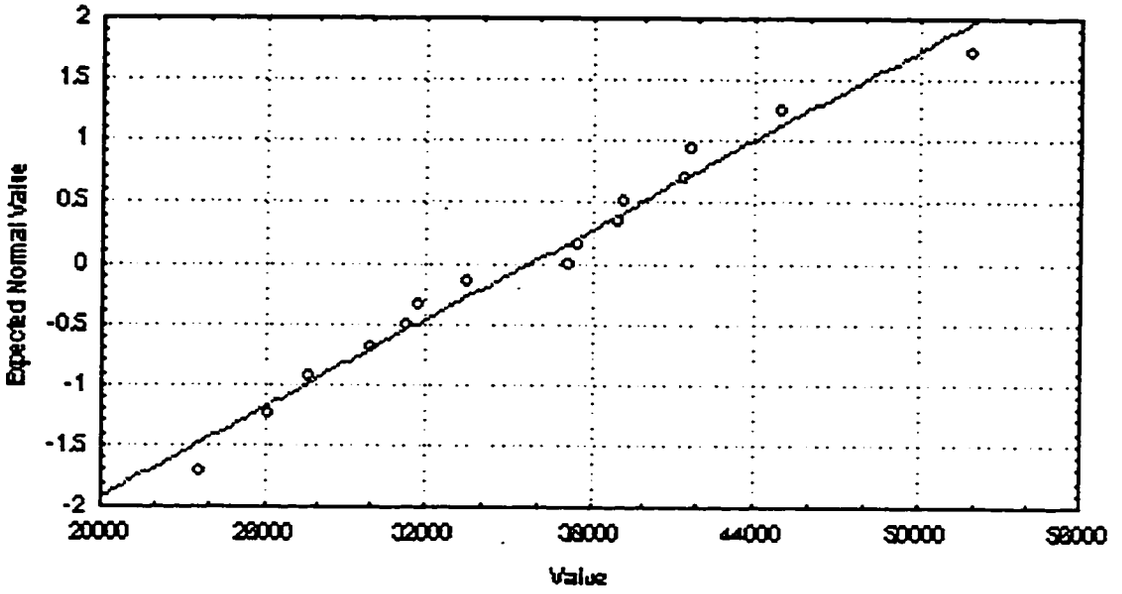


Figure 13: RAW Particulate COD Normal Probability Plot

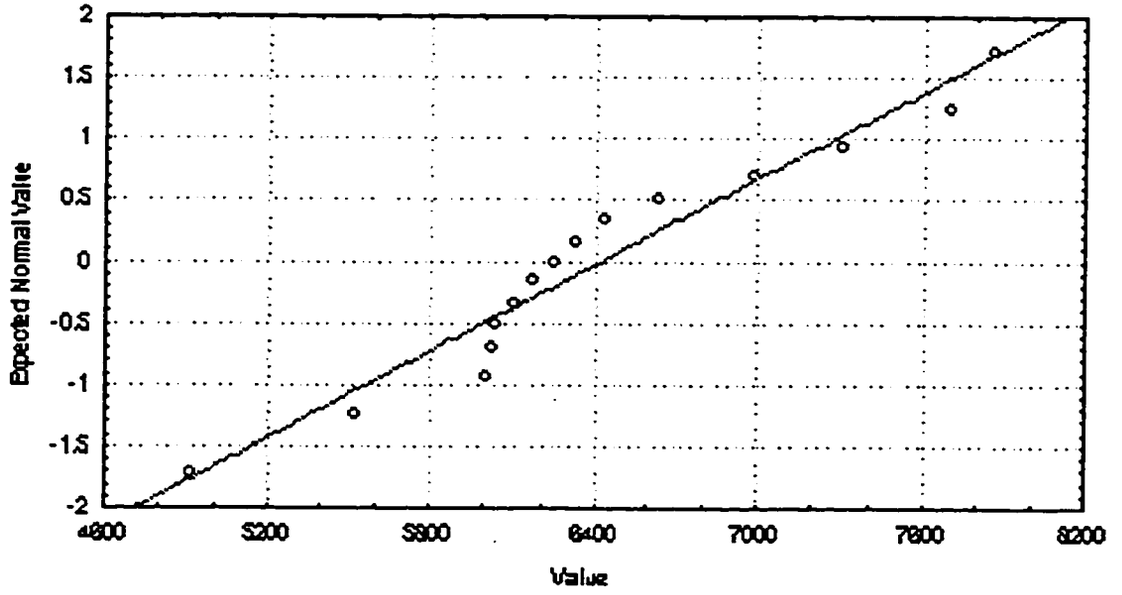


Figure 14: WAS Particulate COD Normal Probability Plot

O_2/L and 6,421 mg O_2/L for WAS compared to the samples used throughout the experiments which had particulate COD concentrations of 42,820 mg O_2/L and 5,513 mg O_2/L for RAW and WAS respectively. Both of these values are within approximately 1 standard deviation from the average particulate COD observed historically.

3.3.2 Determination of Optimal Sludge/OFMSW Mixture

As discussed in section 3.1, there are several inconsistencies in the literature with respect to the optimal mixture of OFMSW and sewage sludge. In this light, an optimization study was undertaken to determine the optimal OFMSW content in the OFMSW/sludge mixture specific to the Ottawa-Carleton region. The raw data from this study are presented in Appendix D.2. From the biogas production data presented in Table D.3, the pooled standard deviation was found to range from 1.5 for the control run to 103.3 for the 25% OFMSW mixture. These values were found to be independent of time and biogas production level. This is exemplified in Figures 15 and 16. In Figure 15, for all runs, the standard deviation appears evenly scattered with the time progression. For the specific case of the 25% OFMSW mixture, the standard deviation is also evenly distributed with respect to biogas production level except for the five high points at approximately 500 mL. There are not enough data points across the entire range of biogas production levels to indicate any systematic trend of the standard deviation.

The average cumulative biogas production profiles for each of the MSW/sludge mixtures are shown in Figure 17. All reactors performed significantly better than the control which consisted of the inoculum and distilled water. As indicated in Figure 18, the cumulative biogas production of the mixtures for approximately the first 170 increased with increasing proportions of OFMSW. This is consistent with the work of Cecchi *et al.* (1988b). However, in this work after 400 hours, the trend remains true for mixtures with 50% OFMSW or higher. The 0% and 25% OFMSW mixtures demonstrate enhanced biological activity after this initial period. Ultimately, the 100% OFMSW mixture performed at the

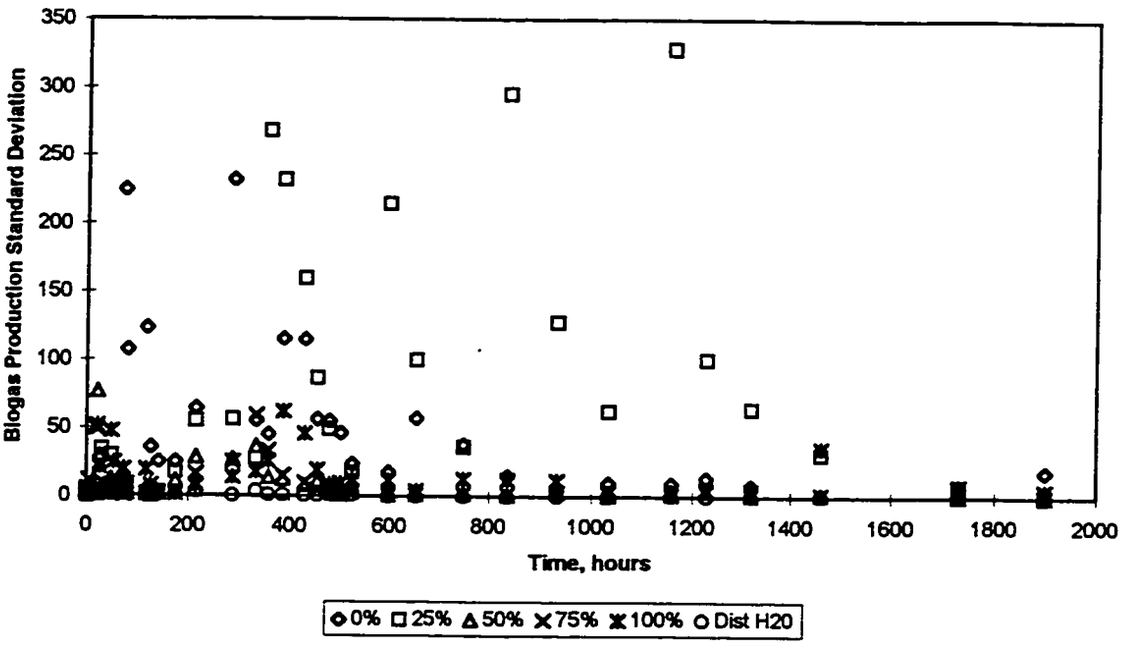


Figure 15: Time Dependence of Biogas Production Standard Deviation for Varying OFMSW/Sewage Sludge Mixtures

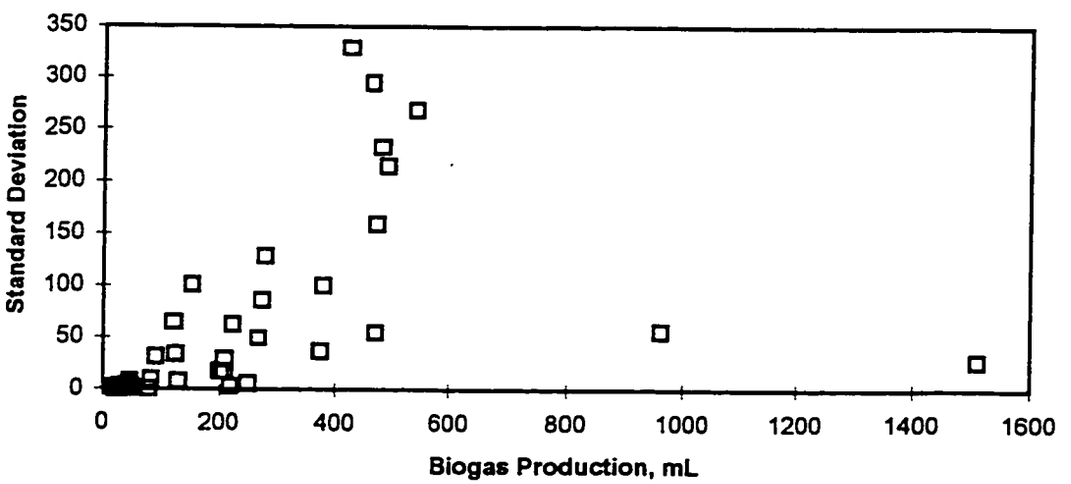


Figure 16: Dependence of Biogas Production Standard Deviation on Biogas Production Volume for the Mixture of 25% OFMSW and 75% Sewage Sludge

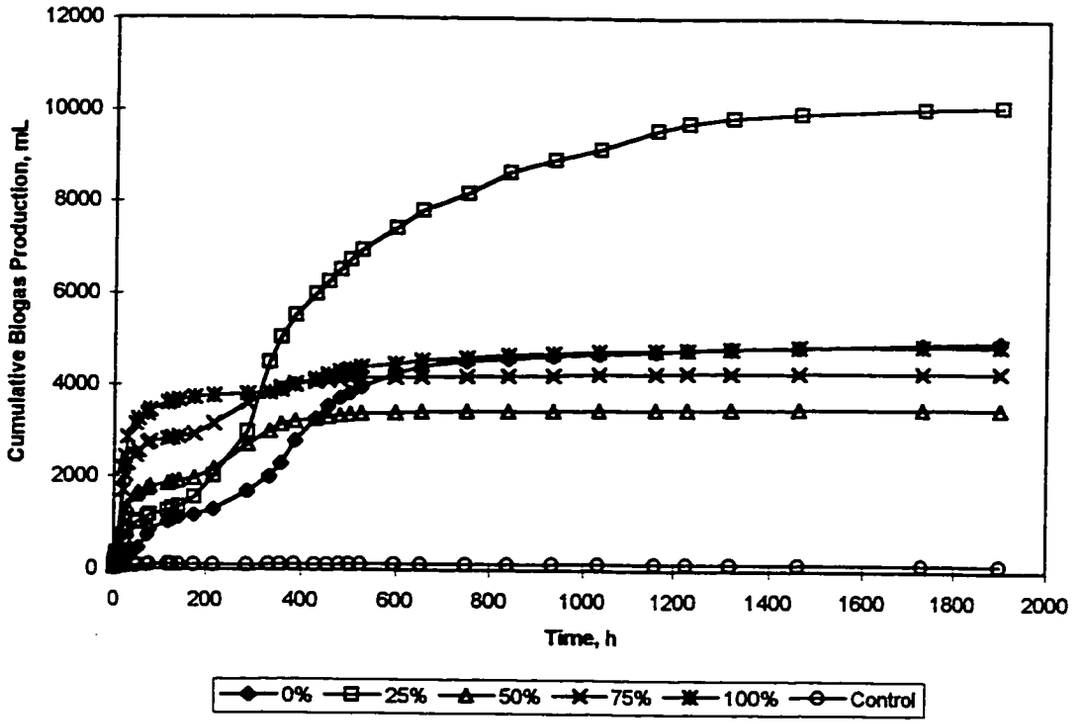


Figure 17: Cumulative Biogas Production Profile for OFMSW/Sludge Mixtures with Varying OFMSW Contents

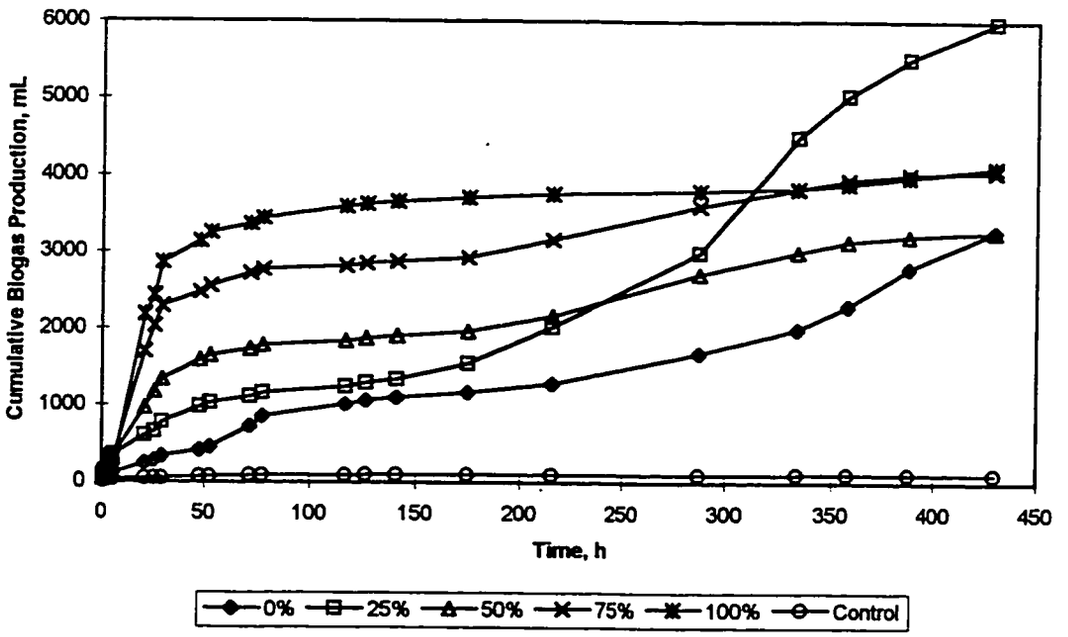


Figure 18: Initial Cumulative Biogas Production Profile for OFMSW/Sludge Mixtures with Varying OFMSW Contents

same level as the digester with no OFMSW added. Of particular significance, is the performance of the mixture with 25% OFMSW. After approximately 400 hours biogas production exceeds all mixtures. These results agree with those of Poggi-Varaldo and Oleszkiewicz (1992) who reported that the richer the mixture is in sludge, the better the reactor performance in terms of volatile solids conversion to biogas. The study by Cecchi *et al.* (1988b) ran for only 600 hours whereas Poggi-Varaldo and Oleszkiewicz(1992) ran their experiments for 1400 hours, similar to the time frame of this study. Differences observed between the studies by Cecchi *et al.* (1988b), Poggi-Varaldo and Oleszkiewicz (1992) and this study are most likely due to the differences in digestion time. It should be noted that Poggi-Varaldo and Oleszkiewicz (1992) reported final results only and did not report the dynamic behaviour reported in this study.

Differences in the magnitudes of the biogas production volumes between different studies can be attributed to differences in feed composition. The increased biogas production at the higher OFMSW concentrations is most likely due to the increasing proportion of very readily biodegradable food waste explaining the relative increases in biodegradability in the region greater than 50% OFMSW. However, for the mixtures with less than 50% OFMSW, the optimal C:N ratio (25 to 80, Kayhanian and Tchobanoglous (1992); Pohland (1992)) is most likely being approached and in the experimental conditions of this study was best attained in the run with an OFMSW content of 25%. The approximate C:N ratio, calculated based on COD and total Kjeldahl nitrogen data for the OFMSW constituents and historical data for the sludges, is 43. Throughout all ensuing experiments, the 25% OFMSW mixture was used. However, further experimentation is required to validate this optimum mixture in terms of other response variables, including indicators of organics removal such as TS, VS and soluble COD.

3.3.3 Component Contribution to Biodegradability

Given the superior performance of the 25% OFMSW mixture, a more thorough analysis of the mixture components was performed. Specifically, the contribution of each of the individual components to the overall biodegradability of the mixture was studied. The raw

data are presented in Appendix D.3. Each of the components was mixed with ROPEC anaerobic digester inoculum and sodium bicarbonate. Batch reactors were run in duplicate at 37 °C, each reactor containing one of the mixture components in the corresponding concentration in which it was represented in the 25% OFMSW/sewage sludge mixture. The biogas production was measured daily with pooled standard deviations ranging from 0.8 for newspaper to 23.0 for RAW. These values were found to be independent of time and biogas production level. This is first exemplified in Figure 19, where for all runs, the standard deviation appears evenly scattered with the time progression. For the specific case of the RAW in Figure 20, the standard deviation is also evenly distributed except for the outlier at approximately 5 mL.

From the progression of the average OFMSW/sewage sludge mixture profile shown in Figure 21, there appear to be two distinct plateaus (0 to 100 hours, and 400 to 500 hours) representing two distinct regions of biological activity. These regions are most likely based on biodegradability of two different classes of substrates: readily biodegradable, and moderate to difficult to biodegrade as postulated by Cecchi *et al.* (1990). The third region

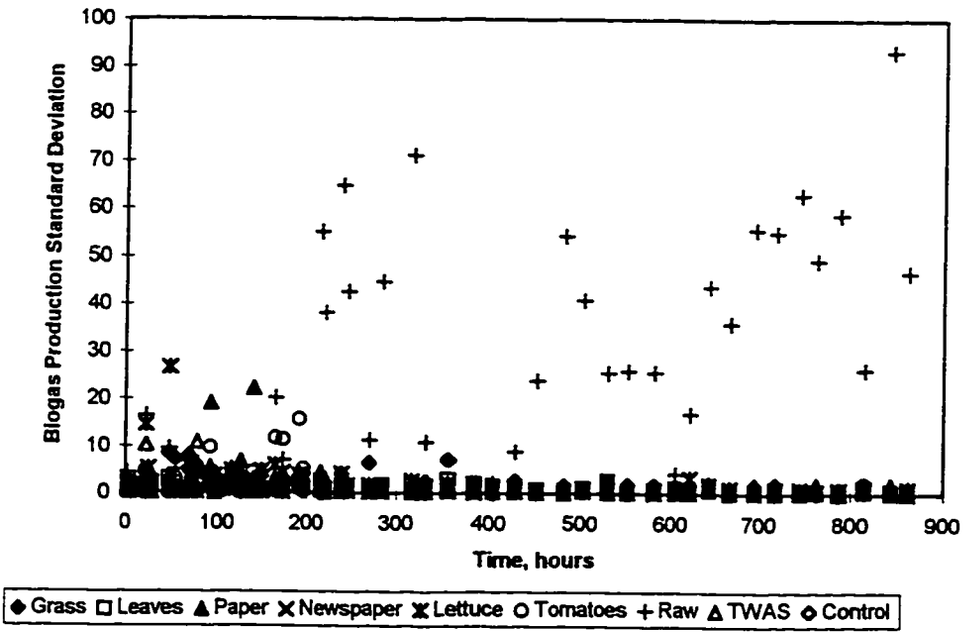


Figure 19: Time Dependence of Biogas Production Standard Deviation of Sewage Sludge and OFMSW Components

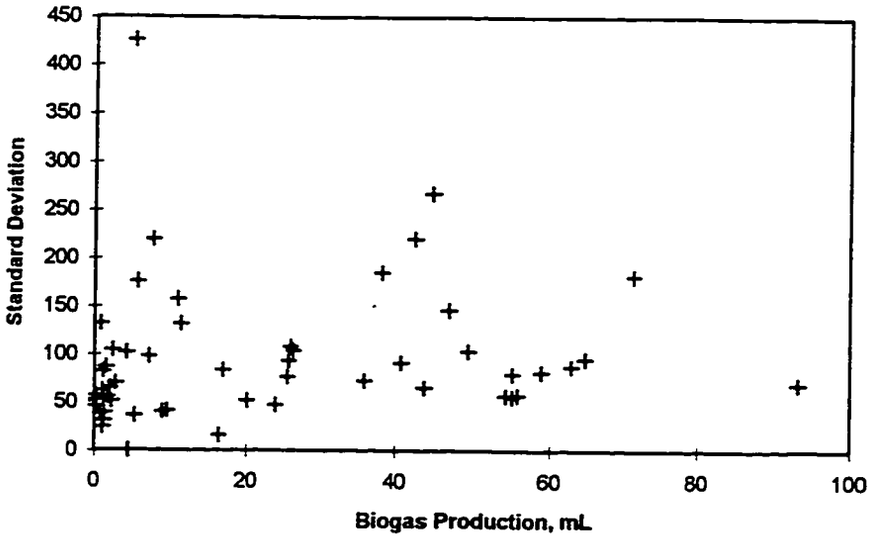


Figure 20: Dependence of Biogas Production Standard Deviation on Biogas Production Volume for RAW

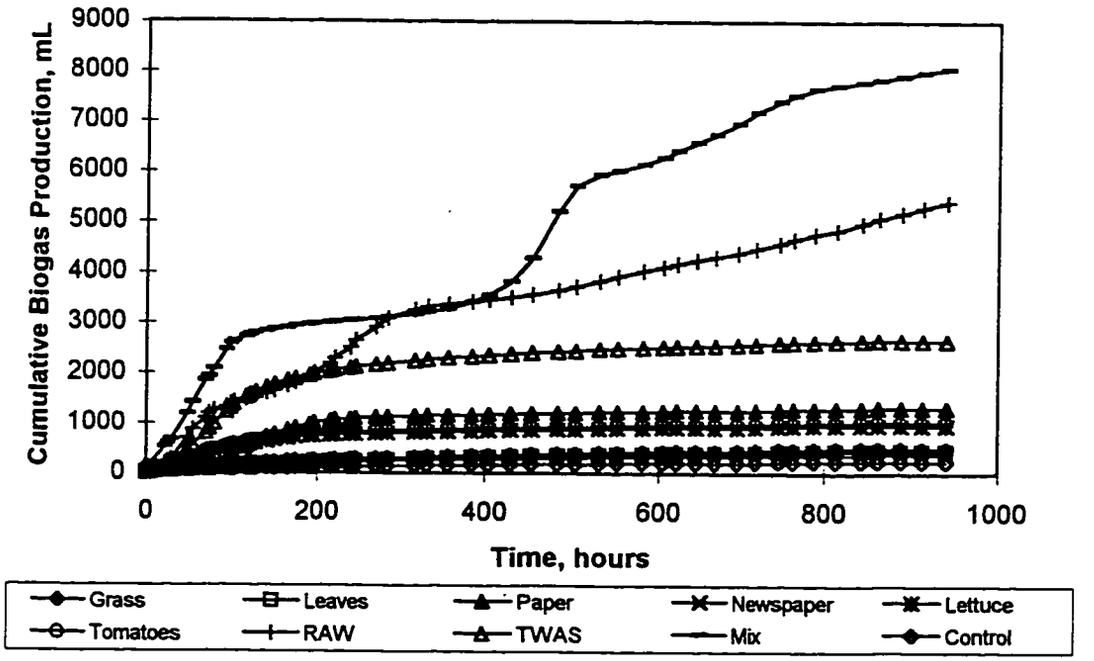


Figure 21: Contribution to Anaerobic Biodegradabilty of Each Component in the 25% OFMSW/Sewage Sludge Mixture

suggested by Cecchi *et al.* (1990) was not clearly identified in this study as reflected in Figure 21. Also, the plateaus of the mixture corresponding to the multiple substrates were not observed in any of the individual component runs, indicating that the microbial population acclimated to the complex mixture differently than when digesting a "pure" component. The single exception is RAW which also appeared to exhibit the two region (0 to 100 hour, and 200 to 300 hour) profile. The complex composition of RAW would suggest the possibility of multiple biodegradabilities.

Additively, the components of the mixture in Figure 21, do not appear to reflect the biodegradability (biogas production) of the OFMSW/sludge mixture. Based on biogas production, the RAW and TWAS appear to be the most biodegradable components. However, this may be confounded by the fact that higher concentrations of these components were used in their respective reactors to reflect the actual component concentration present in the mixture. To clarify this, Figure 22 presents the cumulative biogas production normalized per gram of volatile solids added.

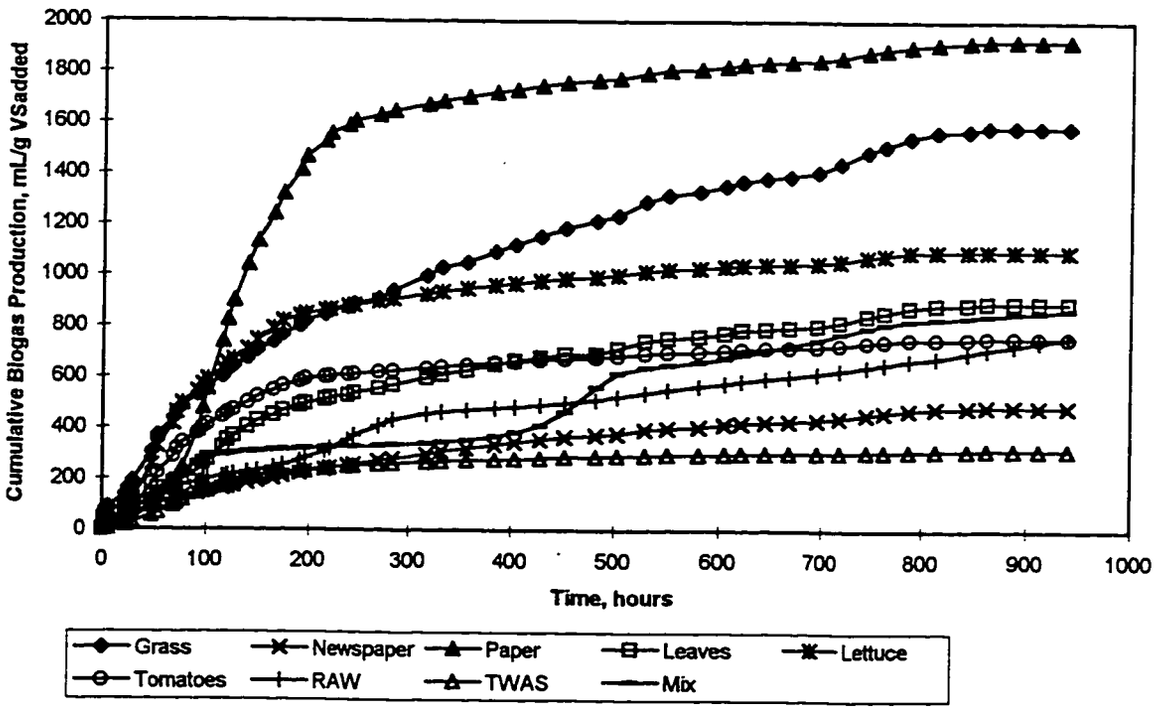


Figure 22: Contribution to Anaerobic Biodegradabilty of Each Component in the 25% OFMSW/Sewage Sludge Mixture Based on VS Addition

From Figure 22, it appears that the paper followed by grass had the highest contributions to biodegradability, whereas the TWAS and newspaper had significantly lower contributions. The grass and lettuce were digested within the first 200 hours at comparable rates, however, beyond that period, the lettuce appeared to be completely biodegraded whereas the grass was continually being digested. TWAS had the lowest anaerobic biodegradability, ultimately plateauing at a level approximately one-tenth of the plateau for the paper. Compared with TWAS, RAW was approximately twice as biodegradable. Typical biodegradabilities of RAW and TWAS are reported to be 40% to 60% and 30% to 50% respectively for conventional anaerobic digestion systems (Grady and Lim, 1980). The OFMSW/sewage sludge mixture appeared to display an average performance; no synergistic behaviour was detected. It approached the anaerobic biodegradability of the sludge fractions which is to be expected due to the high fraction of sewage sludge (75%) in the mixture. From the ultimate biogas production at 950 hours, the expected mixture biogas production was calculated to be 945 mL per g VS added, comparable to the 860 mL per g VS added observed in Figure 22.

Based on Figure 22, the two regions of biological activity identified in Figure 21 would be represented by the paper, grass and possibly lettuce in the first region, and the remaining components in the second region. The slopes of the rise of the two curves in Figure 21 describe the biogas production rates and are indications of the rate of biodegradation of these classes of components. Estimates of these slopes are 21.8 mL/h ± 1.2 mL/h for the first region and 17.4 mL/h ± 1.0 mL/h for the second region which correspond to COD removals of 62.3 mg COD/h ± 3.4 mg COD/h and 49.7 mg COD/h ± 2.9 mg COD/h respectively. A more detailed kinetic study is required to establish kinetic constants for this system.

3.3.4 Batch Test Study on Particle Size and Total Solids Content

As outlined in the experimental protocol in section 3.2.3, a central composite experimental design was used to study the effects of the total solids content and particle size of the feed mixture. The design is presented in Appendix C.2 and the raw data can be found in

Appendix D.4. All batch reactors appeared stable based on the neutral pHs (ranging from 7.6 to 7.9) measured after digestion as reported in Table D.6 and the lack of VFA accumulation as shown in Table D.7. Only two reactors indicated the presence of significant VFAs (48 mg/L and 169 mg/L of acetic acid), both of which had low total solids concentrations (7.9% and 10.0%) and relatively high particle sizes (6.4 mm and 4.0 mm). These reactors did not perform well as indicated by the various performance measures, namely low biogas production and concomitant poor removal of organics, for example, 26.3% and 19.7% TS removal and 0.490 and 0.484 L of biogas per gram of VS removed.

The data collected from the batch runs were used to develop empirical models describing the experimental system. The models were generated using the method of least squares. The technique involves the estimation of model parameters for second order models of the form

$$E(Y) = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \sum_{j \geq i}^{k} \beta_{ij} X_i X_j \qquad [1]$$

where

E(Y) is the expected value of the response variable
β_0, β_i, β_j are the model parameters
X_i and X_j are the coded factors being studied
k is the number of factors being studied

The development of the empirical models for each of the seven measures of process performance are presented in Appendix E with sample calculations presented in Appendix I. Both the results from the initial model as well as the final model are included. The final model was the simplified model after insignificant terms were removed. A model parameter was deemed insignificant if it had a p-level more than 0.05. Table 4 summarizes the parameter estimates obtained by least-squares analysis and their corresponding 95% confidence intervals for the reduced (final) models. The R^2 values are also included and indicate that a large portion of the variance in the data is accounted for by the final

Table 4: Summary of Empirical Models Developed for the Anaerobic Co-Digestion of Sewage Sludge and OFMSW

	Model Response Parameter Estimates ± 95 % Confidence Limits						
Parameter	*Gas Production L*	*Gas Production/ VS added L/g VSa*	*Gas Production/ VS removed L/g VSr*	*Methane Concentration %*	*TS Removal %*	*VS Removal %*	*COD Removal %*
β_0	4.29 ± .31	0.58 ± .04	1.05 ± .04	61.7 ± 1.6	33.5 ± 1.1	53.1 ± 2.5	77.9 ± 8.0
β_1^*	-0.35 ± .26	-0.04 ± .03	-0.07 ± .03	---	---	---	---
β_2^{**}	1.77 ± .30	0.07 ± .04	-0.16 ± .04	---	8.6 ± 1.0	13.0 ± 2.4	13.0 ± 7.3
β_{11}	-0.35 ± .21	-0.04 ± .03	---	---	-2.5 ± 0.7	-3.7 ± 1.6	---
β_{22}	---	---	0.08 ± .04	---	---	---	-9.0 ± 8.0
β_{12}	-0.63 ± .38	-0.06 ± .05	---	---	-3.0 ± 1.3	-4.2 ± 3.1	---
R^2	0.973	0.907	0.950	---	0.983	0.959	0.715
F_{reg}	2620.14	986.74	7834.22	---	10419.29	4608.25	285.09
$F_{.05,v_1,v_2}$	4.12	4.12	4.07	---	4.07	4.07	4.39
F_{lof}	2.27	4.14	0.94	---	2.95	1.94	3.38
$F_{.05,v_1,v_2}$	9.12	9.12	9.01	---	9.01	9.01	9.28

* Factor 1 corresponds to coded particle size

** Factor 2 corresponds to coded TS concentration

models, except for the case of the soluble COD removal model. Its R^2 value of 0.715 reflects the high level of pure error associated with COD measurements. Also, for the biogas methane concentration, a model based on the two factors studied could not be fitted as indicated in the table. The models also indicated no lack of fit since the F_{lof} test statistic was lower than the F-value at a 95% confidence level in all cases.

The residuals for all the models appeared to follow a normal distribution. For example, Figure 23 is a normal probability plot of the residuals for the cumulative biogas production model. The data follow the line representing a normal distribution. Similar trends were observed for the other models.

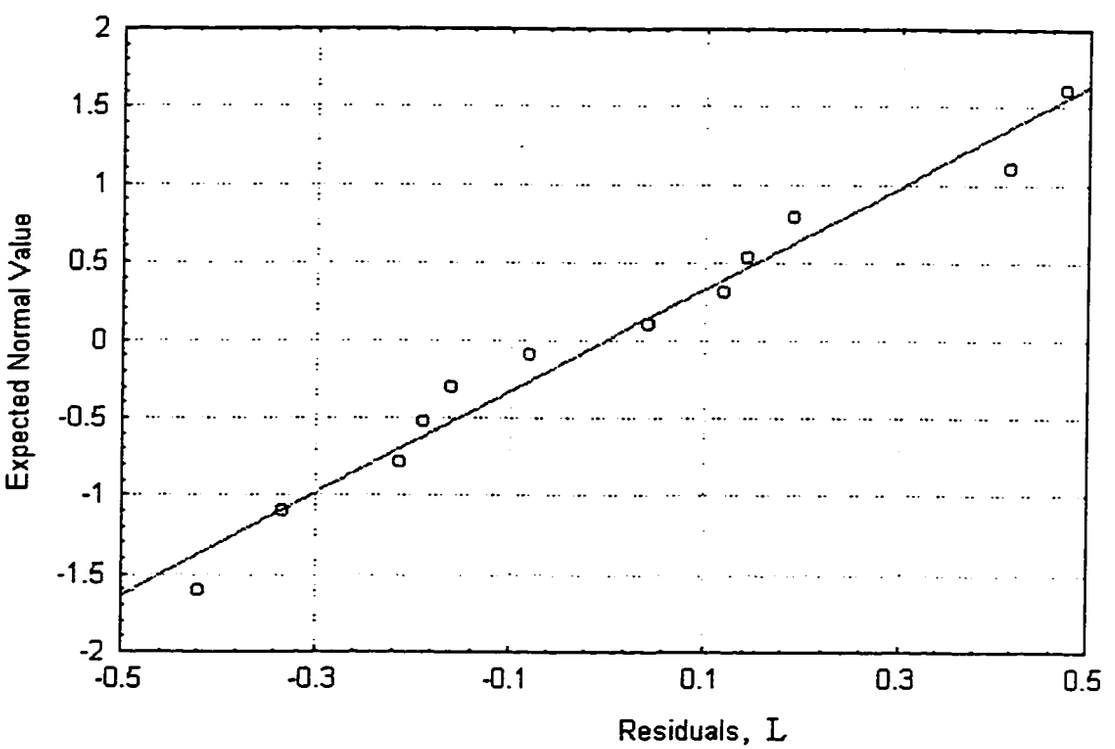


Figure 23: Normal Probability Plot for the Residuals from the Cumulative Biogas Production Model

Also, the residual plots versus the predicted values of the response and run order for each of the models also indicated constant variance. Again considering the case of the cumulative biogas production model, Figure 24 indicates equal scatter of the residual

data above and below the x-axis indicating that the variance was independent of the value of the cumulative biogas production.

The contour plots for all of the six models summarized in Table 4 are shown in Figures 25 and 26, as well as the parabolic plot for soluble COD in Figure 27. The values of the experimental factors shown in the plots are coded, as calculated in Appendix I. To back-calculate to the actual values,

$$\textit{Total Solids Concentration (mg/L)} = 15 + 5\ \textit{(coded TS)} \quad [2]$$

$$\textit{Particle Size (mm)} = 4 + 2\ \textit{(coded Particle Size)} \quad [3]$$

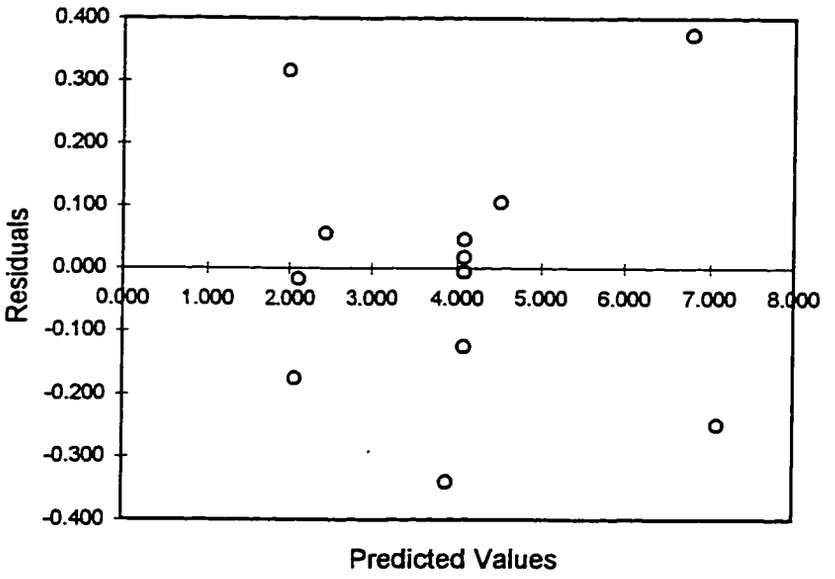
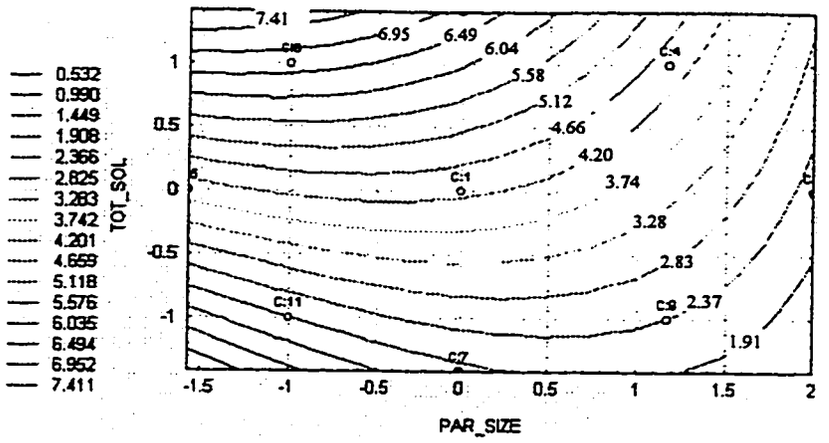


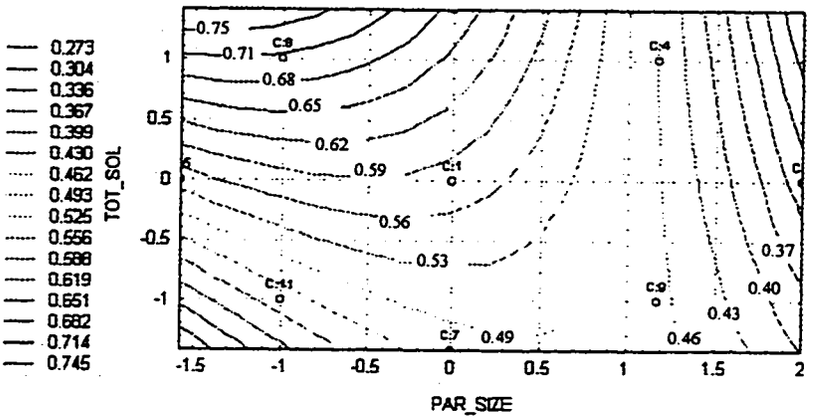
Figure 24: Residual Plot for Cumulative Biogas Production Model

The models for cumulative biogas production and for cumulative biogas production per unit mass of volatile solids fed have very similar responses (Figures 25a) and 25b)). Both indicate optimal operating conditions at high total solids concentrations (22.1%) and small particle sizes (0.85 mm). Both models indicated a strong interaction between the two experimental factors studied. For example, in Figure 25b), the effect of TS concentration on biogas production with respect to VS addition is much more significant at small particle sizes (coded -2.5) than large particle sizes

a. Cumulative Biogas Production (L)



b. Cumulative Biogas Production with Respect to Volatile Solids Addition (L/g VSa)



c. Cumulative Biogas Production with Respect to Volatile Solids Removal (L/g VSr)

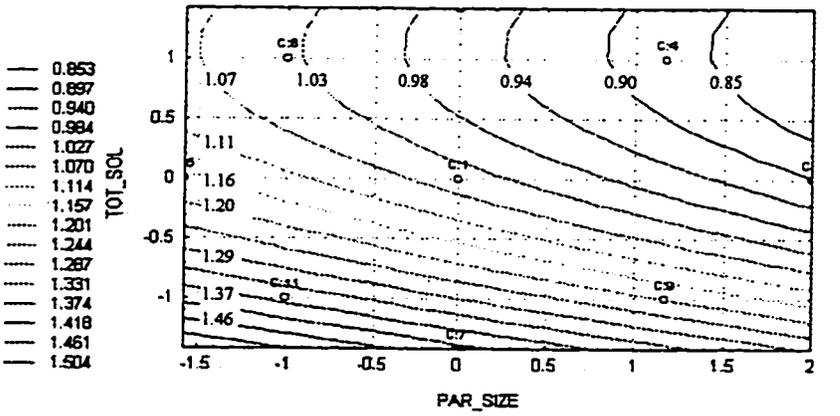
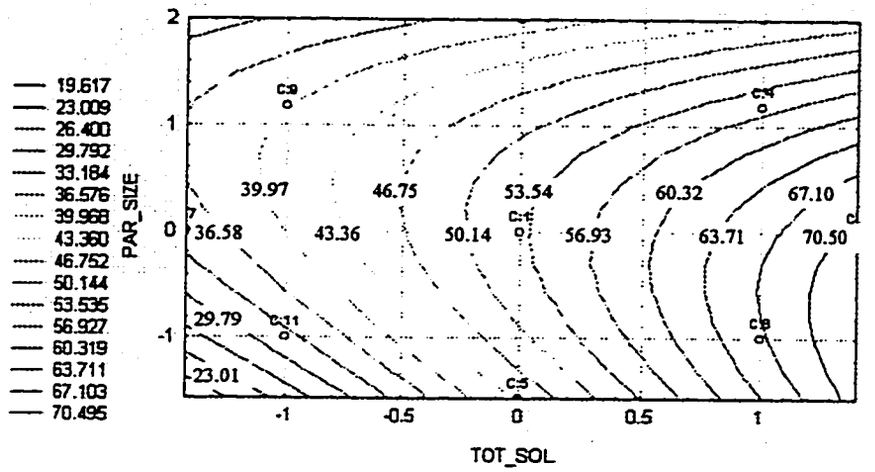


Figure 25: Contour Plots of Empirical Models of the Effect of Total Solids Concentration and Particle Size of the Feed on Biogas Production
Legend: TOT_SOL = Coded TS concentration, PAR_SIZE = Coded particle size

a. Volatile Solids Reduction (%)



b. Total Solids Reduction (%)

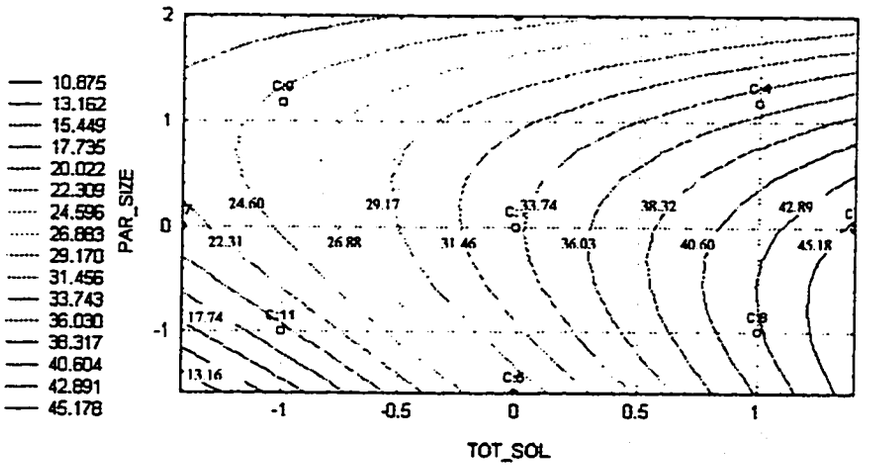


Figure 26: Contour Plots of Empirical Models of the Effect of Total Solids Concentration and Particle Size of the Feed on Organics Removal
Legend: TOT_SOL = Coded TS concentration
PAR_SIZE = Coded particle size

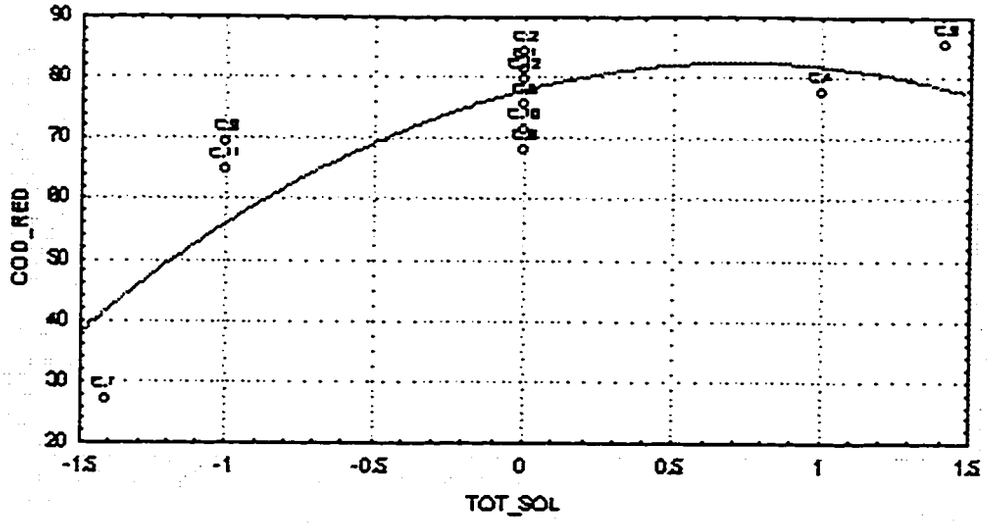


Figure 27: Parabolic Plot of Empirical Models of the Effect of Total Solids Concentration and Particle Size of the Feed on the Soluble COD Removal
Legend: COD_RED = Coded Soluble COD Removal,
PAR_SIZE = Coded Particle Size

(coded 2.0). The model for cumulative biogas production per unit of volatile solids removed (Figure 25c)) generally predicted the same trends as the two preceding models, however, a linear increase of biogas production with respect to volatile solids removal was observed with respect to particle size compared to the parabolic profile of the biogas production model and the biogas production per unit of volatile solids added. Both the VS and TS reduction models, Figures 26a) and 26b), also predicted the same parabolic trend with an optimum at high total solids concentrations (22.1%) and mid-ranged particle sizes (4 mm). Further investigations into the biological and physicochemical basis of this trend are required. Also, since biogas production is expected to be directly proportional to VS removal, the models in Figures 26 and 27a) should be and were indeed found to be consistent. The COD reduction was found to be independent of particle size and based on the parabolic profile in Figure 27, the optimum is at slightly elevated total solids concentrations (19%).

Overall, it was found that optimal operating conditions for anaerobic co-digestion of OFMSW and sewage sludge occurred with small particle sizes (0.85 mm the lowest studied) and high total solids concentrations (22.1% the highest studied). Based on

the initial inoculum used in the reactors, the maximum specific activity achieved, assuming that 50% of the VS was biomass, was found to be 0.38 g COD removed per g biomass per day based on the optimal cumulative biogas production. Reduction of feed particle size influences the HSAD process in two ways: it removes natural barriers to microbial attack, including films, waxy coatings and other surface protectants; and it increases the ratio of surface area to mass (Kayhanian and Hardy, 1994). The larger the surface exposed to microbial attack, the more rapid the biodegradation. However, excessive reduction of feed particle size may become too costly and be economically unjustified. Increased TS concentration in the feed leads to a higher concentration of substrate being available for the anaerobes and consequently increased reactor performance.

Similar trends were observed in the work of Poggi-Varaldo and Oleszkiewicz (1992) on a mixture of Canadian OFMSW and sewage sludge. However, they reported inhibition at TS levels greater than 35%. Further experimentation with the RMOC OFMSW/sludge mixture is required to quantitatively study the effects at higher TS concentrations beyond the range investigated in this study.

3.4 Conclusions and Recommendations

Based on the results reported in this chapter, the anaerobic co-digestion of sewage sludge (RAW and TWAS) and simulated OFMSW was found to be technically feasible. In particular, it was found that:

1. The optimal MSW/sludge mixture as reflected by biogas production consisted of 25% MSW and 75% RAW/TWAS (in a 60:40 volumetric ratio) by volume consistent with the trends reported by other Canadian studies but contrary to the trends reported by European studies;
2. The maximum specific activity achieved, assuming that 50% of the VS was biomass, was found to be 0.38 g COD removed per g biomass per day

3. Of all the components in the mixture the simulated food waste was the most biodegradable whereas the TWAS and paper waste were the least biodegradable;
4. There are two regions of biological activity throughout the anaerobic co-digestion process evaluated in this study, reflected by two classes of compounds characteristic of easy and difficult to biodegrade components, consistent with other studies;
5. The optimal operating conditions for the HSAD process are at low particle sizes (0.85 mm the lowest studied) and high total solids concentrations (22.1% the highest studied). Both particle size and TS concentration of the feed had a significant impact on the performance of the process.

The results presented in this chapter can be used as a basis to design an anaerobic digester to be used within the RMOC to co-treat OFMSW and sewage sludge . However, some considerations in the design that need to be taken into account include the fact that:

1. Further experimentation at higher total solids concentrations in the feed are required to assess reactor performance under potentially stressed conditions;
2. Studies need to be performed on multiple sludges to determine the robustness of HSAD to a wide range of sludge strengths;
3. Further studies need to be performed to gain a better understanding of the parabolic trend of process performance with respect to particle size;

Since both TS concentration of the feed and particle size were found to have significant effects on the process performance, pretreatments to enhance performance through particle size reduction allowing for the maximization of TS concentration of the feed should be further investigated.

CHAPTER 4:
Comparative Study of Pretreatments to the Anaerobic Digestion of Sewage Sludge and Municipal Solid Waste

Based on the results of Chapter 3, further investigation of feed pretreatments to enhance process performance through particle size reduction and maximization of feed TS concentrations was shown to be highly feasible. Historically, attempts to increase anaerobic reactor performance have typically involved increasing the retention time by decreasing either the loading or dilution rates. However, this approach results in an increase in the required reactor volume in order to deal with a given feed rate. In this light, alternative strategies to enhance reactor performance are being investigated. To avoid the high capital costs associated with the large reactor sizes potentially required for HSAD applications, methods to improve digester performance that can lead to both the reduction of reactor volumes and a better process energy balance have been proposed. Based on the optimal mixture and particle size data presented in Chapter 3, this chapter further addresses the effectiveness of alkaline, thermal and thermochemical pretreatments to improve the performance of the anaerobic co-digestion of OFMSW and sewage sludge.

4.1 Literature Survey

Anderson *et al.* (1995) strongly recommended the implementation of pretreatments based on their study of the anaerobic digestion of Canadian sewage sludge in high-rate anaerobic reactors. They found that depending on the sludge ratio, addition of WAS without pretreatment decreased digester operational efficiency; they reported almost 20% less volatile mass reduction, up to 25% reduction in metabolic reaction rates, and reduced

biogas production rates of up to 40%. This was attributed to the presence of the difficult-to-biodegrade cell membrane encapsulating the biodegradable substrates of WAS, making them less available in the digestion process. It was concluded that, unless some type of pretreatment operation is first applied to liberate these substrates, this type of commonly used digestion system will be of less benefit to wastewater treatment plants.

In anaerobic digestion, soluble components such as protein, pectins, starches, lipids, free cellulose and low-lignified materials are preferentially digested (Eastman *et al.*, 1981). Despite the fact that microbial solubilization of more complex organics occurs, the anaerobic treatment of OFMSW typically results in the digestion of less than 50% of the feed materials (Golueke, 1973). Also, the initial solubilization of complex organics is widely recognized as the rate-limiting step of the HSAD process (Eastman and Ferguson, 1981; Gossett *et al.*, 1982; Ray *et al.*, 1989). Hence, attention is now being focused on the facilitation of the solubilization of complex organics in HSAD processes. Pretreatments that enhance solubilization include alkaline, thermal and thermochemical methods.

4.1.1 Alkaline Pretreatment Technologies

Several chemical pretreatments have been suggested including acidic, alkaline and extracellular enzymatic treatments. Comparing acidic and alkaline treatments, the latter has many advantages in light of anaerobic digestion processes. Following acidic treatment (typically pH 3), neutralization of the feed is required before anaerobic treatment to bring the pH up to an appropriate range of 7.0 to 7.4 to allow for microbial activity (Pavlostathis *et al.*, 1985). As a result, there is no possibility of recycling residual acidity for the treatment of fresh feed. However, in the case of alkaline pretreatment, any alkali remaining with the anaerobically treated solids can be readily recycled by subsequent solid-liquid separations.

Pavlostathis and Giraldo-Gomez (1985) studied the alkaline treatment of wheat straw to increase its anaerobic biodegradability. Four solids concentrations (2.5%, 5.0%, 7.5% and 10%) were used with alkali levels ranging from 2 to 50 g of sodium hydroxide per 100 g of dry matter (pH 9.6) for treatment periods of up to 24 hours. They concluded that all levels of alkaline pretreated straw resulted in higher methane production relative to untreated wheat straw. Low alkali levels (2 g of sodium hydroxide per 100 g of dry matter) showed no statistically significant difference (i.e. p-level greater than 0.05) in methane production for differing treatment times. However, with high alkali levels (16 and 50 g sodium hydroxide per 100 g dry matter), an increase in treatment time resulted in increased biogas methane production (by as high as 25%).

Rajan *et al.* (1989) studied low level alkaline pretreatment and reported that the process significantly increased the degree of WAS solubilization. Both sodium hydroxide and lime were studied, with sodium hydroxide yielding higher solubilization levels than lime. Alkali concentrations ranging from 5 meq/L to 80 meq/L were used to treat WAS, with TS concentrations from 0.5% to 4.0%, in one litre mesophilic (35 °C) batch reactors. They found that for a 1% TS feed, solubilization increased from 16 to 44% when sodium hydroxide concentrations were increased from 10 to 30 meq/L. Increase in hydrolysis was not significant beyond this sodium hydroxide concentration; with an addition of 80 meq/L of sodium hydroxide, only 48% solubilization was achieved. Comparing sodium hydroxide to lime treatment, hydrolysis was more effective with sodium hydroxide. Solubilization was 14% with 20 meq/L of lime and only 18% with as high as 40 meq/L lime. Concentrations of feed TS significantly influenced the extent of hydrolysis. For a 30 meq/L sodium hydroxide concentration, hydrolysis decreased from 48% to 31% when the feed TS increased from 0.5% to 3.9%.

Ray *et al.* (1990) worked with the same system as Rajan *et al.* (1989) and reported that a 24-hour treatment of 20 meq/L of sodium hydroxide improved VS removals from 25% to 35% over no pretreatment and increased biogas production from 29% to 112% over the control sludge (at HRTs from 5 to 20 days). They found that in high-rate continuously-

fed digesters, VS and COD removals for sodium hydroxide pretreated sludge at a 7.5-day retention time were comparable to the VS and COD removals without pretreatment at a 20 day retention time.

Knezevic *et al.* (1995) also studied alkaline pretreatment of WAS using sodium hydroxide (15 meq/L for 5 hours). Sodium hydroxide pretreatment was found to enhance anaerobic digestion but not to the extent reported by Ray *et al.* (1990). Using 160-litre pilot-scale, high-rate continuously fed anaerobic digesters, they found that WAS pretreatment enhanced VS reduction (0 to 9% higher than the control), COD removals (0 to 35% higher than the control) and biogas production (29 to 66% higher than the control). Overall, the addition of sodium hydroxide to WAS allowed for a decrease in SRT from 25 to 10 days, without losing overall digester efficiency. However, they reported that, at alkali levels lower than 15 meq/L, the beneficial effects of alkaline pretreatment were not observed.

The positive effect of alkaline pretreatment on anaerobic biodegradability has been clearly demonstrated, however, a cost-benefit analysis of alkaline pretreatment at higher dosages has not yet been performed. Further studies with sodium hydroxide pretreatments at higher levels need to be performed to determine the effectiveness of the pretreatment in light of the required cost for the chemical. Also, based on these preliminary studies, the total solids content of the feed was found to have a significant effect on the effectiveness on the alkaline hydrolysis. However, studies were performed at TS concentrations up to only 10%; further studies with feed TS concentrations of up to 25% need to be performed to assess the effectiveness of alkaline pretreatment on typical HSAD processes.

4.1.2 Thermal Pretreatment

Haug *et al.* (1978) studied the effect of thermal pretreatment on the anaerobic biodegradability of RAW and WAS. Their proposed thermal pretreatment/anaerobic digestion system for processing RAW and WAS is shown in Figure 28. Within this design, residual heat in the sludge was sufficient for both mesophilic and thermophilic

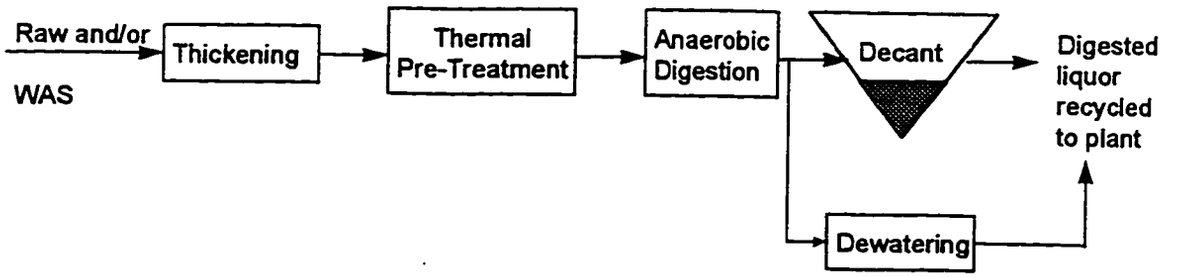


Figure 28: Thermal Pretreatment/Anaerobic Digestion System for Processing RAW and WAS (Haug *et al.*, 1978)

digestion without additional energy requirements. Lab-scale testing in two litre continuous-stirred-tank reactors was performed with 15-day retention times. The thermal pretreatment involved heating the sludge to 175 °C for 0.5 hours. They found that thermal pretreatment had no significant effect on either total biogas production or methane production for RAW. For thermally pretreated WAS, an initial increase in biogas production was observed (88% greater than the control) for approximately eight days followed by a period of inhibition, with biogas production rates only 18% greater than the control. However, after about 30 days, the reactors appeared to acclimate to the feed with biogas production at levels 170% greater than the control. Further testing (Haug *et al.*, 1983) in a pilot-scale facility (45.4 m^3) indicated no inhibition, however, digester performance was not as significantly affected by thermal pretreatment. When a 1:1 volumetric mixture of WAS and RAW was studied, an increase of 14% in biogas production was observed with no periods of inhibition. The effect of pretreatment time was also studied. They found that increased pretreatment time increased biodegradability up until one hour, after which it appeared to have little effect. They also found increased digester performance at increasing pretreatment temperatures up to 175 °C after which production of inhibitory materials adversely affected digester performance.

Hiraoka *et al.* (1984) studied the effectiveness of the treatment of WAS with thermal pretreatment at comparatively lower temperatures, 60 °C to 100 °C. They found an increase of 40% to 50% in biogas production in mesophilic (35 °C) digesters semi-continuously fed (daily). Based on the lab results, a pilot-scale 20 m^3 egg-shaped digester was used to treat a mixture of TWAS and RAW. The TWAS was thermally pretreated at

60 °C for 2 hours. The reactor was operated at a 20-day HRT and organic loading rates of 1 to 1.5 kg of VS per m^3 reactor per day. Biogas production with thermal pretreatment was 0.44 m^3 per m^3 reactor per day whereas a control yielded 0.29 m^3 per m^3 reactor per day. They also demonstrated increased biogas production with increasing TS contents.

"Thermochemical" liquidization of a sludge mixture of RAW and WAS was performed by Sawayama *et al.* (1995). The "thermochemical" liquidization process uses thermal pretreatment to convert sludge into a slurry thereby facilitating pumping. The sludges were heated at 175 °C for one hour and centrifuged at 1000 g for 10 minutes. Two-litre digesters were used at 35 °C at organic loading rates of 1.8 to 2.2 kg of VS per m^3 reactor per day. Anaerobic digestion of the supernatant, particulate fraction and the control (untreated sludge) resulted in biogas productions of 0.55, 0.48 and 0.31 m^3 per m^3 reactor per day respectively. Based on the experimental design, it is difficult to attribute the increase in biogas production to thermal pretreatment; the solid-liquid separation may have also enhanced biodegradability by allowing the microbes to acclimate to more homogeneous feeds.

Thermal pretreatment appears to enhance anaerobic biodegradability of various substrates, but does not seem to be as effective as alkaline pretreatment. Given the varying performances with different substrates, and since no studies have been performed on mixtures of OFMSW and sewage sludge (RAW/TWAS), studies on this mixture should be performed to determine the effectiveness of thermal pretreatment.

4.1.3 Thermochemical Pretreatment

The work by Stuckey and McCarty (1984) evaluated the effect of thermal pretreatment, with and without chemical addition, on the dewaterability and anaerobic digestibility of RAW and WAS. The objective of the study was to understand, at a more fundamental level, the refractory nature of WAS. Hence, their study involved an evaluation of the degradability and toxicity of pure nitrogenous organic compounds present in WAS both

individually and jointly. The effect of thermal pretreatment of these compounds was also assessed to see if the observed behaviour of WAS could be explained from the behaviour of its simpler constituents. They found that, in batch reactors, an optimal pretreatment temperature for bioconvertibility was approximately 175 °C. The effect of alkali addition was insignificant around the optimal temperature, although at higher temperatures it seemed to prevent the sharp decline in bioconvertibility observed in its absence. The effect of pretreatment at 200 °C, with and without sodium hydroxide, was to reduce biodegradability considerably, under both mesophilic and thermophilic conditions. However, pretreatment affected each of the mixtures differently. Collagen bioconvertibility was not affected when sodium hydroxide was used, and only decreased slightly with 200 °C pretreatment alone. The bioconvertibility of other mixtures was decreased in the order: amino acids, RNA and DNA. They indicated that the production of melanoidins (brown nitrogen copolymers) and humic acids can occur during thermochemical pretreatment via the browning reaction. Both these substances are known to be refractory, and could contribute to the decrease in degradability. Overall, the effect of added alkali on bioconvertibility in almost all cases was not significant.

Gossett *et al.*(1982b) studied thermal and alkaline pretreatment of MSW. They studied the effect of solids concentration on biodegradability and consumption of added alkali. Total solids concentrations of 0.79%, 2.25%, 5.15%, 7.43% and 10.0% were treated at each of three alkali concentrations (75 meq/L, 150 meq/L and 300 meq/L sodium hydroxide). Treatments lasted for one hour, in each case, at 25 °C, 133 °C and 200 °C. They found that solubilization was a function of the caustic-to-solids ratio (g NaOH per g TS added). Given these results, costs for a given total mass of solid material requiring treatment can be reduced by treating higher solids concentrations. In semi-continuous digestion studies, treatment was at 200 °C for one hour at an alkali concentration of 185 meq/L using both lime and sodium hydroxide. They also evaluated effluent heat treatment with recycle as opposed to straight-through heat treatment of the feed; solids loading under comparable heat treatment conditions; and, elevated sodium levels on digester performance. Experiments were run in 1.5-litre batch digesters. They reported that, in all

but one of the cases, thermochemical pretreatment resulted in significant increases in methane yields. However, from the VFA (ranging from 1100 mg/L to 2000 mg/L) data, it appeared that the digesters receiving pretreated MSW were being stressed. Biogas data did not show any significant acclimation to the inhibitory substances even after long periods of time. Initially, sodium inhibition was identified as the most likely cause for the reduced performance, however a control run with sodium chloride indicated that the sodium levels had no effect on the performance of the digesters. Gossett *et al.* (1982b) stated that the performance and behaviour of the digesters could largely be explained by the effects of lignin inhibition. Since the alkaline-heat-treated lignin were in concentrations of over 1 g/L (the threshold concentration before inhibition), the anaerobic microorganisms were most likely lignin-inhibited.

Gossett and Belser (1982) studied the thermochemical pretreatment of MSW at different pHs. They found an optimum pretreatment temperature for each pH level: pH 1 (130 °C), pH 3 (175 °C), pH 7 (185 °C), pH 11 (185 °C) and pH 13 (200 °C). They reported that a reduction in pretreatment time tended to shift the optimum to higher temperatures. Overall, the effect of thermal pretreatment on biodegradability was relatively low; the maximum increase in biodegradability was found at pH 13, where the biodegradability increased by 15% over the control after treatment for one hour at 200 °C.

Gossett *et al.* (1982b) also reported that recycling of heat-treated effluent can lead to significant increases in methane production with semi-continuous digestion; biogas production with recycling was reported to be 40% greater than without recycle. Under the conditions studied, this increase was reported to depend on both the concentration of heat-treated lignin in the reactor and the chemical used in the heat treatment. Comparing lime to sodium hydroxide treatments, they reported biogas productions of 17% lower for sodium hydroxide than for lime. Possibly the presence of calcium ion favors formation of different treatment products than are formed in its absence. For example, in the alkaline degradation of 4-0-substituted glucose derivatives (4-0-methyl-D-glucose, maltose, amylose and cellulose), lime favors the formation of D-glucoisosaccharinic acid whereas

fragmentation predominates with sodium hydroxide (Gossett and Belser, 1982). Gossett and Besler (1982) proposed that lime treatment would hence provide a higher inherent biodegradability (due to the lesser degree of inhibition) than does sodium hydroxide treatment due to product differences. However, the apparent increase in biodegradability may simply be due to calcium's ability to precipitate many of the inhibiting compounds, rendering them less of a problem in subsequent digestion.

Based on their findings from both the batch and semi-continuous experiments, Gossett *et al.* (1982b) proposed a two-stage anaerobic digestion process, readily degradable organics being digested in the first digester with the effluent thermochemically treated (to solubilize the remaining organics) and fed to a second digester. They treated a mixture of newsprint and sewage sludge in continuously stirred reactors operated mesophilically (35 °C) with a 15-day retention time. Thermochemical treatment had a significant effect on the performance of the second digester, in a control the methane production was 7 mL/day, the thermochemical (1:1 dilution of water to feed) 76 mL/d and the thermochemical (3:1 dilution of water to feed) 217 mL/d. The lower methane production in the more concentrated mixture of heat-treated feed indicated that some inhibition caused by heat-treated lignin may have resulted. This is also supported by a higher VFA (750 mg/L compared to 140 mg/L) concentration in the concentrated unit.

