

Temperature Dependency of the Rheological Properties and Strength of Cemented Paste Backfill that Contains Sodium Silicate

Ghada Abdulbaqi Ali

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Dr. Mamadou Fall

Department of Civil Engineering
Faculty of Engineering
University of Ottawa

ABSTRACT

Over the past decades, cemented paste backfill (CPB) has become a common, environmentally friendly method of managing mine wastes (such as tailings). This technology allows up to 60% of the total amount of tailings to be reused and filled in the mine stopes after converting them into cemented material. Beside reducing the environmental risks associated with the traditional disposal of these materials, turning them into cemented material and placing them in the underground mine stopes can also provide secondary support for these stopes in addition to minimizing the risk of ground subsidence in the mine area. CPB is an engineered mixture of tailings, water, and hydraulic binder (such as cement, blast furnace slag, and fly ash) that is mixed in the paste plant and delivered into the mine stopes through a gravity or pumping based transportation system. During the transportation of CPB through the delivery system pipelines, the flowability of CPB depends on the rheology of the transported CPB, which is affected by different factors, such as the transportation time, temperature variation, binder type, and chemical composition of these mixtures. In addition, the performance of CPB, after placing the CPB mixture into the mine stopes, is mainly dependent on the role of the hydraulic binder, as it increases the mechanical strength of the mixture through the process of cement hydration. The mechanical strength is also influenced by different factors, such as time progress, temperature variation, and presence of chemical additives. It has previously been found that fresh CPB transported and/or placed in the mine stopes can be susceptible to temperature variation of different sources, such as the climatic effects, heat generated from the surrounding rocks, and heat generated during the process of cement hydration. Unsuitable flowability of CPB through the delivery system might lead to significant financial losses due to clogging of pipelines with unexpected hardening of CPB during transportation, which will cause delay in work and possible damages to the pipelines. Also, failure of CPB structure in the mine stopes due to inappropriate mechanical strength may cause casualties to the mine workers as well as significant environmental and economic damages. Many researchers studied the rheological properties and/or strength development of CPB under the individual effect of any of the aforementioned factors. Additionally, many researchers have evaluated the coupled effect of some of these factors on the rheology and mechanical strength of CPB material. Hitherto, there are currently no studies that addressed the combined effect of all these conditions on the rheological properties and strength development of CPB. At the first stage of this M.A.Sc. study, a series of experimental tests was conducted on fresh CPB in order to determine the combined effect of time, temperature, binder content, and chemical additives on the rheological properties of CPB. These experiments include rheological properties test (yield stress and viscosity), microstructural analysis (thermal analysis and XRD), chemical analysis (pH and Zeta potential), and monitoring tests (electrical conductivity), which were conducted on 125 CPB samples that were mixed and prepared at different temperatures (2°C, 20°C, 35°C) and cured for different curing time (0 hrs., 0.25 hrs., 1 hr., 2hrs, and 4 hrs.). These samples were prepared with different blends of hydraulic binders (PCI, PCI/Slag, and PCI/FA) and contained different dosages of sodium silicate (0%, 0.1%, 0.3%, and 0.5%). The results obtained show that rheology of CPB

increases with the progress of curing time. It also increases with the increase in the initial (mixing and curing) temperature and content of sodium silicate. It was also found that the partial usage of slag and FA reduces the rheological properties. However, CPBs containing PCI/FA as binder have lower rheological properties, and thus better flowability, than those that contain PCI/Slag as binder. At the second stage of this M.A.Sc. study, in order to understand the combined effect of time, temperature and sodium silicate content on the strength development of slag-CPB, unconfined compression (UCS) test, microstructural analysis (thermal analysis and MIP), and monitoring tests (electrical conductivity, suction, and volumetric water content) were conducted on 72 CPB samples that were prepared with PCI-Slag as a binder, cured for different times (1 day, 3 days, 7 days, and 28 days) under different curing temperatures of (2°C, 20°C, 35°C), and contained different dosages of sodium silicate (0%, 0.3% and 0.5%). The results obtained at this stage showed that the strength development of slag-CPB increases with the progress of curing time and temperature. It also increases with the increase in the sodium silicate content. Also, the combined effect of high temperature, high dosage of sodium silicate and longer curing time showed significant enhancement in the mechanical strength of slag-CPB. The findings of this M.A.Sc. research will contribute to cost effective, efficient, and safer design of CPB structures in the mine areas. It will also help in minimizing financial loss associated with unsuitable flowability of CPB transported in the CPB delivery system besides reducing the risks of human loss, and the environmental and economic damages associated with the failure of CPB structures.

DEDICATION

To the souls of my father “**Abdulbaqi Ali**” and my brother “**Hussain Ali**”.

To my mother “**Zahra Ghadeer**”

To my husband, **Imad**, my children, **Hazim, Ahmed, and Mayar**.

To my treasured family.

Thank you all for your love, support, and patience during my study.

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LIST OF SYMBOLS AND ABBREVIATIONS

9MT	: Nine Mine Tailings
AMD	: Acid Mine Drainage
CPB	: Cemented Paste Backfill
DTG	: Differential Thermogravimetry
EC	: Electrical Conductivity
FA	: Fly Ash
hrs	: Hours
min	: Minutes
MIP	: Mercury Intrusion Porosimetry
OPC	: Ordinary Portland Cement
PC	: Portland Cement
PCI	: Portland Cement – type I
sec	: Seconds
Slag	: Blast Furnace Slag
SS	: Sodium Silicate
ST	: Silica Tailings
Temp.	: Temperature
TG	: Thermal Gravimetry
UCS	: Unconfined Compression Strength
UCS _c	: Critical Unconfined Compression Strength
VWC	: Volumetric Water Content
w/c	: Water-to-Cement Ratio
wt%	: percentage of the total dry weight
XRD	: X-ray Diffraction
θ	: Diffraction Angle

CHAPTER 1
General Introduction

1.1 Background and Problem Statement

Mining operations were established in Canada in 1577. Since then, mining industry has become a lead in the economic growth in Canada. For instance, the total Canada-wide mineral production in 2019 was more than 48 billion CAD (Chang 2016, Statista 2020). Moreover, the Canadian mining industry offered around 626,000 jobs in 2018, with an estimation of around 80,000 additional jobs to be offered over the next decade, i.e. up to 2030 (Marshall 2020).

Ore extraction during mining operations create large underground openings (stopes) that might cause several geotechnical engineering issues endangering the safety of the mine workers and the nearby public congregation. These issues include ground subsidence and the instability of these openings (Alainachi and Fall 2019). In addition, mining operations annually generate large amount of mining wastes. The worldwide generation of mine solid waste (e.g. tailings) was estimated to be over 100 billion tons per year (Tayebi-Khorami et al. 2019). Poor disposal of these tailings may harm the environment, as they can generate acid mine drainage (AMD). The AMD has always been a main problem associated with mining activities, as it causes contamination of underground and surface water bodies if deported from mine disposal site (Heikkinen et al. 2009, Abdul-Hussain 2011).

