

Fresh and hardened properties of cemented paste backfill with ternary binder.

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ABSTRACT

The mining industry is a major economic driver and job creator for many countries. However, mining is associated with geo-hazards and environmental issues, such as the disposal of large volumes of waste, acid mine drainage, and ground subsidence. As a result, efficient mining waste management is crucial for sustainable development. The geotechnical, economic, and environmental benefits of cemented paste backfill (CPB) have piqued the interest of researchers and academicians worldwide, making it an essential aspect of underground mining management. CPB is a thickened cementitious combination of dewatered tailings (70 - 85 wt.%), binders (usually 3 to 8% wt.%), and water used to backfill mine waste into underground mining stopes. Despite being used in small amounts, the cost of cement makes up to 80% of the cost of backfilling operations. In addition, clinker production accounts for 5-8% of global human created carbon dioxide (CO₂) emissions. This predicament necessitates the development of a viable alternative to cement. Partially substituting cement with supplementary cementitious materials like fly ash, blast furnace slag, natural pozzolans, and other materials has been increasingly prevalent in CPB.

It is evident that the addition of slag to cement can increase the mechanical strength of CPB at the advanced ages but decreases the strength and suction development due to the slow reaction kinetics in the CPB at the early ages, which may negatively affect the mechanical stability of the CPB, mining cycle, and safety of mineworkers. Moreover, the supply of these materials is limited and may not be enough for the future needs of the industry. Furthermore, there has been a surge in interest in using limestone powder (LS) owing to its abundance, low cost, and lack of environmental costs which are associated with Portland cement – Type 1 (PCI). The addition of LS accelerates hydration at the early ages, thus resulting in high early strength, but the dilution effect can reduce the late strength. The combination of LS and slag in a ternary blended cement can be potentially used as a binder for CPB with acceptable strength development at the early and advanced ages while lowering the cost of the CPB and the carbon footprint of the mining industry.

Nevertheless, the rheology, mechanical strength, and stability are important key performance quality criteria for CPB; however, the effect of ternary cement blends on these parameters is not well known. In this research program, the impact of the binary and ternary cement blends on (i) the fresh properties of CPB, such as the rheological properties (yield stress, viscosity) and setting time, and (ii) the strength and suction development of CPB are investigated. To understand the effect of substituting slag with LS in the binary binder in the first phase of the study, binary binders with two different PCI: Slag proportions of 50/50 and 80/20 are examined with no limestone, followed by replacing slag with an increasing amount of LS from 0 to 20 wt. % of the total binder, with a constant cement content, over a period of 4 hrs (0, 0.25, 1, 2, and 4 hrs) of curing at room temperature. In the second phase, the effect of a ternary binder (PCI-Slag- LS) with varying proportions on the suction development and the mechanical behavior of hardened CPB is investigated over a curing period of up to 90 days. The changes in strength of these binary and ternary binders on the CPB sample are tested for 1, 3, 7, 28, 60, and 90 days. An unconfined compression test (UCS) is conducted to evaluate the strength development. The microstructure of the mixes is examined through mercury intrusion

porosimetry (MIP) for changes to the pore structure. Finally, the results from both phases are validated through monitoring for the development of hydration and suction, electrical conductivity (EC), and temperature, which is carried out for up to 30 days. This is followed by a microstructure analysis with a thermogravimetric/differential thermogravimetric test on fresh and hardened samples.

The results of the first phase show that an increase in the percentage of substituted cement in the binary binder (from PCI/Slag 80/20 to 50/50) increases the yield stress of the CPB but decreases the viscosity of the mix. However, the addition of LS as a substitution for slag shows a considerable decrease in the yield stress of the control mix with an increase in viscosity with increasing dosages of LS, thus indicating an improvement in the flowability of CPB. The second phase results indicate that the slow hydration kinetics of slag influences early age suction and strength changes in the binary sample with a high slag content (50/50); however, its latent hydraulic and pozzolanic properties enhance strength gain after 28 days. The addition of 5% limestone to the ternary blend increases the strength gain by up to 7 days compared to the binary control samples. Indeed, the presence of LS influences the rate of hydration of cement and slag through both physical (filler, nucleation, dilution) and chemical (hydrate) effects. However, substituting more than 10% LS for slag affects the mechanical performance at all ages. Overall, up to 50 wt.% slag and 10 wt.% limestone with cement as a ternary binder can be used without significant compressive strength loss.

This study demonstrates that the partial substitution of ordinary Portland cement (OPC) with varying percentages of slag and LS is complementary, and overall, the interaction of slag and LS is observed. The optimal use of LS and slag in a ternary system may serve as a sustainable alternative to the commonly used OPC and PCI/Slag binders, thereby reducing the energy consumption and carbon footprint associated with cement. The findings of this study will ultimately help to develop a better understanding of the impact of ternary blends with increasing percentages of LS on the rheology and setting time of CPB mixes and mechanical strength changes in designing an efficient mixing plant, particularly its transport system.

DEDICATION

To my beloved daughter Smira

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List of Symbols and Abbreviations

AMD: Acid mine drainage
CH: Calcium hydroxide
CPB: Cemented paste backfill
C-S-H: Calcium silicate hydrate
C₂S: Dicalcium silicate or belite
C₃A: Tricalcium aluminate
C₃S: Tricalcium silicate or alite
C₄AF: Tetracalcium alumina ferrite
DTG: Differential thermogravimetry
EC: Electrical conductivity
FA: Fly ash
GGBS: Ground granular blast-furnace slag
hrs: Hours
LS: Limestone powder
min: Minutes
MIP: Mercury intrusion porosimetry
9MT: Nine mine tailings
OPC: Ordinary Portland cement
PC: Portland cement
PCI: Portland Cement – Type I
PSD: Particle size distribution
sec: Seconds
Slag: Blast furnace slag
ST: Silica tailings
TGA: Thermogravimetric analysis
Temp.: Temperature
TG: Thermal gravimetry
UCS: Unconfined compression strength
UCSc: Critical unconfined compression strength
w/c: Water-to-cement ratio
wt.%: Percentage of total dry weight

1. Chapter 1. Introduction

1.1 General Statement

The mining industry is the primary source and supplier of many types of precious minerals and metals to meet the needs of modern society. The industry creates jobs and is an important sector and a significant contributor to economic development for many countries across the world. Mining is a billion-dollar industry and a pillar of the national economy of Canada. The mineral sector including metallic and non-metallic minerals, directly and indirectly contributed CAN\$125 billion to the economy in 2021, or 5% of the total nominal GDP of Canada (Government of Canada (2022)). The sector has also accounted for over 665,000 jobs in 2021 throughout the country (Government of Canada (2022)). However, extracting and processing mineral resources from rocks generate a tremendous amount of waste in the form of waste soil and rock, and tailings. Also, underground mine operations create huge openings (stopes), which may cause geotechnical problems, like ground subsidence and instability in the nearby areas, and threaten the safety of miners. As tailings can contain residual chemicals, heavy minerals, and poisonous substances, traditional methods of tailings disposal, like in surface tailings ponds/dams, may cause environmental issues and acid mine drainage (AMD); hence the proper transport and disposal of tailings are crucial (Ercikdi et al., 2009; Wu et al., 2013).

Traditional methods of mine waste management like surface storage, including in tailings dams, embankments, or piles, can be a threat to the environment and nearby communities (Jiao et al., 2017; Xiao et al., 2021), and their failure may lead to geotechnical problems and economic losses (Fall & Pokharel, 2010). Two important factors that influence the changes in the modern management practices of mining tailings, particularly in North America, are legislation that results from the failure of tailings surface storage facilities and the current growing public awareness of the environmental effects of acid rock drainage (Cincilla et al., 1997). These factors have significantly contributed to the development of mine backfilling technology. Underground backfilling methods involve returning material removed during extraction to the underground part of the mine, thus reducing the environmental impact, improving the ground conditions, and creating a safer working environment for workers (Benzaazoua et al., 2010; Nasir & Fall, 2008). Moreover, backfilling reduces the acid mine/rock drainage risk and allows restoration of the actual landscape (Potvin et al., 2005). This waste material is often mixed with types of cement and/or pozzolanic binders to enhance its properties. This modern underground backfilling technology, i.e., cemented paste backfilling, is becoming increasingly popular, as the older method, cemented hydraulic backfilling, can cause serious geotechnical technical hazards and incur high costs. Although relatively new, cemented paste backfilling is widely used

around the world, due to its technical, environmental, and economic benefits (Fall & Pokharel, 2010; Kesimal et al., 2005).

Cemented paste backfill (CPB) is a cementitious material made up of three main components: tailings (70%–85% of the total solids), water (fresh or processed), and relatively small amounts of binder (usually 2–8 wt.% of the total solids) (Haruna & Fall, 2020; Jiang & Fall, 2017; Kesimal et al., 2005). The CPB mix is prepared in the mixing plants generally located on the surface of the mine and then transported to the underground stopes by gravity and/or pumping through the pipelines (Ali et al., 2021; Liu & Fall, 2022). Filling the stopes/voids after backfilling provides underground support, thus providing a safe mining environment and reducing the quantity of mine waste on the surface. This also reduces the cost of constructing tailings storage facilities on the ground surface and improves the recovery ratio due to the supporting CPB structures. (Benzaazoua et al., 2004; Cui & Fall, 2016a; Sivakugan et al., 2006). Due to their advantages, many countries, including Canada and Australia, are increasingly using CPB in modern mining as a cleaner and more sustainable tailings management strategy (Sivakugan et al., 2006, 2015). The key engineering design criteria are mechanical stability, transportability or flowability, and cost. The mechanical stability of CPB structures is usually evaluated by using the uniaxial compressive strength (UCS) of the CPB material, because UCS tests are fast, cheap, and reliable (Orejarena & Fall, 2008). In addition, the development of suction in the mass of CPB can lead to the generation of significant effective stresses in CPB slab structures and thus improve their resistance to liquefaction at the early ages, which is critical for the early opening of barricades (Cui & Fall, 2016b). The flowability of CPB is determined by its rheological properties (yield strength, viscosity), while its cost is primarily controlled by the binder consumption. A longer setting time will result in the loss of workability and the start of paste backfill stiffening or strength gain. Knowledge of setting time is important to the overall productivity, and mechanical and economic performances of backfill operations. The changes in the setting time of CPB is investigated by using the Vicat apparatus which is based on the principle of the resistance of the mixture to needle penetration.

1.2 Problem Statement

The most common binder used in CPB is Portland cement (PC). According to the literature, the cement industry currently accounts for up to 8% of total carbon dioxide (CO₂) emissions worldwide (Filippo et al., 2019; Gartner & Hirao, 2015; Kajaste & Hurme, 2016). CPB can account for up to 20% of the total mining costs, with cement accounting for up to 80% of the backfill contents (Benzaazoua et al., 2010; Grice, 1998). As a result, a 1% reduction in cement content may reduce 1.6 to 2% of the overall mining costs (Sadrossadat et al., 2020). To validate this proposition, the use of supplementary cementitious materials (SCMs) like blast-furnace slag (Slag), fly ash (FA), and filler materials, such as limestone (LS) is prevalent and well investigated in concrete and cement-based systems. (Carrasco et al., 2005 ; Courard & Michel, 2014 ; Vance

et al., 2013a; Zheng et al., 2016). In CPB, the use of Slag as a partial substitution for cement has been increasingly adopted in CPB technology, due to its cost-effectiveness and ability to improve the microstructure and strength (at the later ages) of systems (Li & Fall, 2018; Pokharel & Fall, 2011; Solismaa et al., 2021). However, considering the technological advancements in steel recycling plants, the shutdown of coal combustion plants may reduce the availability of Slag and FA at the source, supply of FA, and slag may not be able to meet the future needs of the industry (Bentz et al., 2017; Mehta et al., 2010). Moreover, since slag is a latent hydraulic product, it can negatively affect the early strength of the cementitious mixture. Early strength gain is essential for the mechanical stability of the underground mine cavities as well as for the early opening of the barricades. This latter point is essential to shorten the operating cycles and thus increase the productivity of the mine. Furthermore, in addition to this, increasing the percentage of slag has resulted in increases in the yield stress, which affects the functional transport/pumping delivery of CPB to the stopes. This may possibly cause a delay in flow and/or clogging of the pipeline or damage, thus leading to significant financial losses and production interruption (Haruna & Fall, 2020; Li & Fall, 2016; Wu et al., 2013; Xiao et al., 2019).

In contrast, there is more availability of mineral fillers like limestone which is less expensive than cement and slag (Bentz et al., 2017; Wang et al., 2018). It is observed from the literature that limestone is effective in improving the rheological behavior and workability of (cement, mortar, concrete) cementing systems and also an enhancer of early age strength (Courard & Michel, 2014; Derabla & Benmalek, 2014; Jiang et al., 2020a; Vance et al., 2013a). However, limestone negatively affects the later age strength of the cement mix (Ramezaniapour et al., 2009).

Considering the above advantages and limitations of both slag and LS, a ternary blend of, cement, slag and LS could counterbalance the limitations, and the interactive effect may further improve the overall performance of CPB (rheological and mechanical). Indeed, the use of ternary binders in cement, concrete, and mortar is well established. However, CPB is different from these materials. The changes in the strength of CPB with a ternary mix is not well known, and no studies have been conducted to evaluate the effect of a ternary binder on the rheological properties, setting time and suction of CPB. These knowledge and technological gaps are addressed in this thesis.

1.3 Research Objectives

The main objective of this research is to investigate the influence of a ternary binder (cement, slag, LS) on the fresh (i.e., rheology and setting behavior) properties and mechanical stability, i.e., strength and suction of CPB. This research program is further divided into two phases. The specific objectives of this research work are to evaluate:

- i. the impact of a ternary binder on the rheological behavior of CPB, prepared with 2 different proportions of PCI/Slag, 50/50 and 80/20;
- ii. the effect of different dosages of cement, slag, and LS on the initial and final setting times of CPB;
- iii. the influence of a ternary binder on the changes in the mechanical strength of CPB; and
- iv. the development of suction in CPB samples with a ternary binder and varying dosages of cement slag and LS.

1.4 Research Approach and Methodology

The research problem is identified and formulated as shown in Figure 1.1. The theoretical and technical background information of CPB is first introduced in detail. Subsequently, a comprehensive literature review of previous studies on CPB and its fresh and hardened properties is provided. Furthermore, the use of a binary blend (PCI-Slag) in CPB and its effect on the behaviors of the CPB in fresh and hardened states in the past literature are discussed. Finally, the concept of a ternary binder (PCI-Slag-LS) is described, and its effect on a cementitious system is explained.

Furthermore, an experimentation program to study the effect of the binary and ternary binders on fresh and hardened properties of CPB is discussed. The program is further divided into 2 phases to examine the influence of the ternary binder on fresh and hardened properties. The first phase focuses on the rheological behavior of CPB, and tests are conducted to better understand its yield stress, viscosity, and setting time. The two groups of control CPB samples are prepared by using binary binders with a PCI/Slag ratio of 50/50 (Group A) and 80/20 (Group B) respectively. Additionally, CPB samples with a ternary binder (PCI-Slag-LS), are cast by using an increasing amount of limestone (5,10 and 20 wt.% of the total binder) as a partial substitution for slag. The freshly prepared samples are further tested for yield stress and viscosity over curing times of 0, 0.25, 1, 2 and 4 hrs respectively at room temperature. The aforementioned samples are also examined for changes in setting time (initial and final) and UCS and suction development tests are conducted to understand their mechanical behavior. The details of the experimental program are provided in Chapters 3 and 4.

Finally, all of the results obtained throughout the study are discussed, conclusions are drawn, and future recommendations are provided to advance the current work.

In the second phase of the experimental program, the mechanical stability of the CPB samples is determined by examining their mechanical strength and suction development. To investigate the mechanical strength, the CPB samples are prepared with the same mix proportions used to prepare the samples for the rheological testing, and divided into 2 groups, as explained earlier. The samples are further cured for 1, 3, 7, 28, 60 and 90 days at room temperature, and tested for their UCS. To further investigate the hydration mechanism, the freshly prepared CPB samples are monitored up to 30 days of curing, and their suction development, electrical conductivity (EC), and temperature changes are measured. The suction development is measured by using a dielectric water potential probe (MPS-6), and the temperature and EC are monitored by using a 5TE sensor.

Furthermore, the results obtained from the experimental testing (rheology and mechanical stability) are further validated by conducting microstructural analysis tests, i.e. thermogravimetric/differential thermogravimetric (TG/DTG), mercury intrusion porosimetry (MIP) and monitoring tests, such as monitoring the EC and temperature.

Figure 1.1 Research approach and experimental program

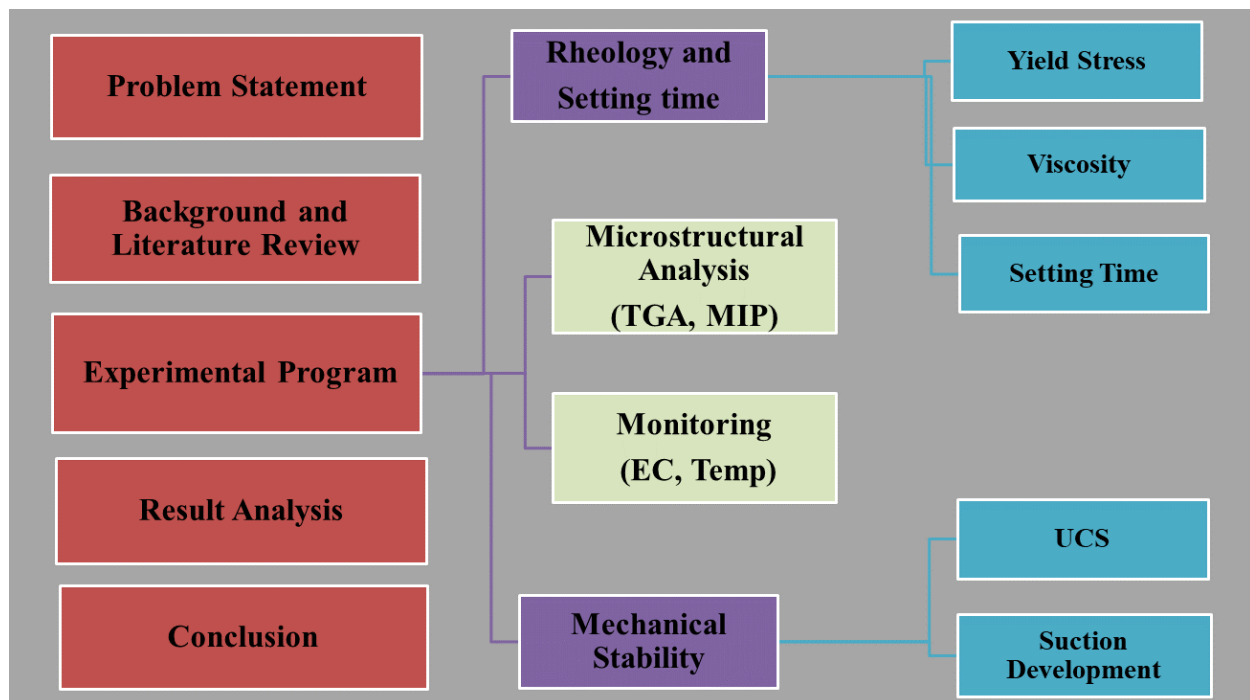


Figure 1.2 Research approach and experimental program

1.5 Thesis Organization

The organization of the thesis is depicted in Figure 1.2. The thesis manuscript consists of 6 chapters, including two research papers.

Chapter 1. Provides a brief introduction about this study and includes the general statement. Defines the problem statement, research objectives and research methods adopted in this study.

Chapter 2. Includes a detailed review of background information on CPB technology. This chapter emphasizes on the importance of studying the fresh and hardened properties of CPB. Furthermore, a review is conducted to study the commonly used binders in preparing CPB, effect of binary binders, and advantages and limitations of these binders. Finally, the concept of a ternary binder (PCI-Slag- LS), and its use in CPB is proposed and a review is conducted based on the past literature.

Chapter 3. Comprises Technical Paper I. This paper mainly presents a study of the effect of the ternary binder on the fresh properties of CPB, i.e., rheological properties and setting time. The effect of using a binary cement blend (PCI-Slag) on the rheological properties are discussed in Section 3.4.1, while the development of the rheological properties of CPB with ternary binders is presented in Section 3.4.2.

Chapter 4. Includes Technical paper 2. In this paper, the effect of ternary binders on mechanical stability (strength and suction development) in CPB is investigated through UCS testing and suction development monitoring. Furthermore, the results obtained for CPB samples prepared with ternary cement binders are compared with CPB made with binary binders.

Chapter 5. Presents a summary of the results obtained in this study and provides insights into the effect of the use of ternary binders on fresh and hardened properties of CPB. This is followed by a discussion of the implications of designing a practical CPB system, its environmental performance and cost management.

Chapter 6. Presents a summary of the conclusion and recommendations for future studies.

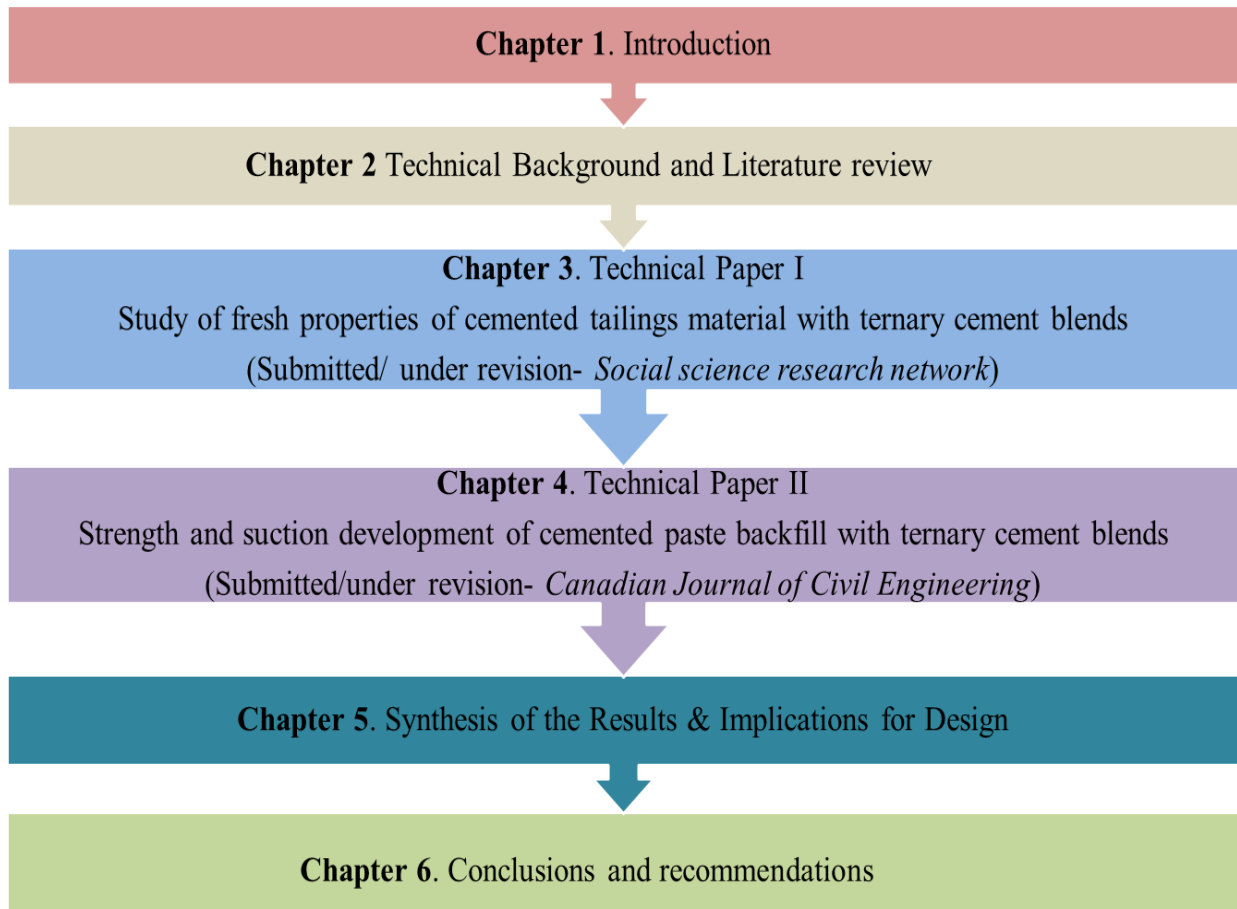


Figure 1.3 Thesis organization flowchart

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2. Chapter 2 Technical Background and Literature Review

2.1 Introduction

This chapter discusses the fundamentals of mining (Section 2.2) and introduction to mine backfilling technology (Section 2.3) with an emphasis on CPB. In addition, the primary binders used in CPB and a mineral filler - limestone, and their hydration process are explained (Section 2.4). Subsequently, the concept of a binary binder (PCI-Slag) and previous studies on the effect of the binary binder (PCI-Slag) on the engineering properties (e.g., rheological, mechanical, suction) of CPB are discussed (Section 2.5). Finally, the concept of a ternary binder (PCI-Slag-Limestone) and its effects as a binder on the cementitious system are explained (Section 2.6). Furthermore, the potential use of PCI-Slag-Limestone in the CPB system is discussed to understand its possible effect on the hydration process, rheology, and strength changes of CPB.

2.2 Fundamentals of Mining

Mining is a multi-billion-dollar industry and major economic activity in many countries like Australia, Canada, China, India, and South Africa. However, mining generates millions of tons of waste each year. The mining industry uses different processes to access, extract and manage valuable mineral ores from the Earth's crust (Hussain et al., 2022). Open-pit mining is used to recover shallow ore, while deep mineral resources are extracted by underground mining. With the scarcity of surface minerals and technological advancements in searching valuable minerals, mining is now advancing into deeper levels that can be up to 4350 m in depth (Mponeng Gold Mine, South Africa) (Ziegler et al., 2015). Millions of metric tons of mining products are extracted worldwide. Figure 2.1 shows the top countries in mining product extraction in 2018. However, the removal of ore from the ground results in significant amounts of mine waste (e.g., tailings) (Kesimal et al., 2005) and significant underground voids. In metal mining, the waste-to-product ratio is frequently 100:1, and in some cases, can be as high as 1,000,000:1 (Dudeney et al., 2013; Qi & Fourie, 2019). Conventional methods of tailings disposal, like piling, stacking in tailings dams, and surface impoundment, may have adverse environmental and geotechnical impacts (Wu et al., 2013; Yilmaz et al., 2003), including groundwater contamination and AMD in some cases (Edraki et al., 2014; Yilmaz & Fall, 2017), and geotechnical hazards like tailings dam failure and large voids that can potentially collapse (Bloss, 2014).

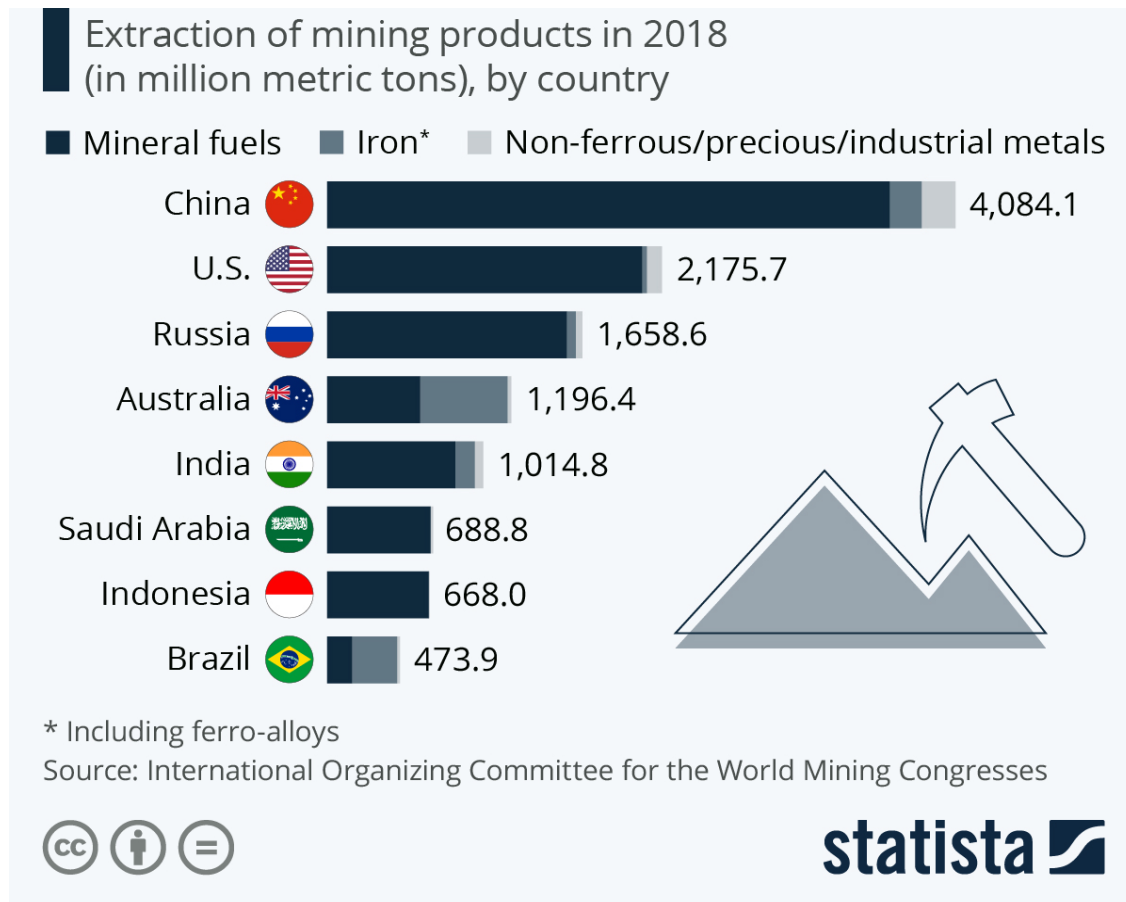


Figure 2.1 Extraction of mining products in 2018 (in million metric tons), by country (Buchholz, K. 2020)

2.3 Introduction to Mine Backfilling and Backfill Types

Backfilling is widely used in Canadian metal mines and defined as the process of filling underground voids with waste material for disposal or engineering purposes (Rankine et al., 2007). Backfilling is the best way to dispose of mine waste, create a safe work environment, and support underground operations (Sivakugan et al., 2015). Approximately 69% of all of the underground mining operations in Ontario (65% of the operations in Canada) have used some form of backfill support (Mining Source Book, 1994). As shown in Figure 2.2, there are three main types of backfills that are commonly used: rock, hydraulic and cemented paste backfills (Table 2.1) (Nasir & Fall, 2008b; Sivakugan et al., 2015). The type of backfill used is based on their characteristics, as well as economic factors and future mine-related goals (Sheshpari, 2015a).



Figure 2.2 Main backfills used: (a) Rock, (b) Hydraulic, and (c) Paste backfills (Yilmaz & Guresci, 2017)

2.3.1 Hydraulic backfill

Hydraulic backfill is a mix of tailings, river or crushed sand and/or rock materials, etc. mixed with water and a binder (Sheshpari, 2015a; Sivakugan et al., 2015). It is prepared on the surface of the mine or underground and transported through the hydraulic head generated by gravity or pipeline distribution systems, with water as the medium of transport. The maximum particle size of the materials is less than $1\text{ }\mu\text{m}$, and less than 10% of the fine particles is smaller than or equal to $10\text{ }\mu\text{m}$, and in most cases, less than that (Potvin et al., 2005). Hydraulic backfill, also called slurry backfill, has high permeability between 10^{-5} to 10^{-6} m/s and low density between 40-50% and 60-70% of solids by weight (Grice, 1998) and can be either cemented or non-cemented.

Some of the advantages of using hydraulic backfill are:

- i. Simple installation and operation,
- ii. Easier to control over backfill material, and
- iii. Less costly transport through gravity.

However, drainage (gravity or mechanical) is required in hydraulic backfilling to remove excess water in the backfill material. Therefore, when small waste particles are available, hydraulic backfilling with non-cemented materials is one of the most cost-effective mining methods (Potvin et al., 2005). Moreover, hydraulic backfill is more susceptible to liquefaction, which may severely threaten work safety in underground mines. The drawbacks of this backfilling method include a significant amount of excess water, cement segregation from the solid phase, and cement washout (Ghirian, 2016; Landriault, 2001).

2.3.2 Rock backfill

Rock backfill contains a mix of materials including waste rocks, stone, gravel, and tailings, which can be used in their cemented or uncemented form (Wang et al., 2013). In the case of cemented backfill, generally 4% to 8% cement is added. Cement paste can be added through pipelines and mixed with waste rock before placement. The raw material is typically prepared by crushing, sieving, and mechanical mixing according

to the particle size distribution (PSD) pattern. This method is feasible and cost-effective, with benefits such as creating usable land by transferring massive waste underground, reducing environmental pollution, increasing stope stability, and reducing the possibility of land subsidence by providing additional support (Table 2.1). However, the disadvantages associated with rock backfilling are (Wang et al., 2013; Belem & Benzaazoua, 2007):

- i. Use of expensive equipment and is highly labor intensive,
- ii. Difficult to control the segregation and achieve good quality control, and
- iii. Complex plant structure and low efficiency.

2.3.3 Cemented paste backfill and key properties.

In 1957, The Bad Grund Mine in Germany used cemented paste backfilling for the first time (Lerche, 1984) to reduce solid waste on the mine surface by reusing up to 60% of the extracted material underground as the primary support element. Then, the Hecla Mining Company implemented cemented paste backfilling at its Lucky Friday Mine in the United States. In the 1990s, CPB successfully replaced rock and hydraulic fills and was used in Australian and Canadian mines (Rankine & Sivakugan, 2007). CPB is an innovative cementitious material made with tailings (70–85 wt.%), water (fresh or processed), and a small dosage of hydraulic binder (2–8 wt.%) (Benzaazoua et al., 2004; Edraki et al., 2014; Fall & Benzaazoua, 2005; Haruna & Fall, 2020a; Jiang & Fall, 2017a) (Figure 2.3, Table 2.1).

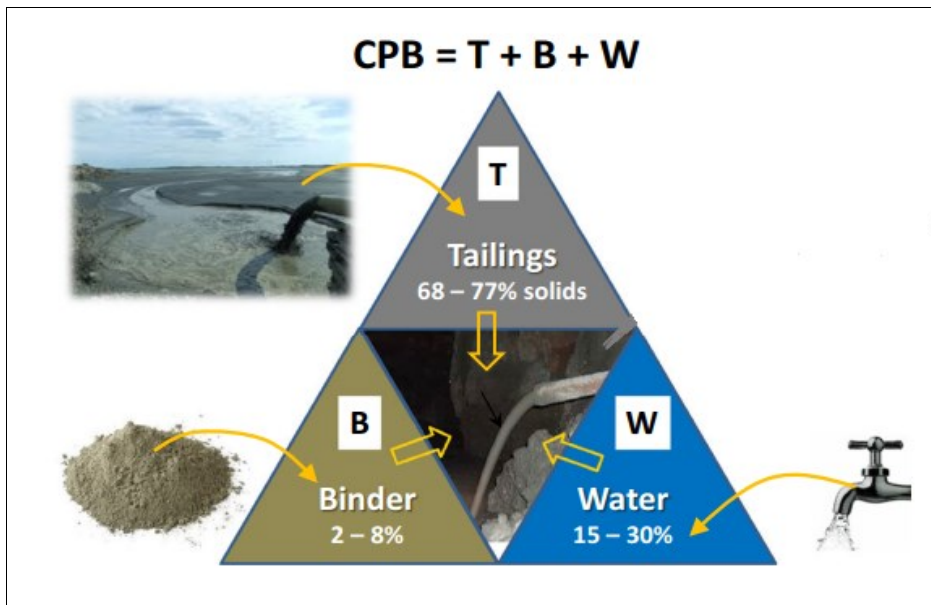


Figure 2.3 Preparation process of cemented paste backfill mixture (modified) (Belem et al., 2016)

This mix behaves like a non-Newtonian fluid and requires less water for its preparation, thus allowing more rapid strength gain (Grice, 1998). CPB mix is usually prepared on the surface of the mine in the backfill plant and transported to the underground stopes through pipes (Figure 2.4). The mixture should contain 70-85% solids of its total mass to avoid segregation and settlement of water and tailings particles during transport. At least 15% of the particles in the backfill must be finer than 20 μm (Klein & Simon, 2006; Landriault, 2001, Grice 2001). Once CPB reaches the stopes, it is cured and hardened and ultimately provides support to the adjacent stopes (Ghirian & Fall, 2014) and a safe working place in underground mines (Ercikdi et al., 2009; Fall & Benzaazoua, 2005; Ghirian & Fall, 2015).

Figure 2.4 Basic configuration for underground placement of CPB (Thomas et al., 1979; Belem & Benzaazoua, 2007; Wu, 2020)

- i. Minimizes the risks associated with tailings ponds (failure, and pollution of land and underground water) (Qi & Fourie, 2019).
- ii. Minimizes the environmental footprint of surface tailings ponds, with the maximum density of mine tailings.
- iii. Increases ore recovery rate due to reduction in the mining life cycle (Benzaazoua, et al., 2004; Fall & Pokharel, 2010).

- iv. Increases the stability (local and regional) of underground voids, thus resulting in an increase in mining safety, efficiency, and productivity (Edraki et al., 2014; Jiang et al., 2020; Rankine & Sivakugan, 2007; Schöler et al., 2015; Tariq & Yanful, 2013).
- v. Co-disposal of chemically challenging industrial by-products or wastes, thus reducing negative impacts on the ecosystem (Bloss, 2014; Tikou & Benzaazoua, 2004).
- vi. No water drainage requirements, unlike hydraulic filling (Simon & Grabinsky, 2013).

Table 2.1 Primary underground backfilling methods and their features (modified) (Landriault, 2006; Yilmaz & Guresci, 2017)

Feature	Rock filling	Slurry filling	Paste filling
Placement state	100% solids	60- 75% solids	75-85% solids
Transport system	Raised mobile equipment separates cement system	Borehole or pipeline via gravity	Borehole or pipeline via gravity
Addition of cement	Cement can be added or not added	Cement can be added or not added	Cement is added
Water:cement ratio (w/c)	Small w/c, higher cement strength	Large w/c, very low cement strength	Small to large w/c, low to high cement strength
Placement rate	100- 400 tons/h	100- 200 tons/h	50- 200 tons/h
Segregation	Stockpile segregation, reduced strength	Slurry settlement segregation, low strength	No segregation
Stiffness	High stiffness	Low stiffness	Low to high stiffness
Strength	High	Low	Moderate
Grain size	>20 cm	>20 μm (≥ 90 wt%)	< 20 μm (≥ 15 wt%)
Capital cost	High	Low	High
Operating cost	High	Moderate	Low
Barricade	No	Yes, but expensive	Yes, but less expensive
Tight filling	Yes - Hard to tight filling	No	Yes

2.3.3.1 Rheology of CPB

As mentioned earlier, CPB mixtures are prepared in a paste backfill plant, typically located on the surface of the mine, and transported underground through pumping, gravity, or a combination of the two (Ali et al., 2021a; Liu & Fall, 2022). The delivery system is chosen based on the amount of energy required to deliver the mixture. Moreover, with technological advancements, the mining industry is advancing into deeper levels. Hence, the practical and efficient transport of CPB is challenging and an economic concern for the overall functioning of the CPB plant (Ali et al., 2021a). Hence, the flowability of fresh CPB is a key concern in the transport of CPB to the mine stope. It is critical as poor flowability can cause clogging

of the pipelines, ultimately a delay in transport, and financial losses (Li & Fall, 2018; Wu et al., 2013). The efficient transport of CPB depends on its rheological or the flowability characteristics of the fresh mix. Rheology is the study of the flow and deformation of materials under the influence of external forces or stresses (Barnes, 2000). The yield stress and viscosity of fresh paste backfills are the main rheological parameters used to determine the flowability, pumpability, and pourability of backfill (Ali et al., 2021b; Jiang et al., 2020; Li & Fall, 2018; Liu & Fall, 2022).