WAS thermochemical pretreatment was studied by Haug *et al.* (1978) in two-litre continuous-stirred-tank reactors. They adjusted the pH to 12 through the addition of 250 meq/L of sodium hydroxide and thermally pre-treated the sludge at 175 °C. The data indicated an initially high performance (biogas production peaking at 900 mL/d at 5 days) and then severely inhibited behaviour (biogas production at 200 mL/d at 43 days). No acclimation to the inhibitory compounds was observed within 43 days. At 43 days, the biogas production was less than 15% of the control and VFAs were as high as 4500 mg/L.

As in the case for thermal pretreatment, reactor performance appears to be dependent on the feed substrate. Hence studies on the OFMSW/sewage sludge mixture are critical to establish the effectiveness of thermochemical pretreatment. The alkaline concentrations in the studies reported in this section were significantly higher than those reported in section 4.1.3 for alkaline pretreatment. Further studies need to be performed at lower alkali levels in thermochemical pretreatment to provide the framework for a more complete design base.

The purpose of this study is to optimize the effectiveness of all three pretreatments specific to the anaerobic co-digestion of OFMSW and RAW/TWAS sewage sludge mixture in the RMOC. To complement the studies reported in the literature, both alkaline and thermochemical pretreatments were performed from the low alkali levels reported for alkaline pretreatments to the high levels reported for thermochemical pretreatments. Through the development of empirical models, optimal operating conditions with respect to feed total solids, its particle size and alkaline dosage were identified based on several response variables: biogas production, biogas methane concentration, and the reduction of chemical oxygen demand, and volatile and total solids concentrations. The experimental design for the analysis of thermal pretreatment is consistent with that presented in Chapter 3. The alkaline and thermochemical pretreatments involved similar experimental designs through the identification of optimal operating levels of feed total solids concentration and particle size as well as alkaline dosage. Larger-scale studies were performed to explore the inconsistencies in reactor performance based on reactor size observed by Haug *et al.* (1978) for thermal pretreatment as discussed in section 4.1.2. Large-scale studies were performed for all three pretreatments as well as for an untreated feed.

4.2 Materials and Methods

4.2.1 Analytical Methods

The analytical methods included the same measurements as presented in section 3.2.1. For detailed descriptions, the methods are presented in Appendix B.

4.2.2 Feedstock Materials

The same feed materials as presented in section 3.2.2 were used throughout the experiments.

4.2.3 Experimental Protocols

Long-term batch tests similar to the ones presented in section 3.2.3, were performed to study the effects of the total solids content of the feed and its particle size on the effectiveness of the thermal pretreatment. The experimental design for the thermal pretreatment involved a two-factor central composite design as described in Appendix C.2. For the alkaline and thermochemical pretreatments, three factors were studied: alkaline dosage, the total solids content of the feed and particle size. The central composite experimental design, described in Appendix C.3, was used for both of these pretreatments. These long-term batch tests were run at five levels of each of the two factors studied: particle sizes ranged from 0.85 mm to 8 mm and total solids contents ranged from 7.0% to 23.4%. The tests performed at 37 °C were based on the 650 mL of optimal 25% OFMSW mixture identified in the optimization study of section 3.3.2, the 150 mL of ROPEC anaerobic inoculum and 2.4 g of sodium bicarbonate. Throughout the experiments, initial and final measurements of soluble COD, VS, TS and daily biogas production readings were reported. The final pH, VFA and biogas methane concentration were also measured. To develop empirical models to describe the effects of particle size and TS on these response variables, a least-squares analysis was performed using STATISTICA Release 4.5. The development of the experimental design and model building methods are presented in Appendix C.

To evaluate all three pretreatments at larger scale, four semi-continuously fed reactors (20 litre working volume) were operated mesophilically (37 °C) at a 20 day HRT. These reactors were 0.18 m wide, 0.18 m deep and 1.20 m high and were fed one litre of substrate daily. Each reactor was fed through a hole in the lid of the reactor and reactor contents were withdrawn from a sampling port 0.25 cm from the bottom of the reactor. Mixing of the reactor contents was achieved by recycling biogas from the top of the

reactor and re-introducing it at the bottom of the reactor. To ensure the reactors achieved steady state conditions, the reactors were operated for 3 HRT's, that is, 60 days. The reactors were started up with four litres of anaerobic seed sludge (2.4% VS) from the anaerobic digesters at ROPEC and 15 litres of the appropriate mixture. The alkaline pretreatment consisted of an alkaline dosage of 185 meq/L for a period of 24 hours. The thermal pretreatment involved heating in an autoclave to 130 °C for one hour at an autoclave pressure of 15 psig. The thermochemical pretreatment involved the thermal treatment followed by the alkaline pretreatment. The feed to the reactor had a TS concentration of 10% and was passed through a 6.35 mm pore-size sieve to ensure particle sizes of no greater than 6.35 mm.

4.3 Results and Discussion

4.3.1 Batch Tests

Details of the experimental design are presented in Appendix C and the raw data can be found in Appendices D.5 through D.7 for the thermal, thermochemical and alkaline pretreatments respectively. The development of the empirical models for each of the pretreatments and corresponding response variables are also respectively presented in Appendices F through H. Results from the initial full second order polynomial and the final reduced models are included. A model was deemed finalized when insignificant (p-value greater than 0.05) parameters were removed.

Table 5 is a summary of the model parameters and ANOVA results presented in Appendices E through H (Appendix E contains data for the control run. For each of the parameters, the 95% confidence intervals are included. All models displayed no significant lack of fit based on internal lack-of-fit tests (95% confidence level) and plots of model residuals. Based on R^2-values and tests of the significance of the regression all but four models were found to account for more than 70% of the variability in the data. Fifteen of the 21 remaining models explained more than 90% of the variability in the data. The pertinent statistics are included in Table 5.

Table 5: Summary of Parameter Estimates with 95% Confidence Limits and Measures of Model Adequacy for the Control, Thermal, Alkaline and Thermochemical Pretreatments

		Model Response	*Parameter Estimates ± 95% Confidence Limits*					
Pretreatment	***Parameter***	***Gas Prod.***	***Gas Prod./VSa***	***Gas Prod./VSr***	***sCOD Removal.***	***VS Removal***	***TS Removal***	***Methane Conc.***
		L	***L/g VSa***	***L/g VSr***	***%***	***%***	***%***	***%***
Control	β_0	4.29 ± .31	0.58 ± .04	1.05 ± .04	77.91 ± 8.03	53.14 ± 2.50	33.51 ± 1.06	61.71 ± 1.56
	β_1^*	-0.35 ± .26	-0.04 ± .03	-0.07 ± .03	---	---	---	---
	β_2^{**}	1.77 ± .30	0.07 ± .04	-0.16 ± .04	13.02 ± 7.33	13.03 ± 2.42	8.56 ± 1.03	---
	β_{11}	-0.35 ± .21	-0.04 ± .03	---	---	-3.72 ± 1.64	-2.45 ± .70	---
	β_{22}	---	---	0.08 ± .04	-9.00 ± 8.03	---	---	---
	β_{12}	-0.63 ± .52	-0.06 ± .05	---	---	-4.16 ± 3.14	-3.02 ± 1.33	---
	R^2	0.973	0.907	0.950	0.715	0.959	0.983	---
	F_{reg}	2620.14	986.74	7834.22	285.09	4608.25	10419.29	---
	$F_{.05,v_1,v_2}$	4.12	4.12	4.07	4.39	4.07	4.07	---
	F_{lof}	2.27	4.14	0.94	3.38	1.94	2.95	---
	$F_{.05,v_1,v_2}$	9.12	9.12	9.01	9.28	9.01	9.01	---
Thermal	β_0	3.51 ± .38	0.49 ± .04	0.93 ± .10	80.32 ± 7.59	55.54 ± 2.41	34.89 ± 1.22	59.35 ± 3.04
	β_1	0.44 ± .29	0.06 ± .03	0.13 ± .07	---	---	---	---
	β_2	1.39 ± .34	---	-0.28 ± .07	14.20 ± 6.93	13.23 ± 2.33	9.52 ± 1.18	---
	β_{11}	---	---	0.06 ± .05	---	-4.29 ± 1.58	-3.17 ± .80	---
	β_{22}	0.50 ± .38	0.07 ± .04	0.15 ± .08	-8.46 ± 7.59	---	---	---
	β_{12}	---	---	0.13 ± .09	---	-4.37 ± 3.02	-7.04 ± 1.53	---
	R^2	0.934	0.750	0.960	0.770	0.966	0.985	---
	F_{reg}	3945.56	594.02	2278.47	540.39	42180.76	9242.80	---
	$F_{.05,v_1,v_2}$	4.07	4.26	4.39	4.26	4.07	4.39	---
	F_{lof}	1.84	1.33	1.44	1.51	2.04	1.34	---
	$F_{.05,v_1,v_2}$	9.01	8.94	9.28	8.94	9.01	9.28	---

* Factor 1 corresponds to coded particle size ** Factor 2 corresponds to coded TS concentration *** Factor 3 corresponds to coded alkaline dose

Table 5: (cont.)	Parameter	Gas Prod.	Gas Prod./VSa	Gas Prod./VSr	sCOD Removal.	VS Removal	TS Removal	Meth. Conc.
		L	L/g VSa	L/g VSr	%	%	%	%
Thermochemical	β_0	2.61 ± .07	0.36 ± .02	1.11 ± .14	47.61 ± 3.53	24.49 ± 1.14	37.46 ± 1.42	58.84 ± .87
	β_1	0.38 ± .07	0.04 ± .01	0.16 ± .12	---	---	---	1.08 ± .98
	β_2	1.02 ± .07	---	-0.14 ± .13	---	3.97 ± .90	4.84 ± 1.12	---
	β_3	-0.17 ± .07	-0.02 ± .01	0.27 ± .13	-6.97 ± 4.27	-5.42 ± .90	-8.57 ± 1.12	---
	β_{11}	---	---	---	---	-1.71 ± .73	-2.21 ± .91	---
	β_{22}	0.38 ± .07	0.05 ± .01	0.17 ± .12	---	---	---	---
	β_{33}	---	---	---	---	-2.07 ± .87	-2.10 ± 1.08	---
	β_{12}	0.19 ± .08	0.02 ± .02	---	---	-1.33 ± 1.08	---	---
	β_{13}	---	---	---	---	---	---	---
	β_{23}	---	---	---	---	-3.09 ± 1.17	-3.69 ± 1.46	---
	R^2	0.988	0.879	0.712	0.375	0.959	0.964	0.216
	F_{reg}	235.13	27.34	9.27	10.81	50.26	75.20	4.97
	$F_{.05,v_1,v_2}$	2.96	3.06	2.96	4.41	2.92	2.96	4.41
	F_{lof}	4.77	3.99	2.65	1.03	1.80	1.22	1.42
	$F_{.05,v_1,v_2}$	4.77	4.74	4.77	4.66	4.82	4.77	4.64
Alkaline	β_0	4.03 ± .18	0.48 ± .04	0.54 ± .07	56.82 ± 3.88	83.76 ± 2.08	51.32 ± 1.84	58.87 ± 1.80
	β_1	0.54 ± .14	0.04 ± .03	0.06 ± .05	---	---	---	---
	β_2	1.35 ± .14	---	-0.06 ± .05	---	3.77 ± 1.97	4.68 ± 1.74	---
	β_3	0.16 ± .14	---	0.15 ± .05	-8.11 ± 4.69	-11.43 ± 1.97	-8.39 ± 1.74	---
	β_{11}	-0.15 ± .12	---	---	---	---	---	---
	β_{22}	0.34 ± .13	0.05 ± .04	0.08 ± .05	---	---	---	---
	β_{33}	---	---	0.09 ± .05	---	-4.93 ± 1.90	-3.10 ± 1.68	---
	β_{12}	0.46 ± .17	0.05 ± .04	0.08 ± .06	---	---	---	---
	β_{13}	---	---	---	---	---	---	---
	β_{23}	---	---	---	-6.07 ± 6.13	---	-3.10 ± 2.27	---
	R^2	0.976	0.571	0.840	0.487	0.918	0.906	---
	F_{reg}	87.31	7.10	11.33	8.06	59.84	36.28	---
	$F_{.05,v_1,v_2}$	3.02	3.02	3.02	3.02	3.02	3.02	---
	F_{lof}	5.01	1.48	5.01	1.31	2.72	1.61	---
	$F_{.05,v_1,v_2}$	5.05	5.05	5.05	5.05	5.05	5.05	---

4.3.1.1 Empirical Models for the Alkaline Pretreatment

The results for the control are presented in section 3.3.4. For all of the response variables, the residuals (i.e., differences between observed and predicted values of the responses) appeared normally distributed with constant variance. Consider the case for the cumulative biogas production, a typical analysis of the residuals is shown by Figures 29 and 30. The residuals in the normal probability plot of Figure 29 follow the linear trend expected for a normal distribution. The residual plot in Figure 30 does not display any significant trends indicating constant variance.

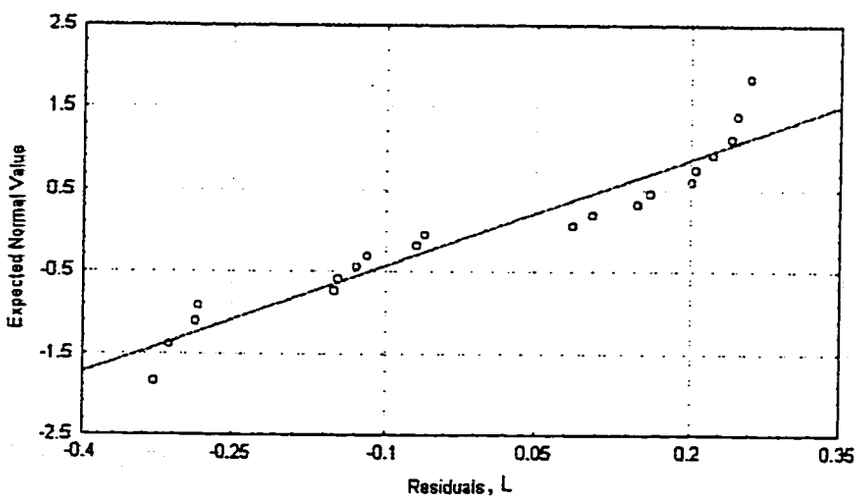


Figure 29: Normal Probability Plot for Alkaline Pretreatment Cumulative Biogas Production Model

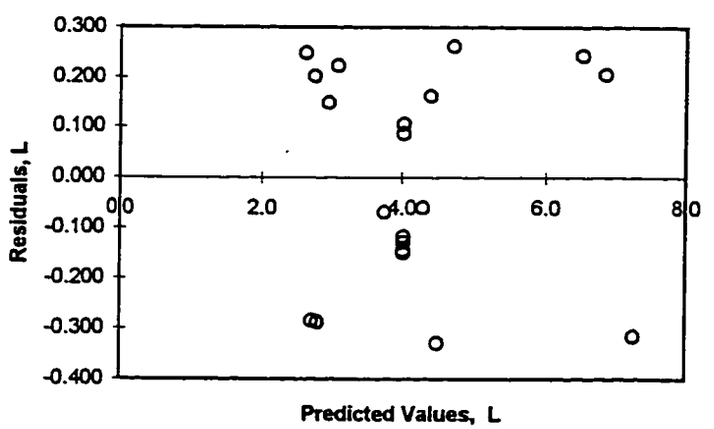
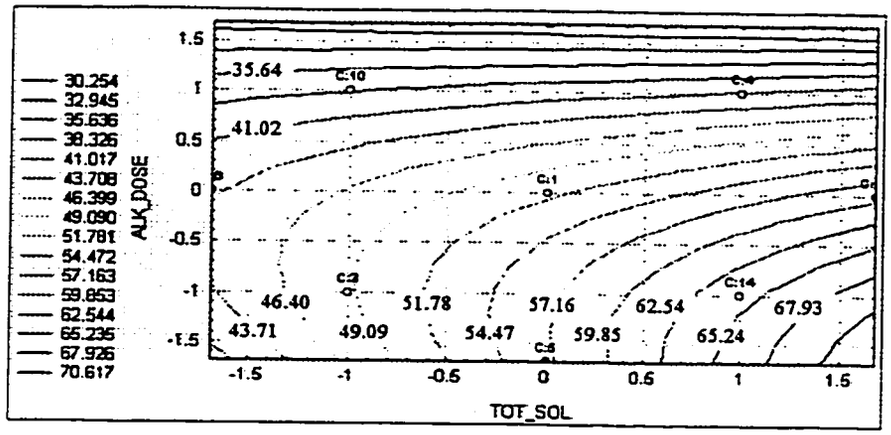


Figure 30: Residual Plot for Alkaline Pretreatment Cumulative Biogas Production Model Based on Total Solids Concentration and Particle Size of the Feed

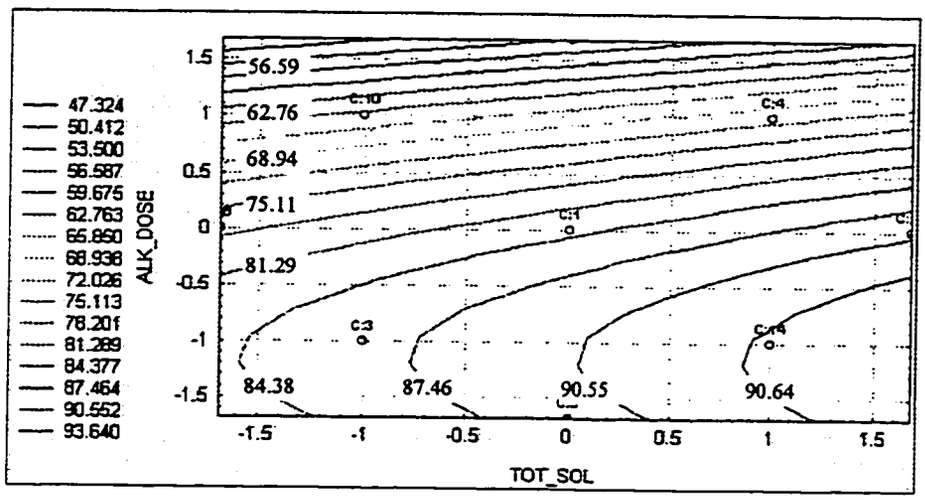
A summary of contour plots for the alkaline pretreatment models are shown in Figure 31. There is a significant difference between the models for the cumulative biogas productions. The cumulative biogas production model and the cumulative biogas production with respect to volatile solids removal were both dependent on all three experimental factors (particle size, TS concentration of the feed and alkaline dose) studied, however, the cumulative biogas production with respect to volatile solids addition was found to be independent of particle size and alkaline dosage. However, the r-squared value for the model with respect to volatile solids addition was very low (0.571). This implies that the pure error associated with the measurement of biogas production is higher or comparable to the variance in the model. The other two biogas models indicate an optimum at high TS (23.4%), large particle sizes (8 mm) and high alkaline doses (185 meq/L). This combination maximizes the alkaline dose on as concentrated a feed as possible leading to the higher biogas production capabilities. However, the reversal in the optima between the control and this pretreatment with respect to particle size may be due to the inability of methanogens to handle the increased hydrolytic activity.

The model for soluble chemical oxygen demand removal also had a low R-squared value (0.49) and hence the usefulness of the model is limited. The high error associated with the COD test is most likely the cause for the low R-squared value. This model was found to be dependent on only feed TS and alkaline dose with an optimum at low alkaline doses (16 meq/L) and high feed TS (23.4%) as indicated in Figure 31c. This is consistent with the trends observed for the other two indicators of organic content, VS and TS concentration. For the case of VS removal, the model also indicated a dependence on alkaline dose and feed TS but with a much higher accountability for variance in the data (R-squared value of 0.918). Increasing reductions of organic content with increasing feed TS is largely due to the increased accessibility of more substrate for the anaerobic bacteria. The process appears to be under optimal operating conditions at low alkaline doses (16 meq/L) with respect to organic content consumption, however, this is masked by the fact that at high alkaline doses, a large proportion of the biodegradable organics are solubilized leading to high biogas productions but differences in solids concentrations as measured by TS and

a. TS Removal (%)



b. VS Removal (%)



c. Soluble COD Removal (%)

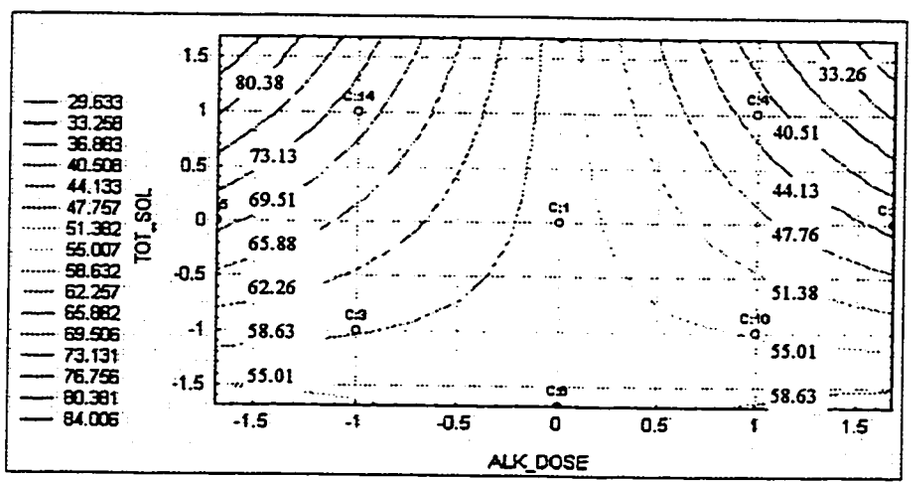


Figure 31: Contour Plots for Alkaline Pretreatment Models of the Effect of Alkaline Dosage, TS Concentration and Particle Size on Various Response Variables
Legend: TOT_SOL=coded TS concentration, ALK_DOSE=coded particle size

d. Biogas Production with Respect to Volatile Solids Addition (L/g VSa)

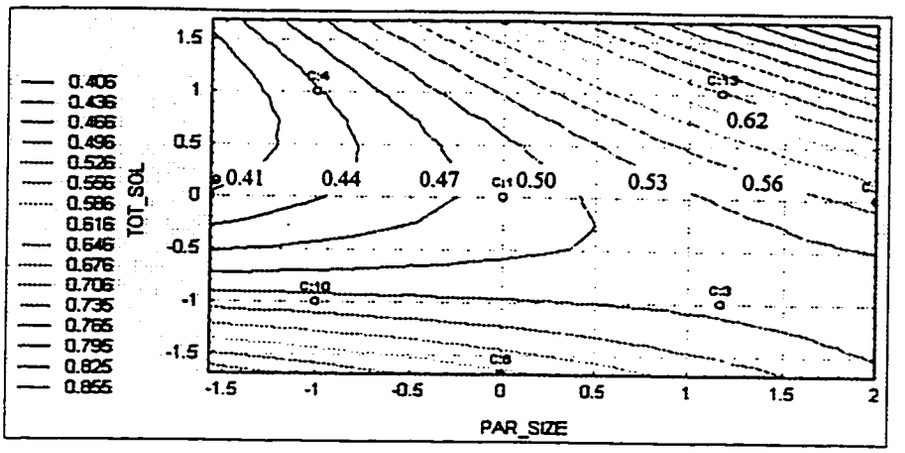


Figure 31(cont.): Contour Plots for Alkaline Pretreatment Models of the Effect of Alkaline Dosage, TS Concentration and Particle Size on Various Response Variables
Legend: TOT_SOL=coded TS concentration, PAR_SIZE=coded particle size

VS are significantly reduced since most of the biodegradable components are solubilized through the alkaline pretreatment. At low (16 meq/L) alkaline doses, a reduction in TS and VS is more pronounced due to the limited solubilization of the feed substrate. This argument is reflected in Figure 31, where there appears to be a strong interaction between the TS and alkaline dose.

As expected, the biogas methane concentration was found to be independent of all three experimental factors studied. The concentration of methane in the biogas should be constant and indicate the metabolic state of the biomass in the reactor. If the anaerobes are active then biogas is produced reflecting the stoichiometric ratios of the biological reaction. Hence, the biogas methane concentration should be constant and independent of the factors studied. Based on the initial inoculum used in the reactors, the maximum specific activity achieved, assuming that 50% of the VS is biomass, was found to be 0.46 g COD removed per g biomass per day based on the optimal cumulative biogas production. The soluble COD removal, in all cases was found to be independent of feed particle size. This is to be expected since the soluble COD concentration is independent of the characteristics of the particulate fraction of the feed.

4.3.1.2 Empirical Models for the Thermal Pretreatment

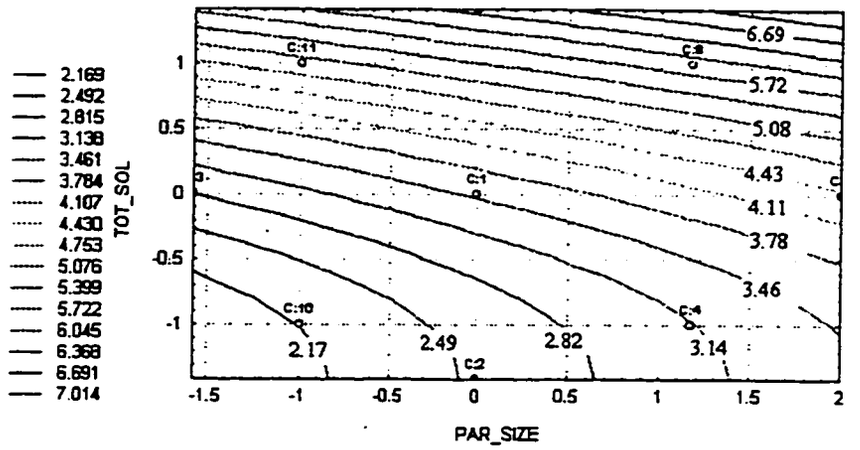
For each of the thermal pretreatment models, the data was found to be normally distributed, with constant variance as indicated in Appendix F. A summary of the contour plots for all of the thermal pretreatment models is shown in Figure 32 and the control results are presented in section 3.3.4.

All cumulative gas production models (Figures 32a, 32b, 32d) were found to be dependent on both particle size and feed TS concentration. As for the alkaline pretreament case, the cumulative biogas production with respect to volatile solids addition had a significantly lower R-squared value (0.750). For all three responses, the optima are at high feed TS concentrations (22.1%). This again maximizes the amount of substrate available per unit volume of feed to the reactor. Each model responds differently to particle size but in all cases, the optima were at large particle sizes (8.0 mm) consistent with the alkaline model. The parabolic trend observed in the case of the control is also apparent in the thermal models.

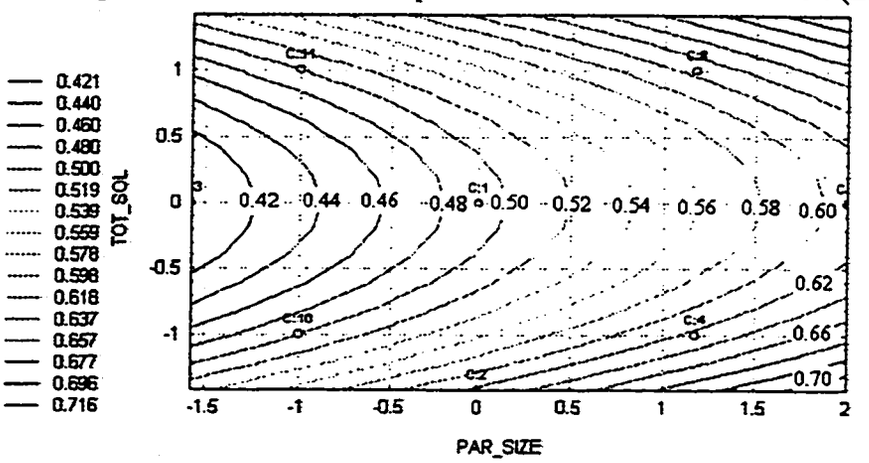
However, the cumulative biogas production model (Figure 32a) indicates a limited effect of particle size on process performance as compared to TS concentration of the feed. Based on the initial inoculum used in the reactors, the maximum specific activity achieved, assuming that 50% of the VS is biomass, was found to be 0.32 g COD removed per g biomass per day based on the optimal cumulative biogas production.

The soluble COD reduction model, consistent with the other soluble COD models, had a low R-squared value of 0.77. The model was found to be solely dependent on feed total solids content as indicated in Figure 32f. The model's independence from particle size is founded since the change in soluble COD would not be expected to be related to the particulate fraction assuming that all of the particulate fraction that is solubilized is digested.

a. Cumulative Biogas Production (L)



b. Cumulative Biogas Production with Respect to Volatile Solids Addition (L/g VSa)



c. Total Solids Removal (%)

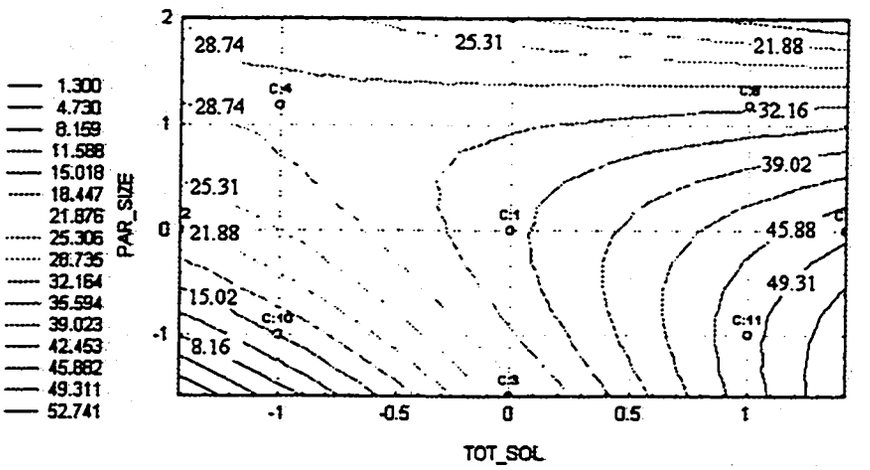
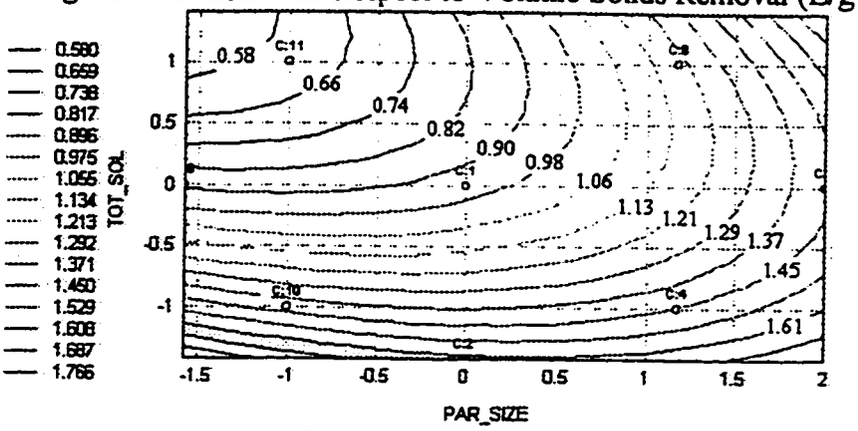
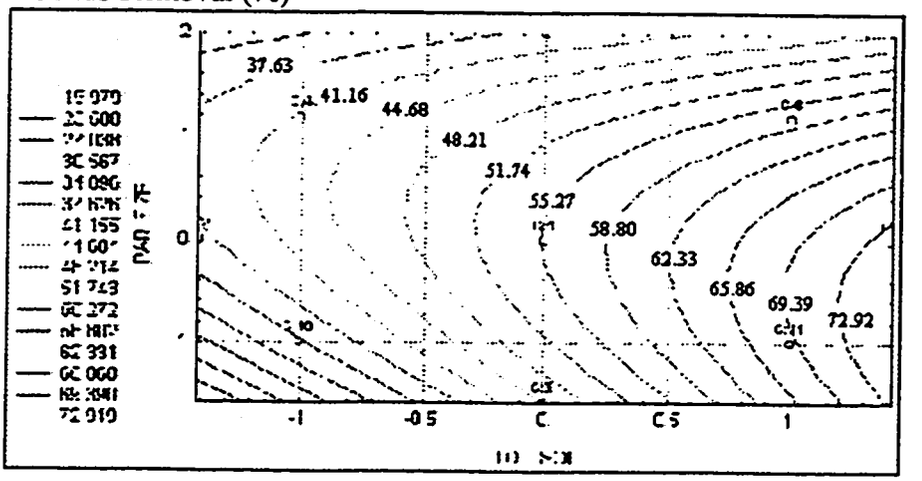


Figure 32: Contour Plots for Thermal Pretreatment Models of the Effect of Total Solids Concentration and Particle Size of the Feed on Various Response Variables
LEGEND: TOT_SOL= Coded TS Concentration, PAR_SIZE= Coded Particle Size
COD_RED= Coded Soluble COD Removal

d. Cumulative Biogas Production with Respect to Volatile Solids Removal (L/g VSr)



e. Volatile Solids Removal (%)



f. Soluble COD Removal (%)

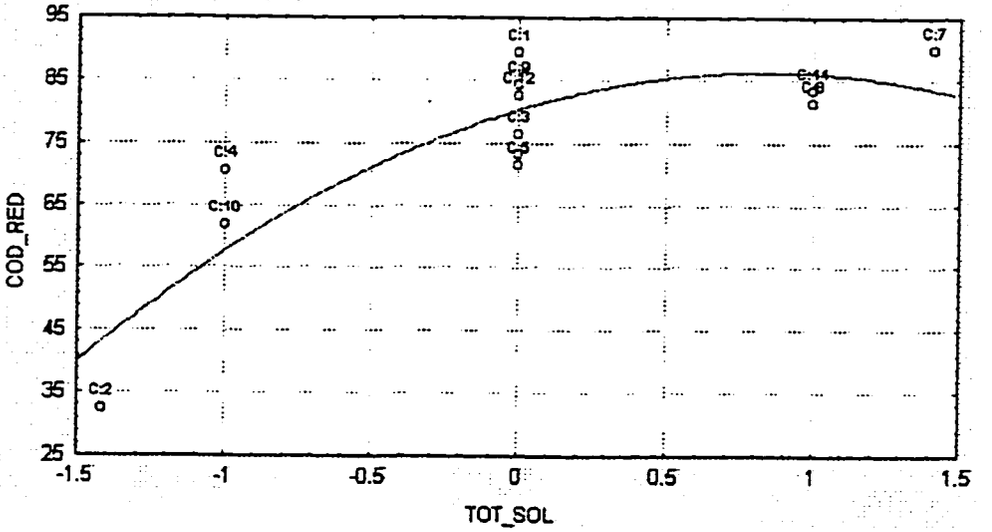


Figure 32 (cont.): Contour Plots for Thermal Pretreatment Models of the Effect of Total Solids Concentration and Particle Size of the Feed on Various Response Variables
LEGEND: TOT_SOL=Coded TS Concentration, PAR_SIZE= Coded Particle Size

The models for VS and TS reduction have very similar contour plots as shown in Figures 32c and 32e. The quadratic profile of the VS reduction model appears dampened in the TS reduction model. This is expected since it is the VS fraction of the TS that is anaerobically digested while the remaining fixed solids in the TS are left undigested. In both cases, optimal operating conditions occur at high feed TS concentrations (23.4%) and mid-range (4.0 mm) particle sizes. This is consistent with the findings for the alkaline pretreatment discussed earlier. Also, the biogas methane concentration was found to be independent of both particle size and feed TS concentrations as observed in the alkaline pretreatment.

4.1.3.3 Empirical Models for the Thermochemical Pretreatment

As in the case for the other pretreatments, all data was found to be normally distributed and of constant variance. All data and plots are presented in Appendix G. All three of the cumulative biogas production, models were dependent on the three factors studied, particle size, TS concentration of the feed and alkaline dose. However, in contrast to the other pretreatments, the cumulative biogas production model with respect to volatile solids addition had a relatively high R-squared value (0.879); it was the model with respect to volatile solids removal that had a lower R-squared value (0.712). The optima for these models were at high feed TS concentrations (23.4%) and large particle sizes (8.0 mm). This trend in the case of TS concentration maximizes available substrate per unit volume and the trend for large particle sizes is consistent with the findings for the other pretreatments studied. The optimum alkaline doses were at mid-range values (4.0 mm). For example, Figures 33 and 34 show the cumulative biogas production profiles for the thermochemical and alkaline pretreatment models respectively. The magnitude of the biogas productions are significantly lower for the thermochemical pretreatment as compared to the alkaline pretreatment. Based on the initial inoculum used in the reactors, the maximum specific activity achieved, assuming that 50% of the VS is biomass, was found to be 0.27 g COD removed per g biomass per day based on the optimal cumulative biogas production.

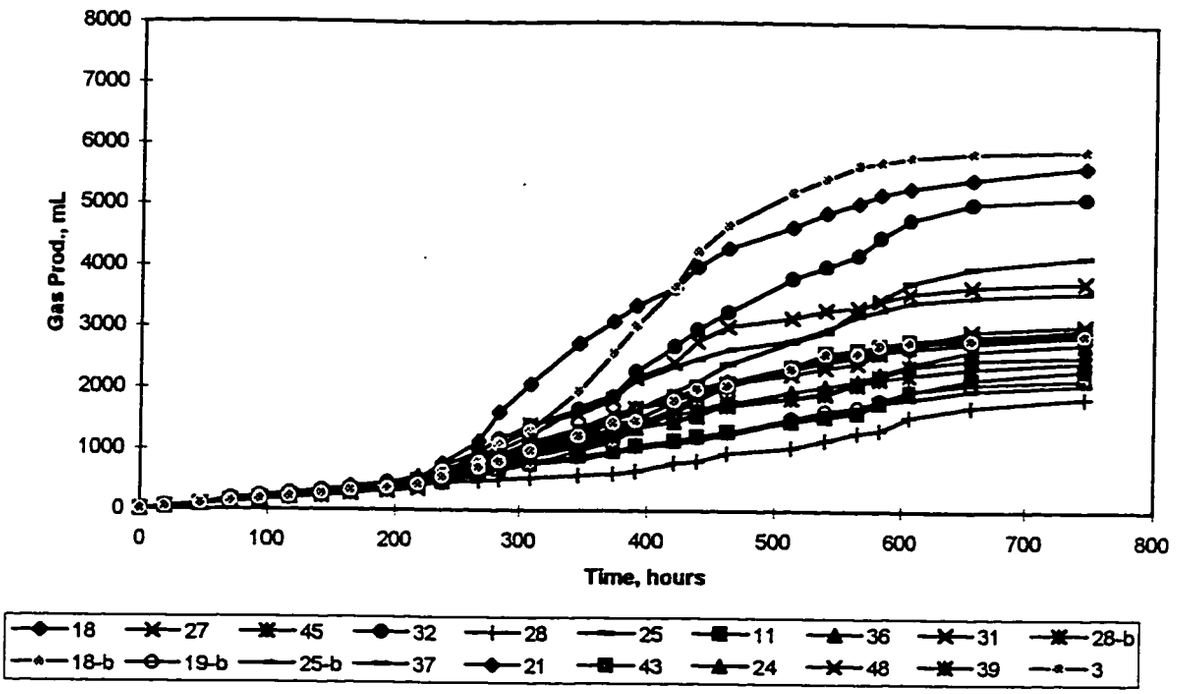


Figure 33: Cumulative Biogas Production Profile for Thermochemically Pretreated Feed for Various Test Bottles

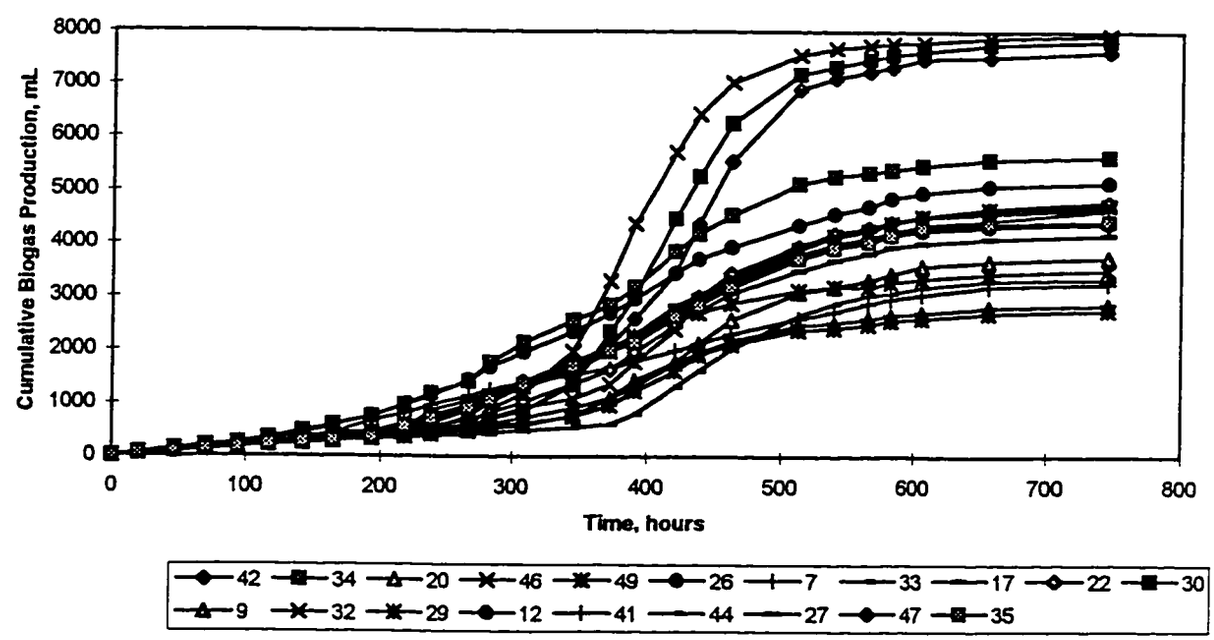


Figure 34: Cumulative Biogas Production Profile for Alkaline Pretreated Feed for Various Test Bottles

The soluble COD reduction model indicated a dependence on only the alkaline dose. The R-squared value for the model was only 0.38 and hence the model was deemed inadequate. Both the VS and TS reduction models indicated a dependence on feed TS, particle size and alkaline dose. Optima for these models are at large particle sizes (8.0 mm), mid-range feed TS (15%) and low alkaline doses (16 meq/L). As such, almost all models developed for thermochemical pretreatment suggest minimizing the alkaline dosage. A slight dependence on particle size was reported in the biogas methane concentration model. The R-squared value for this model was only 0.22 and hence this model was also deemed inappropriate.

4.1.3.4 Comparison of Pretreatments

Table 6 summarizes the optimal operating conditions for each of the response variables for each pretreatment. Overall, models of soluble COD reduction were not found to be useful. Also, biogas methane concentration was found to be independent of feed TS concentration, particle size and alkaline dose. However, optimal conditions were found at high feed TS concentrations. Of the three pretreatments, the alkaline pretreatment had the best overall process performance.

4.3.2 Large-Scale Reactor Testing

To assess the effectiveness of biogas recycling as a means of mixing, a mixing test was first performed in the 20-litre semi-continuous reactors. The reactors were filled with the 25% OFMSW/sludge mixture, and 18 grams of acetic acid were added to the top of the reactor resulting in a reactor acetic acid concentration of 1000 mg/L. Samples were taken at two ports, port A at 25.5 cm from the top of the reactor, and port B at 36.5 cm from the bottom. From Figure 35, the reactor contents appeared to be completely mixed within one minute based on constant acetic acid concentration at both ports.

The specific activities achieved of each of the pretreatments were found to be 0.11, 0.09, 0.09 and 0.08 g of COD removed per g of VS per day for the alkaline, thermal, control and thermochemical reactors, respectively. The cumulative biogas production for each of

Table 6: Optimal Operating Condition for Each of the Pretreatments and the Control Based on the Empirical Models

Pretreatment	Variable	Particle Size (mm)	Total Solids (%)	Alkaline Dose (meq/L)
Control	Gas Production	0.9	22.1	
	Gas Production/VS added	0.9	22.1	
	Gas Production/VS removed	0.9	7.9	
	Soluble COD Removal	—	15.0	
	VS Removal	0.9	22.1	
	TS Removal	0.9	22.1	
	Methane Concentration	—	—	
Thermal	Gas Production	8.0	22.1	
	Gas Production/VS added	8.0/0.9	22.1	
	Gas Production/VS removed	8.0	7.9	
	Soluble COD Removal	—	22.1	
	VS Removal	4.0	22.1	
	TS Removal	0.9	22.1	
	Methane Concentration	—	—	
Thermochemical	Gas Production	8.0	23.4	16.0
	Gas Production/VS added	8.0	23.4	16.0
	Gas Production/VS removed	8.0	15.0	150.0
	Soluble COD Removal	—	—	16.0
	VS Removal	0.9	15.0	16.0
	TS Removal	0.9	23.4	16.0
	Methane Concentration	8.0	—	—
Alkaline	Gas Production	8.0	23.4	150.0
	Gas Production/VS added	8.0	23.4	—
	Gas Production/VS removed	8.0	23.4	150.0
	Soluble COD Removal	—	—	16.0
	VS Removal	—	23.4	16.0
	TS Removal	—	23.4	16.0
	Methane Concentration	—	—	—

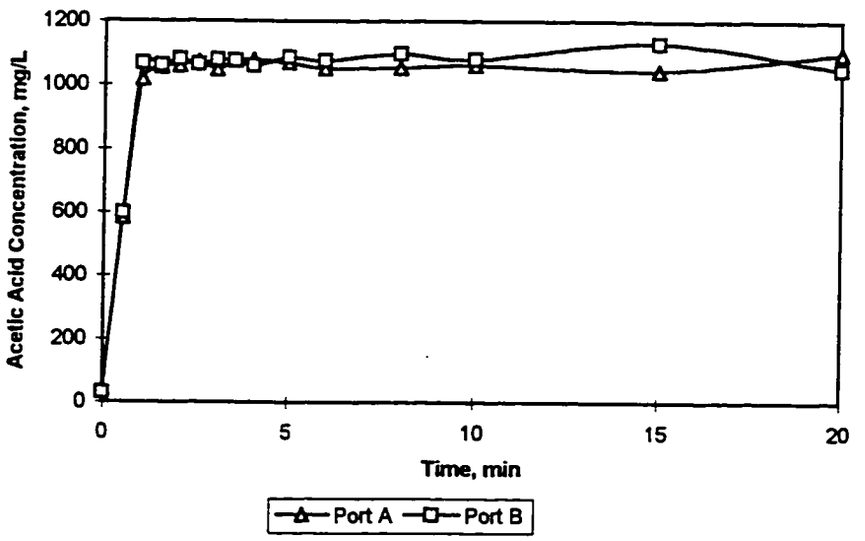


Figure 35: Mixing Test with Acetic Acid in Large-Scale Reactors

the reactors is shown in Figure 36. The plots follow linear trends divided into two regions, the first ends at approximately 200 hours, and the second starts at 200 hours. A linear regression was performed on the data for each of the plots starting at 200 hours. The results are presented in Table 7. From both Figure 36 and Table 7, the alkaline pretreated reactor displayed the highest biogas production (and hence COD removal rate) whereas the thermal, control and thermochemically pretreated mixtures displayed comparable biogas production rates. The hydroytic microbial activity in the alkaline-pretreated reactor was also detected in the relatively higher VFA concentrations (764 mg/L as acetic acid) as compared to the other reactors (maximum of 174 mg/L as acetic acid). The control demonstrated comparable but slightly lower VFA concentrations (169 mg/L as acetic acid). From the soluble COD, TS and VS removal data presented in Table 8, it appears that the alkaline-pretreated reactor performed twice as well as the thermochemically-pretreated reactor. The results reported in Table 8 are from a single reactor and hence

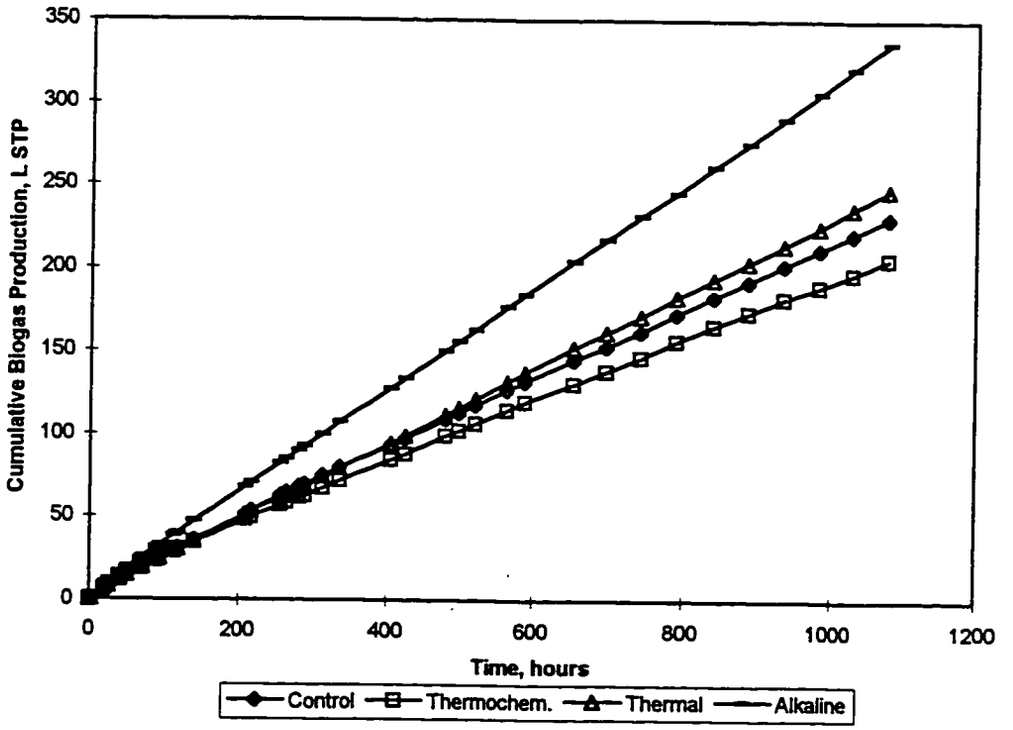


Figure 36: Cumulative Biogas Profile for Each of the Pretreatments in the Large-Scale Reactors

Table 7: Biogas Production Rates for Large-Scale Reactors

Pretreatment	Biogas Production L/hr	COD Removal g COD/hr	R-squared
Alkaline	0.161 ± 0.012	0.46 ± 0.03	0.973
Thermal	0.134 ± 0.011	0.38 ± 0.03	0.971
Control	0.126 ± 0.012	0.36 ± 0.03	0.971
Thermochemical	0.120 ± 0.015	0.34 ± 0.04	0.968

** Based on all data after 200 hours

Table 8: Summary of Soluble COD, TS and VS Percent Removals for the Large-Scale Reactors with the Different Pretreatments

Pretreatment	Soluble COD (%)	TS (%)	VS (%)
Alkaline	52.5	32.9	56.7
Thermal	34.2	19.4	40.4
Thermochemical	25.0	16.2	30.9
Control	46.8	24.9	46.7

there is no estimate of precision. Based on the pH data in Table D.14 (in Appendix D) for the thermochemically-pretreated reactor, the pH was elevated (9.6); on average this reactor was at least one pH unit higher than the other reactors. The bacteria in this reactor are most likely being inhibited by toxic substrates produced during the thermochemical pretreatment as reported by Stuckey *et al.* (1984) or alternatively, the bacteria are not effectively acclimatizing to the feed.

Model predictions, based on the results of the batch tests in section 4.3.1, at the conditions of the large-scale continuous reactors (TS concentration of 10%, particle size of 6.35 mm and alkaline dosage of 185 meq/L) are presented in Table 9. The trends in Table 9 are consistent with those of the large-scale reactor results: alkaline pretreated feed demonstrates the highest anaerobic reactor performance and the thermochemically pretreated, the lowest.

Table 9: Model Predictions for the Large-Scale Operating Conditions

Variable	Control	Thermal	Thermochem	Alkaline
Gas Production (L)	2.4±0.03	3.1±0.08	1.9±0.09	3.2±0.08
Gas Production/VS added (L/mg/L)	0.5±0.07	0.6±0.06	0.4±0.04	0.5±0.04
Gas Production/VS removed (L/mg/L)	1.2±0.06	1.4±0.08	2.1±0.09	1.2±0.07
Soluble COD Removal (%)	55.9±2.8	57.7±3.4	35.9±1.9	53.4±2.4
VS Removal (%)	39.9±1.3	41.5±2.0	39.9±1.7	46.8±2.1
TS Removal (%)	25.1±1.1	29.3±1.4	15.4±0.8	29.0±1.3
Methane Concentration (%)	61.7±4.9	59.4±4.7	60.1±5.2	58.9±4.3

4.4 Conclusions

1. The alkaline pretreated feed performed the best, and the thermochemically pretreated feed performed the worst, displaying inhibited behaviour as compared to the control untreated feed. The maximum specific activities achieved in the batch tests were 0.32, 0.46, 0.32 and 0.27 g COD removed per g biomass per day for the control, alkaline, thermal and thermochemical pretreatments, respectively. For the large-scale reactors the specific activities were 0.09, 0.11, 0.09 and 0.08 g COD removed per g biomass per day respectively.
2. The large-scale continuous reactors and the small-scale batch reactor tests displayed consistent trends in the results observed, that is, biogas production and organics removal.
3. The empirical models developed for biogas production reported in litres were not found to be comparable to biogas production models normalized with respect to VS removal and VS addition.
4. Optimal operating conditions were identified for each of the pretreatments. Overall, the control and thermal pretreatments optimal conditions were at low particle sizes (0.9 mm) and high TS concentrations (22.1%). For thermochemical and alkaline pretreated feeds, the optimal conditions are at high particle sizes (8.0 mm), high TS concentrations (23.4%) and high alkaline doses (150 meq/L).
5. Experiments at higher TS concentrations are required to reflect a wider range of HSAD processes.

CHAPTER 5:
Conclusions and Recommendations

Within the context of typical municipal solid waste and sewage sludge in the RMOC, the anaerobic co-digestion process was found to be highly feasible. In particular, it was found that:

1. Optimal MSW/sludge mixture as reflected by biogas production was 25% OFMSW and 75% RAW/TWAS (in a 60:40 volumetric ratio) by volume;
2. Simulated food waste was the most biodegradable component in the mixture whereas the TWAS and paper waste were the least biodegradable;
3. There are two regions of biological activity throughout the anaerobic co-digestion process, reflected by two classes of biodegradable compounds;
4. The large-scale continuous reactors and the small-scale batch reactor tests displayed consistent trends in the results observed, that is, biogas production and organics removal;
5. The empirical models developed for biogas production reported in litres were not found to be comparable to biogas production normalized with respect to VS removal and VS addition;
6. The alkaline pretreated feed performed the best, and the thermochemically pretreated feed performed the worst, displaying inhibited behaviour as compared to the control untreated feed. The maximum specific activities achieved in the batch tests were 0.32, 0.46, 0.32 and 0.27 g COD removed per g biomass per day for the control, alkaline, thermal and thermochemical pretreatments, respectively. For the large-scale reactors

the specific activities were 0.09, 0.11, 0.09 and 0.08 g COD removed per g biomass per day respectively;

7. Optimal operating conditions were identified for each of the pretreatments. Overall, the control and thermal pretreatments optimal conditions were at low particle sizes (0.9 mm) and high TS concentrations (22.1%). For thermochemical and alkaline pretreated feeds, the optimal conditions are at high particle sizes (8.0 mm), TS concentrations (23.4%) and high alkaline doses (150 meq/L).

However, to integrate anaerobic co-digestion of OFMSW and sewage sludge into the RMOC's longterm solid waste management plan, further research is required. Most importantly:

1. Studies need to be performed on multiple sludges to determine the robustness of HSAD to a wide range of sludge strengths;
2. Further experimentation at higher feed total solids concentrations are required to assess reactor performance under potentially stressed conditions;
3. A kinetic study is required to assess the rate at which the digestion occurs to establish a basis for bioreactor design and sizing;
4. A microbiological study of the biological and physicochemical interactions is required to assess the impact of feed pretreatment protocols, namely, thermal, alkaline and thermochemical pretreatments as presented in this thesis;
5. The technical feasibility of the anaerobic co-digestion process was shown through this study, however, a cost/benefit analysis is required to assess the potential for implementation of the process within the RMOC.

CHAPTER 6:
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APPENDIX A:
Summary of Waste Audit on Ottawa-Carleton Municipal Solid Waste

This appendix provides the results of a waste audit performed by the Regional Municipality of Ottawa-Carleton on its residential, industrial, commercial and institutional solid waste.

Table A.1: Composition of the Residential Waste Stream in Ottawa-Carleton (RMOC, 1992)

Waste Category	Residential Mixed (wt %)	Rural (wt %)	Suburban (wt %)	Urban (wt %)
Paper	**29.7**	**29.5**	**23.6**	**43.6**
Newsprint	5.0	5.7	3.5	10.8
Corrugated & Kraft	5.5	5.2	4.7	6.3
Computer & High Grade	0.7	0.8	1.0	1.1
Boxboard	3.4	3.3	3.4	4.0
Mixed Recyclable	6.2	5.9	4.3	10.9
Non-Recyclable	8.9	8.6	6.7	10.5
Plastic	**8.2**	**7.7**	**7.4**	**7.3**
PET Containers	0.3	0.2	0.2	0.4
HDPE Containers	0.5	0.5	0.5	0.5
Polystyrene	0.4	0.3	0.6	0.3
Film Plastic	3.1	3.0	2.7	2.9
Other Packaging	1.0	0.9	0.9	1.3
Other	2.9	2.8	2.5	1.9
Glass	**2.5**	**4.3**	**2.9**	**4.8**
Liquor & Wine	0.6	1.2	0.9	1.6
Glass Containers	1.3	2.6	1.5	2.5
Other	0.6	0.5	0.5	0.7
Ferrous Metal	**5.0**	**7.8**	**3.2**	**4.6**
Soft Drink	0.4	0.4	0.3	0.5
Food & Beverage Containers	1.3	1.6	0.8	1.8
Aerosol Cans	0.1	0.2	0.1	0.3
Other	3.2	5.6	2.0	2.0
Non-Ferrous Metal	**0.8**	**1.4**	**0.7**	**0.7**
Food & Beverage Containers	0.3	0.4	0.2	0.4
Other	0.5	1.0	0.5	0.3
Yard Trimmings	**14.7**	**10.6**	**25.1**	**4.6**
Grass	5.6	3.5	11.7	2.3
Leaves	1.4	3.5	3.3	0.0
Trimmings	7.7	3.6	10.1	2.3
Other Organics	**31.4**	**30.3**	**27.8**	**29.5**
Food	11.4	12.1	12.3	15.7
Disposable Diapers	2.9	2.6	3.8	1.8
Land Clearing Debris	0.2	0.4	0.3	0.0
Lumber & Wood	4.1	2.7	2.8	1.7
Tires & Rubber	1.0	1.4	0.5	0.0
Textiles	3.6	4.4	2.5	3.4
Other	8.2	6.7	5.6	6.9

Table A.1: (cont.)

Waste Category	Residential Mixed (wt %)	Rural (wt %)	Suburban (wt %)	Urban (wt %)
Other Wastes	**7.4**	**8.1**	**9.6**	**4.9**
Inert Solids	1.6	2.9	2.5	2.3
Brown Goods	0.3	0.0	0.5	0.0
Construction & Demolition	1.9	2.2	3.4	0.5
Fines	3.6	3.0	3.2	2.1
Hazardous Wastes	**0.3**	**0.3**	**0.0**	**0.0**

Table A.2: Composition of IC&I Waste Stream in Ottawa-Carleton (RMOC, 1992)

Waste Category	Education (wt %)	Government (wt %)	Hotels (wt %)	Retail† (wt %)	Office (wt %)	Restaurant (wt %)
Paper	**45.5**	**60.9**	**37.0**	**51.5**	**71.9**	**34.2**
Newsprint	4.6	5.4	5.5	1.5	8.5	1.2
Corrugated & Kraft	7.7	9.9	14.6	30.4	12.4	12.7
Computer & High Grade	4.5	16.8	2.0	4.3	16.7	0.2
Boxboard	2.6	2.1	2.1	2.7	3.2	3.0
Mixed Recyclable	9.5	13.6	3.9	4.5	18.0	1.3
Non-Recyclable	16.6	13.1	8.9	8.1	13.1	15.8
Plastic	**10.5**	**7.5**	**6.1**	**7.2**	**7.4**	**8.2**
PET Containers	0.2	0.1	0.2	0.0	0.1	0.1
HDPE Containers	0.4	0.2	0.5	0.2	0.2	0.6
Polystyrene	1.7	0.6	0.3	0.8	1.0	0.5
Film Plastic	3.7	1.8	2.5	3.5	2.5	4.2
Other Packaging	1.2	0.4	1.1	0.7	0.7	1.1
Other	3.3	4.4	1.5	2.0	2.9	1.7
Glass	**4.7**	**2.9**	**10.9**	**1.5**	**2.7**	**4.9**
Liquor & Wine	0.6	0.1	5.9	0.1	0.1	3.3
Glass Containers	3.5	1.8	3.9	1.1	2.2	1.4
Other	0.6	1.0	1.1	0.3	0.4	0.2
Ferrous Metal	**3.3**	**8.2**	**2.1**	**3.1**	**2.0**	**2.3**
Soft Drink	1.1	0.6	0.3	0.3	0.6	0.2
Food & Beverage Containers	0.5	0.4	1.0	0.2	0.1	1.5
Aerosol Cans	0.0	0.0	0.1	0.1	0.0	0.0
Other	1.7	7.2	0.7	2.5	1.3	0.6
Non-Ferrous Metal	**1.1**	**0.8**	**0.7**	**0.2**	**0.4**	**0.2**
Food & Drink Containers	0.7	0.3	0.4	0.1	0.4	0.1
Other	0.4	0.5	0.3	0.1	0.0	0.1
Yard Trimmings	**0.7**	**0.1**	**0.1**	**1.4**	**0.4**	**3.4**
Grass	0.0	0.1	0.0	0.3	0.3	2.9
Leaves	0.3	0.0	0.0	0.0	0.0	0.0
Trimmings	0.4	0.0	0.1	1.1	0.1	0.5

Table A.2: (cont.)

Waste Category	Education (wt %)	Government (wt %)	Hotels (wt %)	Retail† (wt %)	Office (wt %)	Restaurant (wt %)
Other Organics	**22.0**	**11.4**	**41.3**	**33.3**	**10.3**	**44.5**
Food	15.4	4.4	31.5	23.9	6.9	33.4
Disposable Diapers	0.6	0.0	0.1	1.3	0.0	0.1
Land Clearing Debris	0.0	0.3	0.0	0.0	0.0	1.4
Lumber & Wood	1.5	3.7	1.3	4.1	0.4	0.5
Tires & Rubber	0.0	0.0	0.0	1.0	0.6	0.0
Textiles	1.3	0.8	0.9	0.4	1.2	1.1
Other	3.2	2.2	7.5	2.6	1.2	8.0
Other Wastes	**10.5**	**8.1**	**1.6**	**1.6**	**4.4**	**2.0**
Inert Solids	0.5	0.5	0.4	1.0	0.0	0.1
Brown Goods	0.0	0.0	0.0	0.0	0.9	0.0
Construction & Demolition	6.2	4.0	0.2	0.5	1.1	1.5
Fines	3.8	3.6	1.0	0.1	2.4	0.4
Hazardous Wastes	**1.4**	**0.5**	**0.1**	**0.1**	**0.2**	**0.4**

† Refers to large retail businesses only.

APPENDIX B:
Analytical Methods

This appendix provides a detailed listing of all analytical methods used throughout the experimental program.

B.1 Biogas Production

The volume of biogas produced during the biodegradation process was measured using two different methods based on the reactor configuration.

B.1.1 Batch Reactors

Biogas production was measured periodically throughout each experiment using the apparatus shown in Figure B.1. As shown in the diagram, gas is released from the bioreactor through a septum in the bioreactor lid. The gas is released into a graduated buret filled with coloured, acidified water. An equilibrium is reached between the meniscus in the buret and the separatory flask. The difference in meniscus level is distributed across the cross-sectional area of the separatory flask which is much greater than that of the buret. The water level stabilizes once the pressure inside the bioreactor reaches atmospheric pressure. The difference in the water in the buret constitutes the gas production in millilitres at 35 °C (the temperature of the bioreactor) and atmospheric pressure as per equation B1.

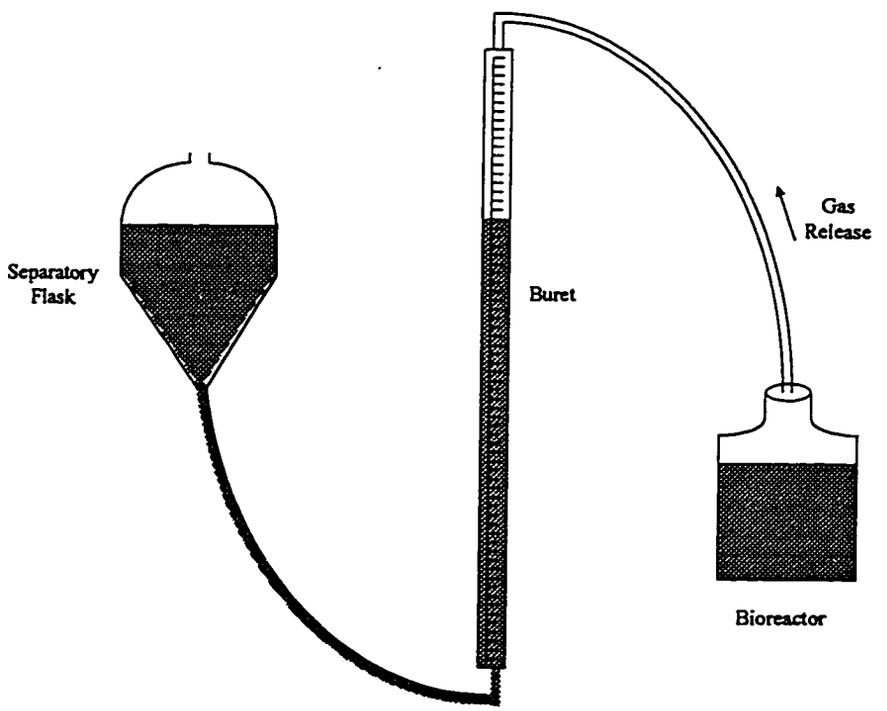

Figure B.1: Biogas Production Apparatus for Batch Reactors

$$Biogas\ Production = V_{after} - V_{before} \quad [B1]$$

where

V_{after} = meniscus reading after gas release, mL

V_{before} = meniscus reading after gas release, mL

B.1.2 Continuous Reactors

Large-scale semi-continuous reactors were connected to wet tip gas meters as shown in Figure B.2. The bioreactor biogas outlet port was connected via tygon tubing to the inlet port. After a certain gas volume is accumulated in the meter, the internal lever *tips* activating a magnetic switch which registers on the readout at the top of the wet tip meter. When the lever tips, the gas is released via the exhaust port at the top of the meter and the cycle repeats itself.

To convert the number of tips to a volume of biogas, the wet tip meter must first be calibrated. A Mark III Flowmeter (Fisher Scientific Company) was used for the calibration. The flowmeter was set at a value of 5 mL/s of air fed to the wet tip meter. The calibration of the wet tip meter is independent of the biogas flow rate. The number of tips was recorded for a period of 60 seconds. Given the air flowrate and the time span, the average volume per tip was calculated using the equation:

$$V = Q * t / N_{tip} \quad [B2]$$

where

V is the volume of air per tip, mL

Q is the air flowrate, mL/s

t is the time for run, seconds

N_{tip} is the number of tips during timing period

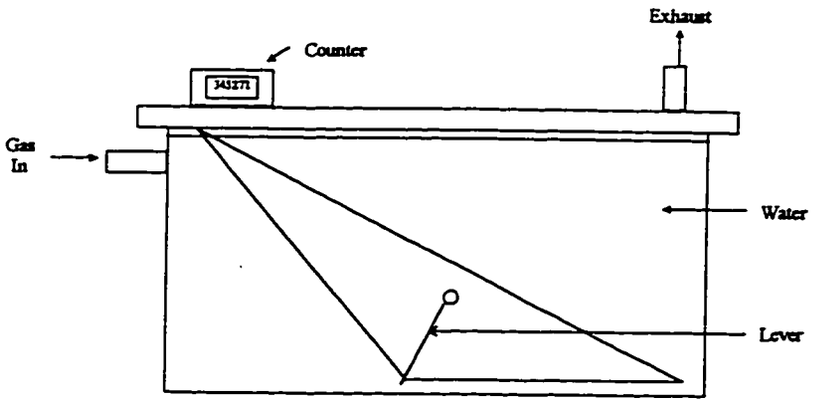

Figure B.2: Wet Tip Gas Meter for Biogas Production Measurement

B.2 Biogas Methane Concentration

Source: Greenberg *et al.*, 1992 (Method 2720C)

As per Standard Methods and the modifications proposed by van Huyssteen (1967), the percent of methane in the biogas was determined using a gas chromatographic method. A Hewlett-Packard (HP) Model 5710A gas chromatograph was fitted with a Poropak T porous polymer packed column (6.35 mm x 304.8 mm) and a thermal conductivity detector. The column was maintained at 70 °C and nitrogen was used as a carrier gas.