An environmentally friendly underground disposal method of mine waste has been extensively used around the world in order to lessen the engineering and environmental problems associated with mining operations. This technology is called the cemented paste backfill (CPB), which is summarized by the reuse of large quantities of the tailings and pumping them back to the mine stope after mixing them with water and binder material (Belem and Benzaazoua 2004, Bull 2019). CPB has been found to be beneficial in increasing the stability of the underground mine in long term (Alainachi and Fall 2019, Ercikdi et al. 2017)

CPB is often prepared and mixed in the surface paste plants, and hydraulically pumped and/or transported by gravity to the underground mine stopes. However, if the transported CPB material does not have suitable flowability, it might cause significant financial losses and production interruption due to the possible flow delay and possible clogging-induced pipelines' damages (Li and Fall 2016, Haruna and Fall 2020). Also, the mechanical strength performance of CPB is significantly affected when exposed to variation of static loading, dynamic loading, temperature variation, and potential chemical interaction of the chemical elements within the CPB components. Inadequate mechanical performance of CPB causes failure of CPB structure and thus endangers the safety of the mine workers and causes negative environmental impacts and economic damages (Fall and Pokharel 2010, Cao et al. 2018, Xue et al. 2018, Alainachi 2020)

Considerable amount of research, (Simon and Grabinsky 2013, Cui and Fall 2016, Haiqiang et al. 2016, Jiang and Fall 2017, Haruna and Fall 2019), has been undertaken to evaluate the flowability and/or the rheological properties of CPB. Other studies (e.g. Kesimal et al. 2005, Nasir and Fall 2010, Li and Aubertin 2012, Ghirian and Fall 2013, Ghirian and Fall 2014, Cui and Fall 2016, Ghirian and Fall 2016, Cihangir et al. 2018, Fang and Fall 2019, Haruna and Fall 2020, Xu et al. 2020) have studied the mechanical behavior or stability of CPB exposed to different

conditions, such as temperature variation, curing time, different CPB mix design, and availability of different chemical components within the CPB. Yet, there is paucity of studies that addressed the combined effect of these conditions on the flowability and/or the mechanical strength of CPB at early age. Thus, the need of more studies to understand the combined effect of these conditions on the flowability and strength of early aged CPB has become critically important.

The engineering properties (such as the mechanical, thermal, and hydraulic behavior etc.) of cementitious material, including CPB, was found to be affected by the progress of cement hydration (i.e. progress of curing time) (Bullard et al. 2011, Scrivener et al. 2015, Aldhafeeri and Fall 2016) and the effect of partial usage of different hydraulic binders (such as slag and fly ash) (Maltais and Marchand 1997, Elakneswaran et al. 2009, Kwan and Chen 2013, Kondraivendhan and Bhattacharjee 2015, Le et al. 2019). Moreover, it was previously observed that the temperature of fresh CPB placed in mine stopes is affected by several heat sources, which might affect the rheological, mechanical, thermal, and hydraulic properties of CPB (Maltais and Marchand 1997, Fall et al. 2010, Nasir and Fall 2010, Aldhafeeri et al. 2016, Cui and Fall 2016, Wang et al. 2016, Fang and Fall 2018, Wang et al. 2018, Haruna and Fall 2019, Liu et al. 2019, Cheng et al. 2020, Haruna and Fall 2020). On the other hand, previous studies have revealed that adding some chemical additives (such as sodium silicate) to the CPB may affect its flow behavior and mechanical strength. It is expected that the flowability of cementitious material drops when it contains sodium silicate, while the mechanical behavior is expected to enhance (Brough and Atkinson 2002, Živica 2007, Abdul-Hussain and Fall 2012, Kermani et al. 2015, Wang et al. 2016, Veenstra et al. 2017, Cihangir et al. 2018, Jiang et al. 2019).

Accordingly, the problem statement of this research, as shown in Figure 1.1, can be summarized as the need of comprehensive study of the rheological properties and mechanical strength of CPB at the early ages by assessing the combined effect of time, temperature, binder type, and chemical additives on these properties.

1.2 Objectives

The primary goal of this M.A.Sc. study is to investigate the temperature dependency of the rheological properties and strength development of CPB under the combined effect of time progress, binder type, and the content of sodium silicate as a chemical additive. Silica tailings are used to determine how the initial (mixing and curing) temperature affects the rheological properties and the mechanical strength of samples prepared with different types of hydraulic binders and different dosages of sodium silicate. The experiment program of this research includes a combination of different tests that are conducted at different temperature conditions. These tests include yield stress and viscosity tests, unconfined compression tests (UCS), microstructure analysis, chemical tests, and monitoring tests. These experiments aim to investigate:

1. The influence of initial (mixing and curing) temperature and the progress of cement hydration process (progress of curing time) on the rheological properties of CPB that contains sodium silicate.

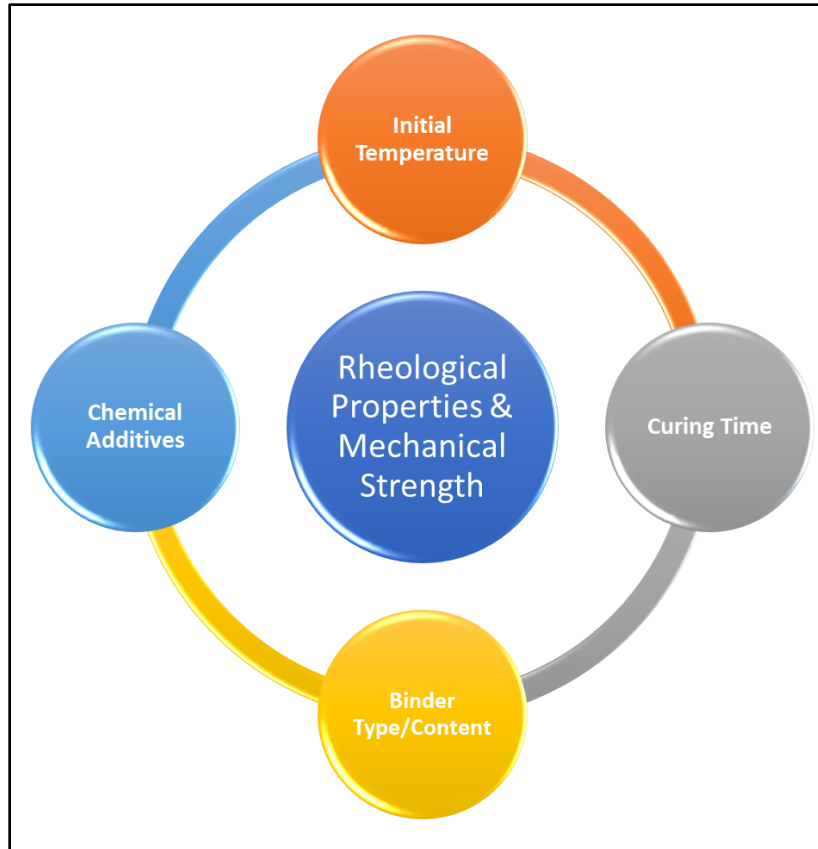


Figure 1.1 Combined conditions studied in this research

2. Whether the type of hydraulic binders of the CPB mixture changes the rheological properties of CPB that contains sodium silicate.
3. The effect of different dosages of sodium silicate on the rheological properties of CPB samples.
4. The influence of curing temperature on the mechanical strength of slag-CPB material that contains sodium silicate.
5. The change in mechanical strength of slag-CPB material with the variation of sodium silicate content.