CPB mixes show non-Newtonian behavior; hence the Bingham plastic flow model is used to analyze the rheological properties of CPB. Several studies found that interparticle interactions dominate the rheology of the paste; additionally, particle characteristics such as their size and distribution (i.e., particle-size distribution (PSD)), surface area, diameter, the distance between the particles, packing fraction, and solid volume fraction all have a significant impact on the rheology of concentrated suspensions, including cemented paste backfill (Mehdipour et al., 2017; Rankine et al., 2007; Shanahan et al., 2016; Sivakugan et al., 2015). The flowability of CPB mixes is mainly related to their yield stress and viscosity of the mix.

a) Yield stress

Yield stress is defined as the ability of a material to flow and can be correlated with its pumpability and transportability (Wang et al., 2018). It can also be defined as the stress required to initiate the flow. Previous research has shown that the yield stress of CPB is time-dependent and influenced by external factors such as temperature and loading pressure as well as internal factors such as pH, density and concentration of the CPB mixtures, characteristics of the CPB mixture constituents, surface potential of the solid particles (Haiqiang et al., 2016; Wu et al., 2013; Yin et al., 2012). Niroshan et al. (2018) studied the effect of two different binders and varying solid contents from 72 to 78% on CPB. An exponential increase in yield stress was observed, while the slump decreases with an increase in solid content. Wang et al. (2018) indicated that the yield stress increases with the mass fraction and cement content in the CPB mix, which supported the findings in Boger (2013).

The yield stress can be measured by using direct (rheometric) or indirect experimental measurement techniques. The latter involves yield stress calculation by extrapolating a set of shear stress vs. shear rate data to zero shear rates. This is followed by direct data extrapolation and extrapolation with flow models, such as the Bingham model based on a linear relationship between shear stress and shear rate, and nonlinear relationship with the Casson model, and Herschel and Bulkley model. The disadvantages of the indirect measurement techniques include sensitivity to the model parameters and lack of data (experimental) availability at low shear rates (Tao et al., 2020). On the other hand, in the direct measurement method, the yield stress is directly measured by shearing material at a slow rate instead of using shear stress and shear rate data. The peak shear rate at which flow is initiated is recorded as the yield stress.

The direct yield stress measurement methods include testing materials through stress relaxation, creep/recovery, stress growth, cone penetration test, the vane method, and methods based on the static equilibrium of an immersed body. In this thesis, the simple vane method is used.

The vane shear method is commonly used to directly measure the yield stress owing to the geometry of the vane which causes the least amount of disturbance when inserted into the sample before measurement to eliminate wall slips. This method is regarded as one of the most effective and precise direct measurement methods for assessing the yield stress of soil and Newtonian fluid (Elaty & Ghazy, 2012).

In recent years, this method has been extensively used for different construction materials, such as concrete and mortars (Bauer et al., 2007). The vane shear test is conducted by completely inserting a four-bladed vane (H:D=2:1) into the cylindrical sample, as shown in Figure 2.5. Subsequently, the vane is rotated at a slow speed. Then, torque is applied to the blades, and the resistance of the sample against the vane blades is measured by using the attached deflection spring. The torque increases continuously until reaching the maximum value, T_m , when the material yields. Furthermore, converting the torque to a unit shearing resistance, the yield stress can be calculated by using:

$$\tau_y = \frac{2T_m}{\pi D^3 \left[\frac{1}{3} + \frac{H}{D} \right]}, \text{ where } \tau_y \text{ is the yield stress, } T_m \text{ is the maximum torque, } H \text{ is the length, and } D \text{ is the}$$

diameter of the vane. Both the viscosity and yield stress tests were repeated two times to ensure the accuracy of the results. As CPB is a non-Newtonian fluid, the Wykeham-Farrance vane shear apparatus which is suitable for soil and non-Newtonian fluids is used in the current study. Figure 2.5 shows how the yield stress of the CPB samples is determined. To measure the resistance of the CPB sample to the applied torque, a 4-bladed vane that is 2.5 cm in diameter with blades that are 2.5 cm in height is used. The test was performed in accordance with ASTM D4648/D4648M-16 (2015).

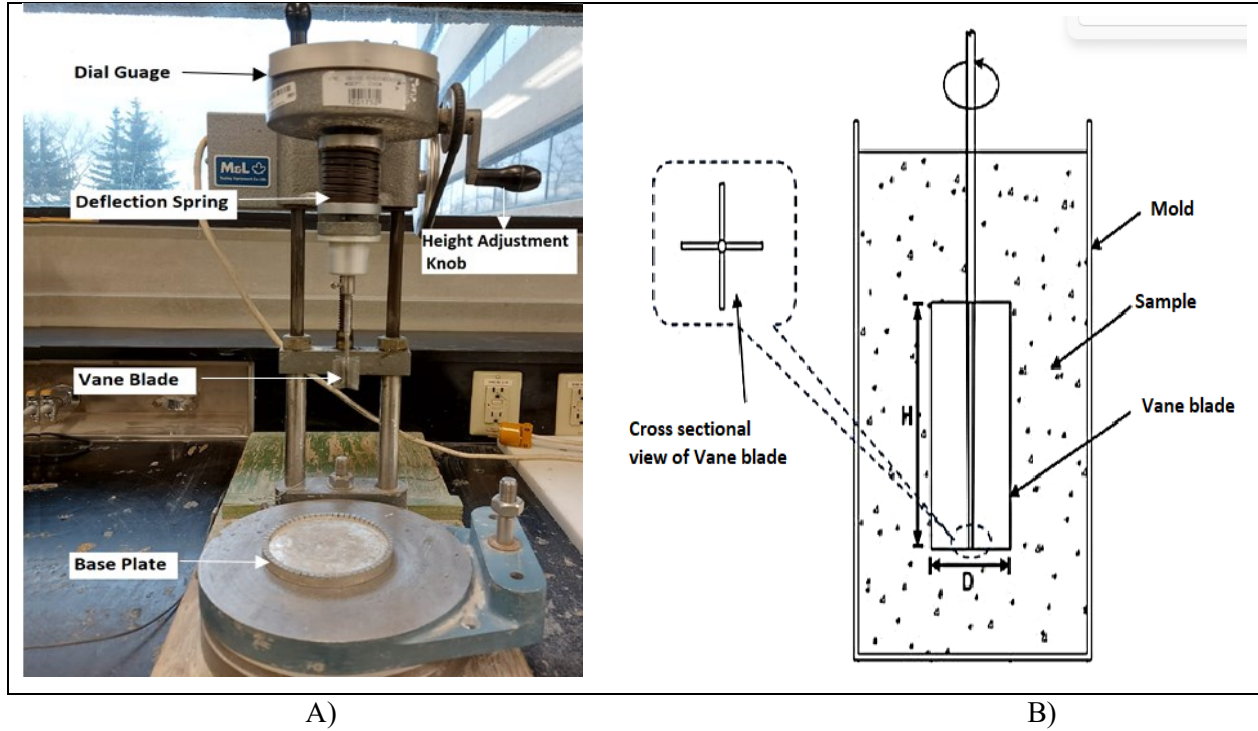


Figure 2.5: A) Vane shear apparatus, and B) Schematic representation of vane test method (Ali, 2021b)

b) Viscosity

Viscosity is a crucial aspect of the rheological behavior of CPB as it is the force required to keep the mix flowing. Viscosity is defined as the measure of the internal frictional resistance of a material and its flow (shear deformation) (Bian et al., 2021; Liu & Fall, 2022). At a specific shear rate, viscosity represents the ratio between the shear stress and shear rate and governs the dynamic flow characteristics of the paste (Ali et al., 2021a; Bian et al., 2021). The viscosity of fluid suspensions can be determined by using different empirical methods like the oscillating-vessel method, during which the viscosity is determined by back-calculating the viscous damping parameters of the material in the vessel with an analytical mathematical model. However, the surface tension and slippage at the liquid/vessel interface may cause a discrepancy in the results. Therefore, a viscometer is generally used to measure the viscosity of the materials. The viscometer measures the shear rate by observing the torque as a function at a predetermined rotational speed. Thus, the viscosity is determined as the ratio of the shear stress to the shear rate at a specific shear rate (Ali et al., 2021b; Sofra, 2017). Different types of viscometers are used, including the Brookfield digital viscometer, Couette viscometer, and coaxial cylinders.

In this study, the Brookfield digital viscometer is used for the viscosity measurements, as shown in Figure 2.6. The device has a rotating spindle and calibrated spring. The spindle number (RV5) is selected based on the paste viscosity ranges, immersed in the paste in a tilted position to prevent air drag, and

rotated at a constant speed (50 rpm). The resistance of the paste to rotation (i.e., viscous drag) is determined by the spring deflection, which is recorded by the rotary transducer (Brookfield, 2014).

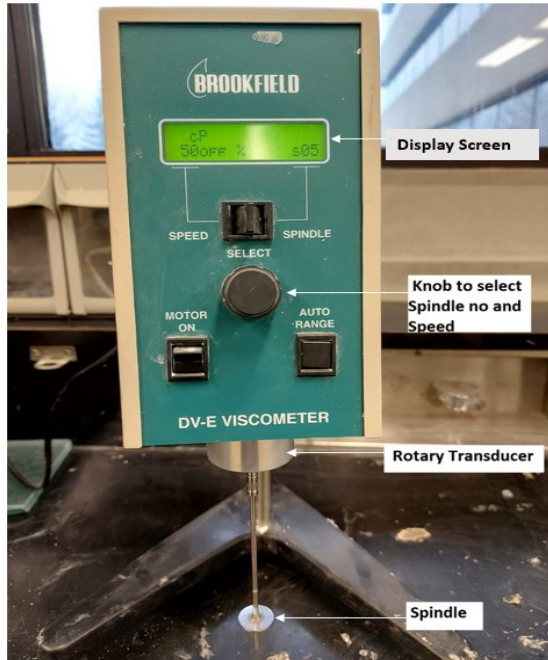


Figure 2.6 Brookfield digital viscometer.

In summary, the rheology of the CPB mix is influenced by several factors, including the mineralogical and chemical compositions of the tailings (Jiang & Fall, 2017b), curing temperature, binder type and dosage, the presence of chemical additives, elapsed time after mix preparation, and mixing time (Ali et al., 2021a; Ercikdi et al., 2009; Fall & Pokharel, 2010; Haiqiang et al., 2016; Haruna & Fall, 2020b; Liu & Fall, 2022; Panchal et al., 2018; Wang et al., 2018; Wu et al., 2013).

Furthermore, Kou et al. (2020) found that both the yield stress and viscosity gradually increase with the progression of curing time and increase in binder content (up to 8%) and temperature (Ali et al., 2021a; Haruna & Fall 2020b; Wu et al., 2013). Additionally, it is observed that adding nano-silica to CPB samples results in an increase in both yield stress and viscosity (Roshani & Fall, 2020); while, adding superplasticizers (ether-based) decreases both the yield stress and viscosity (Haruna & Fall, 2020b). Ali et al. (2021) investigated the effect of adding sodium silicate (SS) on the rheology of CPB and found that increases in yield stress and viscosity are proportional to the increase in dosage of SS, i.e., increasing the amount of SS increases both of these rheological parameters. Li et al. (2020) tested mixes with a cement/tailings ratio that ranged from 1:4 to 1:20, and a slurry concentration of 65, 67, and 69 wt. %. They found that in the fresh state of mixes, the yield stress and viscosity increase with increasing solid content. Liu and Fall (2022) conducted a rheological test on CPB with different mixing times of 1, 2, 4, 7, 10, and 15 min and concluded that the yield stress of the CPB is increased, but the viscosity decreases with increased mixing

time. Panchal et al. (2018) examined the impact of hydration time, binder dosage, and addition of superplasticizer on the rheology of CPB mixtures with a 78% solid mass concentration. They found that the viscosity increases with an increase in hydration time, and solid and binder contents; however, the addition of superplasticizers reduces the yield stress and viscosity of the mix.

2.3.3.2 Setting time

The initial setting time in a cementitious system indicates that the paste has lost its workability and begun to stiffen or gain strength. Therefore, knowledge of the setting time of CPB is critical because it provides vital information on its early age strength development or mechanical stability at the early ages. Furthermore, early strength gains are critical for barricade opening and extraction of neighboring stopes, eventually increasing mining efficiency by shortening the mining cycle (Yin et al., 2012). In this study, the setting time is investigated by using the Vicat apparatus (Figure 2.7). The test with the Vicat apparatus is based on the principle of mixture resistance to needle penetration and performed in accordance with ASTM C191-13. In this test, cylindrical molds of 100 mm in diameter and 40 mm in height were used as the sample containers. The initial setting time was recorded when the needle penetrated a depth of 25 mm from the bottom, and the final setting time was considered when the annular collar attached to the needle did not leave an impression on the specimens.

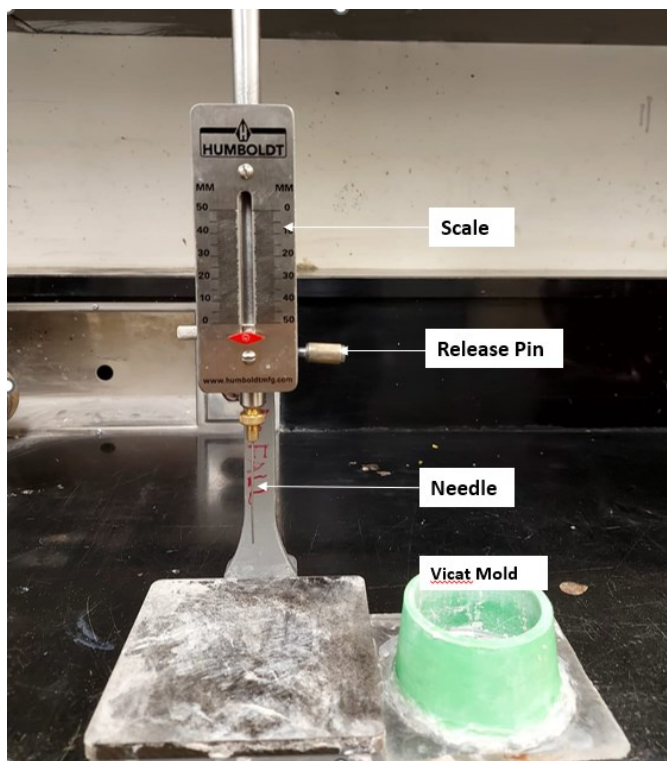


Figure 2.7 Vicat apparatus and mold used for setting time test.

In cementitious systems, many factors influence the setting time, including temperature and the w/c. However, the final setting time of concrete with slag is longer than that of PC concrete, which is approximately 30 minutes longer (The Concrete Society, 2011; cementitious slag makers association, 2012). In another study, Mounanga et al. (2011) found that a mix with 25% slag takes 556 minutes to set, compared to 537 minutes for a control mix with no change in the initial setting time. Furthermore, the addition of 25% and 50% slag resulted in a 21% and 53% reduction in the peak heat output rate, respectively (Mounanga et al., 2011).

The setting of CPB is due to the diffusion of the water molecules and reaction with the solids which result in hydration and hardening of the paste. The setting time may be affected by the type of binder used, its proportion, cement content, solid proportion, water content, and admixture. It has been observed that the setting time is negatively affected by a reduction in the binder (cement) dosage. Klein and Simon (2006) found that reducing the cement content from 7% to 3% increases the initial and final setting times by approximately three times, i.e., reducing the cement content in CPB specimens lengthens the setting time. Furthermore, the amount of water present influences the initial and final setting times of CPB. A 25% increase in water content results in four times longer initial and final setting times. Jafari et al. (2020) made similar observations, in which the setting time of their CPB samples with 3.0%, 5.3%, and 7.5% cement is reduced with an increase in the initial setting time of the cement; that is, initial setting time of 9.18, 7.30, and 5.06 hrs, and final setting time of 28.50, 20.30, and 11.30 hrs, respectively. Indeed, the use of admixtures can also influence the setting process of CPB. Studies in the literature have investigated the effect of adding soluble glass to CPB paste mixtures and noted that the soluble glass acts like an accelerator and improves the initial setting process. When the amount of accelerator is increased from 1.1 wt.% to 3.3 wt.%, the initial setting time decreases from 846 to 108 min (Yin et al, 2012). Chen et al. (2018) examined the possibility of recycling two solid waste materials, phosphogypsum (PG) and construction demolition waste (CDW), as CPB. The results showed that by increasing the solid concentration of CPB from 60% to 70% and adding CDW (from 0% to 40%), the initial/final setting time is decreased from 20% to 30%.

2.3.3.3 Mechanical strength development in CPB

Mechanical stability is the most important quality criteria of hardened CPB and crucial in considering the stability and opening of barricades to prevent collapse, ground subsidence and creating a safe underground work environment. CPB needs to achieve application-specific strength. The most common method to evaluate the mechanical performance/strength of CPB is unconfined compressive strength (UCS) (Fall et al., 2007; Sadrossadat et al., 2020).

In CPB designs, both the early and later age strengths are important. The initial strength is critical for preventing liquefaction and reducing the mining life cycle costs and impacts, and contributes to adequate

long-term strength that would reduce potential collapse and ground subsidence, as well as ensuring the safety of the secondary stopes in mine operations and thus the long-term stability of the mine (Sadrossadat et al., 2020). Researchers have reported a number of strength requirements for cemented paste fills, depending on their specific applications and function in mining operations. In a typical underground mine, the required UCS of CPB usually ranges from 0.7 to 2 MPa (Cui & Fall, 2016). Furthermore, a minimum strength of 0.15 MPa to 0.3 MPa is required at the early curing stages to prevent the risk of liquefaction and barricade collapse. For free-standing CPB structures, the UCS of CPB can be less than 1 MPa; for provision of ground support, a UCS of at least 5 MPa is needed (Li & Fall, 2018; Sheshpari, 2015b; Belem & Benzaazoua, 2004)); for roof support, CPB is required to have a UCS value that exceeds 4 MPa (Grice, 1998); and for stope stability, 0.7 to 2 MPa at 28 days (Belem & Benzaazoua, 2004, Zhao et al., 2019)

Previous studies have also shown that strength development (early and later ages) can be affected by several factors, such as tailings characteristics, chemical composition, mineralogy, fineness and density, type of tailings (Fall & Benzaazoua, 2005; Haruna & Fall, 2021; Kesimal et al., 2005), and curing temperature (Nasir & Fall, 2008). Fall and Benzaazoua (2005) observed that the cement content, tailings fineness, w/c, and tailings density significantly affect the performance of cemented tailings backfill (CTB). An increase in the fineness of the tailings material increases the water demand of the paste backfill, and a higher tailings density increases binder consumption, thus resulting in increased CPB strength (Fall et al., 2004). Haruna and Fall (2021) observed that CPB made from natural tailings (NT) that contain sulphate ions, gained less mechanical strength than CPB prepared from silica tailings (ST).

In addition, as binder plays a vital role in CPB strength development, the binder type, binder content, chemical composition of the binder, and mix proportions of the binders, chemical additives, and filler materials used can significantly affect the strength gain in CPB mixes (Sheshpari, 2015b). For example, the addition of a larger amount of binder results in higher strength (Benzaazoua et al., 2004; Li & Fall, 2016; Simms & Grabinsky, 2009). Furthermore, the binder fineness is found to affect strength development (Benzaazoua et al., 2004). Additionally, the binder type, like cement, pozzolans, and FA, is also a major factor in strength gain and affects the early and later age strength gain differently. Kesimal et al. (2005) conducted UCS tests on different CPB samples and found that the early age strength of CPB tends to increase with a decrease in the w/c. Moreover, compared to the CPB without superplasticizer, increasing the admixture content to 0.125% and 0.25% and temperature from 2°C to 35°C significantly increase the UCS of the CPB samples (Haruna & Fall, 2021).

Furthermore, mixing time significantly affects the strength gain of CPB samples. Liu and Fall (2022) observed that a longer mixing time is beneficial for the early age strength development of CPB specimens and results in the refinement of the CPB pore structure and reduction in hydraulic conductivity.

In this research program, the unconfined compression strength tests of CPB material are performed in accordance with ASTM C39/C39M-21 (2018) by using a computer-controlled mechanical press (ELE Digital Tritest) (Figure 2.8), with a loading capacity and deformation rate of 50 kN and controlled strain rate of 1 mm/min. The height-to-diameter ratio of the tested specimen is 2:1, and the ultimate failure load was recorded.

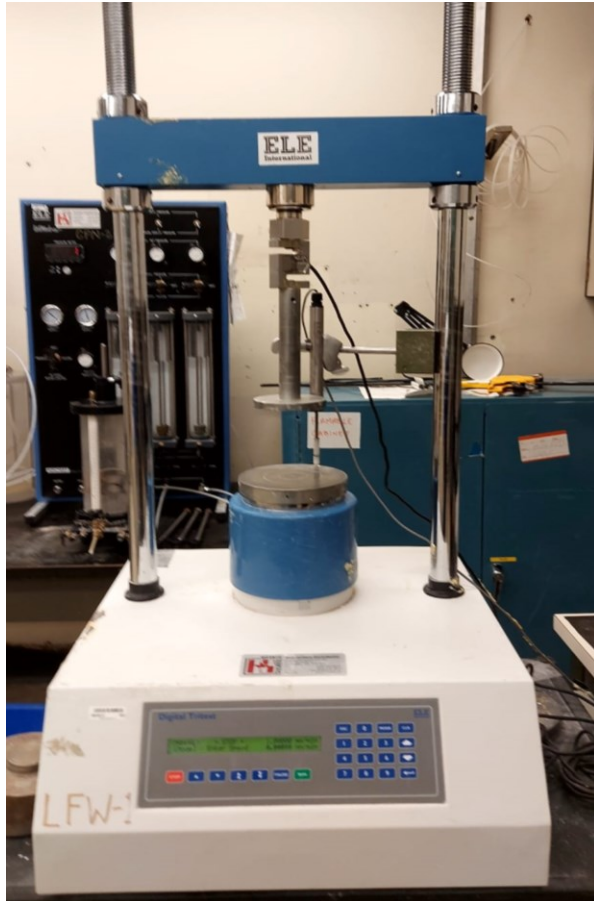


Figure 2.8 Unconfined compressive strength testing machine

2.3.3.4 Suction development in CPB

When the transformation from a hydraulic paste to a solid state begins in CPB, this is followed by the hardening process and strength gain in CPB structures. In addition to cement hydration, CPB gains strength through suction development caused by the self-desiccation of the materials (Ghirian & Fall, 2015; Helinski et al., 2006; Li & Fall, 2016; Liu & Fall, 2022a). The term "self-desiccation" of cementitious materials describes a reduction in the net volume of water and solid particles induced by cement hydration, which results in a decrease in pore water pressure or suction development, and significantly impacts the mechanical performance (Abdul-Hussain & Fall, 2012; Helinski et al., 2006).

Suction development due to self-desiccation plays an important role in the changes of the mechanical properties of CPB. It can potentially reduce the pore pressure and thus increase the effective stress within the CPB material. Furthermore, backfill strength gain is affected by the development of suction (Abdul-Hussain & Fall, 2011; Helinski et al., 2006). Nevertheless, suction development can increase the effective stress, which reduces the vertical stress in the backfill and horizontal stress on a barricade. Therefore, insufficient initial strength and suction development may affect the mechanical stability of CPB, minework safety, barricade openings, mining cycle, ore recovery, and, ultimately, cost of the mining operations (Helinski et al., 2006; Li & Fall, 2018; Liu & Fall, 2022).

Only a few researchers have examined the impact of suction development due to self-desiccation (e.g., Liu and Fall, 2022; Haruna and Fall, 2021; Helinski et al., 2006) on backfill strength. The findings show that an increase in suction could significantly increase the UCS, which results in strength gain at an early age and can lead to the earlier opening of barricades and hence enhance mining efficiency and production (Ghirian & Fall, 2015; Haruna & Fall, 2021; Liu & Fall, 2022). Ghirian and Fall (2014) investigated the changes in suction due to evaporation and self-desiccation in a column experiment by using a CPB mix prepared with 4.5 wt.% PCI and a w/c of 7.6. CPB was loaded into the column, and suction formation began after 3 hours but decreased after 27 hours. They noted that the lower suction range is the result of cement hydration-induced self-desiccation. Desaturation caused by evaporation can result in a larger suction range (300 kPa). The increase in suction values observed corresponds to an increase in strength.

Liu and Fall (2022) also investigated the effect of mixing time on the strength gain, self-desiccation, and other properties of CPB samples. A longer mixing time results in higher self-desiccation, which is beneficial for the early-age mechanical strength of CPB. Furthermore, higher self-desiccation produces higher suction and more hydration products, thus resulting in a more refined pore structure and increased CPB strength.

In this study, the suction development in CPB samples prepared with a binary binder of PCI/Slag and ternary binder of PCI/Slag/LS with different limestone dosages is studied, and its correlation with strength is validated. The monitoring of suction development in the mixes is conducted by using a dielectric water potential sensor (model MPS-6) with a six-point calibration that results in research-grade accuracy of +/- 10% for a measurement range of -9 kPa to -100 kPa.

2.4 Main Types of Backfill Binders and Limestone

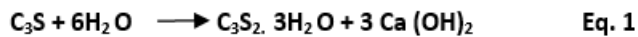
In contrast to the usually thickened tailings for surface disposal applications, the binder significantly contributes to the chemical stability and physical solidification of cemented paste tailings. Binders are also used in backfill to develop acceptable or, in some cases, additional strength to meet the specific

dynamic and static load requirements of backfilling (Tariq & Yanful, 2013) by binding the mix particles together to form a strong matrix of particles. This can be advantageous for the ecologically friendly disposal of sulphide mine waste, as the addition of binders helps to reduce the migration of potentially harmful heavy metals and arsenic (As) (Benzaazoua et al., 2004). Cement is the most common and essential component of CPB mixtures since most CPB properties rely on the binder and its properties. CPB can make up to 20% of the operational cost of mining operations (Fall et al., 2008; Hassani et al., 2007; Tariq & Yanful, 2013), while cement can make up to 75-80% of the total cost of CPB when used as the sole binder (Edraki et al., 2014; Jiang & Fall, 2017). Thus, to reduce the cost and improve the mechanical and overall performances of CPB, various SCMs and mineral fillers are generally added to OPC, which includes Slag, FA, silica fume, limestone, etc. (Li & Fall, 2018 ; Ouellet et al., 2007 ; Pokharel & Fall, 2011 ; Solismaa et al., 2021; Zheng et al., 2016). The properties and hydration process of CPB are discussed below. Subsequently, a mineral filler – limestone is discussed.

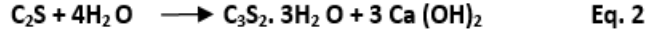
2.4.1 Ordinary Portland cement

Due to its availability and versatility, PC is the most commonly used binder in CPB (Sheshpari, 2015b; Tariq & Yanful, 2013b). When used as a binder, PC improves the shear strength through cohesion, imparts tensile strength, and increases the stiffness of CPB (Hassani et al., 2007). Moreover, it is also responsible for the hydraulic permeability, consolidation/settlement and porosity, and development of the microstructure of CPB (Ouellet et al., 2007; Yilmaz et al., 2008).

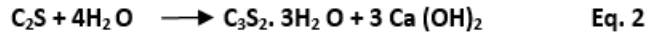
OPC is manufactured by grinding clinkers, which are usually made up of calcium carbonate (CaCO_3), by heating at a high temperature that exceeds 1450°C , some gypsum (CSH_2 , 2-8%) and calcium sulphate. OPC contains five chemical compounds: calcium oxide (CaO), silicon dioxide (SiO_2), aluminium oxide (Al_2O_3), iron oxide (Fe_2O_3), and sulfur trioxide (SO_3) (Figure 2.9). The cement hydration process consists of four phases which involves a heterogeneous mixture and explained in detail as follows (Saleh & Eskander, 2020). The primary mineral component is tricalcium silicate (C_3S or alite), which contributes significantly to the overall strength of the cement paste through the formation of hydration products - calcium silicate hydrate (C-S-H) and portlandite (CH).



The secondary mineral component is di-calcium silicate (acetylene sulfide (C_2S) or belite) which produces the same hydration products as C_3S , but with less CH. Moreover, owing to the slower rate of hydration of C_2S as opposed to C_3S and lower solubility, C_2S contributes less to early age development but more to strength development at the later stages.



Finally, tricalcium aluminate ($\text{Ca}_3\text{Al}_2\text{O}_3$) or C_3A , and calcium aluminoferrite ($\text{Ca}_4\text{Al}_2\text{Fe}_2\text{O}_{10}$) or C_4AF represent the last 2 mineral components of the heterogeneous mixture. Generally, the C_3A content in cement is comparatively lower than the silicates (C_3S). However, C_3A reacts faster and is more soluble than the silicates. Aluminates react immediately after the addition of water in cement and cause flash setting and may affect the workability of cementitious mixes (Neville, 2011). To prevent these issues, CSH_2 is added to the cement. CSH_2 dissolves immediately, releases calcium and sulphate ions into a solution in the pores, reacts with the hydration products, and produces ettringite.



Additionally, as CSH_2 is not sufficient to react with all of the C_3A present, ettringite reacts with C_3A and forms monosulphoaluminates. During hydration, C_3A initiates the chemical reactions in cement (silicate-containing), and tetra-calcium aluminoferrite (C_4AF) controls the rate of heat changes (Neville, 2011; Simon & Grabinsky, 2013).



PC is the reactive phase of CPB that undergoes a hydration reaction when exposed to water.

Furthermore, the main products formed during hydration are C-S-H ($3\text{CaO} \cdot 2\text{SiO}_2 \cdot 3\text{H}_2\text{O}$), which makes up 60 to 70% of the hydration products, (b) CH (also known as calcium hydroxide ($\text{Ca}(\text{OH})_2$), which makes up 20 to 25% of the hydration products, and (c) CSH_2 , ettringite, and calcium aluminate, which make up 15 to 20% of the hydration products (Tariq & Yanful, 2013). Tricalcium aluminate trisulphate hydrate ($\text{C}_3\text{A} \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$) or ettringite (AFt phase) and tricalcium aluminate monosulphate hydrate ($\text{C}_3\text{A} \cdot \text{CaSO}_4 \cdot 12\text{H}_2\text{O}$) or monosulphate (AFm phase) make up the remaining solid products (Saleh & Eskander, 2020, Tariq, 2012).

The cement hydration process comprises five stages: initial mixing reaction (which lasts 15–30 min), induction or dormant period (1–3 h), acceleration period (3 to 17 h after mixing cement and water), deceleration (18 to 48 h; i.e., until both unhydrated cement grains and water are present in the mixture) (Simon & Grabinsky, 2013, Neville 2011, Bullard et al., 2011, Marchon & Flatt, 2016, Soroka, 1979, Ghirian, 2016), and densification (48 h to years). The stages in more detail are as follows.

- (i) Initial mixing reaction - The early hydration reactions include the initial dissolution of PC grains, where the rapid dissolution of highly reactive aluminates and CSH_2 occurs, which results in the release of ions, Ca^{2+} , silicates, aluminate Al^{3+} , hydroxide OH^- , sulphate SO_4^{2-} and alkali ions like K^+ and Na^+ into the pore fluid (Benzazoua et al., 2010, Simon & Grabinsky, 2013);

- (ii) Induction or dormant period - a semi-permeable protective membrane (gel of Al, Si, Ca, and S) forms around the cement particles and begins to form around the hydrating cement grains, thus preventing further hydration. When the membrane that surrounds the cement grain bursts, water penetrates the membrane, following the reaction of the calcium silicates (C_3S and C_2S). Although initial crystallisation starts, the material is primarily in a colloidal form and referred to as the gel phase (Simon & Grabinsky, 2013, Saleh & Eskander, 2020).
- (iii) Acceleration period – The reaction of C_3S and C_2S starts, further resulting in the formation of C-S-H gel and a supersaturated solution of Ca and OH in the pore solution. The amount of C-S-H rapidly increases during this period which comprises up to 60% of the total volume of the cement hydration products. CH, calcium sulphoaluminate, or ettringite (AFt) begins to form at a faster rate. The PC grains (<3-5 mm) are completely dissolved, while C-S-H covers larger grains at the end of the acceleration process (Simon & Grabinsky, 2013, Saleh & Eskander, 2020);
- (iv) Deceleration period - the final stage of cement hydration, during which hydration products such as C-S-H and CH continue to form at a slower rate, followed by the formation of AFm from the reaction between AFt and C_3A . C-S-H accounts for 60% of all hydration products in the hydrated PC pastes, with CH and AFt accounting for the remaining 20%. AFt, which is less stable, is converted into the more stable monosulphate. The heat of hydration peaks and then begins to drop (Simon & Grabinsky, 2013, Saleh & Eskander, 2020); and
- (v) Densification - the belites and alites dissolve slowly, forming a solid mass of C-S-H and CH. The rate of the reaction is entirely controlled through diffusion. This stage may take several years to develop high strength, very low porosity, and low hydraulic conductivity (Simon & Grabinsky, 2013, Saleh & Eskander, 2020).

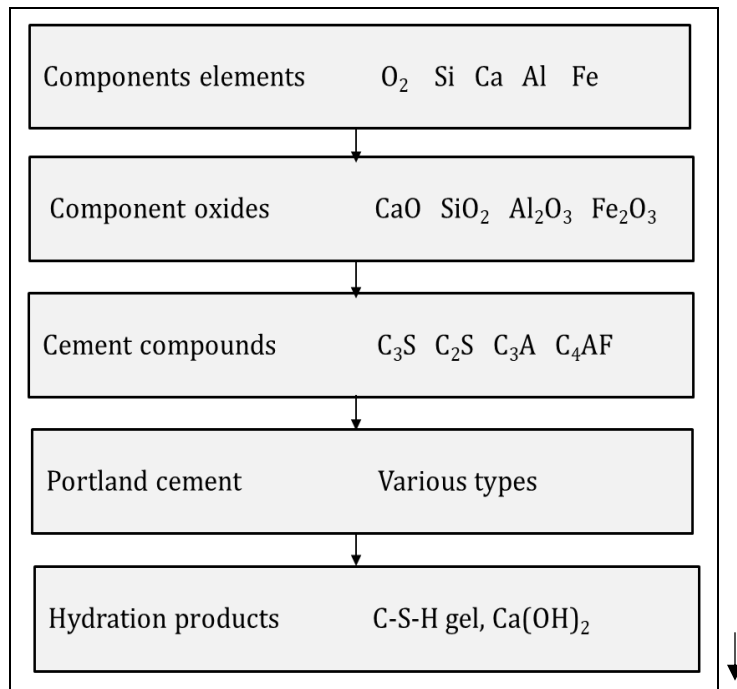


Figure 2.8 Schematic representation of the formation and cement hydration of PC (modified) (Neville & Brooks, 2010)

2.4.2 Granulated blast furnace slag- (Slag)

Slag is one the most common materials added to CPB as a partial substitute for cement, not only to reduce the cost of cement, but also the overall energy required for cement production, which ultimately reduces the carbon footprint of the industry (Solismaa et al., 2021). Using slag as a primary or secondary constituent in cement or concrete reduces the carbon footprint of total cementitious materials, as the embodied carbon content of PC of 913 kg/tonne is higher than that of slag, which is 67 kg/tonne as stated in technical datasheet 2014. Moreover, slag enhances the overall long-term performance of the CPB mix by improving its microstructure and strength at the advanced ages. Generally, slag hydrates more slowly than cement, but can react with lime in the presence of water to form insoluble cementitious products. In some situations, an alkali activator is required to speed up the hydration process (Pokharel & Fall, 2011).

Slag is a by-product of the iron industries and has pozzolanic properties. Pozzolans are materials with siliceous or aluminosilicate materials and are amorphous in nature. Slag is produced through pyrometallurgical processes in a blast furnace used to make iron. It is formed in the molten state simultaneously with iron, then rapidly cooled down with water. The rapid cooling reduces crystallisation and converts molten slag into glassy granular particles (Bouzoubaa & Foo, 2004; Duran Atiş & Bilim, 2007). This is followed by additional grinding to a specific slag fineness (cement fineness or finer), as it influences the hydraulic reactivity of slag (ACI Committee 233). Due to its glassy structure, the reaction of slag is dependent on the breakdown of its glassy structure by hydroxyl ions produced during the hydration of cement. The hydration

process of cement releases hydration products, and CH is produced, which releases alkali (Ca^{2+}), and hydroxyl (OH^-) ions. This increases the pH of the pore solutions, dissolves the glassy structure of slag, and triggers the hydration of slag in cement-slag systems (Sheshpari, 2015b).

Furthermore, activated slag reacts with alkali hydroxide $\text{Ca}(\text{OH})_2$, released by cement hydration reaction to form additional secondary C-S-H gel (Neville, 2010). These C-S-H are similar to those made with PC; however, they have a lower Ca/Si ratio (Hooton, 2000). Additional formed secondary C-S-H gel fills the capillary pore space left by primary cement hydration products (Hooton, 2000).

In cementitious systems as presented in Figure 2.10, the properties of the PC clinker, type, and dosage of the calcium sulphate set modifier, PSD of the cement constituents, and the actual concrete mix design determine the ultimate performance of the mix (Matthes et al., 2018) in addition to the inherent reactivity, fineness, and PSD of the slag,

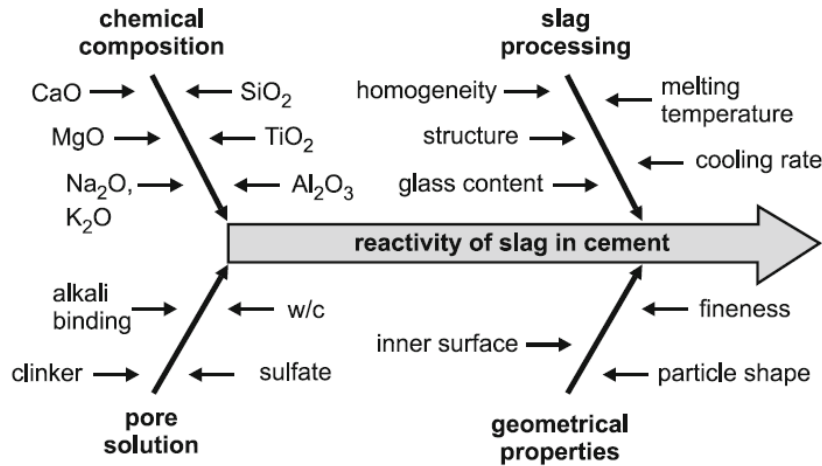


Figure 2.9 Factors that affect reactivity of ground granular blast-furnace slag (after Wolter et al., 2003; Matthes et al., 2018)

The rate of the hydration of slag is related to the glass content (Tanaka, 1983) in the slag and its physical and chemical properties (Roy and Idorn, 1982). The strength development in slag depends on its reactivity, which is related to the grade used. There are three strength grades of slag described in ASTM C989 (2014) and ACI 233R-03, which are mostly influenced by the slag quality, fineness, and potential calcium sulphate additions. Based on the activity index, slag is categorised into three primary grades of 80, 100, and 120. Furthermore, the activity index (relative strength) of slag is calculated by taking the percentile ratio of the UCS of slag- reference (SP) cement cube to the UCS of the reference cement cube (P).

Activity Index = $\frac{SP}{P} * 100$. Also, Grade 80 slag shows a lower strength than the reference cement at all ages. Grade 100 shows a lower strength gain at the early ages from 1 – 21 days, but gains equal or higher strength

at the later ages. Grade 120 slag shows a reduction in strength until the third day but increases in strength at the 7th day and onwards (ACI 233, 2003, Niroshan et al., 2017).

In CPB, the partial substitution of cement with slag is promising for CPB formulations (Benzaazoua et al., 2002). Previous studies have observed that the UCS values in hardened CPB samples are higher for slag-cement binder than PC alone (Ouellet et al., 2007). Also, a 5 wt.% addition of slag shows a reduction in the threshold diameter of the pores from 2 microns to 1 micron (Belem et al., 2001). It is evident from past research that using slag as a partial substitute for cement results in a promising binder for CPB (Benzaazoua, Fall, et al., 2004; Hassani et al., 2007; Yilmaz et al., 2008)

2.4.3 Limestone

Mineral fillers are inert or fine particle materials with low reactivity that are formed during the grinding process of rocks such as limestone, granite, sandstone, dolomite, etc. (John et al., 2018; Scrivener et al., 2015). LS fillers are commonly used in cementitious systems due to their abundance, excellent overall performance, and low cost. However, they do not have pozzolanic properties. Many engineering standards allow the addition of 5% LS with large amounts of CaCO_3 , i.e., >75% (Hooton et al., 2007; John et al., 2018). For example, the Canadian standard, CAN3-A5-M83, has permitted 5% LS in PC since 1983, followed by the Brazilian standard, NBR- 5732, which adopted the use of LS in 1988. Some researchers have argued that LS is simply an adulterant that reduces the quality of the mix. In contrast, others have reported the acceleration of C_3S and addition of calcium carbonate in C-S-H. The substitution of limestone for cement affects the hydration and composition of the cement, as well as the overall performance of the mix. Wang et al. (2018a) showed that in cementitious systems, LS has filler, nucleation, dilution, and chemical effects. Furthermore, Wang et al. (2018b) concluded that cementitious systems are influenced by physical properties like particle size, dosage, rate of dissolution, and mineralogical composition of limestone, cement, and other supplementary cementitious materials.