Procedure:

1. Using a 1 mL syringe with a luar-lok fitting, puncture septum of sampling port.
2. To ensure a representative sample is chosen, move plunger up and down 5 to 10 times.
3. Take a 0.5 mL sample and lock the luar-lok.
4. Inject the needle into the gas chromatograph, un-lock the luar-lok and press down on the plunger.

B.3 Chemical Oxygen Demand (COD)

Source: Greenberg *et al.*, 1992 (Method 5220C)

Reagents:

- Digestion solution, 0.0167 M $K_2Cr_2O_7$
    - To about 500 mL of water, add 4.913 g $K_2Cr_2O_7$ previously dried at 130 °C for 2 hours, 167 mL concentrated H_2SO_4 and 33.3 g $HgSO_4$;

    * Dissolve, cool to room temperature and dilute to 1 L.

- Sulfuric acid reagents
    * Add 11 g Ag_2SO_4 per 4 L bottle of H_2SO_4;
    * Stir on a magnetic stirrer for 1 to 2 days to dissolve Ag_2SO_4.
- Standard potassium hydrogen phthalate (KHP)
    * Lightly crush and then dry KHP to constant weight at 120 °C;
    * Dissolve 425 mg in distilled waster and dilute to 1 L. KHP has a theoretical COD of 1.176 mg O_2/mg and the solution 500 mg O_2/mL.
- 0.10 M Ferrous Ammonium Sulphate (FAS)
    * Dissolve 39.2 mg $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in distilled water;
    * Add 20 mL concentrated sulphuric acid and dilute to 1 L.

Procedure:

1. Wash tubes and caps with H_2SO_4 and add 10 mL of sample, 6 mL of $K_2Cr_2O_7$ and 14 mL of H_2SO_4 reagent. A blank of water and at least one dilution of standard (KHP) should be prepared with each run.
2. Mix, heat at 150 °C for 2 hours and cool to room temperature.
3. Titrate sample with FAS and ferroin indicator

Calculation:

$$M = \frac{V_p * 0.10}{A} \qquad [B2]$$

where:

M is the concentration of FAS, mol/L

Vp is the volume of 0.0167 M $K_2Cr_2O_7$

A is the volume of FAS required in titration for blank, mL

$$COD = \frac{(A-B) * M * 8000}{V_s} \qquad [B3]$$

where:

COD is the chemical oxygen demand, mg O_2/L

B is the volume of FAS required for the sample, mL

V_s is the volume of sample used, mL

B.4 pH

The pH was measured using a Radiometer Copenhagen pH meter model 26. A probe resistant to sulfur-fouling and specific to anaerobic digesters, the Ingold P3504, was also used. The pH meter was calibrated using standards for pH 4 (Canlab), 7 (Canlab) and 10 (Colorkey).

B.5 Volatile Fatty Acid (VFA) Concentration

Acetic, propionic and butyric acids were measured chromatographically using a 2% isobutyric acid solution as an internal standard as per the method of Ackman (1972). A Hewlett-Packard (HP) 5840A gas chromatograph fitted with a packed Chromosorb column (6.35 mm x 365.8mm) maintained at 180 °C, flame ionization detector, an HP model 7671 automatic sampler and an HP model 3380 integrator. The ionization detector was maintained at 350 °C. A flow of 15 mL/min of 99.9% pure helium gas saturated with formic acid was used as a carrier gas. The gas chromatograph was initially calibrated using a mixture of 0.5 mL isobutyric acid solution and a standard mixture composed of 2% of each of acetic, propionic and butyric acids.

Procedure:

1. Centrifuge sample centrifuged in a Fisher micro-centrifuge model 235A, set at high for 5 minutes.
2. Pipet out 0.5 mL of the supernatant and mix in a cuvette with 0.5 mL of the isobutyric acid internal standard.
3. Place in the autosampler of the gas chromatograph and activate the machine.

B.6 Total Solids (TS) Concentration

Source: Greenberg *et al.*, 1992 (Method 2540B)

Apparatus:

Aluminum Dishes

Desiccator

Drying Oven, for operation at 103 to 105 °C

Analytical Balance

Wide-Bore Pipets

Procedure:

1. Heat clean dish to 103 to 105 °C for 1 hour in the drying oven. Cool dish in desiccator and store until needed. Weigh immediately before use.
2. Take a 100 mL sample from the bioreactor and shake well.
3. Pipet 10 mL of well-mixed sample to a pre-weighed dish.
4. Dry sample in the drying oven at 103 to 105 °C for at least 1 hour.
5. Cool dish in desiccator. Weigh dish.
6. Repeat cycle of drying, cooling and weighing until the change in weight is less than 4% of the previous weight or 0.5 mg.

Calculation:

$$TS = \frac{(m_{res} - m_{dish}) \times 1000}{V_s} \qquad [B4]$$

where

TS is the total solids concentration, mg/L

m_{res} is the weight of the dried residue plus the dish, mg

m_{dish} is the weight of the dish, mg

B.7 Volatile Solids (VS) Concentration

Source: Greenberg *et al.*, 1992 (Method 2540E)

Apparatus:

As per B.6

Muffle Furnace, for operation at 500 ± 50 °C

Procedure:

1. Heat clean dish to 500 ± 50 °C for 1 hour in the muffle furnace. Cool dish in desiccator and store until needed. Weigh immediately before use.
2. Follow steps 2 through 6 of Section B.6.
3. Ignite sample by placing in muffle furnace at a temperature of 500 ± 50 °C for 20 minutes.
4. Cool dish in desiccator. Weigh dish.
5. Repeat cycle of ignition, cooling and weighing until the change in weight is less than 4% of the previous weight or 0.5 mg.

Calculation:

$$VS = \frac{(m_{bef} - m_{aft}) \times 1000}{V_s} \qquad [B5]$$

where

VS is the volatile solids, mg/L

m_{bef} is the weight of dried residue plus dish before ignition, mg

m_{aft} is the weight of dried residue plus dish after ignition, mg

APPENDIX C:
The Central Composite Experimental Design

This appendix describes the experimental strategy, the central composite design, and model-building techniques used in this thesis.

C.1 Background

The usefulness of response surface methods in chemical process development and improvement was clearly demonstrated by the work of Box *et al.* (1951). It was this work that introduced the central composite design for second order designs of the form

$$E(Y) = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \sum_{j \geq i}^{k} \beta_{ij} X_i X_j \qquad [C1]$$

where

E(Y) is the expected value of the response variable
β_o, β_i, β_j are the model parameters
X_i and X_j are the coded factors being studied
k is the number of factors being studied

The design consists of a 2^k factorial or fractional factorial design augmented by 2k axial (star) points at $(\pm\alpha, 0, 0, \ldots, 0)$, $(0, \pm\alpha, 0, \ldots, 0)$, $(0, 0, \pm\alpha, \ldots, 0)$, $\ldots$, $(0, 0, 0, \ldots, \pm\alpha)$ and n_o centre points at $(0, 0, \ldots, 0)$, where α is the distance of the star point from the centre. Box *et al.* (1957) also defined an experimental design to be rotatable if the variance of the predicted response, at any point is a function only of the distance of the point from the design centre and not a function of direction. This implies that a rotatable design will leave the variance of the model prediction unchanged when the design is rotated about the centre $(0, 0, \ldots 0)$. The choice of α establishes the rotatability of a central composite design. The design is rotatable when α is equal to the number of points in the factorial positions raised to the power of 0.25 (Montgomery, 1984). Hence, for two factors, α should be set as 1.414.

The central composite designs that were used throughout this work include:

i- a two-factor design for untreated and thermal pretreated runs;

ii- a three-factor design for alkaline and thermochemical pretreatments.

The designs are presented in sections C.2 and C.3 respectively.

C.2 Two-Factor Central Composite Design

As per equation [C1], the proposed model for this two-factor design is

$$E(Y) = \beta_o + \beta_1 X_1 + \beta_2 X_2 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{12} X_1 X_2 \qquad [C2]$$

The run conditions used for the untreated (control) and heat pre-treated mixtures are presented in Table C.2 and are graphically represented in Figure C.1. The design points for particle size, X_1, were set as much as possible with the suggested α of 1.414. However, due to restrictions in the availability of sieves with mesh sizes at all the recommended levels, four design points were run at levels that were not at the suggested α. The implications of the deviations from ideal placement of runs are reflected in the correlation matrices for the actual run conditions, equation [C3], and the optimized design, equation [C4]. There does not appear to be a significant difference between these two matrices.

$$Correlation\ Matrix\ (Actual) = \begin{bmatrix} 1 & 0.164 & 0 & -0.623 & -0.666 & 0 \\ 0.164 & 1 & 0 & -0.336 & -0.071 & 0 \\ 0 & 0 & 1 & 0 & 0 & -0.057 \\ -0.623 & -0.336 & 0 & 1 & 0.262 & 0 \\ -0.666 & -0.071 & 0 & 0.262 & 1 & 0 \\ 0 & 0 & -0.057 & 0 & 0 & 1 \end{bmatrix} \qquad [C3]$$

$$Correlation\ Matrix\ (Optimized) = \begin{bmatrix} 1 & 0 & 0 & -0.632 & -0.632 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 \\ -0.632 & 0 & 0 & 1 & 0.200 & 0 \\ -0.632 & 0 & 0 & 0.200 & 1 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 \end{bmatrix} \qquad [C4]$$

Table C.2: Run Conditions for Two-Factor Central Composite Designs

Particle Size X_1, mm	Total Solids X_2, %	Coded X_1	Coded X_2
2.00	10.0	-1.000	-1.000
6.35	10.0	1.175*	-1.000
2.00	20.0	-1.000	1.000
6.35	20.0	1.175*	1.000
0.85	15.0	-1.575*	0
8.00	15.0	2.000*	0
4.00	7.9	0	-1.414
4.00	22.1	0	1.414
4.00	15.0	0	0
4.00	15.0	0	0
4.00	15.0	0	0
4.00	15.0	0	0

* Could not use optimized run conditions because of physical constraints

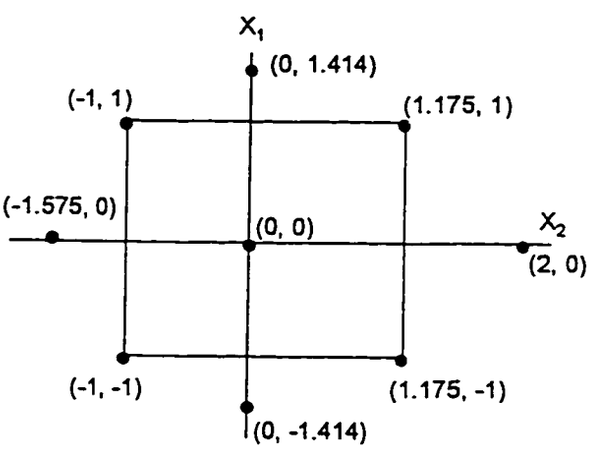

Figure C.1: Central Composite Design for Two Factors

C.3 Three-Factor Central Composite Design

The corresponding model form for the three-factor design based on equation [C1] is

$$E(Y) = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 \quad [C5]$$

The run conditions used for the alkaline and thermochemically pre-treated mixtures are presented in Table C.3 and are graphically represented in Figure C.2. The points at which particle size were set were also not optimized as has been discussed in section C.2. However, there does not appear to be a significant difference between the actual and optimal parameter correlation matrices presented in equations [C6] and [C7].

Table C.3: Run Conditions for Three-Factor Central Composite Designs

Particle Size X_1, mm	Total Solids X_2, %	Alkaline Dose X_3, meq/L	Coded X_1	Coded X_2	Coded X_3
2.00	10.0	50	-1.000	-1.000	-1.000
6.35	10.0	50	1.175*	-1.000	-1.000
2.00	20.0	50	-1.000	1.000	-1.000
6.35	20.0	50	1.175*	1.000	-1.000
2.00	10.0	150	-1.000	-1.000	1.000
6.35	10.0	150	1.175*	-1.000	1.000
2.00	20.0	150	-1.000	1.000	1.000
6.35	20.0	150	1.175*	1.000	1.000
4.00	15.0	16	0.000	0.000	-1.682
4.00	15.0	184	0.000	0.000	1.682
4.00	6.6	100	0.000	-1.682	0.000
4.00	23.4	100	0.000	1.682	0.000
0.85	15.0	100	-1.575*	0.000	0.000
8.00	15.0	100	2.000*	0.000	0.000
4.00	15.0	100	0.000	0.000	0.000
4.00	15.0	100	0.000	0.000	0.000
4.00	15.0	100	0.000	0.000	0.000
4.00	15.0	100	0.000	0.000	0.000
4.00	15.0	100	0.000	0.000	0.000
4.00	15.0	100	0.000	0.000	0.000

* Could not use optimized run conditions because of physical constraints

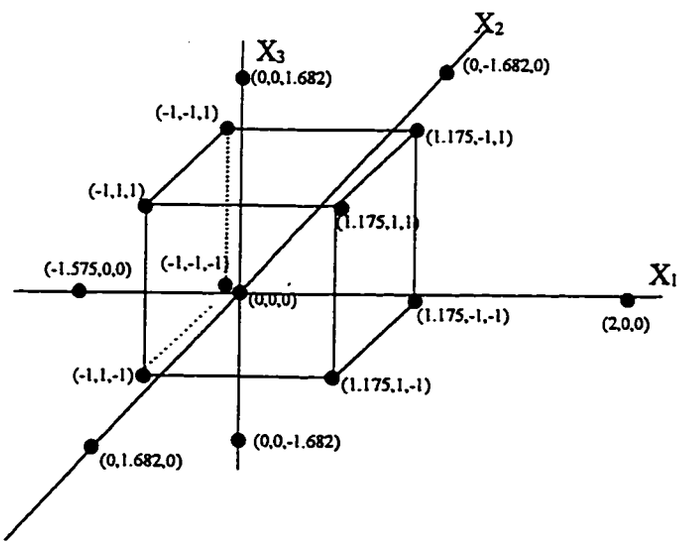

Figure C.2: Central Composite Design for Three Factors

$$Correlation\ Matrix\ (Actual) = \begin{bmatrix} 1 & 0.127 & 0 & 0 & -0.525 & -0.183 & -0.525 & 0 & 0 & 0 \\ 0.127 & 1 & 0 & 0 & -0.024 & 0.021 & -0.024 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 & 0 & -0.061 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & -0.061 & 0 \\ -0.524 & -0.312 & 0 & 0 & 1 & 0.091 & 0.091 & 0 & 0 & 0 \\ -0.525 & -0.024 & 0 & 0 & 0.091 & 1 & 0.098 & 0 & 0 & 0 \\ -0.525 & -0.024 & 0 & 0 & 0.091 & 0.098 & 1 & 0 & 0 & 0 \\ 0 & 0 & -0.061 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & -0.061 & 0 & 0 & 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 \end{bmatrix} \quad [C6]$$

$$Correlation\ Matrix\ (Optimized) = \begin{bmatrix} 1 & 0 & 0 & 0 & -0.517 & -0.492 & -0.492 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\ -0.517 & 0 & 0 & 0 & 1 & 0.019 & 0.019 & 0 & 0 & 0 \\ -0.492 & 0 & 0 & 0 & 0.019 & 1 & 0.091 & 0 & 0 & 0 \\ -0.492 & 0 & 0 & 0 & 0.019 & 0.091 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 \end{bmatrix} \quad [C7]$$

C.4 Analysis of Models

Using least squares analysis, four critical assumptions are made: (Wadsworth, 1990)

1. The values of the experimental factors are known exactly, that is, the associated variance is zero. This essentially means that the uncertainty associated with the value of an experimental factor has much less effect on the response value than the uncertainty associated with the measured value of the response itself.
2. The form of the model is appropriate, that is, the expected value of the random error is zero.
3. The variance of the random error is constant over the range of the experimental factors used to collect the data.
4. There is no systematic association of the random error for any one data point with the random error for any other data point.

A fifth assumption that the random errors are normally distributed, provides a basis for probability statements about model parameters and predictions and for hypothesis tests. Under this assumption, least squares estimates are also maximum likelihood estimates.

To determine the adequacy of a least squares fit, it is necessary to perform an analysis of the residuals from the model. Typically various plots of the residuals, including normal probability plots and plots of residuals versus predicted values of the response variable are first constructed to verify that the random error follows the underlying assumptions.

Analysis of Variance (ANOVA) tables are also constructed. In an ANOVA analysis, the variation is separated into two components, regressional and residual as shown in Table C.4. The sum of squares is used to measure variability (Mason *et al.*, 1989) such that

$$SS = \Sigma(y_i - \bar{y})^2 \qquad [C14]$$

where SS is the sum of squares of deviations of the observed values from their mean value

$$SS_E = \sum (y_i - y_{hat})^2 \qquad [C15]$$

where SS_E is the sum of squares of the residuals

and,

$$SS_R = SS - SS_E \qquad [C16]$$

where SS_R is the sum of squares due to the regression

y_i is the observed value of the response variable at design point I

$\bar{y}$ is the mean value of the observed responses

y_{hat_i} is the predicted response variable at point i

Table C.4: ANOVA Table for Multiple Regression Models (Mason *et al.*, 1989)

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Squares	F
Regression	p	SS_R	$MS_R = SS_R/p$	MS_R/MS_E
Residual	N-p-1	SS_E	$MS_E = SS_E/(n-p-1)$	
(Lack of Fit)	f_{LOF}	SS_{LOF}	$MS_E = SS_{LOF}/ f_{LOF}$	MS_{LOF}/MS_{PE}
(Pure Error)	f_{PE}	SS_{PE}	$MS_E = SS_{PE}/ f_{PE}$	
Total	N-1	SS		

In multiple regression models, there are p degrees of freedom for the sum of squares due to regression, because p coefficients, namely β_1, β_2, ..., β_p, must be estimated to obtain SS_R. Since the degrees of freedom for the entire system is defined as the total number of experimental runs minus 1 (N-1), there are N-p-1 degrees of freedom for SS_E.

The lack of fit test checks whether the order of the model is correct. In this test, the null hypothesis, H_o, proposes that the model adequately fits the data. The test involves dividing the variability of the residuals into two components: pure error and lack of fit. The pure error sum of squares is calculated based on the values of the response variables at the replicate points (Mason *et al.*, 1989):

$$SS_{PE} = \sum_{i=1}^{m}\left(\sum_{j=1}^{n}\left(y_{ij} - \bar{y}_i\right)^2\right) \qquad [C17]$$

where m is the number of points where replicates were taken and n_i the number of replicates taken at point m. The corresponding number of degrees of freedom, f_{PE}, is

$$f_{PE} = \sum_{i=1}^{m} (n_i - 1) \qquad [C18]$$

The corresponding lack of fit terms, the sum of squares, SS_{LOF}, and the degrees of freedom, f_{LOF}, are determined by subtracting the pure error from the residual values. The lack of fit F-statistic shown in Table C.4 provides the basis for the evaluation of lack of fit. The corresponding degrees of freedom for this statistic are f_{LOF} and f_{PE}.

The coefficient of determination, R^2, is another measure of the adequacy of the fit where

$$R^2 = 1 - SS_E/SS = (SS - SS_E)/SS = SS_R/SS \qquad [C19]$$

Values range from 0 to 1, with values closer to 1 indicating high consistencies between the predicted and observed values. R^2 also provides a measure of the fraction of the total variability in the data explained by the regression.

Also in Table C.4, is the regression F-statistic which is related to the null hypothesis that all the model parameters are equal to 0. Since the regression F-statistic is the ratio of the mean square terms for regression to residuals, the corresponding degrees of freedom for this statistic are p and N-p-1.

Confidence intervals for each of the model parameters can also be determined (Wadsworth, 1990). A 95% confidence interval for β_i,

$$\beta_i - t_{0.025} s_e c_{ii}^{0.5} \leq \beta_i \leq \beta_i + t_{0.025} s_e c_{ii}^{0.5} \qquad [C20]$$

where s_e is the square root of the mean squares for the regression and c_{ii} is the diagonal element of the covariance matrix, $(\underline{X}^T\underline{X})^{-1}\sigma^2$ where $\underline{X}$ is a matrix of experimental factors. The variance, σ^2 can be estimated from the pure error variance or if there is no evidence of lack of fit, a better estimate is given by $SS_R/(N-p-1)$.

APPENDIX D:
Raw Data

This appendix lists all of the raw data collected throughout all of the experiments presented in this thesis.

D.1 Study of COD Variability of ROPEC Sewage Sludge

Table D.1: Raw Data for Components of Variance Study on RAW Soluble COD

Day	Time	Sample	TESTS Test ($mg\ O_2/L$) Test (1)	TESTS COD Test (2)	Difference ($mg\ O_2/L$)	Test Variance	SAMPLES Average Test ($mg\ O_2/L$)	SAMPLES Difference ($mg\ O_2/L$)	Sample Variance
M	9:00	1	1878	1890	12	72	1884	13	85
M		2	1870	1872	2	2	1871		
M	12:00	1	2746	2744	-2	2	2745	24	288
M		2	2720	2722	2	2	2721		
T	9:00	1	912	910	-2	2	911	-604	182408
T		2	1518	1512	-6	18	1515		
T	12:00	1	1856	1862	6	18	1859	-85	3613
T		2	1952	1936	-16	128	1944		
W	9:00	1	1850	1846	-4	8	1848	-26	338
W		2	1872	1876	4	8	1874		
W	12:00	1	2000	2002	2	2	2001	-8	32
W		2	2010	2008	-2	2	2009		
Th	9:00	1	1396	1394	-2	2	1395	-12	72
Th		2	1406	1408	2	2	1407		
Th	12:00	1	1094	1094	0	0	1094	-37	685
Th		2	1136	1126	-10	50	1131		
F	9:00	1	1458	1464	6	18	1461	32	512
F		2	1426	1432	6	18	1429		
F	12:00	1	1080	1080	0	0	1080	5	12.5
F		2	1076	1074	-2	2	1075		
			1663	1663	V_T	18	1663	V_S	18804

Table D.1 (cont.): Raw Data for Components of Variance Study on RAW Soluble COD

Day	Time	Sample Average	TIME Difference (mg O_2/L)	Time Variance	Time Average	DAY Difference (mg O_2/L)	Difference Squared
M	9:00	1878	-855	365940.1	2305.3	642.6	412870.5
M							
M	12:00	2733					
M							
T	9:00	1213	-689	237016.1	1557.3	-105.5	11119.7
T							
T	12:00	1902					
T							
W	9:00	1861	-144	10368.0	1933.0	270.3	73062.1
W							
W	12:00	2005					
W							
Th	9:00	1401	288	41616.1	1256.8	-405.9	164795.4
Th							
Th	12:00	1113					
Th							
F	9:00	1445	367	67528.1	1261.3	1261.3	1590751.6
F							
F	12:00	1078					
F							
		1663	V_t	144493.7	1662.7	V_D	563149.8

Table D.2: Raw Data for Components of Variance Study on WAS Soluble COD

Location	Day	Time	Sample	TESTS COD ($mg\ O_2/L$) Test (1)	Test (2)	Difference ($mg\ O_2/L$)	Test Variance	SAMPLES Test Average COD ($mg\ O_2/L$)	Difference ($mg\ O_2/L$)	Sample Variance
North	T	9:00	1	77	77	0	0	77.0	-4.5	10.1
			2	78	85	-7	24.5	81.5		
		12:00	1	83	84	-1	0.5	83.5	2.0	2.0
			2	82	81	1	0.5	81.5		
	W	9:00	1	30	34	-4	8.0	32.0	0	0
			2	33	31	2	2.0	32.0		
		12:00	1	63	64	-1	0.5	63.5	2.5	3.1
			2	59	63	-4	8.0	61.0		
	Th	9:00	1	67	65	2	2.0	66.0	-5.5	15.1
			2	71	72	-1	0.5	71.5		
		12:00	1	62	60	2	2.0	61.0	-1.0	0.5
			2	61	63	-2	2.0	62.0		
South	T	9:00	1	64	66	-2	2.0	65.0	-12.5	78.1
			2	80	75	5	12.5	77.5		
		12:00	1	75	70	5	12.5	72.5	-2.0	2.0
			2	74	75	-1	0.5	74.5		
	W	9:00	1	35	32	3	4.5	33.5	0.5	0.1
			2	32	34	-2	2.0	33.0		
		12:00	1	61	61	0	0	61.0	-1.5	1.1
			2	63	62	1	0.5	62.5		
	Th	9:00	1	67	67	0	0	67.0	-2.0	2.0
			2	69	69	0	0	69.0		
		12:00	1	66	63	3	4.5	64.5	-0.5	0.1
			2	65	65	0	0	65.0		
				63	63	V_T	3.7	63.2	V_S	9.5

Table D.2(cont.): Raw Data for Components of Variance Study on WAS Soluble COD

Day	Time	TIME Sample Average COD	TIME Difference (mg O_2/L)	TIME Time Variance	DAY Average Time COD	DAY Difference (mg O_2/L)	DAY Difference Squared	LOCATION Average Day COD	LOCATION Difference (mg O_2/L)	LOCATION Location Variance
T	9:00	79.3	-3.3	5.3	80.9	17.6	311.4	64.4	2.2	2.6
	12:00	82.5								
W	9:00	32.0	-30.3	457.5	47.1	-16.1	259.3			
	12:00	62.3								
Th	9:00	68.8	7.3	26.3	65.1	1.9	3.6			
	12:00	61.5								
T	9:00	71.3	-2.3	2.5	72.4	9.1	83.6	62.1		
	12:00	73.5								
W	9:00	33.3	-28.5	406.1	47.5	-15.7	247.4			
	12:00	61.8								
Th	9:00	68	3.3	5.3	66.4	3.1	9.9			
	12:00	64.8								
		64.4	V_t	150.5	63.2	V_D	91.5	63.2	V_L	2.6

D.2 Optimization Study for Mix Ratio of Sludge-to-OFMSW

Table D.3: Biogas Production Raw Data for Optimization Study of Ratio of Sludge to OFMSW

	Biogas Production (mL)					
Time(hours)	**0%**	**0%**	**25%**	**25%**	**50%**	**50%**
0.5	27.4	32.6	45.0	50.4	44.9	36.2
1.0	10.8	13.6	26.0	31.4	27.4	26.0
1.5	10.0	10.8	20.8	22.4	22.4	24.0
2.0	9.3	9.3	20.4	19.7	18.8	19.8
2.5	5.7	8.4	18.3	23.1	20.2	19.8
3.0	7.4	6.9	23.4	23.6	19.6	19.4
3.5	6.1	5.2	35.0	32.2	25.2	20.6
4.0	4.5	9.6	33.0	39.1	23.6	25.6
4.5	3.8	4.8	36.2	39.6	34.9	32.1
5.0	8.7	9.5	46.0	39.6	38.6	53.2
5.5	5.6	6.7	35.6	37.0	52.0	53.6
21.0	122.8	140.0	243.7	256.3	558.6	711.2
25.5	32.9	35.3	50.0	53.4	237.5	178.7
29.5	53.7	58.9	159.6	91.2	160.3	173.0
48.0	79.1	81.2	178.8	237.6	269.8	244.6
53.0	39.6	42.1	54.8	39.4	63.8	48.2
72.0	501.4	51.8	73.4	93.5	92.8	70.6
78.0	234.4	19.7	50.9	44.0	51.0	54.0
117.0	281.5	34.6	81.4	79.8	55.0	52.8
126.5	88.7	17.1	58.0	54.2	35.7	35.4
141.0	69.3	18.5	48.9	43.8	26.9	30.2
175.0	88.0	37.2	225.8	190.8	75.2	52.0
215.5	183.2	53.8	525.2	414.8	168.8	225.6
287.5	627.4	163.6	905.3	1017.8	516.4	570.7
333.5	369.0	257.8	1538.1	1482.4	327.6	254.3
357.5	339.8	248.5	808.9	272.8	152.4	124.4
387.0	608.4	377.6	710.8	246.2	83.2	58.2
430.0	371.4	601.6	312.2	631.5	63.8	47.8
456.0	215.0	328.5	187.0	359.8	55.0	31.9
480.5	115.4	225.6	217.4	315.5	45.7	37.0
502.0	71.6	165.0	220.9	215.4	29.8	26.7
525.5	90.8	138.7	218.4	183.0	25.0	9.9
597.0	313.4	278.0	274.4	703.4	19.9	12.4
653.0	235.2	120.4	278.8	478.5	26.4	21.0
750.0	142.2	65.5	337.8	410.9	13.0	8.6
838.0	77.0	46.2	759.0	168.2	2.4	4.2
935.0	67.5	58.1	407.0	150.5	18.4	10.6
1034.0	60.8	40.8	159.2	284.8	10.4	11.2
1158.5	59.7	40.0	94.4	751.0	17.8	9.2
1227.5	49.0	21.4	52.7	253.7	0.0	8.6
1320.0	49.5	33.2	56.6	185.8	10.7	16.4
1462.0	31.8	34.4	59.6	122.0	7.0	3.0
1731.5	80.2	84.2	137.2	122.2	12.2	13.5

Table D.3 (cont.): Biogas Production Raw Data for Optimization Study of Ratio of Sludge to OFMSW

Time	75%	75%	100%	100%	Dist. H_2O	Dist. H_2O
0.5	36.2	43.6	57.3	53.2	11.4	8.8
1.0	24.3	25.8	35.2	29.6	6.0	3.5
1.5	21.6	21.1	23.9	26.3	3.2	2.6
2.0	19.3	19.2	20.4	17.8	3.5	2.5
2.5	20.9	21.4	5.8	13.0	1.9	2.4
3.0	12.3	12.5	6.9	17.6	1.6	1.5
3.5	25.4	23.6	12.0	8.4	0.9	1.6
4.0	19.4	21.0	12.2	4.8	1.7	4.8
4.5	27.8	28.8	5.3	10.5	0.0	0.9
5.0	25.2	48.2	7.8	9.1	0.0	0.0
5.5	43.1	47.4	7.0	13.3	3.0	2.5
21.0	1451.6	1353.1	2034.9	1931.3	15.1	18.6
25.5	357.2	310.6	222.0	263.8	5.7	0.8
29.5	253.2	256.8	423.2	431.1	0.0	0.0
48.0	186.0	170.6	238.9	333.4	19.9	21.4
53.0	93.2	85.1	141.1	92.7	9.6	0.0
72.0	166.1	165.9	135.5	95.9	14.6	6.8
78.0	36.0	53.1	64.3.0	75.0	2.5	4.6
117.0	51.2	53.6	176.1	137.0	0.0	5.6
126.5	31.6	24.3	29.9	45.2	1.3	0.0
141.0	24.5	27.2	26.6	30.0	0.0	0.6
175.0	47.2	50.0	52.2	55.1	0.9	4.8
215.5	258.5	225.0	38.4	52.9	6.6	0.0
287.5	470.6	420.4	32.5	59.9	0.0	0.0
333.5	162.4	281.5	8.3	43.8	0.5	7.7
357.5	80.0	147.4	32.0	83.6	3.1	1.6
387.0	96.2	65.6	36.0	160.2	0.0	1.6
430.0	32.3	53.0	76.3	168.1	0.7	0.0
456.0	25.7	34.8	90.0	128.6	5.1	5.6
480.5	31.2	23.0	78.8	61.4	3.6	5.2
502.0	22.8	21.4	63.1	43.5	5.2	3.7
525.5	12.6	19.4	59.1	34.4	2.4	3.9
597.0	7.5	9.8	85.8	62.0	0.0	0.0
653.0	23.7	21.0	81.2	73.3	2.7	3.1
750.0	13.4	9.8	50.8	76.2	0.0	0.0
838.0	8.4	4.7	42.4	67.7	0.0	0.0
935.0	29.8	22.9	37.1	61.4	3.8	4.2
1034.0	23.6	12.6	32.7	39.4	0.8	0.0
1158.5	13.8	11.4	32.1	35.9	6.2	5.5
1227.5	15.8	8.2	11.2	22.6	0.0	0.0
1320.0	10.8	12.4	29.6	20.6	1.8	2.5
1462.0	7.9	4.0	21.8	94.0	0.8	0.0
1731.5	20.2	15.4	28.4	11.4	0.0	1.2

D.3 Component Contribution to Biodegradability

Table D.4: Biogas Production Raw Data for Batch Tests on Each Mixture Component

	Biogas Production (mL)						
Time (hours)	Grass	Grass	Leaves	Leaves	Paper	Paper	Newspaper
2.0	15.9	18.1	17.9	17.4	18.1	15.6	18.8
4.0	9.2	6.6	3.4	9.3	6.7	6.2	6.8
6.0	2.3	4.3	4.3	2.6	5.0	3.2	4.8
22.0	11.5	20.1	11.4	14.5	15.3	14.4	11.5
25.0	4.8	10.9	5.7	9.3	8.4	6.9	2.7
29.0	5.9	12.2	8.8	6.4	6.1	6.1	0.6
47.5	28.6	45.5	27.7	34.0	21.6	23.5	13.4
53.5	12.9	27.5	10.1	14.1	10.2	9.6	10.6
68.5	16.3	26.0	13.9	12.5	16.8	32.8	9.4
72.0	8.0	9.2	6.0	6.5	20.3	28.1	4.7
77.5	7.7	6.6	4.6	3.8	25.1	35.9	3.7
92.5	11.5	18.4	9.5	14.2	82.8	121.0	16.1
97.3	5.5	5.6	7.1	5.8	59.7	57.3	12.6
101.8	3.3	6.5	2.6	1.6	51.0	52.0	6.3
116.3	9.8	13.9	9.1	10.7	132.9	123.0	16.3
121.0	5.6	8.2	2.6	5.7	63.2	54.5	12.7
126.8	6.3	6.4	5.7	4.3	58.1	44.7	5.9
140.3	10.3	9.4	10.9	10.6	117.8	73.5	15.5
149.5	10.0	10.9	7.7	9.3	67.3	57.9	10.9
164.8	10.1	11.0	9.6	8.1	77.8	71.3	10.8
173.5	7.5	11.7	7.7	8.8	58.9	50.9	6.8
191.0	9.3	11.2	7.7	12.3	67.7	58.9	7.7
196.0	5.8	6.4	7.0	4.6	40.1	33.1	4.4
215.5	8.0	8.1	5.4	10.5	45.7	36.8	5.3
220.5	3.9	5.9	6.0	6.0	22.1	19.0	4.2
239.0	0.5	8.6	7.3	10.3	23.7	19.6	6.2
245.5	2.3	6.2	3.7	5.6	12.7	10.1	2.0
270.0	0.0	12.8	11.4	13.8	17.1	14.1	7.9
284.5	11.2	9.3	6.5	10.1	12.6	11.9	6.7
317.5	19.2	18.4	13.8	17.5	16.0	16.9	11.7
332.3	7.2	12.3	9.4	12.7	10.0	9.3	5.5
356.8	0.0	14.1	7.8	13.8	11.5	10.6	6.6
384.0	11.2	15.6	12.0	16.0	12.7	12.2	9.8
404.0	8.9	8.9	6.3	9.5	8.0	6.5	7.5
428.8	7.5	12.9	8.5	10.6	9.9	8.7	7.2
452.3	9.5	11.0	8.0	9.7	8.0	9.6	4.8
482.0	7.8	11.0	6.9	8.2	6.9	7.0	0.0
503.0	7.6	5.5	4.9	7.0	7.1	5.1	8.9
530.0	15.2	19.8	11.9	17.3	13.6	13.8	12.5
551.8	6.2	10.0	5.0	5.2	7.4	7.6	3.9
581.0	4.2	5.1	3.9	5.2	3.7	3.1	2.8
605.0	5.5	8.8	8.1	10.6	8.4	7.3	6.4
621.0	4.3	7.9	5.4	6.8	7.4	7.1	6.3
643.8	2.3	5.2	4.7	1.8	3.7	2.0	2.8
667.3	1.5	3.4	2.5	2.4	2.2	3.4	2.4
695.0	4.2	7.8	4.7	4.3	4.1	4.9	2.7
718.3	10.9	11.0	9.2	9.1	8.7	8.2	5.4
745.5	13.4	15.1	11.3	12.7	13.2	12.3	12.6
763.3	7.0	7.2	7.2	6.4	7.4	6.7	4.6
787.8	9.8	12.0	11.2	10.7	10.8	8.6	9.6
813.8	5.6	7.7	5.9	5.0	7.4	6.0	3.2
843.5	1.2	2.4	1.5	2.8	4.9	3.3	1.5
861.8	4.2	4.1	3.4	2.6	5.2	3.1	3.3

Table D.4 (cont.): Biogas Production Raw Data for Batch Tests on Each Mixture Component

Time	Newspaper	Lettuce	Lettuce	Tomatoes	Tomatoes	Raw	Raw
2.0	18.1	10.4	14.3	10.5	11.0	50.2	41.6
4.0	8.8	4.0	4.3	4.9	1.6	24.3	23.8
6.0	4.0	1.2	4.3	1.6	1.5	14.7	16.8
22.0	10.5	26.5	55.6	47.5	38.3	88.3	121.0
25.0	2.9	17.1	27.5	25.1	19.3	41.9	37.2
29.0	5.0	18.6	23.0	23.8	21.9	42.7	40.1
47.5	11.6	94.6	147.8	141.4	141.3	416.6	435.5
53.5	7.5	65.3	65.2	57.5	64.0	180.4	170.6
68.5	9.8	70.3	82.8	79.0	94.3	224.9	214.0
72.0	3.3	35.1	43.9	37.1	48.2	79.9	94.9
77.5	3.5	27.3	35.3	28.0	39.2	63.8	66.7
92.5	13.5	38.5	46.5	41.1	60.3	84.5	80.5
97.3	10.4	20.7	28.2	24.1	31.1	56.9	54.6
101.8	8.3	15.6	17.2	15.5	20.6	37.4	34.9
116.3	23.5	31.5	41.1	37.1	40.1	62.3	52.1
121.0	11.4	18.1	22.6	22.1	20.4	38.3	37.7
126.8	8.8	13.6	15.1	15.7	17.5	30.2	31.7
140.3	15.2	32.0	37.8	34.6	43.2	56.8	54.3
149.5	10.5	27.4	36.8	28.6	36.1	49.8	53.2
164.8	9.9	34.4	46.1	23.6	46.9	78.3	118.5
173.5	6.6	20.6	29.5	12.8	35.7	62.9	76.9
191.0	10.8	14.0	21.9	9.3	40.8	129.5	134.9
196.0	2.3	5.3	11.0	5.5	15.8	54.6	56.2
215.5	6.3	8.4	11.8	9.6	13.3	239.9	129.8
220.5	2.4	4.4	4.1	4.4	4.9	133.9	57.6
239.0	3.8	7.2	15.6	8.2	9.0	284.6	155.3
245.5	5.6	3.9	4.6	4.5	3.4	174.4	89.4
270.0	8.3	10.9	14.3	10.6	11.9	255.7	278.2
284.5	6.2	8.4	10.4	7.7	4.5	137.0	226.3
317.5	11.3	12.9	18.4	14.4	13.0	86.3	228.7
332.3	6.1	6.3	9.5	8.3	7.3	51.3	73.0
356.8	7.8	9.0	13.3	9.4	8.1	51.1	53.1
384.0	9.7	11.4	14.0	12.3	12.6	51.1	50.6
404.0	4.9	6.2	7.0	6.9	5.6	41.8	37.5
428.8	5.4	8.6	8.1	8.4	8.0	56.1	38.4
452.3	5.5	8.0	7.0	8.6	7.7	80.9	33.1
482.0	0.0	6.2	6.3	6.1	7.2	145.8	37.4
503.0	9.7	6.5	4.5	5.5	6.3	117.4	35.9
530.0	14.5	12.5	14.7	15.0	13.0	133.6	82.5
551.8	4.8	6.5	7.2	6.5	7.0	67.9	119.8
581.0	2.7	3.6	5.1	2.9	4.6	76.3	127.7
605.0	5.8	7.4	7.8	8.4	8.4	87.8	79.6
621.0	5.8	6.8	0.2	6.8	7.2	82.4	48.7
643.8	0.0	3.9	0.0	3.8	5.2	116.4	29.0
667.3	1.6	2.5	0.0	2.4	3.7	93.7	21.8
695.0	2.9	4.0	3.0	3.1	5.0	135.8	24.3
718.3	7.5	8.8	9.5	8.8	9.8	142.7	32.5
745.5	10.4	11.9	13.7	13.7	13.4	166.6	40.8
763.3	5.8	6.0	7.4	6.6	7.4	131.3	32.8
787.8	7.6	9.6	11.2	10.2	10.1	162.8	45.1
813.8	5.3	0.8	3.5	4.8	0.4	95.8	43.1
843.5	0.7	0.2	0.0	2.7	2.4	239.0	52.5
861.8	1.4	2.3	0.0	2.3	2.9	139.9	46.4

Table D.4 (cont.): Raw Data for Batch Tests on Each Mixture Component

Time	TWAS	TWAS	Mix	Control	Control
2.0	20.1	26.0	73.2	16.6	22.2
4.0	11.7	11.5	73.2	6.4	8.5
6.0	7.2	11.6	52.8	6.2	3.6
22.0	93.8	114.1	319.1	4.5	9.5
25.0	54.9	45.1	70.4	0.7	3.2
29.0	47.5	44.2	77.3	1.9	0.7
47.5	223.2	229.6	514.8	11.8	8.9
53.5	91.2	88.8	231.6	3.1	4.5
68.5	236.8	235.7	434.3	7.3	9.4
72.0	83.0	82.8	85.9	0.0	3.9
77.5	93.8	115.2	145.7	1.7	2.8
92.5	241.2	230.2	380.2	4.0	6.5
97.3	90.2	90.3	127.8	0.0	2.8
101.8	76.8	70.6	51.2	6.2	1.6
116.3	142.9	137.1	83.3	6.9	2.1
121.0	57.3	52.9	44.2	2.9	5.2
126.8	51.1	42.7	32.0	2.6	3.5
140.3	86.7	86.9	40.1	4.9	3.5
149.5	67.8	64.5	31.7	6.7	7.0
164.8	70.3	68.9	34.1	2.3	5.7
173.5	47.5	44.0	30.1	4.7	4.4
191.0	60.0	56.0	27.1	2.7	5.2
196.0	35.4	31.8	17.3	4.4	3.1
215.5	52.3	48.6	23.4	3.6	2.5
220.5	31.8	25.7	14.3	1.7	2.1
239.0	39.1	39.6	20.6	1.3	4.4
245.5	25.0	23.3	15.1	3.0	0.0
270.0	39.9	39.3	29.7	7.7	6.9
284.5	29.9	31.3	29.1	2.4	5.3
317.5	47.8	47.5	56.9	9.5	8.6
332.3	25.3	28.1	44.5	3.1	3.5
356.8	33.6	29.9	66.6	6.8	5.6
384.0	30.7	32.7	136.8	5.5	8.9
404.0	23.2	23.7	137.6	5.9	2.7
428.8	26.7	24.7	267.6	2.5	4.3
452.3	24.3	24.0	470.3	5.9	5.2
482.0	24.4	22.1	913.6	0.0	3.2
503.0	16.7	18.1	510.9	2.1	1.6
530.0	27.7	25.9	223.7	11.7	11.3
551.8	16.5	16.0	78.3	2.7	3.5
581.0	10.5	13.0	123.4	3.6	0.0
605.0	18.2	15.7	140.2	5.2	3.6
621.0	14.2	13.7	141.1	4.6	1.4
643.8	12.0	9.8	154.9	0.0	4.2
667.3	9.2	8.7	161.8	0.0	0.0
695.0	7.7	11.5	225.4	1.9	0.0
718.3	18.6	15.9	216.9	9.6	5.7
745.5	20.7	20.5	222.7	12.6	11.4
763.3	15.0	10.9	124.7	4.0	2.7
787.8	16.8	16.8	122.3	5.1	5.6
813.8	11.5	13.6	74.8	5.9	1.8
843.5	10.5	6.5	65.6	1.0	1.5
861.8	9.3	9.2	53.6	1.8	0.0

D.4 Batch Test Study on Particle Size and Total Solids Content

Table D.5: Design Points For Batch Test Study

Bottle #	Particle Size X_1, mm	Total Solids X_2, %	Coded X_1	Coded X_2
33	2.00	10.0	-1.000	-1.000
3	6.35	10.0	1.175*	-1.000
30	2.00	20.0	-1.000	1.000
39	6.35	20.0	1.175*	1.000
36	0.85	15.0	-1.575*	0
10	8.00	15.0	2.000*	0
29	4.00	7.9	0	-1.414
26	4.00	22.1	0	1.414
48	4.00	15.0	0	0
43	4.00	15.0	0	0
34	4.00	15.0	0	0
41	4.00	15.0	0	0

*Could not use standard coding due to physical constraints

Table D.6: Biogas Methane Concentrations and pH for Batch Tests

Bottle #	Methane (%)	Final pH
33	60.80	7.90
3	61.46	7.80
30	63.20	7.70
39	59.49	7.90
36	64.84	7.80
10	63.21	7.70
29	64.20	7.60
26	65.20	7.65
48	58.06	7.70
43	63.97	7.75
34	57.75	7.60
41	58.35	7.65

Table D.7: Final Volatile Fatty Acid Content of the Batch Reactors

	Concentration, mg/L		
Bottle #	Acetic Acid	Butyric Acid	Propionic Acid
33	0	0	0
3	47.667	0	0
30	0	0	0
39	0	0	0
36	0	0	0
10	0	0	0
29	169.226	1.353	0
26	0	0	0
48	0	0	0
43	0	0	0
34	0	0	0
41	0	0	0

Table D.8: Cumulative Biogas Production for the Batch Tests (37 °C, 1 atm)

↓ Time, h	Cumulative Gas Production, mL											
→ Bottle #	30	39	34	3	26	36	10	29	48	43	33	41
0.00	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15.25	52.1	62.2	65.0	41.2	79.2	60.7	47.2	33.6	60.4	71.1	70.2	46.7
23.25	89.5	96.4	114.8	83.8	150.0	113.9	79.9	51.8	107.2	121.3	125.7	75.1
44.25	239.7	144.6	198.7	167.2	276.1	197.3	125.5	78.4	182.8	208.6	221.7	118.7
62.75	301.6	203.8	333.8	355.0	445.4	322.1	191.9	119.0	310.2	373.7	395.3	183.7
91.75	761.5	332.7	553.8	628.8	822.7	494.9	428.9	238.0	552.0	659.8	683.1	410.4
135.00	1230.6	495.9	960.2	1011.8	1269.0	843.3	648.3	374.5	880.2	1030.9	1047.7	628.5
208.00	2283.3	1169.7	1574.9	1728.4	2110.4	1401.9	1066.0	689.1	1611.6	1820.0	1491.5	1043.9
231.00	2910.3	1684.7	1802.4	1959.9	2639.4	1680.7	1322.9	900.6	1771.6	1960.0	1592.5	1321.8
256.00	3665.0	2051.3	1982.9	2142.4	3172.5	1918.6	1447.3	995.6	2232.9	2072.1	1639.4	2047.6
283.50	4397.9	2767.9	2509.4	2411.3	3730.2	2153.8	1728.2	1190.0	2699.3	2699.3	1991.8	2917.7
306.00	4965.5	3279.8	2969.1	2411.3	4195.2	2305.6	1752.8	1387.6	3172.4	3122.5	2136.0	3343.5
331.00	5538.1	3709.1	3380.2	2497.8	4672.2	2968.6	1792.4	1583.8	3410.4	3579.1	2133.5	3775.3
358.00	6017.6	4180.4	3743.8	2544.2	5075.8	3313.5	1851.7	1702.9	3656.8	3920.9	2378.9	4023.6
375.50	6246.2	4441.5	4064.4	2573.6	5305.7	3438.6	1934.6	1734.0	3870.9	4179.4	2491.2	4215.7
404.50	6587.9	4680.6	4274.7	2606.5	5683.3	3565.4	1951.1	1777.4	4009.5	4330.2	2506.4	4369.6
446.75	7066.3	4781.0	4423.9	2635.9	6454.7	3597.1	2002.9	1844.5	4051.7	4379.5	2520.8	4499.2
474.50	7434.9	4804.6	4456.2	2671.1	6821.5	3642.4	2086.2	1891.5	4108.6	4419.9	2534.5	4501.0
501.25	7596.1	4889.5	4484.7	2716.3	7197.7	3683.4	2143.1	1919.5	4127.7	4459.0	2548.6	4502.6
519.00	7728.1	4912.5	4504.0	2749.7	7250.0	3711.9	2176.3	1937.6	4204.8	4494.2	2563.2	4506.9
549.50	7860.6	4963.7	4525.7	2778.3	7322.9	3749.2	2210.5	2063.7	4238.3	4528.2	2597.4	4512.9
567.25	7894.2	4994.3	4539.0	2798.2	7394.7	3775.9	2229.1	2086.4	4262.2	4551.4	2606.0	4525.9
591.00	7968.2	5007.4	4552.6	2815.6	7493.4	3792.7	2256.5	2105.1	4307.9	4567.4	2614.4	4536.8
641.00	7989.6	5129.4	4568.3	2817.3	7643.2	3813.7	2321.6	2130.3	4328.8	4593.4	2630.4	4549.4
734.5	8080.4	5212.7	4660.4	2819.6	7717.3	4008.8	2370.9	2150.4	4467.7	4602.5	2607.3	4628.7

Table D.9: Soluble Chemical Oxygen Demand of the Batch Tests Before and After Digestion

Bottle #	Initial Volume FAS, mL	Final Volume FAS, mL	Volume FAS Titrated, mL	Soluble COD, mg O_2/L
Before Digestion				
33	10.40	14.60	4.20	1915.2
3	0.90	4.95	4.05	2188.8
30	7.95	9.80	1.85	6201.6
39	15.15	16.90	1.75	6384.0
36	17.80	20.70	2.90	4286.4
10	13.30	16.25	2.95	4195.2
29	0.05	4.65	4.60	1185.6
26	11.80	13.05	1.25	7296.0
48	0.00	2.90	2.90	4286.4
43	4.85	7.70	2.85	4377.6
34	8.80	11.80	3.00	4104.0
41	13.50	16.40	2.90	4286.4
After Digestion				
33	21.00	25.90	4.90	672.0
3	18.40	23.30	4.90	672.0
30	0.10	4.65	4.55	1344.0
39	10.35	14.85	4.50	1440.0
36	0.95	5.65	4.70	1056.0
10	14.20	18.75	4.55	1344.0
29	0.10	4.90	4.80	864.0
26	19.10	23.80	4.70	1056.0
48	9.35	14.25	4.90	672.0
43	16.40	21.00	4.60	1248.0
34	9.70	14.55	4.85	768.0
41	4.90	9.70	4.80	864.0

Table D.10: Total Solids Concentration of the Batch Tests Before and After Digestion

	Before Digestion			*After Digestion*		
Bottle #	Mass Dish Empty, g	Mass After Heat, g	TS, mg/L	Mass Dish Empty, g	Mass After Heat, g	TS, mg/L
33	1.7708	1.8998	12900	1.5848	1.6871	10230
3	1.7626	1.8832	12060	1.6226	1.7115	8890
30	1.7591	1.9944	23530	1.5816	1.7152	13360
39	1.5826	1.8082	22560	1.5739	1.7188	14490
36	1.7701	1.9438	17370	1.6131	1.7377	12460
10	1.602	1.7736	17160	1.7659	1.8996	13370
29	1.7603	1.8633	10300	1.5697	1.6524	8270
26	1.5777	1.8308	25310	1.6224	1.7622	13980
48	1.7503	1.9231	17280	1.5859	1.7001	11420
43	1.5747	1.7478	17310	1.5679	1.6853	11740
34	1.7635	1.9419	17840	1.1972	1.3155	11830
41	1.6155	1.7941	17860	1.5964	1.7144	11800

Table D.11: Volatile Solids Concentration of the Batch Tests Before and After Digestion

	Before Digestion			*After Digestion*		
Bottle #	Mass Before Heat, g	Mass After Heat, g	VS, mg/L	Mass Before Heat, g	Mass After Heat, g	VS, mg/L
33	1.8698	1.7971	7270	1.6796	1.6336	4600
3	1.9032	1.8247	7850	1.7196	1.6727	4690
30	1.9144	1.7716	14280	1.6898	1.6487	4110
39	1.7182	1.5757	14250	1.6637	1.6019	6180
36	1.9138	1.8032	11060	1.7142	1.6527	6150
10	1.7436	1.6365	10710	1.8568	1.7876	6920
29	1.8633	1.8027	6060	1.6427	1.6024	4030
26	1.8308	1.6694	16140	1.7976	1.7495	4810
48	1.9031	1.7934	10970	1.6915	1.6405	5100
43	1.7478	1.6348	11300	1.6802	1.6229	5730
34	1.9219	1.8062	11570	1.3163	1.2606	5570
41	1.7541	1.6395	11460	1.6896	1.6356	5400

Table D.12: Input for Least Squares Analysis of Batch Tests

Bottle #	Particle Size, mm	Total Solids, %	Ult. Gas Prod., L	Ult. Gas at STP, L	Gas (STP) Prod, L/g VS added	Gas (STP) Prod, L/g VS removed
33	2.00	10.0	2.607	2.311	0.489	1.332
3	6.35	10.0	2.820	2.499	0.490	1.217
30	2.00	20.0	8.080	7.163	0.772	1.084
39	6.35	20.0	5.213	4.621	0.499	0.881
36	0.85	15.0	4.009	3.553	0.494	1.113
10	8.00	15.0	2.371	2.102	0.302	0.853
29	4.00	7.9	2.150	1.906	0.484	1.445
26	4.00	22.1	7.717	6.841	0.652	0.929
48	4.00	15.0	4.468	3.960	0.555	1.038
43	4.00	15.0	4.602	4.080	0.555	1.127
34	4.00	15.0	4.660	4.131	0.549	1.059
41	4.00	15.0	4.629	4.103	0.551	1.042
			CH_4 Conc., %	% sCOD Removal	% TS Removal	% VS Removal
33	2.00	10.0	60.80	64.91	20.70	36.73
3	6.35	10.0	61.46	69.30	26.29	40.25
30	2.00	20.0	63.20	78.33	43.22	71.22
39	6.35	20.0	59.49	77.44	35.77	56.63
36	0.85	15.0	64.84	75.36	28.27	44.39
10	8.00	15.0	63.21	67.96	22.09	35.39
29	4.00	7.9	64.20	27.13	19.71	33.50
26	4.00	22.1	65.20	85.53	44.76	70.20
48	4.00	15.0	58.06	84.32	33.91	53.51
43	4.00	15.0	63.97	71.49	32.18	49.29
34	4.00	15.0	57.75	81.29	33.69	51.86
41	4.00	15.0	58.35	79.84	33.93	52.88

Table D.13: Acetic Acid Concentrations for Mixing Test of Continuous Reactors

Time, min	Acetic Acid Concentration, mg/L Port A	Port B
0.0	37.57	29.46
0.5	584.75	598.88
1.0	1018.40	1068.25
1.5	1057.14	1063.04
2.0	1061.06	1080.42
2.5	1071.67	1064.50
3.0	1048.38	1077.52
3.5	1075.94	1076.83
4.0	1078.83	1060.14
5.0	1069.51	1086.31
6.0	1052.37	1076.98
8.0	1053.53	1098.07
10.0	1061.93	1079.49
15.0	1046.46	1133.07
20.0	1099.54	1050.07

Table D.14: pH for The Continuous Digesters

Time, hours	pH				Time, hours	pH			
	Control	Thermochem	Thermal	Alkaline		Control	Thermochem	Thermal	Alkaline
0.00	7.75	7.60	7.80	7.80	265.25	7.05	8.50	7.20	7.30
3.50	7.65	7.55	7.75	7.90	281.50	6.85	8.10	7.10	7.10
19.50	7.05	8.00	7.80	7.40	289.50	6.90	8.10	6.95	7.05
22.50	6.95	8.05	7.45	7.05	313.25	7.05	8.00	7.00	6.90
27.50	6.90	8.70	7.50	7.10	336.75	7.15	8.15	7.40	7.10
41.00	7.10	8.75	7.70	6.85	405.25	7.10	8.35	7.05	7.00
42.50	6.75	9.00	8.05	6.60	424.50	7.10	8.15	7.15	7.00
51.50	6.90	9.10	8.00	6.65	478.25	6.80	8.20	7.40	7.20
52.50	6.85	9.30	8.05	6.65	497.50	7.10	8.00	6.95	6.60
69.50	6.85	9.50	8.25	6.75	520.50	7.05	8.20	7.10	6.80
71.00	6.85	9.55	8.25	6.80	564.50	7.00	7.75	7.35	6.60
73.50	6.85	8.75	8.35	6.80	588.75	7.15	8.20	7.15	6.85
91.25	7.00	9.00	8.35	6.80	655.25	7.25	8.05	7.65	6.60
93.50	6.95	8.75	8.25	6.90	698.00	7.40	7.70	7.50	6.50
96.50	6.95	8.65	7.95	6.90	743.75	7.20	7.85	7.60	6.80
114.50	7.15	8.50	7.15	6.95	791.25	7.55	8.05	7.70	7.10
118.50	7.00	8.65	6.80	6.90	840.40	7.35	8.30	7.30	6.90
120.00	6.90	8.80	7.45	7.20	887.25	7.25	8.25	7.20	6.75
141.50	7.30	8.20	7.70	7.20	935.25	7.20	8.25	7.10	7.05
209.75	7.00	8.25	7.35	7.20	983.50	6.95	8.10	7.05	6.80
218.25	7.10	8.30	7.50	7.20	1031.75	7.15	8.20	7.15	6.95
257.25	6.85	8.20	7.15	7.10	1080.50	7.05	8.15	7.10	6.85

Table D.15: Biogas Production for Continuous Digesters

Time,	Gas Production (mL)				Cumulative Gas Production (L STP)			
hours	Control	Thermochem.	Thermal	Alkaline	Control	Thermochem	Thermal	Alkaline
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3.50	363	520	453	514	0.32	0.46	0.40	0.46
19.50	5855	6387	6272	6866	5.51	6.12	5.96	6.54
22.50	1203	1294	1222	1387	6.58	7.27	7.04	7.77
27.50	1676	2059	1768	2097	8.06	9.09	8.61	9.63
41.00	4500	4893	3731	5586	12.05	13.43	11.92	14.58
42.50	511	526	419	633	12.51	13.90	12.29	15.14
51.50	2984	2829	2515	3648	15.15	16.41	14.52	18.38
52.50	369	307	273	397	15.48	16.68	14.76	18.73
69.50	5165	3984	4542	6582	20.06	20.21	18.79	24.56
71.00	391	326	379	565	20.40	20.50	19.12	25.06
73.50	715	591	606	917	21.04	21.02	19.66	25.88
91.25	4400	4076	4674	6111	24.94	24.64	23.80	31.29
93.50	656	477	546	763	25.52	25.06	24.29	31.97
96.50	744	575	794	1084	26.18	25.57	24.99	32.93
114.50	4511	4366	4732	6508	30.18	29.44	29.19	38.70
118.50	1075	820	1025	1343	31.13	30.16	30.09	39.89
120.00	356	296	383	529	31.44	30.43	30.43	40.36
141.50	5068	4765	5683	8184	35.94	34.65	35.47	47.61
209.75	17542	15310	17748	23092	51.49	48.22	51.20	68.08
218.25	2497	1968	2160	2938	53.70	49.97	53.12	70.69
257.25	10302	7964	9761	13686	62.83	57.03	61.77	82.82
265.25	2193	1777	2076	2744	64.78	58.60	63.61	85.25
281.50	3987	3384	3996	5932	68.31	61.60	67.15	90.51
289.50	1775	1213	2138	2710	69.88	62.68	69.05	92.91
313.25	5858	5467	5934	8120	75.08	67.52	74.31	100.11
336.75	5591	4969	5841	8334	80.03	71.93	79.49	107.50
405.25	15222	14156	16618	22745	93.53	84.47	94.22	127.66
424.50	4246	3845	5044	6947	97.29	87.88	98.69	133.82
478.25	13519	12642	14522	18493	109.27	99.09	111.56	150.21
497.50	4061	3718	4656	6384	112.87	102.38	115.69	155.87
520.50	5166	4647	5968	7921	117.45	106.50	120.98	162.89
564.50	10396	8823	11642	15856	126.67	114.32	131.30	176.94
588.75	5923	5614	6341	8242	131.92	119.30	136.92	184.25
655.25	14539	12492	16973	22274	144.80	130.37	151.96	203.99
698.00	9193	8383	10546	15007	152.95	137.81	161.31	217.30
743.75	10234	9382	11156	15612	162.02	146.12	171.20	231.13
791.25	12121	11290	12711	15808	172.77	156.13	182.47	245.15
840.40	11405	10476	12299	17919	182.88	165.42	193.37	261.03
887.25	10688	9143	11264	16314	192.35	173.52	203.35	275.49
935.25	10461	9242	11746	16483	201.63	181.71	213.77	290.10
983.50	10977	8590	12048	17729	211.36	189.33	224.45	305.82
1031.75	10459	8677	12823	16941	220.63	197.02	235.81	320.83
1080.50	10804	9524	12491	17366	230.20	205.46	246.88	336.23

Table D.16: Volatile Fatty Acid Concentrations for Continuous Digesters

Time, hours	Control				Thermochemical			
	Acetic	Propionic	Butyric	Total VFA	Acetic	Propionic	Butyric	Total VFA
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
22.50	0.00	0.00	0.00	0.00	266.89	0.00	0.00	266.89
41.00	271.76	0.00	0.00	271.76	194.74	0.00	0.00	194.74
91.25	216.48	0.00	0.00	216.48	214.68	0.00	0.00	214.68
114.50	288.99	0.00	0.00	288.99	329.76	0.00	0.00	329.76
141.50	414.38	0.00	0.00	414.38	504.64	0.00	0.00	504.64
194.50	718.93	0.00	0.00	718.93	444.14	0.00	0.00	444.14
218.25	838.97	0.00	0.00	838.97	469.92	0.00	0.00	469.92
257.25	948.93	115.23	0.00	1064.17	431.40	0.00	0.00	431.40
313.25	672.15	0.00	0.00	672.15	484.92	0.00	0.00	484.92
336.75	852.08	0.00	0.00	852.08	392.23	0.00	0.00	392.23
405.25	822.57	0.00	0.00	822.57	346.85	0.00	0.00	346.85
424.50	730.30	0.00	0.00	730.30	396.59	0.00	0.00	396.59
478.25	981.31	125.22	0.00	1106.54	359.06	0.00	0.00	359.06
520.50	716.09	0.00	0.00	716.09	357.16	0.00	0.00	357.16
564.50	886.55	0.00	0.00	886.55	429.25	0.00	0.00	429.25
588.75	795.73	0.00	0.00	795.73	322.50	0.00	0.00	322.50
655.25	599.42	0.00	0.00	599.42	409.58	0.00	0.00	409.58
698.00	650.16	0.00	0.00	650.16	500.00	0.00	0.00	500.00
743.75	705.14	0.00	0.00	705.14	454.60	0.00	0.00	454.60
791.25	514.86	0.00	0.00	514.86	255.73	0.00	0.00	255.73
840.40	538.06	0.00	0.00	538.06	201.38	0.00	0.00	201.38
887.25	378.61	0.00	0.00	378.61	324.45	0.00	0.00	324.45
935.25	311.46	0.00	0.00	311.46	360.42	0.00	0.00	360.42
983.50	997.04	157.97	0.00	1155.01	465.62	0.00	0.00	465.62
1031.75	462.41	0.00	0.00	462.41	471.54	0.00	0.00	471.54
1080.50	243.26	0.00	0.00	243.26	496.19	0.00	0.00	496.19

Time, hours	Thermal				Alkaline			
	Acetic	Propionic	Butyric	Total VFA	Acetic	Propionic	Butyric	Total VFA
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
22.50	197.35	0.00	0.00	197.35	365.92	0.00	0.00	365.92
41.00	174.89	0.00	0.00	174.89	480.01	0.00	0.00	480.01
91.25	185.52	0.00	0.00	185.52	576.30	86.81	0.00	663.11
114.50	410.04	0.00	0.00	410.04	444.82	0.00	0.00	444.82
141.50	297.54	0.00	0.00	297.54	390.18	0.00	0.00	390.18
194.50	401.85	0.00	0.00	401.85	433.42	0.00	0.00	433.42
218.25	356.07	0.00	0.00	356.07	349.62	0.00	0.00	349.62
257.25	399.74	0.00	0.00	399.74	408.78	0.00	0.00	408.78
313.25	504.32	123.51	0.00	627.82	704.60	0.00	0.00	704.60
336.75	334.67	0.00	0.00	334.67	578.95	0.00	0.00	578.95
405.25	447.07	0.00	0.00	447.07	698.21	0.00	0.00	698.21
424.50	369.93	0.00	0.00	369.93	625.30	0.00	0.00	625.30
478.25	353.28	0.00	0.00	353.28	540.55	0.00	0.00	540.55
520.50	479.21	0.00	0.00	479.21	813.48	123.53	0.00	937.01
564.50	459.08	0.00	0.00	459.08	978.05	137.02	0.00	1115.07
588.75	483.14	0.00	0.00	483.14	789.87	0.00	0.00	789.87
655.25	309.47	0.00	0.00	309.47	890.77	0.00	0.00	890.77
698.00	368.17	0.00	0.00	368.17	1087.90	188.96	0.00	1276.86
743.75	326.39	0.00	0.00	326.39	845.50	0.00	0.00	845.50
791.25	275.39	0.00	0.00	275.39	631.32	0.00	0.00	631.32
840.40	314.68	0.00	0.00	314.68	742.98	0.00	0.00	742.98
887.25	454.94	0.00	0.00	454.94	989.64	0.00	0.00	989.64
935.25	513.21	0.00	0.00	513.21	750.50	0.00	0.00	750.50
983.50	583.86	90.61	0.00	674.47	903.18	0.00	0.00	903.18
1031.75	474.32	0.00	0.00	474.32	730.03	0.00	0.00	730.03
1080.50	490.83	0.00	0.00	490.83	644.85	0.00	0.00	644.85

Table D.17: Soluble Chemical Oxygen Demand for Continuous Digesters

	Control				Thermochemical			
Time hours	Initial Vol. (mL)	Final Vol. (mL)	Volume Titr. (mL)	COD (mg O_2/L)	Initial Vol. (mL)	Final Vol. (mL)	Volume Titr. (mL)	COD (mg O_2/L)
0.00	0.10	3.15	3.05	4717	3.15	5.60	2.45	6486
22.50	11.15	14.25	3.10	4570	14.25	16.75	2.50	6339
41.00	0.00	3.15	3.15	4422	3.15	5.70	2.55	6192
91.25	11.45	14.70	3.25	4128	14.70	17.40	2.70	5749
114.50	0.05	3.30	3.25	4128	3.30	5.90	2.60	6044
141.50	11.75	14.95	3.20	4275	14.95	17.55	2.60	6044
194.50	0.30	3.70	3.40	3685	3.70	6.35	2.65	5897
218.25	12.30	15.65	3.35	3833	15.65	18.25	2.60	6044
257.25	0.00	3.30	3.30	3980	3.30	6.00	2.70	5749
313.25	12.20	15.65	3.45	3538	15.65	18.35	2.70	5749
336.75	0.00	3.40	3.40	3685	3.40	6.15	2.75	5602
405.25	12.55	15.95	3.40	3685	15.95	18.65	2.70	5749
424.50	3.55	7.00	3.45	3538	7.00	9.80	2.80	5454
478.25	16.25	19.80	3.55	3243	19.80	22.60	2.80	5454
520.50	6.55	10.15	3.60	3096	10.15	12.90	2.75	5602
564.50	19.50	23.00	3.50	3390	0.10	2.85	2.75	5602
588.75	9.25	12.75	3.50	3390.857	12.75	15.45	2.70	5749
655.25	0.00	3.60	3.60	3096	3.60	6.40	2.80	5454
698.00	13.00	16.55	3.55	3243	16.55	19.35	2.80	5454
743.75	3.35	7.00	3.65	2948	7.00	9.85	2.85	5307
791.25	16.60	20.30	3.70	2801	20.30	23.20	2.90	5160
840.40	6.80	10.50	3.70	2801	10.50	13.45	2.95	5012
887.25	20.30	24.05	3.75	2653	0.20	3.20	3.00	4865
935.25	10.10	13.85	3.75	2653	13.85	16.80	2.95	5012
983.50	0.00	3.80	3.80	2506	3.80	6.80	3.00	4865
1031.75	13.65	17.45	3.80	2506	17.45	20.50	3.05	4717
1080.50	3.70	7.50	3.80	2506	7.50	10.50	3.00	4865