1.3 Research Approaches and Methods

1.3.1 Research Approaches

The research approaches adopted in this study have been illustrated in the schematic flowchart in Figure 1.2. To better understand the temperature and time effect on the rheological properties and the development of the mechanical strength of CPB that is made with different binders and contains different dosages of sodium silicate, this study has been conducted in three main phases:

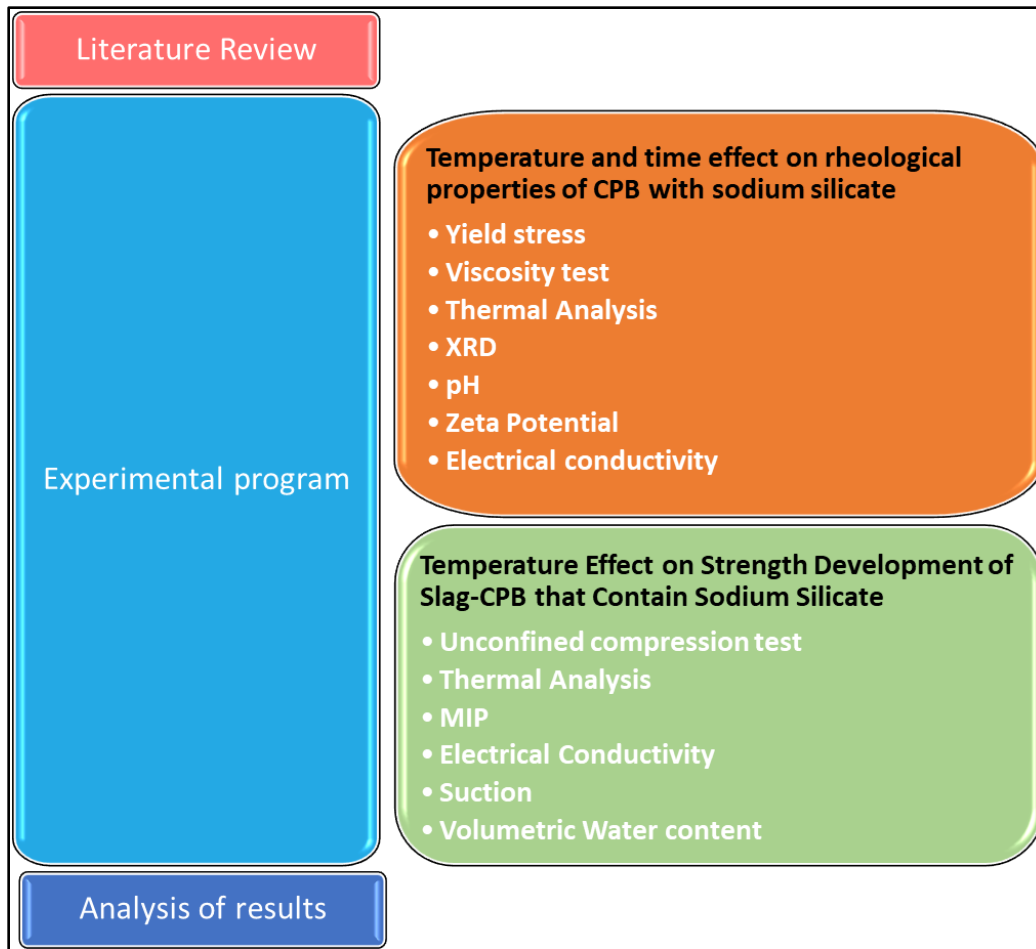


Figure 1.2 Flowchart of research approach

Phase 1 - A comprehensive literature review of (i) the material to be tested (CPB), (ii) different hydraulic binder types, (iii) sodium silicate as chemical additive, (iv) the rheology of CPB, and (v) the compressive strength (UCS).

Phase 2 - Conducting the experimental program that will be divided into two sub-phases to better understand each of the studied properties. These sub-phases are:

1. Conducting rheological (yield stress and viscosity) tests, microstructure (Thermal and XRD) analysis, chemical (pH and zeta potential) tests, and monitoring (electrical conductivity) test on CPB samples that consist of different binders (Portland cement, Slag, and Fly Ash) blends, contain different amount of sodium silicate (0, 0.1%, 0.3%, and 0.5%), and were prepared at different temperatures (2°C, 20°C, and 35°C) and cured to different maturity ages (curing time) of 0, 0.25, 1, 2, and 4 hours.
2. Conducting unconfined compressive strength (UCS) test, microstructure (Thermal and MIP) analysis, and monitoring (suction, volumetric water content and electrical conductivity) tests on CPB samples that consist of 50% of slag and 50% of Portland cement

as a binder, contain different amount of sodium silicate (0, 0.3%, and 0.5%), and cured under different temperatures (2°C, 20°C, and 35°C) to different maturity ages (curing time) of 1, 3, 7, and 28 days.

Phase 3 - Analyzing the experimental results and assessing the rheological properties and strength development of CPB with respect to the tested conditions.

1.4 Thesis Outline

This thesis is organized in the form of technical papers and contains six chapters (Figure 1.3):

Chapter 1: includes a general introduction to the study, the problem statements, objectives, and research approaches and methods adopted in this study.

Chapter 2: reviews technical background and literature review on Cemented Paste Backfill (CPB), hydraulic binders, sodium silicate, rheology, and compressive strength. This information is necessary to facilitate the understanding of the technical issues addressed in this thesis. Moreover, previous studies of rheological properties and strength development of cemented paste backfill have been reviewed and discussed.

Chapter 3: discusses the combined effect of time and temperature on the rheological properties of cemented tailings backfill that contains sodium silicate (Technical Paper I).

Chapter 4: describes the temperature effect on the strength development of slag-cemented paste backfill that contains sodium silicate (Technical Paper II).

Chapter 5: synthesizes the overall thesis results.

Chapter 6: presents the conclusions and recommendations of this thesis.

It should be emphasized that since a paper-based thesis format has been adopted, some of the contents in the papers may be repeated as each paper is independently written and crafted according to the manuscript instructions for the specified publication.

<p>› Chapter 1</p> <ul style="list-style-type: none"> • General Introduction
<p>› Chapter 2</p> <ul style="list-style-type: none"> • Technical Background and Literature Review
<p>› Chapter 3</p> <ul style="list-style-type: none"> • Paper 1 <ul style="list-style-type: none"> • Time- and temperature-dependence of rheological properties of cemented tailings backfill with sodium silicate
<p>› Chapter 4</p> <ul style="list-style-type: none"> • Paper 2 <ul style="list-style-type: none"> • Temperature Dependency of the Strength Development of Slag-Cemented Paste Backfill that Contain Sodium Silicate
<p>› Chapter 5</p> <ul style="list-style-type: none"> • Synthesis of Results and Discussion
<p>› Chapter 6</p> <ul style="list-style-type: none"> • Conclusions and Recommendations

Figure 1.3 Organization of the thesis

1.5 References

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CHAPTER 2
Technical Background and Literature Review

2.1 Introduction

In order to better evaluate the effect of temperature on the rheological properties and strength development of cemented paste backfill (CPB), this chapter provides a review of the fundamental technical background information on CPB. Section 2.2 provides background information on CPB technology, including the mix design and preparation, delivery methods, backfilling strategy, and the design of CPB from geotechnical point of view as well as the source of temperature in backfill operations. In section 2.3, the current knowledge on different hydraulic binders (Portland Cement, Slag, and Fly Ash) and some of the factors that affect the hydration process of these binders have been deliberated. After that, the background of sodium silicate and its effect on cementitious material have been elucidated in section 2.4. Afterwards, the background information to understand the theory of rheology, the equipment and testing methods for rheology, and a review of the previous studies on the rheology of CPB have been described in section 2.5. Furthermore, background of compressive strength and a review of the previous studies that used this technique on CPB have been described in sections 2.6.