2.5 Binary Blended Binder in CPB System and Effect on CPB Properties

According to the Canadian Minerals Yearbook (1993), the mining industry in Ontario alone consumes 5-6% of the total PC produced at the cost of CAN\$75 million each year during backfilling in underground mining operations. The high energy costs associated with cement production, along with the remoteness of the mining site operations, result in the cost of PC typically ranging between CAN\$100-150 per tonne (De Souza et al., 1997). Hence, ordinary PC (OPC) is in general partially substituted with other additives for a cost-effective CPB, such as slag, lime, FA, etc. (Jiang et al., 2020; Schöler et al., 2015; Wu et al., 2017).

German engineer Emil Langen first discovered the potential of slag as an SCM in 1862 and substituted slag for PC in 1880. Since then, slag has been widely used worldwide. The effects of slag as a partial substitute for cement, i.e., a binary mixture of cement with slag, has been very well investigated in cementitious systems in previous studies. The substituted percentages are typically around 50%, but can be up to 95% (BS EN 197-1, 2011, Bilim et al., 2009, Courard & Michel, 2014, Deboucha et al., 2015, Mounanga et al., 2011).

Previous studies on the engineering properties (e.g., rheology, strength) of CPB with a binary binder of OPC and slag are reviewed and discussed below.

2.5.1 Effect of binary blend on rheology of CPB

The addition of slag as a partial substitute for cement in CPB has been found to influence the rheological behavior (yield stress and viscosity) of CPB. The dosage, particle size, presence of chemical additives, and temperature of slag (as the binder) influence its rheological performance in a CPB mix. Some of the findings on the effect that slag has on the rheology of CPB are discussed below.

Simon and Grabinsky (2013)- The impact of slag-containing cement at the early ages is investigated in this study. Although particle packing and electrostatic forces influence the flow behavior at the early ages, it was found that the cement hydration process has minimal effect on the apparent yield stress up to approximately 2 h after mixing the cement and water.

Haiqiang et al. (2016)- Experiments were carried out to investigate the effect of slag on the yield stress of freshly prepared CPB at sub-zero temperatures. It was found that replacing cement with 50 wt.% slag at -6°C reduces the yield stress of the mix up to 0.25 h and then shows a gradual increase in yield stress.

Jiang et al. (2020)- The authors investigated the effect of a mineral admixture on the rheological properties of fresh CPB slurry by conducting a combined assessment of thixotropy with respect to time. Cement is partially replaced by 20, 40, and 60 wt.% coarser slag. With increasing slag dosage, the yield stress and viscosity decrease in the following order: 60%Slag, 40%Slag, 20%Slag, and 100%OPC. This effect was attributed to the coarser nature of the slag particles which resulted in an increase in particle spacing and a decrease in water demand, thus reducing the ability of the suspension to resist shear.

Roshani and Fall (2020)- The effect of nano-silica on the rheological properties of CPB that contains slag or FA as a partial substitute for cement is investigated in this paper. Without the addition of nano-silica, slag blended CPB samples gain yield stress faster than the PCI-CPB samples. However, the addition of nano silica increases the yield stress and viscosity of PCI-CPB compared to Slag-CPB, but it was found to be higher than the samples without nano-silica.

Xiao et al. (2021)- An experiment was carried out to assess the effect of time, temperature, mixing water quality, and proportion of ground granular blast-furnace slag (GGBS) on the rheological properties of Slag-

CPB. Three PCI/Slag weight ratios are employed in this study, i.e., 75/25, 50/50, and 25/75 respectively. Compared to the 100%PCI sample, increasing slag content increases the yield stress and decreases the mix viscosity of the CPB mix as indicated in Figure 2.11. This phenomenon is a result of the slag-induced filler effect, where the slag particles that are finer than cement fill the spaces between the tailings and cement particles, thus resulting in denser packing and higher internal friction among the particles in the mix.

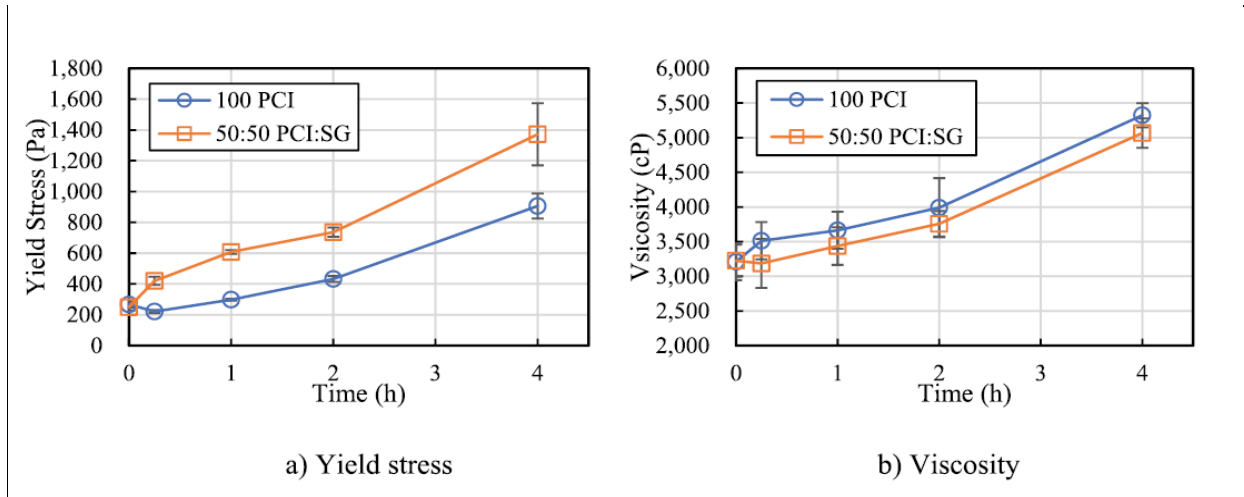


Figure 2.10 Time-dependent changes of the rheological properties of PCI-CPB vs Slag-CPB: a) Yield stress and b) viscosity (Xiao et al., 2021)

Ali et al. (2021a)- The effects of temperature (2°C, 20°C, and 35°C), different amounts of SS (0%, 0.1%, 0.3%, and 0.5%), and different binders, including slag on the rheological properties (yield stress and viscosity) of CPB samples are investigated in this study, with respect to time (0-4 hrs). CPB made with PCI/Slag and containing 0.3% SS has a lower yield stress and viscosity than CPB samples with only PCI after curing for 2 hrs, as shown in Figure 2.12. Furthermore, the yield stress and viscosity of the PCI-CPB samples increase with increases in the SS dosage and temperature.

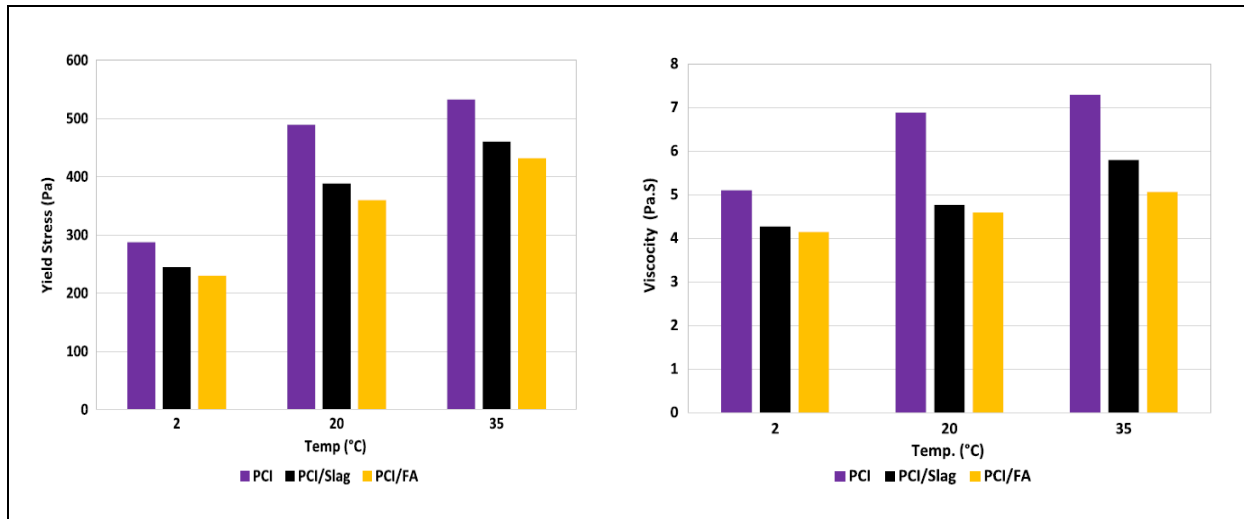


Figure 2.11 Effect of temperature and binder type on the yield stress and viscosity of CPB samples with 0.3% SS cured for 2 h. (Ali et al., 2021)

2.5.2 Effect of binary blend (PCI-Slag) on setting time of CPB

To the best of the author's knowledge, no study has been conducted to investigate the effect of using slag as a partial substitute for cement in a binary binder - PCI/Slag on the changes in setting time of CPB samples.

2.5.3 Effect of binary blend (PCI-Slag) on CPB mechanical strength and suction development

2.5.3.1 Mechanical strength development

The partial substitution of cement with slag influences the strength gain differently at the early and later ages in CPB samples prepared with PCI-Slag binders. As explained earlier, the substitution of slag with cement results in less cementing material to hydrate, and the slow reactivity of slag reduces the initial hydration reaction rate of the mix, thus resulting in low early-age strength. Within the cement matrix, the secondary reaction between slag and CH forms a finely dispersed gel that fills the large pores within the paste matrix, as shown in Figure 2.13, which results in pore refinement and ultimately higher long-term strength (Fall et al., 2010). Past research on the effect of a binary binder system on CPB strength gain (early and later ages) indicates that the strength gain with the addition of slag in CPB mixes is dependent on many factors like binder dosage, curing temperature, and chemical additives. The physical characteristics and chemical composition of slag are discussed below.

Douglas and Malhotra (1989)- The authors investigated slag as a substitute for PC and lime in cemented mine backfill. For this purpose, slags of various particle sizes, tailings binder ratios, and lime contents were

studied. It was found that the performance of the samples prepared with a slag-cement binder exceeded both that of the samples with slag-lime and PC binders.

Belem et al. (2001)- The authors used polymetallic mine tailings that contain 16% and 5% sulphur with PC, slag, and FA-based binders. In a subsequent investigation, a binder with Slag:OPC ratio of 80:20 was used to prepare the CPB samples. It was observed that the PCI/Slag changes the pore size distribution in CPB. However, the addition of only 5 wt.% of slag was observed to reduce the threshold diameter from 2 microns to 1 micron. Samples made with a slag-based binder have the highest strength compared to all of the other binders. Furthermore, pore size refinement due to slag particles reduces the hydraulic conductivity of the CPB effectively, and the suction increases gradually and peaks after 14 days.

Ouellet et al. (2007)- In this study, ground silica is used to simulate tailings in the preparation of paste backfill with cementitious binders. The results of the UCS tests were found to be correlated with those of the MIP tests in terms of the mechanical strength of the backfill. The highest proportion of fine pores was found in CPB specimens made with a mixture of PC-slag binder, thus resulting in the highest UCS values. It was also noted that slag has a more significant impact on pore refinement in PC pastes due to its finer particle size.

Ercikdi et al. (2009)- This study looks at CPB specimens made with ASTM Type I cement and sulphide rich tailings to investigate the effect of adding 20, 40 and 60% wt.% granulated blast-furnace slag on their compressive strength. At all ages, the strength of the CPB specimens decreases with increasing slag content in the binder phase. However, after 56 days, only specimens that contain 20% slag are increased in strength, with an industrially accepted compressive strength threshold of 0.7 MPa.

Fall et al. (2010)- Authors investigated the effect of curing temperatures (2°C, 20°C, 35°C, and 50°C) on the mechanical properties of CPB. Different binder types (Slag, FA), different proportions of PCI/Slag, PCI/FA (50/50), with varying cement contents (PCI-2%, 4.5%, 6%), w/c (5, 7.6, 10), and tailing types were selected and the effect of temperature was investigated over a curing time of up to 90 days. As shown in Figure 2.12. at advanced ages, the addition of slag results in better mechanical performance of the samples compared with those prepared with PCI as the sole binder or PCI blended with FA. The temperature has a significant influence on strength gain. In coupled contexts where the cement content, binder type, and grain size are the other parameters, the increase in strength is more noticeable, and the effect of temperature is more prominent than the other parameters. The addition of slag also reduces the overall porosity by reducing the pore diameter, with increased number of finer pores, as shown in Figure 2.13.

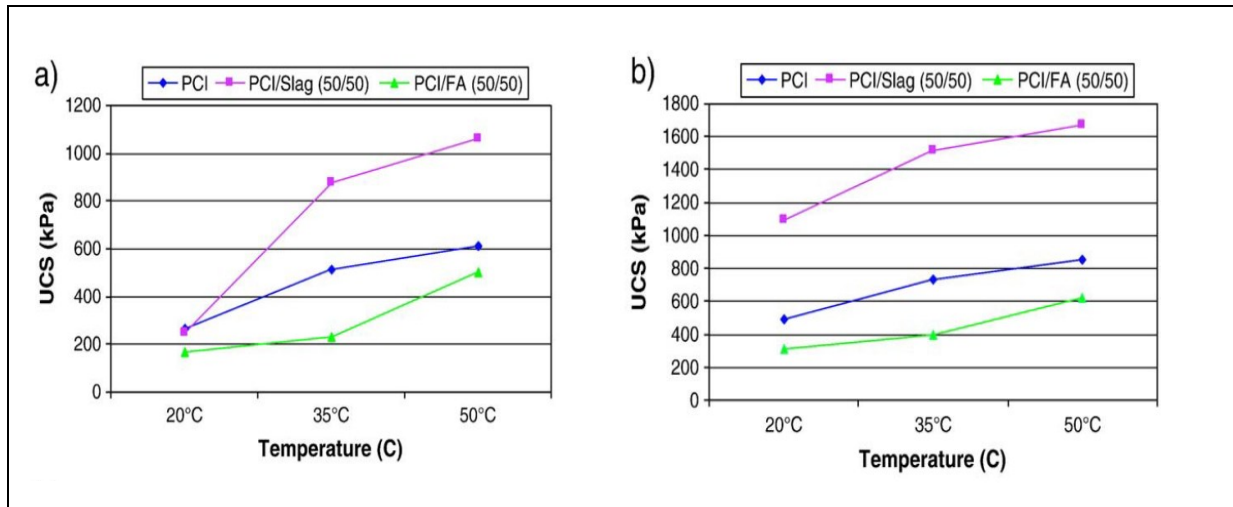


Figure 2.12 Effect of curing temperature on the strength of CPB with various types of binders: a) at 7 days, and b) at 28 days of curing (Fall et al., 2010)

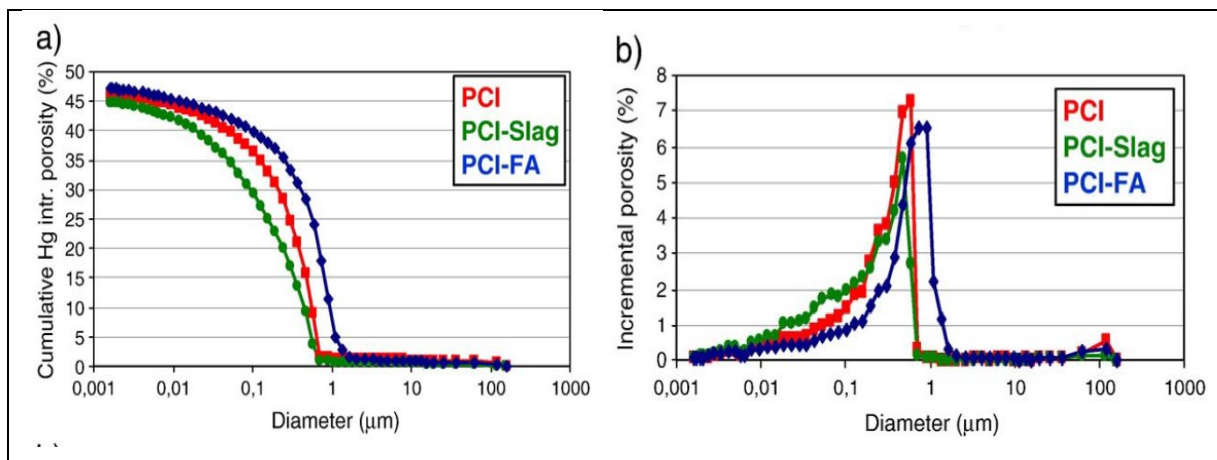


Figure 2.13 a) Pore size distribution (by MIP) and b) Cumulative porosity of CPB at 28 days (Fall et al., 2010).

Pokharel and Fall (2011)- The coupled effect of a chemical factor (sulphate) and thermal factor (curing temperature) and addition of mineral admixture - slag was investigated on the strength development of CPB (Slag-CPB). As shown in Figure 2.14, the combined effects of sulphate and temperature significantly affect the strength development of slag-CPB. Furthermore, the partial substitution of OPC with slag improves the pore refinement in the samples due to the precipitation of AFt which fills the empty pores. The UCS of the Slag-CPB samples at 150 days linearly increases with a higher sulphate dosage (5000 ppm and 15,000 ppm) and temperature (up to 35°C) compared to Slag-CPB with no sulphate. However further increases in sulphate dosage of 25,000 ppm, and temperature (>20°C) negatively affect the strength development.

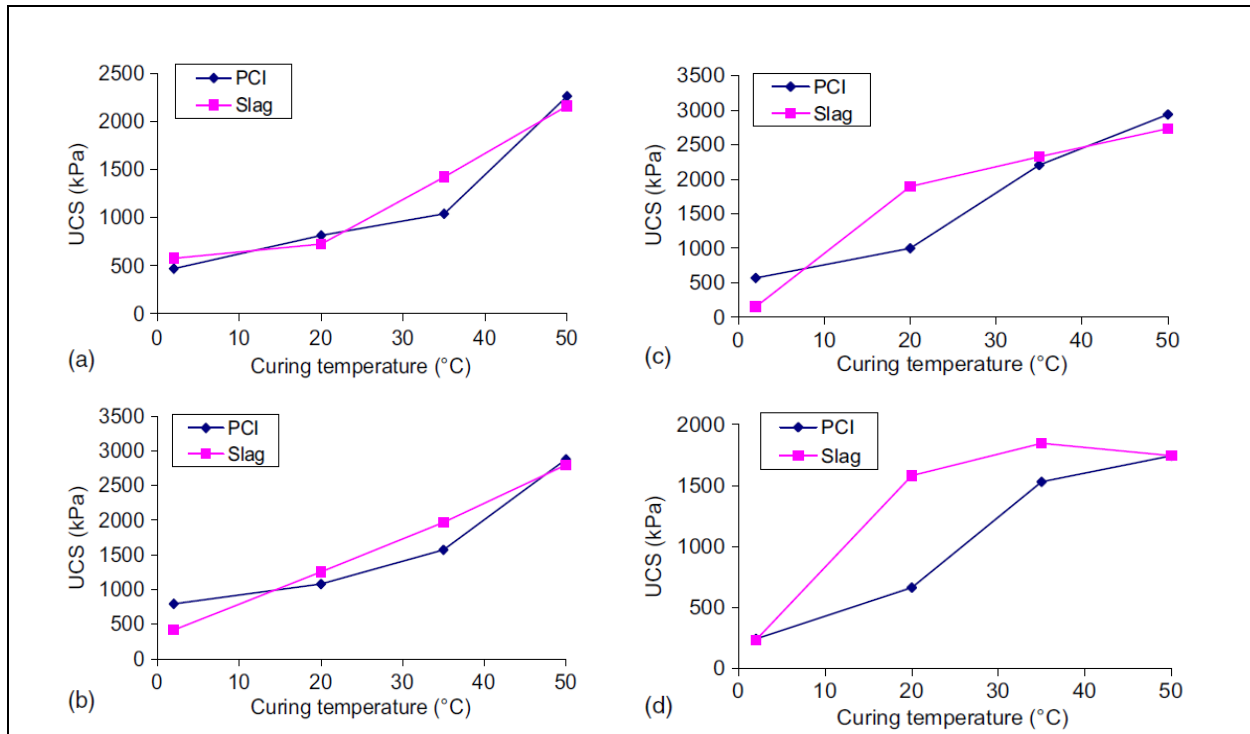


Figure 2.14 28-day UCS values of slag-CPB and PCI-CPB specimens: (a) no sulphate; and (b) 5,000 ppm; (c) 15,000 ppm; and (d) 25,000 ppm of sulphate at different temperatures (Pokharel & Fall, 2011)

Yin et al. (2012)- The effect of the solid components on the mechanical properties of CPB was studied and it was found that the magnitude of the UCS of the CPB samples without slag is almost two times larger than the samples prepared by adding 25% slag. It was also noted that ungrounded slag deteriorates the mechanical properties of CPB. At the same time, the addition of SS contributes to reaching the desired initial strength performance of CPB.

Niroshan et al. (2017)- In this study, a laboratory experimental program is carried out to understand the effect of binder (slag) dosage and solid content on the strength development of CPB. Mixes were prepared with a solid content in the 73–77% range and binder content of 3.0–4.5%. It was noted that with an increase in solid content, the UCS of the CPB samples is increased; similarly, an increase in binder dosage increases the strength of the mix at all ages. Also, the strength gain in the samples with 40 and 60% slag showed a higher strength from 7 days onwards than the pure geopolymer concrete (GPC). This was attributed to the fineness, reactivity index, and pozzolanic effect of slag. Their observations are supported by the findings in Yin et al. (2012a); i.e., an increase in the UCS of CPB samples corresponds to an increase in the solid content.

Solismaa et al. (2021)- In this study, the effect of larger amounts of slag as a substitute for cement (50, 60, and 100%) is studied over the strength gain and porosity of the CPB specimens. It was observed that the

samples with 50 and 60% slag have a higher strength than those with pure OPC at all ages (from 14 days to 91 days). However, only the samples that contain 100% slag exhibited less strength over a period of 56 days but gained higher strength in comparison to the samples with OPC alone after 56 days as presented in Figure 2.15. In addition, the pore structure of all of the slag containing CPB samples is finer than that of the samples with pure OPC.

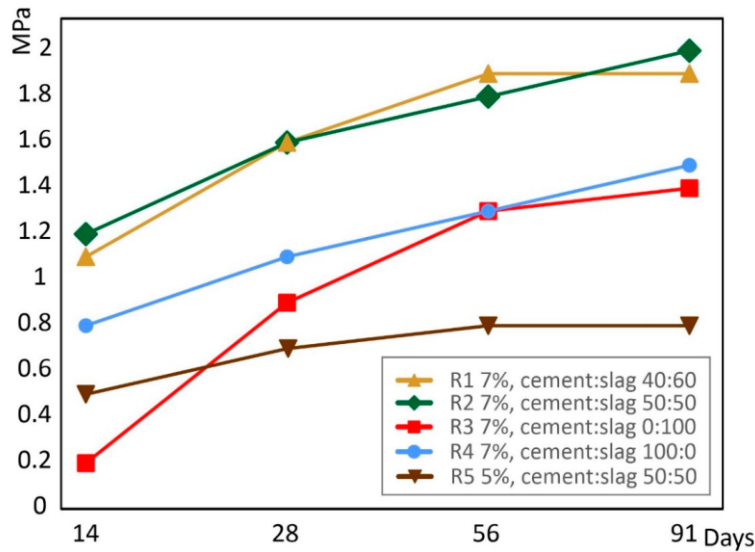


Figure 2.15 Compressive strength of the CPB samples over curing period of 90 days with different binder compositions (R1-R5) (Solismaa et al., 2021b)

2.5.3.2 Suction development

As explained in Section 2.3.3.4, the suction development (due to self-desiccation) is an important factor in the mechanical strength development and stability of CPB structures, and contributes to environmental effects (Fall et al., 2008; Helinski et al., 2006). However, there is a paucity of studies on suction development in CPB with slag except for the few following studies. For instance, Haruna and Fall (2021b) carried out an experimental program to investigate the impact of a polycarboxylate-based superplasticizer on the strength of CPB when subjected to different temperatures (2°C, 20°C, and 35°C) over different curing periods (1, 3, 7, and 28 days). Using different binders including slag, the CPB samples were prepared with a proportion of PCI/Slag 50/50 and superplasticizer percentages of 0, 0.125, and 0.25 of the total weight of the CPB. UCS tests were then performed, and the matric suction in the samples was recorded over the curing periods. It was found that the chemical reaction is elevated in the samples with temperature and the influence of temperature is higher in the presence of the superplasticizer. This increased cement hydration resulted in an increase in free water consumption in the CPB with 0.25% superplasticizer at a later age, followed by an increase in suction development and UCS in the CPB samples. It was also noted that the

strength of Slag-CPB with superplasticizer remained relatively the same in comparison with the CPB samples with PCI as the only binder.

Nevertheless, the early strength gain of the CPB is affected by the addition of slag due to its slow reactivity, so it is important to gain a better understanding of the development of suction in CPB mixtures prepared with a binary binder (PCI/Slag) in varying proportions (80/20, 50/50). This would be essential for the assessment of the mechanical stability of CPB structures at the early ages.

2.5.4 Conclusion

In summary, it is evident from past studies that the substitution of cement with slag in a binary binder may affect the rheology of the CPB samples differently depending on the physical properties of the tailings, slag, and cement used, their proportions, water binder ratio, and solid volume fraction used. Furthermore, the setting time is also influenced by the type of binder used, its proportion, cement content, solid proportion, water content, and admixtures used. Hence, investigations of the fresh properties of CPB prepared with different slag proportions are critical. Furthermore, from the literature, it is noted that even though slag produces the same or higher strength at the later ages, loss of early-age strength is still a concern. At an early age, strength gain and suction development are important factors for preventing liquefaction, and ensuring the workplace safety and productivity of underground mines. Therefore, to increase the early age strength of the PCI-Slag binder, the addition of LS, which can act as a complement to slag, and the formulation of a ternary binder are proposed and studied.

2.6 Ternary Blended Binder (PCI -Slag- LS) in CPB Systems and Review of Previous Studies on the Effect of Ternary Blended Binder on CPB Properties

According to the literature, slag may affect the flowability of CPB samples prepared with a binary binder (PC and Slag), in which cement is partially replaced by slag. Furthermore, slag hydration is slower than cement hydration, thus resulting in lower strength at the early ages but similar or higher strength at the later ages (Courard & Michel, 2014). As a result, the concept of using a ternary binder (PCI-Slag-LS), as depicted in Figure 2.16, is proposed to improve the overall performance of CPB mixes while consuming less cement. In cementitious mixes, a ternary blend of PCI-Slag-LS has numerous advantages. the most notable of which are improved flowability, strength at the longer ages, and pore structure refinement (Adu-Amankwah et al., 2019; Arora et al., 2016; Vance et al., 2013). Limestone accelerates the rate of hydration, which results in more hydration products and higher early-age compressive strength (Hooton et al., 2007). In addition, in small amounts, limestone reacts with the alumina phases of cement and SCMs which results in the formation of an AFm phase (carboaluminate hydrate) (Bonavetti et al., 2001; Voglis et al., 2005). This effect

is primarily due to the nucleation effect (increase in the rate of cement hydration), which causes hydration product growth, and the LS filler effect, which results in improved hydration product assemblage (Kadri et al., 2010; Lothenbach et al., 2008; Menéndez et al., 2003). The addition of limestone to alumina (aluminium oxide) that contains SCMs is usually beneficial given the nature of slag and LS hydration.

Hence, in CPB, a ternary binder (cement, slag, and LS) has the potential to provide both the beneficial effects of early LS hydration and slag hydration at a later age (Adu-Amankwah et al., 2019; Arora et al., 2016; Dhandapani et al., 2021; Vance et al., 2013). The aforementioned effects of limestone when used as binder (in a binary/ternary binder system) are described in more detail below.

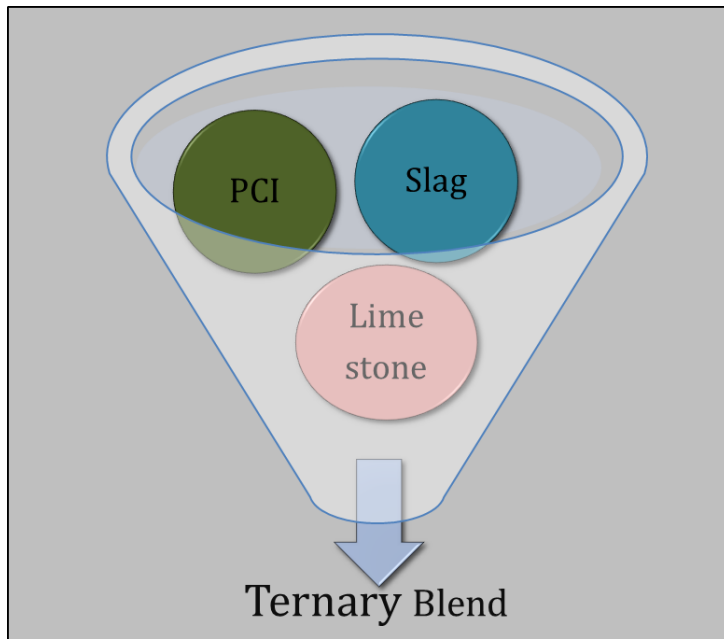


Figure 2.16 Schematic representation of formation of ternary blend (PCI- Slag-LS)

a) Filler Effect

Some researchers (Derabla & Benmalek, 2014, Liu et al., 2018, Sprung & Siebel 1991) consider that incorporating limestone into clinker is similar to adding an inert filler. Even as an inert filler, fine limestone contributes to the performance of the mix as a physical filler by occupying the small pores in the mix and enhancing particle packing. Furthermore, adding limestone also improves the PSD of the mix. The distribution of broader and smoother particles with the addition of limestone enhances particle packing, thus resulting in a reduction in the particle size spacing, subsequently increasing the hydration of cement. Finer limestone (than cement) mainly has filler and dilution effects; however, LS greater than 20 μm shows only dilution effects due to the reduction in the heat of hydration, regardless of the LS dosage (Wang, et al., 2018a, 2018b). The PSD influences both the specific surface area (SSA) and the interparticle spacing. A

larger surface area accelerates the reaction rates, which may increase water demand, thus affecting the workability of the material.

b) Nucleation effect

Limestone with more fines provides more nucleation sites and increases cement hydration by providing a larger surface area for the growth of hydrates. This catalytic effect further accelerates the rate of the hydration of cement. The particle size of the limestone (Oey et al., 2013) and number of particles, and their morphology are the main factors that influence nucleation. Very fine particles (i.e., 5 μm) develop hydration heat peaks that are more rapid and higher (i.e., filler effect for high SSA (Wang et al., 2018a)). During the hydration of cement, the C_3S particles hydrate and rapidly release the Ca^{2+} ions into the solution, which then become absorbed on the surface of the nano- CaCO_3 particles. This phenomenon increases the rate and amount of hydration by decreasing the concentration of Ca^{2+} around C_3S . These particles serve as a nucleation site for the precipitation of CH, which promotes clinker hydration at an early age. However, because CH can form on the surface of limestone particles, the agglomeration of large CH crystals can occur, which can have a negative impact on the performance of the concrete. It should also be noted that both the limestone particles and CH are susceptible to dissolution when undergoing carbonation in the presence of water. In general, the overall result of the nucleation is due to the fact that the limestone particles have increased in rate and degree of hydration and reduced the dormant period at the early ages (Bentz et al., 2017; Lothenbach et al., 2008; Vance et al., 2013).

c) Dilution effect

When cement is partially substituted with limestone in a cementitious system, a reduction in the amount of cement with non cementitious material leads to the dilution effect. This increases the amount of free water available to react with the PC; this phenomenon is called the dilution effect (John et al., 2018; Wang et al., 2018a). This further affects the material properties, which results in lower compressive strength and higher level of porosity. This dilution, or reduction of the reactive component of the binder, may then reduce hydrate production in the cementing medium and the properties of the cementitious system (Wang et al., 2017). Moreover, reducing the amount of cement reduces the w/c, which may result in an increase in the rate of cement hydration at the early ages. Furthermore, this accelerated rate of hydration may somewhat counterbalance the reduction in cementing material at the early ages. However, at the later ages, it may lead to a decrease in strength and increase porosity. When the particle size of LS is similar to or coarser than the cement particles, LS mainly has a dilution effect. Hence, the dilution effect is a function of the amount of cement replaced by limestone, and can also influence the porosity, and subsequently, the strength of the mix.

d) Chemical effect (effect on hydration/phase assemblage)

When limestone dissolves, the carbonate ions become free and available in the pore solution. Once the availability of sulphate in the mix is reduced/limited, calcite from the limestone reacts with C_3A and the additional Al available from the slag, and the resulting hydration product is called a carboaluminate (De Weerd et al., 2011). CO_3^{2-} can react with C_3A to form carboaluminates. Also, the calcium carbonate in limestone is known to interact with the alumina phases (AFm and AFt) of PC, which leads to the formation of carboaluminates instead of monosulphoaluminates (De Weerd et al., 2011; Lothenbach et al., 2008). Depending on the amount of carbonate, two types of carboaluminates can form: hemicarboaluminate (Hc) or monocarboaluminate (Mc). The addition of limestone also causes AFt stabilisation, or the preservation of AFt formed at the early ages (Bonavetti et al., 2001). The primary reaction mechanisms of cement with limestone are AFt preservation, carboaluminate formation, pozzolanic reaction, an increase in the clinker phase and degree of reaction in the hydration of SCMs. Calcite reaction would also be affected by the SO_3/Al_2O_3 binder ratio and the amount of reactive alumina (Al_2O_3) in the system.

Furthermore, the reactivity of LS depends on the fineness of the limestone and the availability of the alumina phases in the cement paste, space/pores for hydrate to precipitate; thus, larger amounts of alumina or very fine concentrations of LS particles enhance the chemical effects of LS. The effects of LS on the workability of concrete are mainly attributed to the morphological, filler, and dilution effects since its nucleation and chemical effects occur over several hours of hydration. When LS is finer than the cement particles, LS mainly shows filler and dilution effects. Limestone reacts with the C_3A component of cement to form carboaluminates (Hooton et al., 2007). Moreover, Bonavetti et al. (2001) confirmed the presence of Mc in limestone-blended cement as early as 3 days which would increase after 28 days of hydration. Voglis et al. (2005) examined 47 limestone cement paste samples and found that carbonate ions are incorporated into the calcium aluminate hydrates to form carboaluminates. After one day of hydration, they found a detectable amount of Mc, which increased over the next 28 days. The effect of the addition of limestone on the formation of CH is also debated.

The effect of ternary binders on the engineering properties of CPB has been rarely addressed. However, there are some previous studies on the engineering properties (e.g., rheology, strength) of CPB with a ternary binder composed of OPC, slag and LS, which are reviewed and discussed below.

2.6.1 Effect of ternary blend (PCI-Slag- LS) on rheology of CPB

Almost all of the previous studies that focus on the effect of adding limestone as a ternary binder on the rheological properties of fresh cementitious materials have focused on conventional cementitious materials such as concrete and mortar. However, these studies cannot be generalized for CPB, as CPB is different

from concrete and mortars. There are very few publications that focus on the use of slag and limestone as a ternary binder in CPB and the impact on its rheology. The few that are available are discussed below.

Zheng et al. (2016)- The combined effects of limestone powder (LS) with a dosage of water-reducing admixture (WRA) on the workability and influence of the microstructure on the UCS of hardened CPB mixtures prepared with 80% of granulated blast-furnace slag and OPC (20 wt.%) were investigated. They found that increasing the LS dosages up to 10 wt.% improves the workability of CPB combinations; beyond that, a decrease in the slump was observed. However, the decrease in slump is lower than that of the control mix. The coupling of LP and WR considerably improves the density and the mechanical performance of the CPB until 22.5% limestone is added to the mix.

Hu et al. (2019) - In this study, the potential of using waste limestone in solid waste cemented paste backfill (SWCPB) is investigated on two different components and grades of tailings. According to the shear test results, adding more LS prolongs the time for the slurry to reach equilibrium. In contrast, the values of the equilibrium shear stress and equilibrium viscosity are found to be lower. This suggests that the addition of LS reduces the dynamic viscosity of a slurry and improves its transportability.

2.6.2 Effect of ternary blend (PCI-Slag- LS) on setting time of CPB

To the best of the author's knowledge, there is no research literature available that has investigated the effect of the ternary binder (such as PCI-Slag-LS), on the setting time of CPB mixes. However, study of impact of using the ternary binder on setting time of CPB is important, and needs to be investigated further. As, understanding the setting time is crucial as it provides information on the development of its early age strength, or its mechanical stability at early ages and overall productivity of mine.

2.6.3 Effect of ternary blend (PCI-Slag-LS) on mechanical strength and suction development of CPB

To the best of the author's knowledge, the impact of a ternary binder (such as PCI-Slag-LS) on the strength and suction development of CPB mixes has not been well investigated with only a relevant study by Hu et al. (2008), who assessed the effectiveness of waste limestone as a cement component in SWCPB. LS was found to contribute to the hydration reaction as a crystal nucleus and incorporated into OPC to increase its strength. An increase in LS content has a negative effect on the strength of the SWCPB up to 7 days of curing; however, after 28 days, LS no longer has any effect on the strength. Furthermore, LS reduces the number of pores and average pore radius values. Porosity, macropore proportion, and average pore radius were all found to be negatively related to LS content.

2.6.4 Conclusion

Many researchers have investigated the rheological properties of CPB, including yield stress and viscosity. These studies examine the effects of initial temperature, binder content, and chemical additives on the rheological properties of slag containing CPB, but the behavior of a CPB mix depends on a number of different factors, as each CPB mix is unique. Furthermore, the effect of the substitution of slag with limestone has not been investigated in the past. The rheological behavior of CPB samples prepared with a binary binder (PCI-Slag) has not been compared with those prepared with a ternary binder with limestone as a substitute for slag (PCI-Slag-LS). The setting time of CPB with the aforementioned binary and ternary binders has not been examined. Most importantly, the mechanical strength gain and suction development in CPB samples prepared with binary slag binders and the effect of partially substituting slag with limestone in a ternary binder have not been investigated. The impact of all these variables on the fresh properties, mechanical strength gain, and suction development of CPB has been neglected. As such, this study examines the effect of a ternary binder on the rheological and mechanical stability of CPB samples, to increase current knowledge and understanding of the use of limestone so as to reduce the use of PCI and slag, thereby encouraging the development of a greener and more energy-efficient binder for CPB technology.

2.6.4 References

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3. Chapter 3. Paper I Study of Fresh Properties of Cemented Tailings Material with Ternary Cement Blends.

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3.1 Abstract

For the sustainable advancement of the mining sector, alternative binders, such as ground granulated blast furnace slag (slag) and fly ash, have been adopted to partially replace ordinary Portland cement (OPC) in the preparation of cemented paste backfill (CPB), a mixture of binder, water and solid mine waste (tailings), which is used as a construction material for underground mine support. However, the supply of these materials is limited and may not be sufficient for the industry's future needs. Limestone (LS) is an inexpensive and abundantly available material. It has the potential to complement when utilized as a binder in a ternary binder of cement slag and limestone. This study is aimed at evaluating the effects of ternary blended cement with slag and limestone powder on key engineering fresh properties of CPB: (i) rheological properties and (ii) setting time. CPB samples with PCI/Slag ratios of 50/50 (Group A) and 80/20 (Group B) were considered in this study. The effect of LS in the ternary mix was investigated by replacing the slag with increasing doses of LS (5, 10, and 20 wt%). The specimens were cast and cured over periods of 0, 0.25, 1, 2 and 4 hours and tested for rheological properties (yield stress, viscosity). In addition, tests were performed to identify the initial and final setting time of the samples. In addition, microstructural tests, zeta potential and pH measurements, and monitoring experiments (temperature, electrical conductivity) were carried out to better comprehend the underlying mechanisms behind the observed changes in rheological behavior and setting time. The findings have shown that the replacement of slag by a higher dosage of LS in the ternary binder significantly affects the rheological properties of the studied CPB. It reduces the yield stress and increases the viscosity of CPB. The effect of LS on the rheological properties of CPB is mainly attributed to the physical (e.g., filler effect, lubrication, particle size) and chemical (nucleation and changes in repulsive forces between CPB particles) effects of LS in the ternary system. The findings also revealed that increasing the proportion of limestone in the CPB ternary binder system prolonged the setting time (initial and final) of the CPB mixture due to the nucleation and dilution effects of LS. Overall, the synergy between slag and LS was observed; the optimal use of LS and slag in the ternary system can serve as a sustainable alternative to the predominantly used OPC or OPC/Slag binary binder, thereby reducing the energy consumption and carbon footprint associated with cement and CPB technology. The results of this research will be valuable for a better comprehension of the impacts of ternary binder with an increasing percentage of LS on the rheology and setting time of the CPB mix, ultimately, the efficient design of the mixing plant, especially the backfill transportation system.