	Thermal				Alkaline			
Time hours	Initial Vol. (mL)	Final Vol. (mL)	Volume Titr. (mL)	COD (mg O_2/L)	Initial Vol. (mL)	Final Vol. (mL)	Volume Titr. (mL)	COD (mg O_2/L)
0.00	5.60	8.50	2.90	5160	8.50	11.15	2.65	5897
22.50	16.75	19.70	2.95	5012	19.70	22.40	2.70	5749
41.00	5.70	8.65	2.95	5012	8.65	11.45	2.80	5454
91.25	17.40	20.30	2.90	5160	20.30	23.05	2.75	5602
114.50	5.90	8.85	2.95	5012	8.85	11.75	2.90	5160
141.50	17.55	20.50	2.95	5012	20.50	23.30	2.80	5454
194.50	6.35	9.50	3.15	4422	9.50	12.30	2.80	5454
218.25	18.25	21.35	3.10	4570	21.35	24.35	3.00	4865
257.25	6.00	9.15	3.15	4427	9.15	12.20	3.05	4717
313.25	18.35	21.55	3.20	4275	21.55	24.55	3.00	4865
336.75	6.15	9.35	3.20	4275	9.35	12.55	3.20	4275
405.25	18.65	21.85	3.20	4275	-- 0.40	3.55	3.15	4422
424.50	9.80	13.05	3.25	4128	13.05	16.25	3.20	4275
478.25	0.00	3.25	3.25	4128	3.25	6.55	3.30	3980
520.50	12.90	16.20	3.30	3980	16.20	19.50	3.30	3980
564.50	2.85	6.00	3.15	4422	6.00	9.25	3.25	4128
588.75	15.45	18.70	3.25	4128	18.70	22.00	3.30	3980
655.25	6.40	9.70	3.30	3980	9.70	13.00	3.30	3980
698.00	19.35	22.55	3.20	4275	0.00	3.35	3.35	3833
743.75	9.85	13.20	3.35	3833	13.20	16.60	3.40	3685
791.25	0.10	3.40	3.30	3980	3.40	6.80	3.40	3685
840.40	13.45	16.80	3.35	3833	16.80	20.30	3.50	3390
887.25	3.20	6.60	3.40	3685	6.60	10.10	3.50	3390
935.25	16.80	20.10	3.30	3980	20.10	23.60	3.50	3390
983.50	6.80	10.10	3.30	3980	10.10	13.65	3.55	3243
1031.75	20.50	23.95	3.45	3538	0.10	3.70	3.60	3096
1080.50	10.50	14.00	3.50	3390	14.00	17.70	3.70	2801

Table D.18: Total Solids Concentrations for Continuous Digesters

	Control			Thermochemical		
Time hours	Mass Empty (g)	Mass After (g)	TS (mg/L)	Mass Empty (g)	Mass After (g)	TS (mg/L)
0.00	1.4531	1.6413	18820	1.4609	1.6779	21700
22.50	1.4519	1.6387	18680	1.4611	1.6932	23210
41.00	1.4722	1.6552	18300	1.7645	1.9765	21200
91.25	1.4544	1.6311	17670	1.4551	1.6591	20400
114.50	1.4576	1.6348	17720	1.4547	1.6559	20120
141.50	1.4592	1.6323	17310	1.4633	1.6658	20250
194.50	1.5673	1.7340	16670	1.5690	1.7701	20110
218.25	1.5668	1.7315	16470	1.4013	1.6046	20330
257.25	1.5548	1.7169	16210	1.5666	1.7697	20310
313.25	1.3858	1.5484	16260	1.2568	1.4587	20190
336.75	1.4156	1.5761	16050	1.5688	1.7648	19600
405.25	1.3914	1.5414	15000	1.4174	1.6162	19880
424.50	1.5854	1.7451	15970	1.3855	1.5882	20270
478.25	1.4092	1.5622	15300	1.3899	1.5865	19660
520.50	1.4657	1.6204	15470	1.4673	1.6614	19410
564.50	1.5408	1.6948	15400	1.3245	1.5229	19840
588.75	1.4717	1.6252	15350	1.4577	1.6567	19900
655.25	1.5244	1.6763	15190	1.4302	1.6331	20290
698.00	1.4278	1.5789	15110	1.2775	1.4685	19100
743.75	1.4719	1.6222	15030	1.3698	1.5700	20020
791.25	1.4459	1.5955	14960	1.4474	1.6418	19440
840.40	1.3229	1.4704	14750	1.4539	1.6477	19380
887.25	1.3588	1.5019	14310	1.3766	1.5669	19030
935.25	1.4667	1.6143	14760	1.3288	1.5143	18550
983.50	1.3695	1.5158	14630	1.4533	1.6447	19140
1031.75	1.4087	1.5507	14200	1.3701	1.5539	18380
1080.50	1.5029	1.6442	14130	1.2649	1.4467	18180

	Thermal			Alkaline		
Time hours	Mass Empty (g)	Mass After (g)	TS (mg/L)	Mass Empty (g)	Mass After (g)	TS (mg/L)
0.00	1.4693	1.6941	22480	1.4565	1.6858	22930
22.50	1.4520	1.6909	23890	1.4548	1.6698	21500
41.00	1.4507	1.7062	25550	1.4521	1.6760	22390
91.25	1.4561	1.7014	24530	1.4561	1.6694	21330
114.50	1.4366	1.6484	21180	1.6033	1.8087	20540
141.50	1.4580	1.6642	20620	1.4722	1.6791	20690
194.50	1.2829	1.4901	20720	1.4309	1.6440	21310
218.25	0.9988	1.2073	20850	1.5726	1.7759	20330
257.25	1.5615	1.7588	19730	1.3777	1.5800	20230
313.25	1.4421	1.6422	20010	1.5706	1.7619	19130
336.75	1.4294	1.6342	20480	1.3989	1.5968	19790
405.25	1.3962	1.6001	20390	1.4305	1.6237	19320
424.50	1.4167	1.6158	19910	1.3706	1.5621	19150
478.25	1.3893	1.5831	19380	1.5754	1.7633	18790
520.50	1.4611	1.6599	19880	1.2729	1.4642	19130
564.50	1.4352	1.6281	19290	1.5431	1.7317	18860
588.75	1.3278	1.5200	19220	1.4578	1.6423	18450
655.25	1.2399	1.4336	19370	1.4412	1.6248	18360
698.00	1.4564	1.6542	19780	1.3765	1.5631	18660
743.75	1.2273	1.4224	19510	1.4599	1.6374	17750
791.25	1.4355	1.6287	19320	1.2744	1.4497	17530
840.40	1.4770	1.6705	19350	1.4461	1.6118	16570
887.25	1.3798	1.5688	18900	1.4533	1.6169	16360
935.25	1.4453	1.6319	18660	1.3669	1.5276	16070
983.50	1.3766	1.5572	18060	1.2603	1.4184	15810
1031.75	1.4499	1.6355	18560	1.4732	1.6279	15470
1080.50	1.4732	1.6544	18120	1.5153	1.6692	15390

Table D.19: Volatile Solids Concentrations for Continuous Digesters

	Control			Thermochemical		
Time hours	Mass Before (g)	Mass After (g)	VS (mg/L)	Mass Before (g)	Mass After (g)	VS (mg/L)
0.00	1.6413	1.5407	10060	1.6779	1.5593	11860
22.50	1.6387	1.5356	10310	1.6932	1.5674	12580
41.00	1.6552	1.5617	9350	1.9765	1.8565	12000
91.25	1.6311	1.5412	8990	1.6591	1.5418	11730
114.50	1.6348	1.5457	8910	1.6559	1.5438	11210
141.50	1.6323	1.5507	8160	1.6658	1.5561	10970
194.50	1.7340	1.6542	7980	1.7701	1.6626	10750
218.25	1.7315	1.6585	7300	1.6046	1.4918	11280
257.25	1.7169	1.6457	7120	1.7697	1.6556	11410
313.25	1.5484	1.4752	7320	1.4587	1.3494	10930
336.75	1.5761	1.5016	7450	1.7648	1.6589	10590
405.25	1.5414	1.4671	7430	1.6162	1.5086	10760
424.50	1.7451	1.6747	7040	1.5882	1.4838	10440
478.25	1.5622	1.4938	6840	1.5865	1.4888	9770
520.50	1.6204	1.5526	6780	1.6614	1.5673	9410
564.50	1.6948	1.6271	6770	1.5229	1.4256	9730
588.75	1.6252	1.5609	6430	1.6567	1.5598	9690
655.25	1.6763	1.6093	6700	1.6331	1.5380	9510
698.00	1.5789	1.5117	6720	1.4685	1.3705	9800
743.75	1.6222	1.5554	6680	1.5700	1.4792	9080
791.25	1.5955	1.5335	6200	1.6418	1.5543	8750
840.40	1.4704	1.4114	5900	1.6477	1.5632	8450
887.25	1.5019	1.4440	5790	1.5669	1.4777	8920
935.25	1.6143	1.5588	5550	1.5143	1.4309	8340
983.50	1.5158	1.4623	5350	1.6447	1.5561	8860
1031.75	1.5507	1.4949	5580	1.5539	1.4711	8280
1080.50	1.6442	1.5906	5360	1.4467	1.3647	8200

	Thermal			Alkaline		
Time hours	Mass Before (g)	Mass After (g)	VS (mg/L)	Mass Before (g)	Mass After (g)	VS (mg/L)
0.00	1.6941	1.5848	10930	1.6858	1.5750	11080
22.50	1.6909	1.5844	10650	1.6698	1.5561	11370
41.00	1.7062	1.5981	10810	1.6760	1.5734	10260
91.25	1.7014	1.6011	10030	1.6694	1.5596	10980
114.50	1.6484	1.5488	9960	1.8087	1.7088	9990
141.50	1.6642	1.5787	8550	1.6791	1.5770	10210
194.50	1.4901	1.3942	9590	1.6440	1.5509	9310
218.25	1.2073	1.1161	9120	1.7759	1.6825	9340
257.25	1.7588	1.6725	8630	1.5800	1.4956	8440
313.25	1.6422	1.5580	8420	1.7619	1.6779	8400
336.75	1.6342	1.5458	8840	1.5968	1.5100	8680
405.25	1.6001	1.5121	8800	1.6237	1.5407	8300
424.50	1.6158	1.5299	8590	1.5621	1.4805	8160
478.25	1.5831	1.4983	8480	1.7633	1.6801	8320
520.50	1.6599	1.5783	8160	1.4642	1.3892	7500
564.50	1.6281	1.5449	8320	1.7317	1.6578	7390
588.75	1.5200	1.4411	7890	1.6423	1.5705	7180
655.25	1.4336	1.3560	7760	1.6248	1.5594	6540
698.00	1.6542	1.5708	8340	1.5631	1.4992	6390
743.75	1.4224	1.3458	7660	1.6374	1.5777	5970
791.25	1.6287	1.5502	7850	1.4497	1.3870	6270
840.40	1.6705	1.5936	7690	1.6118	1.5563	5550
887.25	1.5688	1.4984	7040	1.6169	1.5621	5480
935.25	1.6319	1.5597	7220	1.5276	1.4786	4900
983.50	1.5572	1.4933	6390	1.4184	1.3688	4960
1031.75	1.6355	1.5672	6830	1.6279	1.5764	5150
1080.50	1.6544	1.5893	6510	1.6692	1.6212	4800

D.5 Study of Thermal Pretreatment

Table D.20: Design Points For Batch Test Study

Bottle #	Particle Size X_1, mm	Total Solids X_2, %	Coded X_1	Coded X_2
20	2.00	10.0	-1.000	-1.000
11	6.35	10.0	1.175*	-1.000
12	2.00	20.0	-1.000	1.000
35	6.35	20.0	1.175*	1.000
31	0.85	15.0	-1.575*	0.000
8	8.00	15.0	2.000*	0.000
42	4.00	7.9	0.000	-1.414
16	4.00	22.1	0.000	1.414
46	4.00	15.0	0.000	0.000
24	4.00	15.0	0.000	0.000
9	4.00	15.0	0.000	0.000
17	4.00	15.0	0.000	0.000

* Could not use standard coding due to physical constraints

Table D.21: Biogas Methane Concentrations and pH for Batch Tests

Bottle #	Methane	Final pH
20	60.46	7.90
11	59.72	7.80
12	58.63	7.80
35	59.04	7.65
31	57.27	7.85
8	59.92	7.80
42	60.15	8.00
16	58.81	8.10
46	58.66	7.80
24	59.89	7.60
9	61.11	7.70
17	57.70	7.55

Table D.22: Final Volatile Fatty Acid Content of the Batch Reactors

	Concentration	(mg/L)	
Bottle #	Acetic Acid	Butyric Acid	Propionic Acid
20	32.66	0	0
11	0	0	0
12	0	0	0
35	173.21	0	0
31	0	0	0
8	0	0	0
42	0	0	0
16	0	0	0
46	73.48	0	0
24	0	0	0
9	0	0	0
17	0	0	0

Table D.23: Cumulative Biogas Production for the Batch Tests (37 °C, 1 atm)

Time	Cumulative Gas Production, mL											
Bottle #	12	35	20	11	16	31	8	42	46	24	9	17
0.00	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15.25	456.3	240.0	88.0	122.4	228.8	91.2	131.3	92.2	152.6	156.0	163.4	166.0
23.25	533.8	360.2	150.9	161.2	321.0	145.6	182.9	139.2	224.6	221.2	232.3	238.2
44.25	619.9	546.5	232.2	212.7	445.0	201.2	253.2	180.5	367.3	345.5	369.6	363.2
62.75	766.6	994.5	389.4	346.5	595.2	258.8	426.2	296.0	619.9	533.1	628.2	607.4
91.75	1030.7	1438.9	647.2	601.9	1099.7	352.8	767.5	462.4	951.2	866.7	998.8	981.8
135.00	1258.1	1965.8	975.0	833.5	1466.3	511.8	1007.7	624.5	1379.8	1249.8	1451.8	1365.5
208.00	1774.1	2771.4	1616.4	1197.8	1821.0	878.1	1382.7	948.3	2095.1	2050.9	2275.9	2091.3
231.00	2102.0	3180.2	1881.7	1432.7	2072.2	1111.7	1644.4	1164.8	2463.0	2426.3	2680.7	2459.6
256.00	2448.8	3538.3	2095.6	1678.5	2435.7	1368.7	1954.7	1397.6	2786.9	2755.4	2995.7	2868.7
283.50	2857.9	3917.6	2278.6	1997.9	2964.3	1651.3	2285.1	1664.0	3058.7	3032.3	3358.7	3217.3
306.00	3215.1	4280.8	2432.0	2365.6	3378.7	1842.0	2547.5	1850.9	3231.7	3215.0	3548.1	3469.0
331.00	3565.5	4728.1	2559.8	2554.9	3733.3	1997.3	2800.7	1993.8	3359.1	3374.8	3687.9	3710.6
358.00	3825.2	5236.7	2633.4	2720.0	4058.5	2133.0	3039.0	2112.3	3406.1	3494.1	3760.5	3946.1
375.50	3970.2	5554.3	2671.0	2810.7	4240.9	2215.6	3162.7	2177.4	3438.7	3526.9	3800.3	4067.4
404.50	4149.1	6018.2	2700.4	2984.9	4578.7	2355.9	3303.2	2293.7	3461.7	3562.8	3829.5	4119.0
446.75	4473.5	6479.3	2727.8	3200.8	5115.9	2418.3	3518.9	2355.3	3505.6	3592.8	3869.8	4166.3
474.50	4729.9	6625.8	2754.6	3261.9	5463.1	2450.7	3732.2	2387.6	3538.4	3622.3	3905.0	4201.9
501.25	4913.7	6697.4	2782.4	3295.5	5688.6	2477.6	3950.6	2411.7	3581.1	3655.2	3934.1	4233.9
519.00	4978.1	6749.5	2804.1	3318.4	5783.0	2493.2	4090.7	2428.0	3613.2	3676.1	3961.9	4256.8
549.50	5022.6	6799.9	2831.5	3343.4	5935.1	2513.5	4169.3	2447.5	3660.9	3701.7	3964.5	4291.5
567.25	5047.6	6836.7	2850.9	3358.8	6061.7	2527.6	4207.5	2460.7	3685.9	3719.8	3986.2	4313.9
591.00	5070.6	6868.6	2852.9	3370.9	6227.5	2539.1	4231.7	2469.5	3706.1	3738.6	4013.3	4334.6
641.00	5104.9	6914.5	2888.6	3389.7	6662.0	2558.0	4260.8	2483.8	3736.9	3775.9	4057.7	4368.7
667.25	5130.0	6969.3	2911.9	3409.4	6929.6	2579.6	4291.5	2499.6	3767.1	3805.7	4096.3	4406.5
693.00	5167.1	7012.2	2945.6	3435.6	7109.9	2600.2	4325.9	2518.7	3799.4	3846.9	4136.1	4444.9
710.25	5184.9	7050.4	2978.1	3444.1	7225.8	2624.7	4361.1	2536.4	3836.9	3889.1	4179.5	4481.6
734.50	5246.3	7103.7	3017.5	3469.7	7290.3	2647.9	4397.7	2559.5	3872.7	3937.6	4259.7	4523.9

Table D.24: Soluble Chemical Oxygen Demand of the Batch Tests Before and After Digestion

Bottle #	Initial Volume FAS (mL)	Final Volume FAS (mL)	Volume FAS Titrated (mL)	Soluble COD (mg O_2/L)
Before Digestion				
20	18.15	22.30	4.15	2006.4
11	17.50	21.50	4.00	2280.0
12	4.40	6.20	1.80	6292.8
35	0.00	1.60	1.60	6657.6
31	13.55	16.35	2.80	4468.8
8	8.80	11.65	2.85	4377.6
42	13.05	17.60	4.55	1276.8
16	0.00	1.10	1.10	7569.6
46	8.90	11.65	2.75	4560.0
24	18.60	21.30	2.70	4651.2
9	4.65	7.55	2.90	4286.4
17	4.10	6.95	2.85	4377.6
After Digestion				
20	4.60	9.45	4.85	768.0
11	7.50	12.40	4.90	672.0
12	12.20	16.90	4.70	1056.0
35	0.00	4.60	4.60	1248.0
31	5.65	10.35	4.70	1056.0
8	18.75	23.35	4.60	1248.0
42	14.55	19.35	4.80	864.0
16	18.20	23.05	4.85	768.0
46	9.50	14.50	5.00	480.0
24	14.30	18.90	4.60	1248.0
9	9.50	14.40	4.90	672.0
17	23.50	28.35	4.85	768.0

Table D.25: Total Solids Concentration of the Batch Tests Before and After Digestion

	Before Digestion			*After Digestion*		
Bottle #	Mass Empty (g)	Mass After (g)	TS (mg/L)	Mass Empty (g)	Mass After (g)	TS (mg/L)
20	1.7861	1.9026	11650	1.7599	1.857	9710
11	1.7723	1.9036	13130	1.7423	1.8365	9420
12	1.5036	1.7377	23410	1.629	1.7447	11570
35	1.7762	2.0052	22900	1.5761	1.7331	15700
31	1.5036	1.672	16840	1.5573	1.6824	12510
8	1.5718	1.7454	17360	1.5579	1.6924	13450
42	1.6233	1.7266	10330	1.769	1.8499	8090
16	1.7392	1.9921	25290	1.6382	1.7708	13260
46	1.7369	1.9086	17170	1.7713	1.8811	10980
24	1.5881	1.7527	16460	1.5989	1.709	11010
9	1.7592	1.9236	16440	1.7615	1.8681	10660
17	1.1943	1.3727	17840	1.7782	1.8955	11730

Table D.26: Volatile Solids Concentration of the Batch Tests Before and After Digestion

	Before Digestion			*After Digestion*		
Bottle #	Mass Before (g)	Mass After (g)	VSS (mg/L)	Mass Before (g)	Mass After (g)	VSS (mg/L)
20	1.9026	1.829	7360	1.857	1.8111	4590
11	1.9036	1.8249	7870	1.8365	1.7916	4490
12	1.7377	1.5933	14440	1.7447	1.7055	3920
35	2.0052	1.8613	14390	1.7331	1.6743	5880
31	1.672	1.5615	11050	1.6824	1.6179	6450
8	1.7454	1.638	10740	1.6924	1.6252	6720
42	1.7266	1.6672	5940	1.8499	1.812	3790
16	1.9921	1.8302	16190	1.7708	1.7281	4270
46	1.9086	1.7953	11330	1.8811	1.8313	4980
24	1.7527	1.6443	10840	1.709	1.6574	5160
9	1.9236	1.8146	10900	1.8681	1.8177	5040
17	1.3727	1.2604	11230	1.8955	1.8445	5100

Table D.27: Input for Least Squares Analysis of Batch Tests

Part. Size coded X_1	Total Solids coded X_2	Ult. Gas Prod. (L)	Ult. Gas @ STP	Gas Prod. (L/g VSS added)	Gas Prod. (L/g VSS removed)
2.00	10.0	3.018	2.675	0.559	1.486
6.35	10.0	3.470	3.076	0.601	1.400
2.00	20.0	5.246	4.650	0.495	0.680
6.35	20.0	7.104	6.297	0.673	1.138
0.85	15.0	2.648	2.347	0.327	0.785
8.00	15.0	4.398	3.898	0.558	1.492
4.00	7.9	2.560	2.269	0.588	1.624
4.00	22.1	7.290	6.462	0.614	0.834
4.00	15.0	3.873	3.433	0.466	0.832
4.00	15.0	3.934	3.487	0.495	0.945
4.00	15.0	4.260	3.776	0.533	0.991
4.00	15.0	4.524	4.010	0.549	1.006
		CH_4 Conc. (%)	% COD Removal	% TS Removal	% VS Removal
2.00	10.0	60.46	61.72	16.65	37.64
6.35	10.0	59.72	70.53	28.26	42.95
2.00	20.0	58.63	83.22	50.58	72.85
6.35	20.0	59.04	81.25	31.44	59.14
0.85	15.0	57.27	76.37	25.71	41.63
8.00	15.0	59.92	71.49	22.52	37.43
4.00	7.9	60.15	32.33	21.68	36.20
4.00	22.1	58.81	89.85	47.57	73.63
4.00	15.0	58.66	89.47	36.05	56.05
4.00	15.0	59.89	73.17	33.11	52.40
4.00	15.0	61.11	84.32	35.16	53.76
4.00	15.0	57.70	82.46	34.25	54.59

D.6 Study of Thermochemical Pretreatment

Table D.28: Design Points For Batch Test Study

Bottle #	Part. Size X_1, mm	Total Solids X_2, %	Alk. Dose X_3, meq/L	Coded X_1	Coded X_2	Coded X_3
11	2.00	10.00	50	-1.000	-1.000	-1.000
36	6.35	10.00	50	1.175	-1.000	-1.000
25	2.00	20.00	50	-1.000	1.000	-1.000
18	6.35	20.00	50	1.175	1.000	-1.000
28	2.00	10.00	150	-1.000	-1.000	1.000
45	6.35	10.00	150	1.175 *	-1.000	1.000
27	2.00	20.00	150	-1.000	1.000	1.000
32	6.35	20.00	150	1.175 *	1.000	1.000
31	4.00	15.00	16	0.000	0.000	-1.682
28-b	4.00	15.00	184	0.000	0.000	1.682
37	4.00	6.59	100	0.000	-1.682	0.000
18-b	4.00	23.41	100	0.000	1.682	0.000
19-b	0.85	15.00	100	-1.575 *	0.000	0.000
25-b	8.00	15.00	100	2.000 *	0.000	0.000
21	4.00	15.00	100	0.000	0.000	0.000
43	4.00	15.00	100	0.000	0.000	0.000
24	4.00	15.00	100	0.000	0.000	0.000
48	4.00	15.00	100	0.000	0.000	0.000
39	4.00	15.00	100	0.000	0.000	0.000
3	4.00	15.00	100	0.000	0.000	0.000

* Could not use standard coding due to physical constraints

Table D.29: Biogas Methane Concentrations and pH for Batch Tests

Bottle #	Methane (%)	Final pH
11	56.88	7.80
36	58.62	8.30
25	57.67	8.15
18	60.10	8.10
28	61.34	7.85
45	63.22	7.65
27	57.50	7.75
32	60.90	7.85
31	58.78	7.55
28-b	58.70	7.65
37	59.49	8.30
18-b	53.82	7.80
19-b	56.40	7.55
25-b	60.18	7.45
21	59.45	7.45
43	58.14	8.40
24	56.63	7.45
48	58.99	7.50
39	59.51	7.55
3	61.69	7.45

Table D.30: Final Volatile Fatty Acid Content of the Batch Reactors

Bottle #	Concentration (mg/L)		
	Acetic Acid	Butyric Acid	Propionic Acid
11	0.0	0.0	0.0
36	0.0	0.0	0.0
25	0.0	0.0	0.0
18	0.0	0.0	0.0
28	0.0	0.0	0.0
45	0.0	0.0	0.0
27	0.0	0.0	0.0
32	0.0	0.0	0.0
31	670.3	0.0	0.0
28-b	0.0	0.0	0.0
37	0.0	0.0	0.0
18-b	0.0	0.0	0.0
19-b	593.0	0.0	0.0
25-b	564.0	0.0	0.0
21	560.7	0.0	0.0
43	0.0	0.0	0.0
24	531.5	0.0	0.0
48	587.1	0.0	0.0
39	528.7	0.0	0.0
3	516.7	0.0	0.0

Table D.31: Cumulative Biogas Production for the Batch Tests (37 °C, 1 atm)

Time	Gas	Prod.	mL																	
hours	18	27	45	32	28	25	11	36	31	28-b	18-b	19-b	25-b	37	21	43	24	48	39	3
0.00	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
20.25	41.4	38.5	36.7	52.3	43.1	41.6	46.1	36.4	40.4	39.8	45.4	37.6	46.0	45.5	39.1	40.8	36.4	38.8	37.0	40.2
48.00	90.9	83.0	80.1	97.4	95.6	85.4	88.7	91.7	90.5	89.8	99.1	81.9	85.2	89.0	83.7	81.8	88.2	83.4	85.6	90.3
71.00	131.7	116.8	119.4	182.5	148.7	129.7	125.8	127.6	133.3	144.7	139.1	119.3	132.8	135.6	127.8	124.2	127.9	123.4	126.8	130.7
94.00	176.5	157.7	153.2	221.7	185.5	164.5	162.7	163.2	162.7	181.3	198.4	161.2	174.5	168.3	170.5	167.8	165.3	166.9	170.2	171.3
117.25	216.8	198.3	193.4	277.6	222.3	205.7	199.6	211.4	198.4	225.4	239.2	205.7	216.3	213.4	219.8	208.7	212.8	220.7	214.9	218.6
142.75	273.4	233.6	227.8	331.5	257.4	262.0	257.6	289.8	246.9	251.7	282.8	249.8	283.7	266.7	271.6	262.4	265.7	268.9	270.0	268.3
165.00	320.9	274.7	267.4	372.5	311.7	303.6	294.5	394.7	274.7	304.9	314.7	297.1	331.1	312.3	324.3	311.3	318.9	320.4	315.2	317.8
194.00	373.7	318.4	316.8	451.8	358.5	342.1	358.7	440.2	311.5	354.0	361.7	344.4	375.6	359.6	369.7	345.9	352.3	361.1	349.5	355.7
218.75	502.3	484.3	475.3	513.9	424.6	389.8	391.7	552.3	349.2	402.6	403.3	391.6	429.8	403.8	416.0	397.6	403.3	413.9	399.8	418.3
238.50	770.6	645.1	565.8	731.4	451.7	563.6	480.4	664.4	576.3	612.2	645.6	512.3	595.6	485.6	564.1	449.7	532.4	506.7	487.9	544.7
267.50	1136.7	882.6	651.5	952.7	477.6	741.2	573.3	771.3	792.4	748.5	788.9	591.6	704.3	553.0	712.3	589.0	687.9	644.5	613.0	701.2
283.75	1603.6	1035.9	749.7	1175.6	504.2	928.5	668.8	887.1	913.3	856.3	1086.4	703.7	992.2	657.1	876.0	684.6	855.0	823.5	745.9	798.6
309.00	2074.4	1404.3	937.3	1387.9	536.1	1104.3	765.2	1037.1	1121.2	978.4	1307.3	810.7	1223.5	765.8	1032.8	757.3	954.1	906.3	886.5	987.4
346.25	2729.6	1572.8	1025.6	1653.1	572.4	1283.6	897.4	1141.4	1342.4	1131.8	1948.5	884.4	1686.4	836.4	1298.5	989.1	1197.3	1105.8	1045.7	1222.8
373.00	3094.3	1821.6	1201.8	1866.5	612.5	1467.2	980.1	1256.7	1577.6	1245.1	2578.4	973.8	1891.5	944.9	1517.6	1134.0	1342.4	1298.7	1214.8	1445.6
390.75	3359.5	2173.4	1481.6	2271.4	647.6	1651.4	1072.0	1362.3	1674.9	1361.9	3011.8	1056.3	2124.7	1026.4	1682.7	1355.6	1494.2	1411.5	1387.6	1467.8
421.25	3655.9	2423.3	1568.5	2683.3	778.7	1948.1	1153.3	1459.0	1796.5	1473.2	3668.9	1138.5	2357.8	1100.7	1856.9	1590.8	1786.5	1675.9	1645.0	1804.6
439.00	3993.0	2774.7	1646.7	2967.9	809.8	2113.7	1224.5	1552.2	1984.6	1597.6	4243.2	1202.2	2479.6	1166.5	1997.8	1809.6	1942.7	1889.1	1876.3	1989.6
462.75	4307.1	3020.8	1789.9	3258.2	951.5	2394.6	1301.4	1748.8	2085.3	1714.3	4679.5	1287.6	2632.3	1247.3	2116.5	2034.5	2110.9	2087.4	2065.2	2043.9
512.75	4651.3	3161.4	1840.1	3813.8	1036.4	2771.2	1463.7	1943.5	2229.8	1839.7	5218.7	1511.8	2789.8	1494.6	2312.8	2348.6	2352.8	2316.7	2328.9	2320.7
539.00	4885.9	3294.8	1918.7	4004.6	1162.3	2937.9	1548.8	2039.6	2343.0	1954.0	5455.3	1633.2	2976.5	1586.8	2437.0	2586.5	2486.0	2472.8	2511.3	2563.8
564.75	5060.1	3336.2	2090.4	4197.3	1291.6	3302.6	1609.1	2136.2	2417.8	2071.7	5676.2	1695.7	3204.7	1639.5	2519.7	2635.4	2617.1	2563.4	2533.2	2581.7
582.00	5202.9	3452.1	2164.2	4486.5	1328.4	3474.3	1768.3	2241.4	2596.7	2206.4	5723.9	1811.3	3296.5	1782.7	2604.2	2712.6	2678.3	2635.4	2649.8	2708.8
606.25	5299.3	3566.8	2223.8	4772.4	1545.2	3742.2	1942.7	2403.7	2702.3	2372.6	5805.7	1962.8	3422.7	1834.5	2656.7	2783.8	2756.9	2721.3	2743.7	2758.6
657.00	5456.5	3676.9	2338.7	5048.2	1712.1	3981.1	2159.2	2629.1	2945.6	2481.9	5888.5	2084.3	3514.8	1978.7	2743.9	2875.6	2804.4	2845.9	2813.3	2800.4
745.25	5667.2	3745.7	2441.6	5135.1	1853.7	4164.0	2311.4	2734.6	3034.1	2533.7	5944.6	2156.7	3579.3	2064.5	2875.3	2946.9	3088.0	2976.6	2907.8	2999.4

Table D.32: Soluble Chemical Oxygen Demand of the Batch Tests Before and After Digestion

Bottle #	Initial Volume FAS, mL	Final Volume FAS, mL	Volume FAS Titrated, mL	Soluble COD mg O_2/L
Before Digestion				
11	3.25	11.50	8.25	1960.8
36	15.50	23.40	7.90	2280.0
25	8.00	9.85	1.85	6201.6
18	13.80	15.55	1.75	6384.0
28	0.10	4.30	4.20	1915.2
45	4.50	8.50	4.00	2280.0
27	11.85	13.70	1.85	6201.6
32	5.40	7.15	1.75	6384.0
31	12.90	18.70	5.80	4195.2
28-b	0.40	6.25	5.85	4149.6
37	10.00	19.20	9.20	1094.4
18-b	0.00	0.90	0.90	7843.2
19-b	0.15	5.90	5.75	4240.8
25-b	15.40	21.15	5.75	4240.8
21	20.85	26.55	5.70	4286.4
43	9.90	15.40	5.50	4468.8
24	11.10	16.70	5.60	4377.6
48	0.35	6.00	5.65	4332.0
39	6.20	11.70	5.50	4468.8
3	11.60	17.15	5.55	4423.2
After Digestion				
11	13.20	18.55	5.35	1282.3
36	2.60	8.05	5.45	1049.1
25	10.40	13.90	3.50	2880.0
18	0.00	3.65	3.65	2592.0
28	4.60	9.00	4.40	1152.0
45	0.00	4.30	4.30	1344.0
27	10.90	13.80	2.90	4032.0
32	14.25	17.05	2.80	4224.0
31	12.90	18.05	5.15	1748.6
28-b	0.00	4.70	4.70	2797.7
37	0.00	5.60	5.60	699.4
18-b	21.15	25.70	4.55	3147.4
19-b	17.05	22.00	4.95	2214.9
25-b	15.90	20.80	4.90	2331.4
21	5.60	10.70	5.10	1865.1
43	11.50	16.50	5.00	2098.3
24	3.65	8.70	5.05	1981.7
48	0.00	4.90	4.90	2331.4
39	9.15	14.25	5.10	1865.1
3	15.85	20.85	5.00	2098.3

Table D.33: Total Solids Concentration of the Batch Tests Before and After Digestion

	Before Digestion			*After Digestion*		
Bottle #	Mass Empty (g)	Mass After (g)	TS (mg/L)	Mass Empty (g)	Mass After (g)	TS (mg/L)
11	1.8126	1.9503	13190	1.8241	1.9303	10620
36	1.8415	1.9727	12540	1.6468	1.7461	9930
25	1.4806	1.7192	23860	1.7460	1.8974	15140
18	1.6116	1.8357	22410	1.5707	1.7299	15920
28	1.5993	1.7277	12840	1.5646	1.6751	11050
45	1.6164	1.7383	12190	1.7365	1.8404	10390
27	1.5571	1.7959	23880	1.6597	1.8619	20220
32	1.5720	1.7952	22320	1.6101	1.8023	19220
31	1.9847	2.1631	17260	2.0369	2.1699	12720
28-b	1.8636	2.0445	17510	1.8965	2.0528	15630
37	2.0438	2.1443	9470	1.6679	1.7475	7960
18-b	2.0457	2.3200	26850	1.8008	1.9825	18170
19-b	1.7955	1.9814	18010	1.6347	1.7780	14330
25-b	1.8490	2.0267	17190	1.6869	1.8287	14180
21	2.0097	2.1972	18170	1.5435	1.6792	13570
43	2.0241	2.2030	17310	1.8873	2.0242	13110
24	1.8769	2.0594	17670	1.8757	2.0124	13670
48	1.9949	2.1761	17540	2.0460	2.1805	12870
39	1.8097	1.9943	17880	1.6454	1.7814	13600
3	1.6961	1.8795	17760	1.4996	1.6340	13440

Table D.34: Volatile Solids Concentration of the Batch Tests Before and After Digestion

	Before Digestion			*After Digestion*		
Bottle #	Mass Before (g)	Mass After (g)	VS (mg/L)	Mass Before (g)	Mass After (g)	VS (mg/L)
11	1.9503	1.4628	7550	1.9303	1.8792	5110
36	1.9727	1.4828	7790	1.7461	1.6942	5190
25	1.7192	1.5727	14650	1.8974	1.8244	7300
18	1.8357	1.6920	14370	1.7299	1.6535	7640
28	1.7277	1.6559	7180	1.6751	1.6189	5620
45	1.7383	1.6554	8290	1.8404	1.7762	6420
27	1.7959	1.6495	14640	1.8619	1.7496	11230
32	1.7952	1.6520	14320	1.8023	1.6917	11060
31	2.1631	1.6379	11320	2.1699	1.6972	6070
28-b	2.0445	1.5178	11470	2.0528	1.9608	9200
37	2.1443	1.6763	5600	1.7475	1.7074	4010
18-b	2.3200	1.7260	18200	1.9825	1.8879	9460
19-b	1.9814	1.4581	11130	1.7780	1.7036	7440
25-b	2.0267	1.4971	11760	1.8287	1.7469	8180
21	2.1972	1.6729	11230	1.6792	1.6085	7070
43	2.2030	1.6784	11260	2.0242	1.5425	6970
24	2.0594	1.5397	10770	2.0124	1.9452	6720
48	2.1761	1.6508	11330	2.1805	1.6942	7430
39	1.9943	1.4727	10960	1.7814	1.7141	6730
3	1.8795	1.3562	11130	1.6340	1.5674	6660

Table D.35: Input for Least Squares Analysis of Batch Tests

Bottle #	Particle Size coded X_1	Total Solids coded X_2	Alk. Dose coded X_3	Ult. Gas Prod. (L)	Ult. Gas @ STP	Gas Prod. (L/g VS added)	Gas Prod. (L/g VS removed)	Methane Conc. (%)	% sCOD Removal	% TS Removal	% VS Removal
11	-1.000	-1.000	-1.000	2.311	2.049	0.417	1.292	56.88	34.60	19.48	32.32
36	1.175	-1.000	-1.000	2.736	2.425	0.479	1.435	58.62	53.98	20.81	33.38
25	-1.000	1.000	-1.000	4.164	3.691	0.388	0.773	57.67	53.56	36.55	50.17
18	1.175	1.000	-1.000	5.667	5.023	0.538	1.148	60.10	59.40	28.96	46.83
28	-1.000	-1.000	1.000	1.854	1.643	0.352	1.621	61.34	39.85	13.94	21.73
45	1.175	-1.000	1.000	2.442	2.165	0.402	1.781	63.22	41.05	14.77	22.56
27	-1.000	1.000	1.000	3.745	3.320	0.349	1.498	57.50	34.98	15.33	23.29
32	1.175	1.000	1.000	5.135	4.552	0.489	2.148	60.90	33.83	13.89	22.77
31	0.000	0.000	-1.682	3.034	2.689	0.366	0.788	58.78	58.32	26.30	46.38
28-b	0.000	0.000	1.682	2.534	2.246	0.301	1.522	58.70	32.58	10.74	19.79
37	0.000	-1.682	0.000	2.064	1.830	0.503	1.770	59.49	36.09	15.95	28.39
18-b	0.000	1.682	0.000	5.945	5.270	0.445	0.928	53.82	59.87	32.33	48.02
19-b	-1.575	0.000	0.000	2.157	1.912	0.264	0.797	56.40	47.77	20.43	33.15
25-b	2.000	0.000	0.000	3.579	3.172	0.415	1.363	60.18	45.02	17.51	30.44
21	0.000	0.000	0.000	2.875	2.548	0.349	0.942	59.45	56.49	25.32	37.04
43	0.000	0.000	0.000	2.947	2.612	0.357	0.937	58.14	53.05	24.26	38.10
24	0.000	0.000	0.000	3.088	2.737	0.391	1.040	56.63	54.73	22.64	37.60
48	0.000	0.000	0.000	2.977	2.639	0.358	1.041	58.99	46.18	26.62	34.42
39	0.000	0.000	0.000	2.908	2.578	0.362	0.938	59.51	58.26	23.94	38.59
3	0.000	0.000	0.000	2.999	2.658	0.367	0.915	61.69	52.56	24.32	40.16

D.7: Study of Alkaline Pretreatment

Table D.36: Design Points For Batch Test Study

Bottle #	Part. Size X_1, mm	Total Solids X_2, %	Alk. Dose X_3, meq/L	Coded X1 *	Coded X_2	Coded X_3
7	2	10	50	-1	-1	-1
33	6.35	10	50	1.175	-1	-1
26	2	20	50	-1	1	-1
42	6.35	20	50	1.175	1	-1
49	2	10	150	-1	-1	1
20	6.35	10	150	1.175	-1	1
34	2	20	150	-1	1	1
46	6.35	20	150	1.175	1	1
17	4	15	16	0	0	-1.682
22	4	15	184	0	0	1.682
29	4	6.59	100	0	-1.682	0
30	4	23.41	100	0	1.682	0
9	0.85	15	100	-1.575	0	0
32	8	15	100	2	0	0
12	4	15	100	0	0	0
41	4	15	100	0	0	0
44	4	15	100	0	0	0
27	4	15	100	0	0	0
47	4	15	100	0	0	0
35	4	15	100	0	0	0

* Could not use standard coding due to physical constraints

Table D.37: Biogas Methane Concentrations and pH for Batch Tests

Bottle #	Methane (%)	Final pH
7	58.62	7.8
33	56.88	8.3
26	57.67	8.15
42	60.1	8.1
49	54.51	7.85
20	63.22	7.65
34	57.5	7.75
46	60.9	7.85
17	53.73	7.55
22	60.18	7.65
29	59.82	8.3
30	61.13	7.8
9	58.7	7.55
32	59.49	7.45
12	56.44	7.45
41	58.99	7.4
44	59.51	7.45
27	57.32	7.5
47	58.14	7.55
35	62.41	7.45

Table D.38: Final Volatile Fatty Acid Content of the Batch Reactors

Bottle #	Concentration (mg/L)		
	Acetic Acid	Butyric Acid	Propionic Acid
7	0.0	0.0	0.0
33	728.0	0.0	0.0
26	0.0	0.0	0.0
42	0.0	0.0	0.0
49	0.0	0.0	0.0
20	0.0	0.0	0.0
34	0.0	0.0	0.0
46	0.0	0.0	0.0
17	570.3	0.0	0.0
22	0.0	0.0	0.0
29	0.0	0.0	0.0
30	0.0	0.0	0.0
9	693.0	0.0	0.0
32	764.0	0.0	0.0
12	0.0	0.0	0.0
41	760.7	0.0	0.0
44	0.0	0.0	0.0
27	0.0	0.0	0.0
47	0.0	0.0	0.0
35	0.0	0.0	0.0

Table D.39: Cumulative Biogas Production for the Alkaline Pretreated Batch Tests (37 °C, 1 atm)

Time	42	34	20	46	49	26	7	33	17	22	30	9	32	29	12	41	44	27	47	35
0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
20.25	54.6	59.7	49.8	51.7	56.6	54.9	53.6	47.9	51.4	46.7	51.3	51.1	49.8	50.5	48.6	51.1	53.5	47.9	52.4	50.6
48.00	125.9	138.3	113.7	124.5	131.8	127.5	127.3	116.7	94.5	89.3	90.2	93.5	89.4	91.8	89.7	88.4	93.6	91.5	88.3	90.7
71.00	186.3	195.6	162.3	167.3	187.9	181.6	169.9	158.4	132	125.7	131.7	128.5	121.9	132.4	129.5	133.4	137.6	128.4	125.1	130.7
94.00	234.5	253.8	204.8	213.9	247.5	237.9	211.2	190.3	176.3	168.4	170.9	172.4	167.5	177.9	176.4	181.9	185.6	172.7	183.2	180.5
117.25	291.1	342.5	254.3	262.4	292.3	289.2	265.7	213.8	209.7	211.2	213.4	217.2	208.6	213.7	223.9	231.3	227	216.5	227.9	241.8
142.75	365.8	477.9	302.2	312.3	356.1	423.7	342.6	256.9	253.6	245.7	242.5	251.6	251.3	257.6	278.4	265.9	261.4	278.4	272.3	269.1
165.00	423.4	597.6	346.1	384.6	413	582.4	421.5	298.5	308.4	292.7	286.4	283.5	288.2	289.3	311.2	305.7	297.8	308.2	312.8	298.7
194.00	477.9	743.2	398.7	431.7	465.8	746.6	672.9	321.7	356.8	349.1	327.8	331.7	325.7	328.3	354.8	347.6	341.4	357.6	362.8	341.5
218.75	544.2	968.4	443.5	479.8	623.7	975.1	803.5	343.2	396.7	436.1	369.7	385.6	372.4	369.7	541.3	523.1	526.5	503.8	476.9	562.9
238.50	696.5	1179.5	497.8	531.9	719.8	1146.5	967.7	375.8	514.9	583.2	432.9	424.8	433.8	412.4	687.8	762.8	661.1	863.2	611.5	713.8
267.50	895.8	1413.7	554.4	590	903.7	1436.7	1132.8	403	756.3	675.4	567.2	473.7	552.5	527.8	896.3	965	874.2	1054.7	874.2	911.6
283.75	1073.1	1764.9	612.9	876.7	1084.6	1675.9	1256.6	436.7	1012.6	818.6	694.5	515.5	711.2	574.9	1073	1109.4	1048	1176.5	1023.9	1075.1
309.00	1306.7	2142.9	723.7	1203.8	1324.6	1963.3	1392.4	487.1	1347.8	1034.1	863.7	576.4	923.9	628.5	1392.5	1411.2	1376.5	1413.8	1398.5	1346.7
346.25	1617	2551.6	911.4	1978.3	1621.4	2346.8	1534.5	546.7	1762.7	1364.8	1346.8	766.9	1087.1	743.2	1755.9	1783.2	1712.9	1788.4	1756.8	1688.9
373.00	2083.8	2835.1	1099.3	3284.7	1972.4	2681.9	1661.4	602.6	2034.1	1631.3	2344.6	1087.4	1364.6	953.6	2057.7	2064.9	2034.8	2024.3	2089.1	1983.2
390.75	2592.6	3176.8	1433.6	4368	2267.5	2957.8	1804.5	787.1	2218.3	1967.2	3017.6	1323.3	1776.5	1227.3	2188.8	2198.3	2176.7	2297.1	2205.7	2136.5
421.25	3442.7	3843.5	1774.4	5719.1	2553.8	3446.2	1987.2	1312.8	2588.9	2476.8	4458.9	1745.8	2387.3	1613.7	2699.4	2737.6	2685.2	2832.6	2712.4	2627.6
439.00	4366.3	4176.7	2123.2	6428.7	2697.2	3703.7	2113	1607.3	2717.8	2972.5	5263.1	1937.6	2911.6	1908.5	2965.9	2992.8	2917.6	3016.2	2954.8	2845.9
462.75	5549	4537.4	2564.3	7033.4	2875.6	3934.9	2311.6	2017.9	3066.9	3426.5	6255.8	2182.2	3323.9	2110.4	3247.6	3268.9	3211.4	3347.5	3269	3176.4
512.75	6903.5	5123.9	3072.7	7546.8	3116.7	4346.7	2586.7	2654.7	3484.1	3943.2	7187.5	2463.9	3877.5	2374.6	3743.4	3785.1	3727.6	3978.6	3745.6	3705.1
539.00	7107.8	5267.8	3184.4	7685.9	3177.6	4552.8	2717.8	2875.4	3675.2	4186.4	7330.9	2511.3	4144.8	2417.9	3965.9	3992.6	3946.4	4113.4	3987.6	3908.2
564.75	7247.6	5334.3	3297.9	7741.8	3185.4	4699.4	2874.4	3026.3	3814.5	4297	7463.6	2588.4	4231.1	2487.1	4092.1	4116.7	4087.3	4245.8	4053.1	4017.9
582.00	7321.8	5389.1	3425.6	7776.4	3267.7	4847.4	2961.5	3067.8	3928.6	4404.5	7551.3	2647.5	4376.5	2557.8	4187.6	4205.9	4193.2	4387.6	4138.4	4153.7
606.25	7483.1	5462.8	3574.3	7804.3	3342.8	4956.3	3046.6	3185.4	4001.4	4512.1	7623.5	2693.2	4503.7	2604.5	4234.8	4346.2	4281.9	4494.7	4245.9	4281.4
657.00	7511.2	5574.9	3656.8	7877.6	3415.5	5075.4	3178.9	3286.4	4085.7	4654.3	7754.3	2787.4	4635.5	2688.9	4303.1	4426.7	4364.8	4573.1	4317.5	4366.2
745.25	7619.3	5627.7	3722.4	7946	3493.1	5145.6	3237.2	3327.9	4154.3	4782.5	7819.1	2840.3	4717	2745.8	4376.4	4639.6	4396.7	4660.2	4372.6	4409.8

Table D.40: Soluble Chemical Oxygen Demand of the Batch Tests Before and After Digestion

Bottle #	Initial Volume (mL)	Final Volume (mL)	Volume Titrated (mL)	COD (mg O_2/L)
Before Digestion				
7	0.00	4.25	4.25	1732.8
33	4.25	8.25	4.00	2188.8
26	8.25	12.10	3.85	5973.6
42	12.10	13.95	1.85	6110.4
49	13.95	18.25	4.30	1641.6
20	18.25	22.20	3.95	2280.0
34	0.10	3.90	3.80	6019.2
46	3.90	7.60	3.70	6110.4
17	7.60	10.60	3.00	4012.8
22	10.60	16.70	6.10	3921.6
29	16.70	21.40	4.70	912.0
30	0.00	1.10	1.10	7478.4
9	1.10	4.15	3.05	3921.6
32	4.15	7.25	3.10	3830.4
12	7.25	12.90	5.65	4332.0
41	12.90	15.85	2.95	4104.0
44	15.85	18.55	2.70	4560.0
27	18.55	21.50	2.95	4104.0
47	0.90	3.80	2.90	4195.2
35	3.80	6.65	2.85	4286.4
After Digestion				
7	0.00	5.45	5.45	1049.1
33	5.45	11.00	5.55	816.0
26	11.00	15.95	4.95	2214.9
42	15.95	21.05	5.10	1865.1
49	0.00	5.55	5.55	816.0
20	5.55	10.95	5.40	1165.7
34	10.95	15.30	4.35	3613.7
46	15.30	19.65	4.35	3613.7
17	0.20	5.60	5.40	1165.7
22	5.60	10.45	4.85	2448.0
29	10.45	16.15	5.70	466.3
30	16.15	21.20	5.05	1981.7
9	0.00	5.20	5.20	1632.0
32	5.20	10.35	5.15	1748.6
12	10.35	15.60	5.25	1515.4
41	15.60	20.85	5.25	1515.4
44	0.60	5.80	5.20	1632.0
27	5.80	10.90	5.10	1865.1
47	10.90	16.25	5.35	1282.3
35	16.25	21.45	5.20	1632.0

Table D.41: Total Solids Concentration of the Batch Tests Before and After Digestion

Bottle #	*Before Digestion* Mass Empty (g)	Mass After (g)	TS (mg/L)	*After Digestion* Mass Empty (g)	Mass After (g)	TS (mg/L)
7	1.8734	1.9882	11480	1.8774	1.9343	5690
33	1.8456	1.9575	11190	1.8932	1.9512	5800
26	1.6734	1.8937	22030	1.9012	1.9859	8470
42	1.8934	2.1039	21050	1.8743	1.9534	7910
49	1.8684	1.9864	11800	1.9138	1.9873	7350
20	1.7293	1.8397	11040	1.8127	1.8826	6990
34	1.9833	2.1904	20710	1.8352	1.9623	12710
46	1.8938	2.0951	20130	1.8293	1.9574	12810
17	1.9012	2.0678	16660	1.8298	1.9009	7110
22	1.8659	2.0311	16520	1.8923	2.0035	11120
29	1.8632	1.9623	9910	1.7974	1.8557	5830
30	1.9647	2.2059	24120	1.7982	1.8856	8740
9	1.9643	2.1328	16850	1.8138	1.9033	8950
32	1.9648	2.1402	17540	1.8829	1.9661	8320
12	1.9283	2.1061	17780	1.8365	1.9232	8670
41	1.8628	2.0410	17820	1.8826	1.9628	8020
44	1.8212	1.9923	17110	1.8372	1.9138	7660
27	1.8324	2.0097	17730	1.8643	1.9484	8410
47	1.8026	1.9788	17620	1.8816	1.9692	8760
35	1.8356	2.0123	17670	1.8635	1.9457	8220

Table D.42: Volatile Solids Concentration of the Batch Tests Before and After Digestion

Bottle #	*Initial* Mass Before (g)	Mass After (g)	VS (mg/L)	*Final* Mass Before (g)	Mass After (g)	VS (mg/L)
7	1.9882	1.9138	7440	1.9343	1.9251	920
33	1.9575	1.8789	7860	1.9512	1.9387	1250
26	1.8937	1.7322	16150	1.9859	1.9760	990
42	2.1039	1.9357	16820	1.9534	1.9405	1290
49	1.9864	1.9143	7210	1.9873	1.9608	2650
20	1.8397	1.7559	8380	1.8826	1.8504	3220
34	2.1904	2.0313	15910	1.9623	1.9097	5260
46	2.0951	1.9405	15460	1.9574	1.9002	5720
17	2.0678	1.9382	12960	1.9009	1.8863	1460
22	2.0311	1.9027	12840	2.0035	1.9486	5490
29	1.9623	1.8973	6500	1.8557	1.8385	1720
30	2.2059	2.0019	20400	1.8856	1.8704	1520
9	2.1328	2.0026	13020	1.9033	1.8786	2470
32	2.1402	2.0075	13270	1.9661	1.9468	1930
12	2.1061	1.9773	12880	1.9232	1.9048	1840
41	2.0410	1.9128	12820	1.9628	1.9451	1770
44	1.9923	1.8621	13020	1.9138	1.8963	1750
27	2.0097	1.8782	13150	1.9484	1.9300	1840
47	1.9788	1.8457	13310	1.9692	1.9504	1880
35	2.0123	1.8816	13070	1.9457	1.9278	1790

Table D.43: Input for Least Squares Analysis of Batch Tests

Bottle #	Part. Size coded X_1	Tot. Solids coded X_2	Alk. Dose coded X_3	Ult. Gas Prod. (L)	Ult. Gas @ STP	Gas Prod. (L/g VS added)	Gas Prod. (L/g VS removed)	Max. CH_4 Conc. (%)	% COD Removal	%TS Removal	%VS Removal
7	-1	-1	-1	3.237	2.869	0.593	0.677	58.62	39.45	50.43	87.63
33	1.175	-1	-1	3.328	2.950	0.577	0.687	56.88	62.72	48.1	84.09
26	-1	1	-1	5.146	4.562	0.435	0.463	57.67	62.92	61.55	93.87
42	1.175	1	-1	7.619	6.754	0.618	0.669	60.10	69.48	62.42	92.33
49	-1	-1	1	3.493	3.096	0.661	1.045	54.51	50.29	37.71	63.245
20	1.175	-1	1	3.722	3.299	0.606	0.984	63.22	48.87	36.68	61.57
34	-1	1	1	5.628	4.989	0.482	0.721	57.50	39.96	38.62	66.93
46	1.175	1	1	7.946	7.043	0.701	1.113	60.90	40.86	36.36	63.00
17	0	0	-1.682	4.154	3.682	0.437	0.493	53.73	70.95	57.32	88.73
22	0	0	1.682	4.783	4.240	0.508	0.887	60.18	37.58	32.68	57.24
29	0	-1.682	0	2.746	2.434	0.576	0.783	59.82	48.87	41.17	73.53
30	0	1.682	0	7.819	6.931	0.523	0.565	61.13	73.50	63.76	92.54
9	-1.575	0	0	2.84	2.517	0.297	0.367	58.70	58.38	46.88	81.02
32	2	0	0	4.717	4.181	0.485	0.567	59.49	54.35	52.56	85.45
12	0	0	0	4.376	3.879	0.463	0.541	56.44	65.02	51.23	85.71
41	0	0	0	4.639	4.112	0.493	0.573	58.99	63.07	54.99	86.19
44	0	0	0	4.396	3.897	0.460	0.532	59.51	64.21	55.23	86.55
27	0	0	0	4.66	4.131	0.483	0.562	57.32	54.55	52.56	86.00
47	0	0	0	4.373	3.876	0.448	0.522	58.14	69.43	50.28	85.87
35	0	0	0	4.409	3.908	0.460	0.533	62.41	61.93	53.48	86.30

APPENDIX E:
Control Model Results

This appendix contains all of the empirical modeling data and results for the control.

E.1 Cumulative Gas Production at STP

a) Initial Model

Table E.1: Parameter Estimates for Initial Cumulative Gas Production Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	4.0855	-0.3651	1.7714	-0.3092	0.2504	-0.6257
Std.Err.	0.1406	0.0899	0.1003	0.0744	0.1137	0.1300
t(8)	29.0677	-4.0619	17.6642	-4.1534	2.2023	-4.8135
p-level	0.0000	0.0066	0.0000	0.0060	0.0699	0.0030
Variance	explained	98.484%		R	0.9924	

Table E.2: ANOVA Table for Initial Cumulative Gas Production Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{0.01,v1,v2}$	$F_{0.05,v1,v2}$
Regression	5	1836.10	367.22	4579.77	8.75	4.39
Residual	6	0.48	0.08			
(Lack of Fit)	3	0.46	0.15	24.87	29.46	9.28
(Pure Error)	3	0.02	0.01			
Total	11					

Table E.3: Correlation Matrix of Parameter Estimates for Initial Cumulative Gas Production Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
β_0	1.0000	0.1636	0.0000	-0.6235	-0.6662	0.0000
β_1	0.1636	1.0000	0.0000	-0.3363	-0.0705	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	-0.0567
β_{11}	-0.6235	-0.3363	0.0000	1.0000	0.2618	0.0000
β_{22}	-0.6662	-0.0705	0.0000	0.2618	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	0.0000	1.0000

Table E.4: Pure Error Variance for Initial Cumulative Gas Production Model

For replicate set	
Mean	4.0685
sum of residuals squared	0.0181
degrees of freedom	3
pure error variance	0.0060

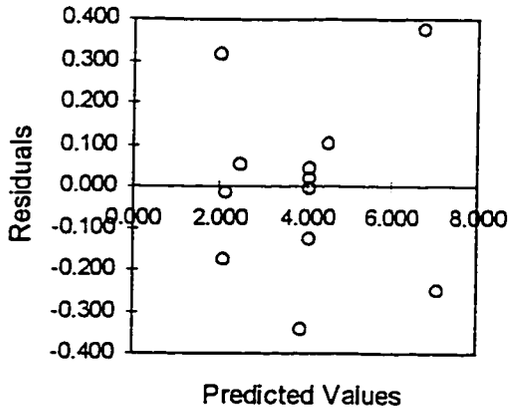

Figure E.1: Residuals Versus Predicted Values for Initial Cumulative Gas Production Model

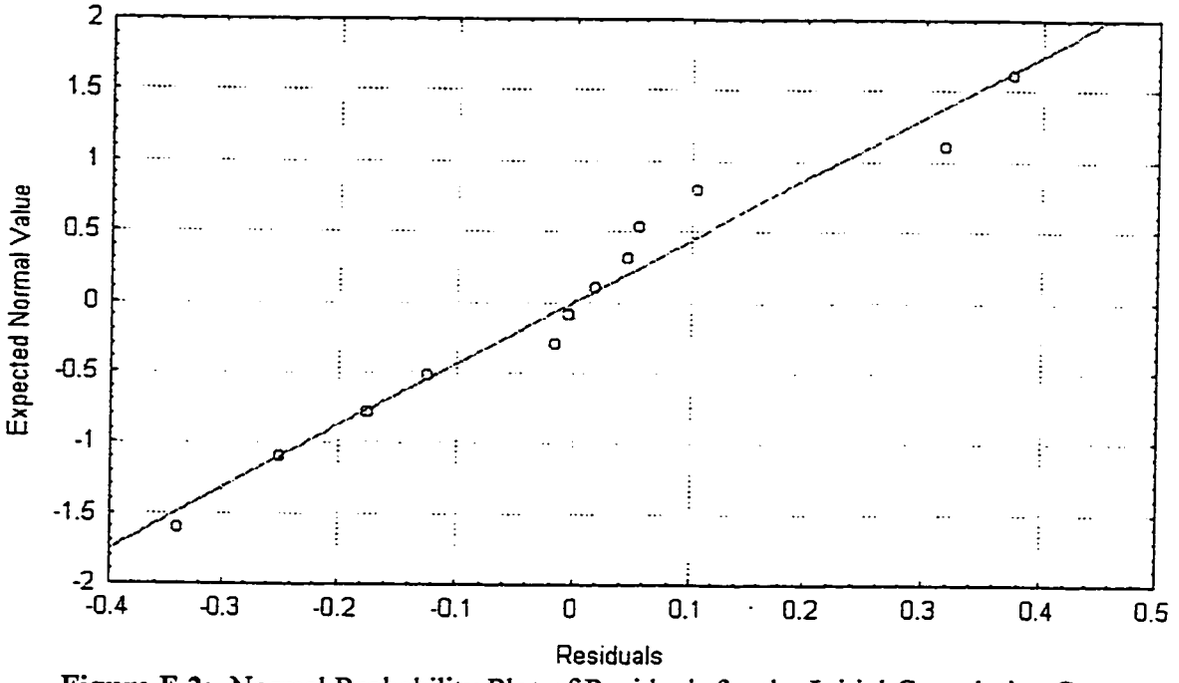

Figure E.2: Normal Probability Plot of Residuals for the Initial Cumulative Gas Production Model

b) Final Model

Table E.5: Parameter Estimates for Final Cumulative Gas Production Model

	β_0	β_1	β_2	β_{11}	β_{12}
Estimate	4.2917	-0.3512	1.7714	-0.3521	-0.6257
Std.Err.	0.1305	0.1116	0.1249	0.0894	0.1618
t(8)	32.8851	-3.1457	14.1882	-3.9366	-3.8663
p-level	0.0000	0.0162	0.0000	0.0056	0.0062
Variance	explained	97.259%		R	0.9862

Table E.6: ANOVA Table for Final Cumulative Gas Production Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{.01,v1,v2}$	$F_{.05,v1,v2}$
Regression	4	1302.57	325.64	2620.14	7.85	4.12
Residual	7	0.87	0.12			
(Lack of Fit)	4	0.65	0.16	2.27	28.71	9.12
(Pure Error)	3	0.22	0.07			
Total	11					

Table E.7: Correlation Matrix of Parameter Estimates for Final Cumulative Gas Production Model

	β_0	β_1	β_2	β_{11}	β_{12}
β_0	1.0000	0.1567	0.0000	-0.6238	0.0000
β_1	0.1567	1.0000	0.0000	-0.3301	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	-0.0567
β_{11}	-0.6238	-0.3301	0.0000	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	1.0000

Table E.8: Pure Error Variance for Final Cumulative Gas Production Model

For replicate set	
Mean	*4.0065*
sum of residuals squared	0.216211
degrees of freedom	3
pure error variance	0.0721

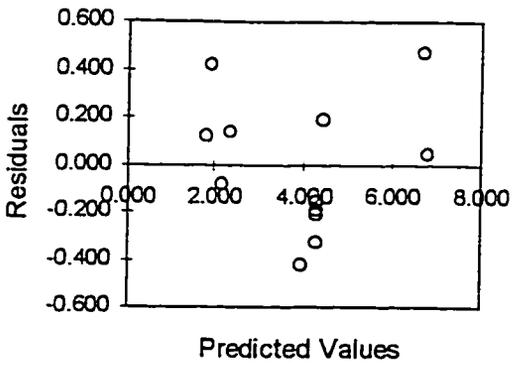

Figure E.3: Residuals Versus Predicted Values for Final Cumulative Gas Production Model

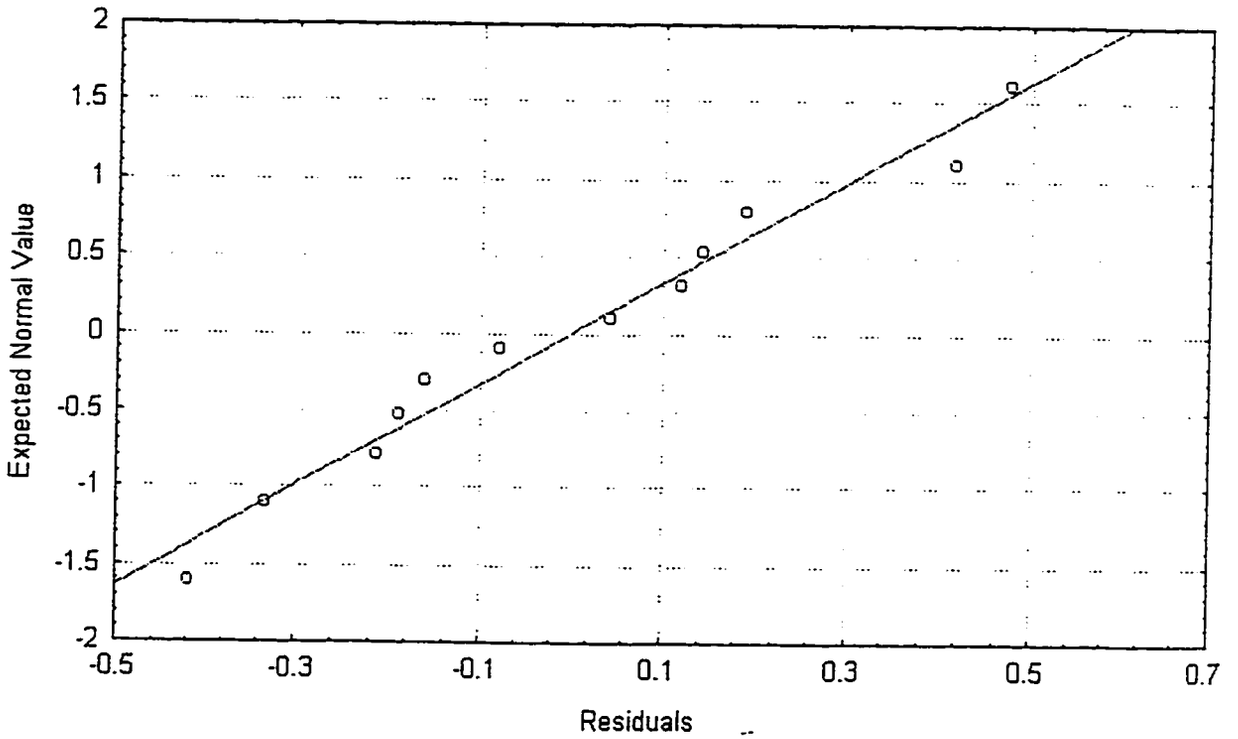

Figure E.4: Normal Probability for Plot of Residuals for the Final Cumulative Gas Production Model

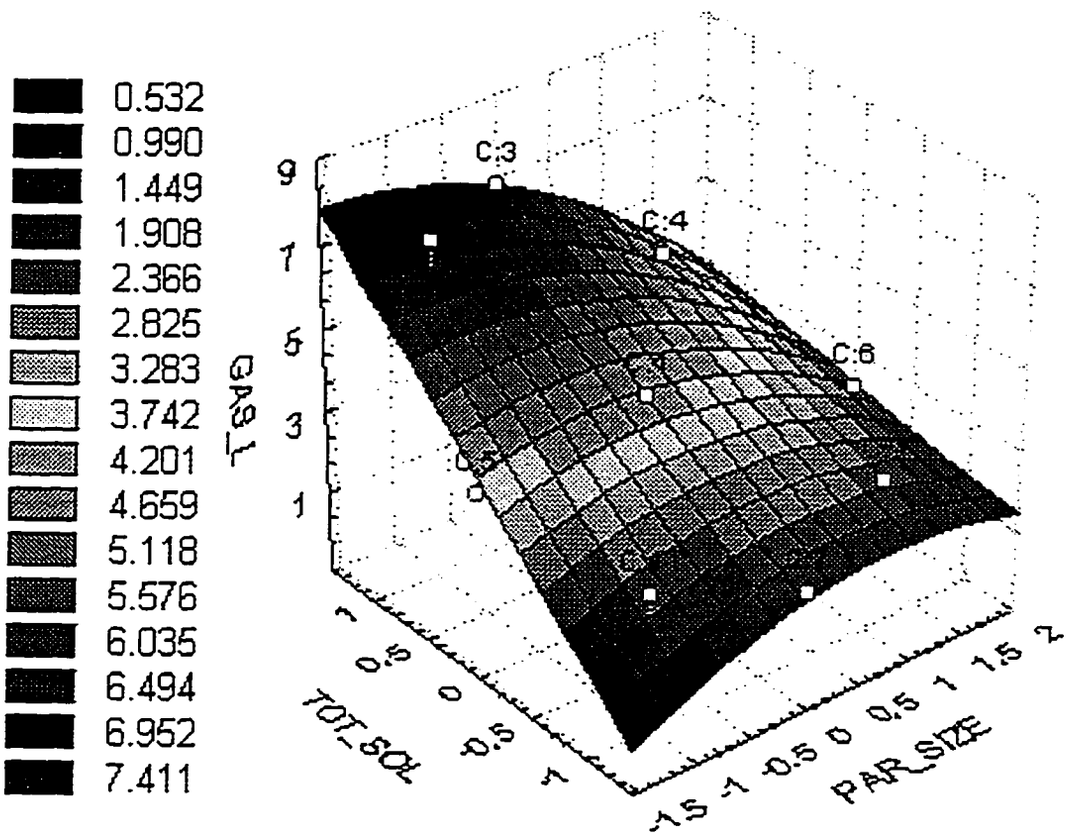

Figure E.5: Surface Plot of Final Cumulative Gas Production Model

E.2 Cumulative Gas Production at STP with Respect to Volatile Solids Addition

a) Initial Model

Table E.9: Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	0.5561	-0.0455	0.0689	-0.0378	0.0223	-0.0622
Std.Err.	0.0198	0.0126	0.0141	0.0105	0.0160	0.0183
t(8)	28.1551	-3.5979	4.8888	-3.6106	1.3983	-3.4043
p-level	0.0000	0.0114	0.0027	0.0112	0.2115	0.0144
Variance	explained	92.961%		R	0.9642	