2.2 Background of Cemented Paste Backfill (CPB)

Underground mining operations produce a huge quantity of mine wastes. Beside not having any benefit to the industry, mishandling of these wastes makes them highly dangerous to the environment as they may generate several hazards, such as acid mine drainage (AMD). Also, mining activities generate large underground voids that may aggravate the risks of ground subsidence (Chang 2016, Cui 2017). Although there are several waste management techniques that are often used, but each type of these “traditional” methods is associated with its own environmental, geotechnical, and/or economic challenges. For instance, surface disposal, such as stacking tailings in a series of dams, is one of the common tailing disposal methods. However, beside the fact that this method requires a large (over ground) area, these dams might fail resulting in multiple environmental and economic damages, as the tailings might rapidly drift for several kilometers (Chang 2016, Bull 2019). Therefore, in order to minimize these challenges, a need for new solution has been felt. One of these solutions is the cemented paste backfill (CPB), which is a modern technology of mine waste management that was developed in the early 1980s in Germany. This technology has been extensively used in several underground mines around the world as it allows big quantities of tailings to be turned into cemented backfill and returned to the mine voids. This method is environmentally friendly as it reduces the risk of generating acid mine drainage (AMD) and/or other toxic materials. Furthermore, from the geotechnical point of view, the use of CPB prevents the collapse and/or roof fall of the mine stope by providing ground support to the walls and pillars. Economically, on the other hand, CPB can be produced and delivered to the mine stope in a short period, which reduces the backfilling process (mining cycle) in a matter of days and consequently increases the mine productivity (Belem and Benzaazoua 2004, Abdul-Hussain 2011, Cui 2017, Aldhafeeri 2018, Alainachi and Fall 2019a).

2.2.1 Mix design of CPB

The identical CPB mixture consists of mine tailings with a solid percentage of 70% - 85%, fresh or mine processed water with a water-to-cement ratio (w/c) that can range between 5.5 and 9.6, and around 2% - 9% (by total weight of solid) of hydraulic binder (Figure 2.1). The content of these ingredients is often selected based on the primary and tertiary requirements of the mine stope as well as the tailings fineness and density which affect CPB transportability (slump) (Alakangas et al. 2013, Yilmaz et al. 2013, Aldhafeeri and Fall 2016, Haiqiang et al. 2016, Koohestani et al. 2018). Ordinary Portland Cement (OPC) is perceived to be the most hydraulic binder used in CPB mixture. However, a partial replacement for the OPC with pozzolanic materials, such as blast furnace slag (Slag) and/or fly ash (FA), has become a common practice in CPB technology in order to reduce the cost due to cement consumption without affecting the desired strength of CPB (Tariq and Yanful 2013, Cui 2017).



Figure 2.1 Components of cemented paste backfill (CPB)

CPB mixture is often prepared in paste backfill plant, which is usually located at the surface of the mine. Produced CPB is then transported through underground distribution system. Based on the amount of energy required to deliver it, CPB is often delivered into the mine stope by pumping, gravity and/or combination of both (Figure 2.2) (Belem and Benzaazoua 2004, Cui 2017). Therefore, the flowability of fresh CPB is a key concern in CPB transportation to the mine stope.

If transported material did not have suitable flowability, pipelines' clogging might occur, which will cause flow delays, temporary disruption of the progress of CPB production, and/or possible replacement of clogged pipelines, resulting in significant financial losses (Wu et al. 2013, Li and Fall 2016, Haruna and Fall 2019). To ensure better flowability of transported fill in the pipelines, it is imperative to study the rheological properties of these fills, such as yield stress and viscosity. To maintain appropriate flowability, the pumping pressure of fresh CPB must be larger than the yield stress of the produced paste. In order to optimize the delivery system, appropriate balance between pumping velocity, the pipe diameters, and relative density of the fill has to be taken into consideration. It is also worth noting that the rheological properties of CPB, and consequently its flowability through the pipelines, are significantly affected by several factors, such as temperature, binder type, the presence of chemical additives, and the elapsed time after mix preparation (Wu et al. 2013, Haiqiang et al. 2016, Cui 2017, Wang et al. 2018, Haruna and Fall 2019, Alainachi 2020). Further details on the rheological properties of CPB will be discussed in section 2.5.

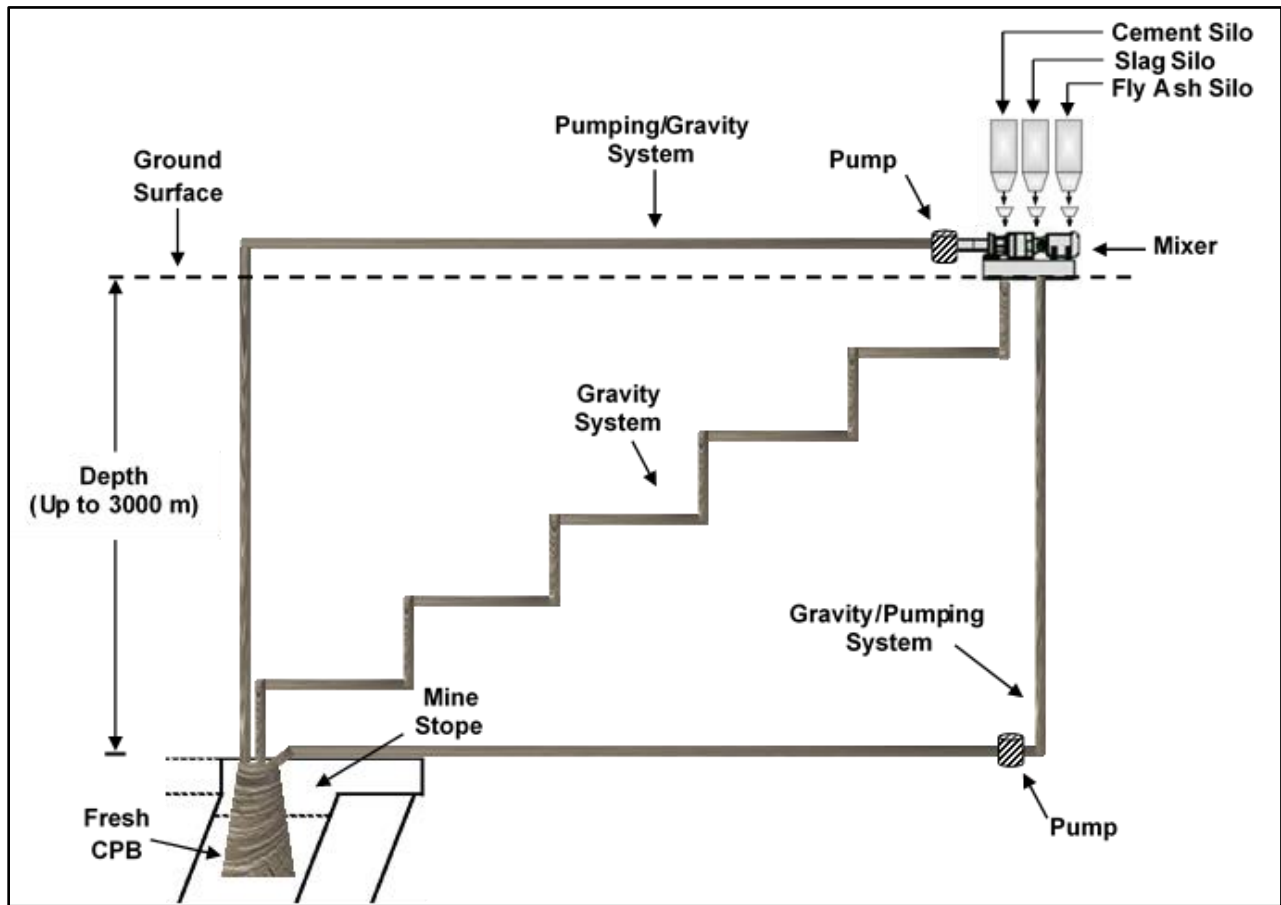


Figure 2.2 Underground delivery systems of CPB

2.2.2 Geotechnical design of CPB

While designing a CPB structure, it is vitally important to take into consideration the economic efficiency of the CPB structure as well as the stability of the structure, and consequently the safety