Keywords: cemented paste backfill; tailings; rheology; setting time; ternary binder; sustainable mining

3.2 Introduction

The mining sector contributes considerably to the economy of numerous nations worldwide. For sustainable mining, managing huge amounts of mining waste (e.g., tailings) is one of the most critical issues and challenges facing the mining sector (Liu & Fall, 2022; Jiang et al., 2020; Yang et al., 2018). Traditional practices such as surface waste disposal often cause geotechnical and geo-environmental hazards, such as acid mine/rock drainage and tailings dam failures (Ercikdi et al., 2009; Wu et al., 2013; Yilmaz et al., 2003). Therefore, innovative mine waste management technologies, such as cemented paste backfill technology, have been developed over the recent decades to reduce and tackle the above-mentioned problems related to the surface disposal of mine waste (tailings).

Cement paste backfill (CPB) has proven to be effective for mine waste management and ground support. Furthermore, due to its economic, environmental, and social benefits, it is a common practice in modern mining industries. Thus, it is widely used in several nations, such as Australia, Canada, China, Germany, and South Africa (Cihangir et al., 2012; Fall & Benzaazoua, 2005; Haiqiang et al., 2016; Sivakugan et al., 2006; Wu et al. 2013). CPB is a composite mixture of tailings (70-85% of total solids), water (processed or fresh), and a relatively low amount of binder (typically 2-8% by weight of total solids) (Fall & Benzaazoua, 2005; Haiqiang et al., 2016; Haruna & Fall, 2020; Yilmaz et al., 2018).

Important performance and technical properties of fresh CPB include flowability and setting time. The fresh CPB mixture is typically prepared on the ground surface in a backfill plant and then transported by means of gravity and/or pumping through the pipeline to the excavated cavities underground (Liu & Fall, 2022). The successful transport of CPB, which is a function of its fluidity, is key to the successful application of CPB technology in any mine, as poor fluidity can lead to pipe clogging, delayed transport, disruption of mining operations, and ultimately financial losses (Li & Fall, 2018; Simon & Grabinsky, 2013; Wu et al., 2013). The fluidity of CPB is mainly related to its yield stress and viscosity (Ali et al., 2021; Li & Fall, 2018; Liu & Fall, 2022). Practical and efficient transportation of CPB is a challenge and an economic concern for the overall operation of the CPB plant and mining activities. This challenge is growing as many mines are located at greater and greater depths, often resulting in longer transport distances, which requires fresh CPB to be prepared with excellent flowability. Indeed, with technological advances and the progressive exhaustion of ore at shallow depths in numerous regions of the world, the mining activities are going deeper, with a maximum depth of up to 4350 m deep (Mponeng, South Africa) (Ziegler et al., 2015). The setting time of CPB provides crucial information on its early age strength development, in other words, its mechanical stability at early ages. A rapid gain in early age strength is important to reduce the risk of

liquefaction of CPB to ensure or improve the safety of working conditions and increase mine productivity (Yin et al., 2012). Therefore, a faster rate of strength gain at first age is a key objective in mine backfill and mining.

Ordinary Portland cement (OPC) is the predominant binder used in the production of CPB. But OPC is costly and its consumption accounts for 75-80% of the cost of CPB (Grice, 2001; Haiqiang et al., 2016). In addition, clinker production accounts for 5-8% of the world's man-made CO₂ emissions (Damtoft et al., 2008; Filippo et al., 2019; Gartner & Hirao, 2015). The above factors have driven mining firms to look for cement substitutes that decrease the Portland cement content (and thus the cost of CPB) and reduce the mining industry's carbon footprint, while keeping or enhancing the mechanical and rheological properties of CPB. Thus, in recent decades, the use of a binder consisting of Portland cement and supplementary cementitious material (SCM), such as blast furnace slag (slag), also known as binary cement, has been increasingly adopted in CPB technology and practice. These SCMs are often utilized in CPB to lower the cost and improve some material performance properties (Haiqiang et al., 2020; Kou et al., 2020; Niroshan et al., 2017; Schöler et al., 2015; Wu et al., 2017). For example, replacing Portland cement with slag or fly ash in the binary mix format has been shown to be an effective way to improve the long-term mechanical strength and microstructural properties of CPB (at older ages), and consequently improving the mechanical stability and environmental performance of the CPB structure at advanced ages, thereby reducing the overall cost (Fall & Pokharel, 2010; Li & Fall, 2018) and carbon footprint of CPB. However, due to the poor reaction kinetics of slag or fly ash, their use as a binary binder with cement in CPB results in low compressive strength at young ages and a low rate of strength gain of CPB in comparison to those of CPB with cement only (Snellings et al., 2022). In other words, these SCMs decrease the mechanical stability of CPB structures at early ages, which negatively affects worker safety and mine productivity. Additionally, the supply of fly ash and slag may be limited and insufficient to meet future industry needs (Bentz et al., 2017; Mehta et al., 2010; Snellings et al., 2022). Moreover, Xiao (2021) noted an increase in yield stress and viscosity with increasing slag percentage, which reduces the fluidity of CPB, ultimately negatively affecting CPB flowability and delivery to stopes (Wu et al., 2013; Xiao et al., 2019). These problems and drawbacks associated with the use of slag or fly ash have motivated mining backfill researchers and industry to search for other binder alternatives or combinations that could eliminate or reduce the aforementioned problems while maintaining or improving the positive impacts of using slag or fly ash (binary binder) on CPB properties.

Consequently, in the past few years, the use of limestone powder (LS) as a partial replacement material for cement has received increasing interest. Limestone has the benefits of being widely available, cheap, and without the environmental burdens of Portland cement (Bentz et al., 2017; Wang et al., 2018a). Limestone has been effectively used in cementitious materials, such as concrete and mortar, in recent years (Cyr et al.,

2006; Ye et al., 2007). Several previous investigations have reported that limestone has been effective in improving the rheological behavior and workability of cement paste, concrete and mortar materials (Courard and Michel, 2014; Derabla & Benmalek, 2014; Jiang et al., 2020; Sua-Iam, 2013; Vance et al., 2013a). This is mainly attributed to its filler and nucleating effect (Kumar et al., 2013; Vance et al., 2013b). Wang et al. (2018a) recommended the addition of LS aluminum rich SCMs into cementitious systems for overall performance improvement. These studies revealed that the addition of LS to Portland cement systems leads to an increase in the rate of hydration at early ages, thus causing high early strength, but it can diminish the subsequent strength because of the dilution effect when added at a higher replacement (Menéndez et al., 2003; Mounanga et al., 2011; Ramezaniapour & Hooton, 2014).

From the preceding discussion, it can be deduced that the combined use of limestone powder and slag in a ternary blended cement (LS, slag, OPC) can be helpful in formulating a CPB binder with adequate early age strength development and suitable rheological properties while decreasing the cost of CPB and the carbon footprint of the mining industry. However, the effect of ternary cement mix (Portland cement, slag, limestone) on fresh CPB properties is not well understood, and no studies on the development of rheological properties (yield stress, viscosity) and setting time (initial, final) of CPB with ternary cement mixes have been conducted. All previous studies on the impact of this ternary cement mixture on the fresh properties of the cementitious system have been conducted on different cementitious materials, such as concrete or mortar. However, concrete and mortar are different from CPB. These studies on concrete and mortar cannot be directly transferred to CPB. The aims of this research are to experimentally examine the effects of the ternary cement mixture (Portland cement, slag, limestone) on the fresh properties of CPB. These properties include rheological properties (viscosity and yield stress) and setting time.

3.3 Experimental Program

3.3.1 Materials

Tailings: Silica tailings (ST) with 99.8 wt% quartz are the main constituents of the CPB preparation for this experimental program. Their physical characteristics are presented in Table 3.1. Figure 3.1 compares the particle size distributions of the STs and the combined average particle size of the tailings from 9 mines in eastern Canada. Quartz is the most common mineral in tailings from Canadian hard rock mines; therefore, the use of ST is most appropriate to correlate the results of the experimental work with the actual Canadian mining scenario. In addition, its chemically inert nature reduces the interference from additional chemical reactions that may occur in the presence of other chemically active minerals or impurities from the natural tailings.

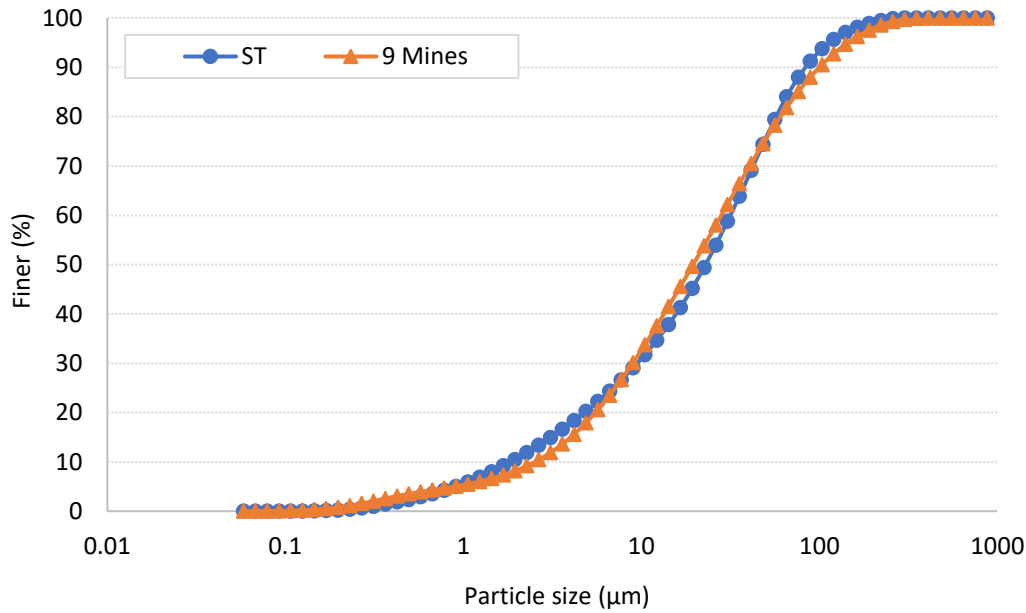


Figure 3.1 Particle size distribution of silica tailings and average particle size distribution of tailings from 9 Canadian mines

Table 3.1 Physical characteristics of tailings

Element	G_s	D_{10}	D_{30}	D_{50}	D_{60}
Unit	-	μm	μm	μm	μm
ST	2.7	1.9	9.0	22.5	31.5
Average of 9 types of tailings	-	1.8	9.1	20.0	30.8

Notes: G_s : spec. gravity; ST: silica tailings; and wt.: weight.

Binders and mixing water

In this experimental program, CPB specimens were made using PCI as the main binder with varying weight percentages of slag and limestone powder (CaCO_3) in place of cement. Figure 3.2 displays the particle size distribution of ST and all binders. Two mix proportions using 4.35 wt.% binder and 7.35 water/binder ratio were chosen in this study, with PCI/Slag proportions of 50/50, and 80/20 by weight. In addition, slag was replaced with 5, 10, and 20% limestone in the ternary binder. The chemical compositions and physical characteristics of the binders are listed in Tables 3.2 and 3.3, respectively. To study the rheological properties of the sample, distilled water is used to avoid the impact of impurities from tap water.

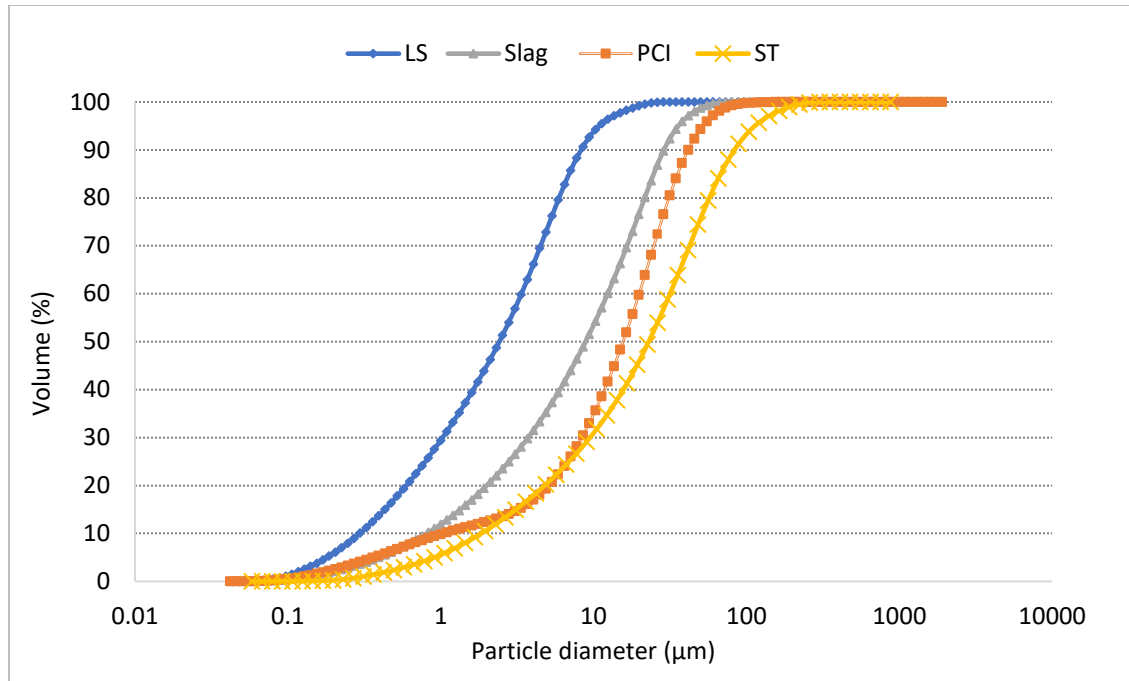


Figure 3.2 Grain size distribution of silica tailings ST, PCI, Slag, and LS

Table 3.2 Chemical composition of PCI and Slag

	S (wt.%)	Ca (wt.%)	Si (wt.%)	Al (wt.%)	Mg (wt.%)	Fe (wt.%)
PCI	1.5	44.9	8.4	2.4	1.6	1.9
Slag	12	26.6	18.9	3.9	6.9	0.5

Note- S- Sulphur, Ca- Calcium, Si- Silicon, Al- Aluminium, Mg- Magnesium, Fe- Iron

Table 3.3 Physical characteristics of the binders

Element	RD	S _s	D ₁₀	D ₅₀	D ₉₀
Unit			μm	μm	μm
Slag	2.8	2.10	0.77	8.4	27.6
LS	2.93	2.69	0.28	2.31	7.94
PCI	3.2	1.30	0.99	14.9	39.76

Note- RD- Relative density S_s: spec. surface area(m²/g)

3.3.2 Specimen preparation

Two groups of samples A and B with 50 wt.% and 80 wt.% CPB, respectively, were prepared using a binder content of 4.35 wt.% and a water-to-binder ratio of 7.35. The design of the CPB mix is presented in Table

3.4. The fresh samples were prepared by dry mixing predetermined proportions of the materials in the mixer for 1 minute, followed by wet mixing for a total of 7 minutes to ensure homogeneity of the mix.

Table 3.4 Mix composition of the CPB samples

Groups	Sample nomenclature	Type of Tailing Binder	Content (%)	%PCI in the binder	%Slag in the binder	%LP in the binder	W/B	Type of water
A	50P-50S-0L	ST	4.5	50	50	0	7.35	Distilled water
	50P-45S-5L			50	45	5		
	50P-40S-10L			50	40	10		
	50P-30S-20L			50	30	20		
B	80P-20S-0L			80	20	0		
	80P-15S-5L			80	15	5		
	80P-10S-10L			80	10	10		
	80P-0S-20L			80	0	20		

3.3.3 Test methods

3.3.3.1 Rheological tests

The prepared samples were then used to perform viscosity and yield stress tests to investigate the rheology of fresh CPB using a 10 cm x 10 cm mold for the yield stress and viscosity tests. The specimens were covered with a plastic film throughout the test. In addition, to account for the continuous movement of the CPBs during transport and to ensure homogeneity, the samples were shaken for 1 minute before performing each yield stress or viscosity test and then stabilized for 30 seconds. This process mimics the shearing of paste backfill during transportation and avoids the settlement of tailings particles (due to self-weight) during curing (Haiqiang et al., 2016). The yield stress and viscosity tests were conducted twice to ensure the accuracy of the results.

a) Yield Stress Test:

Yield stress and viscosity are the primary parameters that represent the flowability and transportability of fresh CPBs (Liu & Fall, 2022, Wang et al., 2018b). Yield stress is the lowest stress needed to initiate flow, indicating the magnitude of particle-particle forces (Simon & Grabinsky, 2013). Since CPB is a non-Newtonian fluid, the Wykeham-Farrance vaneshear apparatus suitable for soil and non-Newtonian fluids was used for this study. The apparatus consists of a vane arrangement with four 2.5cm x 2.5cm blades powered by an electric motor. Then the torque was applied and the resistance of the sample against the blades of the vane was measured by the attached deflection spring. The maximum torque reading was measured on the comparator. The test was performed on all CPB specimens with different proportions of binder mixture over a curing period of 0, 0.5, 1-, 2-, and 4-hours following ASTM D4648/D4648M-16 (ASTM 2011). The specimens were covered with a thin plastic film. The yield stress was determined based on the following equation:

$$\tau_y = \frac{2T_m}{\pi D^3 \left[\frac{1}{3} + \frac{H}{D} \right]}$$

here τ_y is the yield stress, T_m is the maximum torque, H is the length, and D is the diameter of the vane, respectively.

b) Viscosity test:

Viscosity is a crucial aspect of CPB behavior. It corresponds to the force required to maintain the flow of the mixture. The viscosities of the samples were measured using the Brookfield digital viscometer (model LVDV-E, with a base spring torque of 673.7 dynes-cm). The principle involves, measuring the viscous drag of the fluid measured against the rotating spindle through the spring deflection; finally, the viscosity was reported in cp (Brookfield, 2016). The size and shape of the spindle and the spindle speed are chosen in

rpm depending on the type of mixture. For this study, spindle number 5 and a rotational speed of 50 rpm were chosen. Fresh samples were poured into a 10cm x 10cm mold and capped with thin plastic wraps to prevent water evaporation, as when the backfill was transported in practice by pipes. The samples were tested for 0, 0.5, 1, 2, 4 hours curing time, 3 times for accuracy of results.

3.3.3.2 Setting time measurements

Knowledge of setting time is important to the overall productivity, mechanical and economic performance of the backfill operation. Initial set denotes the loss of workability and the start of paste backfill stiffening or strength gain. In addition, early strength gains are crucial for barricade opening, and extraction of neighboring stopes, to ultimately increase mining efficiency by reducing the mining cycle (Yin et al., 2012). Therefore, the impact of ternary binder (PCI-Slag-LS) on setting time was investigated using the Vicat apparatus. The test is based on the principle of mixture resistance to needle penetration and is performed according to ASTM C191-13. In this test, cylindrical molds of 100 mm diameter and 40 mm height were used as sample containers. The initial setting time was recorded when the needle reached 25 mm penetration from the bottom, and the final setting time was considered when the outer collar of the needle did not leave an impression on the specimens.

3.3.3.3 Microstructural analyses

To comprehend the evolution of the hydration process of CPB with the ternary binder at the microstructural level, by measuring the sudden mass changes under the application of heat, thermogravimetry (TG) and differential TG (DTG) were performed on cement paste specimens with considered binder proportions. The specimens were made with a binder-to-water ratio of 1:1 to accelerate the hydration process. After the target curing period, the samples were subjected to oven drying at 45°C until the mass stabilized, then powdered and sieved through a 40-micron sieve. The thermal analyzer, TGA Q5000 V20.13, was used to carry out thermogravimetric analysis on the cement paste samples up to a temperature of 1000°C. The progression of cement hydrate decomposition is represented by the TGA curve and the endothermic peak by the derivative of the TGA curve. Furthermore, the typical cement hydration curve is divided into three phases. The phase between 50°C and 200°C, 400°C and 500°C, and 600°C and 800°C indicates free water and calcium silicate hydrates, ettringite, monosulfate decomposition, Portlandite (CH) dihydroxylation, and CaCO₃ decarbonization, respectively (Deboucha et al., 2017; Ma et al., 2021).

3.3.3.4 pH and zeta potential measurements

pH – To track the hydration process through the hydration product in the interstitial solution and the alkalinity level of the mixture, a pH test was performed on freshly prepared CPB samples throughout the 0.5-,

1-, 2-, and 4-hour cure period. The samples were kept covered in plastic wrapping paper during the cure period. pH measurements were performed using Oakton pH Tester 10 (Thomas Scientific) with a range of -1.00 to 15.00 and an accuracy of ± 0.01 Ph.

Zeta potential- The potential difference at the boundary between the inner stern layer and the outer diffused region in the colloidal system is called the zeta potential (zeta potential manual). It indicates the stability of the colloidal system and the microscale interaction of the particles in terms of attractive and repulsive forces, which are closely related to the rheological (fluidity) behavior of the mixture (Xiao et al., 2021). The Zetasizer Nano series Malvern Instrument was used for this research. Considering the particle size of CPB, the specimens were prepared with solid/water ratios of 0.5 g/L, and distilled water was used as the solvent (Ali et al., 2021; Haiqiang & Fall, 2017). The test was repeated 3 times on each specimen.

3.3.3.5 Monitoring experiments

A set of monitoring experiments were performed on fresh CPBs prepared in the same way as the samples used for rheological testing. An electrical conductivity (EC) test was done on fresh CPB specimens to examine the progress of the cement hydration process by monitoring changes in EC caused by ion movement and changes in water content with time. In addition, changes in hydration rate can be reliably related to the heat released during the hydration period and can be measured by monitoring the temperature change. Changes in EC and temperature were monitored using the 5TE sensor. The sensor has an accuracy of $\pm 10\%$ for EC measurement, ranging from 0 dS/m to 23 dS/m. The freshly prepared samples were poured into a 20cm x 10cm mold, the sensors were inserted into the mold in the center and closed tightly with the cap. EC and temperature changes were tracked by an EM50 data logger.

3.4 Results and Discussion

3.4.1 Development of the rheological properties of CPB with binary cement blends (PCI/Slag)

The impact of binary binder on CPB samples, prepared with different ratios of PCI/Slag mixture, 50/50 and 80/20 wt.%, on the evolution of yield stress and viscosity is depicted in Figure 3.3 and Figure 3.4, respectively. Replacing cement with slag has a noticeable effect on the rheological behavior of CPB samples of both groups, A and B, i.e., with PCI/Slag 50/50 and 80/20, respectively. As expected, the yield stress of all samples increased during the curing period with time. From Figure 3.3, it is noted that the yield stress of sample 50P-50S-0L (50%PCI and 50% slag) was 231 Pa at 0 hour, it increased to 489 Pa at the end of 4 hours, and the yield stress of sample 80P-20S-0L (80%PCI and 20% slag) was 216 Pa at 0 hour, it increased to 461 Pa at the end of 4 hours of curing. The yield stress of 50P-50S-0L with 50% slag was observed to

be higher than that of 80P-20S-0L with 20% slag. Since the early hydration reactions of slag are slower than those of PCI, due to its glassy and pozzolanic nature (Arora et al., 2016; Deboucha et al., 2015; Haruna & Fall, 2021), in the 50P-50S-0L sample with higher slag content, less hydration products were formed. Thus, the interparticle forces present in a sample with a higher percentage of slag control the yield stress at this age (Haiqiang & Fall, 2017; Roshani & Fall, 2020; Xiao et al., 2021). In addition, the finer slag particles fill the spaces between the tailings or cement particles and refine the pore structure of the mix, leading to denser matrix packing. This causes a decrease in the distance between tailings particles, increasing the interparticle friction between particles in samples with a higher percentage of slag, resulting in higher yield stress in the sample (Haiqiang et al., 2016; Xiao et al., 2021). Furthermore, the slow hydration is validated by the results of monitoring the electrical conductivity of the specimens with 50% slag and 20% slag (Figure 3.5). From Figure 3.5, it is noticed that the electrical conductivity of sample 80P-20S-0L attains a maximum value of 4.02 mS/cm at 4 hours, while the EC of 50P-50S-0L reaches a peak at 6 hours with 3.56 mS/cm. This suggests that the hydration reaction is delayed and there is less generation of hydration products in the sample with higher percentage of slag, which is consistent with the higher yield stress in sample 50P-50S-0L with 50% slag than in sample 80P-20S-0L with only 20% slag.

Similar to the yield stress, the viscosity of all specimens was noted to increase with time during the 4-hour curing period (Figure 3.4). In addition, the viscosity of the fresh sample 50P-50S-0L was noted 3400cp, increasing to 4840cp over 4 hours of curing time, for the freshly prepared sample 80P-20S-0L, it was noted 3720cp, increasing to 5298cp at the end of 4 hours of curing. However, unlike the yield stress, as shown in Fig. 3.4, the viscosity of the Group A sample (with 50% slag), 50P-50S-0L with 50% slag, was observed to be lower than the Group B sample, 80P-20S-0L, with 20% slag. In other words, the CPB with higher slag content has lower viscosity. This behavior is ascribed to the effects of the following mechanisms: (i) The particles in the mix are in the kinetic stage; at this stage, the physical characteristics of the mix govern the flow (Rahman et al, 2014); (ii) Higher cement content in sample 80P-20S-0L (80 wt.%) gives more hydration products (e.g., CH, C-S-H, ettringite) at a faster rate, leading to C-S-H gel causing higher attractive forces between the tailings and cement particles in comparison to the Group A sample (50% cement) ; (iii) solids volume affects the viscosity of the mixture, more hydration products increase the solids volume and higher solid volume fractions and interparticle friction, resulting in higher viscosity in 80P-20S-0L samples (Ali et al. , 2021; Liu & Fall, 2022; Xiapeng et al., 2019); iv) the lower reactivity of slag results in less hydration product, thus, in the 50P-50S-0L sample with 50% slag, less hydration product was formed compared to the 80P-20S-0L sample with 20% slag. Thus, it absorbs comparatively less water for the chemical reaction, making more water available in the mix matrix, forming a water film that provides additional lubrication to the mix and reduces the viscosity (Jiang & Fall, 2017).

The aforementioned higher hydration rate in the sample with 80% PCI is confirmed by the change in the pH of the CPB over the curing time, as shown in Figure 3.6. During the chemical hydration reaction, a higher percentage of cement results in a higher hydration product and the release of alkali ions, portlandite ($\text{Ca}(\text{OH})_2$), into the pore solution. The hydration product increases the alkalinity of the solution, resulting in a higher pH. Figure 3.6 shows that the pH of 80P-20S-0L was 12.10 and 12.34 at 0 and 4 hours, respectively, higher than that of 50P-50S-0L, 12.02 and 12.24 at 0 and 4 hours, respectively. Furthermore, this higher generation of hydration products in samples with more Portland cement or less slag (80P-20S-0L) is corroborated by the results of DT/DTG analyses carried out on the cement pastes at 4h of specimens 80P-20S-0L with 20% slag and 50P-50S-0L with 50% slag, as illustrated in Figure 3.7. Sudden changes in weight loss in the form of a major peak are seen. The first peak is observed in the temperature range of 60-150°C and is related to the dehydration of the hydration products, calcium silicate hydrate (C-S-H), ettringite, gypsum and associated free water. In contrast, the second peak located between 430°C and 460°C results from the dihydroxylation of calcium hydroxide (CH). In addition, the third peak in the range of 620°C to 680°C for samples without LS is due to the decomposition of calcite in cement. The fourth peak is between 920°C and 960°C, indicating some carbonation of the calcite. Comparison of the peaks at 60-150°C and 430-450°C and corresponding weight losses indicates that the CPB with more cement or less slag (80P-20S-0L) has higher peaks and weight losses than the one with less cement 50P-50S-0L, indicating the formation of less hydration product in the sample with a higher proportion of slag (50P-50S-0L) or vice versa.

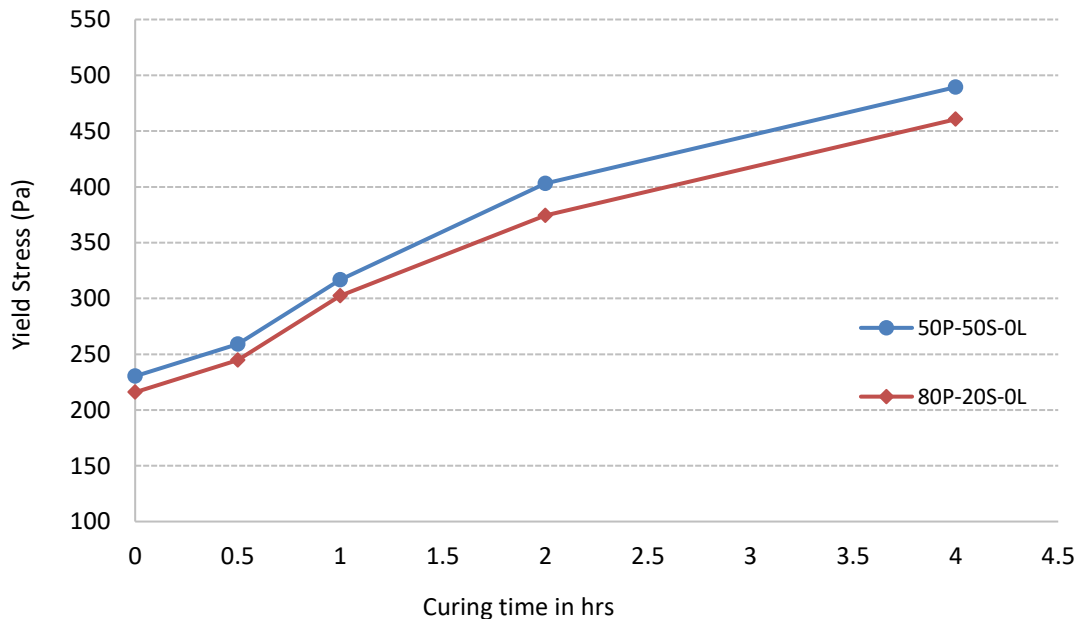


Figure 3.3 Comparison of yield stress of specimens with PCI/Slag (50/50 and 80/20 wt.%)

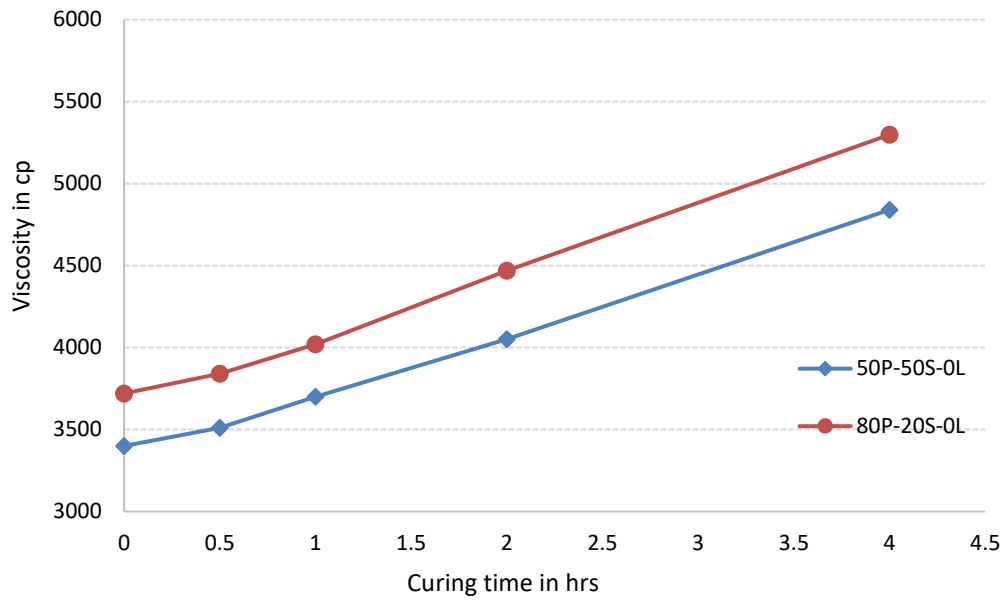


Figure 3.4 Comparison of viscosity of samples with PCI/Slag (50/50 and 80/20 wt.%)

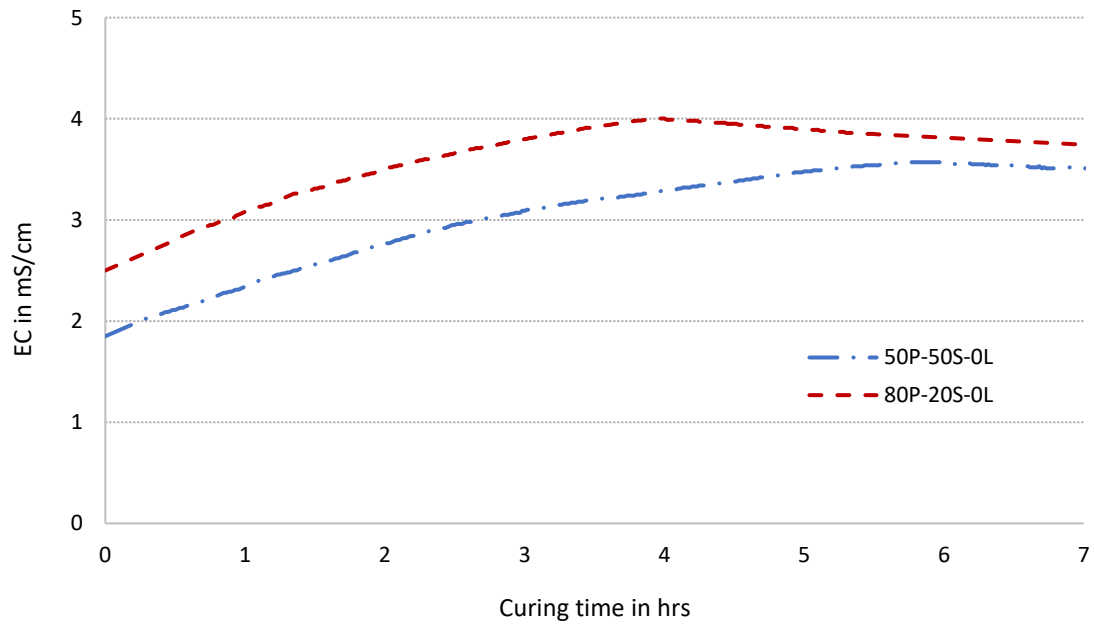


Figure 3.5 Electrical conductivity changes of samples with PCI/Slag (80/20 and 50/50 wt.%)

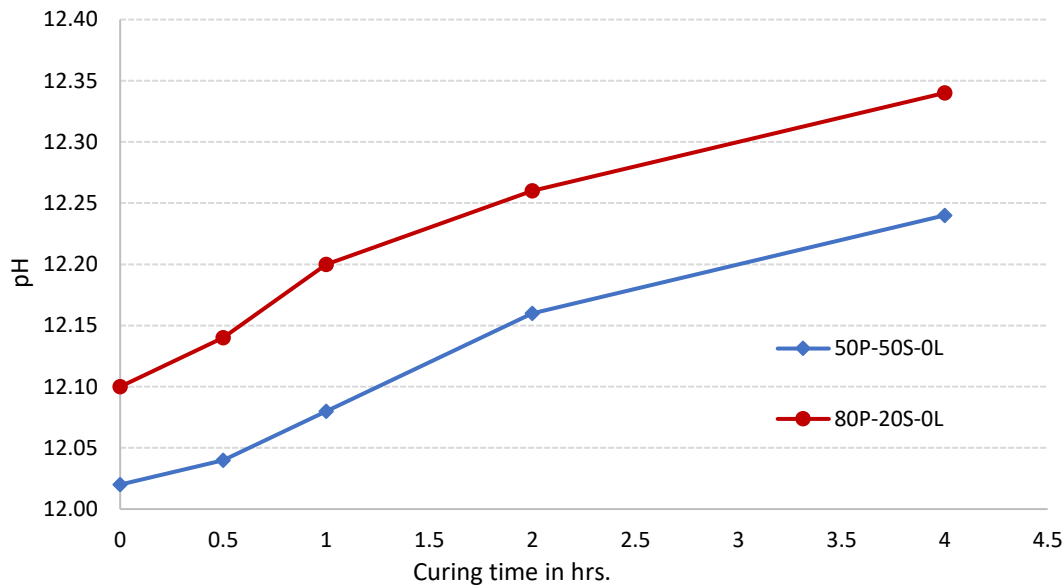


Figure 3.6 pH changes of samples with PCI-Slag (80/20 and 50/50 wt.%)

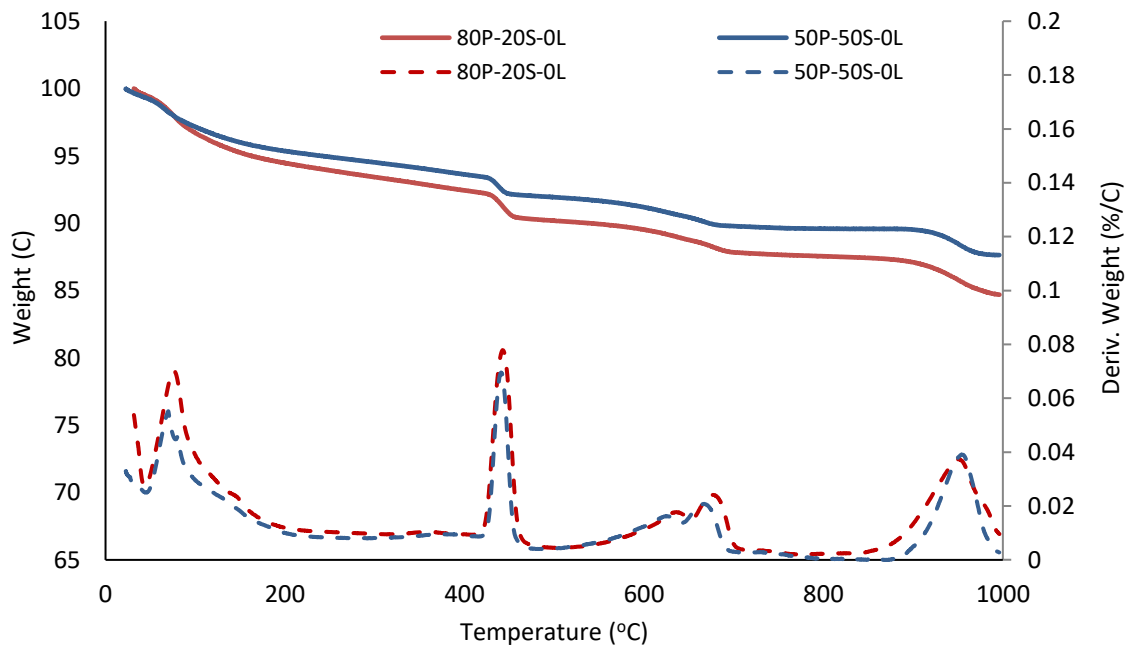


Figure 3.7 Results of TG/DTG analysis for samples with PCI-Slag (80/20 and 50/50 wt.%) cured for 4 hrs.