Table E.10: ANOVA Table for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	5	8.94	1.79	1128.98	8.75	4.39
Residual	6	0.01	0.00			
(Lack of Fit)	3	0.01	0.00	123.55	29.46	9.28
(Pure Error)	3	0.00	0.00			
Total	11					

Table E.11: Correlation Matrix of Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
β_0	1.0000	0.1636	0.0000	-0.6235	-0.6662	0.0000
β_1	0.1636	1.0000	0.0000	-0.3363	-0.0705	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	-0.0567
β_{11}	-0.6235	-0.3363	0.0000	1.0000	0.2618	0.0000
β_{22}	-0.6662	-0.0705	0.0000	0.2618	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	0.0000	1.0000

Table E.12: Calculation for Pure Error Variance for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

For replicate set	
Mean	*0.5527*
sum of residuals squared	7.63E-05
degrees of freedom	3
pure error variance	2.54E-05

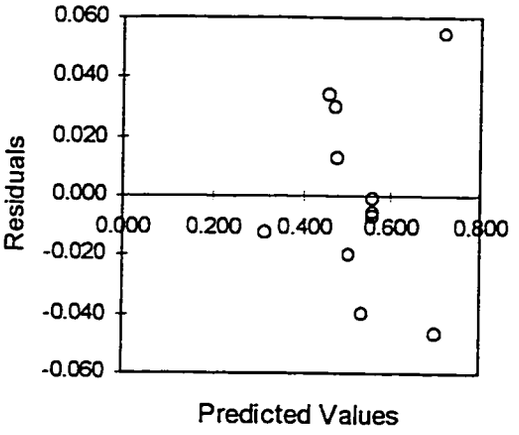

Figure E.6: Residuals versus Predicted Values for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

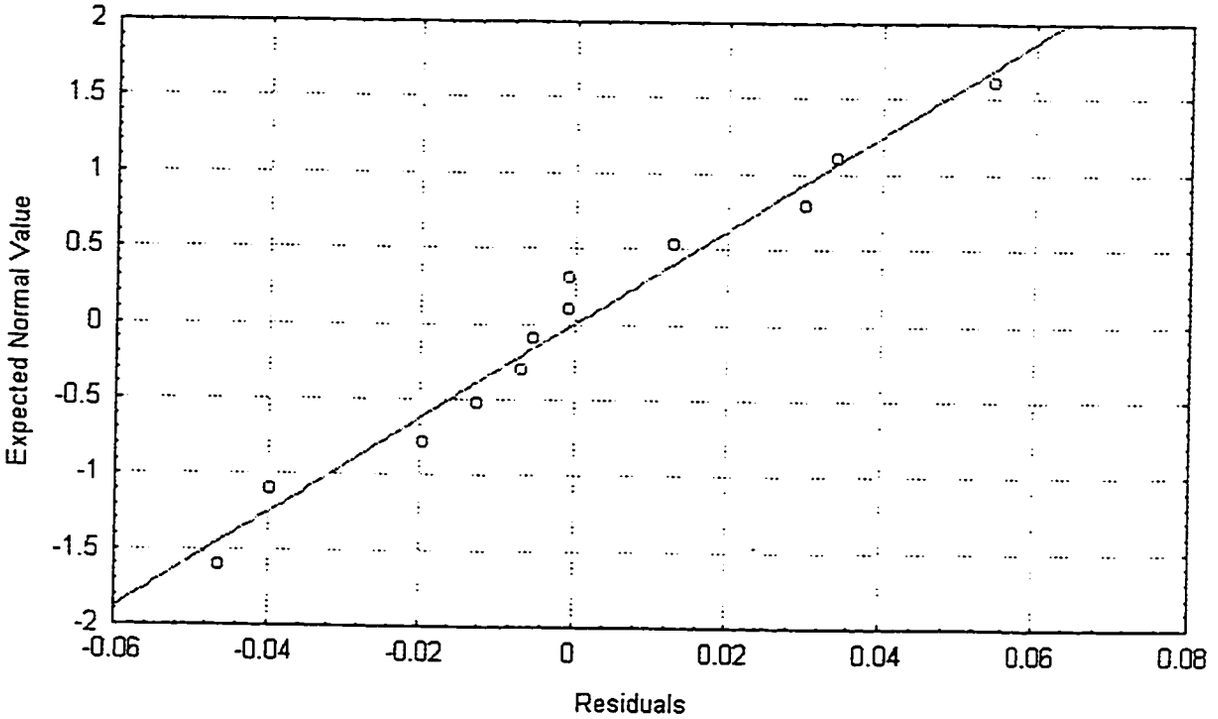

Figure E.7: Normal Probability for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

b) Final Model

Table E.13: Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_2	β_{11}	β_{12}
Estimate	0.5745	-0.0442	0.0689	-0.0416	-0.0622
Std.Err.	0.0157	0.0134	0.0150	0.0108	0.0195
t(8)	36.5835	-3.2906	4.5859	-3.8653	-3.1934
p-level	0.0000	0.0133	0.0025	0.0062	0.0152
Variance	explained	90.668%		R	0.9522

Table E.14: ANOVA Table for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	4	7.10	1.78	986.74	7.85	4.12
Residual	7	0.01	0.00			
(Lack of Fit)	4	0.01	0.00	4.14	28.71	9.12
(Pure Error)	3	0.00	0.00			
Total	11					

Table E.15: Correlation Matrix of Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_2	β_{11}	β_{12}
β_0	1.0000	0.1567	0.0000	-0.6238	0.0000
β_1	0.1567	1.0000	0.0000	-0.3301	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	-0.0567
β_{11}	-0.6238	-0.3301	0.0000	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	1.0000

Table E.16: Pure Error Variance for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

For replicate set	
Mean	*0.5527*
sum of residuals squared	0.001932
degrees of freedom	3
pure error variance	0.0006

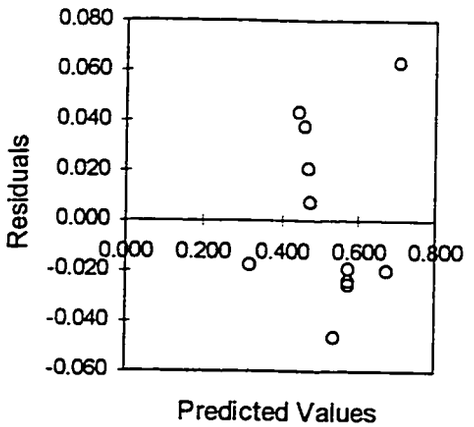

Figure E.8: Residuals Versus Predicted Values for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

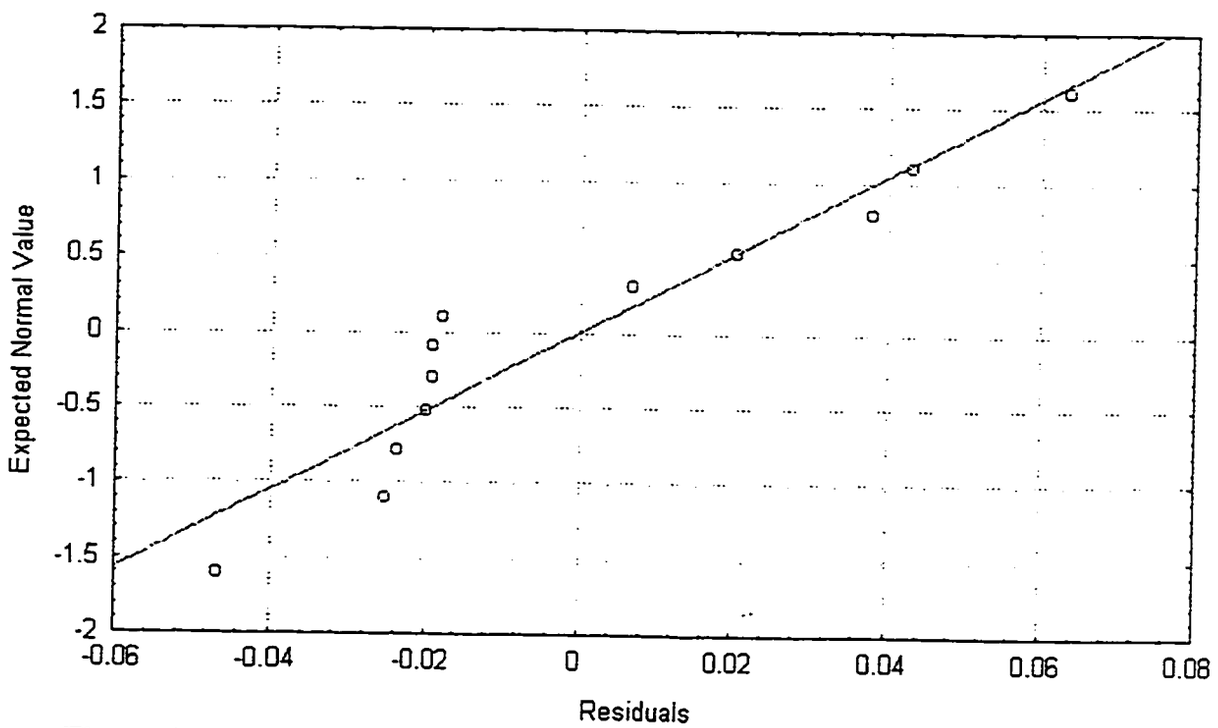

Figure E.9: Normal Probability Plot of the Residuals for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

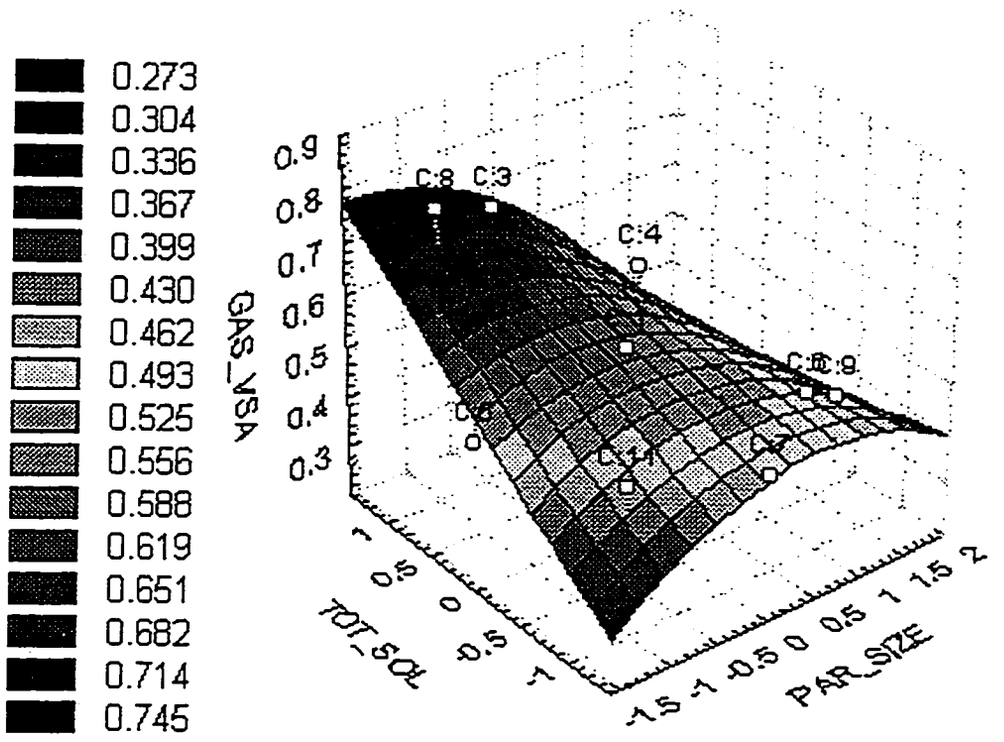

Figure E.10: Surface Plot for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

E.3 Cumulative Gas Production at STP with Respect to Volatile Solids Removal

a) Initial Model

Table E.17: Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	1.0686	-0.0672	-0.1634	-0.0176	0.0682	-0.0188
Std.Err.	0.0208	0.0133	0.0148	0.0110	0.0168	0.0192
t(8)	51.4924	-5.0647	-11.0334	-1.6054	4.0648	-0.9781
p-level	0.0000	0.0023	0.0000	0.1595	0.0066	0.3658
Variance	explained	96.859%		R	0.9842	

Table E.18: ANOVA Table for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	5	-45.84	-9.17	-5244.75	8.75	4.39
Residual	6	0.01	0.00			
(Lack of Fit)	3	0.01	0.00	1.04	29.46	9.28
(Pure Error)	3	0.01	0.00			
Total	11					

Table E.19: Correlation Matrix of Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
β_0	1.0000	0.1636	0.0000	-0.6235	-0.6662	0.0000
β_1	0.1636	1.0000	0.0000	-0.3363	-0.0705	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	-0.0567
β_{11}	-0.6235	-0.3363	0.0000	1.0000	0.2618	0.0000
β_{22}	-0.6662	-0.0705	0.0000	0.2618	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	0.0000	1.0000

Table E.20: Pure Error Variance for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

For replicate set	
Mean	*1.0664*
sum of residuals squared	0.005148
degrees of freedom	3
pure error variance	0.0017

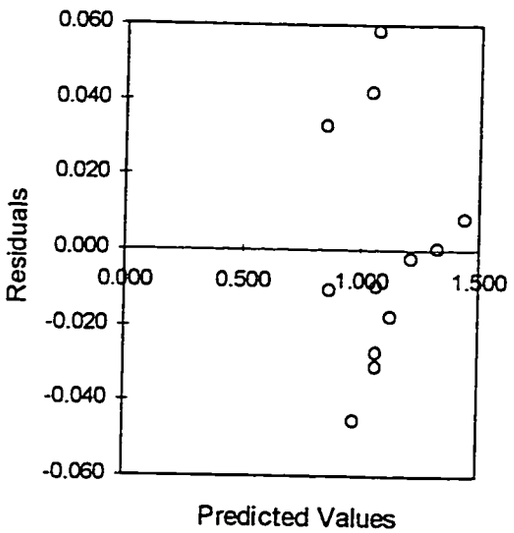

Figure E.11: Residual versus Predicted Values for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

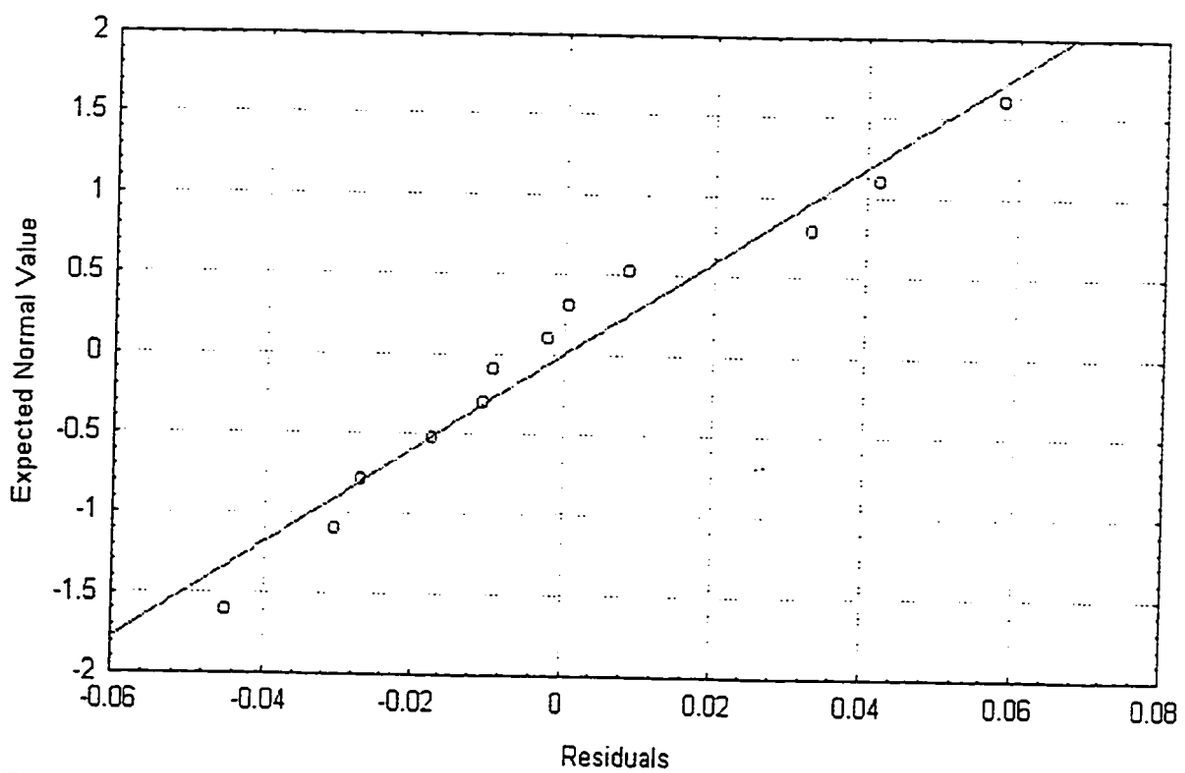

Figure E.12: Normal Probability Plot of Residuals for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

b) Final Model

Table E.21: Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_{22}
Estimate	1.0478	-0.0744	-0.1642	0.0753
Std.Err.	0.0177	0.0136	0.0161	0.0177
t(8)	59.1566	-5.4514	-10.1740	4.2571
p-level	0.0000	0.0006	0.0000	0.0028
Variance	explained	95.008%	R	0.9747

Table E.22: ANOVA Table for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	3	-48.96	-16.32	-7834.22	7.59	4.07
Residual	8	0.02	0.00			
(Lack of Fit)	5	0.01	0.00	0.94	28.24	9.01
(Pure Error)	3	0.01	0.00			
Total	11					

Table E.23: Correlation Matrix of Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_{22}
β_0	1.0000	-0.0626	0.0000	-0.6665
β_1	-0.0626	1.0000	0.0000	0.0193
β_2	0.0000	0.0000	1.0000	0.0000
β_{22}	-0.6665	0.0193	0.0000	1.0000

Table E.24: Pure Error Variance for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

For replicate set	
Mean	*1.0664*
sum of residuals squared	0.006509
degrees of freedom	3
pure error variance	0.0017

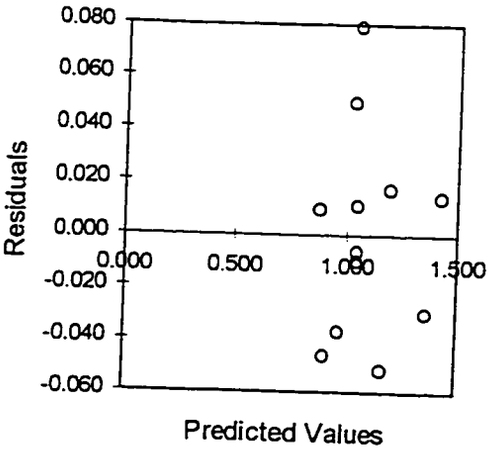

Figure E.13: Residuals Versus Predicted Values for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

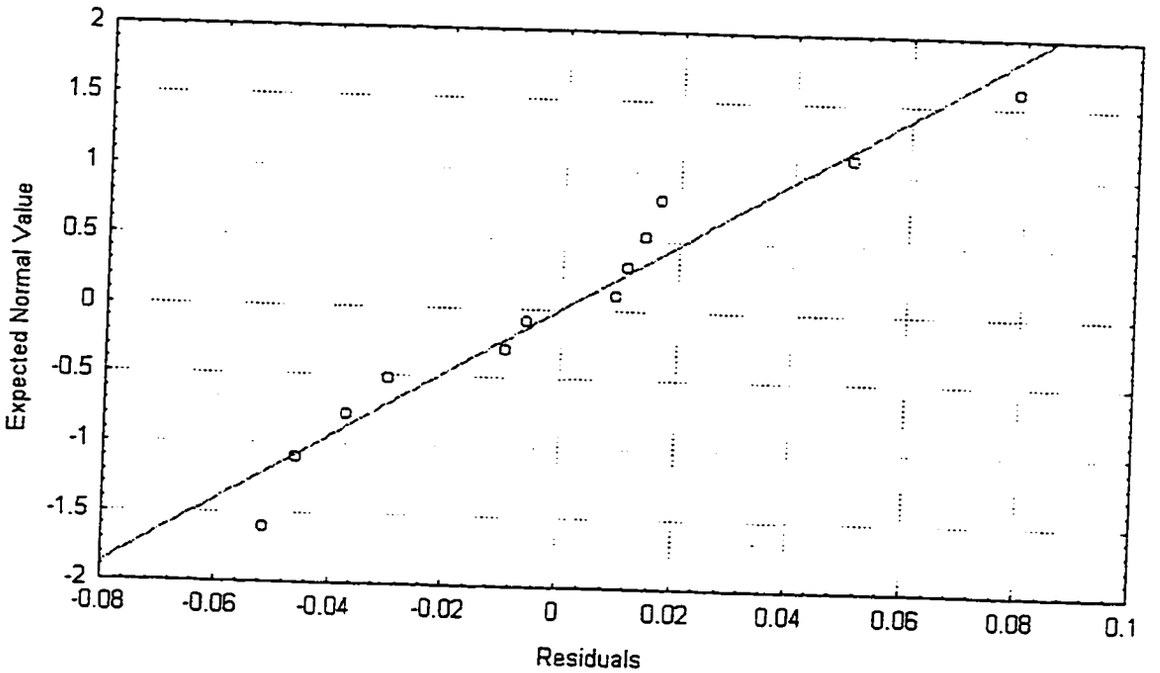

Figure E.14: Normal Probability Plot of Residuals for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

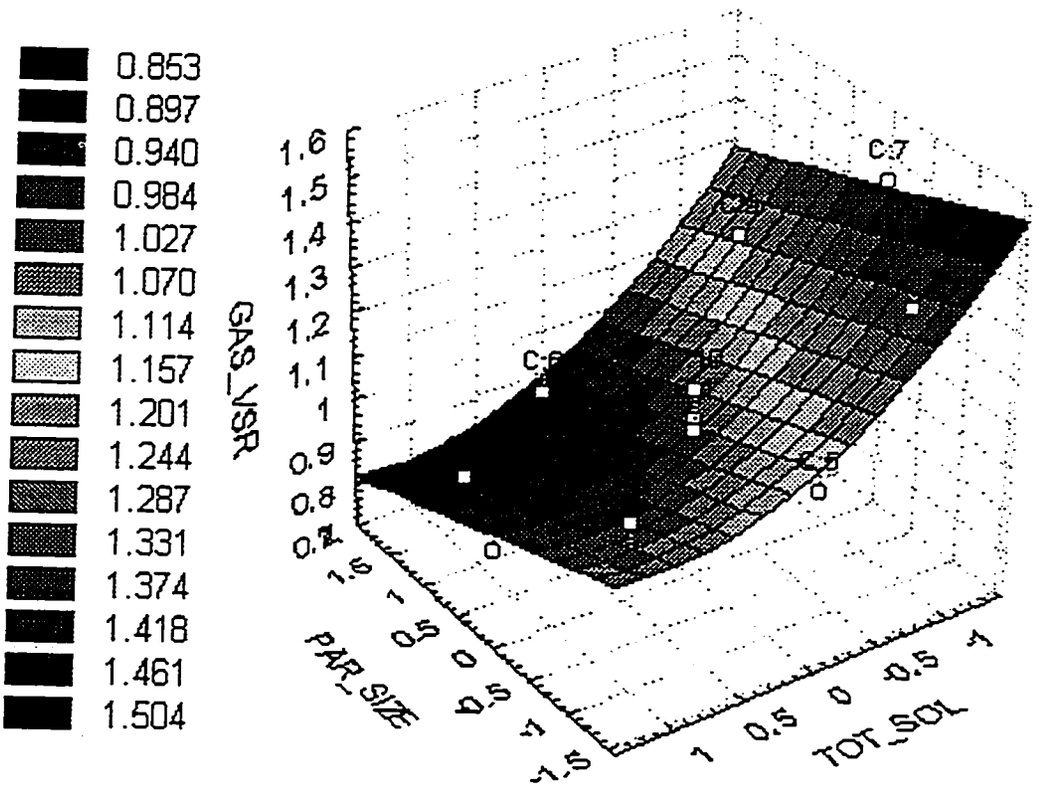

Figure E.15: Surface Plot for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

E.4 Chemical Oxygen Demand

a) Initial Model

Table E.25: Parameter Estimates for Initial Chemical Oxygen Demand Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	79.9338	-0.2950	13.0969	-1.6549	-9.6850	-1.7703
Std.Err.	5.3260	3.4064	3.8001	2.8209	4.3079	4.9255
t(8)	15.0082	-0.0866	3.4465	-0.5866	-2.2482	-0.3594
p-level	0.0000	0.9338	0.0137	0.5788	0.0656	0.7316
Variance	explained	73.944%			R	0.8599

Table E.26: ANOVA Table for Initial Chemical Oxygen Demand Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	5	35317.71	7063.54	61.35	8.75	4.39
Residual	6	690.82	115.14			
(Lack of Fit)	3	598.45	199.48	6.48	29.46	9.28
(Pure Error)	3	92.38	30.14			
Total	11					

Table E.27: Correlation Matrix of Parameter Estimates for Initial Chemical Oxygen Demand Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
β_0	1.0000	0.1636	0.0000	-0.6235	-0.6662	0.0000
β_1	0.1636	1.0000	0.0000	-0.3363	-0.0705	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	-0.0567
β_{11}	-0.6235	-0.3363	0.0000	1.0000	0.2618	0.0000
β_{22}	-0.6662	-0.0705	0.0000	0.2618	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	0.0000	1.0000

Table E.28: Pure Error Variance for Initial Chemical Oxygen Demand Model

For replicate set	
Mean	*79.2359*
sum of residuals squared	92.37613
degrees of freedom	3
pure error variance	30.1425

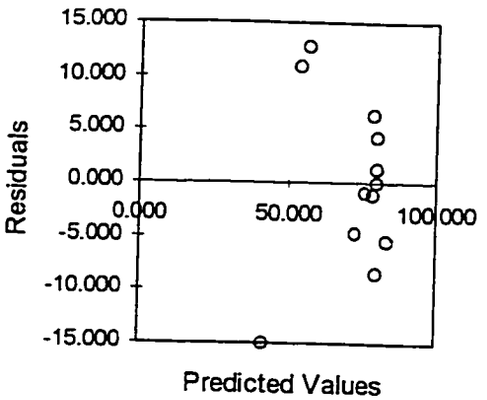

Figure E.16 Residuals versus Predicted Values for Initial Chemical Oxygen Demand Model

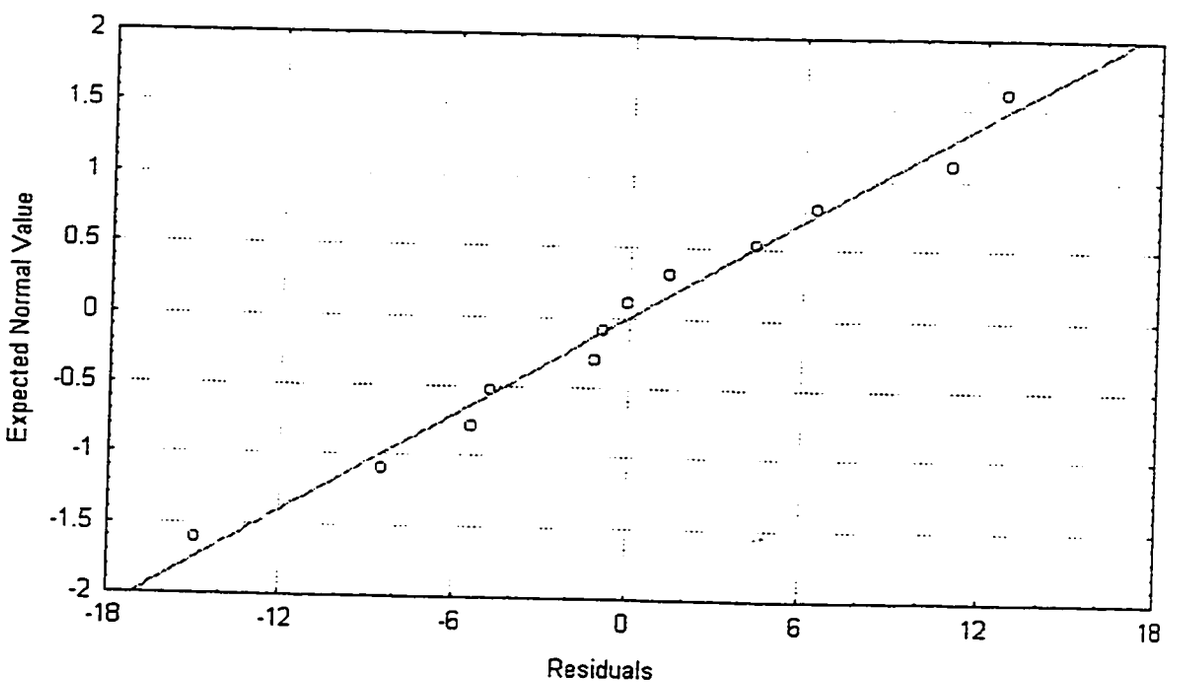

Figure E.17 Normal Probability Plot of Residuals for the Chemical Oxygen Demand Model

b) Final Model

Table E.29: Parameter Estimates for Final Chemical Oxygen Demand Model

	β_0	β_2	β_{22}
Estimate	77.9073	13.0195	-8.9991
Std.Err.	3.5493	3.2402	3.5500
t(8)	21.9501	4.0182	-2.5350
p-level	0.0000	0.0030	0.0320
Variance	explained	71.493%	R 0.8455

Table E.30: ANOVA Table for Final Chemical Oxygen Demand Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	2	47882.19	23941.10	285.09	8.02	4.26
Residual	9	755.78	83.98			
(Lack of Fit)	6	658.29	109.72	3.38	27.91	8.94
(Pure Error)	3	97.49	32.50			
Total	11					

Table E.31: Correlation Matrix of Parameter Estimates for Final Chemical Oxygen Demand Model

	β_0	β_2	β_{22}
β_0	1.0000	0.0000	-0.6667
β_2	0.0000	1.0000	0.0000
β_{22}	-0.6667	0.0000	1.0000

Table E.32: Calculations for Pure Error Variance for Final Chemical Oxygen Demand Model

For replicate set	
Mean	*79.2359*
sum of residuals squared	97.48843
degrees of freedom	3
pure error variance	30.496

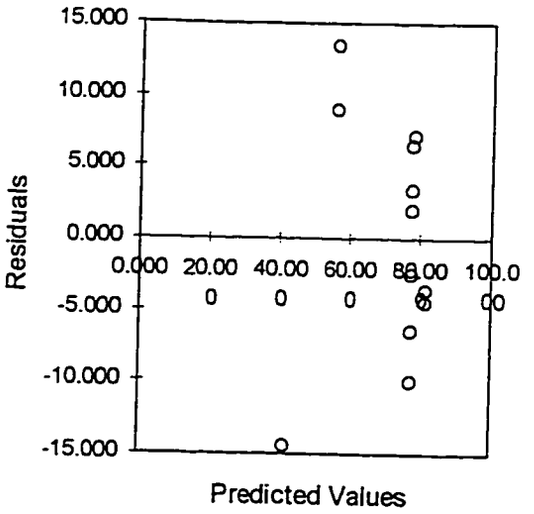

Figure E.18: Residuals Versus Predicted Values for Final Chemical Oxygen Demand Model

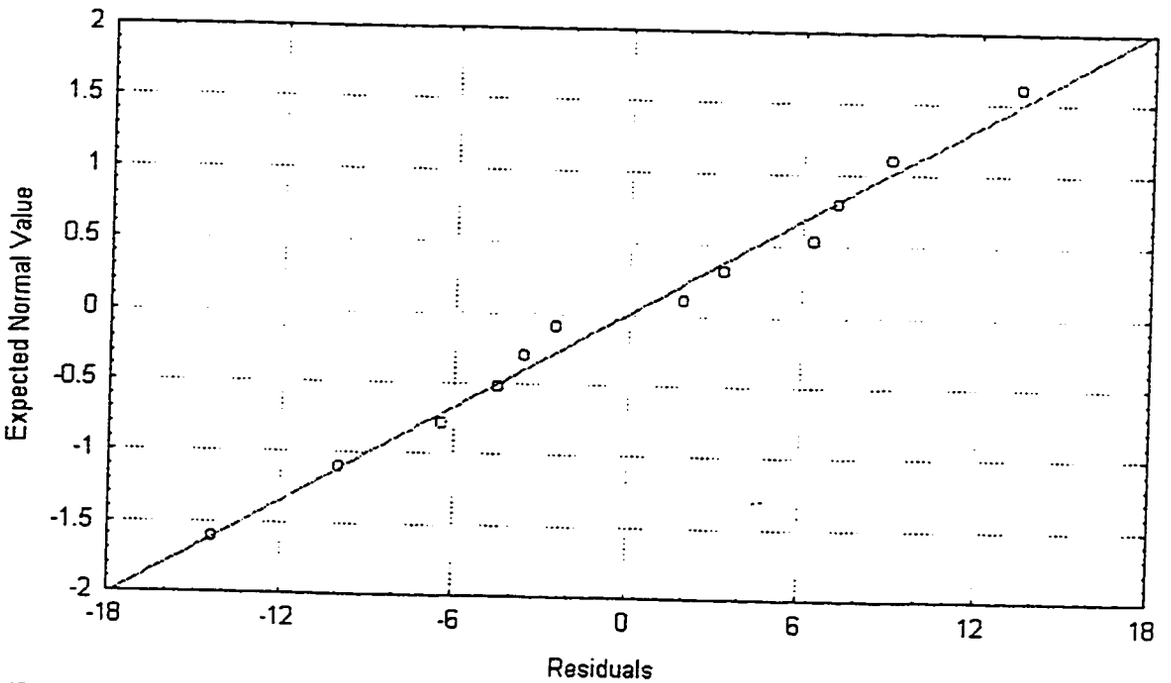

Figure E.19: Normal Probability Plot of Residuals for Final Chemical Oxygen Demand Model

E.5 Volatile Solids Reduction

a) Initial Model

Table E.33: Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	52.1200	-1.5134	13.0293	-3.1752	0.9097	-4.1606
Std.Err.	1.3420	0.8583	0.9575	0.7108	1.0855	1.2411
t(8)	38.8363	-1.7631	13.6070	-4.4670	0.8380	-3.3523
p-level	0.0000	0.1283	0.0000	0.0043	0.4341	0.0154
Variance	explained	97.440%			R 0.9871	

Table E.34: ANOVA Table for Volatile Solids Reduction Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	5	132448.58	26489.72	3623.53	8.75	4.39
Residual	6	43.86	7.31			
(Lack of Fit)	3	33.29	11.10	3.15	29.46	9.28
(Pure Error)	3	10.57	3.52			
Total	11					

Table E.35: Correlation Matrix of Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
β_0	1.0000	0.1636	0.0000	-0.6235	-0.6662	0.0000
β_1	0.1636	1.0000	0.0000	-0.3363	-0.0705	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	-0.0567
β_{11}	-0.6235	-0.3363	0.0000	1.0000	0.2618	0.0000
β_{22}	-0.6662	-0.0705	0.0000	0.2618	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	0.0000	1.0000

Table E.36: Pure Error Variance for Volatile Solids Reduction Model

For replicate set	
Mean	*51.849*
sum of residuals squared	10.57384
degrees of freedom	3
pure error variance	3.5246

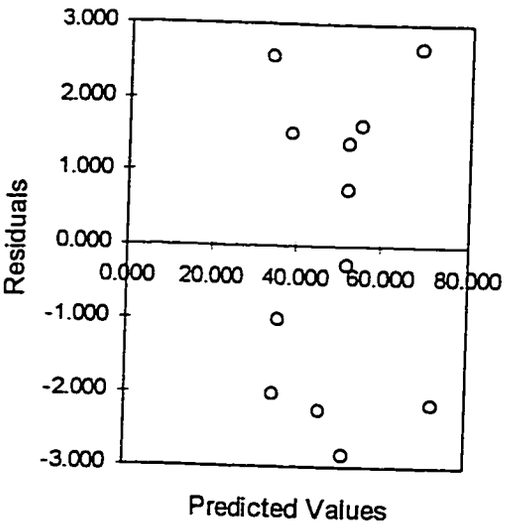

Figure E.20: Residuals Versus Predicted Values for Volatile Solids Reduction Model

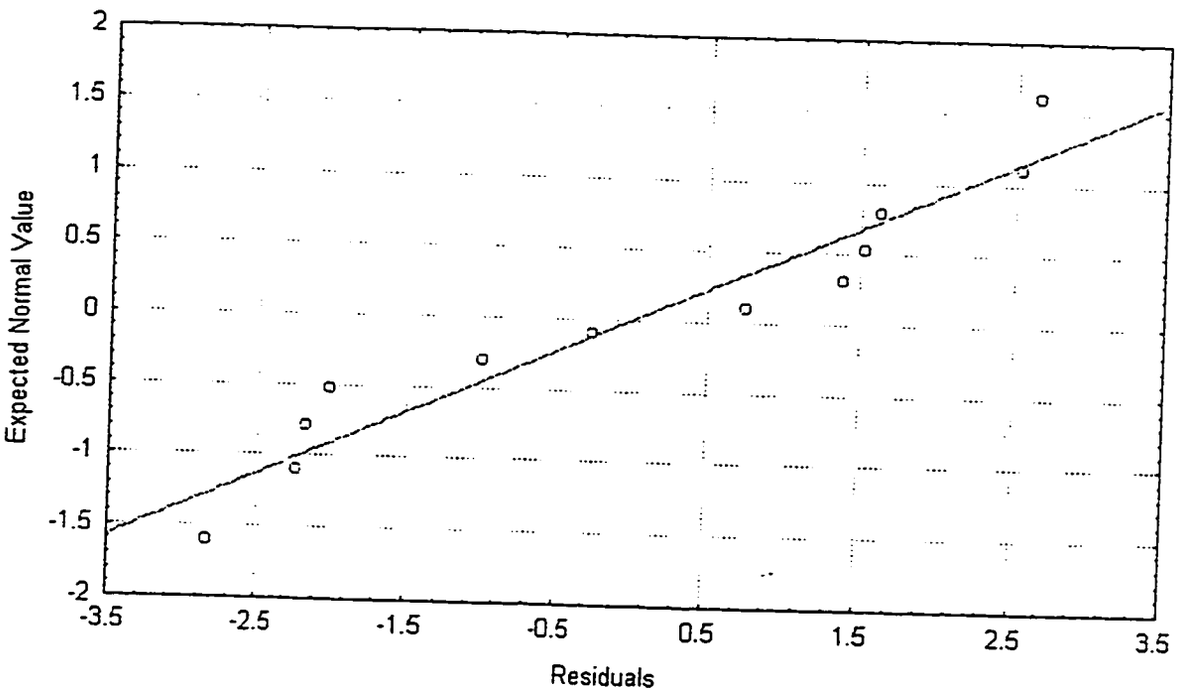

Figure E.21: Normal Probability Plot of Residuals for Volatile Solids Reduction Model

b) Final Model

Table E.37: Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_2	β_{11}	β_{12}
Estimate	53.1372	13.0293	-3.7181	-4.1606
Std.Err.	1.0840	1.0501	0.7101	1.3610
t(8)	49.0173	12.4082	-5.2359	-3.0570
p-level	0.0000	0.0000	0.0008	0.0157
Variance	explained	95.896%	R	0.9793

Table E.38: ANOVA Table for Volatile Solids Reduction Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	3	121537.62	40512.54	4608.25	7.59	4.07
Residual	8	70.33	8.79			
(Lack of Fit)	5	53.70	10.74	1.94	28.24	9.01
(Pure Error)	3	16.63	5.54			
Total	11					

Table E.39: Correlation Matrix of Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_2	β_{11}	β_{12}
β_0	1.0000	0.0000	-0.6137	0.0000
β_2	0.0000	1.0000	0.0000	-0.0567
β_{11}	-0.6137	0.0000	1.0000	0.0000
β_{12}	0.0000	-0.0567	0.0000	1.0000

Table E.40: Calculations for Pure Error Variance for Volatile Solids Reduction Model

For replicate set	
Mean	*51.8849*
sum of residuals squared	16.62639
degrees of freedom	3
pure error variance	5.5421

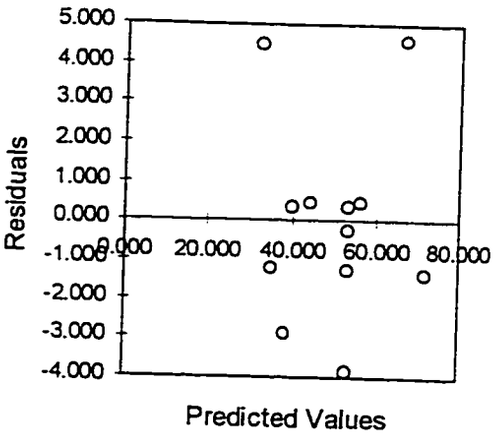

Figure E.22: Residuals Versus Predicted Values for Volatile Solids Reduction Model

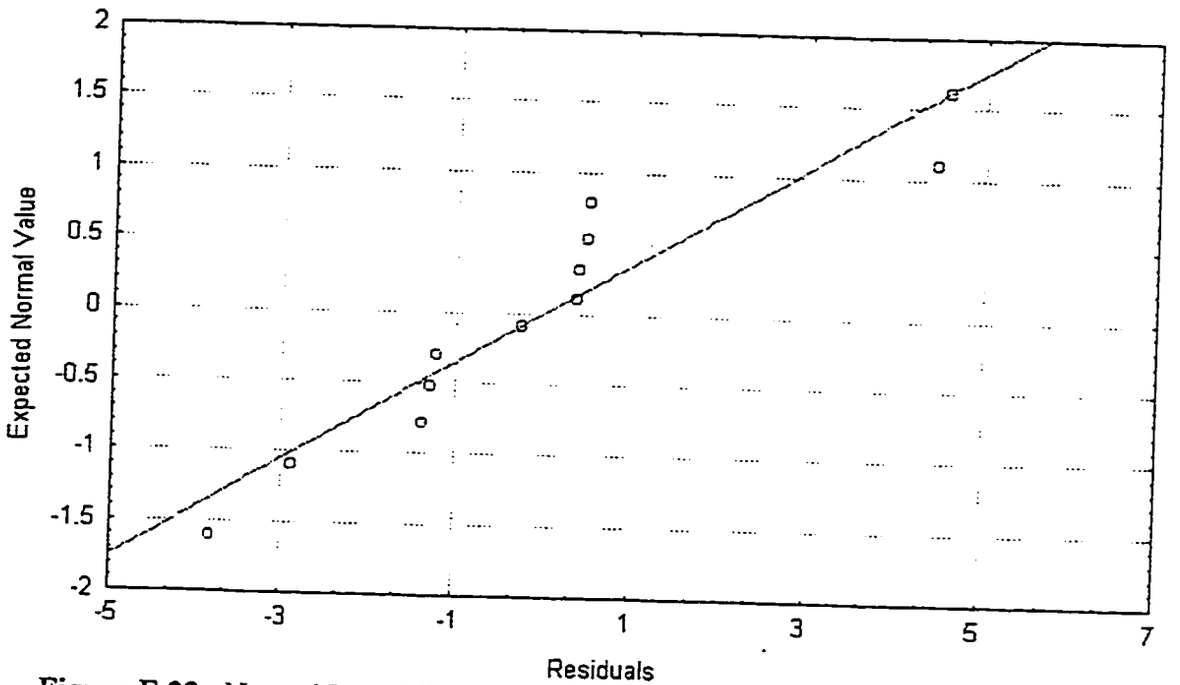

Figure E.23: Normal Probability of Residuals for Volatile Solids Reduction Model

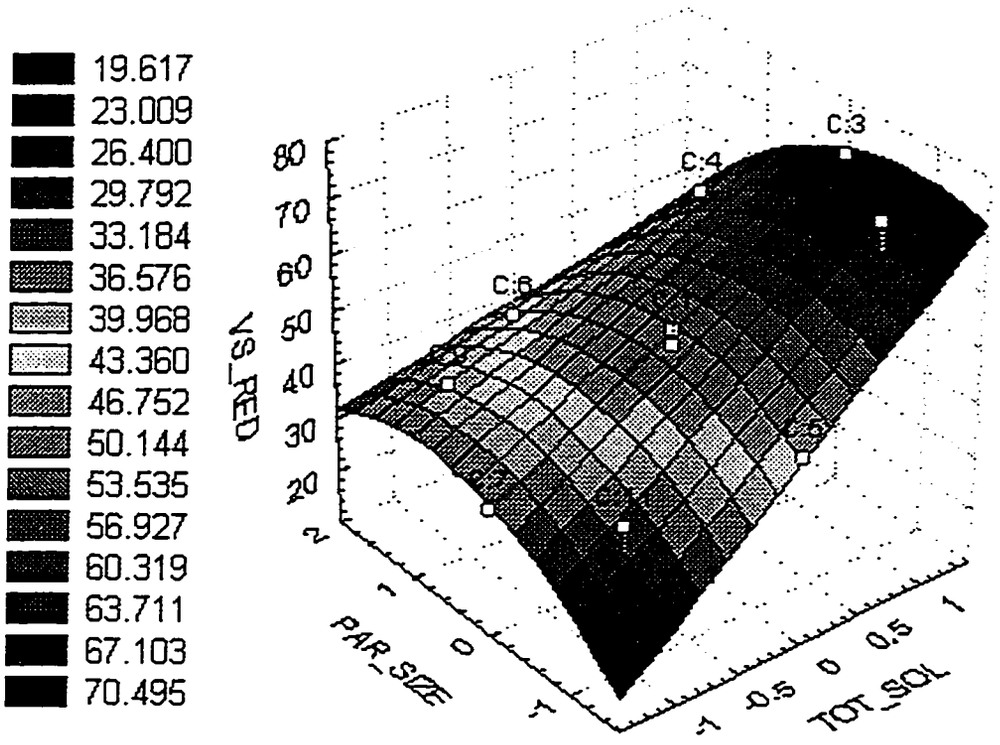

Figure E.24: Surface Plot for Volatile Solids Reduction Model

E.6 Total Solids Reduction

a) Initial Model

Table E.41: Parameter Estimates for Initial Total Solids Reduction Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	33.5935	-0.4122	8.5633	-2.3763	-0.1972	-3.0191
Std.Err.	0.6610	0.4228	0.4716	0.3501	0.5346	0.6113
t(8)	50.8236	-0.9750	18.1576	-6.7876	-0.3688	-4.9391
p-level	0.0000	0.3672	0.0000	0.0005	0.7250	0.0026
Variance	explained	98.542%		R	0.9927	

Table E.42: ANOVA Table for Initial Total Solids Reduction Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	5	47027.24	9405.45	5303.83	8.75	4.39
Residual	6	10.64	1.77			
(Lack of Fit)	3	8.41	2.80	3.78	29.46	9.28
(Pure Error)	3	2.23	0.74			
Total	11					

Table E.43: Correlation Matrix of Parameter Estimates for Initial Total Solids Reduction Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
β_0	1.0000	0.1636	0.0000	-0.6235	-0.6662	0.0000
β_1	0.1636	1.0000	0.0000	-0.3363	-0.0705	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	-0.0567
β_{11}	-0.6235	-0.3363	0.0000	1.0000	0.2618	0.0000
β_{22}	-0.6662	-0.0705	0.0000	0.2618	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	0.0000	1.0000

Table E.44: Pure Error Variance for Initial Total Solids Reduction Model

For replicate set	
Mean	*33.4272*
sum of residuals squared	2.234463
degrees of freedom	3
pure error variance	0.7448

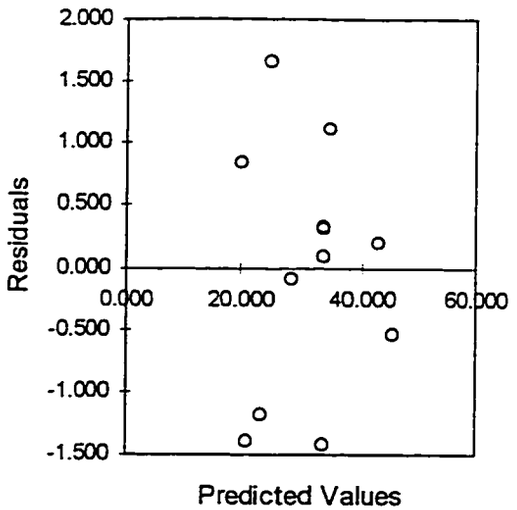

Figure E.25: Residuals versus Predicted Values for Initial Total Solids Reduction Model

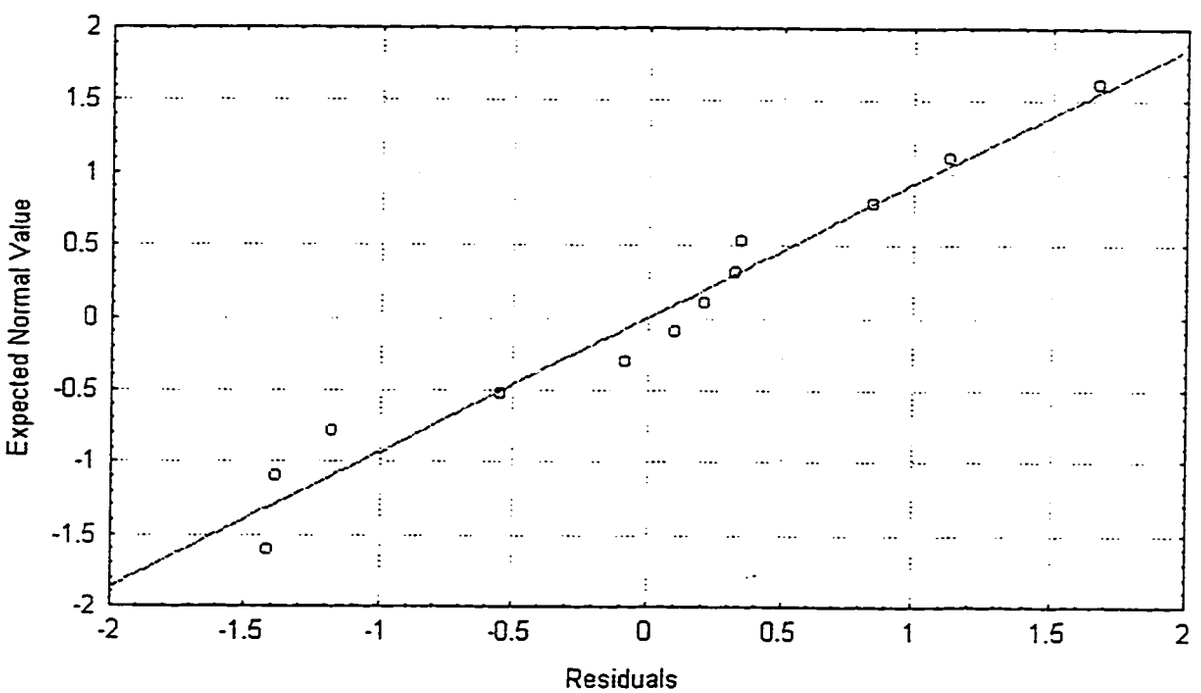

Figure E.26: Normal Probability Plot of Residuals for Initial Total Solids Reduction Model

b) Final Model

Table E.45: Parameter Estimates for Final Total Solids Reduction Model

	β_0	β_2	β_{11}	β_{12}
Estimate	33.5086	8.5633	-2.4544	-3.0191
Std.Err.	0.4601	0.4456	0.3014	0.5776
t(8)	72.8357	19.2160	-8.1443	-5.2270
p-level	0.0000	0.0000	0.0000	0.0008
	Variance explained	98.264%	R	0.9913

Table E.46: ANOVA Table for Final Total Solids Reduction Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	3	49492.44	16497.48	10419.29	7.59	4.07
Residual	8	12.67	1.58			
(Lack of Fit)	5	10.52	2.10	2.95	28.24	9.01
(Pure Error)	3	2.14	0.71			
Total	11					

Table E.47: Correlation Matrix of Parameter Estimates for Final Total Solids Reduction Model

	β_0	β_2	β_{11}	β_{12}
β_0	1.0000	0.0000	-0.6137	0.0000
β_2	0.0000	1.0000	0.0000	-0.0567
β_{11}	-0.6137	0.0000	1.0000	0.0000
β_{12}	0.0000	-0.0567	0.0000	1.0000

Table E.48: Pure Error Variance for Final Total Solids Reduction Model

For replicate set	
Mean	*33.4272*
sum of residuals squared	2.143816
degrees of freedom	3
pure error variance	0.7058

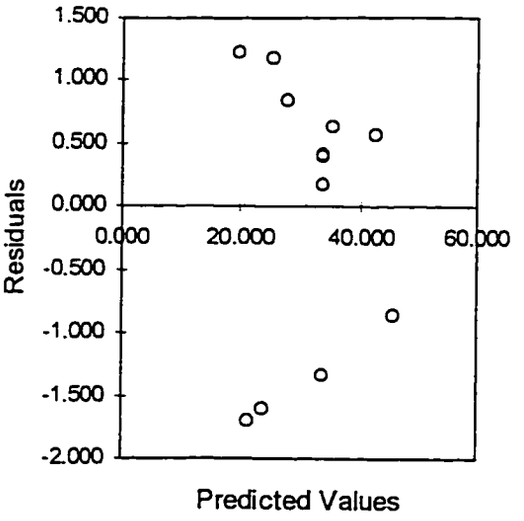

Figure E.27: Residuals Versus Predicted Values for Final Total Solids Reduction Model

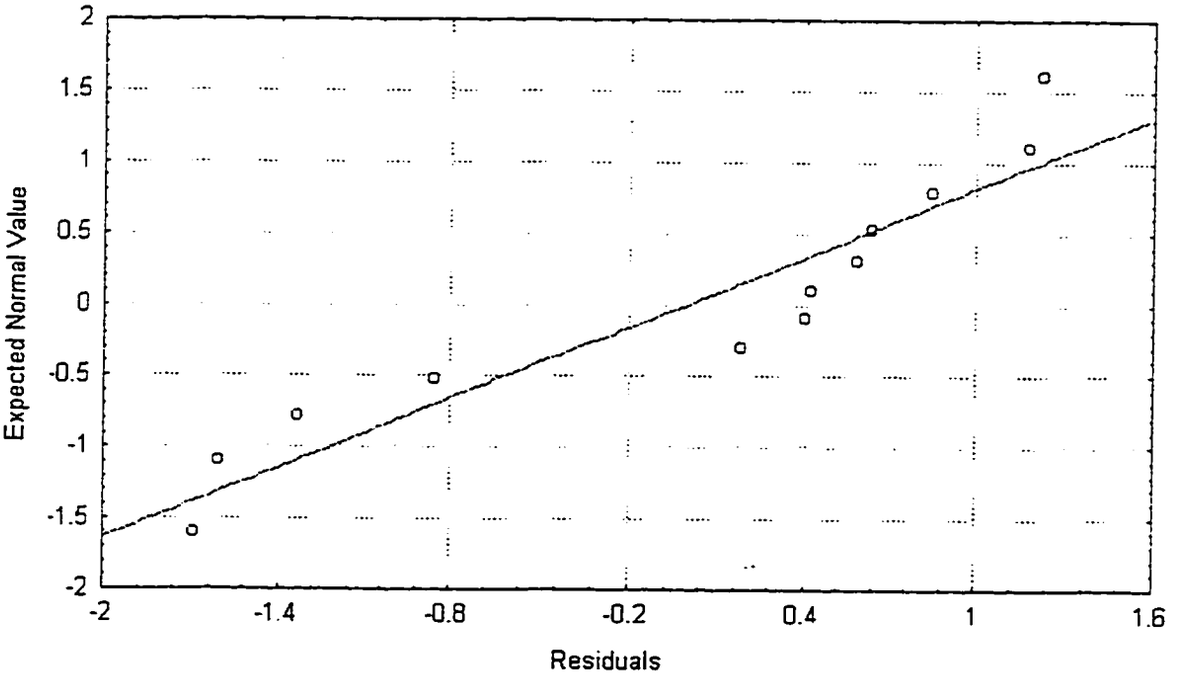

Figure E.28: Normal Probability Plot of Residuals for Final Total Solids Reduction Model

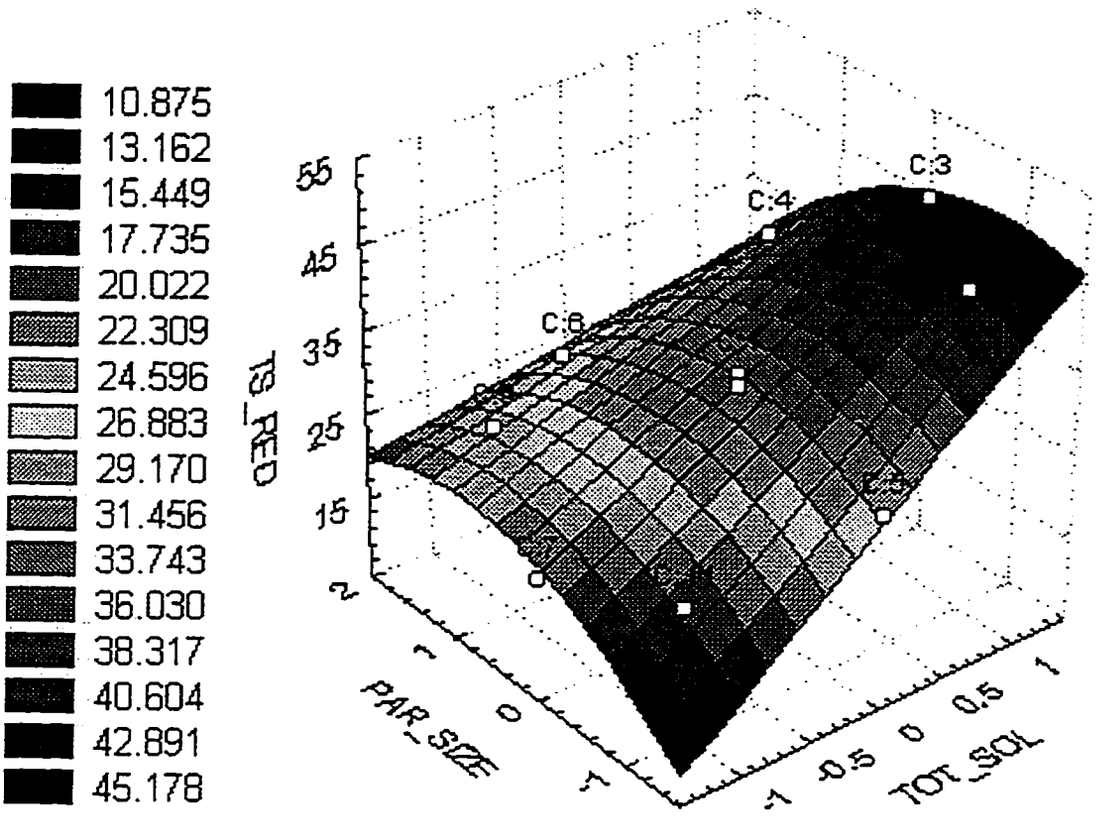

Figure E.29: Surface Plot for Final Total Solids Reduction Model

E.7 Biogas Methane Concentration

Table E.49: Parameter Estimates for Biogas Methane Concentration Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	59.3471	-0.9425	0.2747	1.1850	1.9720	-1.0104
Std.Err.	1.2929	0.8269	0.9225	0.6848	1.0458	1.1957
t(8)	45.9009	-1.1398	0.2978	1.7304	1.8857	-0.8451
p-level	0.0000	0.2978	0.7759	0.1343	0.1083	0.4305

Variance explained 51.457% R 0.7173

Table E.50: Pure Error Variance for Biogas Methane Concentration Model

For replicate set	
Mean	*59.5325*
sum of residuals squared	26.57275
degrees of freedom	3
pure error variance	8.8118

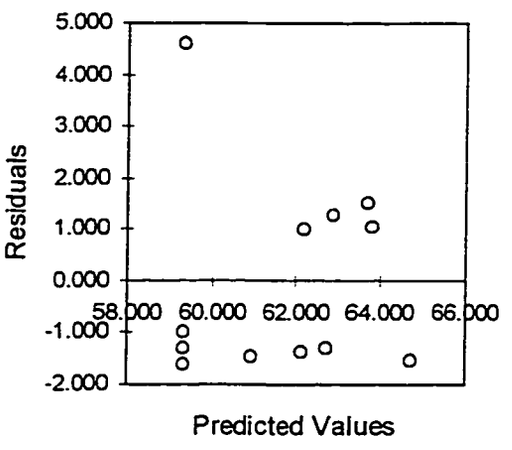

Figure E.30: Residuals Versus Predicted Values for Biogas Methane Concentration

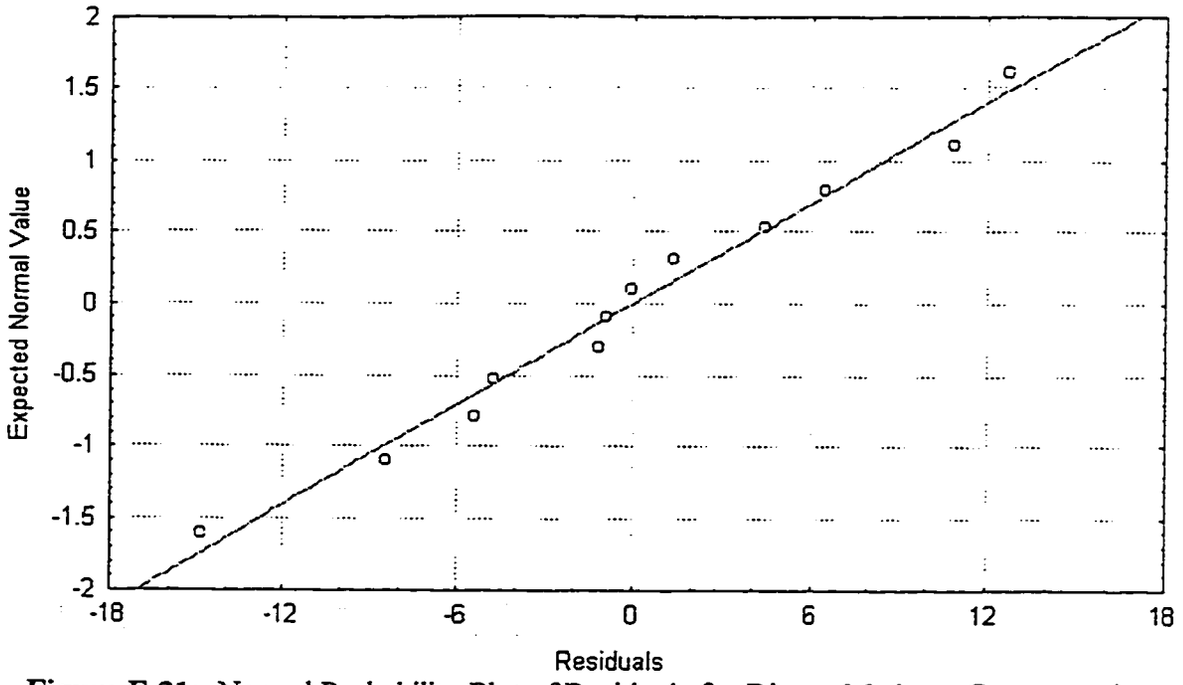

Figure E.31: Normal Probability Plot of Residuals for Biogas Methane Concentration

APPENDIX F:
Thermal Model Results

This Appendix includes all of the empirical modeling data and results for the thermal pretreatment

F.1 Cumulative Gas Production at STP

a) Initial Model

Table F.1: Parameter Estimates for Initial Cumulative Gas Production Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	3.7033	0.5036	1.3786	-0.1676	0.4297	0.2787
Std.Err.	0.1542	0.0987	0.1101	0.0817	0.1248	0.1426
t(8)	24.0086	5.1050	12.5267	-2.0511	3.4444	1.9541
p-level	0.0000	0.0022	0.0000	0.0861	0.0137	0.0985
Variance	explained	97.177%		R	0.9858	

Table F.2: ANOVA Table for Initial Cumulative Gas Production Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	5	1986.62	397.32	4114.32	8.75	4.39
Residual	6	0.58	0.10			
(Lack of Fit)	3	0.36	0.12	1.66	29.46	9.28
(Pure Error)	3	0.22	0.07			
Total	11					

Table F.3: Correlation Matrix of Parameter Estimates for Initial Cumulative Gas Production Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
β_0	1.0000	0.1636	0.0000	-0.6235	-0.6662	0.0000
β_1	0.1636	1.0000	0.0000	-0.3363	-0.0705	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	-0.0567
β_{11}	-0.6235	-0.3363	0.0000	1.0000	0.2618	0.0000
β_{22}	-0.6662	-0.0705	0.0000	0.2618	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	0.0000	1.0000

Table F.4: Pure Error Variance for Initial Cumulative Gas Production Model

For replicate set	
Mean	3.6772
sum of residuals squared	0.218
degrees of freedom	3
Pure error variance	0.0727

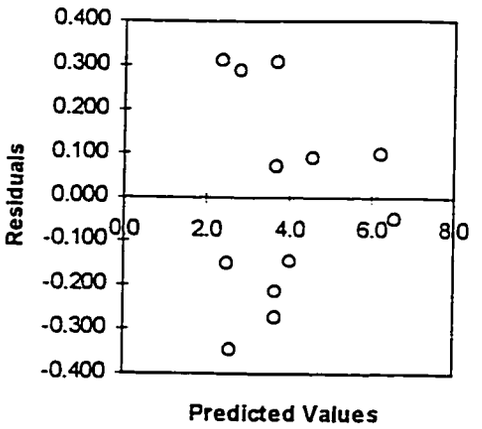

Figure F.1: Residuals Versus Predicted Values for Initial Cumulative Gas Production Model

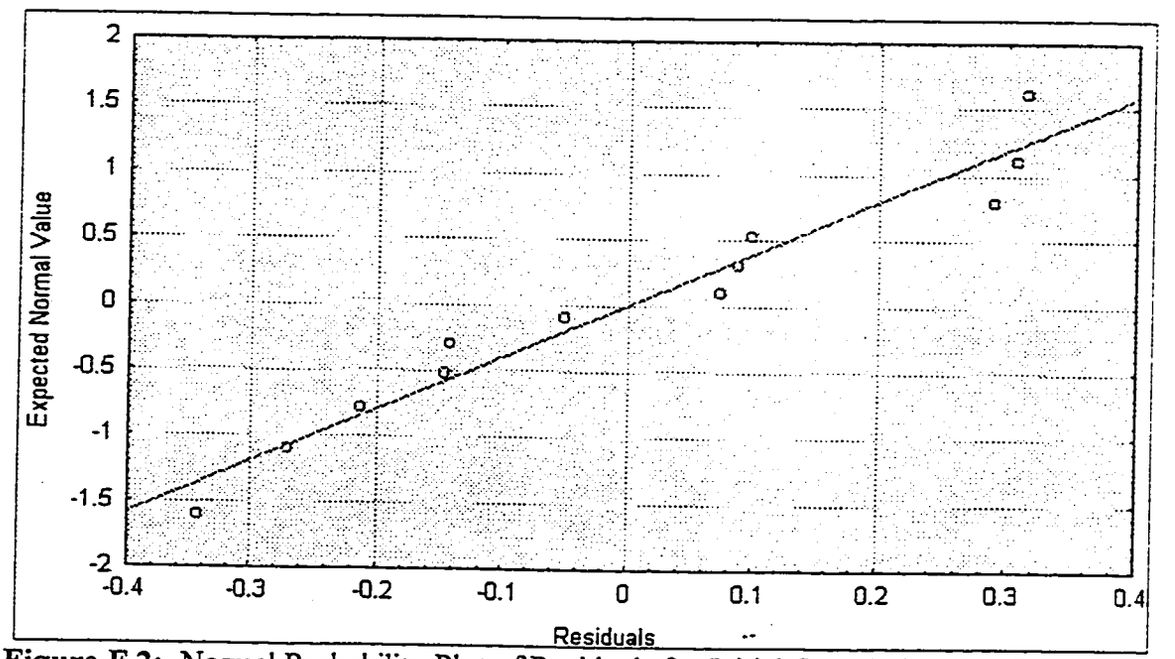

Figure F.2: Normal Probability Plot of Residuals for Initial Cumulative Gas Production Model

b) Final Model

Table F.5: Parameter Estimates for Final Cumulative Gas Production Model

	β_0	β_1	β_2	β_{22}
Estimate	3.5060	0.4356	1.3908	0.4967
Std.Err.	0.1597	0.1230	0.1455	0.1594
t(8)	21.9565	3.5408	9.5598	3.1157
p-level	0.0000	0.0076	0.0000	0.0143
Variance	explained	93.400%	R	0.9664