of the workers in the mine workplace and the surrounding area. Economically, it is important to shorten the completion time of the backfill process in order to reduce the mining cycle time that will consequently boost the mine production. From the safety standpoint, the CPB structures should be designed to have a sufficient strength in order to provide a safe and stable platform for mine workers besides preventing caving of the mine stope and/or subsidence of the ground of the mine and the neighboring area. Accordingly, the strength and stability of the CPB structures play the major role in achieving the best CPB performance with respect to workers' safety and mine productivity, and thus in designing the CPB structures (Belem and Benzaazoua 2004, Chang 2016, Lu 2017). Strength performance of CPB structures is also found to be significantly affected by the temperature, binder type, the post-mix and preparation time, and the presence of chemical additives (Fall et al. 2010, Nasir and Fall 2010, Ghirian and Fall 2013, Chang 2016, Wang et al. 2016, Fang and Fall 2018). Further details on the strength of CPB will be discussed in section 2.6.

2.3 Source of Temperature in Mine Backfill

During its delivery and/or when it is placed in mine stopes, several sources might affect the temperature of CPB in the field (Figure 2.3). These sources might be internal, such as the heat generated during hydration of the hydraulic binders, or external, such as the climatic influence based on the geographic location, temperature variation with stope depth, heat from the interaction between CPB and the rock mass, and heat from paste-pipe friction as along with the temperature variation produced by the mining operations (Fall et al. 2007, Fall et al. 2010, Wu et al. 2013, Aldhafeeri et al. 2016, Wang et al. 2016, Alainachi and Fall 2019b, Bull 2019).

2.3.1 Hydration heat

There is a common consensus that exothermic reaction of the components of cement or any other hydraulic binder with water generates a significant amount of heat. This generated heat is associated with the formation of cement hydration products that are responsible for the hardening of cementitious materials, such as CPB (Fall et al. 2010). Due to the large size of CPB structures, the generated heat does not easily dissipate through these structures. So, there will be a noticeable increase in temperature of the CPB placed in the mine stope through its curing process (Bull 2019). However, the rate of heat evolution or the amount of released heat, in other words, varies with the progress of time and/or the type and content of the hydraulic binder used in the mix (Figure 2.4) (Schindler and Folliard 2003, Bullard et al. 2011, Han et al. 2017).

2.3.2 Climatic influence and geographic location

The temperature of CPB transported through the pipeline and/or placed in the mine stope is significantly affected by the climate fluctuations (e.g. winter/summer) and the geographic location of the mine itself, especially in the extremely hot regions and/or permafrost regions (Wang et al. 2016, Aldhafeeri 2018, Alainachi and Fall 2019b).

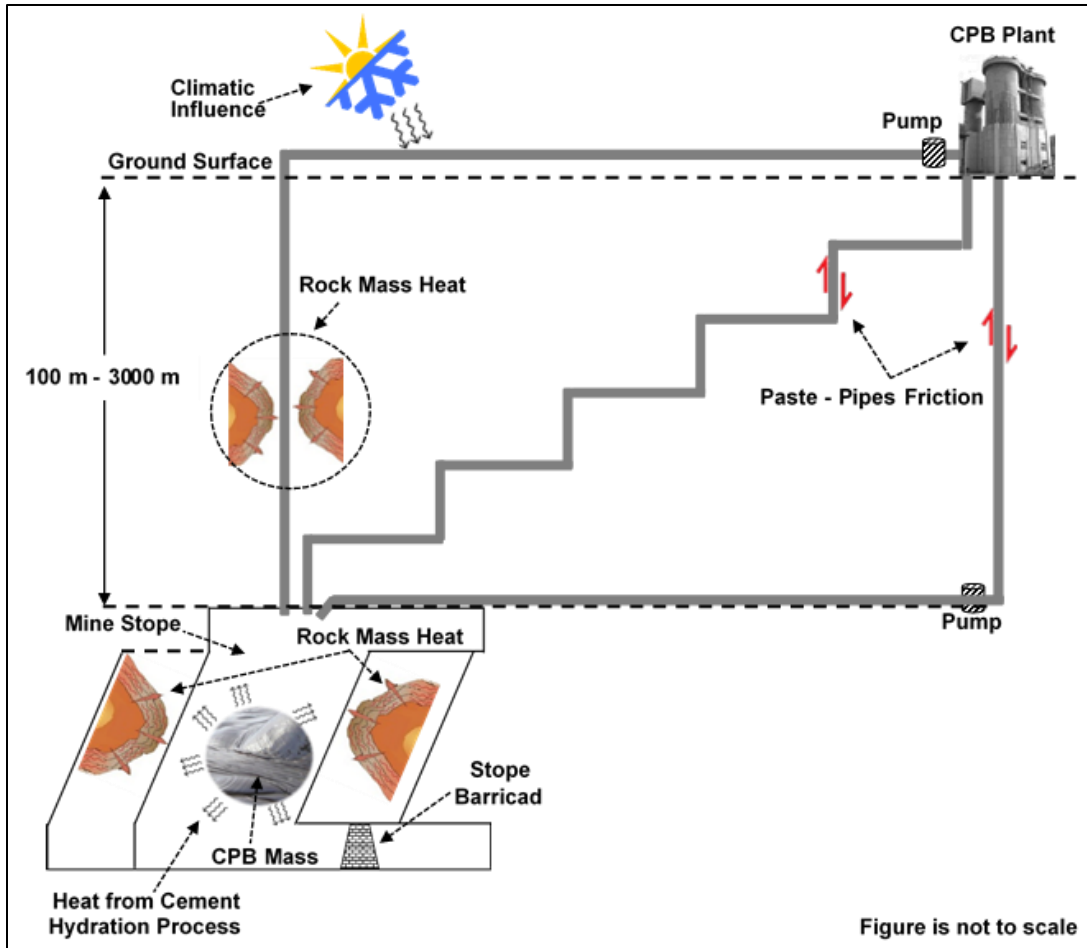


Figure 2.3 Sources of temperature in mine backfill

2.3.3 Geological condition and mine stope depth

Simultaneously with the high demand on the extraction of more ores, there is a gradual reduction of ore available at shallow depths in many underground mines around the world. Hence, underground mine operations are being extensively undertaken at greater depths (Johnson 2015). Generally, the depth increase is naturally associated with increase in heat flux, which is attributed to the geothermal gradient (Wu et al. 2013). As shown in Figure 2.5, in deep mines (more than 2500 m depth), the temperature is expected to exceed 45°C, while it might be less than 20°C at shallow depths (0 m – 500 m) (Fall et al. 2014).