3.4.2 Effect of ternary binder system (PCI-Slag-LS) on rheological behavior of CPB

It was noticed that the partial substitution of slag by LS powder, with PCI as ternary binder, has a considerable impact on the overall rheology of CPB (yield stress and viscosity). Figure 3.8 illustrates the influence

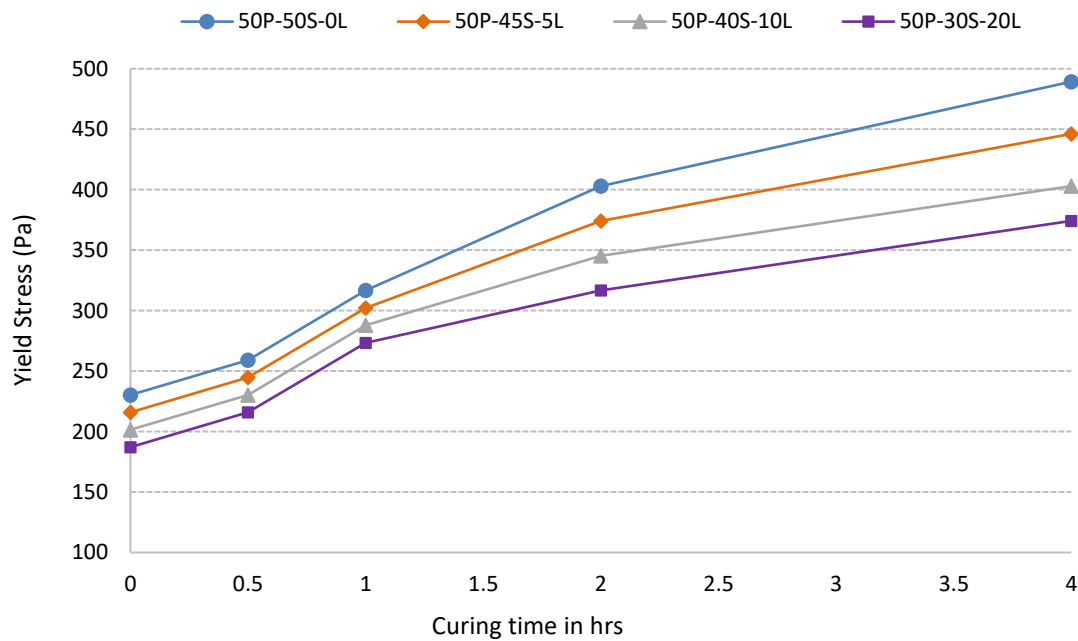
of the ternary binder with varying dosage of LS on the yield stress evolution in the CPB samples. The yield stress of the mixtures in both groups A and B, with PCI/Slag ratios of 80/20, and B 50/50, respectively, decreased with increasing dosage of limestone in place of slag. Regardless of the cement and slag content, i.e., the yield stress of the freshly prepared sample (0 hours), after the addition of 0-20 wt% LS, decreased from 230 Pa to 187 Pa, in group A and from 216 Pa to 158 Pa in group B, respectively. In addition, during 4 hours of curing, the yield stress reduced from 489 Pa to 374 Pa in group A and from 461 Pa to 360 Pa in group B, respectively. In other words, the addition of LS to CPB improves its flowability, which is beneficial in mine backfilling practice. This yield stress reduction effect due to LS addition is attributed mainly to the following physical effects or factors (morphological, filler, and dilution) of limestone as explained below:

(i) The lubrication provided by the limestone particles to the mixture (Bentz et al., 2017); in other words, during the hydration process, different hydration products are produced, and ions are released in suspension, the location of hydroxyl groups (OH^-) on the surface of calcite (Ca^{+2}), can give a means of electrostatic repulsion between the particles and thus, improve the flowability of the suspension by reducing the interaction between cement and cement particles and further flocculation (Bentz et al., 2017 ; Sekkal & Zaoui, 2013); ii) LS finer than cement acts as a filler, fills water-filled pores and expels excess water, thereby increasing the fluidity and decreasing the yield stress of the mixture (Zheng et al., 2016). However, further increasing the dosage of fine LS may increase the surface area and water demand, which could increase the yield stress. Therefore, an additional factor (chemical factor) should be considered as contributing to the decrease in the yield stress of the CPB mixture when adding LS up to 20 wt.%. This factor is the change in the repulsive forces between particles in the CPB system due to the addition of LS. This change can be assessed through the zeta potential, a good indicator of the magnitude of electrical repulsion or attraction between suspended particles (Ali et al., 2021; Jiang & Fall, 2017). Figure 3.10 shows the zeta potential of the CPB samples at the end of 4h of the curing period, where the absolute value of the zeta potential of sample 50P-50S-0L with 0% limestone was 35.5 mV; however, the absolute value of the zeta potential of sample 50P-50S-10L with 10% limestone was observed to increase to 38.1 mV. Similarly, for sample 80P-20S-0L with 0% limestone, the absolute value was 33.8 mV, which increased to 35.3 mV for sample 80P-20S-10L with 10% limestone. The absolute zeta potential of samples from both groups increased when replacing slag with incremental LS dosage. This indicates that the double-layer electrical repulsion forces increased upon LS addition, ultimately reducing the yield stress of the mixture. On the contrary, the viscosity of the samples in both groups, A with PCI/Slag 50/20, and B with 80/50, increased, with an increase in the percentage of limestone replacing slag, regardless of the PCI/Slag ratio in the ternary binder.

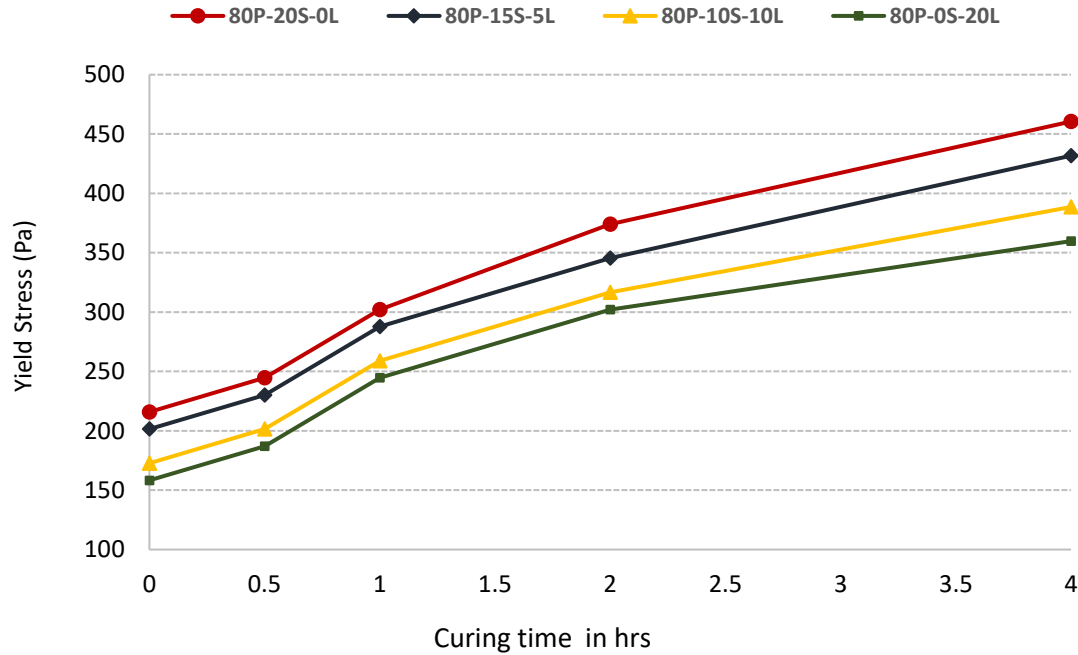
For example, from Figure 3.9, the viscosity of the freshly prepared control sample 50P-50S-0L (at 0 hours) was 3400 cp, after the addition of 5%, 10% and 20% LS dosage, it increased to 3480 cp, 3560 cp, 3648cp, respectively. At the end of the 4-hour curing period, the viscosity of the control sample 50P-50S-0L was 4830 cp, and after the addition of 5, 10 and 20% LS dosage, it increased to 5036 cp and 5260 cp, 5430 cp, respectively. This indicates that the viscosity of all samples increased over the curing time, regardless of the proportion of binder mixture. A similar trend was observed in Group B samples, i.e., with 80% Portland cement, where the viscosity of the 80P-20S-0L control samples at 4 hours was 5298cp and then increased to 5520 cp, 5732 cp, and 5882cp upon the addition of 5, 10, 20% LS, respectively.

The increase in viscosity upon addition of LS is mainly attributed to its filler and nucleating effect. The viscosity is mainly a function of solid content, particle size distribution in the mixture, and water demand (Deng et al., 2018; Vance et al., 2013; Xiao et al., 2021; Yang et al., 2020), which can be correlated with the surface area of LS. Indeed, the SSA of LS is 2.69 (m²/g) and higher than that of slag and cement, which may increase the water demand of the mixture, increasing the viscosity. The finer limestone induces the nucleation effect in the mixture (Kumar et al., 2013; Oey et al., 2013), which accelerates the degree of hydration and can produce more hydration products (C-S-H, C-H). Thus, higher hydration products increase the volume and concentration of solids in the mixture, and ultimately, the viscosity of the samples. However, this can also increase the amount of free water on the top surface, which can increase the thickness of the water film. The dominant phenomenon of these two effects decides the ultimate increase or decrease in viscosity (Hoshino et al., 2006). Indeed, the addition of LS dosage increased the viscosity of the samples in groups A and B with PCI/Slag (50/20 and 80/20). However, the influence of LS viscosity on the samples in group B with 80% PCI was noted to be higher than on the samples in group A with 50% PCI. This may be due to greater availability of cement and reactive alumina, making it available for the hydration reaction with limestone. In addition, more cement in the system utilizes all available LS as nucleation sites, enhancing the hydration reaction and potentially forming more hydration products. The acceleration of the hydration rate due to the nucleation effect of LS is also correlated with the monitoring of EC, as shown in Figure 3.11a, it is seen that the conductivity of the samples with 5 and 10 wt% LS, reached a maximum value of 3.90 mS/cm and 3.76 mS/cm at 5 hours and 5.4 hours, while the EC of 50P-50S-0L peaked at 6 hours with 3.56 mS/cm, which is lower compared to the ternary mixture samples, indicating a slow hydration rate. A similar pattern is observed in Group B samples, from Figure 3.11b, the samples with 5 and 10 wt% LS, peaked before 4 hours with peak values, 4.74 mS/cm and 4.26 mS/cm, respectively, while the EC of 80P-20S-0L peaked after 4 hours. The EC results are also validated by temperature monitoring. As presented in Figure 3.12, the temperature of the two CPB mixtures (50P-45S-5L, 80P-15S-5L) with 5 wt.% LS is higher than that of the samples without LS, indicating a greater heat release due to an accelerated hydration reaction induced by the nucleation effect of the addition of fine limestone.

This rise in hydration products due to LS addition mentioned above is also observed and confirmed by the DTG/TG results, as shown in Figure 3.13. The first and second peak, which represent the hydration products (e.g., C-S-H, ettringite, and CH) in the system, were found to be slightly higher for the samples with 20% LS 80P-0S-20L, than the control sample 80P-20S-0L, respectively, this indicates the acceleration of cement hydration in the presence of limestone. Also, as expected, an intense increase during the 3rd peak, which represents the decomposition of CaCO_3 , was noted for the samples with LS because it added CaCO_3 into it (Feng et al., 2022). However, the first and second peak for sample 50P-50S-0L were higher than sample 50P-30S-20L with 20% LS. This indicates that the physical effect (particle size, SSA, packing density, morphology) of LS dominates the viscosity behavior of the mixture.

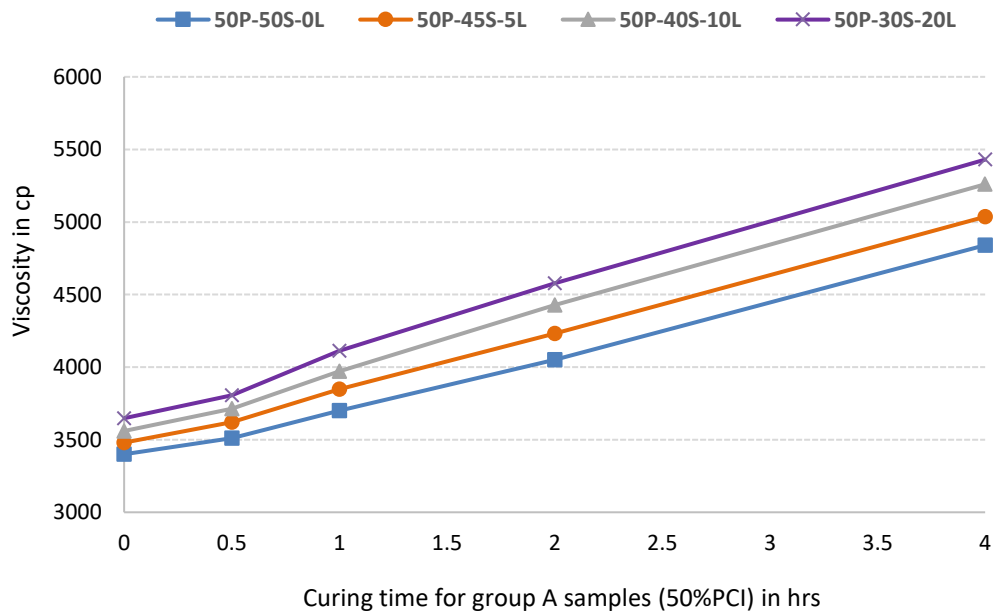


a) Group A CPB samples (binder with 50% Portland cement)

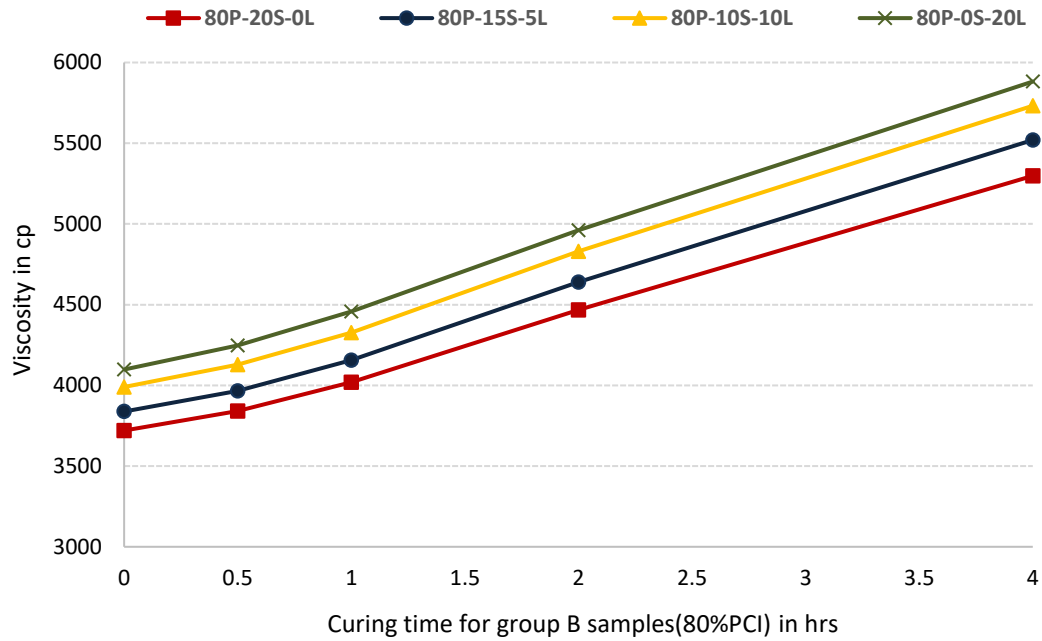


b) Group B CPB samples (binder with 80% Portland cement)

Figure 3.8 Influence of LS on yield stress of CPB samples: a) Group A (binder with 50% Portland cement), and b) Group B (binder with 80% Portland cement).



a) Group A CPB samples (binder with 50% Portland cement)



b) Group B CPB samples (binder with 80% Portland cement)

Figure 3.9 Influence of LS on viscosity of CPB specimens: a) Group A, and b) Group B.

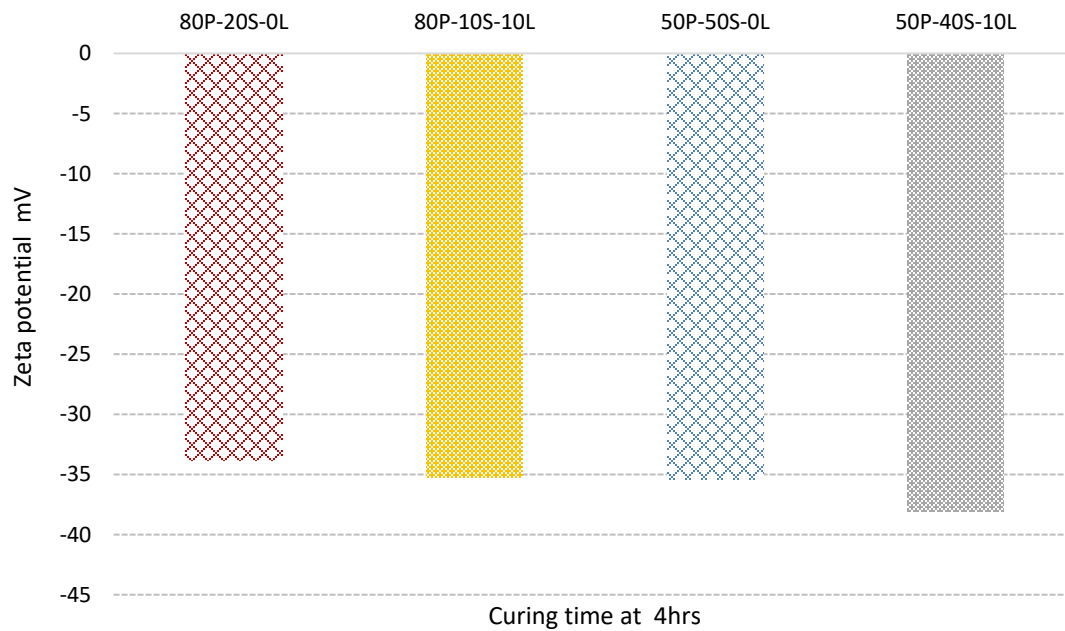
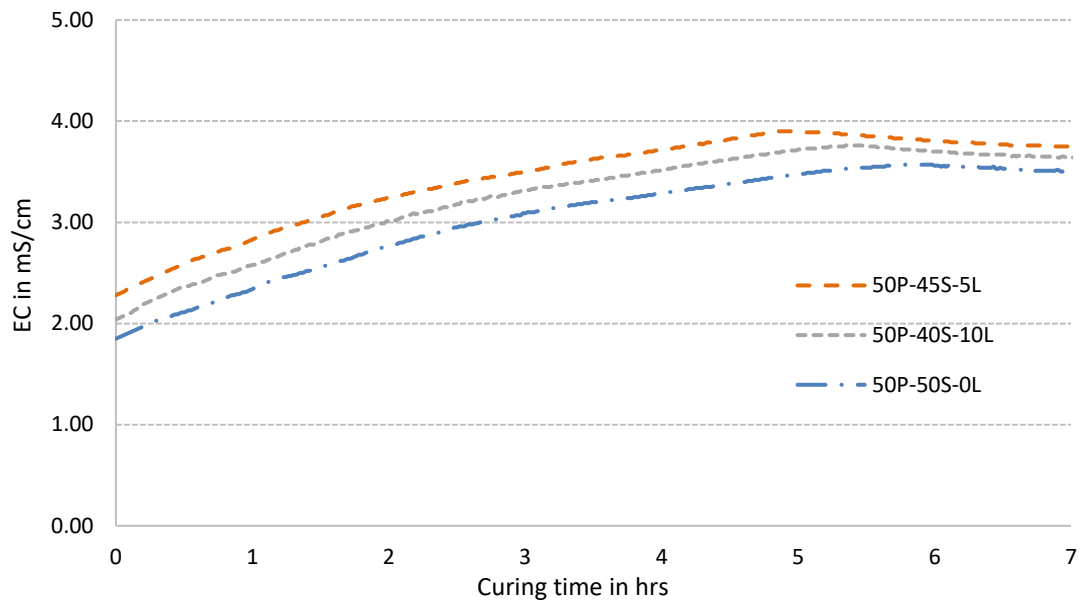
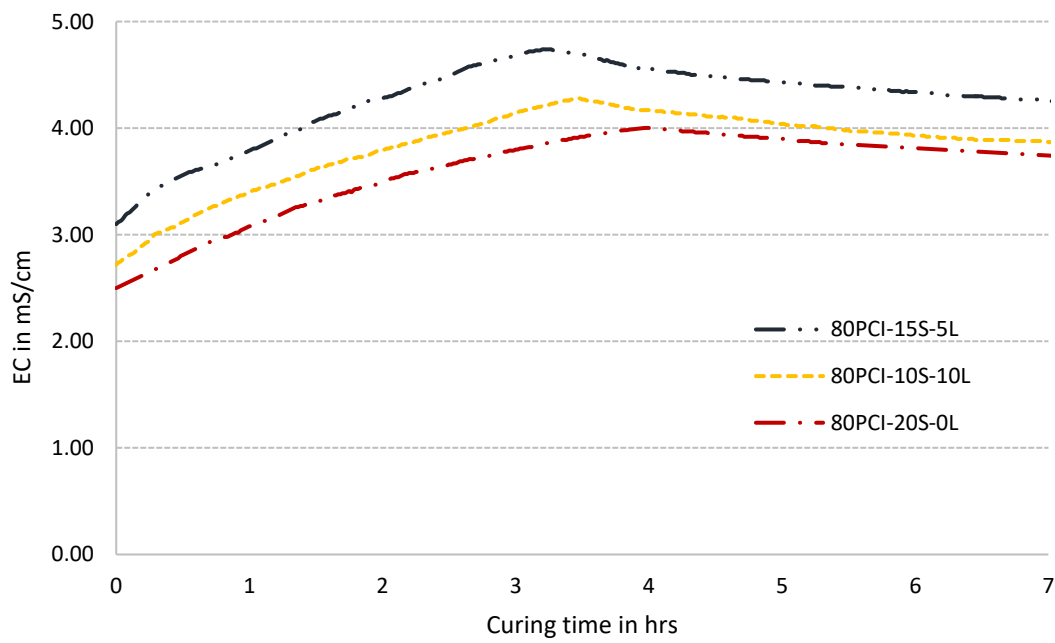


Figure 3.10 Zeta potential of Groups A and B CPB samples (binder with 50% Portland cement) cured for 4 h.



a) Group A CPB samples (binder with 50% Portland cement)



b) Group B CPB samples (binder with 80% Portland cement)

Figure 3.11 Changes in electrical conductivity of CPB samples with 0%, 5%, and 10% LS: a) Group A, and b) Group B.

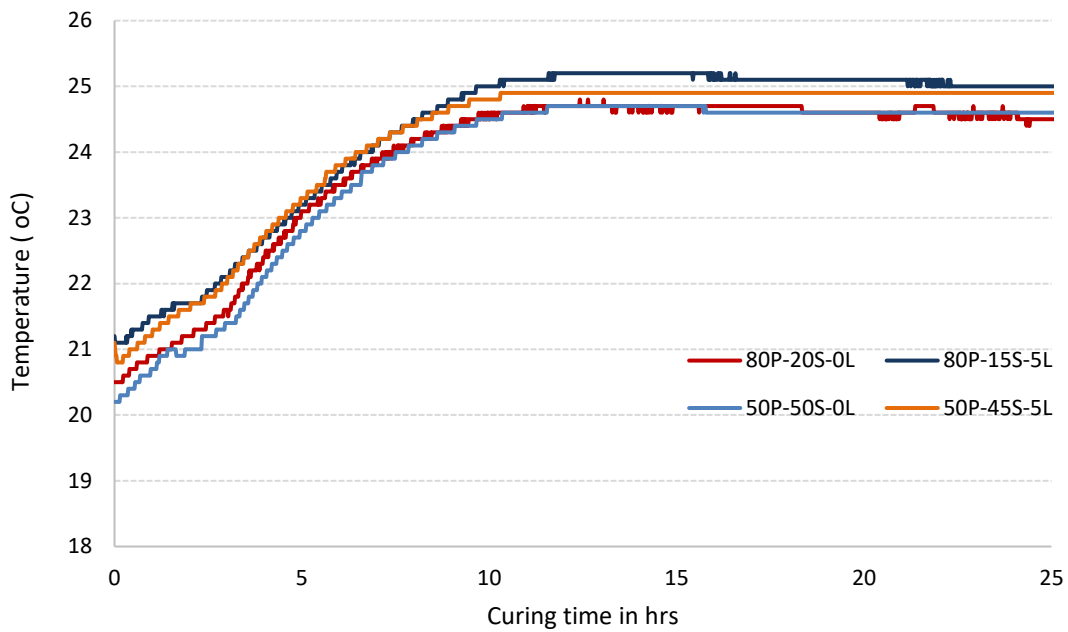


Figure 3.12 Temperature changes in CPB samples with 5 wt.% addition of LS

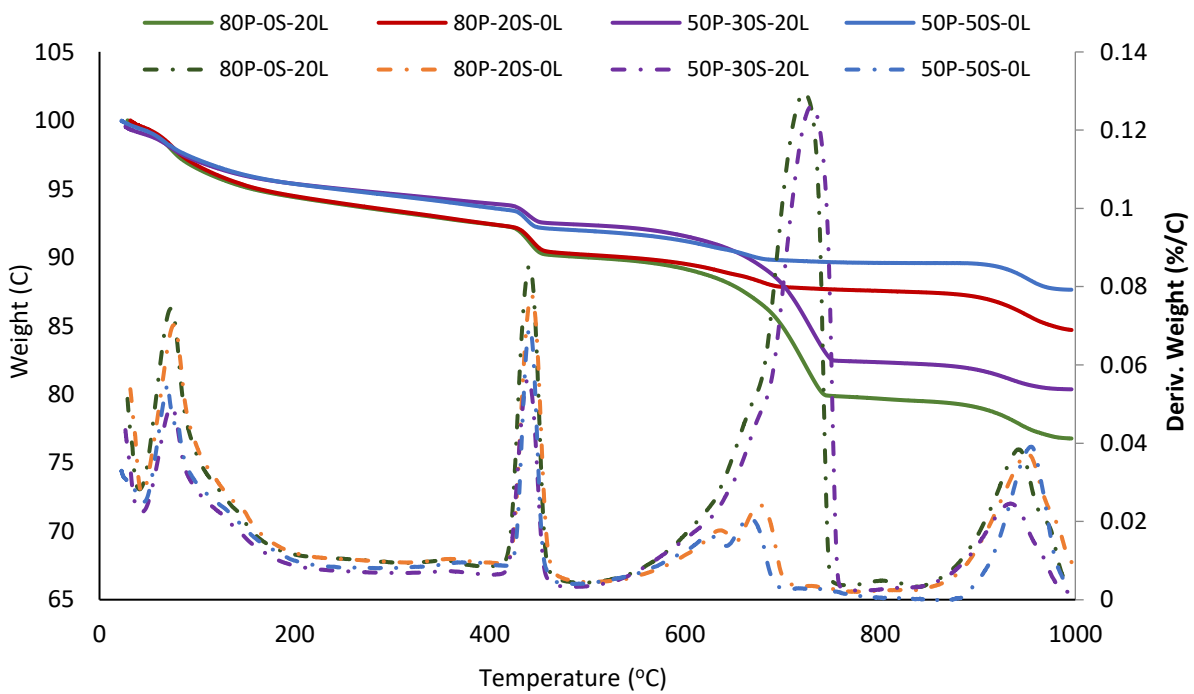


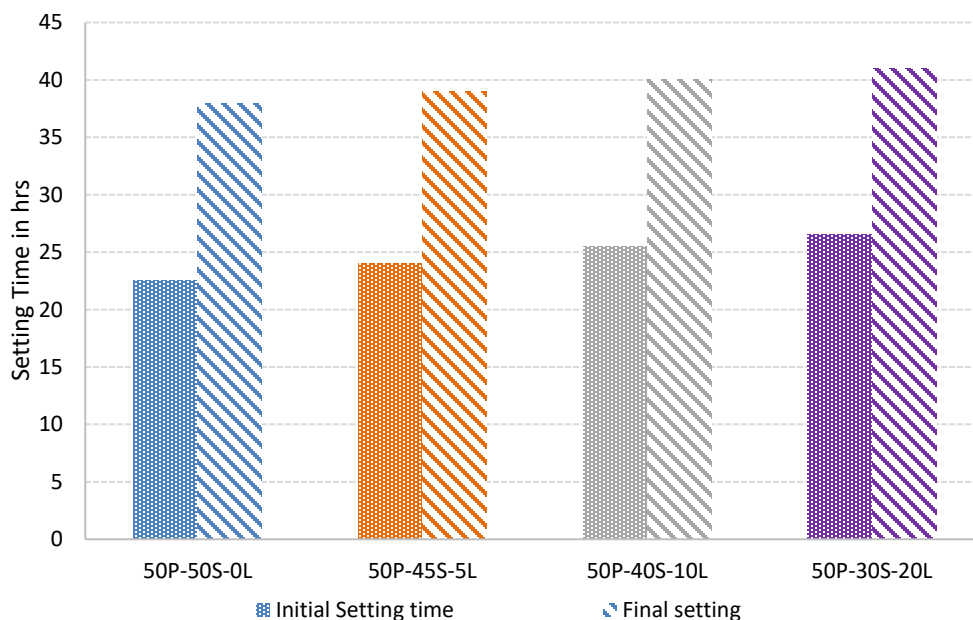
Figure 3.13 TG/DTG analysis results of specimens after 4 hrs of curing

3.4.3 Development of initial and final setting times of CPB with binary and ternary cement blends

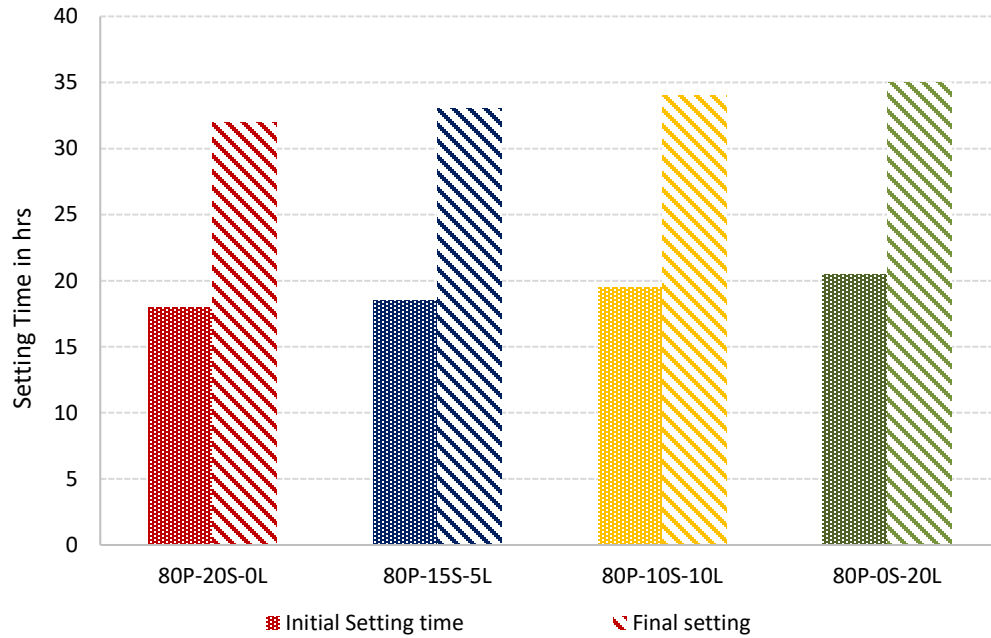
The results of the measurements of the initial and final setting time of all the CPB samples with binary and ternary cement blends are shown in Figure 3.14. It indicates the trend of increase/retardation in the setting time with a decrease in the amount of cement content, irrespective of the variation in slag and limestone content. Control sample 80P-20S-0L from group B (binder with 80% Portland cement) has a shorter initial and final setting time (at 18 hrs and 32 hrs) than that of the control sample of group A 50P-50S-0L (binder with 80% Portland cement) (at 22 hrs and 38 hrs) and the shortest of all the samples under study. This effect was clearly the result of the generation of more hydration products in the samples with higher cement content, leading to early setting in the samples with higher cement content, as already demonstrated with DT-DTG results discussed above. Further, hardening/solidification of the mix containing cement mainly occurs due to the creation of hydration products of the cement-like C-S-H, Portlandite, and ettringite (Klein & Simon, 2006). As in the hardening pastes, the setting is the process of connection of isolated or weakly bound particles which form solid paths in paste (Yin et al., 2012). Higher quantity of hydration products formation was also explained through EC results. Fig 3.5 shows higher EC for samples with PC/Slag 80/20 than 50/50. More EC indicates the generation of more hydration products, i.e., a faster hardening process and short setting time.

Figure 3.14 also shows the influence of the ternary binder, with a varying dosage of LS in replacement with slag, on the setting time of the CPB mix. It is noticed that the increase in the percentage of limestone had prolonged the setting time (initial and final) of the CPB mix. The initial and final setting time of group A control sample 50P-50S-0L was 22.5 hrs and 38hrs respectively, upon addition of 5% to 20% LS, the initial setting of samples was noted as 23.5 and 26.5 hrs resp., and final setting time at 39 hrs and 41 hrs respectively. This indicates that upon adding 5% to 20% LS, the setting time was delayed with increased dosage. The initial setting time was delayed by 4hrs and the final setting time by 3hrs, compared to the control sample with 0% LS. The same pattern was observed in the samples of group B; the initial and final setting time of control sample 80P-20S-0L was observed at 18 and 31hrs, respectively. Further, upon addition of incremental dosage of LS from 5% to 20%, initial setting time was noted at 18.5 hrs and 31.5 hrs and final setting time at 20.5 and 33 hrs. This infers the maximum delay with 20% of LS in the initial and final setting time was 2.5 and 2 hrs, respectively. It was also seen that the delay in setting time upon addition of LS dosage in group B (PCI/Slag -80/20) samples was lesser than the delay in group A(PCI/Slag-50/50) samples, which indicates the impact of nucleation effect was higher with higher cement content which balances the dilution effect at some level.

This delay in setting time upon the addition of LS is primarily due to the nucleation and dilution effects as previously observed in cement-based materials. From Figure 3.12, it was seen increase in temperature in samples with addition of 5% LS, over control sample 50P-50S-0L with 0% LS, it inferred the limestone primarily influences the cement hydration by acting as nucleation sites for cement and expected to produce more hydration products to shorten the setting time; however, from Figure 3.13, TG results of samples, it is seen that less product of hydration formed in 50P-30S-20L, with 20wt% of LS, than 50P-50S-0L, no such formation of additional hydration products are reported. Indeed, in a sample from group B, sample with 20 wt.% LS, 80P-0S-20L, somewhat higher peak was observed than 80P-20S-0L. However, the amount of the additional hydration products was not sufficient to shorten the setting time of the sample. Further, as indicated in Figure 3.11, increase in electrical conductivity was noted when LS powder was added in control samples from both groups. However, higher EC may not be directly correlated with the formation of a higher product of hydration as it may not be the result of only additional hydration product but due to finer nature of LS with higher surface area, which may influence the movement of ions in pore solution. Hence it can be inferred that mainly nucleation and dilution effects are dominating and influencing setting time, as the chemical effect of LS seems to have negligible or little impact (delay) on the setting time of CPB as the chemical reaction between cement hydrate and LS does not occurs before 24hours (Ren et al., 2020). The Delay in setting time observed upon the addition of LS is similar to elsewhere (Meddah et al., 2014).



a) Group A CPB samples (binder with 50% Portland cement)



b) Group A CPB samples (binder with 80% Portland cement)

Figure 3.14 Effect of addition of LS on initial and final setting times in CPB samples: a) Group A, and b) Group B.

3.5 Summary and Conclusions

In this manuscript, experimental work was performed on cemented paste backfill samples to study the evolution of rheological properties and setting time as a function of two parameters considered, a variable mixture of PCI/Slag and the effect of ternary binder (PCI-Slag-LS) with increasing dosage of limestone (5, 10, 20%).

Several CPB specimens with variable PCI/Slag content (80/20 and 50/50) and increasing dosage of LS in place of slag were cast and cured, and their rheological properties were evaluated for 4 hours (0, 0.25, 1, 2, 4 hours). The impact of LS on the flowability of CPB and the microstructure of CPB at different curing times is revealed by rheological tests (yield stress and viscosity) and microstructural analysis of these CPB combinations. In order to gain an understanding of the causes of the nature of the results, other tests were undertaken, including electrical conductivity monitoring, TG/DTG microstructural analysis, pH and zeta potential measurements. A summary of the major findings from the results is as follows,

- i.) Regardless of the different dosages of cement, slag and LS, an increase in yield stress and viscosity with an increase in curing time was observed. This was due to the increase in hydration products, with the progress of the cement hydration process and the self-desiccation process with time.

- ii.) In CPB specimens with binary binders containing different proportions of PCI/Slag, the samples with PCI/Slag, i.e., 50/50, gained higher yield stress with time than the samples with PCI/Slag 80/20. The mechanism responsible for this phenomenon includes interparticle friction resulting from the filler effect of the slag and a denser matrix due to its finer nature.
- iii.) The addition of LS with incremental dosage (5-20 wt%) in the form of a ternary binder impacts the rheology (flowability) by decreasing the yield stress and increasing the viscosity of fresh CPB. Interestingly, the effect of LS is complementary to the effect of slag on the rheology of the mix.
- (iv.) This effect of LS on the rheology of CPB, the decrease in yield stress was mainly attributed to the physical factors or effects of LS, i.e., the finer limestone fills the void spaces and causes water to be available in more pores, which provides additional lubrication to the mix, and additional repulsive forces. However, the viscosity increases due to the nucleation effect and the higher water demand due to the finer limestone powder with higher surface area.
- v.) The setting time (initial and final) of the samples with 80/20 PCI/Slag mix was found to be shorter than the sample with 50/50, because more hydration products (C-S-H, ettringite, AFm) were formed in the sample with higher cement content with higher rate, resulting in stronger matrix of the mix.
- vi.) The delay in setting time of CPB samples when LS was added in increasing dosage as a replacement for slag was observed and is consistent with the DTG/TG results. This indicates the influence of LS on cement hydration by nucleation effect, however more hydration product was not formed in all samples.

Despite the results obtained in this work, more detailed chemical and microstructural studies are recommended to gain a deeper insight into the impact of LS on the microstructure and chemical composition of the CPB system with a ternary binder (PC, slag, LS). In addition, it will be useful to develop a modeling tool to optimize the use of LS in CPB with a ternary binder.

3.6 References

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4. Chapter 4. Paper II Strength and Suction Development of Cemented Paste Backfill with Ternary Cement Blends.

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4.1 Abstract

To reduce the cost of backfill operations and promote sustainable mining, cheaper and more environmentally friendly binders than Portland cement are increasingly used in the preparation of cementitious paste backfill (CPB), a mixture of tailings, binder, and water, widely used as construction in underground mines. This has powered the idea of the use of ternary binders made of Portland cement, ground blast furnace slag (Slag), and fine limestone (LS) powder to prepare more sustainable CPB. However, the effect of this ternary cement blend on key engineering properties and design parameters (mechanical strength, suction) is not well known. In this paper, the effect of ternary binder containing Portland cement (PCI), Slag, and LS, with varying proportions, on the suction development and the mechanical strength of CPB was investigated over a curing period of up to 90 days. Mechanical (uniaxial compressive strength) tests, microstructural analyses (Thermo-gravimetry analysis, TG/DTG, mercury intrusion porosimetry, MIP), and monitoring (suction, electrical conductivity, temperature) experiments were conducted on CPB. In the first part of this study, binary binders with two different PCI: Slag proportions, 50:50 and 80:20, were examined with no limestone, followed by the slag replacement with increasing weight percentages of LS from 0 to 20 % by wt. of the total binder, with constant cement content. Results indicate that slow slag hydration kinetics affected the early age suction and strength evolution in the binary sample with high slag content (50 wt.%); however, its latent hydraulic and pozzolanic properties enhanced the strength gain at a later age from 28 days and onwards. The addition of 5 wt.% of limestone in the ternary blend improved the strength up to 7 days compared to binary control samples. Overall, 10% of binder replacement levels by adding LS does not affect the mechanical strength of CPB at an early age or later age. Indeed, the presence of limestone influences the rate of hydration of cement and slag both by physical effect (filler, nucleation, dilution) and by the chemical by hydrate assemblage; however, beyond 10% addition of LS affected the mechanical performance at all ages. Overall, the strength development reveals that up to 50 wt. % of slag can be used, with up to 10 wt.% of limestone with cement as a ternary binder without significant loss in compressive strength. The results of this research could significantly benefit the mining industry with a ternary binder for cement replacement, which may enhance the overall performance of the CPB system, facilitate the environmental-friendly design of CPB, and improve tailings management practice and the overall economy.

4.2 Introduction

The mining industry is the source of minerals and metals, which are critical for the economic development of nations. Nevertheless, the extraction process of minerals produced over 14 billion tons of tailings globally in 2010, resulting in huge mine waste and underground voids (Jones & Boger, 2012; Zhao et al., 2018). With increased environmental awareness, stringent laws, regulations, and policies mining industry is adopting advanced waste management technology like underground backfilling. Cemented Paste Backfill (CPB), the novel technology of mine waste backfilling, facilitates the safe disposal of mine waste and underground support with environmental, economic, and technical benefits (M. Fall & Pokharel, 2010; Kesimal et al., 2005). CPB is a combination of tailings (70% - 85% by weight), binders (3% - 7% by weight), and water used to backfill mine waste into underground mining stopes (Benzazoua, Fall, et al., 2004; Edraki et al., 2014; Haruna & Fall, 2020a; H. Jiang & Fall, 2017a). Portland cement (PC), the most commonly used binder in CPB preparation PC, is expensive. Clinker production accounts for 5-8% of global anthropogenic CO₂ emissions (Vance, Aguayo, et al., 2013b; Vance, Kumar, et al., 2013a). Moreover, cement consumption accounts for 75% to 80% of CPB's production cost (H. Jiang & Fall, 2017a).