Table F.6: ANOVA Table for Final Cumulative Gas Production Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	3	2004.00	668.00	3945.56	7.59	4.07
Residual	8	1.35	0.17			
(Lack of Fit)	5	1.02	0.20	1.84	28.24	9.01
(Pure Error)	3	0.33	0.11			
Total	11					

Table F.7: Correlation Matrix of Parameter Estimates for Final Cumulative Gas Production Model

	β_0	β_1	β_2	β_{22}
β_0	1.0000	-0.0626	0.0000	-0.6665
β_1	-0.0626	1.0000	0.0000	0.0193
β_2	0.0000	0.0000	1.0000	0.0000
β_{22}	-0.6665	0.0193	0.0000	1.0000

Table F.8: Pure Error Variance for Final Cumulative Gas-Production Model

For replicate set	
mean	3.67725
sum of residuals squared	0.332
degrees of freedom	3
pure error variance	0.1107

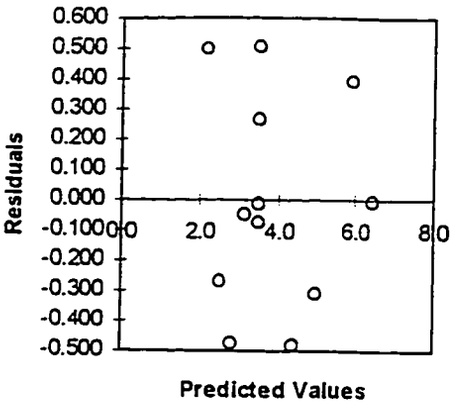

Figure F.3: Residuals Versus Predicted Values for Final Cumulative Gas Production Model

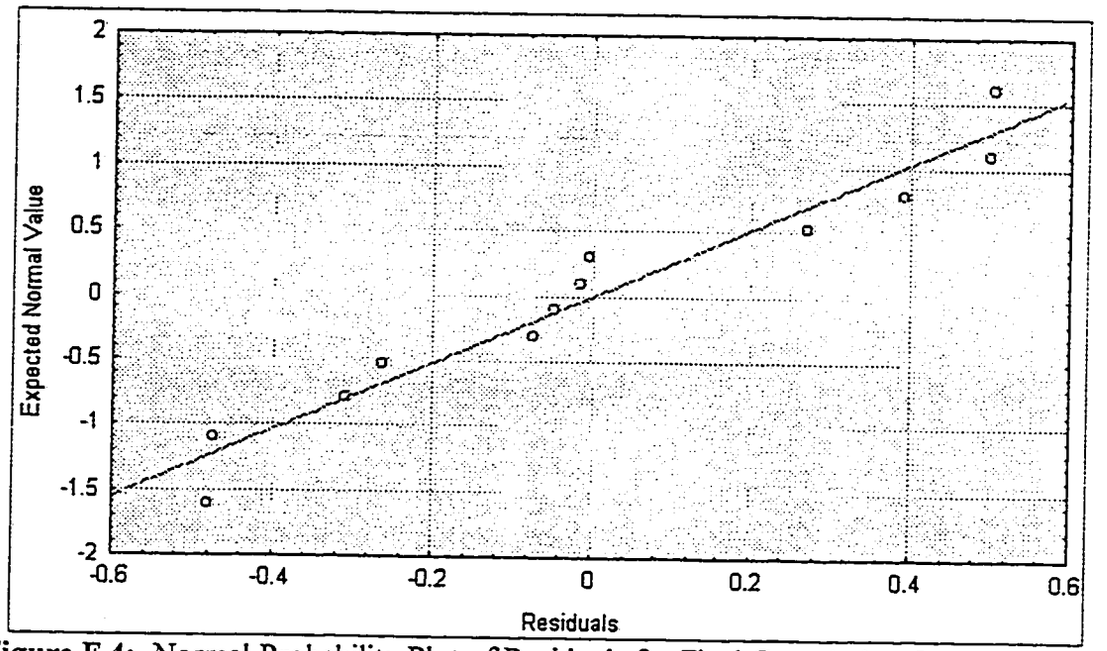

Figure F.4: Normal Probability Plot of Residuals for Final Cumulative Gas Production Model

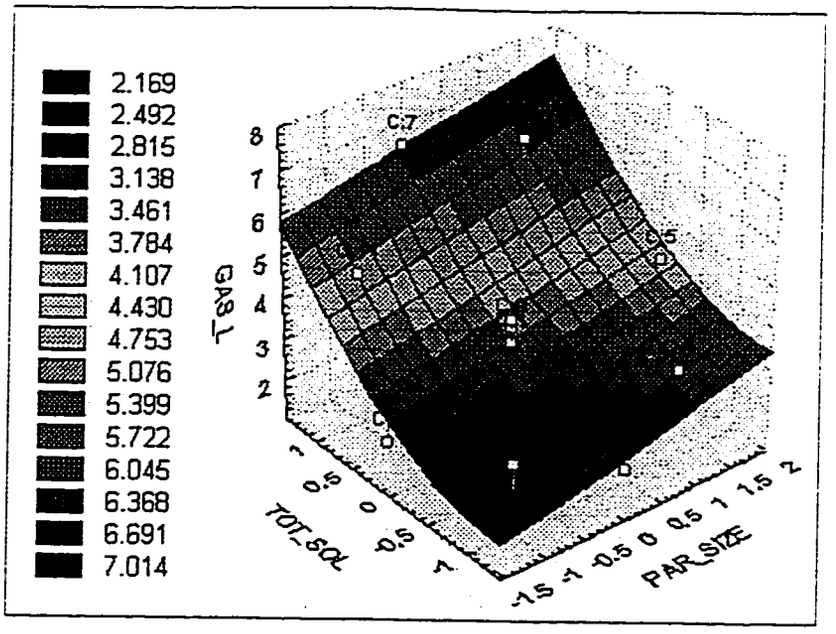

Figure F.5: Surface Plot of Final Cumulative Gas Production Model

F.2 Cumulative Gas Production at STP with Respect to Volatile Solids Addition

a) Initial Model

Table F.9: Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	0.5135	0.0648	0.0042	-0.0192	0.0578	0.0309
Std.Err.	0.0214	0.0137	0.0152	0.0113	0.0173	0.0197
t(8)	24.0476	4.7437	0.2786	-1.6983	3.3447	1.5647
p-level	0.0000	0.0032	0.7899	0.1404	0.0155	0.1687
Variance	explained	86.916%		R	0.9323	

Table F.10: ANOVA Table for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	5	3.76	0.75	406.57	8.75	4.39
Residual	6	0.01	0.00			
(Lack of Fit)	3	0.01	0.00	1.62	29.46	9.28
(Pure Error)	3	0.00	0.00			
Total	11					

Table F.11: Correlation Matrix of Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
β_0	1.0000	0.1636	0.0000	-0.6235	-0.6662	0.0000
β_1	0.1636	1.0000	0.0000	-0.3363	-0.0705	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	-0.0567
β_{11}	-0.6235	-0.3363	0.0000	1.0000	0.2618	0.0000
β_{22}	-0.6662	-0.0705	0.0000	0.2618	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	0.0000	1.0000

Table E.12: Calculation for Pure Error Variance for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

For replicate set	
Mean	0.51075
sum of residuals squared	0.004
degrees of freedom	3
pure error variance	0.00133

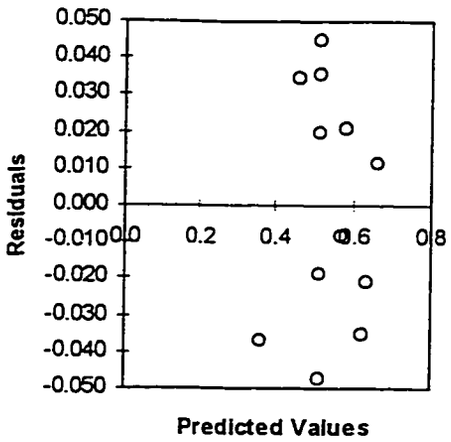

Figure F.6: Residuals versus Predicted Values for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

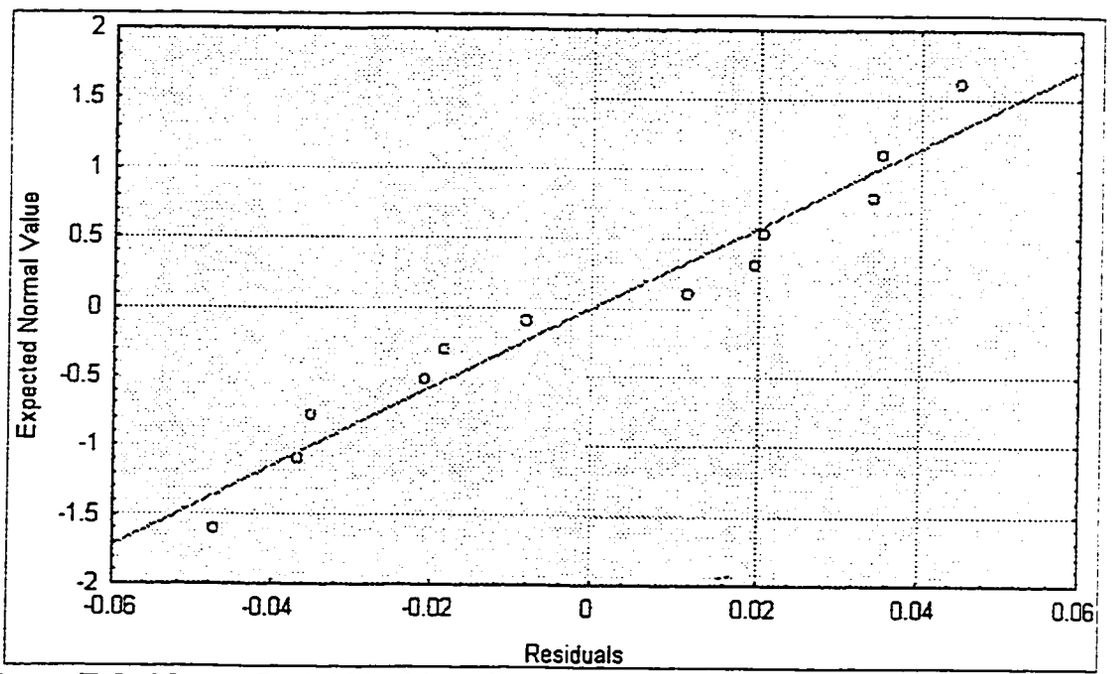

Figure F.7: Normal Probability Plot of Residuals for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

b) Final Model

Table F.13: Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_{22}	
Estimate	0.4909	0.0570	0.0654	
Std.Err.	0.0188	0.0145	0.0188	
t(8)	26.0479	3.9251	3.4783	
p-level	0.0000	0.0035	0.0070	
Variance	explained	74.992%		R 0.8960

Table F.14: ANOVA Table for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	2	2.80	1.40	594.02	8.02	4.26
Residual	9	0.02	0.00			
(Lack of Fit)	6	0.02	0.00	1.33	27.91	8.94
(Pure Error)	3	0.01	0.00			
Total	11					

Table F.15: Correlation Matrix of Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_{22}
β_0	1.0000	-0.0626	-0.6665
β_1	-0.0626	1.0000	0.0193
β_{22}	-0.6665	0.0193	1.0000

Table F.16: Pure Error Variance for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

For replicate set	
mean	0.51075
sum of residuals squared	0.006
degrees of freedom	3
pure error variance	0.002

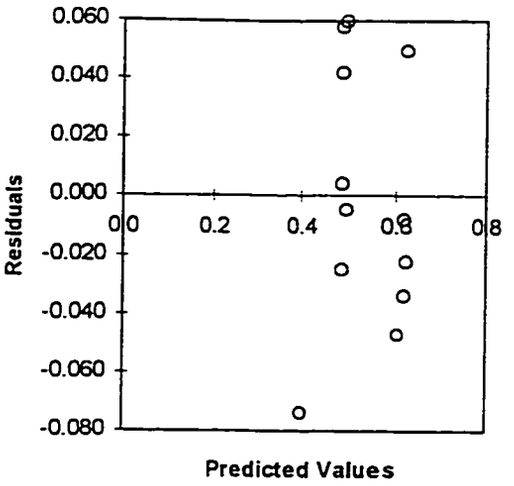

Figure F.8: Residuals Versus Predicted Values for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

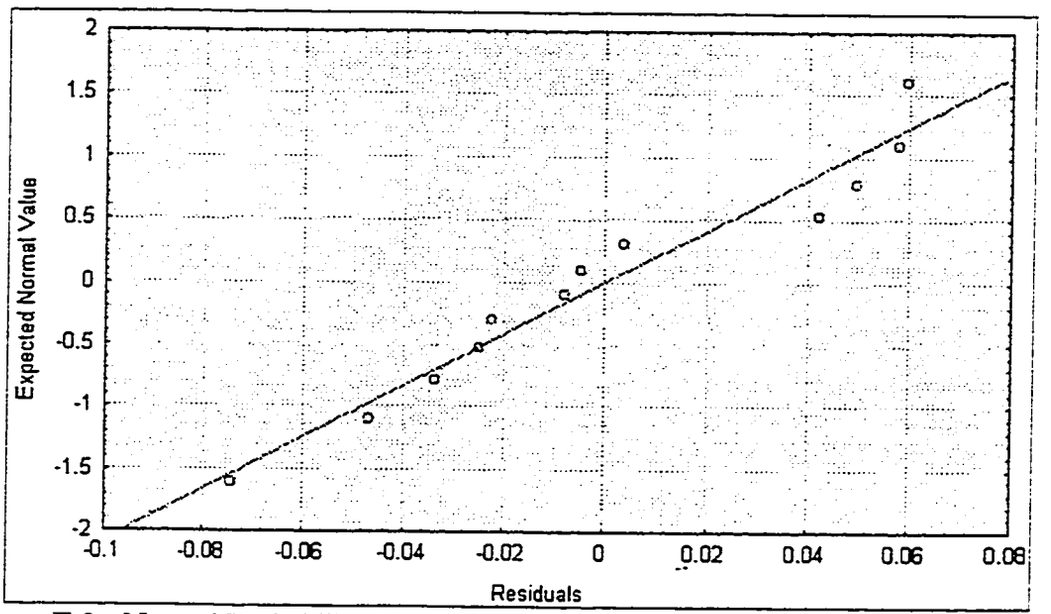

Figure F.9: Normal Probability Plot of Residuals for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

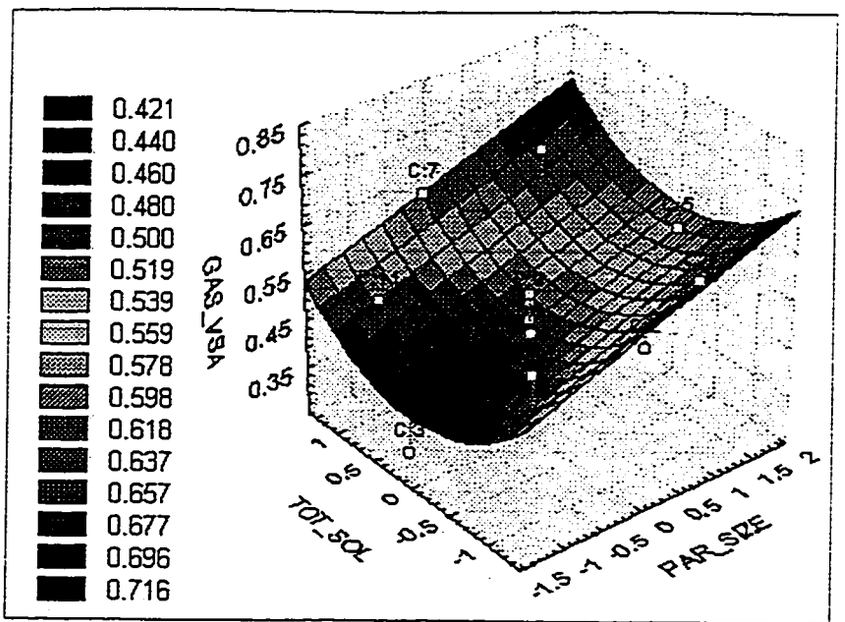

Figure F.10: Surface Plot for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

F.3 Cumulative Gas Production at STP with Respect to Volatile Solids Removal

Table F.17: Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	0.9393	0.1312	-0.2785	0.0579	0.1485	0.1251
Std.Err.	0.0432	0.0277	0.0309	0.0229	0.0350	0.0400
t(8)	21.7195	4.7434	-9.0246	2.5262	4.2465	3.1278
p-level	0.0000	0.0032	0.0001	0.0449	0.0054	0.0204
Variance	explained	95.971%		R	0.9797	

Table F.18: ANOVA Table for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	5	-86.48	-17.30	-2278.47	8.75	4.39
Residual	6	0.05	0.01			
(Lack of Fit)	3	0.03	0.01	1.44	29.46	9.28
(Pure Error)	3	0.02	0.01			
Total	11					

Table F.19: Correlation Matrix of Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
β_0	1.0000	0.1636	0.0000	-0.6235	-0.6662	0.0000
β_1	0.1636	1.0000	0.0000	-0.3363	-0.0705	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	-0.0567
β_{11}	-0.6235	-0.3363	0.0000	1.0000	0.2618	0.0000
β_{22}	-0.6662	-0.0705	0.0000	0.2618	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	0.0000	1.0000

Table F.20: Pure Error Variance for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

For replicate set	
mean	0.9435
sum of residuals squared	0.019
degrees of freedom	3
pure error variance	0.0063

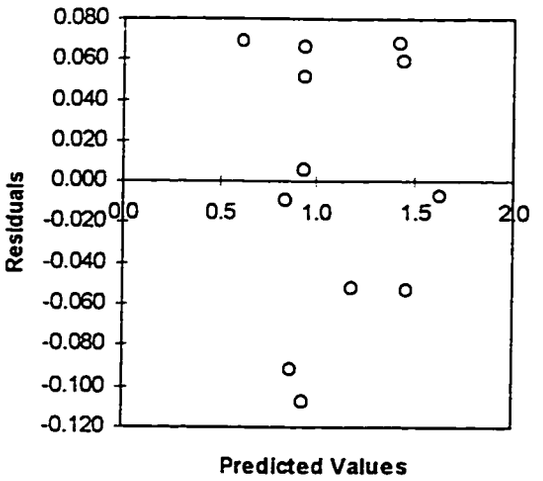

Figure F.11: Residual versus Predicted Values for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

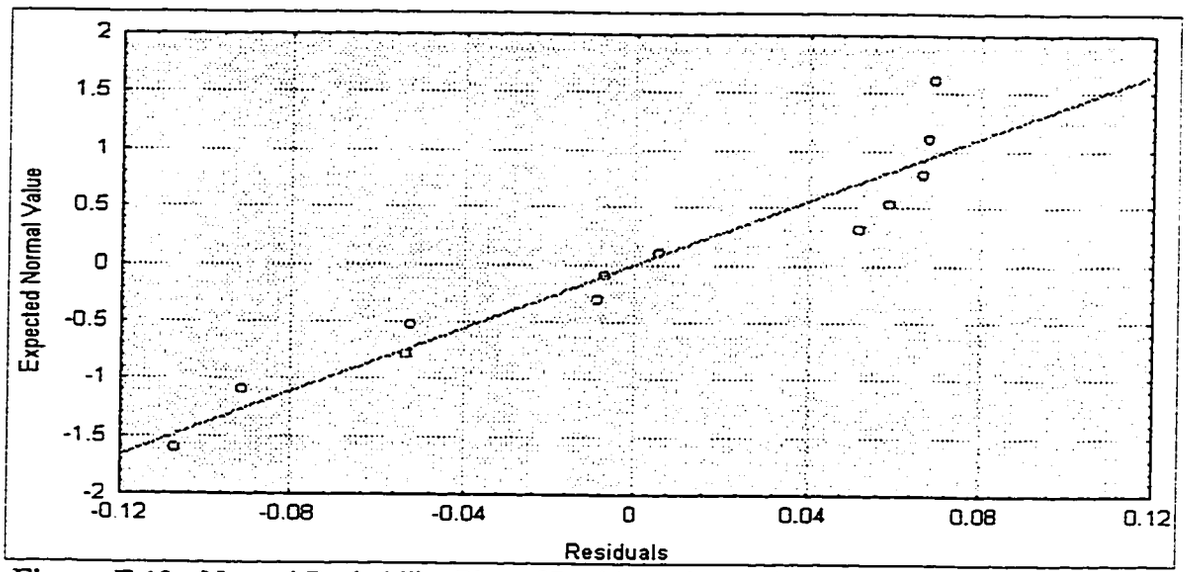

Figure F.12: Normal Probability Plot of Residuals for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

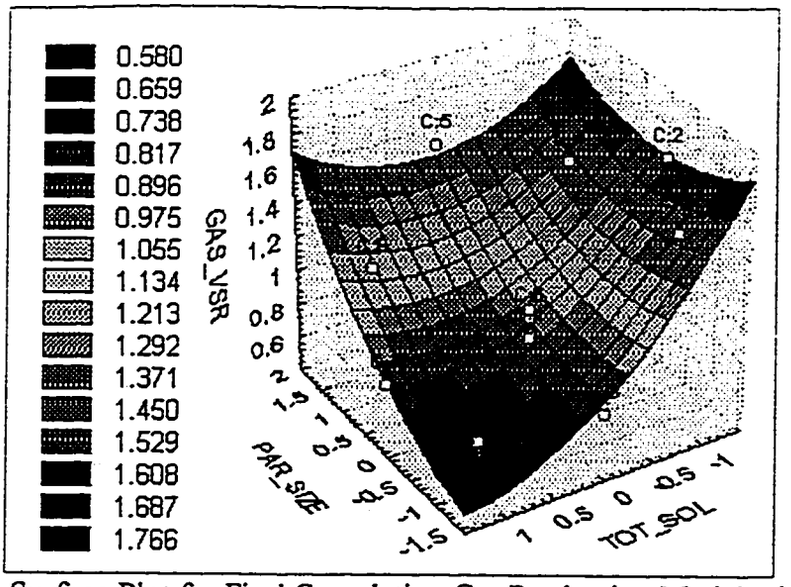

Figure F.13: Surface Plot for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

F.4 Chemical Oxygen Demand

a) Initial Model

Table F.2 1: Parameter Estimates for Initial Chemical Oxygen Demand Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	82.9034	0.5985	14.3242	-2.1756	-9.3347	-2.9231
Std.Err.	4.5933	2.9378	3.2773	2.4328	3.7152	4.2478
t(8)	18.0488	0.2037	4.3707	-0.8943	-2.5126	-0.6881
p-level	0.0000	0.8453	0.0047	0.4056	0.0457	0.5171
Variance	explained	81.057%		R	0.9003	

Table F.22: ANOVA Table for Initial Chemical Oxygen Demand Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	5	58308.13	11661.63	136.18	8.75	4.39
Residual	6	513.82	85.64			
(Lack of Fit)	3	373.76	124.59	2.67	29.46	9.28
(Pure Error)	3	140.06	46.69			
Total	11					

Table F.23: Correlation Matrix of Parameter Estimates for Initial Chemical Oxygen Demand Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
β_0	1.0000	0.1636	0.0000	-0.6235	-0.6662	0.0000
β_1	0.1636	1.0000	0.0000	-0.3363	-0.0705	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	-0.0567
β_{11}	-0.6235	-0.3363	0.0000	1.0000	0.2618	0.0000
β_{22}	-0.6662	-0.0705	0.0000	0.2618	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	0.0000	1.0000

Table F.24: Pure Error Variance for Initial Chemical Oxygen Demand Model

For replicate set	
Mean	82.355
sum of residuals squared	140.063
degrees of freedom	3
pure error variance	46.6877

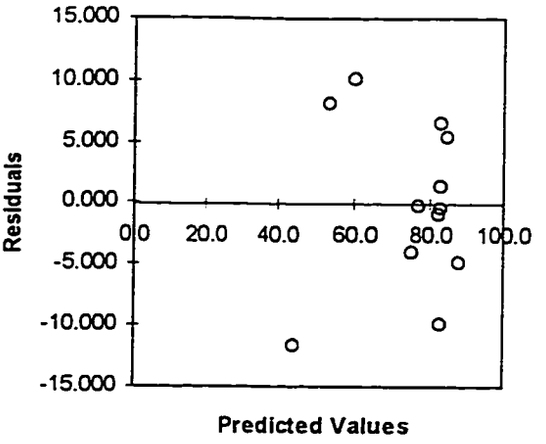

Figure F.14: Residuals versus Predicted Values for Initial Chemical Oxygen Demand Model

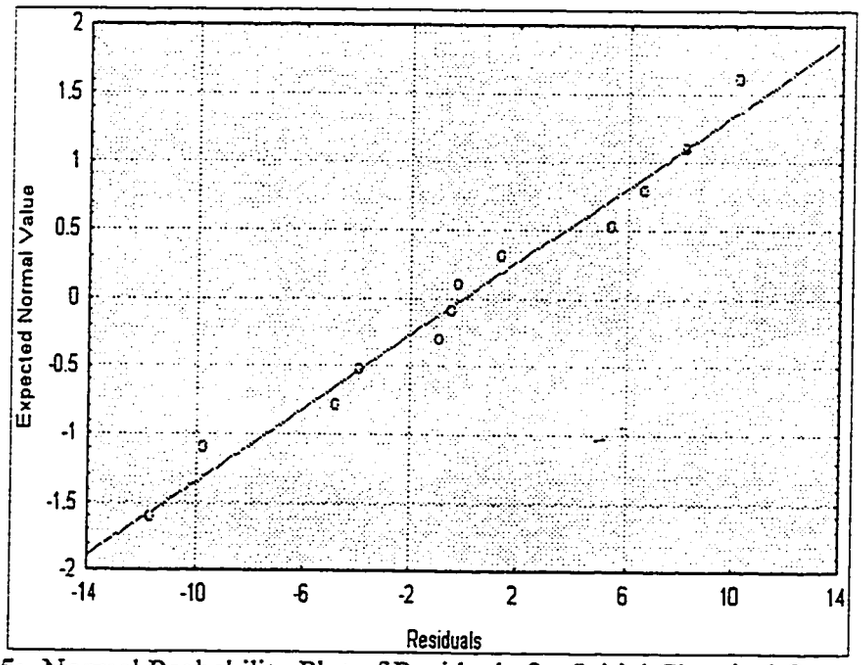

Figure F.15: Normal Probability Plot of Residuals for Initial Chemical Oxygen Demand Model

b) Final Model

Table F.25: Parameter Estimates for Final Chemical Oxygen Demand Model

	β_0	β_2	β_{22}
Estimate	80.3192	14.1963	-8.4576
Std.Err.	3.2244	2.9436	3.2251
t(8)	24.9096	4.8228	-2.6225
p-level	0.0000	0.0009	0.0277
	Variance explained	77.004%	R 0.8775

Table F.26: ANOVA Table for Final Chemical Oxygen Demand Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	2	74906.28	37453.14	540.39	8.02	4.26
Residual	9	623.76	69.31			
(Lack of Fit)	6	468.33	78.05	1.51	27.91	8.94
(Pure Error)	3	155.44	51.81			
Total	11					

Table F.27: Correlation Matrix of Parameter Estimates for Final Chemical Oxygen Demand Model

	β_0	β_2	β_{22}
β_0	1.0000	0.0000	-0.6667
β_2	0.0000	1.0000	0.0000
β_{22}	-0.6667	0.0000	1.0000

Table F.28: Calculations for Pure Error Variance for Final Chemical Oxygen Demand Model

For replicate set	
mean	82.355
sum of residuals squared	155.437
degrees of freedom	3
pure error variance	51.8123

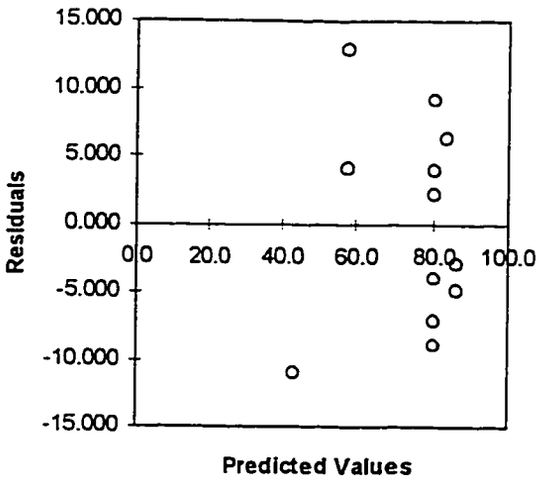

Figure F.16: Residuals Versus Predicted Values for Final Chemical Oxygen Demand Model

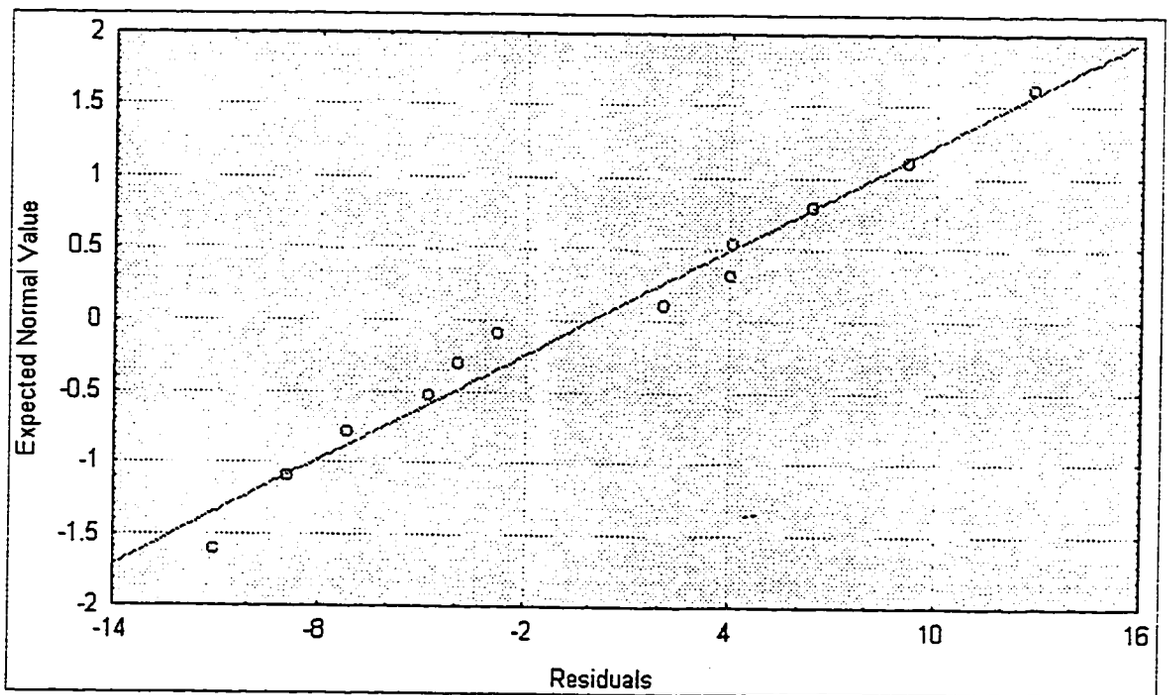

Figure F.17: Normal Probability Plot of Residuals for Final Chemical Oxygen Demand Model

F.5 Volatile Solids Reduction

a) Initial Model

Table F.29: Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	54.3985	-0.2151	13.2340	-4.0201	1.3561	-4.3725
Std.Err.	1.4464	0.9251	1.0320	0.7661	1.1699	1.3376
t(8)	37.6099	-0.2325	12.8237	-5.2476	1.1592	-3.2689
p-level	0.0000	0.8239	0.0000	0.0019	0.2904	0.0171
Variance	explained	97.223%		R	0.9860	

Table F.30: ANOVA Table for Volatile Solids Reduction Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	5	147712.51	29542.50	3479.10	8.75	4.39
Residual	6	50.95	8.49			
(Lack of Fit)	3	43.78	14.59	6.11	29.46	9.28
(Pure Error)	3	7.17	2.39			
Total	11					

Table F.31: Correlation Matrix of Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
β_0	1.0000	0.1636	0.0000	-0.6235	-0.6662	0.0000
β_1	0.1636	1.0000	0.0000	-0.3363	-0.0705	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	-0.0567
β_{11}	-0.6235	-0.3363	0.0000	1.0000	0.2618	0.0000
β_{22}	-0.6662	-0.0705	0.0000	0.2618	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	0.0000	1.0000

Table F.32: Pure Error Variance for Volatile Solids Reduction Model

For replicate set	
mean	54.2
sum of residuals squared	7.166
degrees of freedom	3
pure error variance	2.38867

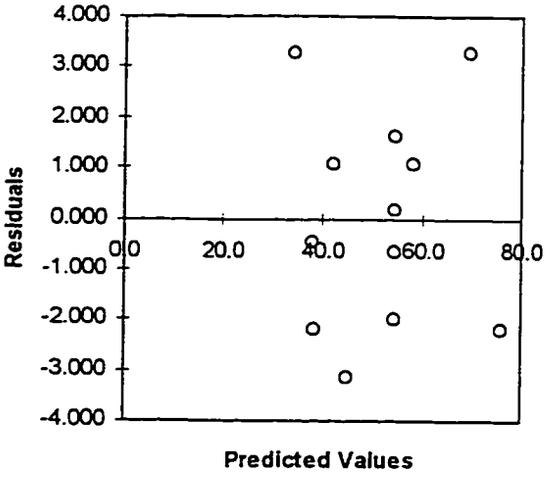

Figure F.19: Residuals Versus Predicted Values and Run Number for Volatile Solids Reduction Model

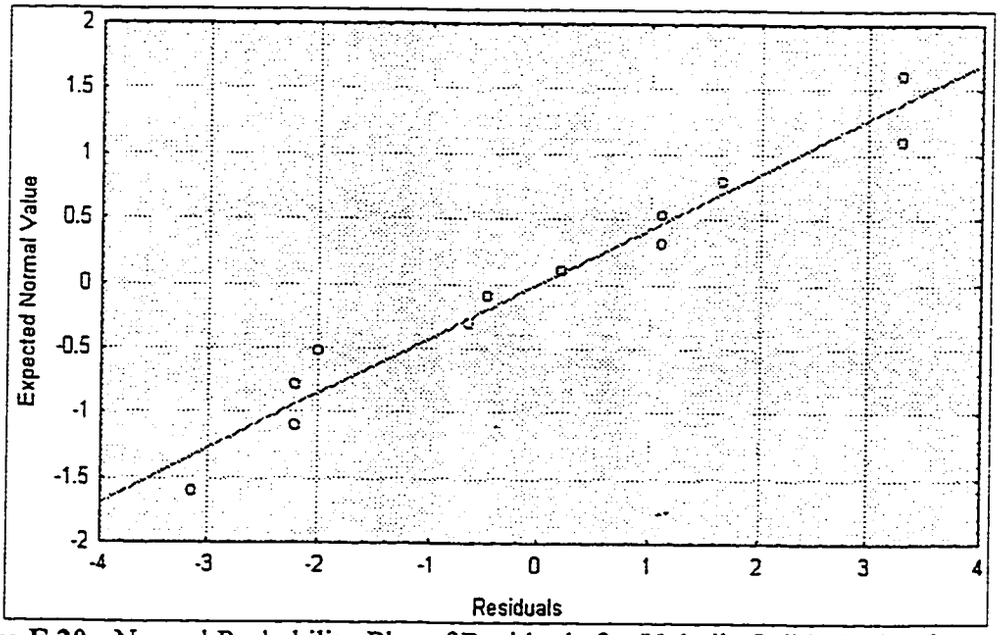

Figure F.20: Normal Probability Plot of Residuals for Volatile Solids Reduction Model

b) Final Model

Table F.33: Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_2	β_{11}	β_{12}
Estimate	55.5410	13.2340	-4.2895	-4.3725
Std.Err.	1.0224	0.9903	0.6697	1.2836
t(8)	54.3266	13.3637	-6.4051	-3.4066
p-level	0.0000	0.0000	0.0002	0.0093
Variance	explained	96.591%	R	0.9828

Table F.34: ANOVA Table for Volatile Solids Reduction Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	3	126542.29	42180.76	5394.59	7.59	4.07
Residual	8	62.55	7.82			
(Lack of Fit)	5	48.35	9.67	2.04	28.24	9.01
(Pure Error)	3	14.20	4.73			
Total	11					

Table F.35: Correlation Matrix of Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_2	β_{11}	β_{12}
β_0	1.0000	0.0000	-0.6137	0.0000
β_2	0.0000	1.0000	0.0000	-0.0567
β_{11}	-0.6137	0.0000	1.0000	0.0000
β_{12}	0.0000	-0.0567	0.0000	1.0000

Table F.36: Calculations for Pure Error Variance for Volatile Solids Reduction Model

For replicate set	
mean	54.2
sum of residuals squared	14.201
degrees of freedom	3
pure error variance	4.7337

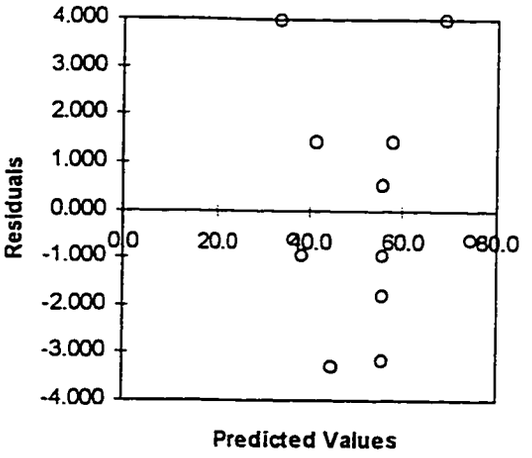

Figure F.21: Residuals Versus Predicted Values for Volatile Solids Reduction Model

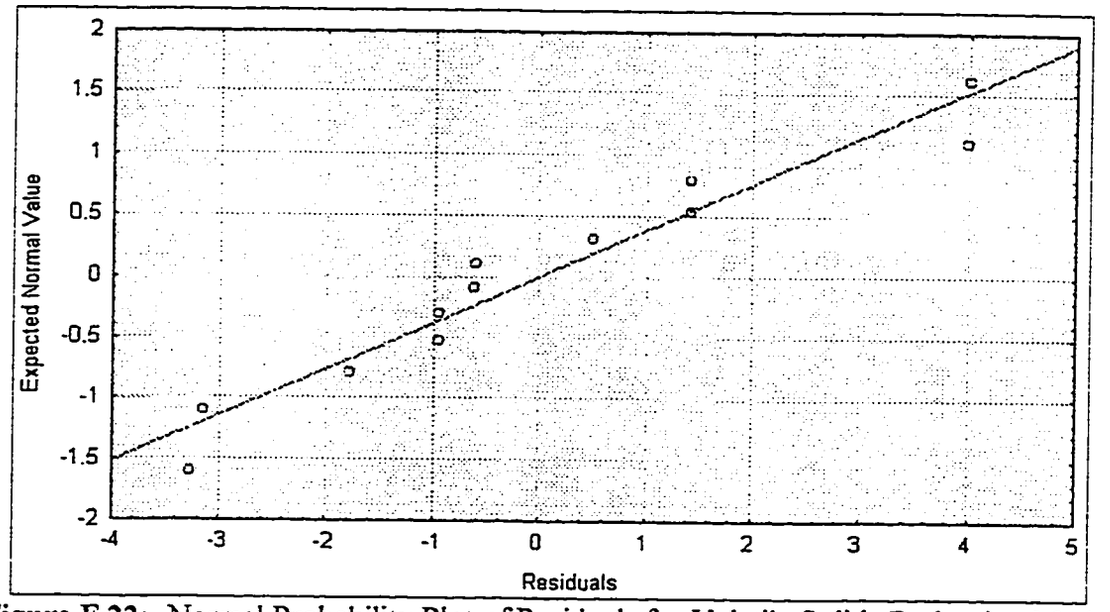

Figure F.22: Normal Probability Plot of Residuals for Volatile Solids Reduction Model

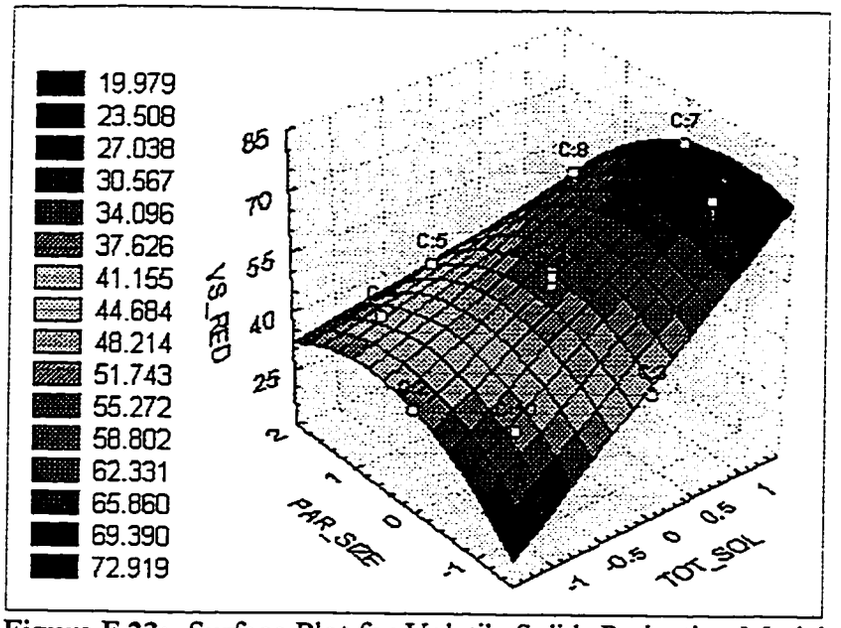

Figure F.23: Surface Plot for Volatile Solids Reduction Model

F.6 Total Solids Reduction

a) Initial Model

Table F.37: Parameter Estimates for Initial Total Solids Reduction Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	34.6251	-0.2800	9.5243	-3.0562	0.2566	-7.0416
Std.Err.	0.7851	0.5021	0.5602	0.4158	0.6350	0.7260
t(8)	44.1039	-0.5576	17.0030	-7.3498	0.4041	-9.6987
p-level	0.0000	0.5973	0.0000	0.0003	0.7001	0.0001
Variance	explained	98.643%			R	0.9932

Table F.38: ANOVA Table for Initial Total Solids Reduction Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	5	58346.23	11669.25	4664.46	8.75	4.39
Residual	6	15.01	2.50			
(Lack of Fit)	3	10.26	3.42	2.16	29.46	9.28
(Pure Error)	3	4.75	1.58			
Total	11					

Table F.39: Correlation Matrix of Parameter Estimates for Initial Total Solids Reduction Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
β_0	1.0000	0.1636	0.0000	-0.6235	-0.6662	0.0000
β_1	0.1636	1.0000	0.0000	-0.3363	-0.0705	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	-0.0567
β_{11}	-0.6235	-0.3363	0.0000	1.0000	0.2618	0.0000
β_{22}	-0.6662	-0.0705	0.0000	0.2618	1.0000	0.0000
β_{12}	0.0000	0.0000	-0.0567	0.0000	0.0000	1.0000

Table F.40: Pure Error Variance for Initial Total Solids Reduction Model

For replicate set	
mean	34.6425
sum of residuals squared	4.753
degrees of freedom	3
pure error variance	1.5843

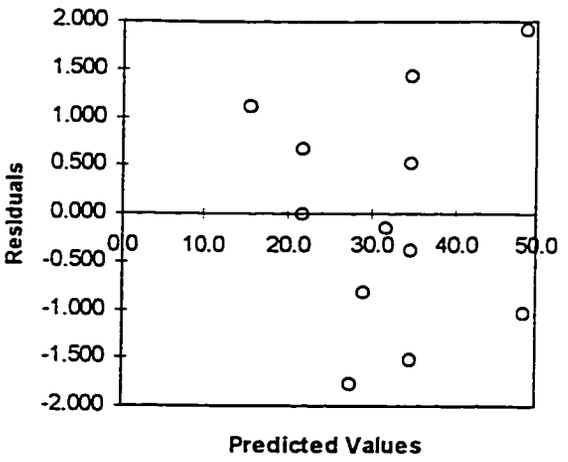

Figure F.24: Residuals versus Predicted Values for Initial Total Solids Reduction Model

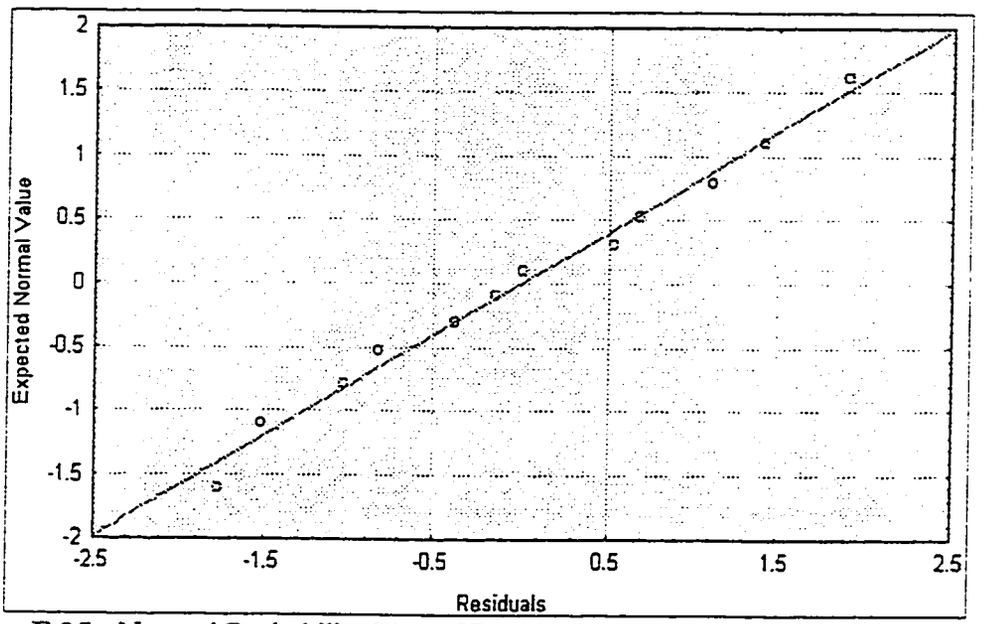

Figure F.25: Normal Probability Plot of Residuals for Initial Total Solids Reduction Model

b) Final Model

Table F.41: Parameter Estimates for Final Total Solids Reduction Model

	β_0	β_2	β_{11}	β_{12}
Estimate	34.8852	9.5243	-3.1705	-7.0416
Std.Err.	0.5190	0.5028	0.3400	0.6517
t(8)	67.2112	18.9439	-9.3250	-10.8058
p-level	0.0000	0.0000	0.0000	0.0000
Variance	explained	98.542%	R	0.9927

Table F.42: ANOVA Table for Final Total Solids Reduction Model

Source of Variation	*Degrees of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	3	55882.72	18627.57	9242.80	8.75	4.39
Residual	8	16.12	2.02			
(Lack of Fit)	5	11.14	2.23	1.34	29.46	9.28
(Pure Error)	3	4.99	1.66			
Total	11					

Table F.43: Correlation Matrix of Parameter Estimates for Final Total Solids Reduction Model

	β_0	β_2	β_{11}	β_{12}
β_0	1.0000	0.0000	-0.6137	0.0000
β_2	0.0000	1.0000	0.0000	-0.0567
β_{11}	-0.6137	0.0000	1.0000	0.0000
β_{12}	0.0000	-0.0567	0.0000	1.0000

Table F.44: Pure Error Variance for Final Total Solids Reduction Model

For replicate set	
mean	34.6425
sum of residuals squared	4.987
degrees of freedom	3
pure error variance	1.6623

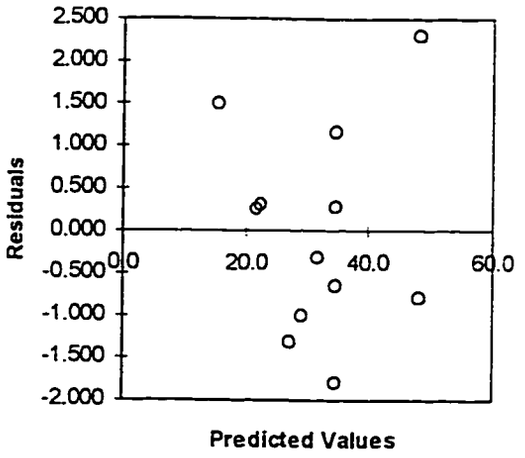

Figure F.25: Residuals Versus Predicted Values for Final Total Solids Reduction Model

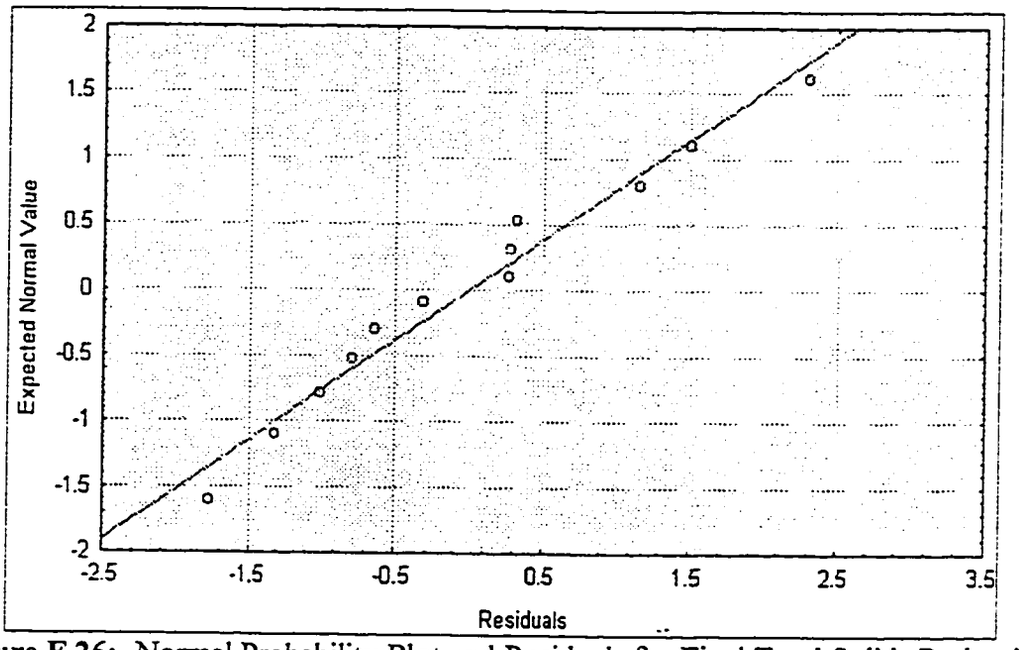

Figure F.26: Normal Probability Plot and Residuals for Final Total Solids Reduction Model

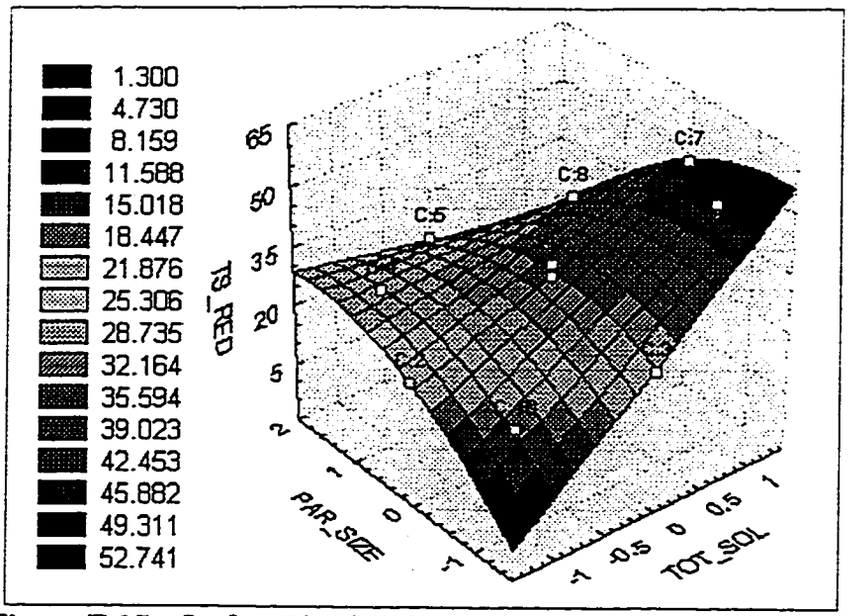

Figure E.27: Surface Plot for Final Total Solids Reduction Model

F.7 Biogas Methane Concentration

Table F.45: Parameter Estimates for Biogas Methane Concentration Model

	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Estimate	59.3471	-0.9425	0.2747	1.1850	1.9720	-1.0104
Std.Err.	1.2929	0.8269	0.9225	0.6848	1.0458	1.1957
t(8)	45.9009	-1.1398	0.2978	1.7304	1.8857	-0.8451
p-level	0.0000	0.2978	0.7759	0.1343	0.1083	0.4305
Variance	explained	51.457%			R	0.7173

Table F.46: Pure Error Variance for Biogas Methane Concentration Model

For replicate set	
mean	59.5325
sum of residuals squared	26.57275
degrees of freedom	3
pure error variance	8.85758

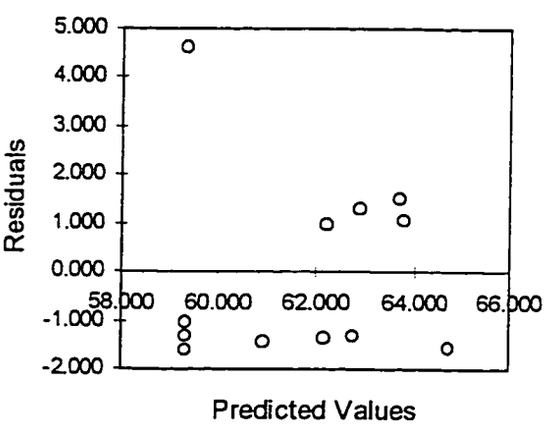

Figure F.28: Residuals Versus Predicted Values and Run Number for Biogas Methane Concentration

APPENDIX G:

Thermochemical Model Results

This appendix includes all of the data and results from the empirical models for the thermochemical pretreatment.

G.1 Cumulative Gas Production at STP

a) Initial Model

Table G.1: Parameter Estimates for Initial Cumulative Gas Production Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	2.6215	0.3826	1.0218	-0.1651	-0.0086	0.3757	-0.0069	0.1915	0.0035	-0.0220
Std.Err.	0.0614	0.0398	0.0409	0.0409	0.0349	0.0397	0.0397	0.0490	0.0490	0.0534
t(8)	42.6884	9.6166	24.9771	-4.0368	-0.2472	9.4600	-0.1749	3.9076	0.0722	-0.4123
p-level	0.0000	0.0000	0.0000	0.0024	0.8098	0.0000	0.8647	0.0029	0.9439	0.6888

Variance explained 98.85% R 0.9925

Table G.2: ANOVA Table for Initial Cumulative Gas Production Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	9	19.63	2.18	95.76	4.94	3.02
Residual	10	0.23	0.02			
(Lack of Fit)	5	0.21	0.04	9.18	10.97	5.05
(Pure Error)	5	0.02	0.00			
Total	19					

Table G.3: Correlation Matrix of Parameter Estimates for Initial Cumulative Gas Production Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0000	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0000	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0614	0.0000	0.0000
β_3	-4E-08	-4E-08	2.2E-09	1	1.3E-07	-1E-08	-4E-08	-1E-10	-0.0614	-4E-08
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	0.0000	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	1.4E-08	-4E-08	0.09104	0.09798	1	-1E-07	2.2E-08	-3E-09
β_{12}	5.2E-08	3.2E-09	-0.0614	-1E-10	-9E-09	-8E-09	-1E-07	1	1.2E-09	2.1E-08
β_{13}	-1E-07	-2E-09	-2E-08	-0.0614	1E-08	2E-07	2.2E-08	1.2E-09	1	2.5E-09
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table G.4: Pure Error Variance for Initial Cumulative Gas Production Model

For replicate set	
Mean	2.6287
sum of residuals squared	0.022
degrees of freedom	5
Pure error variance	0.0044

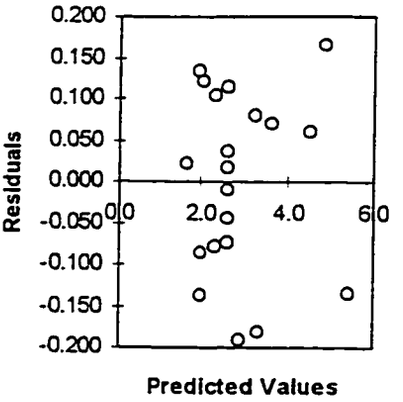

Figure G.1: Residuals Versus Predicted Values for Initial Cumulative Gas Production Model

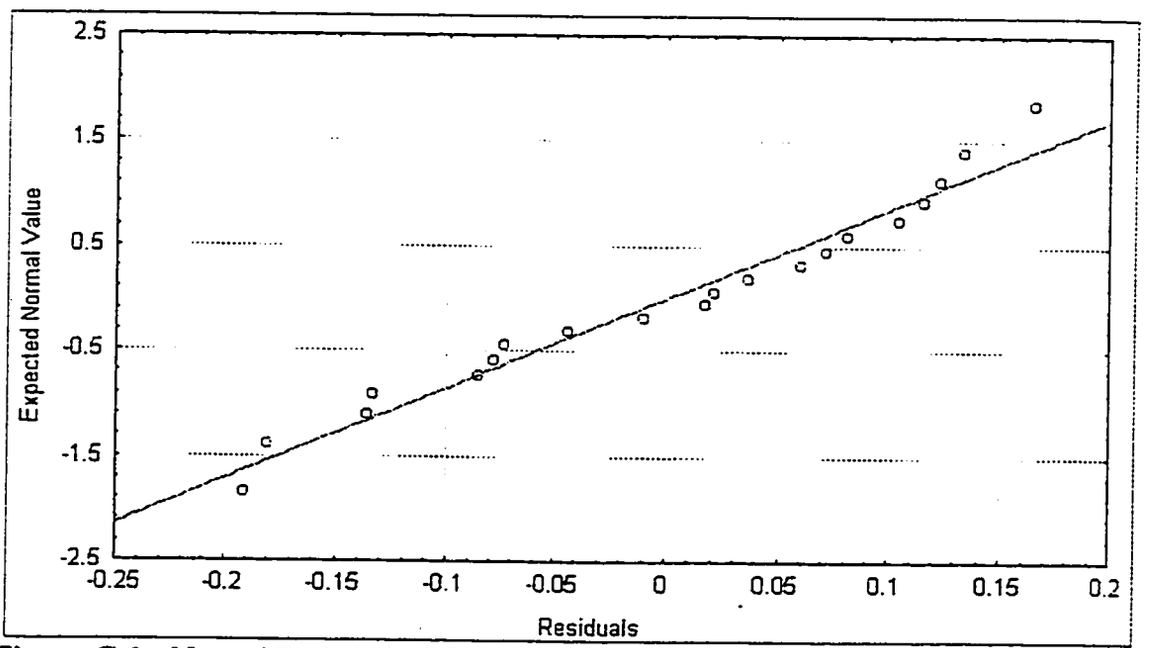

Figure G.2: Normal Probability Plot of Residuals for Initial Cumulative Gas Production Model

b) Final Model

Table G.5: Parameter Estimates for Final Cumulative Gas Production Model

	β_0	β_1	β_2	β_3	β_{22}	β_{12}
Estimate	2.6091	0.3796	1.0218	-0.1650	0.3771	0.1915
Std.Err.	0.0370	0.0324	0.0350	0.0350	0.0337	0.0419
t(8)	70.5335	11.7306	29.1768	-4.7193	11.1851	4.5647
p-level	0.0000	0.0000	0.0000	0.0003	0.0000	0.0004

Variance explained 98.82% R 0.9941

Table G.6: ANOVA Table for Final Cumulative Gas Production Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	5	19.62	3.92	235.13	4.69	2.96
Residual	14	0.23	0.02			
(Lack of Fit)	9	0.21	0.02	4.77	10.16	4.77
(Pure Error)	5	0.02	0.00			
Total	19					

Table G.7: Correlation Matrix of Parameter Estimates for Final Cumulative Gas Production Model

	β_0	β_1	β_2	β_3	β_{22}	β_{12}
β_0	1.0000	-0.0520	0.0000	0.0000	-0.6227	0.0000
β_1	-0.0520	1.0000	0.0000	0.0000	0.0045	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	-0.0614
β_3	4.4E-09	-3E-09	-1E-09	1	-7E-09	-2E-09
β_{22}	-0.6227	0.0045	0.0000	0.0000	1.0000	0.0000
β_{12}	-1E-09	-9E-11	-0.0614	-2E-09	1.5E-09	1

Table G.8: Pure Error Variance for Final Cumulative Gas Production Model

For replicate set	
Mean	2.6287
sum of residuals squared	0.024
degrees of freedom	5
Pure error variance	0.0048

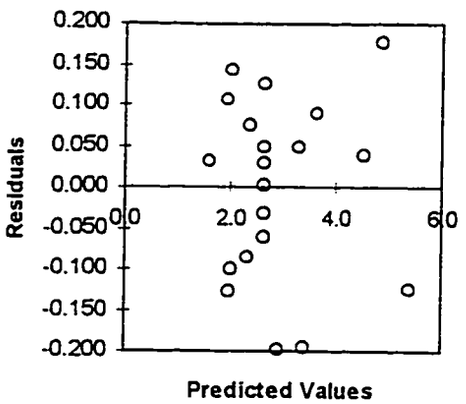

Figure G.3: Residuals Versus Predicted Values for Final Cumulative Gas Production Model

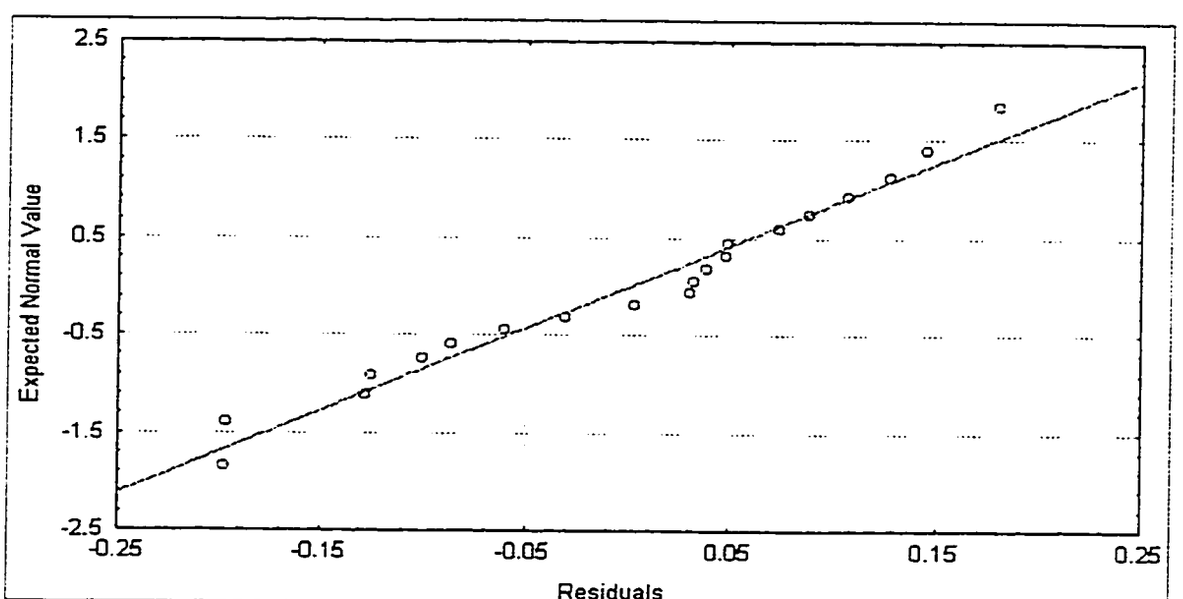

Figure G.4: Normal Probability Plot of Residuals for Final Cumulative Gas Production Model

G.2 Cumulative Gas Production at STP with Respect to Volatile Solids Addition

a) Initial Model

Table G.9: Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	0.3625	0.0454	0.0001	-0.0247	-0.0025	0.0479	-0.0017	0.0214	-0.0028	0.0067
Std.Err.	0.0130	0.0084	0.0087	0.0086	0.0074	0.0084	0.0084	0.0103	0.0103	0.0113
t(8)	27.9461	5.4012	0.0125	-2.8596	-0.3409	5.7141	-0.2062	2.0637	-0.2718	0.5984
p-level	0.0000	0.0003	0.9903	0.0170	0.7402	0.0002	0.8407	0.0660	0.7913	0.5629

Variance explained 88.60% R 0.9413

Table G.10: ANOVA Table for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{.01,v1,v2}$	$F_{.05,v1,v2}$
Regression	9	0.08	0.01	8.63	4.94	3.02
Residual	10	0.01	0.00			
(Lack of Fit)	5	0.01	0.00	8.54	10.97	5.05
(Pure Error)	5	0.00	0.00			
Total	19					

Table G.11: Correlation Matrix of Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0000	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0000	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0615	0.0000	0.0000
β_3	9.7E-08	4.7E-09	1.3E-05	1	-2E-08	8E-09	-2E-07	-7E-07	-0.0614	1.9E-07
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	0.0000	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	-4E-07	-2E-07	0.09104	0.09798	1	1.7E-08	1.3E-08	2.1E-06
β_{12}	-1E-07	-6E-08	-0.0615	-7E-07	3.1E-08	3E-07	1.7E-08	1	1.6E-06	9.1E-07
β_{13}	-6E-09	-4E-10	-4E-05	-0.0614	1.1E-09	-3E-10	1.3E-08	1.6E-06	1	-8E-07
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table G.12: Calculation for Pure Error Variance for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

For replicate set	
Mean	0.3640
sum of residuals squared	0.001
degrees of freedom	5
Pure error variance	0.0002

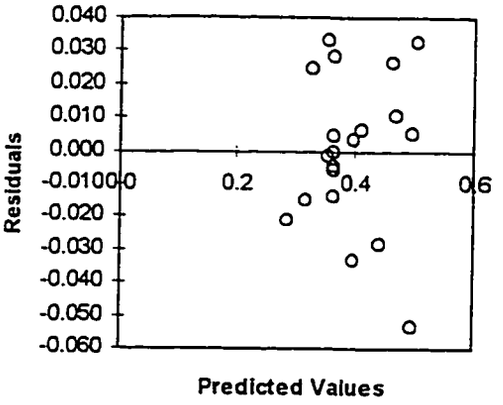

Figure G.5: Residuals versus Predicted Values for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

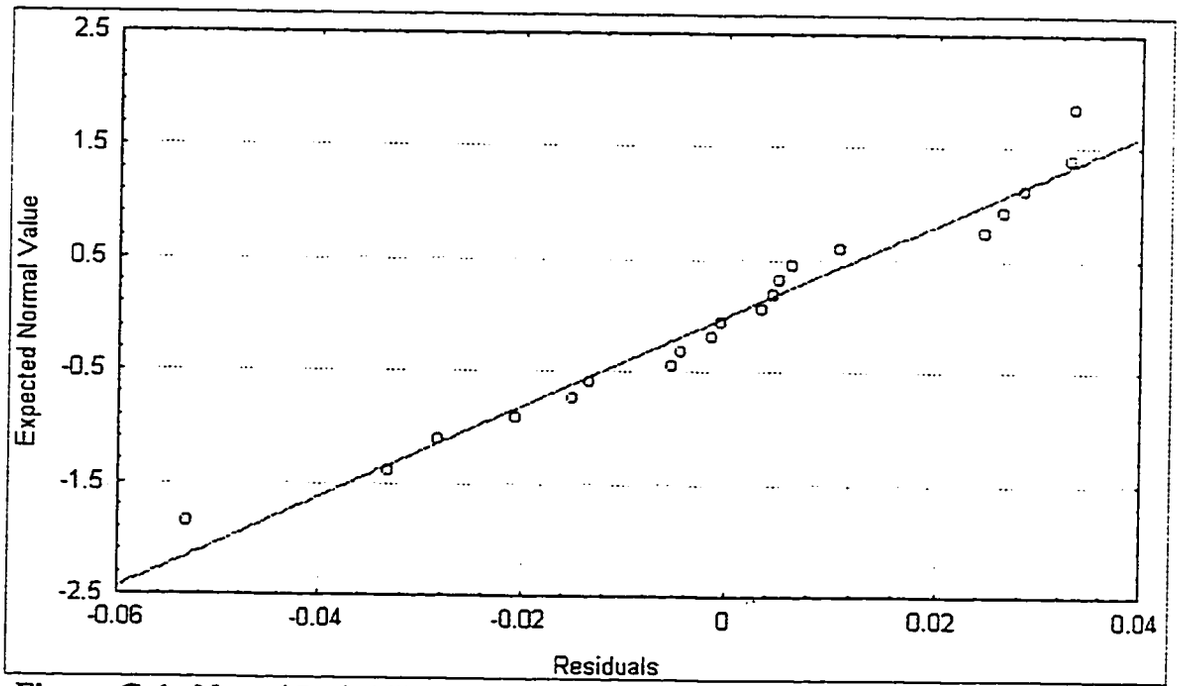

Figure G.6: Normal Probability Plot of Residuals for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

b) Final Model

Table G.13: Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_3	β_{22}	β_{12}
Estimate	0.3591	0.0445	-0.0248	0.0483	0.0214
Std.Err.	0.0077	0.0067	0.0072	0.0070	0.0087
t(8)	46.8484	6.6383	-3.4300	6.9175	2.4641
p-level	0.0000	0.0000	0.0037	0.0000	0.0263
Variance	explained	87.94%		R	0.9377