2.3.4 Rock mass heat

The surrounding rock mass is another major heat source for CPB in any deep level mining operation. The thermal interaction between the CPB transported/placed within the mined-out stope and the exposed rock mass will obviously change the temperature of the CPB (Wu et al. 2013).

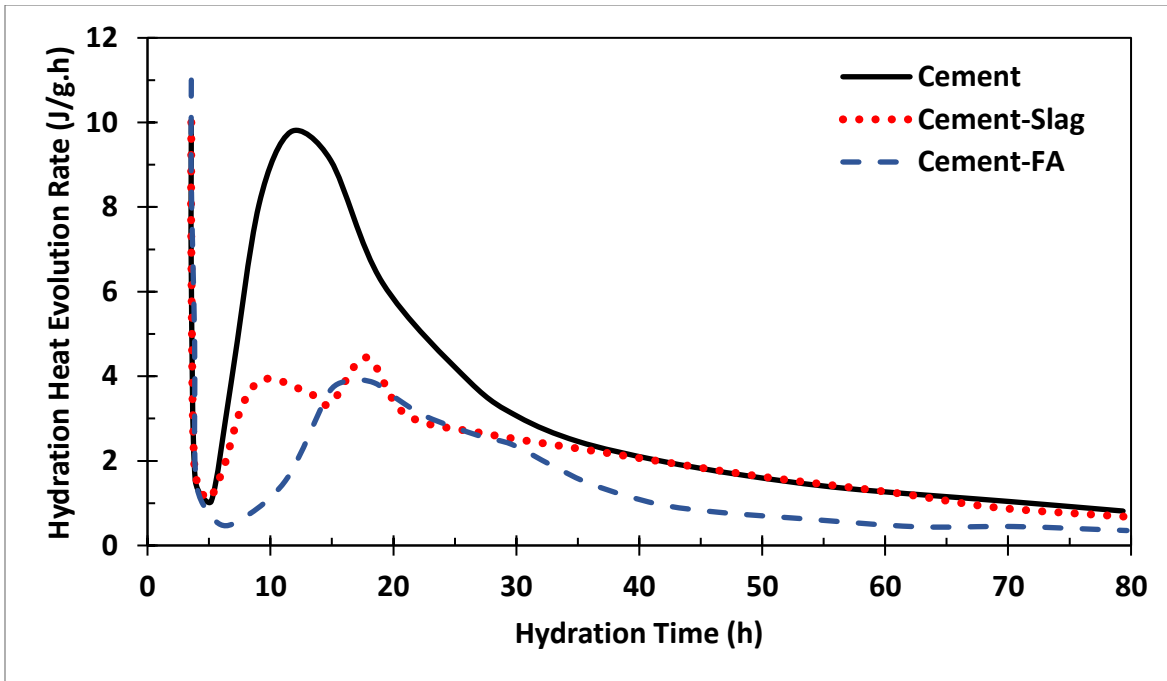


Figure 2.4 Variation of hydration heat evolution rate for different binder composite (modified from: Han et al. 2017).

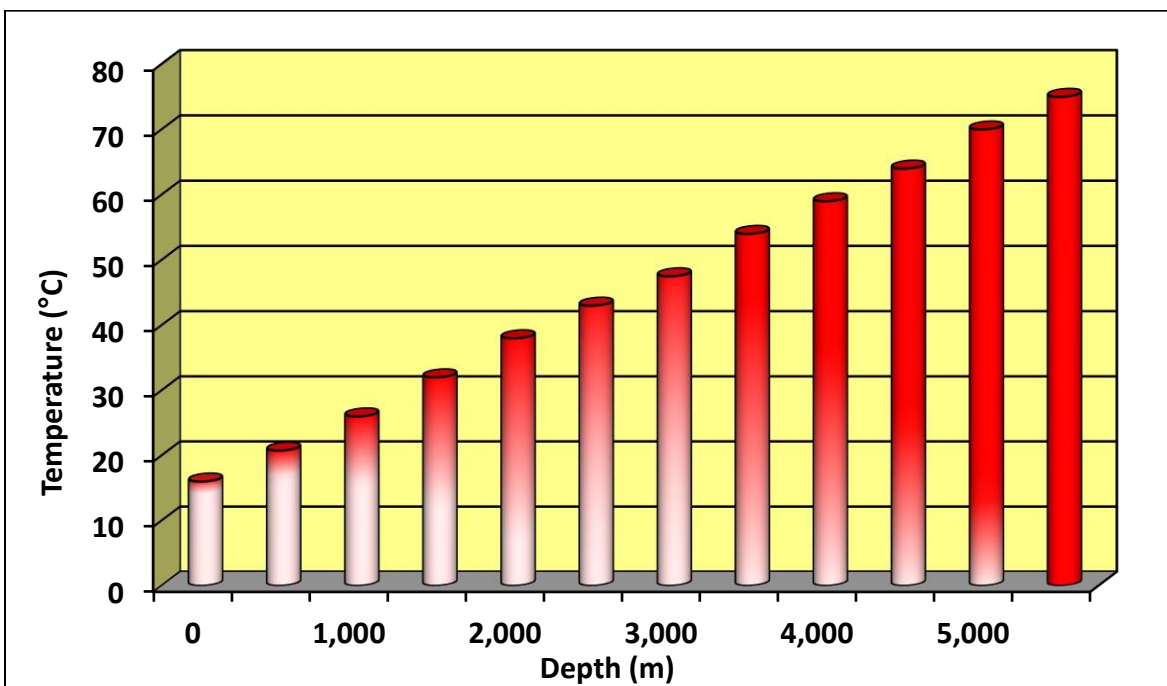


Figure 2.5 Temperature variation with depth in South African gold mine (modified from: Fall et al. 2014).

2.3.5 Paste-pipe friction

Regardless of the adopted delivery and distribution system of the CPB, there is always friction between the transported CPB and the inner sidewalls of the pipelines during the transportation of the fill from the production plant and the stope. This friction often elevates the temperature of the fresh transported CPB (Wu et al. 2013).

2.3.6 Temperature variation produced by the mining operations

Mining operations may use some technologies that can contribute to temperature variation of CPB within the mine stope. These technologies include mining machinery, blasting operations, fires, ventilation, and lighting. However, these heat sources may cause insignificant changes to the temperature of CPB because of the considerably large size of the mine stopes and/or the CPB structure (Fall et al. 2010).

2.4 Background of Hydraulic Binders, Its Hydration and Key Influential Factors

Hydraulic binders are the most important ingredient of any cementitious materials, such as CPB. The main task of hydraulic binders is to increase the strength of these materials by bonding their particles together (Landriault et al. 1997, Persson 1997). In CPB, there are quite a few types of hydraulic binders that are usually used, such as Ordinary Portland Cement (OPC), blast furnace slag (Slag), and Fly Ash (FA) (Haiqiang et al. 2016). An overview of these hydraulic binders, the basic hydration reactions, and the key influential factors have been briefly described in the following subsections.

2.4.1 Ordinary Portland Cement (OPC)

Ordinary Portland cement (OPC) is a hydraulic cement that experiences a dissolution and precipitation while reacting with water, forming different water-resistant hydrates that are responsible for the setting and hardening of the cement. The products of cement hydration include poorly crystalline gel of calcium silicate hydrate (C-S-H), thin to large hexagonal crystals of portlandite (CH), and needle-like morphological crystals of ettringite (Marchon and Flatt 2016, Cui 2017).