In recent decades, for the partial replacement of cement, the use of secondary cementitious materials (SCM) like blast furnace slag has been increasingly adopted in CPB technology, being cost-effective and its ability to improve microstructure and strength (later age) (Li & Fall, 2018; Pokharel & Fall, 2011; Solismaa et al., 2021). When cement is partially replaced by slag, it reacts with the (Ca (OH₂)) released in cement hydration process and produces additional C-S-H, and induces increase in compressive strength at advanced ages (Solismaa et al., 2021a). Further, due to its latent hydraulic property, slag hydrates slowly at an early age, reducing the strength of CPB at an early age (Arora et al., 2016c; Castellano et al., 2016). However, early age strength is important for mechanical stability and to eliminate the risk of liquefaction (Pokharel & Fall, 2011). In addition to cement hydration, CPB gains strength through suction development caused by self-desiccation of the materials (Ghirian & Fall, 2015; Helinski et al., 2006; W. Li & Fall, 2016; Liu & Fall, 2022). Insufficient initial strength and suction development may affect the CPB's mechanical stability, minework safety, barricade openings, the mining cycle, ore recovery, and, ultimately, the economy of the mining operation (M. Fall & Pokharel, 2010; Helinski et al., 2006; W. Li & Fall, 2018; Liu & Fall, 2022a).

Recently ternary binders been used in cementitious systems to overcome the limitations of binary binders' and maximize benefits by complementing one another. This has increased interest in using limestone powder (LP) as a partial cement replacement material, owing to its potential to improve the performance of the cementitious system in the presence of slag at an early age (Adu-Amankwah et al., 2017; Arora et al., 2016a; Deboucha et al., 2022; Li & Jiang, 2020). Moreover, LS and slag together in blended cement binder were observed to accelerate the early hydration (Mounanga et al., 2011b), thus, the strength of the cementitious system. This phenomenon is attributed to the physical and chemical effects of LS. Finer limestone

particles act as a nucleation site for hydrates, accelerating the rate of hydration, and fills the voids between coarser particles, resulting in better particle packing, which may increase the compressive strength at early ages (Deboucha et al., 2022; Li & Jiang, 2020; Carrasco et al., 2005a; Vance et al., 2015). Furthermore, it was also observed that LS chemically reacts with the aluminate phase of slag to produce mono and hemi-carboaluminate (Adu-Amankwah et al., 2017; Arora et al., 2016). Carboaluminates, being more stable than monosulphoaluminate, increase the total volume of hydrates and thus may induce pore refinement and increase compressive strength (Adu-Amankwah et al., 2017).

From the discussion above, it can be inferred that incorporating limestone powder and blast-furnace slag together could complement each other and bring out the synergy. This ternary blend (PCI-Slag-LS) has the potential to develop adequate CPB strength at an early age, without affecting strength at advanced ages, while decreasing the cost of CPB and the carbon footprint of the mining industry. The use of supplementary cementitious materials (SCM) like blast-furnace slag (Slag), fly ash (FA), and filler materials limestone (LS) is very well investigated in cement-based systems, such as concretes and mortars (Carrasco et al., 2005a; Courard & Michel, 2014; Vance, Aguayo, et al., 2013b; Zheng et al., 2016a), but rarely addressed in cemented tailings backfill systems. CPB is different from concrete and other cement-based mixes; the effect of ternary cement blends (Portland cement, limestone powder, slag) on the critical CPB performance properties, mechanical strength, and suction development is not well-known and needs to be investigated further. The objective of this research is to investigate the effect of the ternary cement blends on mechanical stability of CPB, by understanding the strength evolution and suction development over curing time, through laboratory experimental program.

4.3 Materials and Methods

4.3.1 Materials

4.3.1.1 Tailings materials

Silica Tailings (ST) is a synthetic tailing, a product of the US Silica Co. is used for specimen preparation. Note that the particle size distribution of ST is comparable to the average of nine types of tailings from mines in eastern Canada, as shown in Figure 4.2. The physical characteristics of the tailings are presented in Table 4.1. The STs contain 99.8% quartz (SiO_2), the most common mineral in hard rock mine tailings in Canada. In addition, quartz is chemically inert, which reduces the amount of uncertainty in the results that can be created by the chemical reaction; therefore, it is a ST-controlled medium and is best suited for this experimental work.

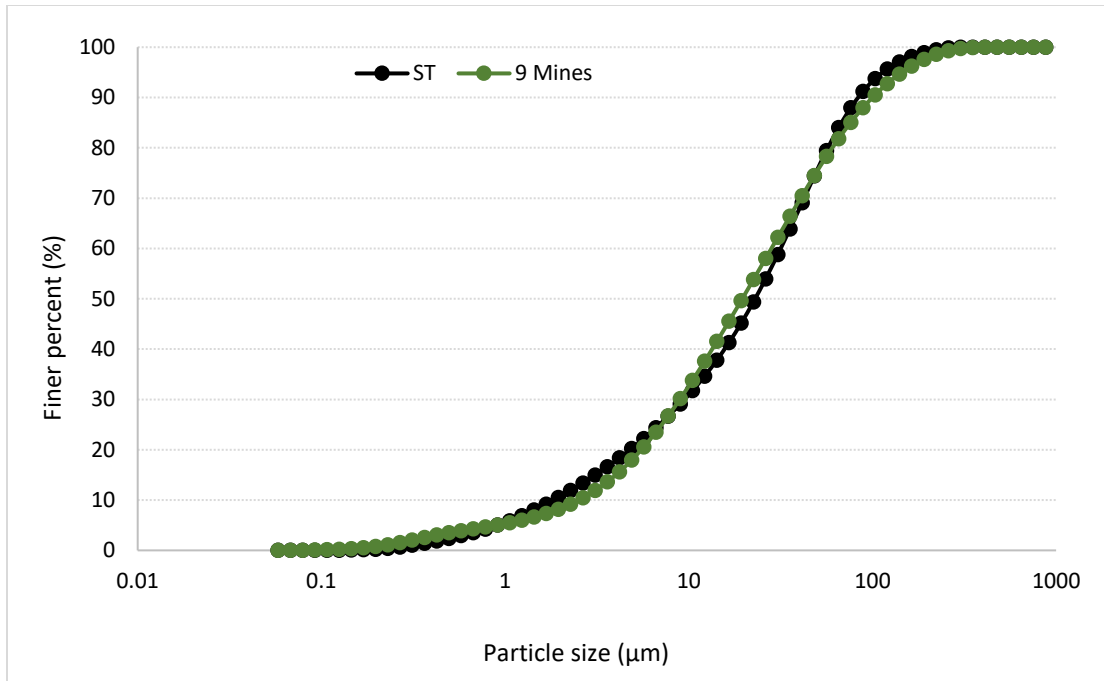


Figure 4.1 Grain size distribution of silica tailings and average grain size distribution of tailings from 9 Canadian mines

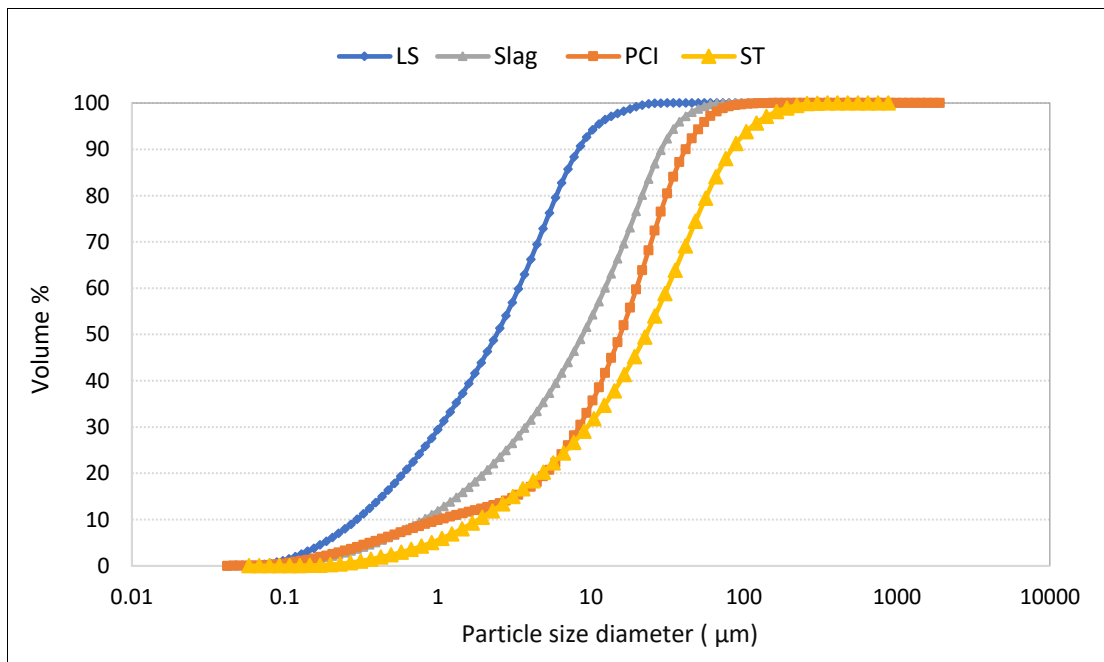


Figure 4.2 Particle size distribution of binders in this study: PCI, Slag, and limestone powder.

4.3.1.2 Binders and water

The most common binding agents in modern mine backfilling, Ordinary Portland cement type I and ground granulated blast furnace slag (GGBFS), are used in this study. The physical and chemical properties of the

two binders are reported in Tables 4.1 and 4.2, respectively. All CPB samples, with both binary and ternary binder, are prepared with tap water.

4.3.1.3 Limestone powder

Limestone is used as a filler material in the form of pure CaCO_3 to avoid the effect of chemically active clay minerals and other impurities that may be present in raw limestone. The density of the LS used in this study was 2.93 g/L, with a specific surface area of 2.69 (m^2/kg), the Fisher Science Education product. The particle size distribution of LS powder compared to ST and binders is shown in Figure.4.2. It is observed that the limestone powder is finer than slag and cement with a d_{50} of 2.31 μm .

Table 4.1 Physical composition of tailings and binders used.

Element Unit	RD (g/lit)	Ss (m^2/g)	D ₁₀ μm	D ₅₀ μm	D ₆₀ μm
ST	-	-	1.9	22.5	31.5
Avg of 9 types of tailings			1.8	20	30.8
PCI	3.1	1.3	0.99	14.9	39.76
Slag	2.8	2.1	0.77	8.48	27.6
LS	2.93	2.69	0.28	2.31	7.94

Notes: RD: relative density, Ss: specific surface area. (m^2/g)

Table 4.2 Main chemical composition of binders

(wt.%)	MgO	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	CaCO ₃
PCI	2.65	62.82	18.03	4.53	2.70	3.82	-
Slag	10.98	41.14	34.32	9.54	-	3.87	-
LS	-	-	-	-	-	-	99.9

Notes: MgO: magnesium oxide, CaO: Calcium oxide, SiO₂: Silicon dioxide, Al₂O₃: Aluminium oxide, Fe₂O₃: Ferric oxide, SO₃: Sulfur trioxide, CaCO₃: Calcium carbonate

4.3.2 Sample preparation and curing

The experimental laboratory program consists of different binary and ternary mixes, cast, cured and tested over different curing time as shown in Table 4.3, 4.4. CPB paste mixtures with PCI/Slag proportion, 50/50 and 80/20, and ternary mixes (PCI-Slag-LS) with 5, 10, and 20% of LS in replacement of slag. Constant water/ binder (W/B) ratio of 7.35 and 4.5 weight % of the binder was used to prepare CPB samples for UCS tests and monitoring experiments.

Table 4.3 Summary of binder mix composition of CPB specimens and curing times for UCS testing.

Group	Sample nomenclature	Binder ^(a) Content (%)	%PCI in the binder	%Slag in the binder	%LP in the binder	W/B	Curing times (days)	No. of samples
A	50P-50S-0L	4.5	50	50	0	7.35	1, 3, 7, 28, 60,90	18
	50P-45S-5L		50	45	5		1, 3, 7, 28, 60,90	18
	50P-40S-10L		50	40	10		1, 3, 7, 28, 60,90	18
	50P-30S-20L		50	30	20		1, 3, 7, 28, 60,90	18
B	80P-20S-0L		80	20	0		1, 3, 7, 28, 60,90	18
	80P-15S-5L		80	15	5		1, 3, 7, 28, 60,90	18
	80P-10S-10L		80	10	10		1, 3, 7, 28, 60,90	18
	80P-0S-20L		80	0	20		1, 3, 7, 28, 60,90	18

PCI, Portland cement type I; Slag, ground blast furnace slag; W/B ratio, the water mass divided by the binder mass. Binder Content (%) = Mass of binder x100/ (Mass of dry soil + Mass of binder); LP: limestone powder; Binder^(a) = PCI + Slag + LP; % is a weight %

Table 4.4 Summary of binder mix composition of CPB specimens and curing times for monitoring EC, temperature and suction.

Group	Sample nomenclature	Binder ^(a) Content (%)	%PCI in the binder	%Slag in the binder	%LP in the binder	W/B	Duration (days)	Number of samples
A	50P-50S-0L	4.5	50	50	00	7.35	Up to 30	1
	50P-45S-5L		50	45	05	7.35		1
	50P-40S-10L		50	40	10	7.35		1
	50P-30S-20L		50	30	20	7.35		1
B	80P-20S-0L	4.5	80	20	00	7.35	Up to 30	1
	80P-15S-5L		80	15	05	7.35		1
	80P-10S-10L		80	10	10	7.35		1
	80P-0S-20L		80	00	20	7.35		1

4.3.3 Mechanical tests

To investigate the effect of binary and ternary binders on the mechanical properties of CPB, unconfined compressive strength (UCS) was performed on all the samples. All the ingredients in predetermined proportions were dry mixed for 2 minutes, followed by 7 minutes of wet mixing in the mixture to ensure the

homogeneity of the mix (Bian et al., 2019). Molds of size 5cm x 10cm were filled with freshly prepared CPB, followed by tapping to remove entrapped air, and sealed with plastic caps. The UCS test is conducted using the automated testing machine of 50 kN capacity at the rate of 1mm/min. Further, 144 samples with different mix proportions, as presented in Table 4.4, were cured for 1, 3, 7, 28, 60, and 90 days at 20 °C, room temperature, and tested for UCS in accordance with ASTM-C39/C39M-21. The ultimate failure load and pattern were recorded, and stress and stress-strain curves were plotted. Each sample was tested thrice for accuracy and repeatability of the test results.

4.3.4 Microstructural tests and analysis

To understand the phases of hydration product development in the CPB mixes with binary and ternary binder, microstructural analysis was carried out using thermogravimetry (TG/DTG). Samples were prepared using a water-to-binder ratio of 1 and cured in a temperature-controlled oven at about 40 °C for four days. The cured specimen is then crushed and sieved through a sieve size of 40 microns, and about 20 gm of powder is tested for changes in mass while the temperature of a sample is changed over time. The mercury intrusion porosimeter (MIP) test was also conducted over CPB samples to understand the pore structure evolution. Cured CPB samples for a predetermined period of age 3 and 28 days were oven-dried, cut into 2cm x 2cm cubes, and tested for pore size and pore volume distribution using Micromeritics Auto Pore III 9420 mercury porosimeter.

4.3.5 Monitoring

To investigate the progress of hydration product development (hydration mechanism) and changes in the structure of CPB samples over time, specimens were monitored for up to 30 days by measuring suction development, Electrical Conductivity (EC), and temperature changes. Fresh CPB mix was prepared, and a mold of size 20cm x 10cm was used for the monitoring program. The temperature, EC, and suction were monitored using a sensor 5TE (Pessl Instruments GmbH, Weiz, Austria) that measures the EC in the range of 0–23 ds/m with an accuracy of +/- 0.1. Readings were recorded using a data logger (Em50, Decagon Devices, Inc.). Changes in electrical conductivity during monitoring indicate hydration progress by measuring the ions released during the chemical reaction of binder hydration with water. To measure the suction development in the mixes, dielectric water potential sensor MPS-6, with six-point calibration that results in research-grade accuracy of +/- 10% for a measurement range of –9 kPa to –100 kPa, is used in this study.

4.4 Experimental Results and Discussion

4.4.1 CPB with binary cement blends

4.4.1.1 Strength development in CPB with binary cement blends

Figure 4.3 shows typical results of the effect of the binary binder, prepared with different proportions of PCI/Slag mix, 50/50 and 80/20 wt.%, on the strength development of CPB during the curing time up to 90 days. It is observed that increasing the levels of cement replacement by slag had a negative effect on the strength development at early ages. The early age compressive strengths of all samples in Group A, with PCI/Slag 50/50 (50P-50S-0L), were observed to be lower than those in Group B, with PCI/Slag 80/20 (PCI/Slag 80/20). Figure 4.3 shows that sample 80P-20S-0L (Group B PCI/Slag 80/20) has compressive strength values of 176 kPa, 348 kPa, 517 kPa at 1, 3, and 7 days of curing, respectively, which are higher than sample 50P-50S-0L (Group A, PCI/Slag 50/50), 145 kPa, 309 kPa, and 458 kPa at 1, 3, and 7 days of curing, respectively. This higher strength and rate of strength increase in the Group B specimens at early ages is related to their higher cement content (80% cement in Group B versus 50% cement in Group A). Since higher cement content increases the rate of hydration reaction, it leads to the formation of more hydration products, such as calcium silicate hydrate (C-S-H), ettringite, and calcium hydroxide (CH), in the samples with higher cement content. In addition, it increases the volume of the cement paste. It forms a stronger cementitious matrix structure due to improved cementation and pore structure refinement, which ultimately results in higher mechanical strength at early age (Ali et al., 2021). In contrast, CPB with higher slag content (Group A) generates a lower amount of hydration products due to the slow hydration mechanisms of slag at early ages, which has also been demonstrated in many previous studies (e.g., Haruna and Fall, 2021).

The formation of more hydration products in the higher cement content (80 wt.%) samples, 80P-20S-0L, than in the lower cement content (50 wt.%) samples, 50P-50S-0L, is confirmed by the results of the TG/DTG test, as shown in Figure 4.4 a, for 3 days of curing. The peak between 50 °C and 100 °C represents the mass loss by decomposition of chemically bound water, mainly from, C-S-H, ettringite, and the peak between 420 °C and 450 °C represents the dihydroxylation of $\text{Ca}(\text{OH})_2$ (Arora et al., 2016c; Deboucha et al., 2017b; Xu et al., 2022). It is observed that the peak mass loss between 50 °C and 100 °C is higher for samples 80P-20S-0L than 50P-50S-0L, indicating the decomposition of more hydration products formed in the former than in the latter. Furthermore, in sample 80P-20S-0L, the sharp drop in mass observed between 420 °C and 450 °C represents the dihydroxylation of $\text{Ca}(\text{OH})_2$. Higher $\text{Ca}(\text{OH})_2$ in the 80P-20S-0L system indicates an increased rate and amount of chemical reaction and the formation of more hydration products, in the sample with higher cement (Deboucha et al., 2018).

On the contrary, sample 50P-50S-0L, with higher slag content (50 wt.%) acquired higher strength at later ages from 28 days and increased rapidly up to 90 days compared to sample 80P-20S-0L with lower slag content (20 wt.%). The UCS values for the later ages of 28, 60, and 90 days for sample 50P-50S-0L were

noted at 966 kPa, 1338 kPa, and 1480 kPa, respectively, and were observed to be significantly higher than in sample 80P-20S-0L, i.e., 881 kPa, 1140 kPa, and 1281 kPa, respectively. This effect was attributed to the latent pozzolanic and hydraulic effect of the slag. Slag reacts with Ca(OH)_2 formed during the cement hydration reaction and produces secondary C-S-H, in higher amounts the higher the slag content in the mix (Arora et al., 2016c). The formation of more hydration products is validated by the TG/DTG results. As presented in Figure 4.4 b, the first peak at 50-100°C is related to the dehydration of hydration products and ettringite, where the sudden mass loss occurred in sample 50P-50S-0L. This clearly indicates the higher amount of hydration products in the mixture. In addition, the second peak between 420 and 450°C, associated with the decomposition of calcium hydroxide (portlandite), was lower in sample 50P-50S-0L, compared to sample 80P-20S-0L, indicating greater CH consumption, due to a higher proportion of slag. Further, Figure 4.5 shows the pore refinement in the samples at 3 and 28 days of curing. Pore refinement is evident in both samples with curing time. Initially, the higher cement content resulted in a finer pore structure; however, the samples with higher slag content observed pore refinement at a later age. This indicates the production of more hydration products in the sample with higher slag content due to the secondary C-S-H production capacity of the slag at later age, resulting in pore refinement. In addition to the chemical reaction, the physical characteristics of the slag (i.e., finer than the cement) also influenced the refinement of the pore structure and ultimately the strength improvement due to better packing of the particles in the mix as a result of the filler effect. In addition, in the literature, it is observed that slag particles smaller than 10 mm participate in the strength improvement at young age, while those from 10 to 45 mm do so after 28 days, respectively (Carrasco et al., 2005b; Menéndez et al., 2003). In agreement with these results, the slag used in this research work contains more than 50% of 8.25 mm particles, which contributed to the improvement of the young age strength of CPB samples. A similar effect of adding slag in place of cement has also been noted elsewhere (Arora et al., 2016c; Panesar & Francis, 2014).

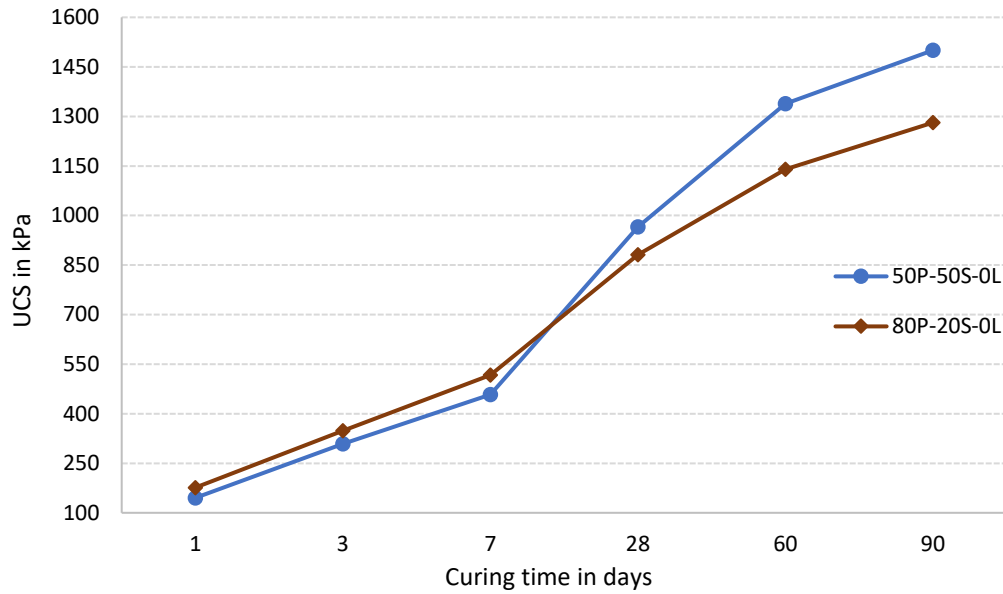
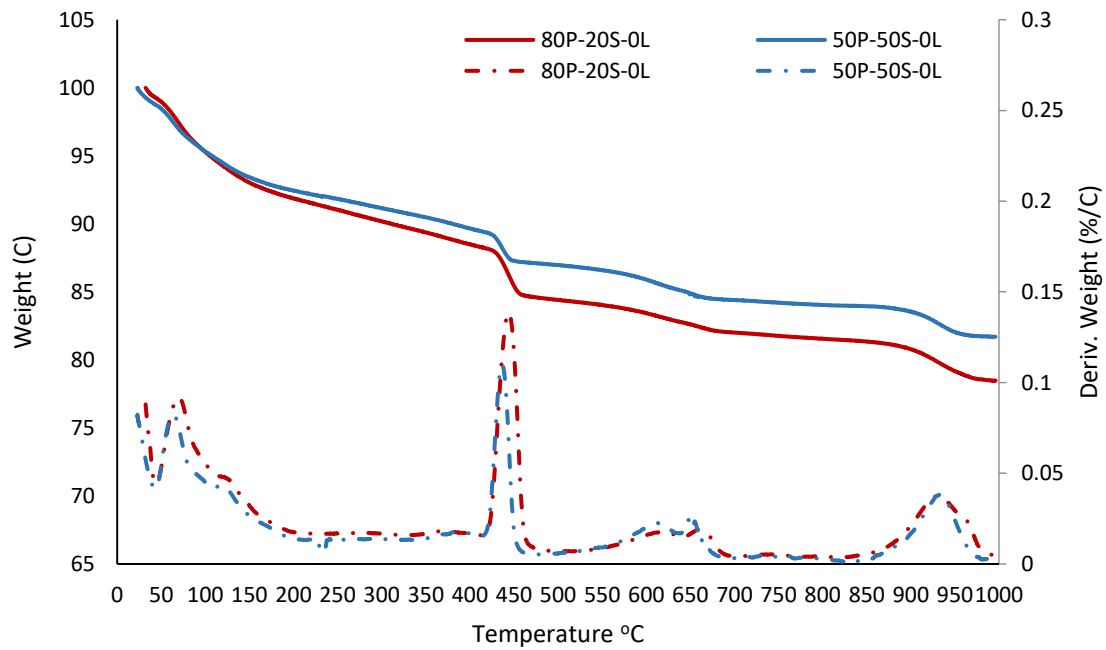
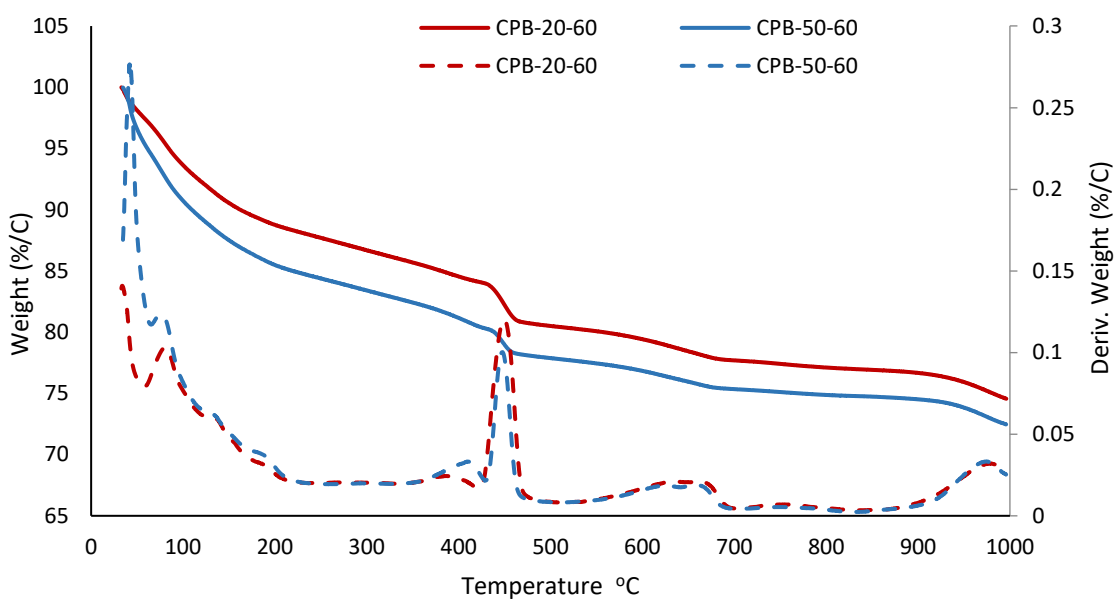


Figure 4.3 Effect of PCI/Slag ratio (50/50 and 80/20) on strength development with time up to 90days

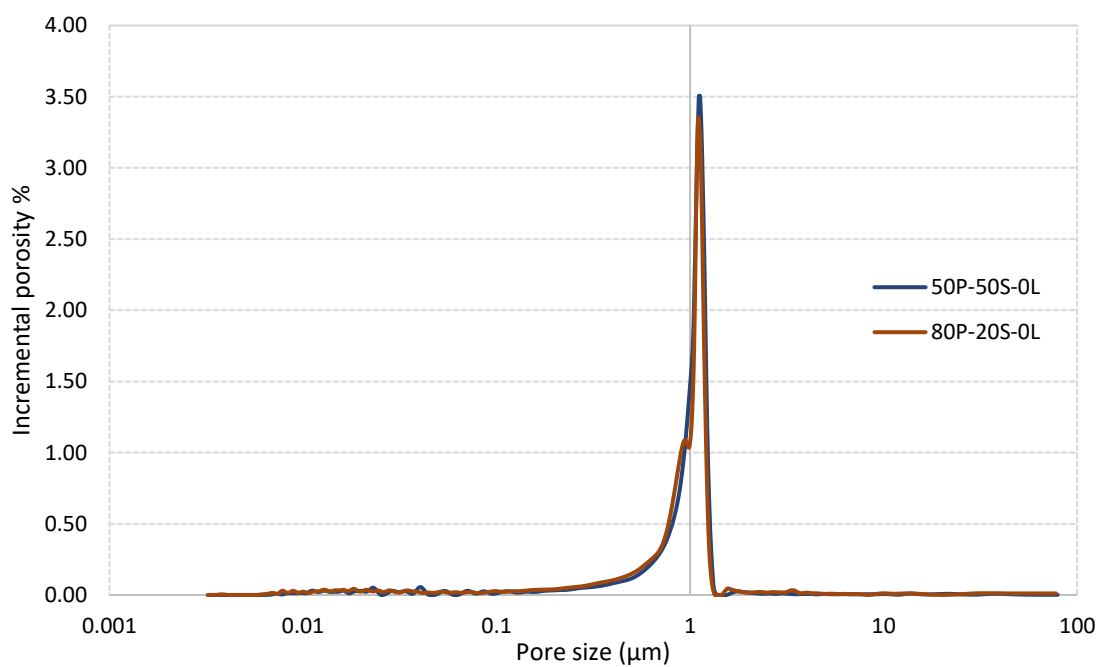


a) 3 days



b) 28 days

Figure 4.4 TG/DTG profiles/curves for binary control samples at (a) 3 days, and (b) 28 days



a) 3 days

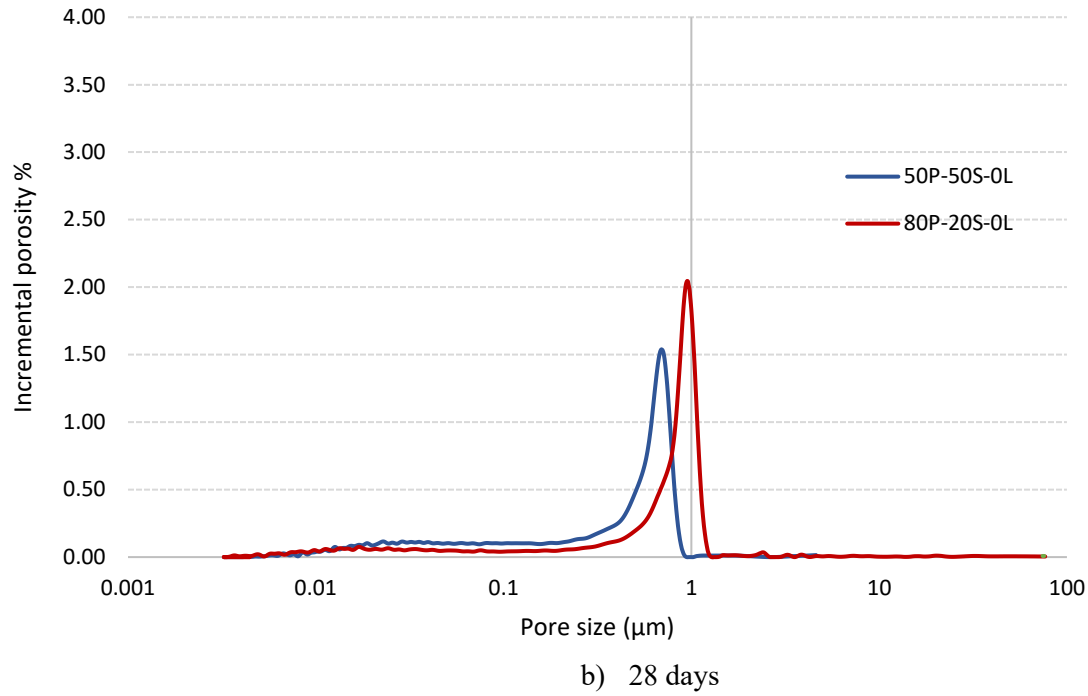


Figure 4.5 Effect of PCI/Slag proportion on pore size distribution: (a) 3 days, and (b) 28 days

4.4.1.2 Suction development in CPB with binary cement blends

The effect of binary binders with PCI/Slag (50/50, 80/20) on the suction development as a function of time is presented in Figure 4.6. It is observed that the addition of increasing percentages of slag in place of cement has a negative effect on the rate of suction development at early ages. In CPB samples 50P-50S-0L with higher proportions of slag (50 wt.%), the rate of suction development is lower than that of sample 80P-20S-0L, with lower proportion of slag (20%). The difference in suction values between the two aforementioned samples was not significant at very early ages; however, it increases drastically over the curing time. Subsequently, on day 7, higher rate and suction values were observed in sample 80P-20S-0L compared to sample 50P-50S-0L, i.e., 92 and 52 kPa, respectively. This is again attributed to the higher reactivity of cement and slag's slower reaction kinetics at an early age (Deboucha et al., 2015, 2018). In addition, the reduction in cementitious material (dilution effect) (Cyr et al., 2005)), reduces the rate of hydration reaction, resulting in less water consumption, and lower suction development in the mix and with a higher amount of slag. Since suction development leads to an increase in effective strength development, in agreement with this statement, lower suction development in sample 50P-50S-0L (50 wt.% slag), resulted in lower UCS values: 144 kPa, 325 kPa, 460 kPa, at 1, 3 and 7 days, respectively. Conversely, samples with higher

cement content 80P-20S-0L, developed higher suction and gave higher mechanical strength 176 kPa, 359 kPa, 510 kPa at 1, 3 and 7 days respectively.

On the contrary, after 14 days of curing, the rate of suction development in sample 50P-50S-0L increased significantly compared to sample 80P-20S-0L. Subsequently, the suction values of both samples were found to be equal at day 21 of curing and a higher suction rate was observed in the sample with higher slag content. The development of suction is observed to correlate with the development of mechanical strength over the curing time, i.e., at 28 days, the suction value of sample 50P-50S-0L, noted 160 kPa, is higher than that of sample 80P-20S-0L, 152 kPa, following UCS values of 966 and 882 kPa, respectively. This trend of increasing suction and UCS values in samples with higher slag content is again attributed to the pozzolanic and latent hydraulic properties of the slag. Slag reacts with CH produced by cement hydration, resulting in increased water consumption, self-desiccation and reduction of pore water, causing an increase in suction. This reaction is followed by the production of additional secondary C-S-H, which increases strength, as explained in Section 3.1.1 above.

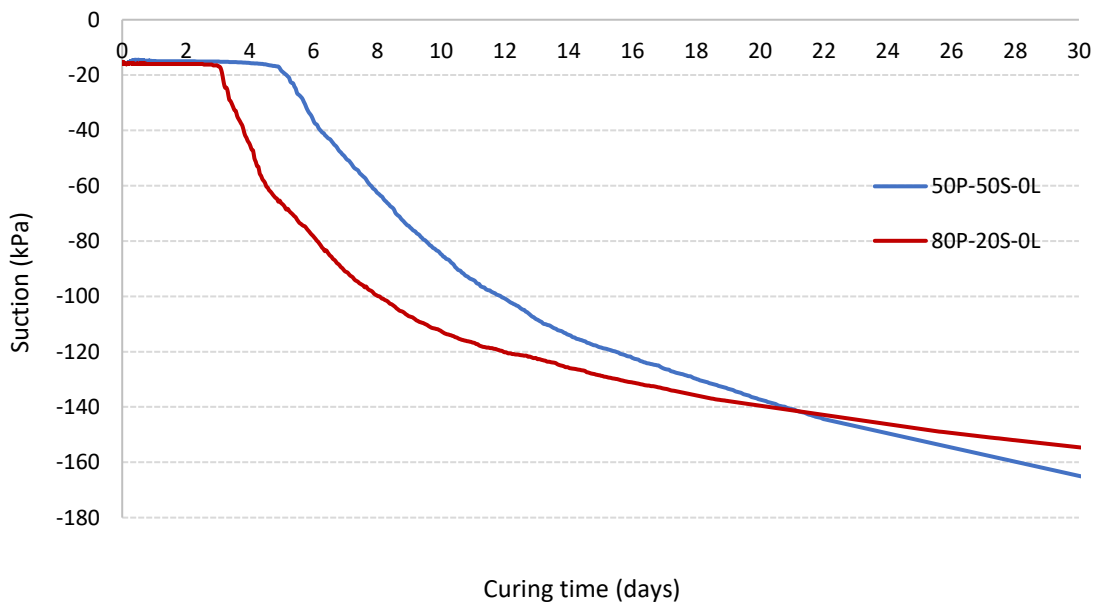


Figure 4.6 Suction development in binary control sample with PCI/Slag (50/50 and 80/20) up to 30days

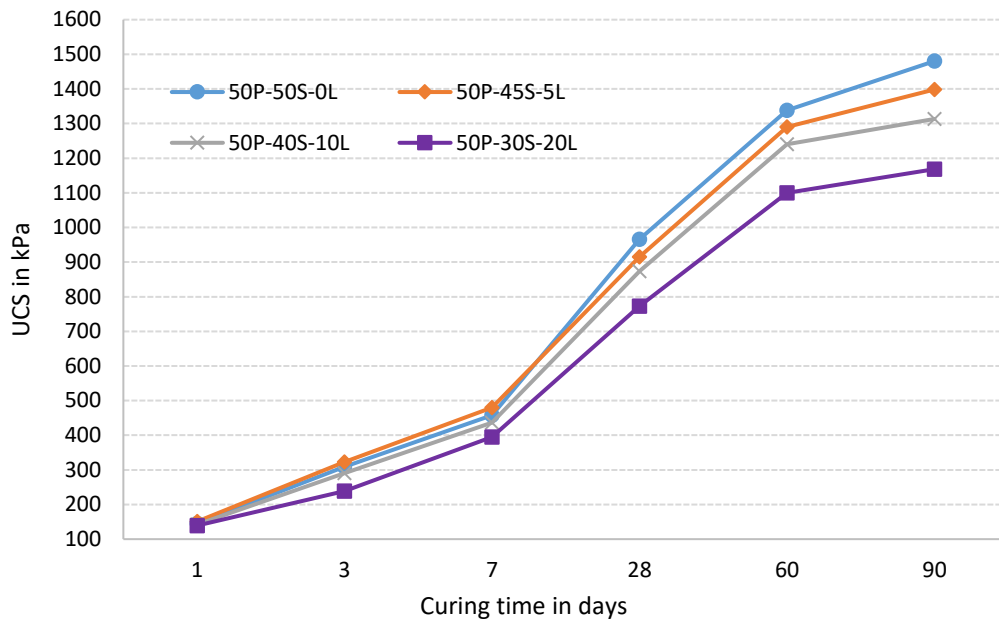
4.4.2 CPB with ternary cement blends

4.4.2.1 Strength development of CPB with ternary cement blends

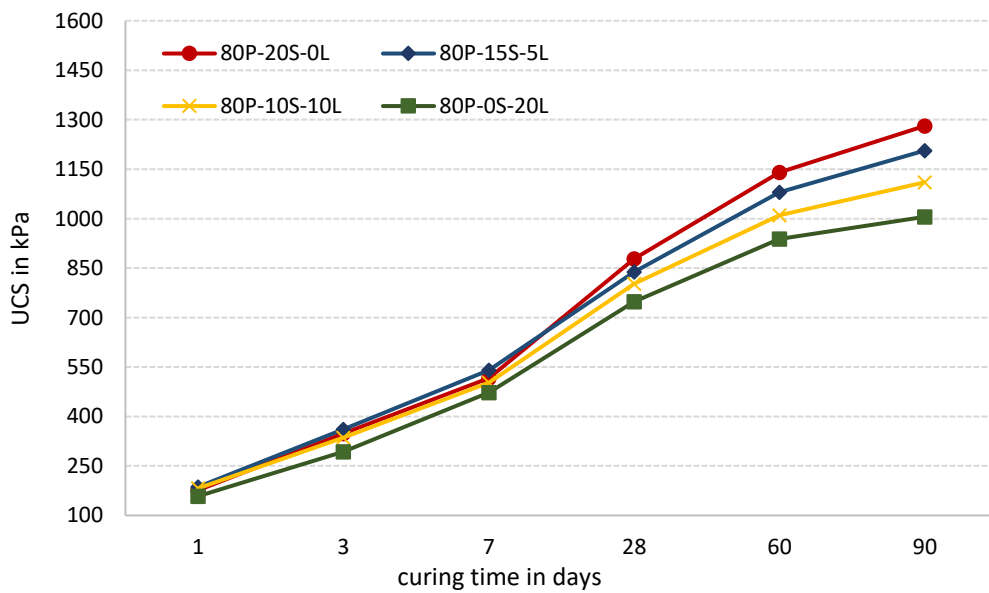
Figure 4.7 Shows the influence of ternary binders (cement, slag and LS) and their proportion on the mechanical strength of CPB specimens over a curing period up to 90 days. The gradual addition of LS (5, 10,

20 wt% of total binder), replacing slag, influenced the hydration rate of cement and slag and the strength evolution of CPB specimens.