Table G.14: ANOVA Table for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	4	0.08	0.02	27.34	4.89	3.06
Residual	15	0.01	0.00			
(Lack of Fit)	10	0.01	0.00	3.99	10.05	4.74
(Pure Error)	5	0.00	0.00			
Total	19					

Table G.15: Correlation Matrix of Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_3	β_{22}	β_{12}
β_0	1.0000	-0.0520	0.0000	-0.6227	0.0000
β_1	-0.0520	1.0000	0.0000	0.0045	0.0000
β_3	1.3E-10	6.9E-09	1	-3E-10	6.7E-17
β_{22}	-0.6227	0.0045	0.0000	1.0000	0.0000
β_{12}	-5E-10	9.7E-09	6.7E-17	4.3E-11	1

Table G.16: Pure Error Variance for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

For replicate set	--
Mean	0.3640
sum of residuals squared	0.001
degrees of freedom	5
Pure error variance	0.0002

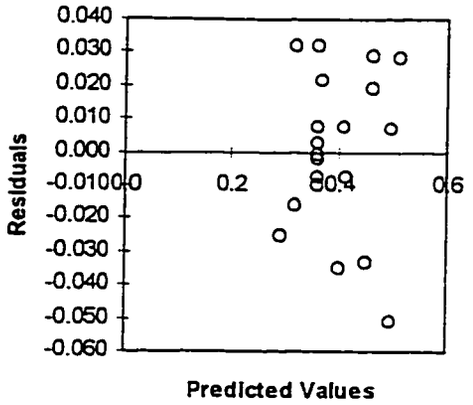

Figure G.7: Residuals Versus Predicted Values for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

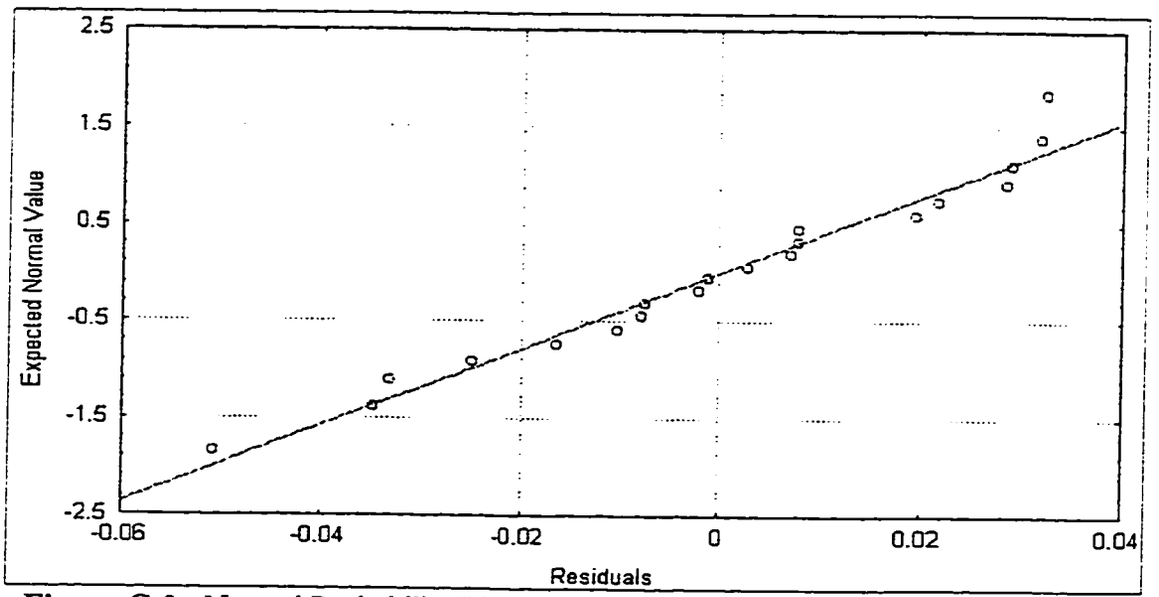

Figure G.8: Normal Probability Plot of Residuals for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

G.3 Cumulative Gas Production at STP with Respect to Volatile Solids Removal

a) Initial Model

Table G.17: Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	0.9598	0.1355	-0.1494	0.2643	0.0724	0.1870	0.1184	0.0883	0.0360	0.1313
Std.Err.	0.0752	0.0487	0.0501	0.0501	0.0428	0.0486	0.0486	0.0600	0.0600	0.0654
t(8)	12.7582	2.7802	-2.9804	5.2733	1.6911	3.8435	2.4339	1.4707	0.5994	2.0082
p-level	0.0000	0.0194	0.0138	0.0004	0.1217	0.0032	0.0352	0.1721	0.5622	0.0724

Variance explained 88.32% R 0.9398

Table G.18: ANOVA Table for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	9	2.58	0.29	8.40	4.94	3.02
Residual	10	0.34	0.03			
(Lack of Fit)	5	0.33	0.07	19.90	10.97	5.05
(Pure Error)	5	0.02	0.00			
Total	19					

Table G.19: Correlation Matrix of Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0000	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0000	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0614	0.0000	0.0000
β_3	-1E-09	1.8E-08	-2E-08	1	-6E-09	7.4E-09	2.7E-09	-3E-09	-0.0614	-1E-16
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	0.0000	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	4.6E-09	2.7E-09	0.09104	0.09798	1	-2E-08	1.2E-09	-1E-08
β_{12}	2.1E-08	-7E-09	-0.0614	-3E-09	-2E-08	-1E-08	-2E-08	1	5.8E-08	-5E-10
β_{13}	5.2E-09	-1E-09	2.7E-08	-0.0614	1.9E-09	-2E-08	1.2E-09	5.8E-08	1	3.2E-16
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table G.20: Pure Error Variance for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

For replicate set	
Mean	0.9688
sum of residuals squared	0.016
degrees of freedom	5
Pure error variance	0.0032

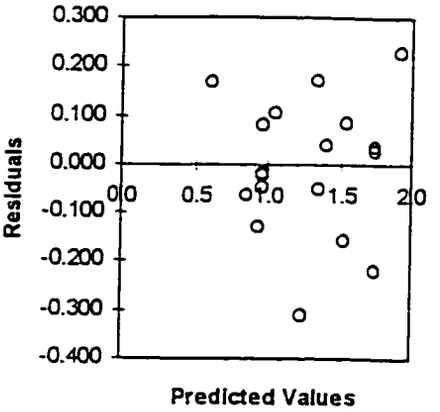

Figure G.9: Residual versus Predicted Values for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

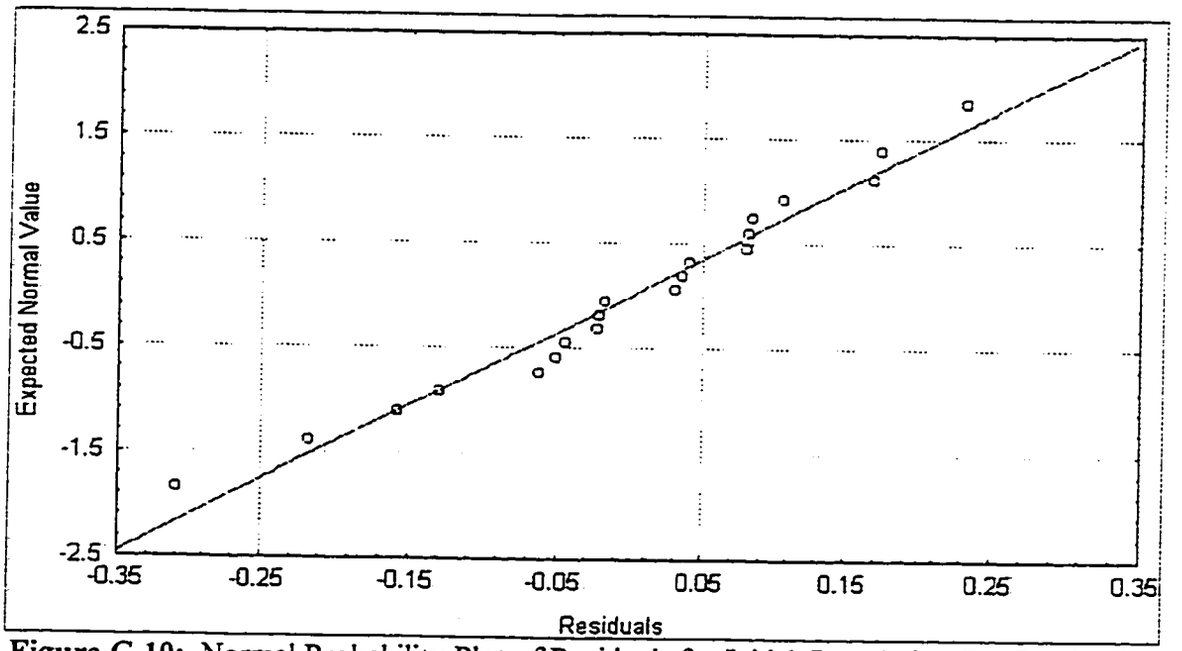

Figure G.10: Normal Probability Plot of Residuals for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

b) Final Model

Table G.21: Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_3	β_{22}
Estimate	1.1091	0.1607	-0.1448	0.2661	0.1695
Std.Err.	0.0679	0.0594	0.0641	0.0641	0.0619
t(8)	16.3427	2.7073	-2.2585	4.1495	2.7395
p-level	0.0000	0.0162	0.0392	0.0009	0.0152
Variance	explained	71.20%		R	0.8438

Table G.22: ANOVA Table for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	4	2.08	0.52	9.27	4.69	2.96
Residual	15	0.84	0.06			
(Lack of Fit)	10	0.71	0.07	2.65	10.16	4.77
(Pure Error)	5	0.13	0.03			
Total	19					

Table G.23: Correlation Matrix of Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_3	β_{22}	β_{33}
β_0	1.0000	-0.0457	0.0000	0.0000	-0.5633	-0.5633
β_1	-0.0457	1.0000	0.0000	0.0000	0.0049	0.0049
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000
β_3	2.8E-08	-9E-11	3E-15	1	-3E-09	-5E-08
β_{22}	-0.5633	0.0049	0.0000	0.0000	1.0000	0.0904
β_{33}	-0.5633	0.00489	-6E-08	-5E-08	0.09044	1

Table G.24: Pure Error Variance for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

For replicate set	
Mean	0.9688
sum of residuals squared	0.036
degrees of freedom	5
Pure error variance	0.0072

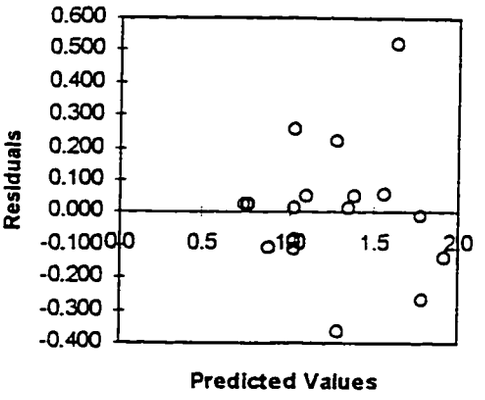

Figure G.11: Residuals Versus Predicted Values for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

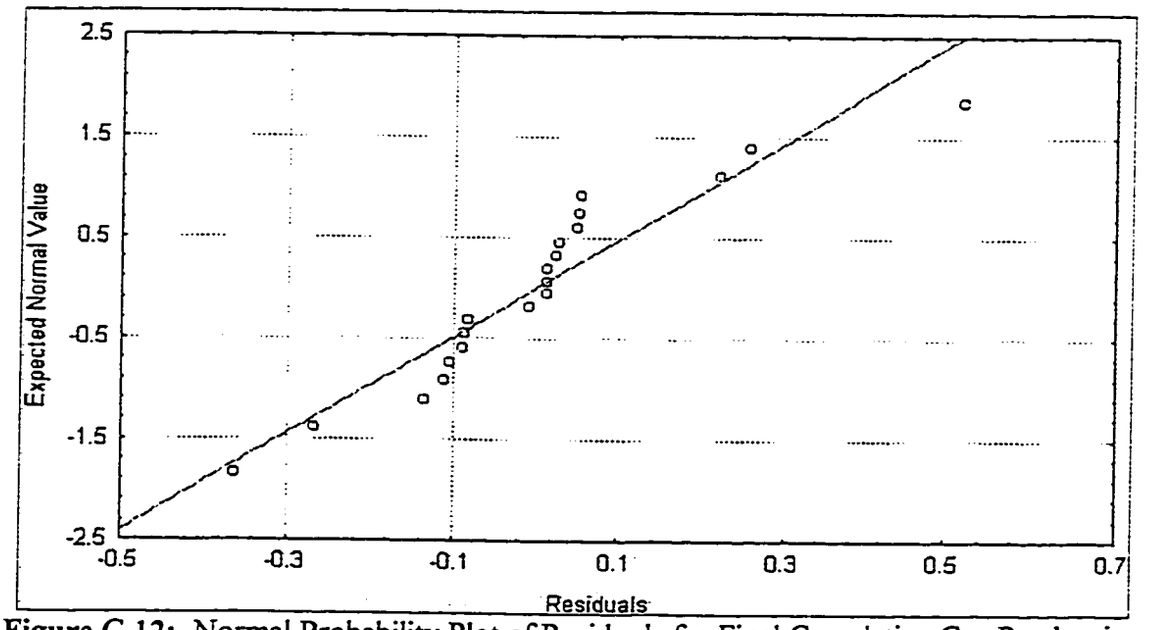

Figure G.12: Normal Probability Plot of Residuals for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

G.4 Chemical Oxygen Demand

a) Initial Model

Table G.25: Parameter Estimates for Initial Chemical Oxygen Demand Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	53.7703	2.2369	3.9303	-6.8201	-2.9205	-2.4461	-3.3382	-1.9907	-2.8494	-4.5587
Std.Err.	2.0524	1.3297	1.3673	1.3673	1.1675	1.3273	1.3273	1.6375	1.6375	1.7831
t(8)	26.1983	1.6822	2.8746	-4.9881	-2.5015	-1.8429	-2.5151	-1.2157	-1.7401	-2.5566
p-level	0.0000	0.1234	0.0165	0.0005	0.0314	0.0951	0.0306	0.2520	0.1125	0.0285

Variance explained 85.60% R 0.9252

Table G.26: ANOVA Table for Initial Chemical Oxygen Demand Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	9	1512.26	168.03	6.61	4.94	3.02
Residual	10	254.37	25.44			
(Lack of Fit)	5	166.30	33.26	1.89	10.97	5.05
(Pure Error)	5	88.07	17.61			
Total	19					

Table G.27: Correlation Matrix of Parameter Estimates for Initial Chemical Oxygen Demand Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0000	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0000	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0614	0.0000	0.0000
β_3	-1E-09	-7E-10	-4E-10	1	1.4E-09	7.1E-10	3.2E-10	-2E-10	-0.0614	2E-10
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	0.0000	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	-7E-10	3.2E-10	0.09104	0.09798	1	1.5E-09	2.6E-09	7.5E-10
β_{12}	-3E-09	-1E-09	-0.0614	-2E-10	3.4E-09	1.9E-09	1.5E-09	1	4.5E-09	2E-09
β_{13}	-3E-09	-2E-09	-9E-10	-0.0614	2.2E-09	2.4E-09	2.6E-09	4.5E-09	1	6.9E-10
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table G.28: Pure Error Variance for Initial Chemical Oxygen Demand Model

For replicate set	
Mean	53.5450
sum of residuals squared	88.072
degrees of freedom	5
Pure error variance	17.6144

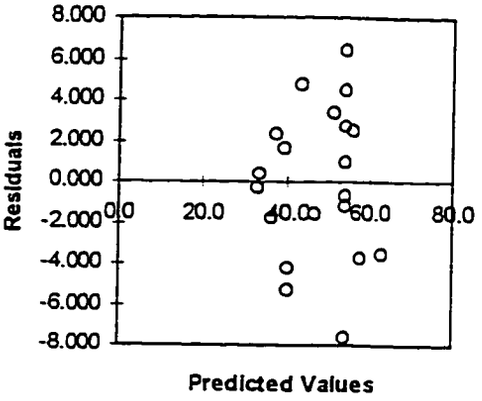

Figure G.13: Residuals versus Predicted Values for Initial Chemical Oxygen Demand Model

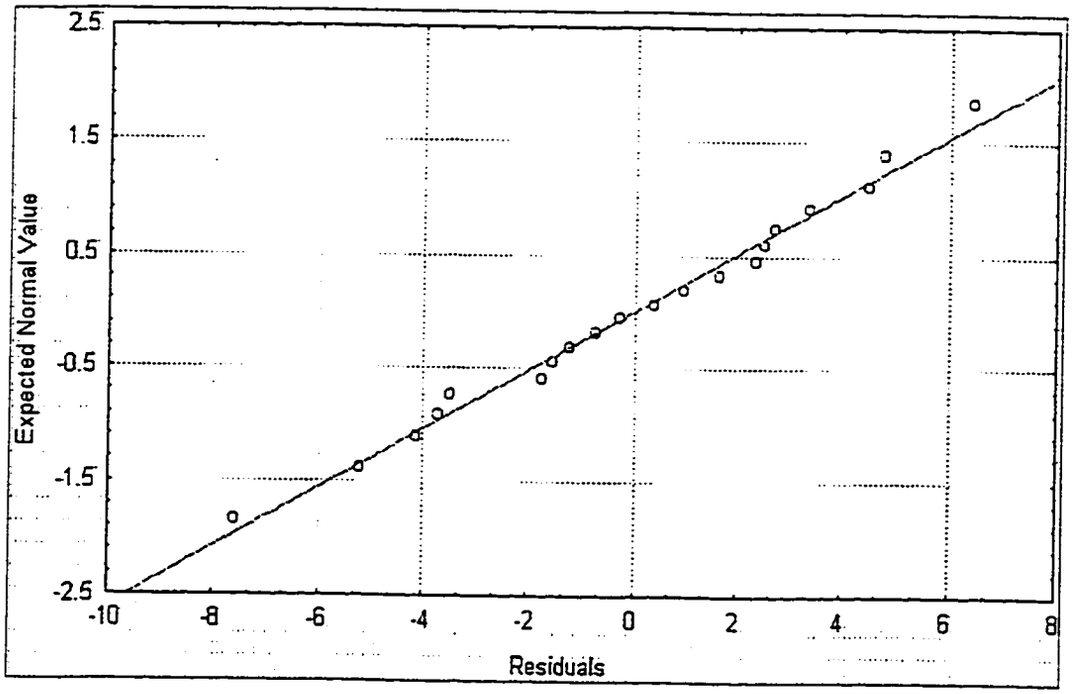

Figure G.14: Normal Probability Plot of Residuals for Initial Chemical Oxygen Demand Model

b) Final Model

Table G.29: Parameter Estimates for Final Chemical Oxygen Demand Model

	β_0	β_3
Estimate	47.6091	-6.9661
Std.Err.	1.7511	2.1189
t(8)	27.1887	-3.2875
p-level	0.0000	0.0041
	Variance explained	37.52% R 0.8625

Table G.30: ANOVA Table for Final Chemical Oxygen Demand Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	1	662.79	662.79	10.81	8.29	4.41
Residual	18	1103.84	61.32			
(Lack of Fit)	13	804.66	61.90	1.03	9.83	4.66
(Pure Error)	5	299.18	59.84			
Total	19					

Table G.31: Correlation Matrix of Parameter Estimates for Final Chemical Oxygen Demand Model

	β_0	β_3
β_0	1.0000	0.0000
β_3	0	1

Table G.32: Pure Error Variance for Final Chemical Oxygen Demand Model

For replicate set	
Mean	53.5450
sum of residuals squared	299.176
degrees of freedom	5
Pure error variance	59.8352

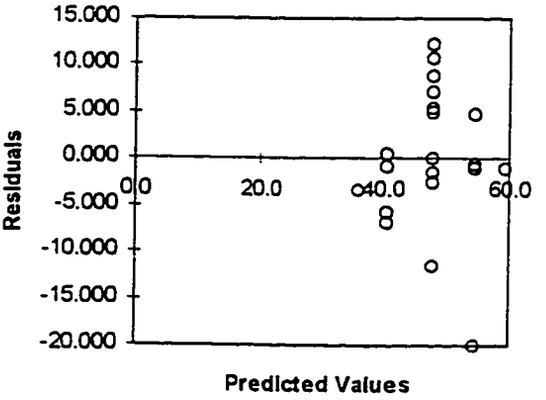

Figure G.15: Residuals Versus Predicted Values for Final Chemical Oxygen Demand Model

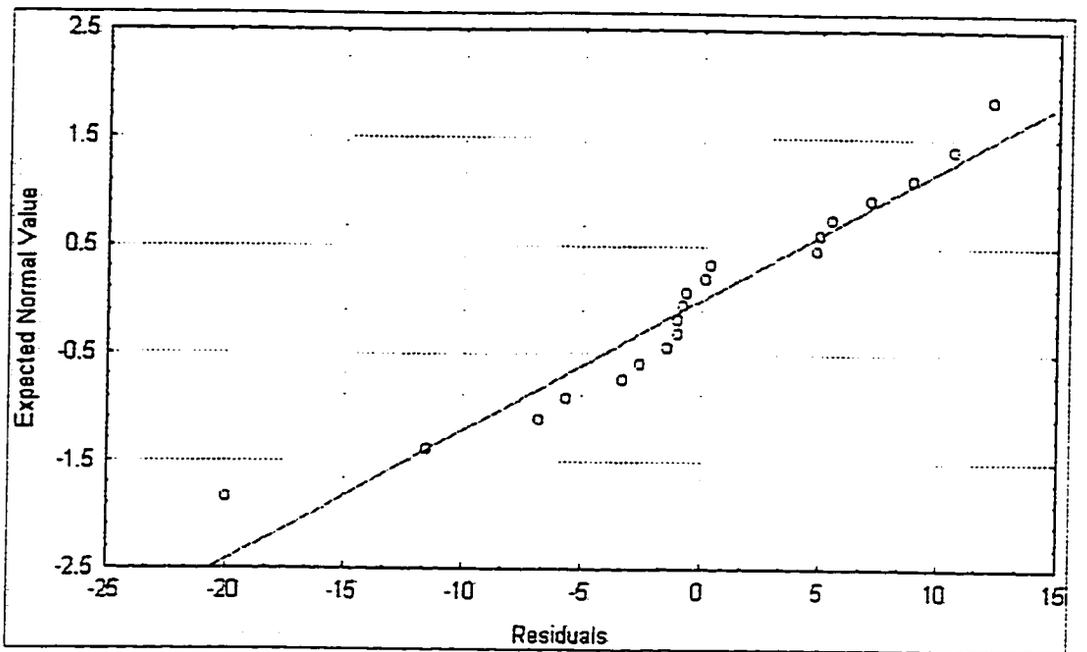

Figure G.16: Normal Probability Plot of Residuals for Final Chemical Oxygen Demand Model

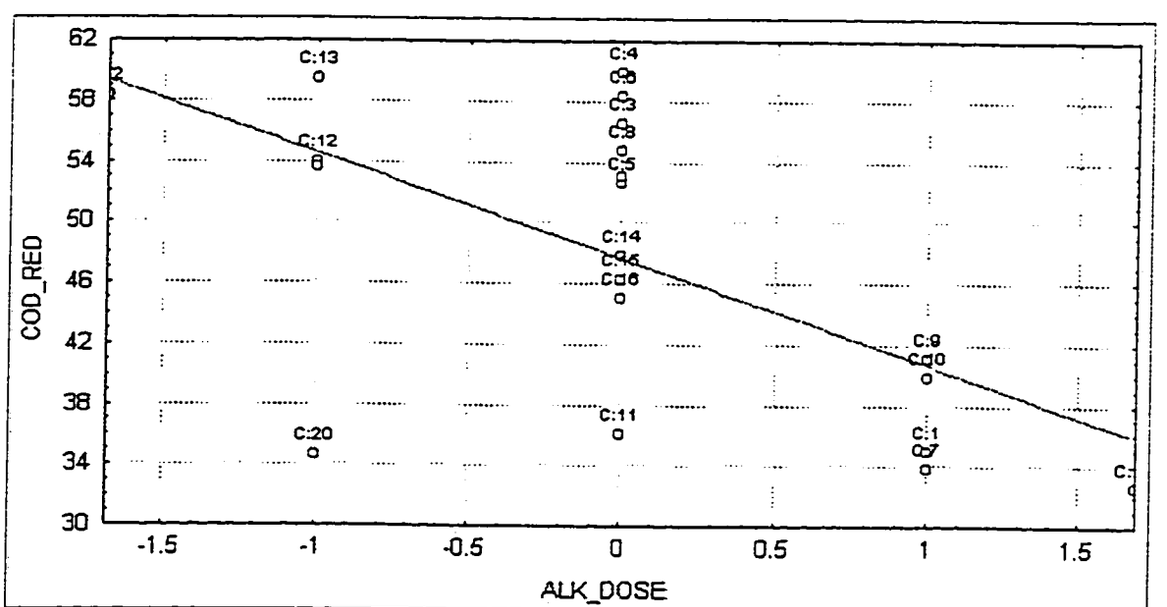

Figure G.17: Parabolic Plot for Final Chemical Oxygen Demand Model

G.5 Volatile Solids Reduction

a) Initial Model

Table G.33: Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	24.4896	-0.3546	3.9688	-5.4538	-1.6229	-0.0814	-2.0664	-1.3320	0.6057	-3.0875
Std.Err.	0.7026	0.4552	0.4680	0.4680	0.3996	0.4543	0.4543	0.5605	0.5605	0.6104
t(8)	34.8571	-0.7790	8.4798	-11.6527	-4.0608	-0.1791	-4.5482	-2.3763	1.0806	-5.0582
p-level	0.0000	0.4540	0.0000	0.0000	0.0023	0.8615	0.0011	0.0389	0.3052	0.0005

Variance explained 96.50% R 0.9824

Table G.34: ANOVA Table for Volatile Solids Reduction Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{.01,v1,v2}$	$F_{.05,v1,v2}$
Regression	9	822.22	91.36	30.65	4.94	3.02
Residual	10	29.81	2.98			
(Lack of Fit)	5	20.75	4.15	2.29	10.97	5.05
(Pure Error)	5	9.06	1.81			
Total	19					

Table G.35: Correlation Matrix of Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0000	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0000	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0614	0.0000	0.0000
β_3	-2E-09	-4E-09	-2E-10	1	1.6E-09	2.9E-09	-1E-10	-5E-10	-0.0614	1E-10
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	0.0000	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	-2E-09	-9.9E-11	0.09104	0.09798	1	2E-09	1.2E-08	3.6E-09
β_{12}	-1E-08	9.4E-09	-0.0614	-5.3E-10	-1E-09	2.2E-08	2E-09	1	3.4E-09	3.9E-11
β_{13}	-5E-08	-2E-08	1.8E-09	-0.06138	1.7E-08	9.3E-08	1.2E-08	3.4E-09	1	-2E-09
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table G.36: Pure Error Variance for Volatile Solids Reduction Model

For replicate set	
Mean	24.5170
sum of residuals squared	9.059
degrees of freedom	5
Pure error variance	1.8118

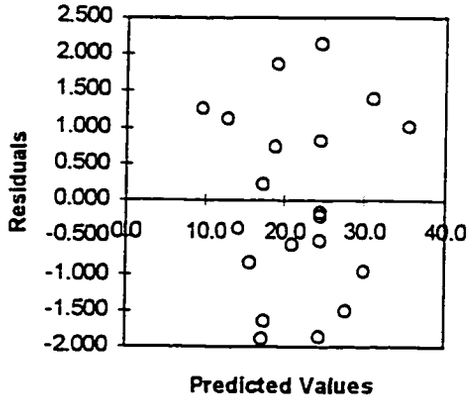

Figure G.18: Residuals Versus Predicted Values for Volatile Solids Reduction Model

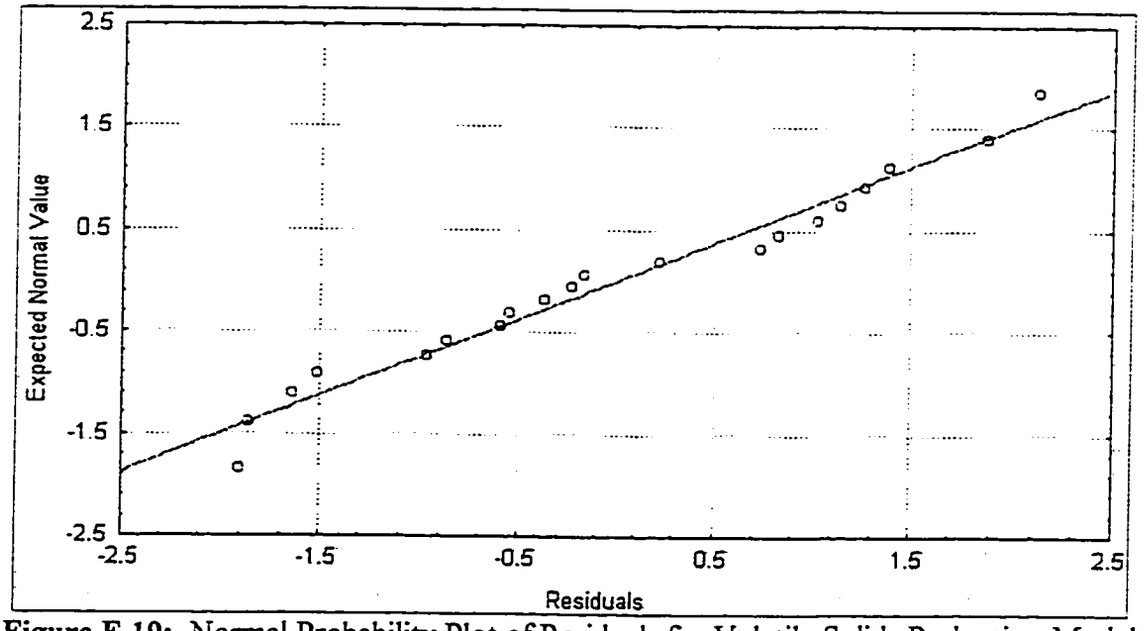

Figure E.19: Normal Probability Plot of Residuals for Volatile Solids Reduction Model

b) Final Model

Table G.37: Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_2	β_3	β_{11}	β_{33}	β_{12}	β_{23}
Estimate	24.4864	3.9688	-5.4228	-1.7135	-2.0661	-1.3320	-3.0875
Std.Err.	0.5647	0.4462	0.4453	0.3605	0.4309	0.5343	0.5819
t(8)	43.3633	8.8953	-12.1772	-4.7526	-4.7946	-2.4928	-5.3061
p-level	0.0000	0.0000	0.0000	0.0004	0.0004	0.0270	0.0001

Variance explained 95.87% R 0.9791

Table G.38: ANOVA Table for Volatile Solids Reduction Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{.01,v1,v2}$	$F_{.05,v1,v2}$
Regression	6	816.82	136.14	50.26	4.62	2.92
Residual	13	35.21	2.71			
(Lack of Fit)	8	26.15	3.27	1.80	10.29	4.82
(Pure Error)	5	9.06	1.81			
Total	19					

Table G.39: Correlation Matrix of Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_2	β_3	β_{11}	β_{33}	β_{12}	β_{23}
β_0	1.0000	0.0000	0.0000	-0.5526	-0.5621	0.0000	0.0000
β_2	0.0000	1.0000	0.0000	0.0000	0.0000	-0.0614	0.0000
β_3	-3E-10	-4E-11	1	1.1E-10	4.2E-10	6.5E-10	3.1E-10
β_{11}	-0.5526	0.0000	0.0000	1.0000	0.0801	0.0000	0.0000
β_{33}	-0.5621	-2E-09	4.23E-10	0.08014	1	3.8E-09	5.6E-09
β_{12}	-3E-09	-0.0614	6.52E-10	2.3E-09	3.8E-09	1	4.5E-09
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table G.40: Calculations for Pure Error Variance for Volatile Solids Reduction Model

For replicate set	
Mean	24.5170
sum of residuals squared	9.060
degrees of freedom	5
Pure error variance	1.8120

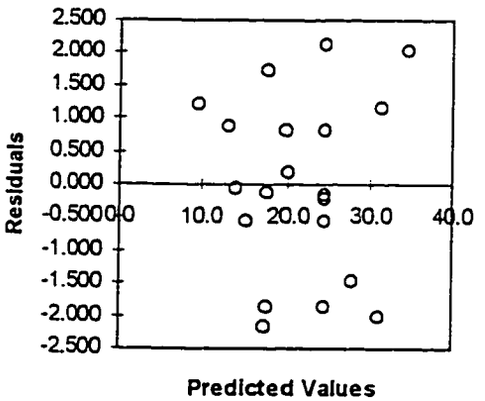

Figure G.20: Residuals Versus Predicted Values and Run Number for Volatile Solids Reduction Model

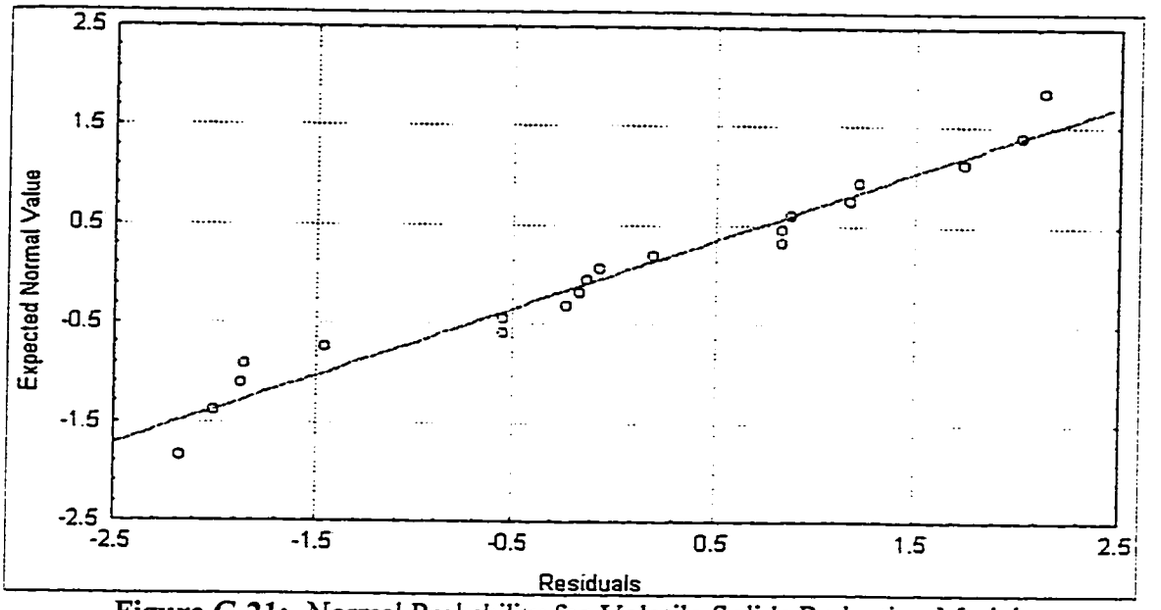

Figure G.21: Normal Probability for Volatile Solids Reduction Model

G.6 Total Solids Reduction

a) Initial Model

Table G.41: Parameter Estimates for Initial Total Solids Reduction Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	37.7471	0.1834	4.8760	-8.5853	-2.2830	-0.3198	-2.1306	-0.7113	0.2611	-3.6923
Std.Err.	0.9253	0.5995	0.6164	0.6164	0.5263	0.5984	0.5984	0.7382	0.7382	0.8039
t(8)	40.7942	0.3059	7.9104	-13.9279	-4.3375	-0.5344	-3.5606	-0.9636	0.3537	-4.5930
p-level	0.0000	0.7660	0.0000	0.0000	0.0015	0.6048	0.0052	0.3580	0.7309	0.0010

Variance explained 96.86% R 0.9842

Table G.42: ANOVA Table for Initial Total Solids Reduction Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{.01,v1,v2}$	$F_{.05,v1,v2}$
Regression	9	1593.73	177.08	34.25	4.94	3.02
Residual	10	51.70	5.17			
(Lack of Fit)	5	33.45	6.69	1.83	10.97	5.05
(Pure Error)	5	18.25	3.65			
Total	19					

Table G.43: Correlation Matrix of Parameter Estimates for Initial Total Solids Reduction Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0000	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0000	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0614	0.0000	0.0000
β_3	5.4E-09	7E-09	6.6E-10	1	-4E-09	-7E-09	-2E-09	-4E-09	-0.0614	1.7E-09
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	0.0000	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	1.4E-09	-2E-09	0.09104	0.09798	1	-2E-08	3.2E-08	3.9E-10
β_{12}	3.8E-08	6.9E-08	-0.0614	-4E-09	-3E-08	-4E-08	-2E-08	1	5.9E-08	4.5E-09
β_{13}	-9E-08	-2E-07	-1E-08	-0.0614	8.2E-08	1.1E-07	3.2E-08	5.9E-08	1	-1E-08
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table G.44: Pure Error Variance for Initial Total Solids Reduction Model

For replicate set	
Mean	37.6545
sum of residuals squared	18.246
degrees of freedom	5
Pure error variance	3.6492

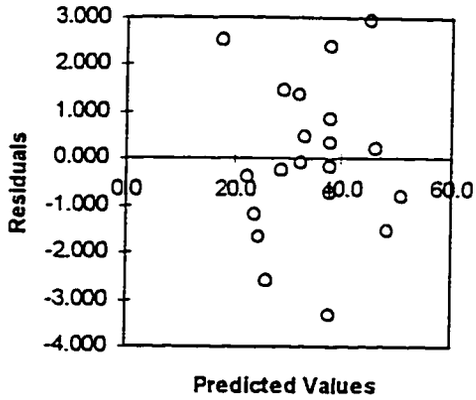

Figure G.22: Residuals versus Predicted Values for Initial Total Solids Reduction Model

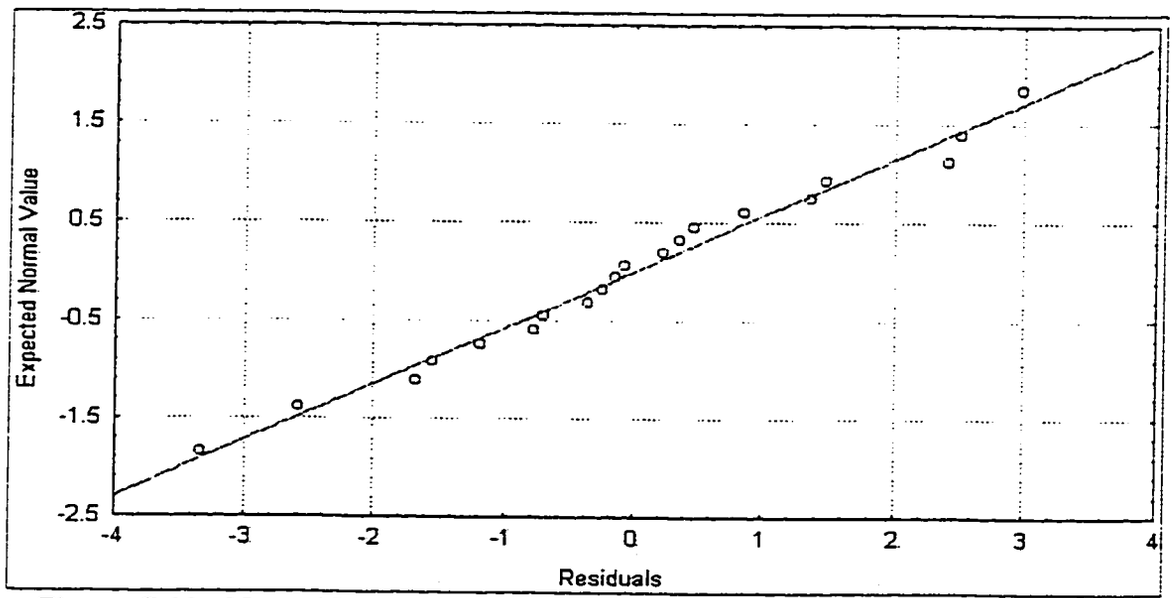

Figure G.23: Normal Probability Plot of Residuals for Initial Total Solids Reduction Model

b) Final Model

Table G.45: Parameter Estimates for Final Total Solids Reduction Model

	β_0	β_2	β_3	β_{11}	β_{33}	β_{23}
Estimate	37.4563	4.8396	-8.5719	-2.2095	-2.0955	-3.6923
Std.Err.	0.7048	0.5558	0.5558	0.4500	0.5378	0.7262
t(8)	53.1477	8.7075	-15.4228	-4.9104	-3.8962	-5.0842
p-level	0.0000	0.0000	0.0000	0.0002	0.0016	0.0002
Variance explained		96.41%			R	0.9819

Table G.46: ANOVA Table for Final Total Solids Reduction Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	5	1586.36	317.27	75.20	4.69	2.96
Residual	14	59.07	4.22			
(Lack of Fit)	9	40.64	4.52	1.22	10.16	4.77
(Pure Error)	5	18.43	3.69			
Total	19					

Table G.47: Correlation Matrix of Parameter Estimates for Final Total Solids Reduction Model

	β_0	β_2	β_3	β_{11}	β_{33}	β_{23}
β_0	1.0000	0.0000	0.0000	-0.5526	-0.5621	0.0000
β_2	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000
β_3	-6E-10	-3E-10	1	5E-10	6.1E-10	-4E-10
β_{11}	-0.5526	0.0000	0.0000	1.0000	0.0801	0.0000
β_{33}	-0.5621	2.7E-09	6.14E-10	0.08014	1	-4E-09
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table G.48: Pure Error Variance for Final Total Solids Reduction Model

For replicate set	
Mean	37.6545
sum of residuals squared	18.431
degrees of freedom	5
Pure error variance	3.6862

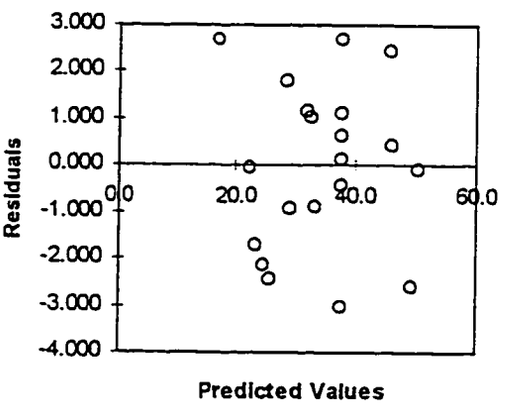

Figure G.24: Residuals Versus Predicted Values for Final Total Solids Reduction Model

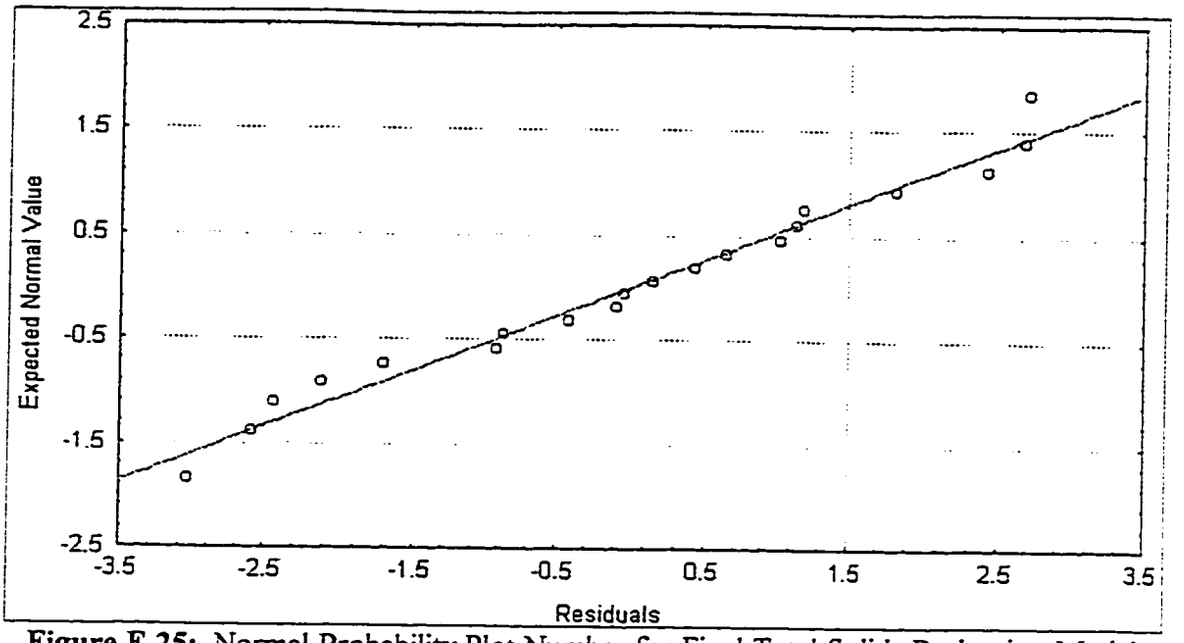

Figure E.25: Normal Probability Plot Number for Final Total Solids Reduction Model

G.7 Methane Concentration

a) Initial Model

Table G.49: Parameter Estimates for Methane Concentration Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	59.0042	1.0749	-0.9979	0.6912	0.0111	-0.4952	0.2418	0.2900	0.1650	-1.0538
Std.Err.	0.7368	0.4774	0.4908	0.4908	0.4192	0.4765	0.4765	0.5878	0.5878	0.6401
t(8)	80.0786	2.2517	-2.0331	1.4081	0.0264	-1.0393	0.5074	0.4933	0.2807	-1.6462
p-level	0.0000	0.0480	0.0694	0.1894	0.9795	0.3231	0.6229	0.6325	0.7847	0.1308

Variance explained 61.87% R 0.7866

Table G.50: ANOVA Table for Methane Concentration Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	9	53.20	5.91	1.80	4.94	3.02
Residual	10	32.78	3.28			
(Lack of Fit)	5	18.73	3.75	1.33	10.97	5.05
(Pure Error)	5	14.05	2.81			
Total	19					

Table G.51: Correlation Matrix of Parameter Estimates for Methane Concentration Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0000	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0000	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0614	0.0000	0.0000
β_3	-8E-07	-5E-07	-1E-08	1	1.5E-06	1E-07	1.5E-07	7E-08	-0.0614	-1E-08
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	0.0000	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	8.8E-08	1.5E-07	0.09104	0.09798	1	-1E-08	-6E-07	1.6E-07
β_{12}	4.4E-08	3.2E-08	-0.0614	7E-08	-4E-08	-5E-08	-1E-08	1	-4E-09	-3E-08
β_{13}	1.9E-06	1.1E-06	7.8E-10	-0.0614	-3E-06	-3E-07	-6E-07	-4E-09	1	5.3E-08
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table G.52: Pure Error Variance for Methane Concentration Model

For replicate set	
Mean	59.0683
sum of residuals squared	14.052
degrees of freedom	5
Pure error variance	2.8104

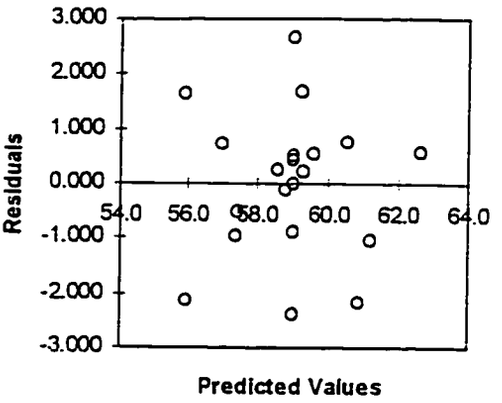

Figure G.26: Residuals versus Predicted Values for Initial Methane Concentration Model

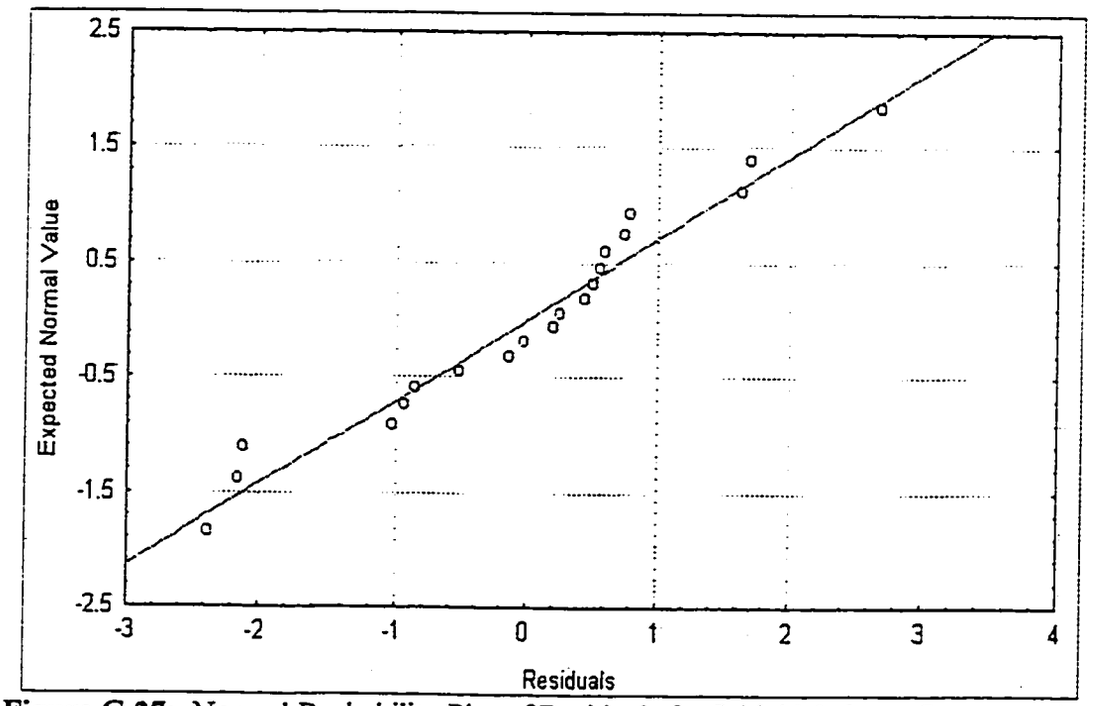

Figure G.27: Normal Probability Plot of Residuals for Initial Methane Concentration Model

b) Final Model

Table G.53: Parameter Estimates for Final Methane Concentration Model

	β_0	β_1
Estimate	58.8398	1.0799
Std.Err.	0.4335	0.4846
t(8)	135.7239	2.2283
p-level	0.0000	0.0389
Variance explained	21.62%	R 0.4650

Table G.54: ANOVA Table for Final Methane Concentration Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{.01,v1,v2}$	$F_{.05,v1,v2}$
Regression	1	18.59	18.59	4.97	8.29	4.41
Residual	18	67.39	3.74			
(Lack of Fit)	13	53.05	4.08	1.42	9.83	4.64
(Pure Error)	5	14.34	2.87			
Total	19					

Table G.55: Correlation Matrix of Parameter Estimates for Final Methane Concentration Model

	β_0	β_1
β_0	1.0000	-0.0629
β_1	-0.0629	1.0000

Table G.56: Pure Error Variance for Final Methane Concentration Model

For replicate set	
Mean	59.0683
sum of residuals squared	14.341
degrees of freedom	5
Pure error variance	2.8682

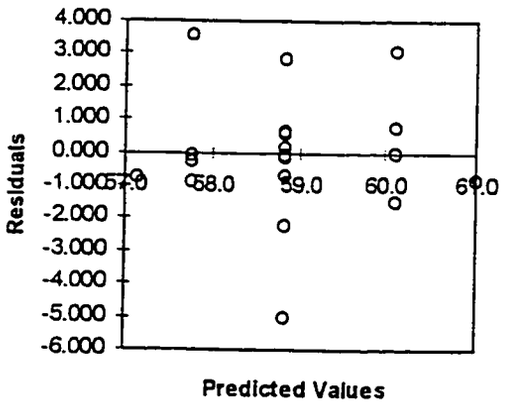

Figure G.28: Residuals Versus Predicted Values for Final Methane Concentration Model

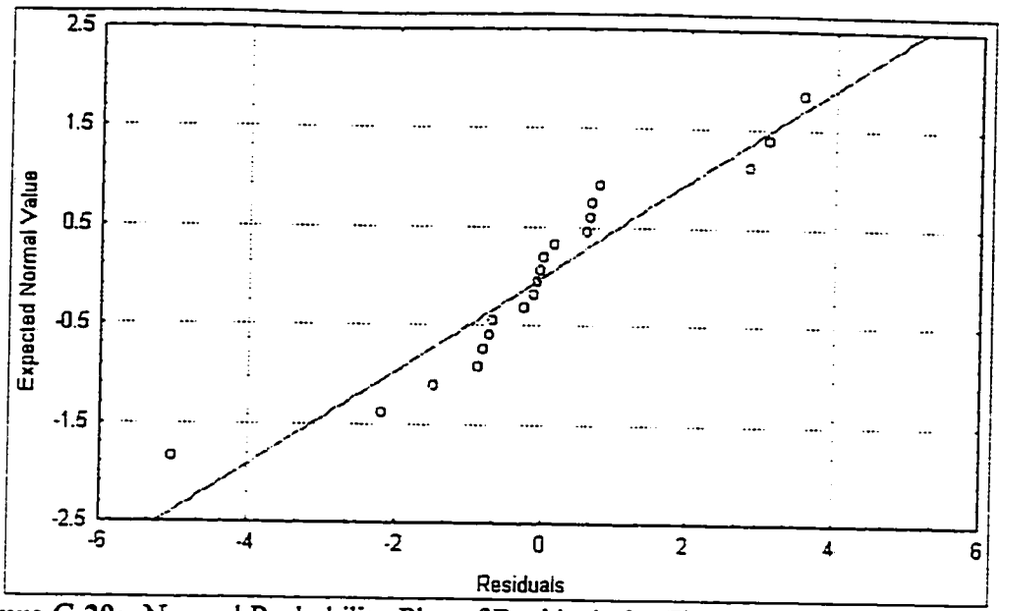

Figure G.29: Normal Probability Plot of Residuals for Final Methane Concentration Model

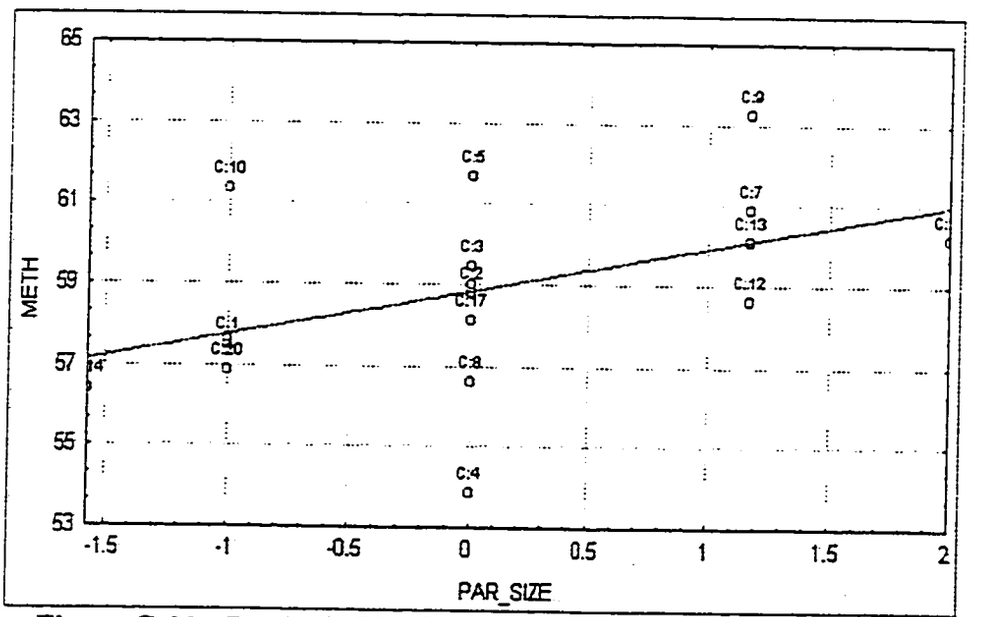

Figure G.30: Parabolic Plot for Final Methane Concentration Model

APPENDIX H:
Alkaline Model Results

This Appendix includes all data and results for the empirical models for the alkaline pretreatment

H.1 Cumulative Gas Production at STP

a) Initial Model

Table H.1: Parameter Estimates for Initial Cumulative Gas Production Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	3.9510	0.5385	1.3456	0.1634	-0.1419	0.3470	0.0920	0.4559	-0.0020	0.0175
Std.Err.	0.1090	0.0706	0.0726	0.0726	0.0620	0.0705	0.0705	0.0870	0.0870	0.0947
t(8)	36.2468	7.6249	18.5308	2.2506	-2.2890	4.9231	1.3050	5.2420	-0.0227	0.1845
p-level	0.0000	0.0000	0.0000	0.0481	0.0451	0.0006	0.2211	0.0004	0.9823	0.8573

Variance explained 97.94% R 0.9896

Table H.2: ANOVA Table for Initial Cumulative Gas Production Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	9	34.06	3.78	52.75	4.94	3.02
Residual	10	0.72	0.07			
(Lack of Fit)	5	0.63	0.13	7.43	10.97	5.05
(Pure Error)	5	0.09	0.02			
Total	19					

Table H.3: Correlation Matrix of Parameter Estimates for Initial Cumulative Gas Production Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0000	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0000	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0614	0.0000	0.0000
β_3	4.1E-07	3.9E-08	-7E-09	1	-2E-07	-2E-07	-6E-07	-8E-08	-0.0614	-1E-06
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	0.0000	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	-4E-08	-6E-07	0.09104	0.09798	1	-9E-08	3.2E-06	-3E-07
β_{12}	5.9E-08	-1E-09	-0.0614	-8E-08	-3E-09	-4E-08	-9E-08	1	7.6E-07	-9E-08
β_{13}	-3E-06	-6E-07	-3E-07	-0.0614	2E-06	5.1E-07	3.2E-06	7.6E-07	1	2.1E-05
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table H.4: Pure Error Variance for Initial Cumulative Gas Production Model

For replicate set	
Mean	3.9673
sum of residuals squared	0.085
degrees of freedom	5
Pure error variance	0.017

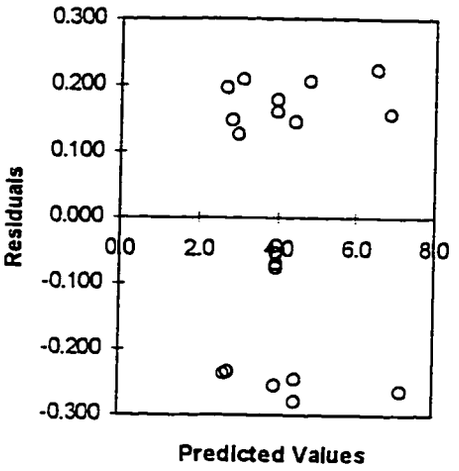

Figure H.1: Residuals Versus Predicted Values for Initial Cumulative Gas Production Model

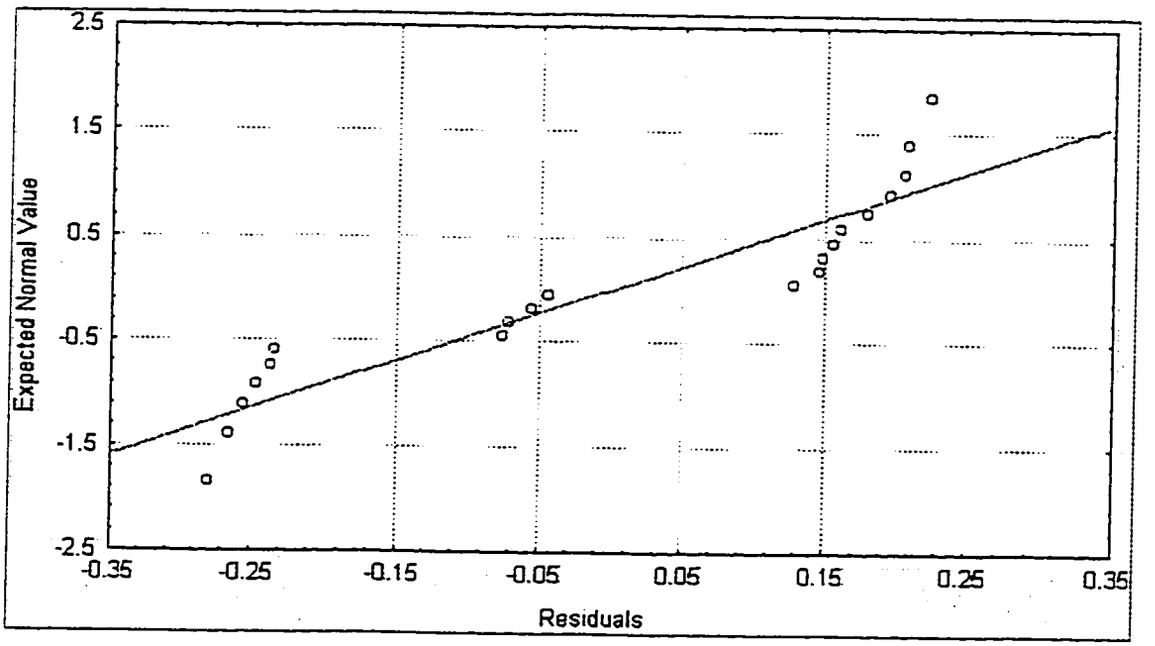

Figure H.2: Normal Probability Plot of Residuals for Initial Cumulative Gas Production Model

b) Final Model

Table H.5: Parameter Estimates for Final Cumulative Gas Production Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{12}
Estimate	4.0257	0.5407	1.3456	0.1633	-0.1493	0.3380	0.4559
Std.Err.	0.0881	0.0671	0.0690	0.0689	0.0587	0.0667	0.0826
t(8)	45.6813	8.0595	19.5015	2.3713	-2.5442	5.0709	5.5164
p-level	0.0000	0.0000	0.0000	0.0339	0.0245	0.0002	0.0001
Variance	explained	97.58%		R	0.9878		

Table H.6: ANOVA Table for Final Cumulative Gas Production Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	6	33.94	5.66	87.31	4.94	3.02
Residual	13	0.84	0.06			
(Lack of Fit)	8	0.75	0.09	5.01	10.97	5.05
(Pure Error)	5	0.09	0.02			
Total	19					

Table H.7: Correlation Matrix of Parameter Estimates for Final Cumulative Gas Production Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{12}
β_0	1.0000	0.1342	0.0000	0.0000	-0.5622	-0.5598	0.0000
β_1	0.1342	1.0000	0.0000	0.0000	-0.3114	-0.0216	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	-0.0614
β_3	2.6E-08	1.1E-08	3.3E-09	1	-4E-08	-1E-08	7.9E-09
β_{11}	-0.5622	-0.3114	0.0000	0.0000	1.0000	0.0829	0.0000
β_{22}	-0.5598	-0.0216	0.0000	0.0000	0.0829	1.0000	0.0000
β_{12}	5.3E-09	2.2E-09	-0.0614	7.9E-09	-7E-09	-3E-09	1

Table H.8: Pure Error Variance for Final Cumulative Gas Production Model

For replicate set	
Mean	3.9673
sum of residuals squared	0.093
degrees of freedom	5
Pure error variance	0.0186

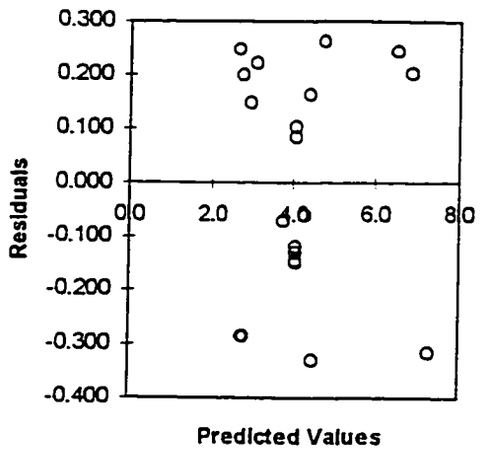

Figure H.3: Residuals Versus Predicted Values Number for Final Cumulative Gas Production Model

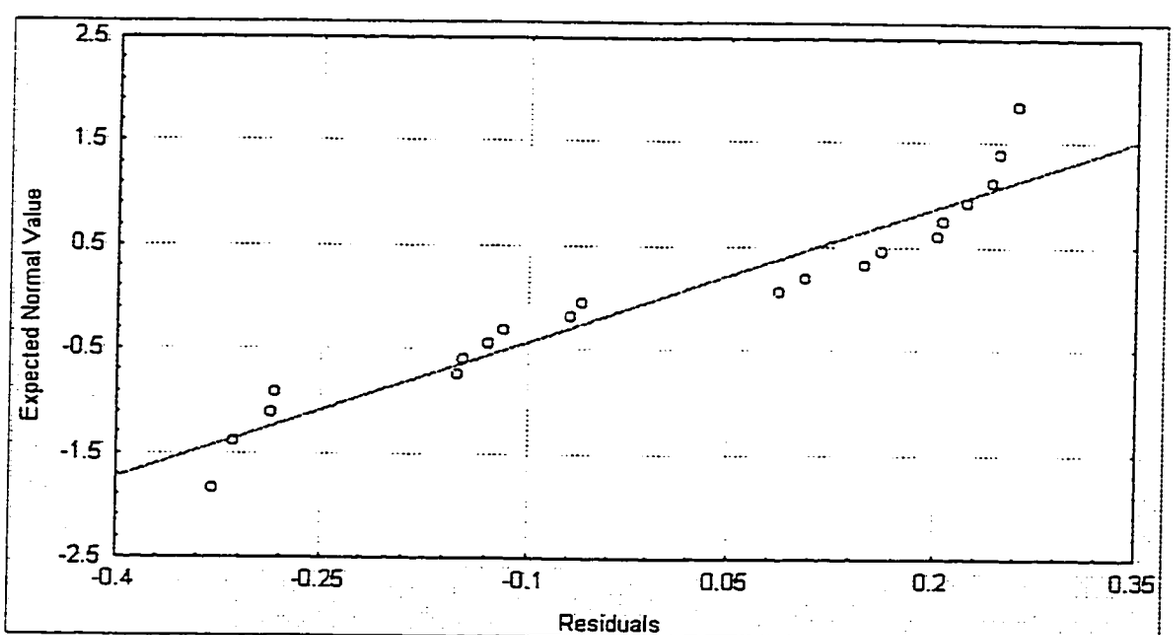

Figure H.4: Normal Probability Plot of Residuals for Final Cumulative Gas Production Model

H.2 Cumulative Gas Production at STP with Respect to Volatile Solids Addition

a) Initial Model

Table H.9: Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	0.4617	0.0452	-0.0240	0.0254	-0.0041	0.0531	0.0259	0.0539	-0.0001	0.0041
Std.Err.	0.0269	0.0174	0.0179	0.0179	0.0153	0.0174	0.0174	0.0215	0.0214	0.0234
t(8)	17.1552	2.5900	-1.3409	1.4146	-0.2661	3.0514	1.4877	2.5117	-0.0068	0.1754
p-level	0.0000	0.0270	0.2096	0.1875	0.7956	0.0122	0.1677	0.0308	0.9947	0.8642
Variance explained		73.53%			R	0.8575				

Table H.10: ANOVA Table for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{.01,v1,v2}$	$F_{.05,v1,v2}$
Regression	9	0.12	0.01	3.09	4.94	3.02
Residual	10	0.04	0.00			
(Lack of Fit)	5	0.04	0.01	6.46	10.97	5.05
(Pure Error)	5	0.01	0.00			
Total	19					