2.4.2 Blast Furnace Slag (Slag)

Blast-furnace slag cement (Slag) is a pozzolanic mineral admixture that has been extensively used in the past decades as a partial replacement of Portland Cement. In CPB operations, the utilization of pozzolanic admixtures (such as slag) can reduce the binder cost due to cement consumption, while maintaining the desired strength of CPB mixture (Daube and Bakker 1986, Cui 2017). The reactivity of slag is often activated by alkalinity which results from the hydration of clinker during the early hydration process of cement-slag blends (Roy and Idorn 1982, Daube and Bakker 1986).

2.4.3 Fly Ash (FA)

Fly Ash (FA) is a by-product pozzolanic material that is usually generated from coal firing for power generation. FA consists of fine, powdery particles that have spherical shape. There is an

increasing demand of using FA as it is considered to be highly contaminating material. However, the current utilization of FA in the form of alternative to cement and concrete products depends on several factors, such as the design strength and workability of concrete as well as the water demand. As pozzolanic admixture, FA can also be activated by alkalinity, and it is significantly affected by temperature (Palomo et al. 1999, Siddique 2004, Ahmaruzzaman 2010).

2.4.4 Hydration Process

Several researches (e.g. Schindler and Folliard 2003, Gruskovnjak et al. 2006, Bullard et al. 2011, Kolani et al. 2012, Scrivener et al. 2015, Marchon and Flatt 2016, Le et al. 2019) have explained the mechanism of hydration process of cement, slag, FA, and different blends of these binders. In general, there are four periods (Figure 2.6), which the progress of cement hydration with time passes through. The heat flow and the amount of cement hydration change with time at each stage. These four periods are:

- 1) Initial reaction period, which is the period of immediate reaction of cement when exposed to water. In this stage, significant amount of heat is released because of the rapid dissolution of gypsum and aluminite. This period lasts up to 1 hour after mixing.
- 2) Slow reaction period, wherein the hydration products (C-S-H and ettringite) will be formed around the calcium silicate. So, the magnitude of cement hydration will slowly increase, and low rate of hydration heat will be released. This period occurs between 1 and 2 hours after mixing.
- 3) Acceleration period, wherein a significant amount of hydration heat will be released. Thus, the hydration process rapidly increases. In this period, the peak level of hydration process is reached, the pore spaces will be further refined, and the bonds strength between particles will significantly improve. This period occurs between 2 to 9 hours after mixing.
- 4) Deceleration period, wherein the heat evolution rate will decrease because the hydration products (C-S-H and CH) will continue to form around the particles. Hence, there will be a noticeable reduction in cement hydration. This period begins 9 hours after mixing and continue until the hydration process reaches its minimum magnitude at around 24 hours after mixing.

When Slag is partially added to the blend, the hydration of slag begins during the initial reaction period of the cement hydration process. The early hydration of clinker releases alkalinity, which is responsible for activating the slag, synergistic with temperature rise and provides energy to activate alkali-hydroxide attack on the slag particles (Kolani et al. 2012).

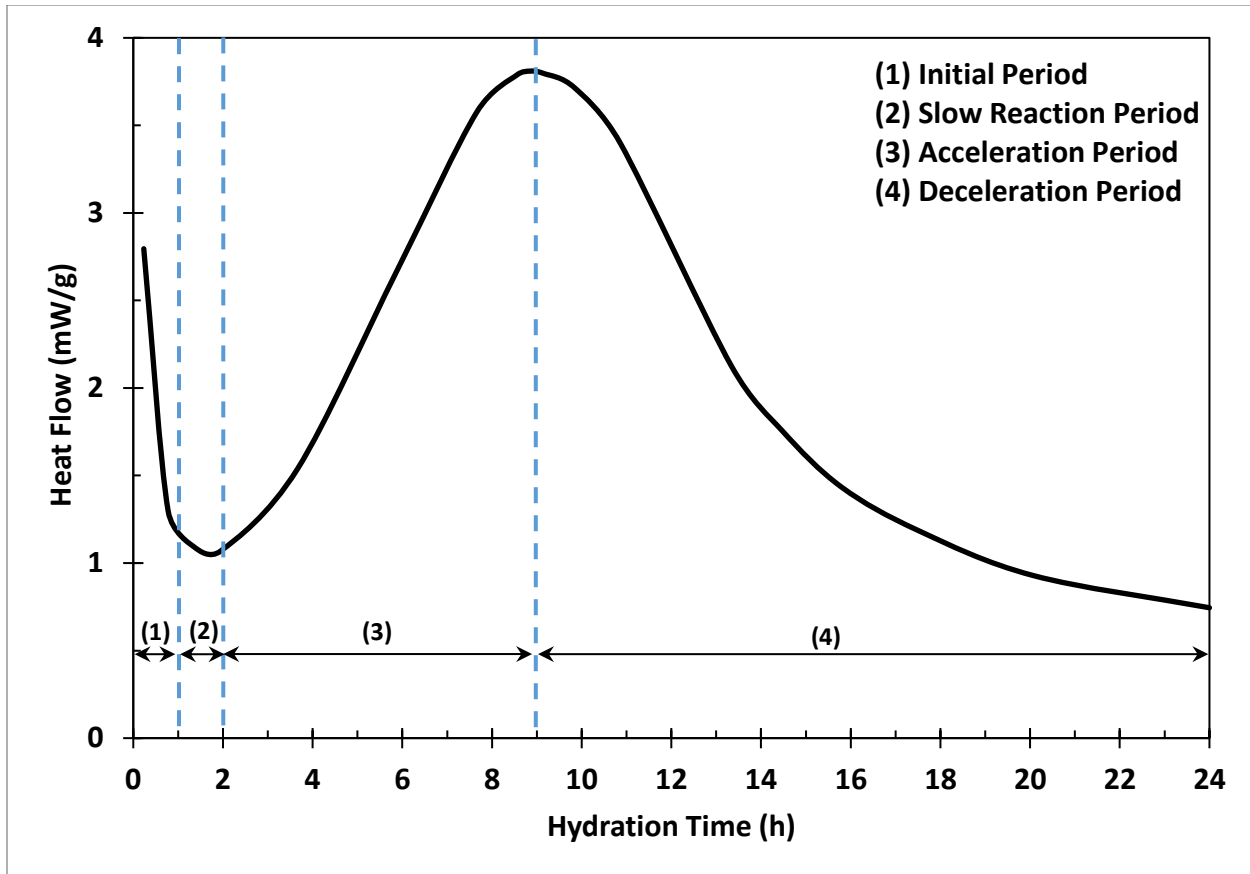


Figure 2.6 Progress of cement hydration with time (modified from: Bullard et al. 2011).

On the other hand, when FA is partially added to the cement blend, the early hydration of cement alite will be faster than its hydration with the pure OPC mixture, because the fine particles of FA will result in the formation of hydration products on the surface of the FA and the thin layer of the hydrates formed on the surface of alite (Sakai et al. 2005). However, the slow reaction of FA within the cement-FA blend makes the heat released during the initial hydration uncountable (Han et al. 2017).

2.4.5 Key Factors that Affect Binder Hydration

There are several factors that affect binder hydration, for example fineness of the binder material, amount of mixing water (i.e. w/c ratio), mixing and curing temperature, and chemistry of mixing water (Sakai et al. 2005, Bentz 2006, Lin and Meyer 2009, Fall et al. 2010, Li and Fall 2016, Wu et al. 2016, Han et al. 2017).