As shown in Figure 4.7, at 1 day, CPB ternary samples, group A, 50P-45S-5L, 50P-40S-10L (5 and 10% LS), and group B, 80P-15S-5L, 80P-10S-10L, achieved higher strength than the binary control samples 50P-50S-0L, 80P-20S-0L, (0% LS) of groups A and B, respectively. In addition, the addition of only 5 wt.% LS increased the strength of the samples up to 7 days, while the addition of 10 wt.% LS resulted in a slight decrease in strength. However, the gain and reduction in strength caused by the addition of LS up to 10% is only on the order of +/- 5-10% compared to the control binary sample. This slight gain in strength due to the addition of 5% LS is due to the increased reactivity of LS in the presence of an alumina-rich system (aluminates), where LS acts as heterogeneous nucleation sites for cement and slag, accelerates hydration, and also participates in the formation of carboaluminate and stabilization of ettringite (Lothenbach et al., 2008; Ramezaniapour & Hooton, 2014). This is also supported by the electrical conductivity (EC) results, as shown in Figure 4.8. The evolution of EC in the samples with LS peaked faster, with higher EC values, than its binary control sample, i.e., the EC of the ternary sample of group A, 50P-45S-5L, is noted 3.89 mS/cm, higher than the binary sample 50P-50S-0L, 3.54 mS/cm, and the CE of the group B ternary sample, 80P-15S-5L, is noted 4.56 mS/cm, higher than the sample 80P-20S-0L, 4mS/cm. This indicates a faster rate of hydration in the samples containing added limestone. After 1 day, the higher electrical conductivity pattern with 10% LS persisted, but no increase in strength was observed. This could be due to the larger surface area of the LS particles, which caused increased particle movement in the pore solution. In addition to the acceleration effect, the packing effect caused by the fine limestone particles enhanced the strength gain by improving the packing of the particles. However, the participation of limestone in the hydration process depends on and is limited by the availability of dissolved Al_2O_3 in the mixture available from SCMs with aluminosilicate (Adu-Amankwah et al., 2019; Schöler et al., 2015). Therefore, as expected, additional replacement of slag with limestone (>10%) did not improve strength proportionally with increasing dosages of limestone (Schöler et al., 2015). The formation of higher hydration products in the presence of limestone at early ages is confirmed by the TG/DTG test results, as presented in Figure 4.9. The weight loss at the 25-100°C peak represents the decomposition of chemically bound water primarily from the C-S-H, ettringite, and aluminum ferrite AFm phases. In group A, the control samples 50P-50S-0L, and the sample with 5% LS addition 50P-45S-5L, the weight loss at 25 to 100°C, observed to be almost the same. A similar trend was observed for the Group B samples. In addition, a slightly higher peak and a sharp decrease in mass, observed at the 420°C - 450°C peak, which corresponds to the dihydroxylation of $Ca(OH)_2$, in both samples 50P-45S-5L and 80P-15S-5L, with 5 wt% LS. This confirms the acceleration of the rate of the hydration reaction after the addition of 5 % LS.

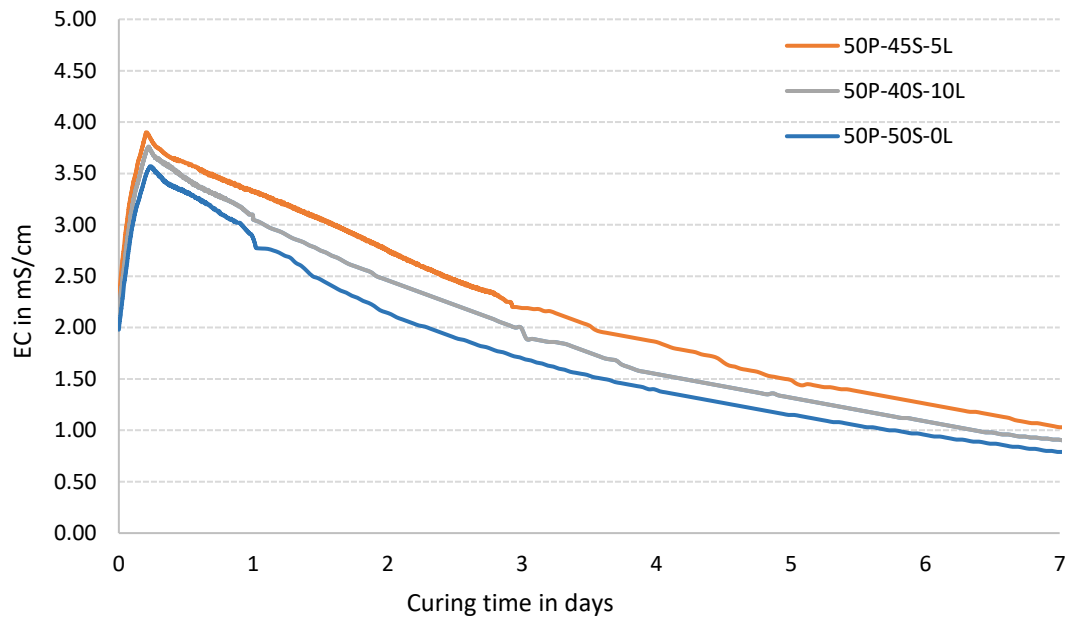


a) Group A CPB samples (binder with 50% Portland cement)

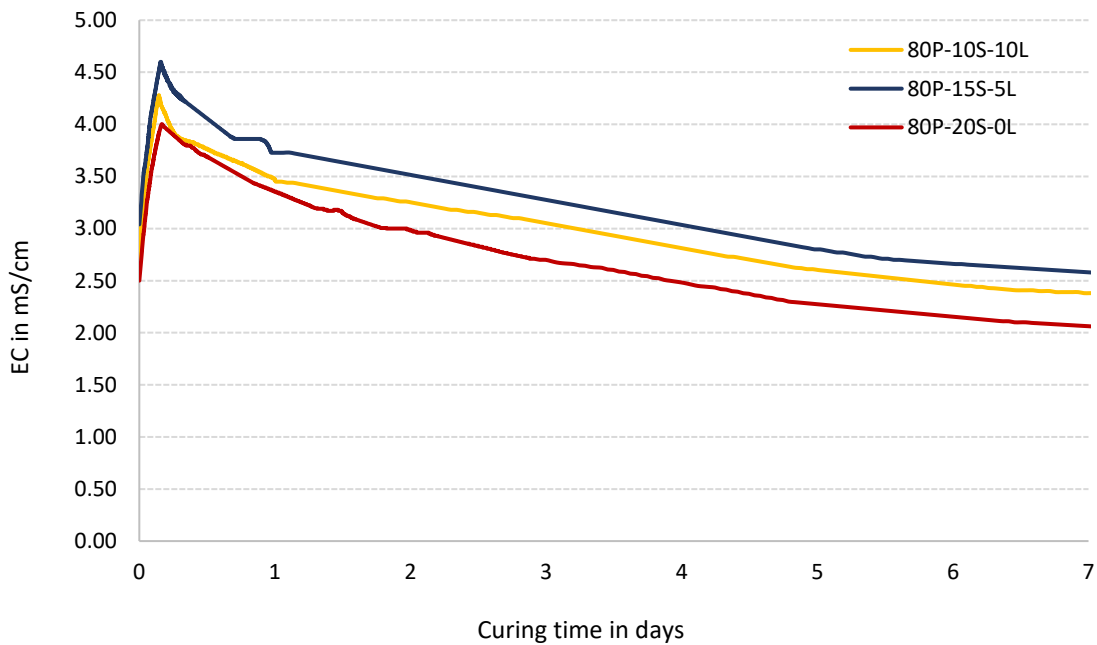


b) Group B CPB samples (binder with 80% Portland cement)

Figure 4.7 Comparison of strength development with increasing LS percentages by weight (0, 5, 10, 20 wt.%): (a) Group A, and (b) Group B samples



a) Group A CPB samples (binder with 50% Portland cement)



b) Group B CPB samples (binder with 80% Portland cement)

Figure 4.8 Comparison of strength development with increasing LS percentages by weight (0, 5, 10, 20 wt.%): (a) Group A, and (b) Group B samples

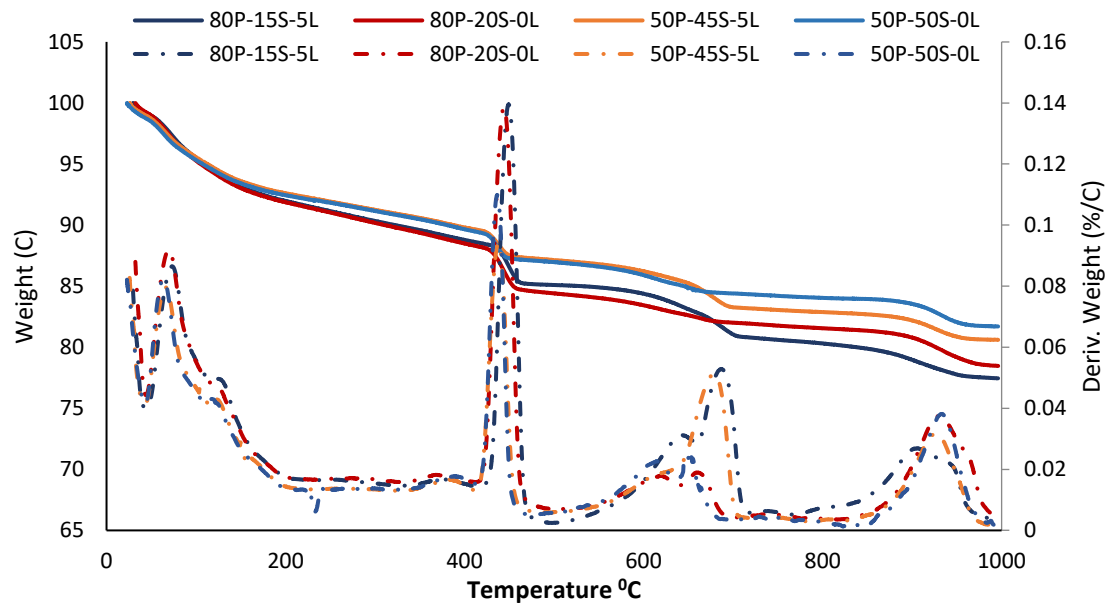
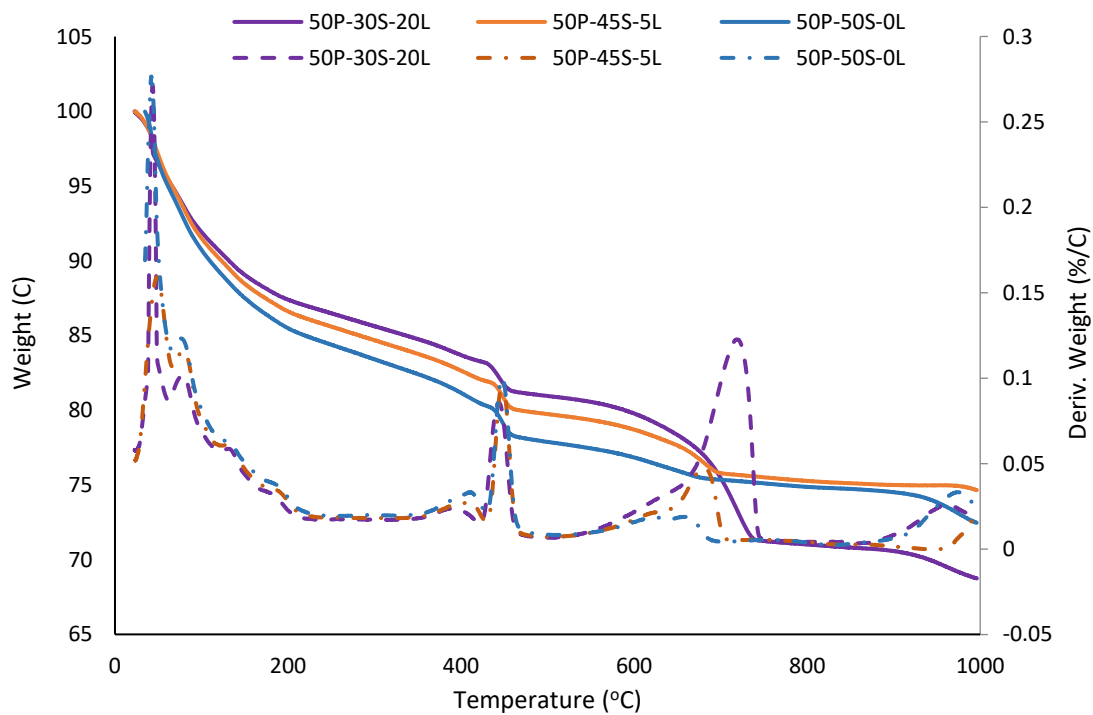


Figure 4.9 Comparison of TG/DTG profiles of binary control samples and samples with 5 wt%. LS at 3 days: Group A, and Group B



a) Group A CPB samples (binder with 50% Portland cement)

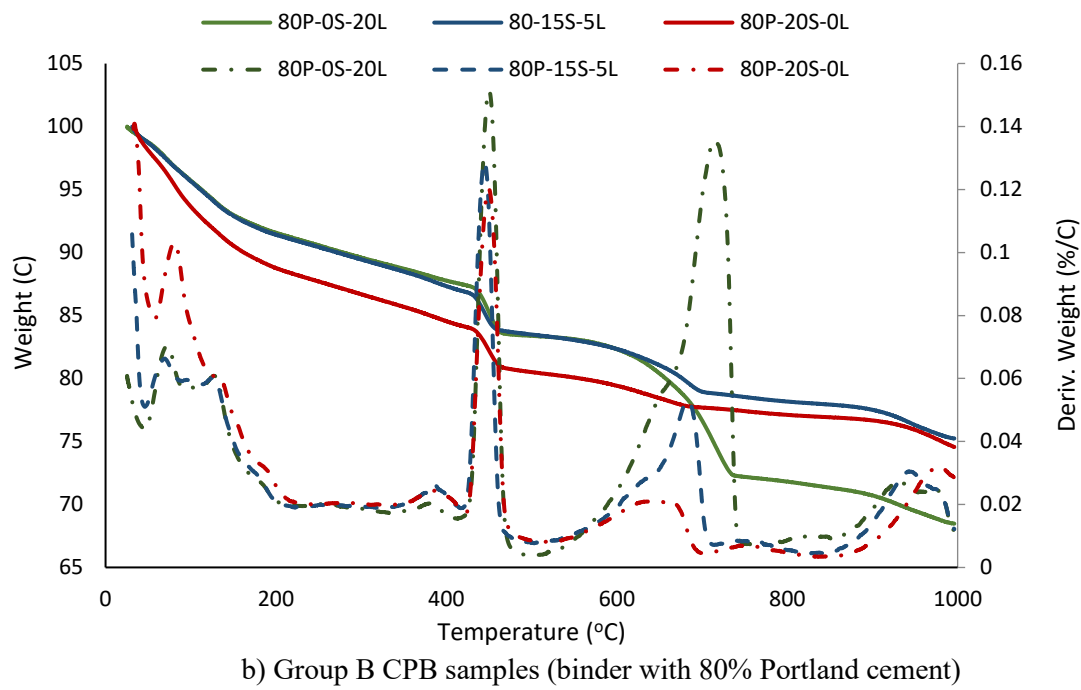
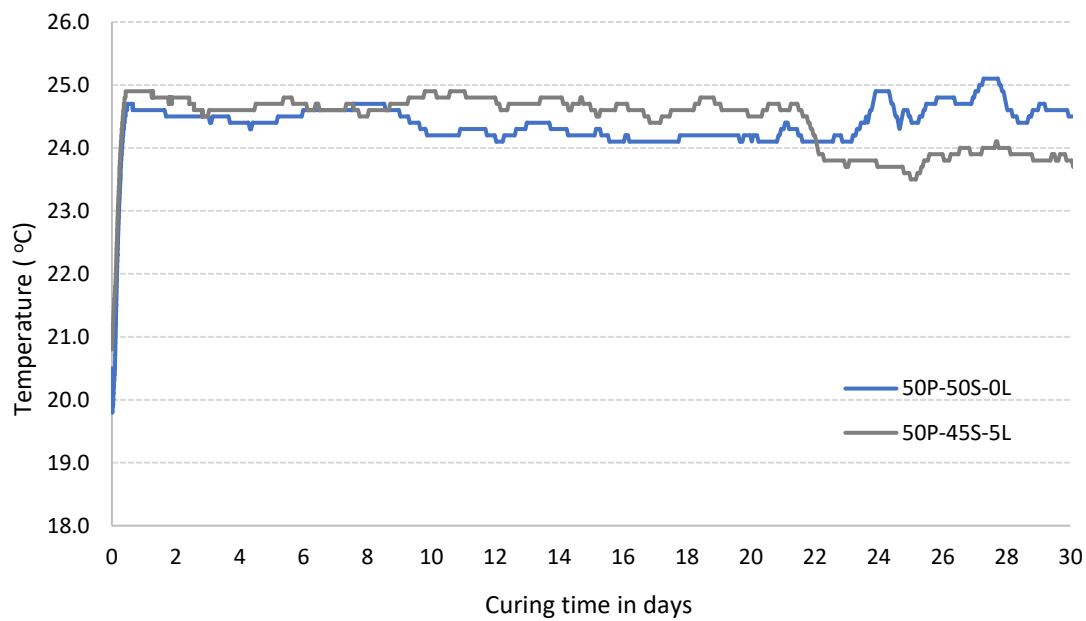
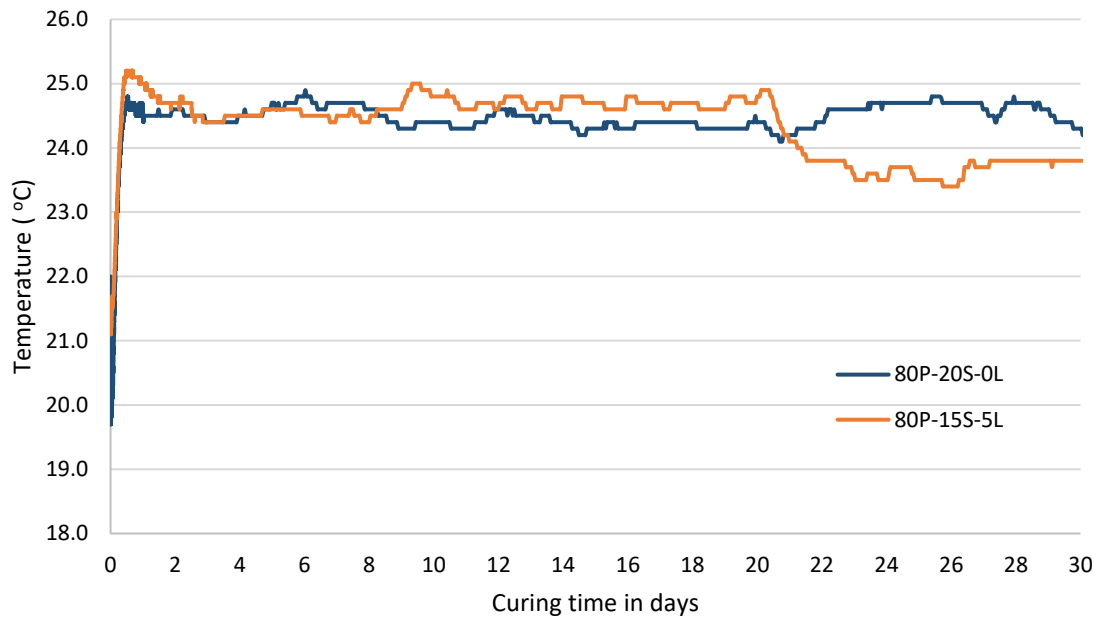


Figure 4.10 TG/DTG profiles/curves - effect of adding LS at 28 days: (a) Group A, and (b) Group B.





b) Group B CPB samples (binder with 80% Portland cement)

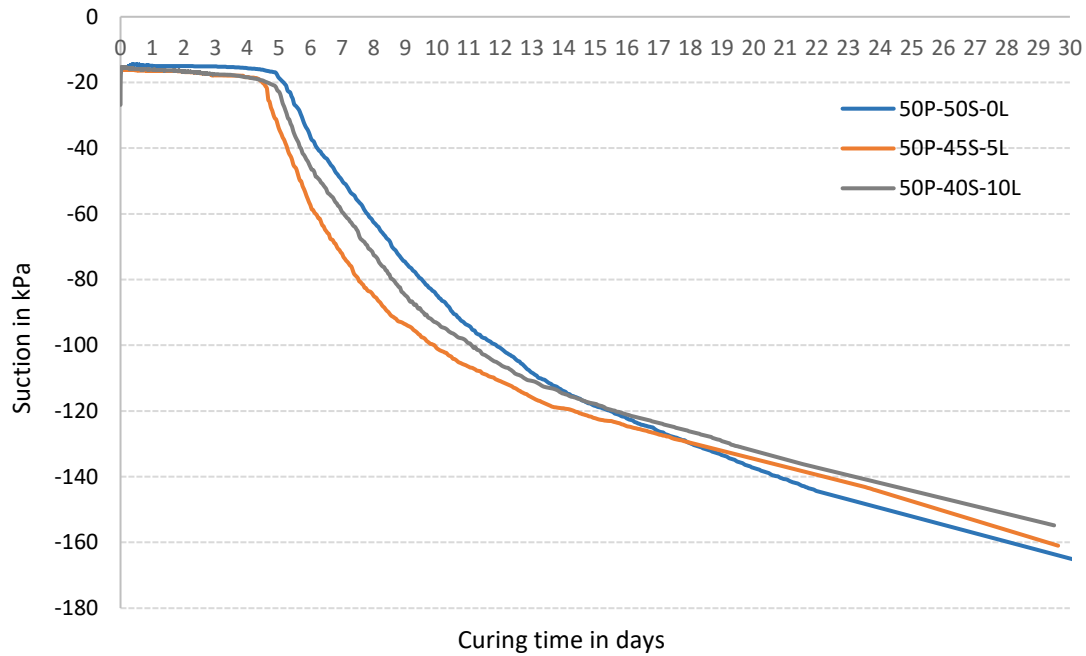
Figure 4.11 Temperature changes over a curing period of 30 days: (a) Group A, and (b) Group B

4.4.2.2 Suction development of CPB with ternary cement blends

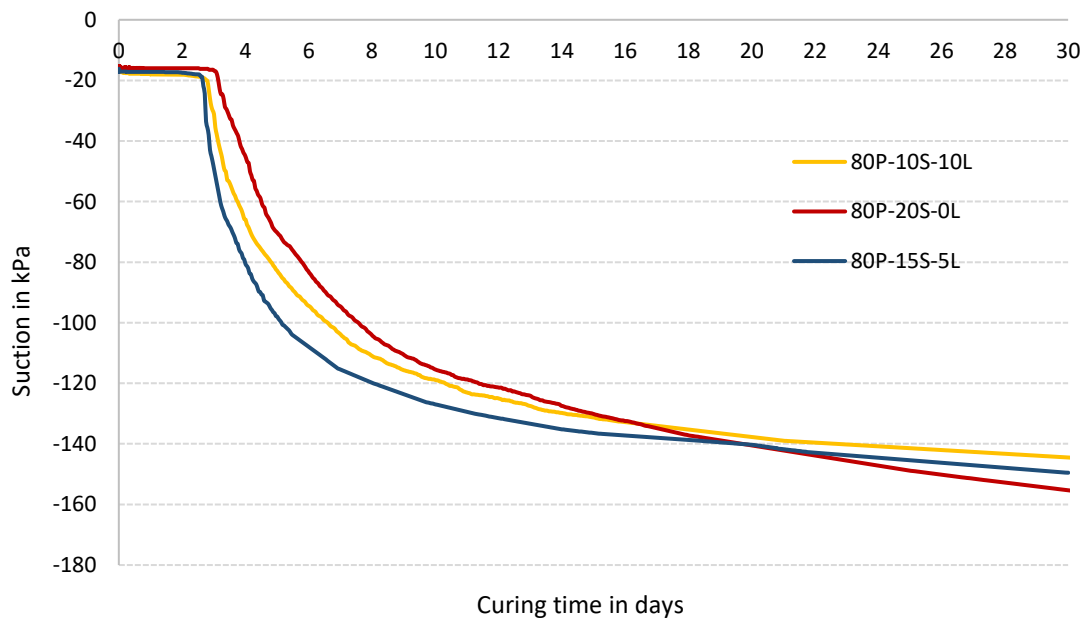
As shown in Figure 4.12, the suction development in CPB specimens with ternary binders exhibits different suction rate and strength gain behavior at early and later ages. The addition of limestone (up to 10%) improved the suction development, in ternary CPB samples at early ages up to 7 days. That is, the suction in sample 50P-50S-0L is observed at 51 kPa, lower compared to the sample with the addition of 5% and 10% LS and a suction of 60 and 73 kPa, respectively. This is consistent with the increase in strength due to the addition of finer LS, as explained in the previous section 3.2.1. A higher suction increase rate is also attributed to the addition of finer limestone. The higher specific surface area of LS provides nucleation sites for the precipitation of cement hydrates and influences the reactivity of cement and slag. This results in an increase in the hydration reaction, greater water consumption, and self-desiccation, which increases pore water dissipation.

However, after 14 days of cure, the sample with added limestone slowed down the suction. On the other hand, the suction is increased in the sample with higher slag content. This trend is followed until the end of the 30-day cure period, i.e., the suction of sample 50P-50S-0L is observed at 160 kPa, compared to samples 50P-45S-5L and 50P-40S-10L, with 157 and 150 kPa, respectively. A similar pattern of suction development is observed in the Group B samples. At 28 days, the binary samples without LS developed greater suction than the samples with ternary binders. This pattern of suction development is the same as the strength development phenomenon at later ages. The increase in suction at later ages in the samples with

slag is attributed to the pozzolanic and latent hydraulicity of the slag. Overall, it is deduced and confirmed that after 14 days, the slag reacts with the CH produced by the cement hydration, consumes more water due to the renewed hydration reaction, and the dissipation of pore water, increases the suction. It is observed that this renewed slag activity is lower in mixes where LS replaces slag, i.e., less slag is available to produce additional C-S-H when almost all of the available cement is already hydrated.



a) Group A CPB samples (binder with 50% Portland cement)



b) Group B CPB samples (binder with 80% Portland cement)

Figure 4.12 Suction development in ternary samples with incremental LS percentages by weight (0 to 10 wt.%): (a) Group A, and (b) Group B.

4.5 Conclusion

In this research, the effect of binary and ternary blended cement binders with varying proportions of slag and cement with cement substitution up to 50%, and incremental percentages of LS (5% up to 20% total binder wt.), in the ternary blend, on properties of CPB mix have been studied for the effects of varying proportions of slag in a binary binder and the effect addition of LS in the ternary binder, on the early-age hydration kinetics, the evolution of mechanical properties and suction development of blended binders have been studied.

From the tests conducted and analysis of the results following conclusions were drawn:

- i) The replacement of cement by slag substitution affected the early-age hydration kinetics of the CPB mix by reducing the rate of hydration reaction subsequently, mechanical properties and suction development at an early age. This strength reduction was attributed to the dilution of potential cementing material and slow hydration of slag. At a later age, however, latent hydraulicity of slag, secondary C-S-H was produced, and samples with higher slag, i.e., 50% slag in it, gained greater strength and suction than mix with 20% slag.
- ii) In comparison to binary binders, ternary binders with moderate slag replacements by LS up to 10% of total binders improved the reaction rate, early-age mechanical performance, and suction of the mixes. This is the result of a combination of physical and chemical effects.
- iii) MIP results shows, continuous progressive pore refinement in all the samples with the curing time. At early ages, up to 7 days, in binary binders, the pore refinement was slightly higher in the sample with higher cement content. However, it was not proportional with cementing material as finer slag also induced the filler effect, filling the pores. However, LS addition induced pore refinement in a ternary binder at an early age but later reduced porosity by diluting slag.
- iv) Overall slag replacement by LS with 10% wt. of total binder is noted as optimum limestone content resulting in the best synergy in the ternary binder to obtain similar or improved properties than binary samples in the CPB mixes.
- v) Further, a detailed study on the chemical effect of limestone, i.e., phase assemblage and LS induced products formed during the hydration reaction and its quantification, actual particle packing, is recommended.

The factors affecting the physical effect of LS on strength evolution can be investigated further by using coarser LS; waste limestone is incorporated to correlate the actual LS and used to compare its effect on mechanical behavior to pure CaCO_3 .

4.6 References

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5. Chapter 5. Synthesis of the Results & Implications for Design

5.1 Introduction

This thesis investigates the effect of a ternary binder that contains PCI, slag, and limestone. Limestone is used in place of slag in CPB mixes up to 20% by weight. The engineering properties of CPB with a ternary binder are also compared with those with a binary binder (control). Chapter 3 details an investigation on the effect of binary and ternary binders on the rheological properties and setting time of CPB material, while Chapter 4 discusses its mechanical strength and suction development. Table 5.1 summarises and details the performed experimental tests as described in Chapters 3 and 4.

Table 5.1 Summary of laboratory tests

Chapter	Test	Cement Content (%)	Tailings	W/C ratio	Binder Content	Binder blends	Water	Curing time
3	Rheology	Yield stress	ST	7.35	4.5	PCI/Slag PCI/Slag/ LS	Distilled water	0, 0.25, 1,2,4 (hrs)
		Viscosity						
		pH & Zeta potential						
		Monitoring Tests (EC, Temp, Suction)						
		Microstructural analyses (MIP, TG/DTG)						
4	Mechanical Strength	UCS					Tap water	1, 3, 7, 28, 60,90 (days)
		Suction						
		Monitoring Tests (EC, Temp, Suction)						
		Microstructural Analyses (MIP, TG/DTG)						

UCS: Unconfined Compression Strength; TG/DTG: Thermogravimetric/Differential Thermogravimetric Analysis; MIP: Mercury Intrusion Porosimetry; EC: Electrical Conductivity.

5.2 Effect of Binary and Ternary Binders on the Rheological Properties and Setting Time of Cemented Paste Backfill

The effect of the addition of binders (cement/slag/limestone), and their dosage, as a binary and ternary binder, on rheological properties (yield stress and viscosity) of CPB samples with respect to curing time and influence on setting time were studied in Chapter 3. Binary samples were prepared with different PCI/Slag proportions, 50/50, 80/20, followed by ternary binders with an incremental dosage of LS 5, 10, 20 wt.% of binder, in replacement with slag. Samples were prepared per the details in Table 5.2, and tests were performed to investigate the impact on rheology. Influence of binary and ternary binders on setting time of CPB samples was examined by conducting setting time test by using Vicat apparatus. Further, microstructural analysis (MIP, TG/DTG) and monitoring tests (EC, Temp, Suction), pH, and zeta potential test were conducted to understand and identify the fundamental mechanisms responsible for the differences observed in the yield and viscosity test results.

The rheological properties of CPB have been observed to increase with increasing curing time as a result of the progressing cement hydration process, consuming water and forming more hydration products with time, regardless of binder and its dosages. For example, in binary binders, as shown in Figure 5.1, the yield stress in samples from both group A and B is increased upon replacing cement with slag from 20 to 50% at all ages. However, the viscosity values decreased in samples from both group A and B when the slag dose was increased from 20 wt.% to 50 wt.%, as shown in Figure 5.2.

In contrast, the addition of limestone results in a decrease in yield stress in both samples from groups A and B as shown in Figure 5.1. With increasing the dosage of LS, the yield stress is reduced even further. This phenomenon may cause an increase in the flowability of the mix. The reduction in yield stress is attributed to the finer particle size of LS, which filled the voids between the coarser tailing and cement particles, allowing more water to enter the mix and providing additional lubrication. However, higher LS dosages may cause an increase in viscosity due to increased SSA availability, which may increase water absorption. Furthermore, due to the nucleation effect, limestone may accelerate the cement hydration process and produce more hydration products, resulting in increase in viscosity as shown in Figure 5.2.

As shown in Figure 5.3, in binary CPB samples the initial and final setting time of CPB samples observed to be increased with increasing percentages of addition of slag in replacement to cement. The initial and final setting time of control sample 80P-20S-0L from group B (binder with 80% PCI) reported at 18 hrs and 32 hrs shorter than that of the control sample of group A 50P-50S-0L (binder with 80%PCI) at 22 hrs and 38 hrs respectively.

Moreover, In CPB samples with ternary binder, it is also noted that the initial and final setting times of the CPB mix were extended by the increase in the amount of limestone. After adding 5% to 20% LS, the initial setting of the samples was recorded as 23.5 and 26.5 hrs, respectively, and the final setting time at 39 hrs

and 41 hrs, respectively. Figure 5.3 shows that the setup time was prolonged with higher dosage when LS was increased from 5% to 20%.

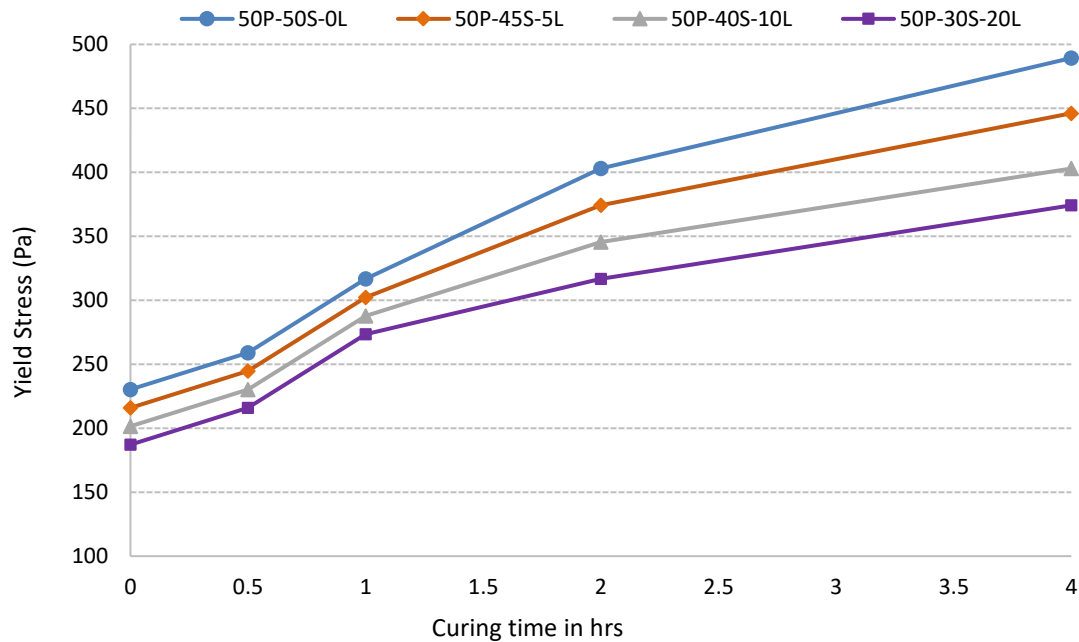
This study helps understand the rheology of CPB samples, which can be used to determine their transportability and setting behaviour. Further, the results obtained from this study will help design a CPB system with binary and a ternary binder containing PCI/Slag/ LS, CPB samples, and factors that influence the flow behavior of the CPB backfill. It is evident that using higher cement-slag (50/50) binder blends increases the yield stress and viscosity of the fill mixture; however, a reduction in yield stress values is noted upon the addition of LS. As yield stress is one of the fundamental parameters for design and backfill (paste), a decrease in yield stress will improve the flowability upon the addition of LS. Further, this will accelerate the backfill process, reducing the risk of damaging the pipes and economic ramifications. Furthermore, these implementations can aid in avoiding laminar flow settings without incurring the high cost of using large-diameter pipes and/or high-pressure pumps.

This study may help understand the design of CPB mixtures with improved flowability with minimum energy and workability, increasing the transportation system's efficiency.