Table H.11: Correlation Matrix of Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0001	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0001	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0614	0.0000	0.0000
β_3	-7E-06	-6E-06	2.7E-06	1	1.4E-05	1.3E-06	1.4E-06	-1E-07	-0.0612	2.3E-05
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	-0.0002	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	-2E-07	1.4E-06	0.09103	0.09798	1	1.2E-07	-2E-05	8E-07
β_{12}	-6E-08	-2E-09	-0.0614	-1E-07	7.5E-09	8.9E-09	1.2E-07	1	2.8E-06	-9E-10
β_{13}	0.00012	9.4E-05	-4E-05	-0.0612	-0.0002	-2E-05	-2E-05	2.8E-06	1	-0.0003
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	-0.0003	1.0000

Table H.12: Calculation for Pure Error Variance for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

For replicate set	
Mean	0.4668
sum of residuals squared	0.006
degrees of freedom	5
Pure error variance	0.0012

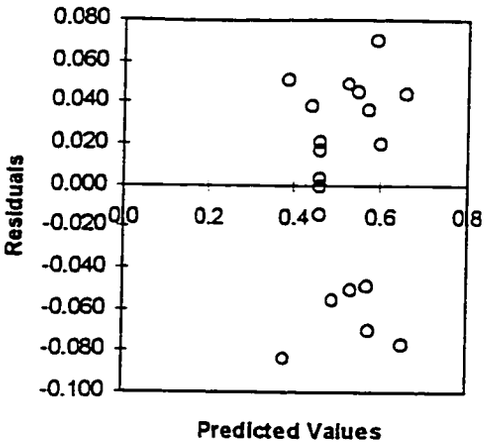

Figure H.5: Residuals versus Predicted Values for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

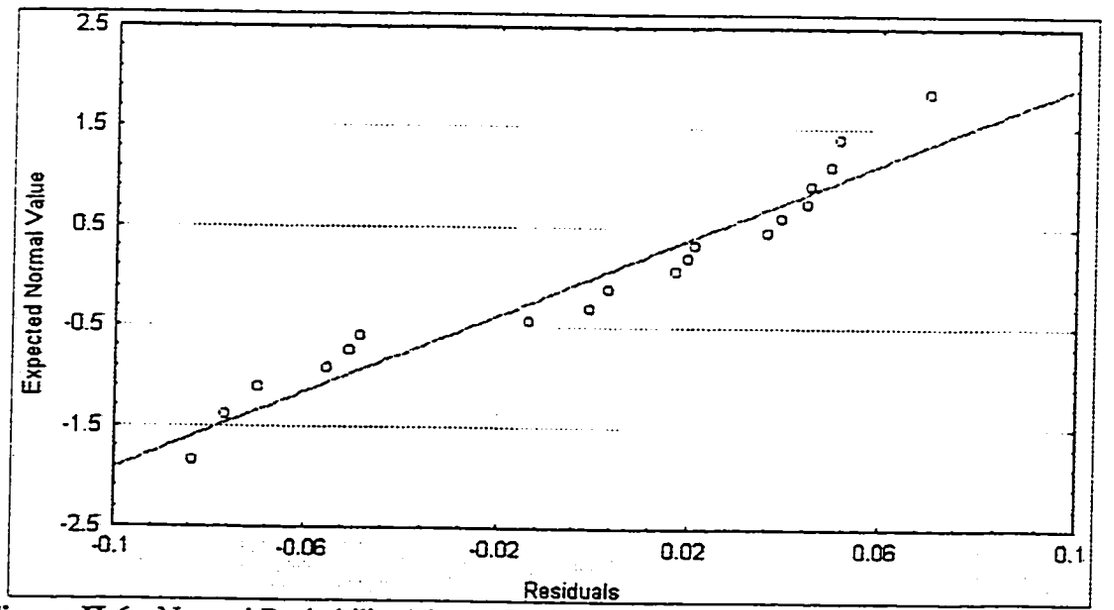

Figure H.6: Normal Probability Plot of Residuals for Initial Cumulative Gas Production Model with Respect to Volatile Solids Addition

b) Final Model

Table H.13: Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_{22}	β_{12}
Estimate	0.4776	0.0436	0.0512	0.0522
Std.Err.	0.0191	0.0167	0.0174	0.0216
t(8)	25.0541	2.6155	2.9449	2.4182
p-level	0.0000	0.0187	0.0095	0.0279

Variance	explained	57.10%	R	0.7556

Table H.14: ANOVA Table for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{.01,v1,v2}$	$F_{.05,v1,v2}$
Regression	3	0.09	0.03	7.10	4.94	3.02
Residual	16	0.07	0.00			
(Lack of Fit)	11	0.05	0.00	1.48	10.97	5.05
(Pure Error)	5	0.02	0.00			
Total	19					

Table H.15: Correlation Matrix of Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

	β_0	β_1	β_{22}	β_{12}
β_0	1.0000	-0.0520	-0.6227	0.0000
β_1	-0.0520	1.0000	0.0045	0.0000
β_{22}	-0.6227	0.0045	1.0000	0.0000
β_{12}	1.3E-08	-2E-08	-2E-08	1

Table H.16: Pure Error Variance for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

For replicate set	
Mean	0.4668
sum of residuals squared	0.017
degrees of freedom	5
Pure error variance	0.0034

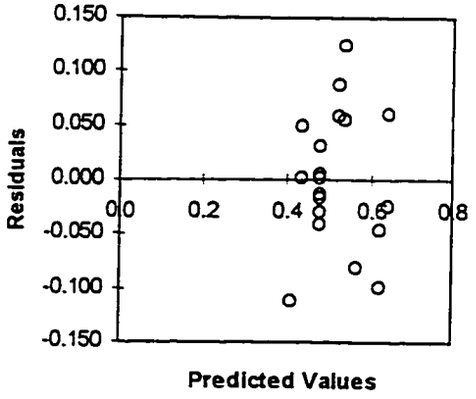

Figure H.7: Residuals Versus Predicted Values for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

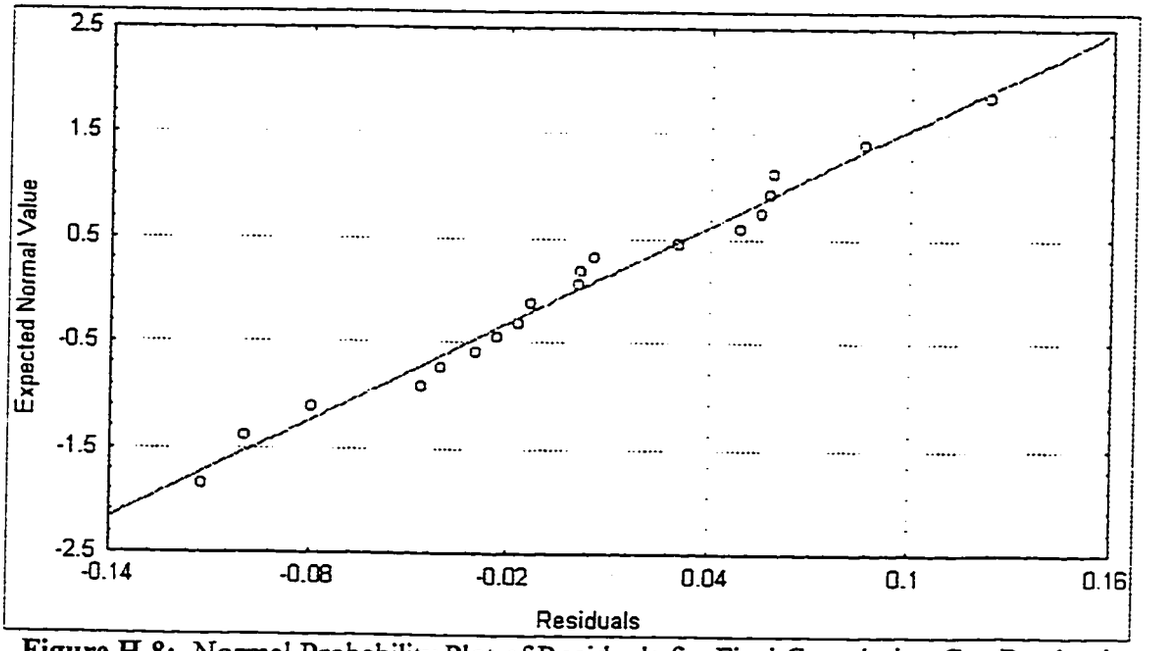

Figure H.8: Normal Probability Plot of Residuals for Final Cumulative Gas Production Model with Respect to Volatile Solids Addition

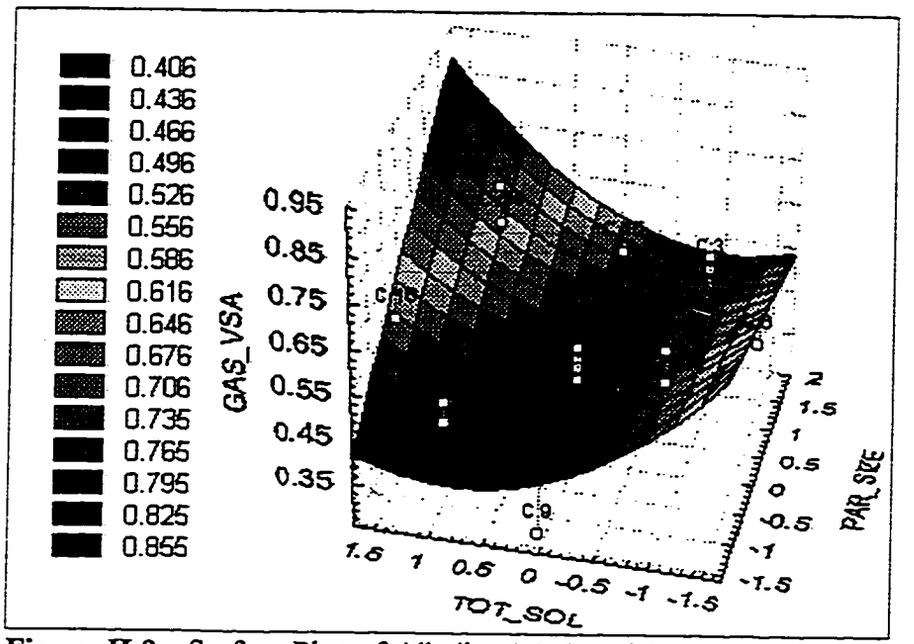

Figure H.9: Surface Plot of Alkaline Model of Gas Production with Respect to Volatile Solids Addition

H.3 Cumulative Gas Production at STP with Respect to Volatile Solids Removal

a) Initial Model

Table H.17: Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	0.5373	0.0587	-0.0619	0.1478	0.0063	0.0840	0.0896	0.0747	0.0148	0.0046
Std.Err.	0.0451	0.0292	0.0300	0.0300	0.0256	0.0291	0.0291	0.0360	0.0360	0.0392
t(8)	11.9216	2.0108	-2.0631	4.9246	0.2473	2.8813	3.0753	2.0789	0.4123	0.1181
p-level	0.0000	0.0721	0.0660	0.0006	0.8097	0.0163	0.0117	0.0643	0.6888	0.9083

Variance explained 84.33% R 0.9183

Table H.18: ANOVA Table for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{.01,v1,v2}$	$F_{.05,v1,v2}$
Regression	9	0.66	0.07	5.98	4.94	3.02
Residual	10	0.12	0.01			
(Lack of Fit)	5	0.11	0.02	6.25	10.97	5.05
(Pure Error)	5	0.02	0.00			
Total	19					

Table H.19: Correlation Matrix of Parameter Estimates for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0000	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0000	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0614	0.0000	0.0000
β_3	2.4E-08	5.3E-09	5.2E-09	1	-4E-08	-1E-08	-7E-09	4.2E-09	-0.0614	-7E-08
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	0.0000	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	3.2E-08	-7E-09	0.09104	0.09798	1	-2E-08	-1E-07	-6E-07
β_{12}	4.3E-08	-2E-09	-0.0614	4.2E-09	-6E-08	-1E-08	-2E-08	1	-7E-08	-8E-09
β_{13}	3.5E-07	1.6E-07	-3E-08	-0.0614	-6E-07	-6E-08	-1E-07	-7E-08	1	-6E-07
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table H.20: Pure Error Variance for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

For replicate set	
Mean	0.5457
sum of residuals squared	0.017
degrees of freedom	5
Pure error variance	0.0034

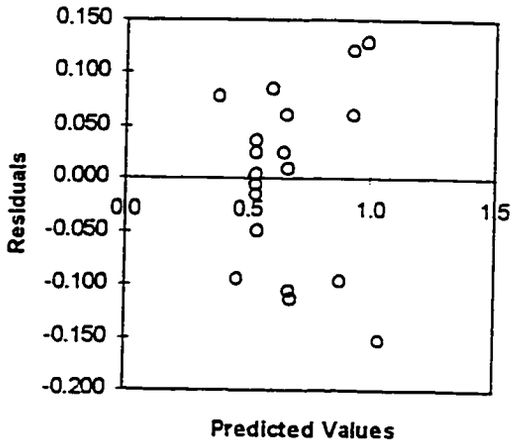

Figure H.9: Residual versus Predicted Values for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

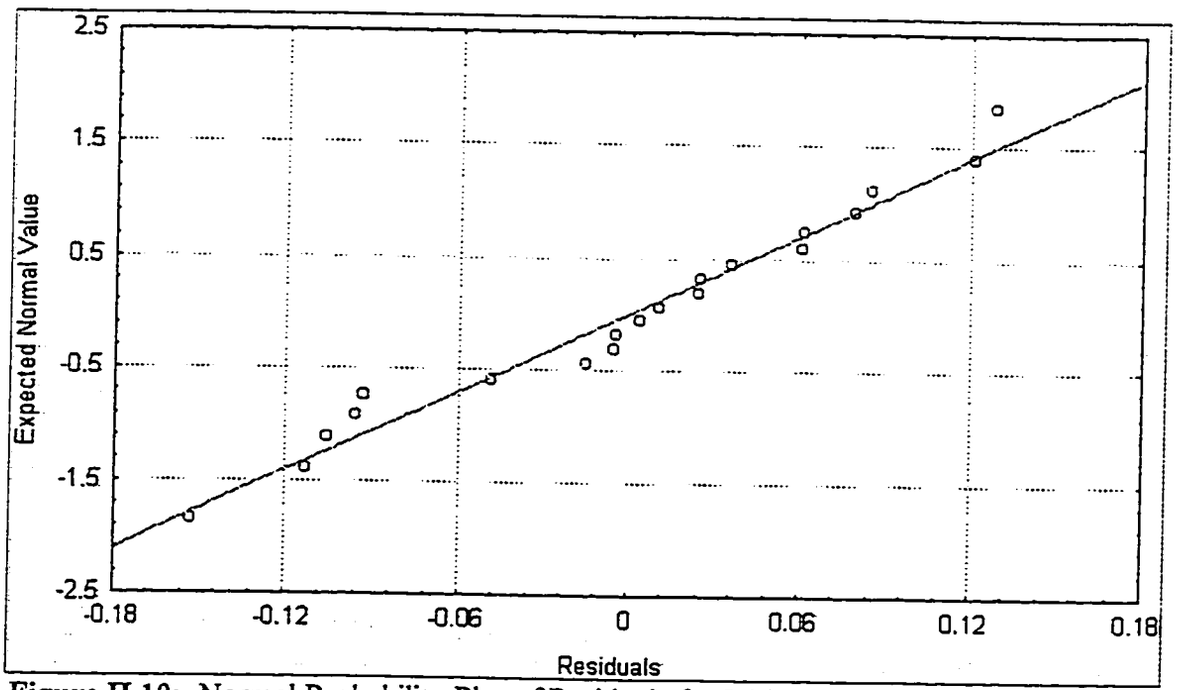

Figure H.10: Normal Probability Plot of Residuals for Initial Cumulative Gas Production Model with Respect to Volatile Solids Removal

b) Final Model

Table H.21: Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_3	β_{22}	β_{33}	β_{12}
Estimate	0.5431	0.0610	-0.0619	0.1486	0.0833	0.0890	0.0747
Std.Err.	0.0341	0.0246	0.0267	0.0266	0.0258	0.0258	0.0319
t(8)	15.9406	2.4758	-2.3240	5.5863	3.2337	3.4532	2.3418
p-level	0.0000	0.0278	0.0370	0.0001	0.0065	0.0043	0.0358
Variance explained		83.95%			R	0.9162	

Table H.22: ANOVA Table for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	6	0.66	0.11	11.33	4.94	3.02
Residual	13	0.13	0.01			
(Lack of Fit)	8	0.11	0.01	5.01	10.97	5.05
(Pure Error)	5	0.01	0.00			
Total	19					

Table H.23: Correlation Matrix of Parameter Estimates for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

	β_0	β_1	β_2	β_3	β_{22}	β_{33}	β_{12}
β_0	1.0000	-0.0457	0.0000	0.0000	-0.5633	-0.5633	0.0000
β_1	-0.0457	1.0000	0.0000	0.0000	0.0049	0.0049	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	-0.0614
β_3	-4E-10	8.2E-09	8.7E-09	1	4E-11	4E-11	-5E-10
β_{22}	-0.5633	0.0049	0.0000	0.0000	1.0000	0.0904	0.0000
β_{33}	-0.5633	0.00489	-1E-09	4E-11	0.09044	1	1.2E-08
β_{12}	-6E-09	2.2E-08	-0.0614	-5E-10	-3E-10	1.2E-08	1

Table H.24: Pure Error Variance for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

For replicate set	
Mean	0.5457
sum of residuals squared	0.013
degrees of freedom	5
Pure error variance	0.0026

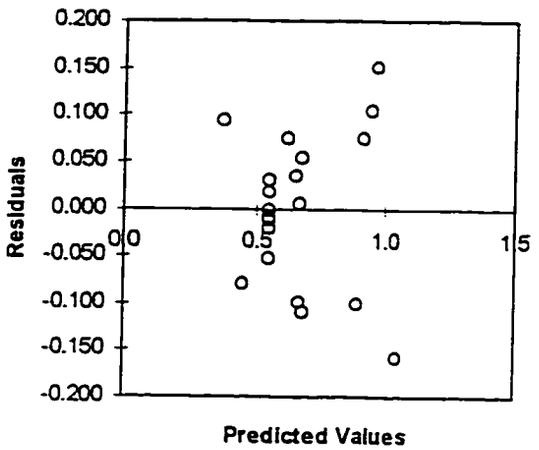

Figure H.11: Residuals Versus Predicted Values for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

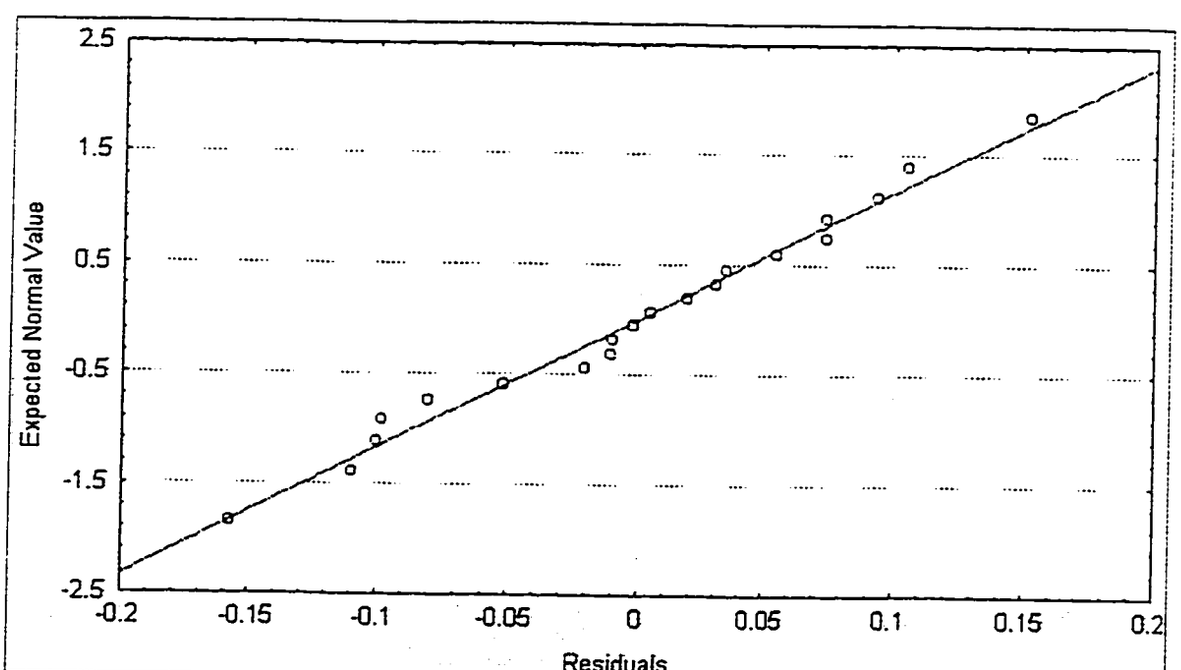

Figure H.12: Normal Probability Plot of Residuals for Final Cumulative Gas Production Model with Respect to Volatile Solids Removal

H.4 Chemical Oxygen Demand

a) Initial Model

Table H.25: Parameter Estimates for Initial Chemical Oxygen Demand Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	63.4031	2.4888	3.9974	-7.9329	-3.3613	-1.7306	-4.1766	-1.8280	-3.3845	-6.0713
Std.Err.	2.7032	1.7514	1.8008	1.8008	1.5377	1.7481	1.7481	2.1567	2.1567	2.3486
t(8)	23.4544	1.4211	2.2198	-4.4052	-2.1859	-0.9900	-2.3892	-0.8476	-1.5693	-2.5851
p-level	0.0000	0.1857	0.0507	0.0013	0.0537	0.3455	0.0380	0.4165	0.1476	0.0272

Variance explained 81.99% R 0.9055

Table H.26: ANOVA Table for Initial Chemical Oxygen Demand Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	9	2008.57	223.17	5.06	4.94	3.02
Residual	10	441.26	44.13			
(Lack of Fit)	5	314.94	62.99	2.49	10.97	5.05
(Pure Error)	5	126.32	25.26			
Total	19					

Table H.27: Correlation Matrix of Parameter Estimates for Initial Chemical Oxygen Demand Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0000	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0000	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0614	0.0000	0.0000
β_3	-7E-09	-4E-09	-2E-09	1	8.6E-09	4.6E-09	3.3E-09	8E-09	-0.0614	1.4E-09
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	0.0000	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	-8E-09	3.3E-09	0.09104	0.09798	1	3.8E-09	5.7E-09	8.1E-10
β_{12}	-2E-08	-8E-10	-0.0614	8E-09	2.3E-08	1.4E-08	3.8E-09	1	1.9E-08	2.7E-10
β_{13}	-9E-09	-5E-09	-1E-08	-0.0614	6.8E-09	6.5E-09	5.7E-09	1.9E-08	1	2E-09
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table H.28: Pure Error Variance for Initial Chemical Oxygen Demand Model

For replicate set	
Mean	63.0350
sum of residuals squared	126.316
degrees of freedom	5
Pure error variance	25.2632

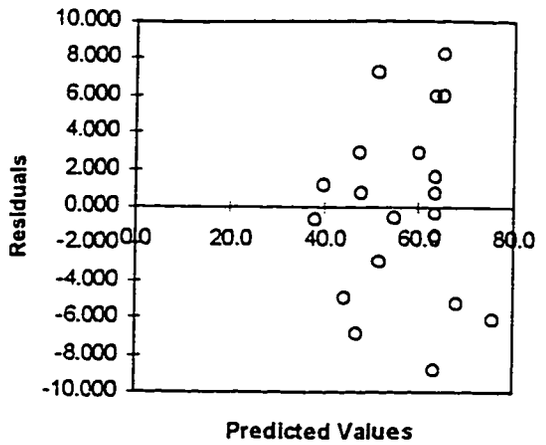

Figure H.13: Residuals versus Predicted Values for Initial Chemical Oxygen Demand Model

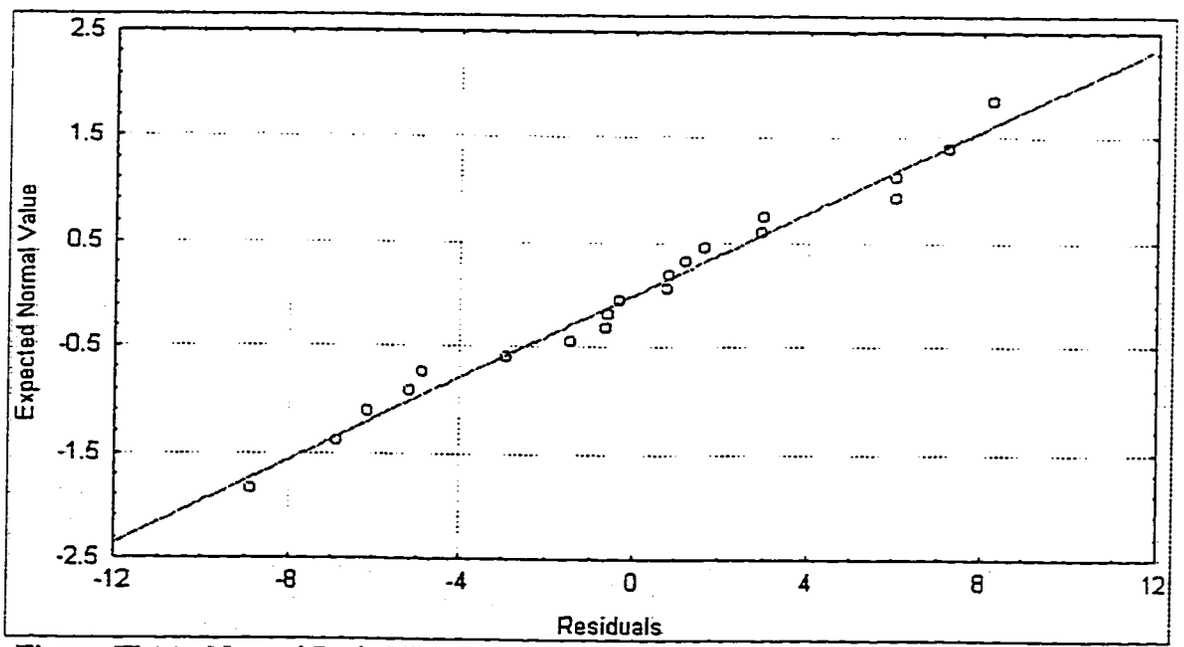

Figure H.14: Normal Probability Plot of Residuals for Initial Chemical Oxygen Demand Model

b) Final Model

Table H.29: Parameter Estimates for Final Chemical Oxygen Demand Model

	β_0	β_3	β_{23}
Estimate	56.8195	-8.1063	-6.0713
Std.Err.	1.9231	2.3271	3.0407
t(8)	29.5458	-3.4834	-1.9967
p-level	0.0000	0.0028	0.0621
	Variance explained	48.67%	R 0.6977

Table H.30: ANOVA Table for Final Chemical Oxygen Demand Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	2	1192.40	596.20	8.06	4.94	3.02
Residual	17	1257.42	73.97			
(Lack of Fit)	12	953.24	79.44	1.31	10.97	5.05
(Pure Error)	5	304.18	60.84			
Total	19					

Table H.31: Correlation Matrix of Parameter Estimates for Final Chemical Oxygen Demand Model

	β_0	β_3	β_{23}
β_0	1.0000	0.0000	0.0000
β_3	-1E-10	1	-9E-10
β_{23}	0.0000	0.0000	1.0000

Table H.32: Pure Error Variance for Final Chemical Oxygen Demand Model

For replicate set	
Mean	63.0350
sum of residuals squared	304.181
degrees of freedom	5
Pure error variance	60.8362

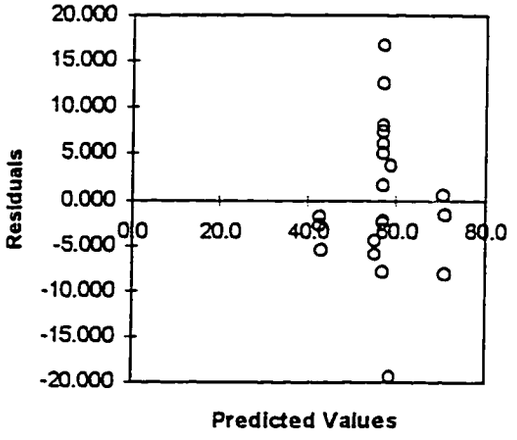

Figure H.15: Residuals Versus Predicted Values for Final Chemical Oxygen Demand Model

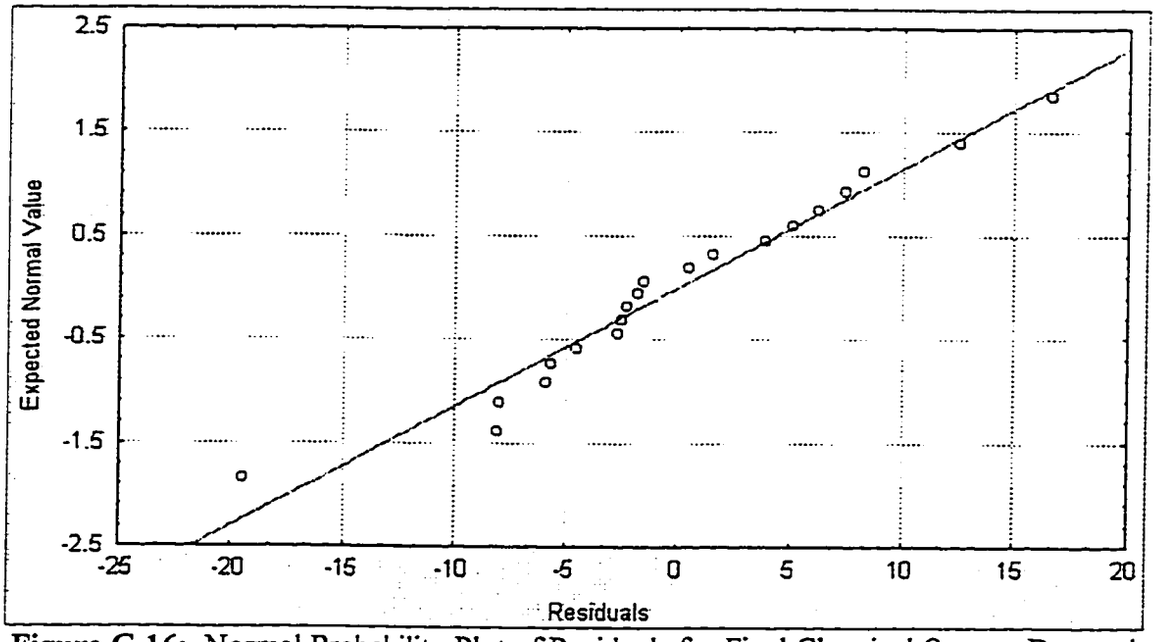

Figure G.16: Normal Probability Plot of Residuals for Final Chemical Oxygen Demand Model

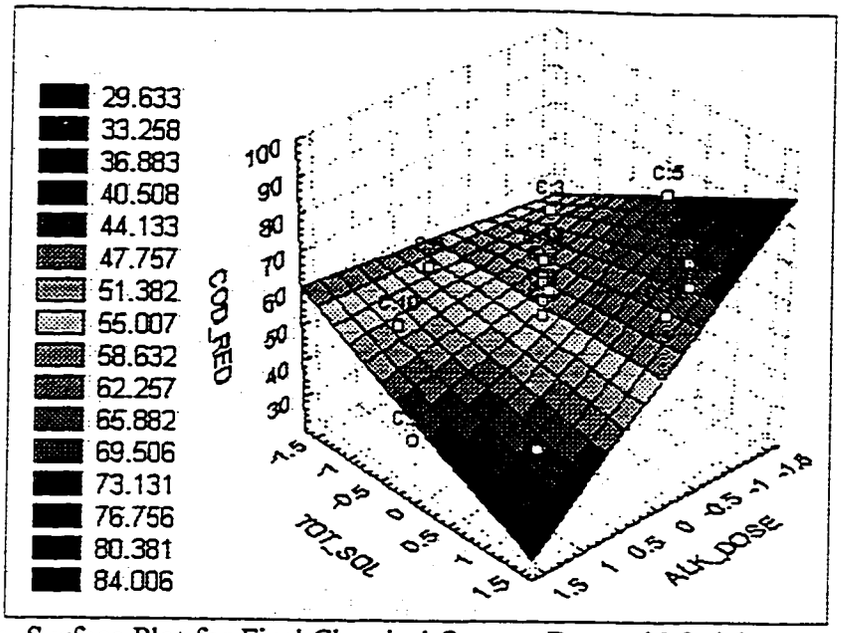

Figure H.17: Surface Plot for Final Chemical Oxygen Demand Model

H.5 Volatile Solids Reduction

a) Initial Model

Table H.33: Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	86.0813	0.1006	3.7813	-11.4222	-1.2708	-1.6450	-5.2009	-0.1300	-0.1699	-1.1700
Std.Err.	1.4754	0.9559	0.9829	0.9829	0.8393	0.9541	0.9541	1.1771	1.1771	1.2818
t(8)	58.3435	0.1052	3.8472	-11.6212	-1.5142	-1.7241	-5.4510	-0.1105	-0.1444	-0.9128
p-level	0.0000	0.9183	0.0032	0.0000	0.1609	0.1154	0.0003	0.9142	0.8881	0.3828
Variance explained		94.83%			R	0.9738				

Table H.34: ANOVA Table for Volatile Solids Reduction Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{.01,v1,v2}$	$F_{.05,v1,v2}$
Regression	9	2412.97	268.11	20.40	4.94	3.02
Residual	10	131.45	13.14			
(Lack of Fit)	5	130.73	26.15	181.73	10.97	5.05
(Pure Error)	5	0.72	0.14			
Total	19					

Table H.35: Correlation Matrix of Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0000	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0000	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0614	0.0000	0.0000
β_3	9.4E-09	6.5E-08	9.8E-15	1	-3E-08	1.8E-09	-2E-09	-1E-13	-0.0614	-1E-08
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	0.0000	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	-2E-09	-1.9E-09	0.09104	0.09798	1	4.1E-08	3.1E-08	-1E-10
β_{12}	-2E-07	-2E-06	-0.0614	-1.1E-13	5.3E-07	4.1E-08	4.1E-08	1	2.2E-12	3.4E-13
β_{13}	-2E-07	-1E-06	-2E-13	-0.06138	4.8E-07	-4E-08	3.1E-08	2.2E-12	1	1.5E-07
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table H.36: Pure Error Variance for Volatile Solids Reduction Model

For replicate set	
Mean	86.1083
sum of residuals squared	0.719
degrees of freedom	5
Pure error variance	0.1438

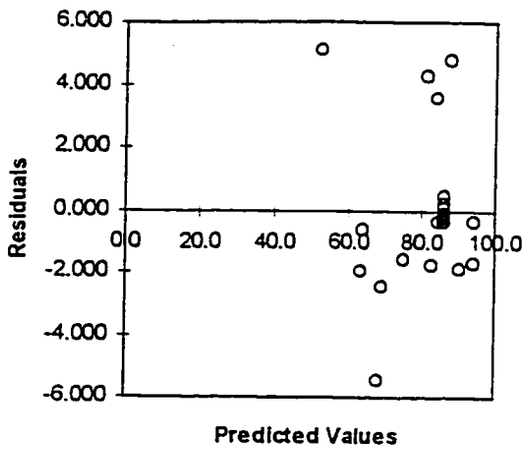

Figure H.18: Residuals Versus Predicted Values and Run Number for Volatile Solids Reduction

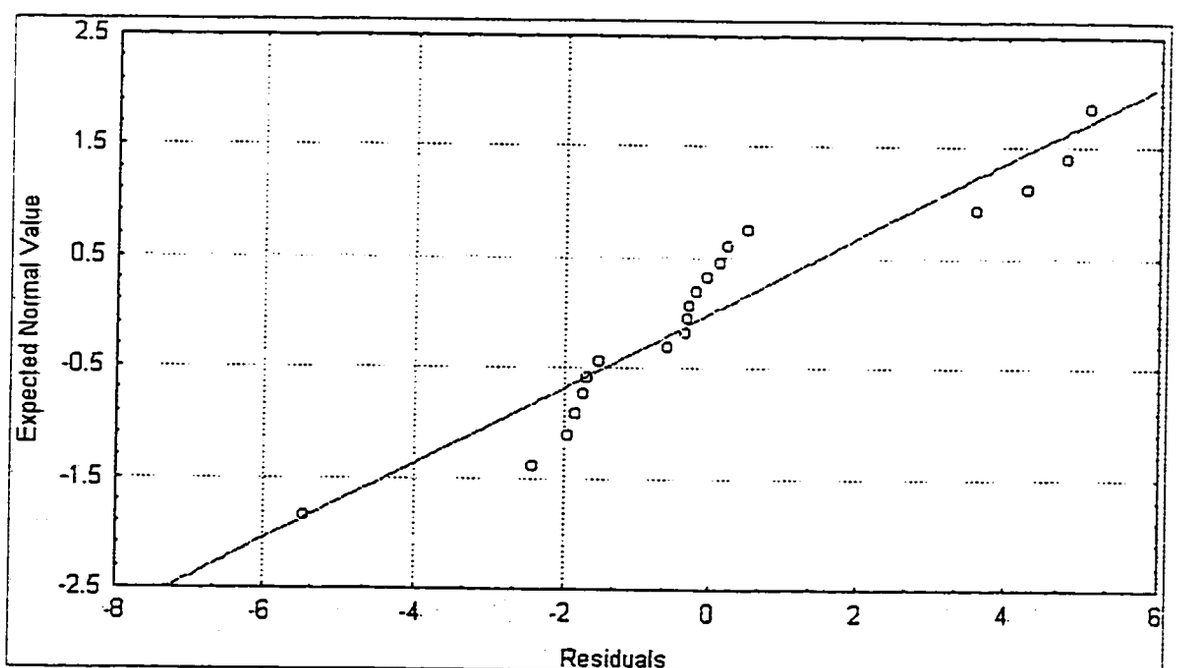

Figure H.19: Normal Probability Plot of Residuals for Volatile Solids Reduction Model

b) Final Model

Table H.37: Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_2	β_3	β_{33}
Estimate	83.7624	3.7746	-11.4309	-4.9309
Std.Err.	1.0315	0.9761	0.9761	0.9415
t(8)	81.2016	3.8670	-11.7108	-5.2372
p-level	0.0000	0.0014	0.0000	0.0001
Variance explained	91.82%		R	0.9582

Table H.38: ANOVA Table for Volatile Solids Reduction Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	3	2336.20	778.73	59.84	4.94	3.02
Residual	16	208.21	13.01			
(Lack of Fit)	11	178.37	16.22	2.72	10.97	5.05
(Pure Error)	5	29.84	5.97			
Total	19					

Table H.39: Correlation Matrix of Parameter Estimates for Volatile Solids Reduction Model

	β_0	β_2	β_3	β_{33}
β_0	1.0000	0.0000	0.0000	-0.6233
β_2	0.0000	1.0000	0.0000	0.0000
β_3	-1E-11	2.5E-10	1	7.4E-12
β_{33}	-0.6233	-2E-11	7.36E-12	1

Table H.40: Calculations for Pure Error Variance for Volatile Solids Reduction Model

For replicate set	
Mean	86.1083
sum of residuals squared	29.838
degrees of freedom	5
Pure error variance	5.9676

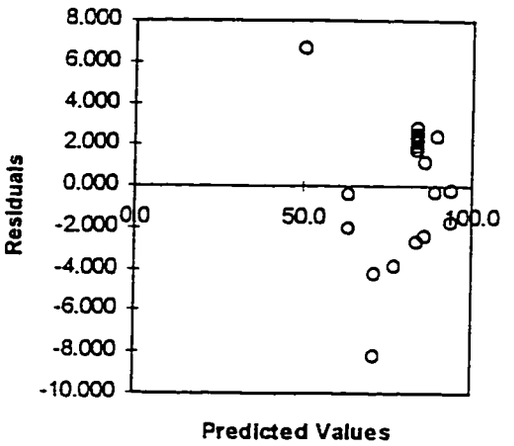

Figure H.21: Residuals Versus Predicted Values for Volatile Solids Reduction Model

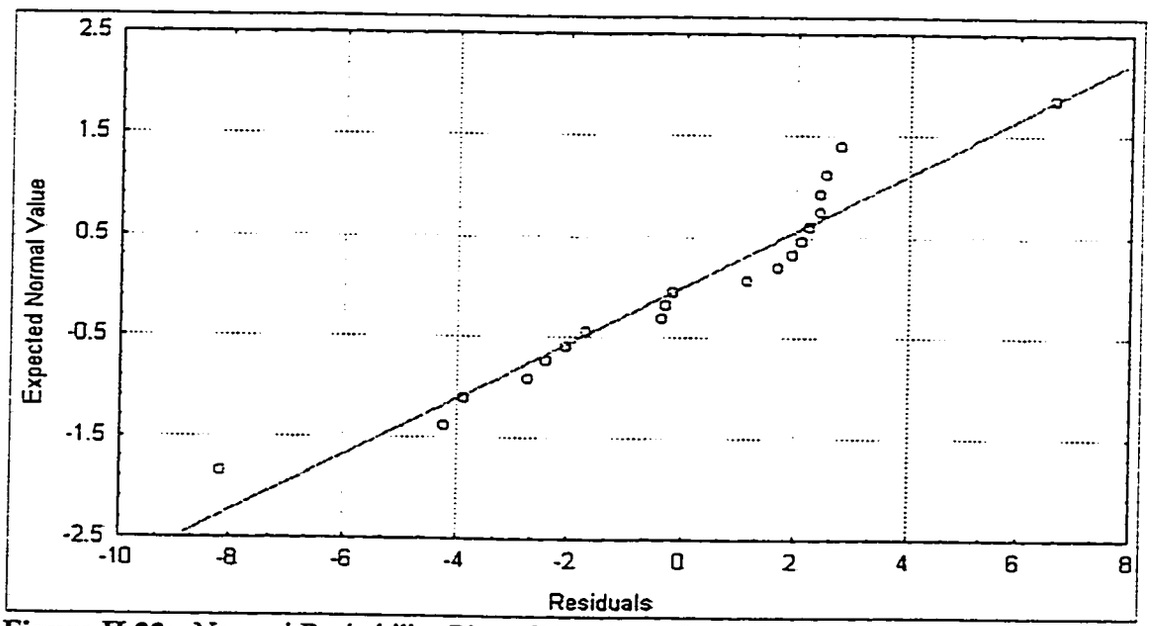

Figure H.22: Normal Probability Plot of Residuals for Volatile Solids Reduction Model

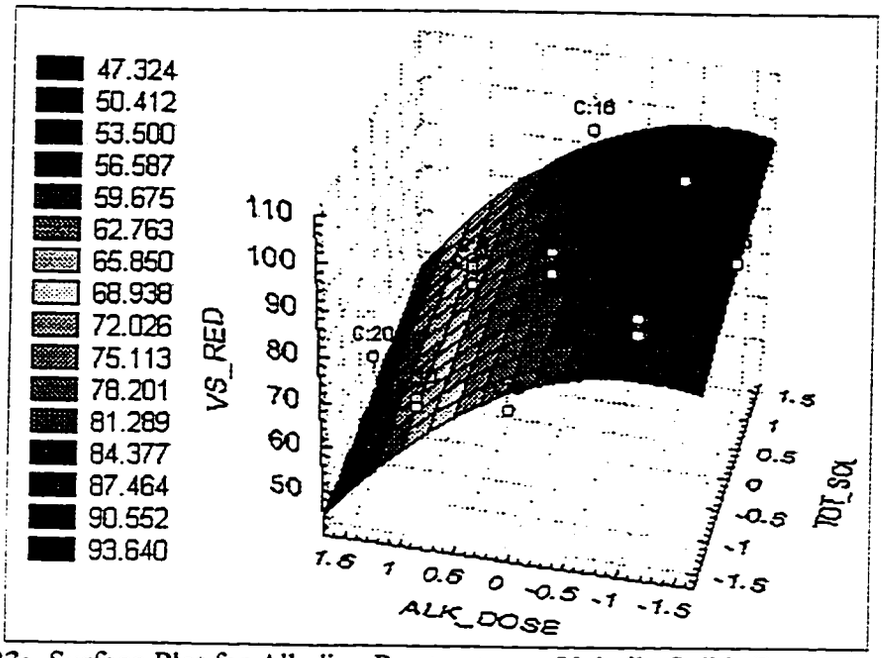

Figure H.23: Surface Plot for Alkaline Pre-treatment Volatile Solids Reduction Model

H.6 Total Solids Reduction

a) Initial Model

Table H.41: Parameter Estimates for Initial Total Solids Reduction Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	52.9355	0.6783	4.6769	-8.3785	-1.3550	-0.6456	-3.2824	0.1117	-0.2737	-3.0950
Std.Err.	1.3707	0.8880	0.9131	0.9131	0.7797	0.8864	0.8864	1.0935	1.0935	1.1908
t(8)	38.6203	0.7638	5.1220	-9.1760	-1.7378	-0.7283	-3.7032	0.1022	-0.2503	-2.5990
p-level	0.0000	0.4626	0.0004	0.0000	0.1129	0.4831	0.0041	0.9206	0.8074	0.0265

Variance explained 93.05% R 0.9646

Table H.42: ANOVA Table for Initial Total Solids Reduction Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	9	1518.42	168.71	14.87	4.94	3.02
Residual	10	113.45	11.34			
(Lack of Fit)	5	96.38	19.28	5.65	10.97	5.05
(Pure Error)	5	17.06	3.41			
Total	19					

Table H.43: Correlation Matrix of Parameter Estimates for Initial Total Solids Reduction Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0000	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0000	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0614	0.0000	0.0000
β_3	-3E-09	-1E-08	3.8E-09	1	6.4E-09	-4E-10	6.4E-10	-6E-08	-0.0614	1.5E-09
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	0.0000	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	-1E-09	6.41E-10	0.09104	0.09798	1	2.7E-08	-2E-08	-6E-10
β_{12}	-2E-07	-6E-07	-0.0614	-6.42E-08	3.9E-07	-3E-08	2.7E-08	1	1.7E-06	5.6E-08
β_{13}	7.5E-08	2.9E-07	-1E-07	-0.06138	-2E-07	1.1E-08	-2E-08	1.7E-06	1	-4E-08
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table H.44: Pure Error Variance for Initial Total Solids Reduction Model

For replicate set	
Mean	52.9650
sum of residuals squared	17.064
degrees of freedom	5
Pure error variance	3.4128

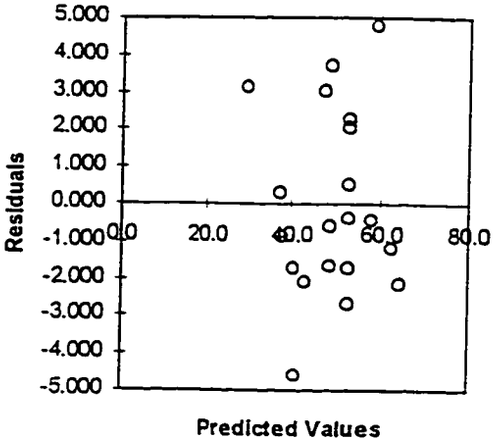

Figure H.24: Residuals versus Predicted Values for Initial Total Solids Reduction Model

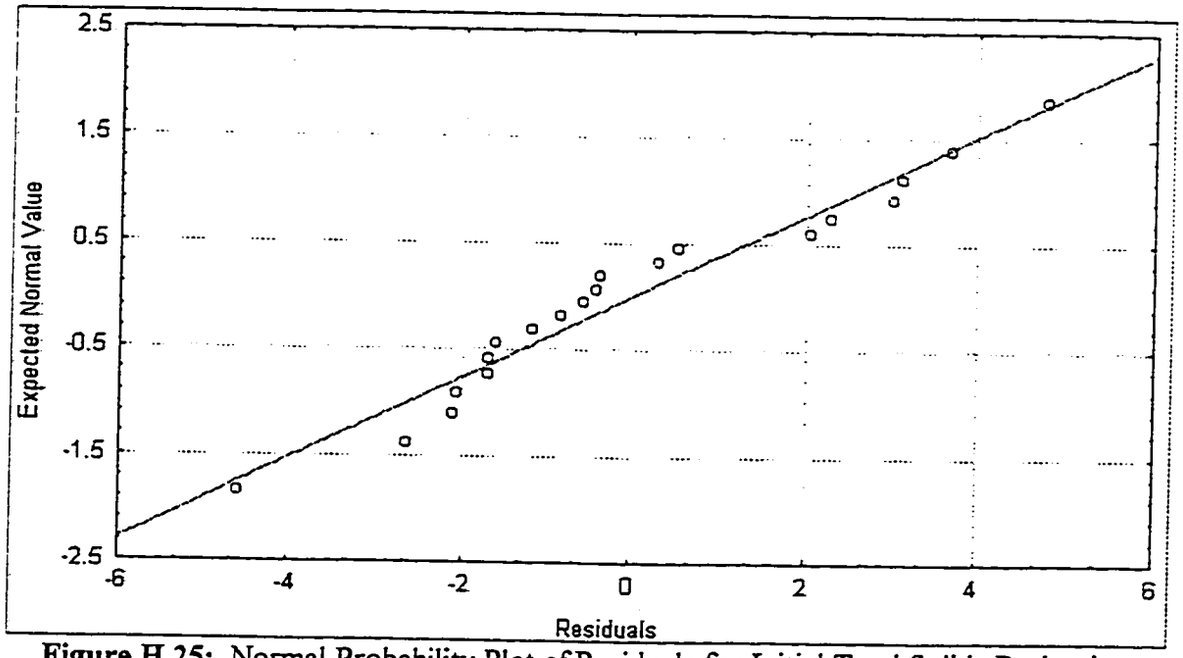

Figure H.25: Normal Probability Plot of Residuals for Initial Total Solids Reduction Model

b) Final Model

Table H.45: Parameter Estimates for Final Total Solids Reduction Model

	β_0	β_2	β_3	β_{33}	β_{23}
Estimate	51.3223	4.6826	-8.3926	-3.0975	-3.0950
Std.Err.	0.9129	0.8638	0.8638	0.8332	1.1287
t(8)	56.2184	5.4207	-9.7153	-3.7174	-2.7420
p-level	0.0000	0.0001	0.0000	0.0021	0.0151
Variance explained		90.63%		R	0.9520

Table H.46: ANOVA Table for Final Total Solids Reduction Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{01,v1,v2}$	$F_{05,v1,v2}$
Regression	4	1478.98	369.75	36.28	4.94	3.02
Residual	15	152.88	10.19			
(Lack of Fit)	10	116.58	11.66	1.61	10.97	5.05
(Pure Error)	5	36.31	7.26			
Total	19					

Table H.47: Correlation Matrix of Parameter Estimates for Final Total Solids Reduction Model

	β_0	β_2	β_3	β_{33}	β_{23}
β_0	1.0000	0.0000	0.0000	-0.6233	0.0000
β_2	0.0000	1.0000	0.0000	0.0000	0.0000
β_3	-6E-10	-6E-10	1	9.18E-10	-1E-09
β_{33}	-0.6233	-3E-09	9.2E-10	1	2.6E-10
β_{23}	0.0000	0.0000	0.0000	0.0000	1.0000

Table H.48: Pure Error Variance for Final Total Solids Reduction Model

For replicate set	
Mean	52.9650
sum of residuals squared	36.307
degrees of freedom	5
Pure error variance	7.2614

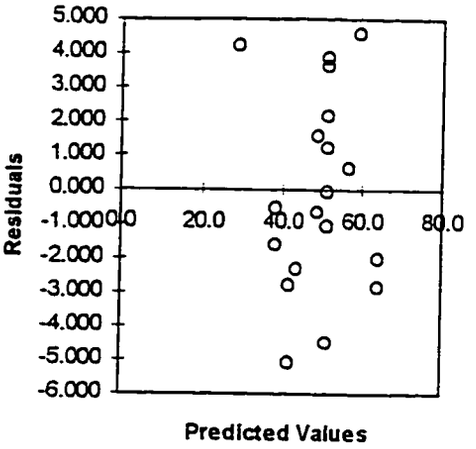

Figure H.26: Residuals Versus Predicted Values for Final Total Solids Reduction Model

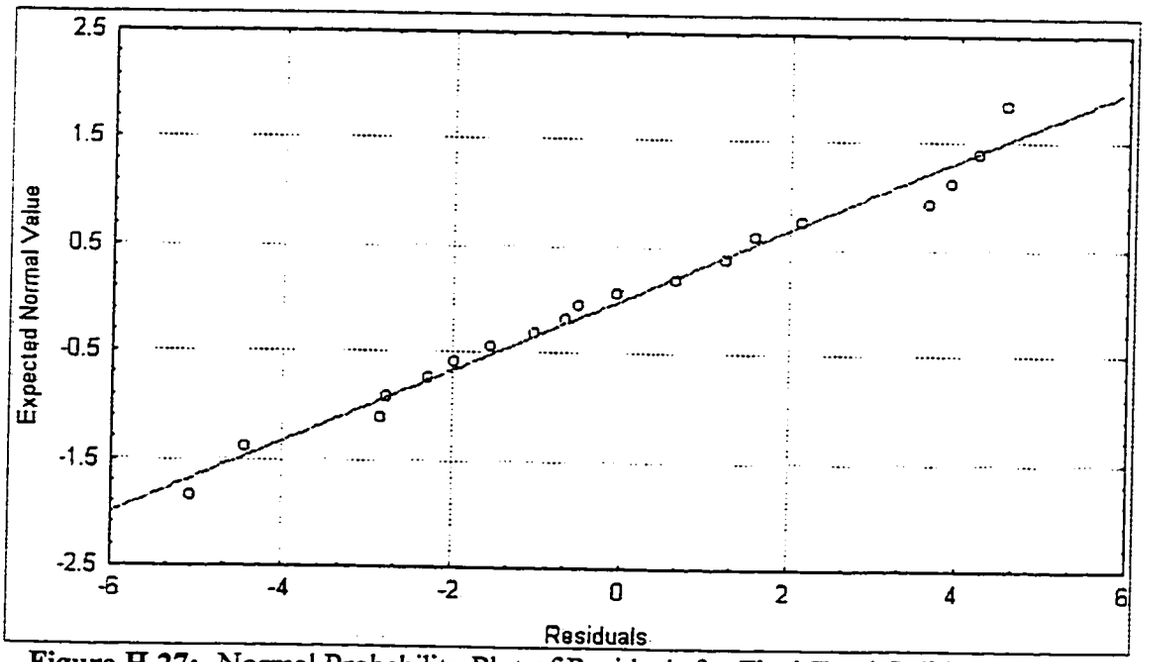

Figure H.27: Normal Probability Plot of Residuals for Final Total Solids Reduction Model

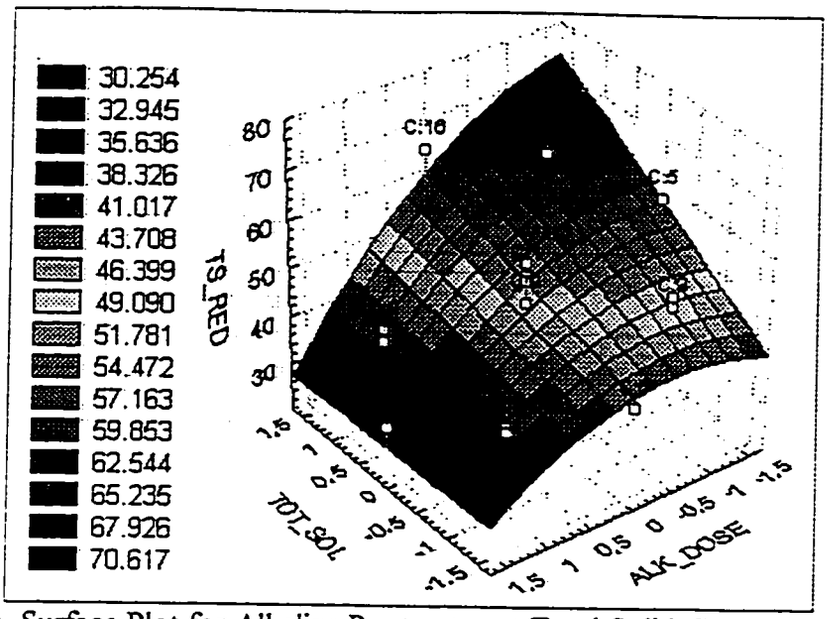

Figure H.28: Surface Plot for Alkaline Pre-treatment Total Solids Reduction Model

H.7 Methane Concentration

a) Initial Model

Table H.49: Parameter Estimates for Methane Concentration Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Estimate	58.8675	0.9917	0.3833	0.9391	-0.0714	0.5466	-0.6976	-0.1314	1.2615	-0.2000
Std.Err.	0.8939	0.5791	0.5955	0.5955	0.5085	0.5780	0.5780	0.7131	0.7131	0.7766
t(8)	65.8582	1.7124	0.6437	1.5770	-0.1404	0.9456	-1.2069	-0.1842	1.7689	-0.2575
p-level	0.0000	0.1176	0.5342	0.1459	0.8911	0.3666	0.2553	0.8575	0.1073	0.8020

Variance explained 54.92% R 0.7411

Table H.50: ANOVA Table for Methane Concentration Model

Source of Variation	*Deg. of Freedom*	*Sum of Squares*	*Mean Square*	*F*	$F_{.01,v1,v2}$	$F_{.05,v1,v2}$
Regression	9	58.79	6.53	1.35	4.94	3.02
Residual	10	48.25	4.82			
(Lack of Fit)	5	26.06	5.21	1.17	10.97	5.05
(Pure Error)	5	22.18	4.44			
Total	19					

Table H.51: Correlation Matrix of Parameter Estimates for Methane Concentration Model

	β_0	β_1	β_2	β_3	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
β_0	1.0000	0.1267	0.0000	0.0000	-0.5242	-0.5255	-0.5255	0.0000	0.0000	0.0000
β_1	0.1267	1.0000	0.0000	0.0000	-0.3122	-0.0238	-0.0238	0.0000	0.0000	0.0000
β_2	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	0.0000	-0.0614	0.0000	0.0000
β_3	-8E-08	-5E-08	4.9E-15	1	1.5E-07	1.4E-08	1.4E-08	-8E-14	-0.0614	9.1E-10
β_{11}	-0.5242	-0.3122	0.0000	0.0000	1.0000	0.0910	0.0910	0.0000	0.0000	0.0000
β_{22}	-0.5255	-0.0238	0.0000	0.0000	0.0910	1.0000	0.0980	0.0000	0.0000	0.0000
β_{33}	-0.5255	-0.0238	4.7E-09	1.4E-08	0.09104	0.09798	1	-8E-08	-8E-10	-3E-09
β_{12}	2.9E-07	1.6E-07	-0.0614	-8E-14	-5E-07	-5E-08	-8E-08	1	4.5E-15	1.4E-15
β_{13}	4.6E-09	6.9E-10	-3E-16	-0.0614	-9E-09	-8E-10	-8E-10	4.5E-15	1	-1E-08
β_{23}	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	1.0000

Table H.52: Pure Error Variance for Methane Concentration Model

For replicate set	
Mean	58.8017
sum of residuals squared	22.183
degrees of freedom	5
Pure error variance	4.4366

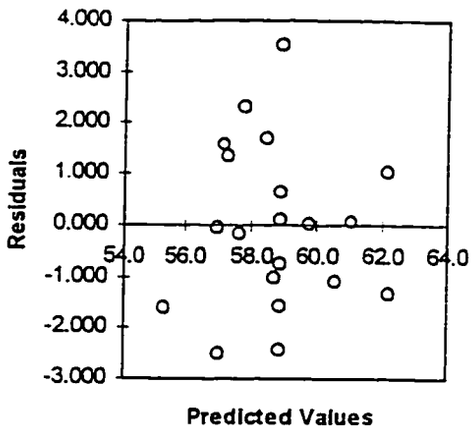

Figure H.29: Residuals versus Predicted Values for Initial Methane Concentration Model

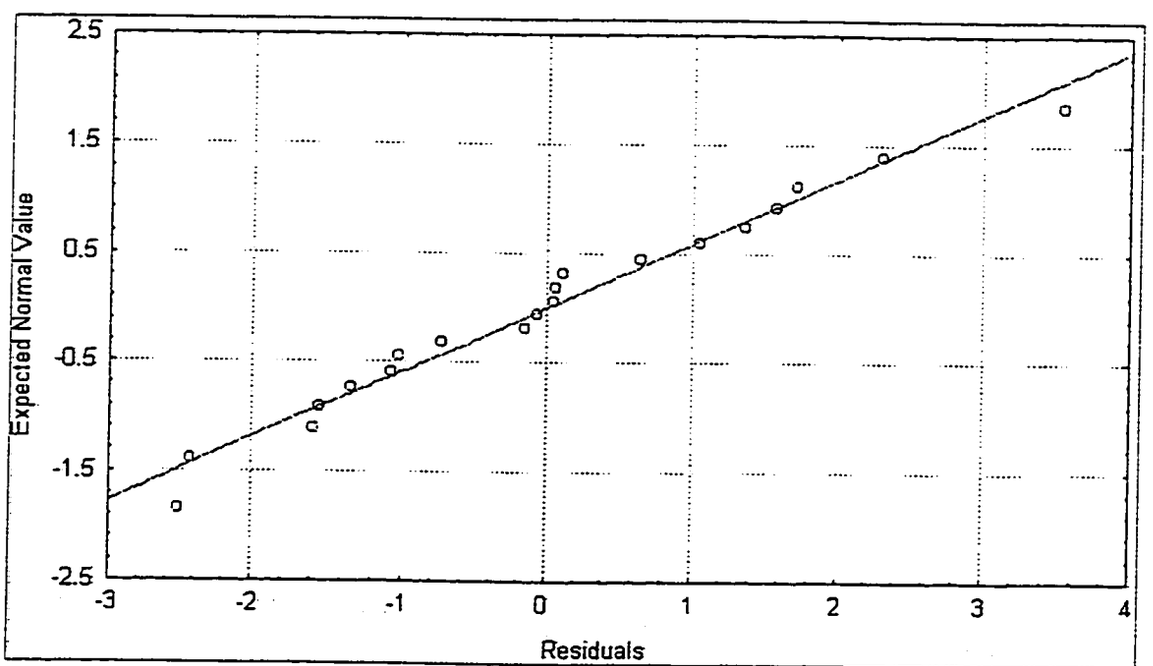

Figure H.30: Normal Probability Plot of Residuals for Initial Methane Concentration Model

APPENDIX I:
Sample Calculations

This appendix is a listing of sample calculations for all calculated variables reported in this work.

I.1 Study of COD Variability of Sewage Sludge

In the case of RAW on Monday at 9:00 am, the test variance is calculated as:

$$\sigma_T^2 = (\text{difference})^2/2$$
$$\sigma_T^2 = (12)^2/2$$
$$\sigma_T^2 = 72$$

The pooled variance is

$$V_T = \frac{\Sigma(\sigma_T^2)}{DT(T-1)}$$

where D = number of days

T = number of time of days studied

$$V_T = [72 + 2 + 2 + 2 + \ldots + 18 + 0 + 2] / [5(3)(2-1)]$$
$$V_T = 18$$

I.2 Batch Test Study

All calculations are carried out for the control study bottle 33.

$$\text{Coded } X_1 = (X_1 - 4) / 2$$
$$\text{Coded } X_1 = (2-4) / 2$$
$$\text{Coded } X_1 = -1.000$$

Consider the cumulative biogas production model.

$$Y = \begin{bmatrix} 2.607 \\ 2.820 \\ 8.080 \\ 5.213 \\ 4.009 \\ 2.371 \\ 2.150 \\ 7.717 \\ 4.468 \\ 4.602 \\ 4.660 \\ 4.629 \end{bmatrix}, \quad X = \begin{bmatrix} 0 & -1.000 & -1.000 & 1.000 & 1.000 & 1.000 \\ 0 & 1.175 & -1.000 & 1.381 & 1.000 & -1.175 \\ 0 & -1.000 & 1.000 & 1.000 & 1.000 & -1.000 \\ 0 & 1.175 & 1.000 & 1.381 & 1.000 & 1.175 \\ 0 & -1.575 & 0 & 2.481 & 0 & 0 \\ 0 & 2.000 & 0 & 4.000 & 0 & 0 \\ 0 & 0 & -1.414 & 0 & 1.999 & 0 \\ 0 & 0 & 1.414 & 0 & 1.999 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 \end{bmatrix}$$

The least squares estimates of the parameters are estimated by minimizing the sum of squares of residuals, that is,

$$\beta_{hat} = (\underline{X}_T\underline{X})^{-1}X^TY$$

$$X_T X = \begin{bmatrix} 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 11.242 & 0 & 5.338 & 0.350 & 0 \\ 0 & 0 & 7.999 & 0 & 0 & 0.350 \\ 0 & 5.338 & 0 & 27.970 & 4.762 & 0 \\ 0 & 0.350 & 0 & 4.762 & 11.992 & 0 \\ 0 & 0 & 0.350 & 0 & 0 & 4.761 \end{bmatrix}$$

$$X_T Y = \begin{bmatrix} 0 \\ -2.820 \\ 15.738 \\ 41.211 \\ 38.444 \\ -2.661 \end{bmatrix}$$

Hence,

$$\beta_{hat} = \begin{bmatrix} 4.086 \\ -0.365 \\ 1.771 \\ -0.309 \\ 0.250 \\ -0.626 \end{bmatrix}$$

Subtracting the observed from the predicted values for the biogas production results in

$$\text{Re}siduals = \begin{bmatrix} 0.612 \\ 0.375 \\ 1.291 \\ 0.697 \\ 0.115 \\ 0.251 \\ 0.068 \\ 0.627 \\ 0.382 \\ 0.516 \\ 0.574 \\ 0.543 \end{bmatrix}$$

Calculating the mean,
Mean = (2.607 + 2.820 + 8.08 + ... + 4.660 + 4.629) / 11
Mean = 4.848

Mean of replicates = (4.468 + 4.602 + 4.660 + 4.629) / 3
Mean of replicates = 4.069

Sum of residuals squared = $(0.612)^2 + (0.375)^2 + (1.291)^2 + \ldots + (0.574)^2 + (0.543)^2$
Sum of residuals squared = 4.78

Sum of squares = $[(2.607-4.848)^2+(2.820-4.848)^2+ \ldots + (4.660-4.848)^2 + (4.629-4.848)^2]$
Sum of squares = 42.351

Sum of squares of regression = 42.351 - 4.78
Sum of squares of regression = 37.571

Pure error variance = $[.382^2 + .516^2 + .574^2 + .543^2]/3$
Pure error variance = 1.037

So, calculating the co-variance matrix,

$$(X^T X)^{-1} \sigma^2 = \begin{bmatrix} 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0.067 & 0 & 0.032 & 0.002 & 0 \\ 0 & 0 & 0.048 & 0 & 0 & 0.002 \\ 0 & 0.032 & 0 & 0.168 & 0.029 & 0 \\ 0 & 0.002 & 0 & 0.028 & 0.072 & 0 \\ 0 & 0 & 0.002 & 0 & 0 & 0.029 \end{bmatrix}$$

The correlation between any two parameters can be estimated by

$$\rho_{hat}(\beta_{i,hat}\beta_{j,hat}) = c_{ij}/(c_{ii}c_{jj})^{0.5}$$

where
cij is the element of the covariance matrix in row i and column j.

Hence, the correlation matrix is

$$\rho_{hat} = \begin{bmatrix} 1.000 & 0.164 & 0 & -0.624 & -0.666 & 0 \\ 0.164 & 1.000 & 0 & -0.336 & -0.071 & 0 \\ 0 & 0 & 1.000 & 0 & 0 & -0.057 \\ -0.624 & -0.336 & 0 & 1.000 & 0.262 & 0 \\ -0.666 & -0.071 & 0 & 0.262 & 1.000 & 0 \\ 0 & 0 & -0.057 & 0 & 0 & 1.000 \end{bmatrix}$$

Calculating the coefficient of determination

$R^2 = 37.571 / 42.351$

$R^2 = 0.8871$

$R = 0.942$

Calculating lack-of-fit,

$SS_{lof} = SS_{residuals} - SS_{pure\ error}$

$SS_{lof} = 4.78\text{-}1.04$

$SS_{lof} = 3.74$

Calculating mean squares,

$\text{Mean Square}_{regression} = 37.571 / 5$

$\text{Mean Square}_{regression} = 7.514$

$\text{Mean Square}_{residuals} = 4.78/6$

$\text{Mean Square}_{residuals} = 0.80$

$\text{Mean Square}_{lof} = 3.74/3$

$\text{Mean Square}_{lof} = 1.24$

$\text{Mean Square}_{pure\ error} = 1.037/3$

$\text{Mean Square}_{pure\ error} = 0.346$

Calculating F,

$F_{regression} = 7.514/0.80$

$F_{regression} = 9.39$

$F_{residuals} = 1.24/0.346$

$F_{residuals} = 3.584$