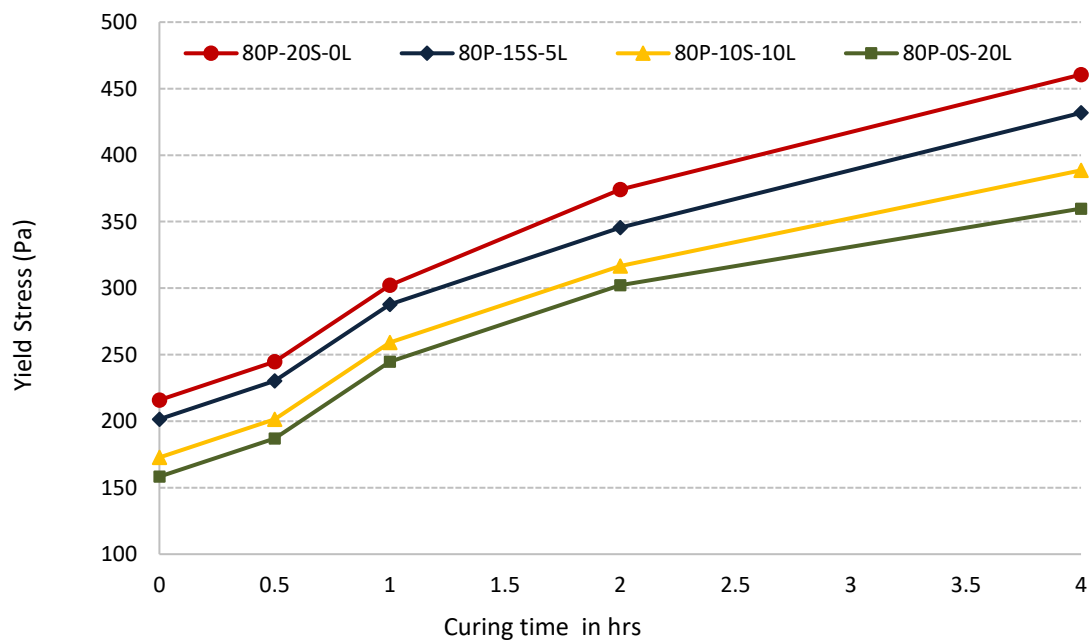
Table 5.2 Summary of binder mix composition of CPB specimens and curing time for rheological properties

CPB Group	Rheological Property	Binder type	Sample	PCI	Slag	LS	Curing Time (hrs)				
							0	0.25	1	2	4
A	Yield stress (kPa)	Binary	50P-50S-0L	50	50	0	230	259	317	403	489
		Ternary	50P-45S-5L	50	45	5	216	245	302	374	446
			50P-40S-10L	50	40	10	201	230	288	345	403
			50P-30S-20L	50	30	20	187	216	273	317	374
B		Binary	80P-20S-0L	80	20	0	216	245	302	374	461
		Ternary	80P-15S-5L	80	15	5	201	230	288	345	432
			80P-10S-10L	80	10	10	173	201	259	317	389
			80P-0S-20L	80	0	20	158	187	245	302	360
A		Binary	50P-50S-0L	50	50	0	3400	3510	3700	4052	4840
		Ternary	50P-45S-5L	50	45	5	3480	3620	3848	4232	5036
			50P-40S-10L	50	40	10	3560	3714	3972	4428	5260
			50P-30S-20L	50	30	20	3648	3806	4112	4578	5430

B	Viscosity (cp)	Binary	80P-20S-0L	80	20	0	3720	3840	4020	4468	5298
		Ternary	80P-15S-5L	80	15	5	3838	3966	4156	4640	5520
			80P-10S-10L	80	10	10	3990	4128	4326	4830	5732
			80P-0S-20L	80	0	20	4098	4248	4458	4960	5882

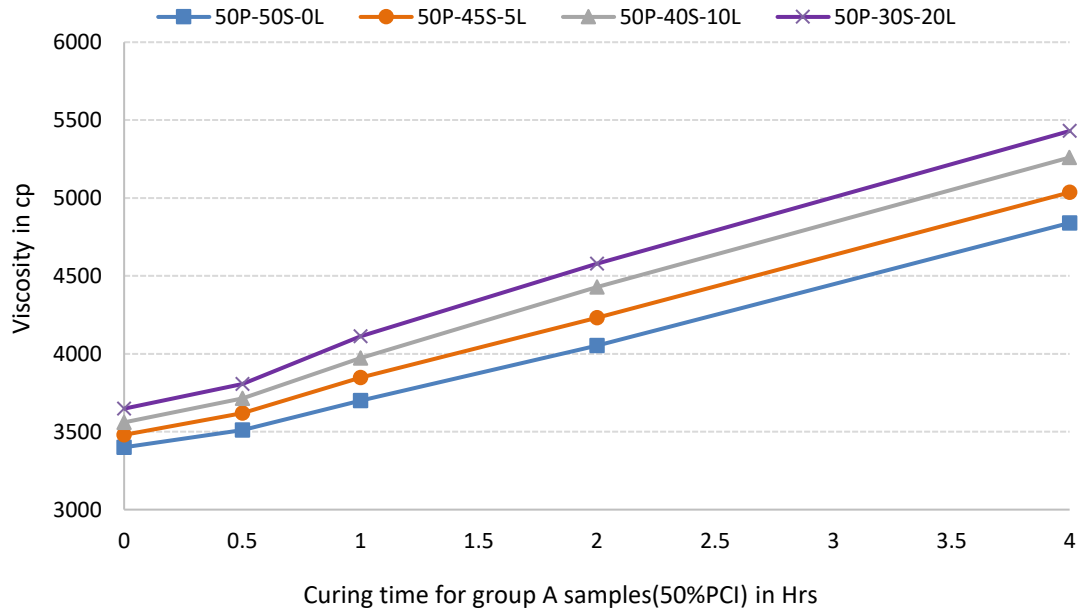


a) Group A CPB samples (binder with 50% Portland cement)

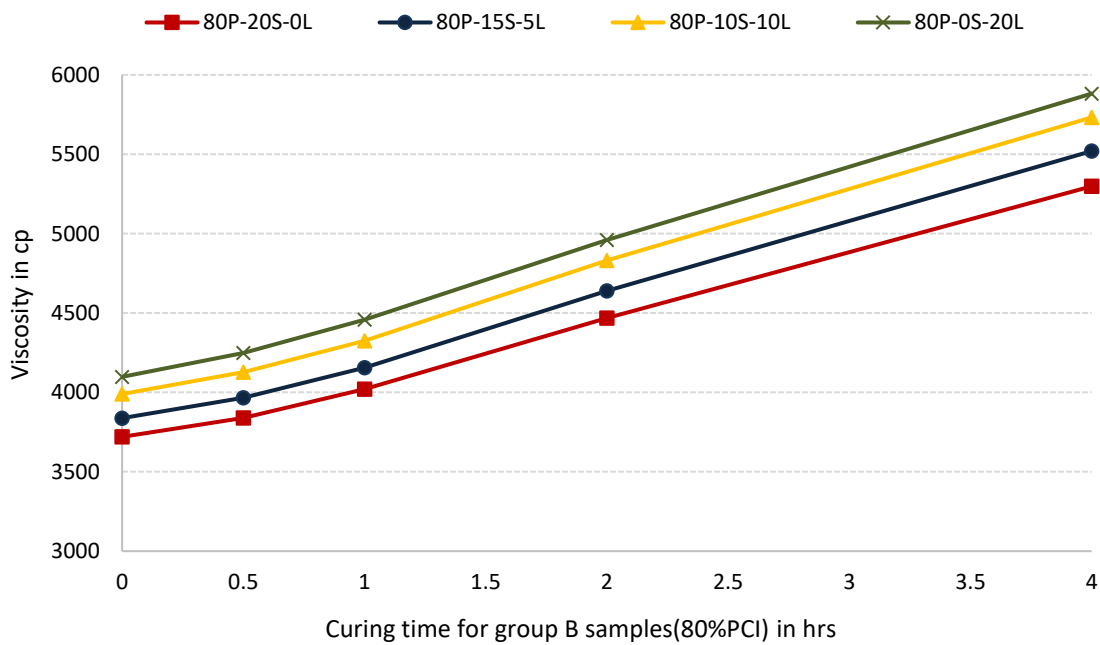


b) Group B CPB samples (binder with 80% Portland cement)

Figure 5.1 Influence of increasing dosage of LS on yield stress of CPB samples: (a) Group A, and (b) Group B.



a) Group A CPB samples (binder with 50% Portland cement)



b) Group B CPB samples (binder with 80% Portland cement)

Figure 5.2 Influence of increasing dosage of LS on viscosity of CPB samples: (a) Group A, (b) Group B.

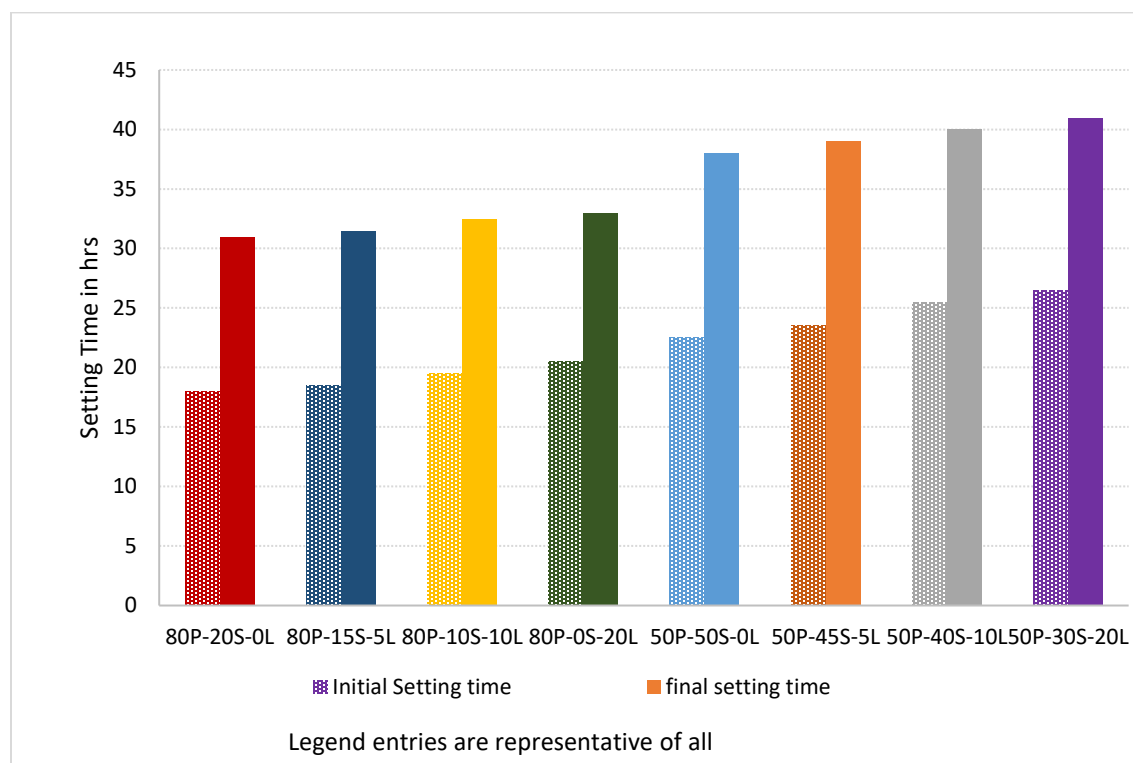


Figure 5.3 The effect of the addition of LS in the CPB samples on the evolution of initial and final setting time a) Group A CPB samples and b) Group B CPB samples

5.3 Effect of Binary and Ternary Binders on the Mechanical Stability (Strength and Suction) of Cemented Paste Backfill

This section compares the mechanical strength of ternary mixes with increasing limestone dosages in replacement with slag to the control binary mixes. Furthermore, the phenomenon of suction development is investigated, as is its relationship with strength gain. At early ages, the compressive strength was reported to be reduced in the CPB samples made using binary binders (Portland cement and slag), with increase in replacement levels of slag. Contrary, at later ages, from 28 days and onwards, the replacement of slag has increased the strength, and strength is higher with an increase in dosage of slag, as summarised in Table 5.3 and depicted in Figure 5.4. Similarly, the suction values were observed less in CPB samples with higher percentages of slag at early ages, and trend is reversed after 28 days (Higher suction values in samples with higher percentages of slag) as shown in Figure 5.5.

Furthermore, when incremental dosages of limestone were added, the early and later age strength of CPB samples was affected differently. Comparing the performance of the samples with 3 dosages, i.e., 5, 10, and 15wt %, of limestone, the sample with 5% addition of LS enhanced the strength gain, and sample with 10

wt.% of addition of LS slightly reduced the strength up to 7 days (+/- 5%). This is attributed to the nucleation effect of finer LS particles. However, further, increase in LS dosage, i.e., 20 wt.% caused a reduction in strength at all ages. The effect of acceleration in the cement hydration by fine limestone in the presence of slag is limited by the availability of dissolved alumina in the solution. Further, an acceleration in hydration cannot compensate for a loss in strength.

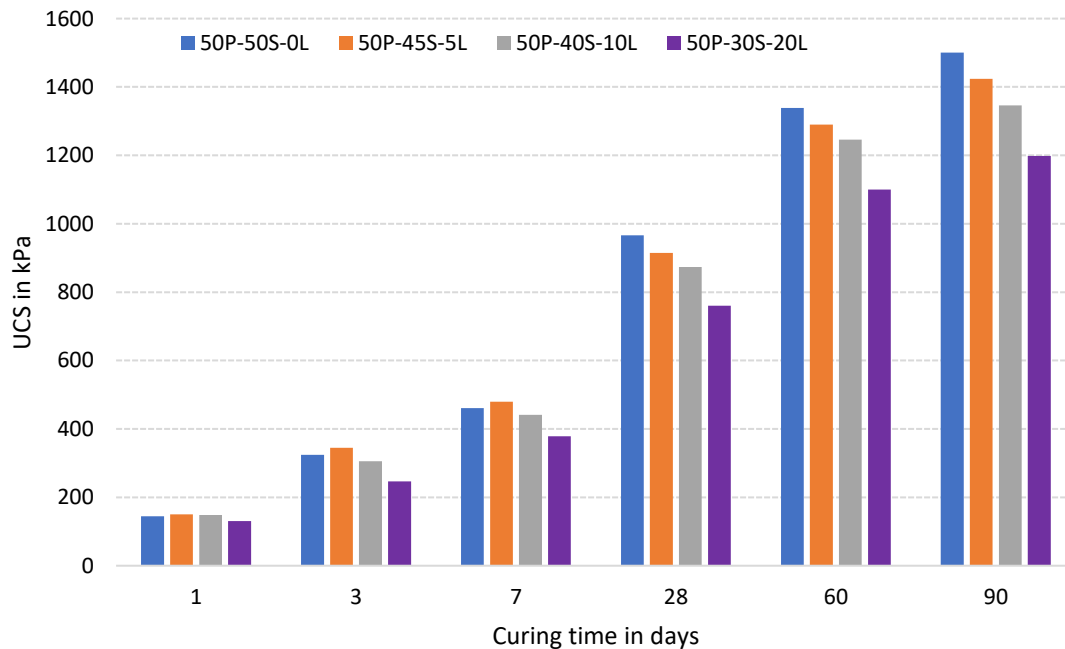
As shown in figure 5.4, a and b, at 28 days and onwards, mixes with higher slag replacement level, i.e., 50 wt.%, achieved higher strengths compared to the blend with 20 wt. % of slag. This was attributed to the latent hydraulicity of slag, which produces additional C-S-H reaction with CH available from cement hydration reaction. Whereas for CPB samples with ternary binders, the slag hydration process dominates the strength evolution of ternary binder mixes after 7 days, outweighing the impact of limestone addition. As a result, at 28 days, CPB samples with a ternary binder achieved lower strengths than their respective control mixes (binary binder mixes). However, with addition 5wt% of LS in ternary binder, the decrease in later age strength was a mere -5%, followed by the addition of 10wt% of LS, causing -10% reduction of UCS. This could be attributed to the dilution of secondary C-S-H producing slag by LS, resulting decrease in strength gain.

Suction development was observed consistent with the increase in strength due to the addition of finer LS as shown in Figure 5.5. In ternary CPB samples, the addition of limestone (up to 10%) increased the suction development at early age of up to 7 days. In other words, sample 50P-50S-0L has a suction of 51 kPa, which is lower than the samples with 5% and 10% LS additions, which have suctions of 60 and 73 kPa, respectively. This is in line with the increased strength brought on using finer LS. However, after 14 days of cure, the sample with added limestone slowed down the suction, and at 28 days, the binary samples without LS developed greater suction than the samples with ternary binders in both the group A and B, as depicted in Figure 5.5.

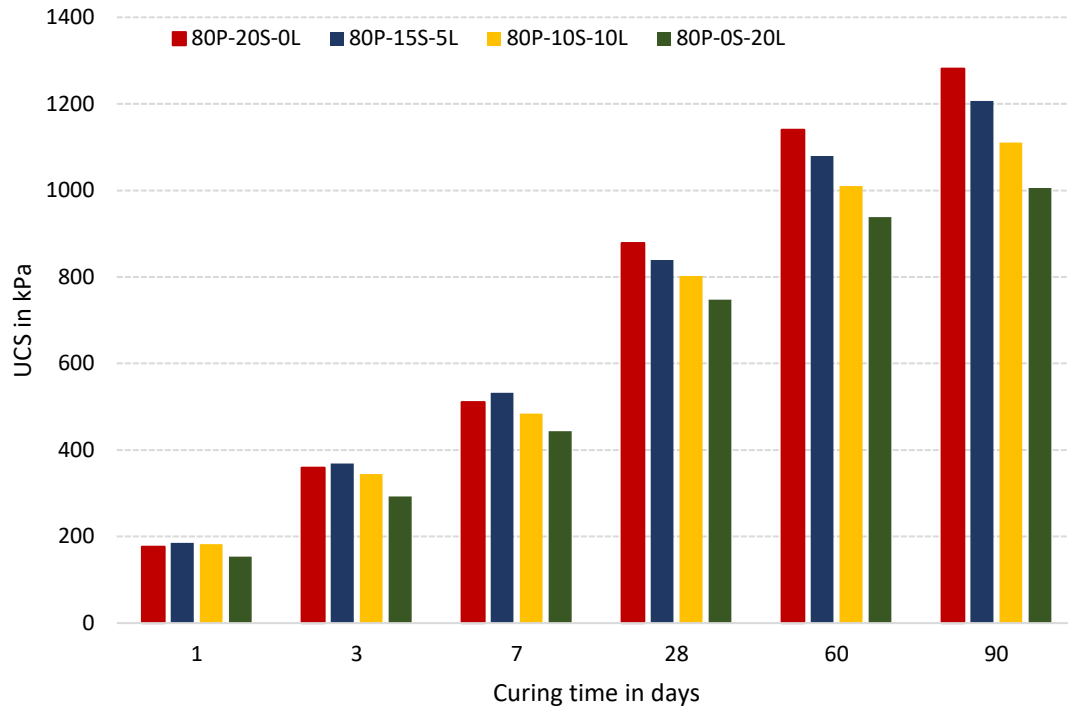
Therefore, it can be inferred from the result that up to 10 wt.% replacement of LS addition is possible to incorporate in replacement to slag with mere strength loss at early age and up to 10% loss in strength at later ages. These findings may help design more efficient and secure CPB structures.

Table 5.3 Summary of binder mix composition of CPB specimens and curing time for UCS testing

CPB Group	Binder type	Sample name	PCI	Slag	LS	Mechanical strength (UCS in kPa)					
						Early age (days)			Later age (days)		
						1	3	7	28	60	90
A	Binary	50P-50S-0L	50	50	0	144	325	460	966	1338	1500
	Ternary	50P-45S-5L	50	45	5	150	344	480	915	1290	1424
		50P-40S-10L	50	40	10	149	305	442	873	1246	1347
		50P-30S-20L	50	30	20	130	247	379	760	1100	1198
B	Binary	80P-20S-0L	80	20	0	176	359	510	878	1140	1281
	Ternary	80P-15S-5L	80	15	5	186	369	532	839	1080	1206
		80P-10S-10L	80	10	10	182	345	484	802	1010	1110
		80P-0S-20L	80	0	20	153	293	443	748	938	1006

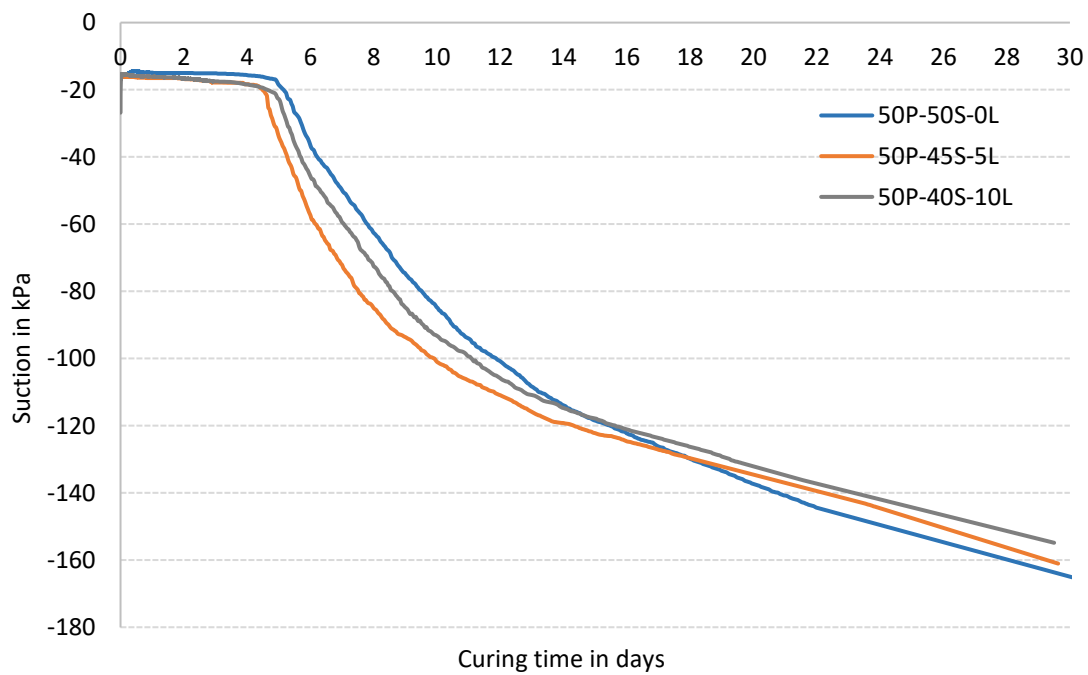


a) Group A CPB samples (binder with 50% Portland cement)

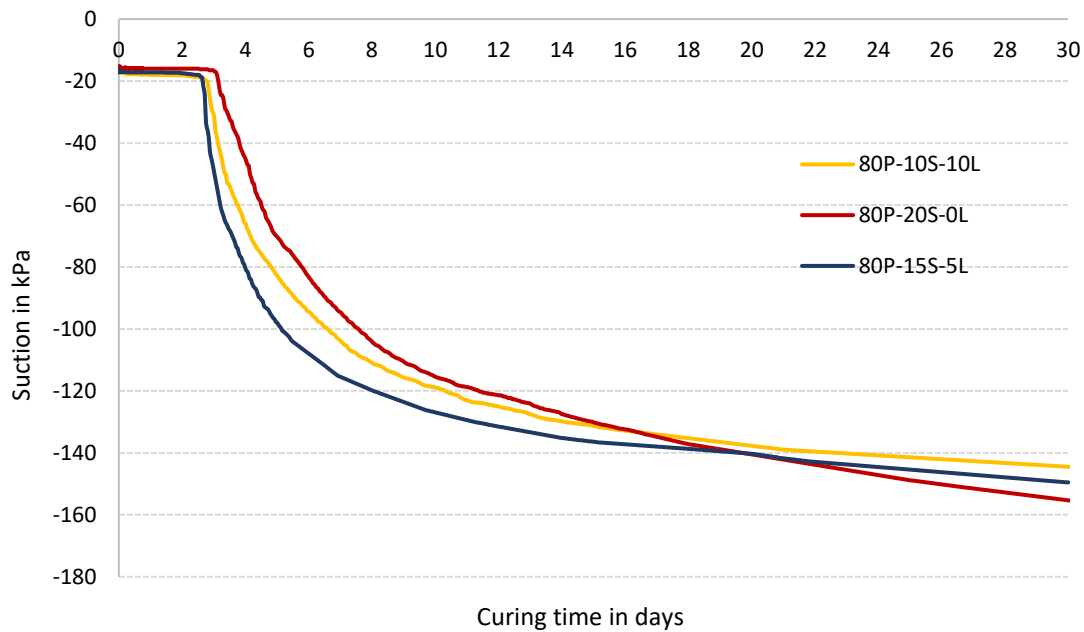


b) Group B CPB samples (binder with 80% Portland cement)

Figure 5.4 Influence of increasing dosage of LS on mechanical strength changes of CPB samples: (a) Group A, and (b) Group B.



a) Group A CPB samples (binder with 50% Portland cement)



b) Group B CPB samples (binder with 80% Portland cement)

Figure 5.5 Suction development in ternary samples with incremental LS percentages by weight (0 to 10%)
a) Group A b) Group B.

5.4 Implications for Geotechnical Design of CPB Structures

The implications of the obtained results for the geotechnical design of CPB structures are discussed and summarized in the following sections.

5.4.1 Mechanical stability

Mechanical stability is one of the most important CPB design criteria (Fall et al., 2007; Ghirian & Fall, 2017), with UCS being the most frequently used to evaluate the strength of CPB mixes. Furthermore, the shear strength properties or parameters, stress-strain behavior, and modulus of elasticity are all related to the mechanical properties of CPB. Mechanical performance in CPB technology is critical for providing ground support that would reduce mining-induced geotechnical issues, such as ground subsidence and mine stope instability, and for the safety of mine workers and the surrounding area. Therefore, the mechanical stability or strength of a CPB structure must be maintained to high standards to achieve the desired mechanical performance of CPB.

It is therefore critical to design backfill those results in enhanced mechanical stability and performance of CPB structure, and knowledge and understanding of the mechanical properties are required. Furthermore, for early age strength assessment, the effect of various parameters like the type of binders used, the impact

of their proportion on the rate of hydration, and suction development due to self-desiccation must be considered.

The strength requirements (UCS) for CPB may vary depending upon its function; for example, CPB used for void filling requires a very low compressive strength of 150–300 kPa, while free-standing or roof support backfill requires a high compressive strength in the range of 1 and 4 MPa (Fall et al. 2005).

Numerous studies have been conducted on the compressive strength to evaluate the mechanical stability in a more timely and cost-effective manner. For instance, Mitchell (1983) calculated the critical UCS for a freestanding CPB structure as $UCS_c = \gamma H(1+H/L)$ where H is the height of the stope, L is the width of the stope, and γ is the unit weight of the fill, as shown in Figure 5.4.

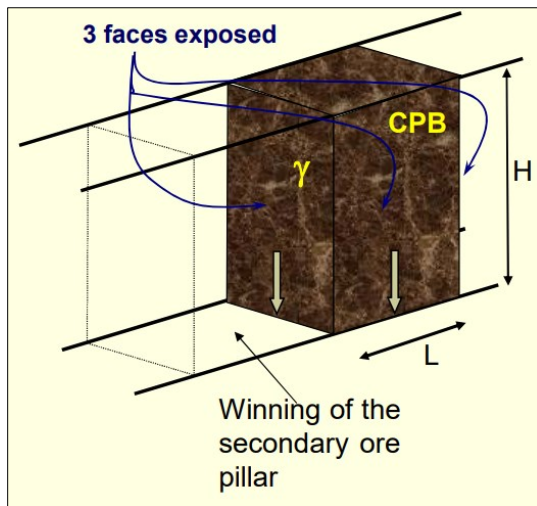


Figure 5.6 Critical UCS of CPB with respect to width L and height H of the stope (Fall, 2021)

In this study, the use of higher proportions of slag reduces the rate of hydration and suction development due to its slow reactivity. As the changes in suction of the backfill is directly related to the changes in strength, the addition of slag causes a reduction in strength at the early ages. However, with the addition of 5 wt. % of LS, the rate of hydration of the mix is enhanced owing to the nucleation effect, and finer particles of LS compared to cement and slag. Furthermore, some strength reduction occurs up to 10 wt.% limestone addition. This is very important to note for CPB, which has a high strength requirement. Indeed, in the CPB samples with higher slag percentages (50 wt.%), increased suction is reported after 21 days and higher strength is gained at 28 days, followed by 60 and 90 days, compared to the CPB samples with 20 wt.% of slag. However, the addition of limestone up to 5 wt.% causes a mere 5% reduction in strength compared to the samples with a binary binder. An increase in limestone dosages and a continued decrease in strength are observed. Hence, as demonstrated from the results of this study, where different combinations of binary and ternary binders are used, it is possible to find and use optimum percentages of slag and limestone as

substitutions for cement, which may help to prepare binder for formulating CPB with the required mechanical strength criteria. Suction has a significant impact on the mechanical and shear strengths during the early ages (both hours and days) of curing.

5.4.2 Barricade stability

Barricades are constructed to prevent failure caused by high pressure generated by saturated fill mass in order to retain the CPB during open stope filling (excess pore water pressure) (Koupouli et al., 2016). The provision of a safe and stable barricade is critical in underground stopes because the risks associated with barricade failure which causes “mudrush” pose a significant concern to the safety of mine workers who are working in the adjacent stopes. In addition, the constructed barricades must be capable of containing the CPB that will be placed into a single stope with a backfill volume of up to 25,000 cubic meters.

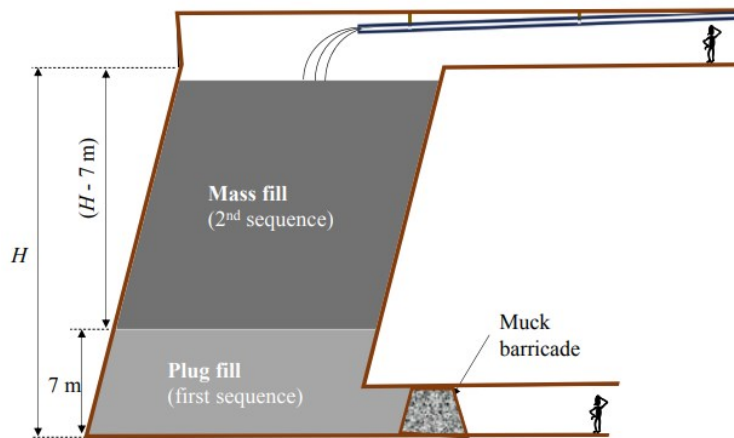


Figure 5.7 Typical underground stope filling sequence (Koupouli et al., 2016)

As shown in Figure 5.5, a barricade that is built in a stope across the undercut to retain the fresh CPB mix supports the mine site after solidification, which is directly related to backfill production rates. The horizontal effective stress and pore water pressure (PWP) in the backfill near the bottom of the stope directly contribute to the stress on the barricade. As a result, pressure is exerted onto the barricade. However, the pressure exerted is a very complex process which is influenced by several other field conditions, such as arching effects, stope geometry, physical properties of tailings, overburden pressure, and so on and so forth, all of which should be considered for each mine site. As a result, understanding how to limit stress is essential for designing safe yet cost-effective barricades and ensuring structural integrity. Previous research has found that temperature, PWP, suction, and effective stress all have a direct impact on backfill stress. Furthermore, the filling rate and sequence, as well as the binder content and type, affect cement hydration reactions, which in turn affect the suction development by reducing the PWP that acts on the barricades.

The current study has found that a higher dosage of slag as a partial substitute for cement at the early ages reduces the cement hydration process in binary binders, thus there is less water consumption which causes slower self desiccation and thereby reducing the pore water dissipation. However, substituting up to 10 % limestone for slag accelerates the hydration reaction, produces more cement hydration products, and reduces the water content of the CPB material (and thus reduces the PWP and total stress). This significantly raises the hydration temperature, speeds up the hydration process, and reduces the PWP and total stress applied to the barricade. The findings of this study can be used for backfill designs for stable barricades and thus a safe work environment, which can be achieved by using optimised percentages of cement, slag, and limestone.

5.4.3 Environmental performance and sustainability

The safe and secure management of tailings is required within the context of stringent modern environmental and regulatory standards; hence a safe environmental performance in the underground voids is one of the important characteristics that speaks to the success of tailings backfill design and operation processes. Backfill susceptibility to AMD is a critical issue that must be addressed for long-term and efficient performances. The fluid transportability (or permeability) of hardened CPB directly influences the environmental performance of mine backfilling. When water and oxygen enter the CPB matrix, reactivity or oxidation occurs, which results in the oxidation of the sulphate minerals in the tailings, and results in the generation of AMD. CPB technology has a number of environmental benefits, including improved water management, the elimination of the need for complex retaining impoundments, a relatively small footprint of the tailings disposal area, and the ability to use progressive reclamation (De Souza et al., 2003; Yilmaz et al., 2015). In the later stages, costly environmental problems and long-term liabilities associated with AMD treatment from surface ponds can be avoided, as reducing the amount of reactive tailings reduces both their environmental impact and future capital expenses. Due to its low permeability, CPB acts as a barrier to prevent underground water seepage. It also inhibits oxygen diffusion, which prevents AMD. Mechanical strength and hydraulic conductivity are dependent on each another. It is observed that pore refinement occurs with the commencement of the hydration process. As hydration proceeds with curing time, the pore size becomes finer, which reduces the pore openings and permeability. Furthermore, the use of slag reduces the larger pores and increases the finer pores, especially at the later ages. The addition of finer limestone also refines the pore structure owing to the filler effect, which reduces the permeability and generation of leachate, thereby reducing potential impacts on the receiving water bodies (Verburg & Golder Associates, 2001). In addition, the disposal of slag, which is an industrial by-product, may pose a threat to the ecosystem if stockpiled on the surface (Benzaazoua et al., 2004).

Furthermore, in the use of cement as a primary binder in CPB, the production of clinker is an energy-intensive process, and clinker is formed at 1450°C. Cement production accounts for about 5% of the total CO₂ emitted and is associated with global warming. Hence, reducing the use of cement as a binder can help to reduce the carbon footprint. In this study, slag is used as a partial substitute for cement, and limestone as a partial substitute for slag. The use of slag as a secondary constituent in cement or concrete production decreases the overall carbon footprint of cementitious materials. Due to the large amount of slag used in cementitious materials, the carbon footprint of cementitious materials can be reduced to less than 30% of that of cement (CEM I), starting with 913 kg/tonne PCI, 75 kg/tonne limestone, and 67 kg/tonne slag, as mentioned in the UK technical datasheet (Technical datasheet 2014). Moreover, limestone requires grinding, which is less energy-consuming compared to cement and slag, considering its soft form. Furthermore, limestone produces the lowest transport-related CO₂ emissions because the raw material source is the same as the source for the clinker, thus making it a more energy-efficient material.

The findings of this study suggest that when used effectively, slag and limestone not only reduce the cost of CPB but also have the potential to reduce CO₂ emissions associated with the production of PC when used as a substitute in clinker. Therefore, this study contributes towards the sustainable growth of the mining industry.

5.4.4 Cost and management

As mentioned earlier, the cost of backfilling could constitute up to 20% of the cost of mining operations, whereas cement can account for up to 75-80% of the cost of backfilling operations (Grice, 1998). Additionally, a paste backfill mix with a 1 wt.% binder typically costs CAN\$1/tonne (Naylor et al., 1997). According to De Souza et al. (2003), the cost of a paste backfill mix that contains 3 wt.% of cement accounts for 42% of the overall cost of the paste backfilling operation. To reduce costs, mining operations must be productive. Mine production can be optimized by constructing a strong barricade, using a higher backfill density, accelerating the stope filling cycle time, and utilising improved paste backfill delivery and positioning procedures. Additionally, costs can be reduced by ensuring that the tailings and cement are appropriately integrated, choosing a more effective binder type, binder content, and dosage for an equivalent or higher strength, and avoiding more conservative backfill designs. In conclusion, given the facts and figures presented, it is critical to control the cost of mining operations through two key criteria: shorter mining cycles and optimising binder consumption to improve the overall productivity of mining operations, as discussed in detail below.

5.4.4.1 Mining cycle

As mentioned earlier, the mining cycle time can influence the cost of mining operations. Hence, reducing the mining cycle time is a very important aspect towards cost saving. A shorter cycle time enables the opening of barricades for mining in adjacent stopes and the following sequence of mining operations. A shorter mining cycle can be obtained through a continuous flow of CPB materials into the stope with efficient transport processes, i.e., those that do not cause any clogging in the pipes, with less friction to avoid damage to the pipes. This can save time that might be otherwise spent to unclog or undo the damage caused. From the results of this study, it can be observed that replacing cement with 50 wt.% of slag increases the yield stress; however, replacing slag with LS helps to reduce the yield stress, which will improve the flowability of the mix and ease the transport process. Moreover, the setting time of the mixes is not affected by the addition of up to 10 wt.% of LS in a ternary binder. The results of this study can be used to design and manage the transport of CPB mixes efficiently. Furthermore, sufficient early age strength gain in barricades is crucial for their opening to commence ore extraction operations in adjacent stopes. Hence knowledge of strength gain and suction development may help in designing a productive mine cycle. As demonstrated in this study, a larger percentage of slag use, or 50 wt.% slag causes a reduction in early age strength as opposed to samples with 20 wt.% slag. Hence selection of the optimum amount of slag as a substitute may prevent the reduction in strength at the early ages, and then satisfying the strength criteria at the later ages. Furthermore, the addition of up to 5 wt.% LS enhances the cement hydration, which increases the water consumption and leads to an acceleration in pore water dissipation and suction development. Overall enhancement in early age strength subsequently enables the desired strength gain in a shorter period of time, with a shorter mining cycle and savings in cost.

5.4.4.2 Binder consumption

Despite the fact that the binder used in CPB preparation typically costs around 75- 80% of the backfilling process, it is a common practice in many underground mines to use a high binder content (7 wt.% on average) within the fill to accelerate its strength development, particularly when placing the first layer of 2-3 m in thickness (known as the plug fill), and in the residual fill, not more than 5 wt.%. As a result, optimising binder consumption can result in significant cost savings. According to the findings of this study, the use of slag in optimum percentages to substitute for cement will reduce the cost of an otherwise expensive binder, i.e., cement. The site-specific cost estimates for cemented paste systems are prepared based on the unit price of the binder (s). The reported unit prices of PC and slag are CAN\$104/tonne, and \$90/tonne, respectively (Tyson, 2012). Furthermore, the UCS of CPB cured in the presence of 10 wt.% of LS improves the strength and suction of the mix on the first day. Further enhancements in strength are obtained with the addition of 5 wt.% of LS until the 7th day. Hence, further reduction in cement consumption can be obtained.

Therefore, using a ternary binder with slag and LS as a partial substitute for cement can help to reduce the cost of mining operations since the cost of the binder has been reduced.

5.5 References

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6. Chapter 6. Conclusions and Recommendations

This section presents a conclusion of the results along with recommendations for future studies. The main objective of this research is to study and understand the effect of slag in a binary binder - PCI/Slag (80/20 and 50/50), as well as the effectiveness of using limestone in a ternary binder (PCI/Slag/LS) as a substitute for slag with increasing dosages (5, 10, 20 wt.% of the total binder), on the changes of the rheological properties and mechanical behavior of the CPB samples. To investigate the above-mentioned parameters, a test program is carried out in two phases.

In the first phase, several CPB specimens are cast and cured for 0, 0.25, 1, 2, and 4 hours with a binary binder (PCI/Slag) and increasing dosages of LS in place of slag, and their rheological properties are evaluated by examining the yield stress and testing the viscosity, followed by examining the setting time. In the second phase, UCS tests are performed to determine strength gain and suction development over a curing time of up to 90 days to investigate changes in the mechanical behaviour. The microstructural properties are determined using MIP and TG/DTG analyses, and the results are supported by the findings from the EC, temperature, and suction monitoring experiments. The following are the key findings from Phases I and II.

Phase I

- i. Irrespective of the type of binder and their proportion, an increase in yield stress and viscosity can be observed with an increase in curing time. This is due to the increase in hydration products, with the progression of cement hydration and self-desiccation with time.
- ii. CPB specimens with 50/50 PCI/Slag gain a higher yield stress with time as opposed to 80/20 PCI/Slag. This phenomenon is related to the fines of slag, which cause interparticle friction and the formation of a denser matrix as a result of the filler effect.
- iii. The addition of LS with incremental dosages (5-20 wt.%) in the form of a ternary binder impacts the rheology (flowability) by decreasing the yield stress and increasing the viscosity of fresh CPB. Interestingly, the effect of LS complements that of slag on the rheology of the mix.
- iv. This effect of LS on the rheology of CPB - a decrease in yield stress, is mainly attributed to the physical factors or effects of LS, i.e., the finer limestone fills the void spaces and causes water to be available in more pores, which provides additional lubrication to the mix, and additional repulsive forces. However, the viscosity increases due to the nucleation effect and the higher water demand due to the finer limestone powder with a larger surface area.
- v. The setting time (initial and final) of the samples with 80/20 PCI/Slag is found to be shorter than that with 50/50 PCI/Slag because more hydration products (C-S-H, ettringite, AFm) form in the sample with a higher cement content at a higher rate, thus resulting in a stronger matrix of the mix.

- vi. A delay in the setting time of the CPB samples when LS is added in increasing dosages as a substitution for slag is observed and consistent with the DTG/TG results. This indicates the influence of LS on cement hydration through a nucleation effect. However, not all of the samples produced more hydration products.

Phase II

- i) The substitution of cement with slag affects the early-age hydration kinetics of the CPB mix by subsequently reducing the rate of the hydration reaction, mechanical strength and suction development at an early age. This strength reduction is attributed to the dilution of potential cementing material and slow hydration of slag. At a later age, however, there is the latent hydraulicity of slag, secondary C-S-H is produced, and samples with a higher slag proportion in the binary binder, (PCI/Slag) i.e., 50/50 gain higher strength and suction than the sample with 80/20 PCI/Slag.
- ii) In comparison to binary binders, ternary binders with moderate substitutions of slag with LS up to 10 wt.% of the total binder improve the reaction rate, early-age mechanical performance, and suction of the mixes. This is the result of a combination of the physical (filler, particle packing) and chemical (nucleation and chemical interaction) effects of limestone.
- iii) The MIP results show continuous and progressive pore refinement in all of the samples as the curing time increases. Pore refinement is slightly higher in the sample with higher cement content at the early ages, up to 7 days, in binary binders. However, it is not proportional to cementing material because finer slag also induces a filler effect, filling the pores, and resulting in a larger number of fine pores at the later ages. In contrast, LS addition induces pore refinement in a ternary binder at the early stages but later reduces porosity due the loss of slag, and eventually the secondary C-S-H produced by slag.
- iv) The overall substitution of slag with 10 wt.% of LS in the total binder is noted as the optimum limestone content which provides the most ideal combination in the ternary binder to obtain similar or improved properties as opposed to the binary samples in the CPB mixes.

Although it is considered that this work brings new insights and understanding of the fresh properties and mechanical strength gain behaviours of these mixtures, further investigation is still required. Detailed chemical and microstructural studies are recommended to gain a deeper insight into the impact of LS on the microstructure and chemical composition of CPB systems with a ternary binder (PC, slag, and LS).

It is also recommended that the chemical effect of limestone is investigated, i.e., phase assemblage and LS induced products formed during the hydration reaction, as well as their quantification – the actual particle packing density. In addition to this, it is recommended that the factors that affect the physical effect of LS on strength changes can be examined by using coarser LS. Waste limestone can be incorporated to correlate with actual LS and used to compare its effect on the mechanical behavior with pure CaCO_3 .

Most importantly, it will be useful to develop a modeling tool to optimize the dosage of slag and LS, thus minimising the shortcomings of each component, and providing the optimal binder that can improve the rheological and mechanical properties of CPB mix.