Several studies (Bentz 2006, Lin and Meyer 2009) have found that fineness of cement particles improves the mechanical strength of cemented materials as it accelerates the cement hydration process because the finer cement particles have larger surface area. This will provide more contact area for the mixing water that increases the amount of reaction between the water and cement particles. In addition, the more hydration of fine cement particles, the more production

of less thick cement hydration products. It reduces the setting time of cemented particles, and accelerates the reaction between water and fine cement particles. Also, beside the acceleration of the water-binder reaction due to the increase in production of thin layers of hydrates, the fine size and spherical shape of the FA particles will increase the packing density of the soils that are cemented with cement-FA blend and reduce the friction forces between these particles. This will reduce the volume of pore-water required to fill the voids between particles, and thus enhance the workability and flowability of the cemented material by reducing its yield stress and viscosity (Siddique 2004, Sakai et al. 2005, Kwan and Chen 2013). On the other hand, it was found that the presence of coarse slag particles within the cement-slag blends increases the reactivity of slag and significantly increases the hydration rate of slag particles (Tan et al. 2014), which will consequently increase the mechanical strength of soils cemented with cement-slag blend (Pokharel and Fall 2011).

Similarly, the degree of cement hydration was found to be another factor that significantly affects the hydration of soil-hydraulic binder mixes. It was found that the increase in w/c increases the porosity and hydraulic conductivity of the cemented material, and thereby increases the flowability (reduces the rheological properties) (Figure 2.7) and reduces the mechanical strength of these materials (Figure 2.8) (Bentz 2006, Fall et al. 2008, Harini et al. 2011, Ramezani et al. 2016)..

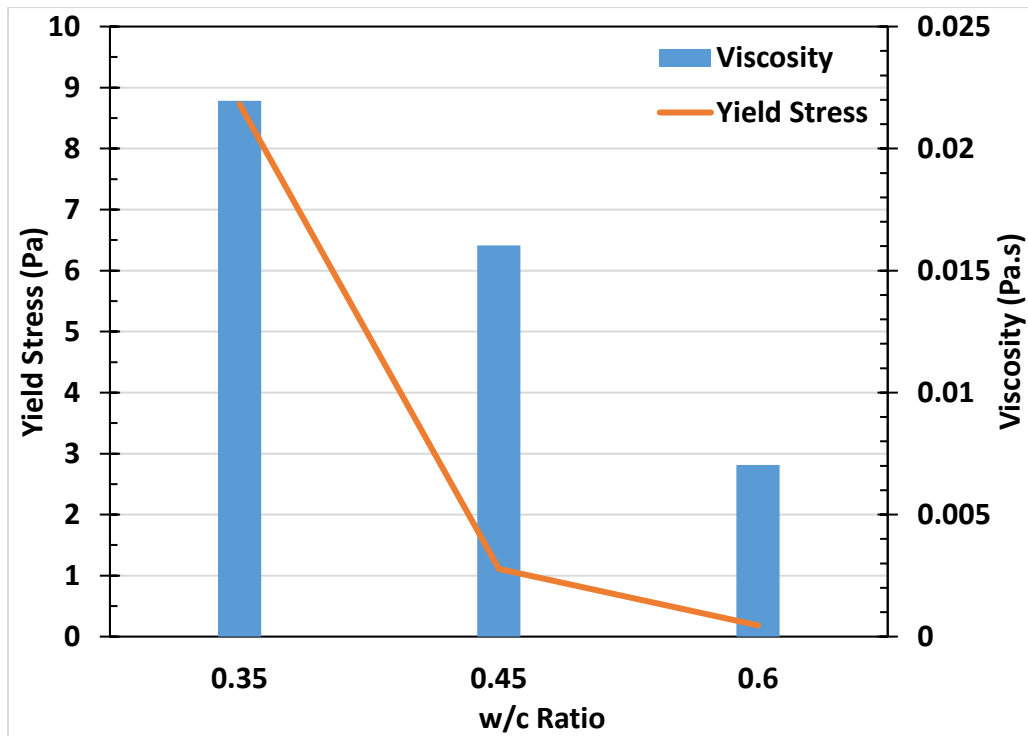


Figure 2.7 Effect of water-to-cement (w/c) ratio on the rheological properties of self compacted concrete (modified from: Ramezani et al. 2016).

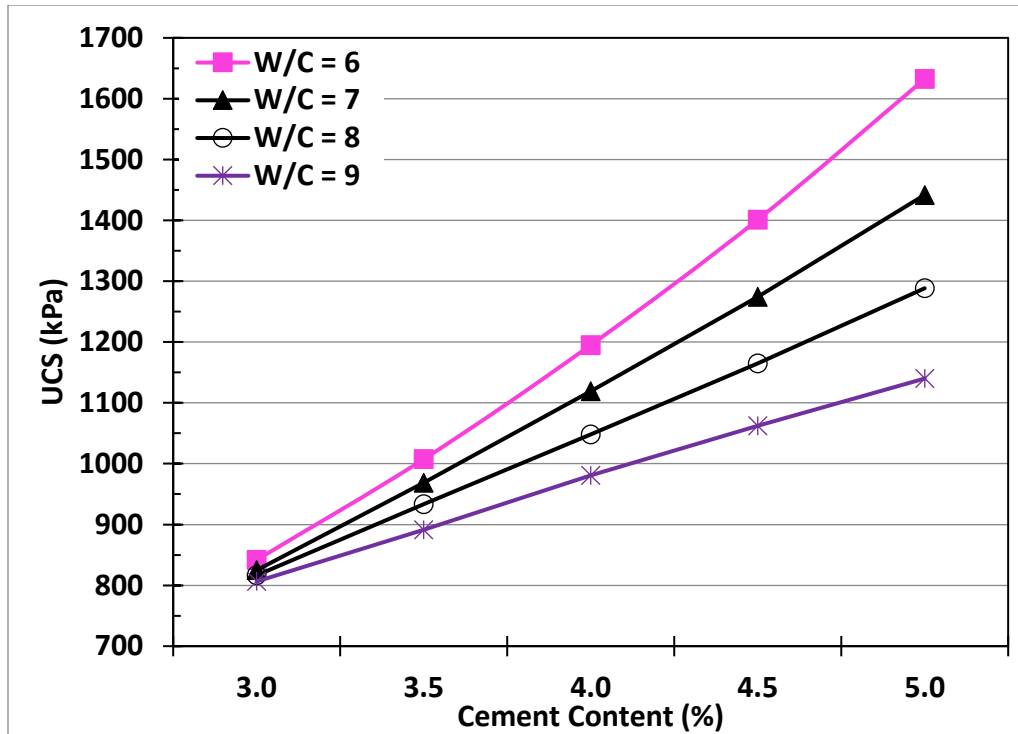


Figure 2.8 Effect of water-to-cement (w/c) ratio on the mechanical strength of cemented Paste Backfill (modified from: Fall et al. 2008).

The initial (mixing and curing) temperature of cementitious material is considered to be another important factor that influences the hydration process. It has been found that high initial and/or curing temperature will reduce the pore spaces between particles and reduce the water content. Thus, the cement hydration process will accelerate, and more cement hydration products will be generated within the pores between the particles. Consequently, the high initial and/or curing temperature will improve the mechanical strength of cemented soil at early ages (Figure 2.9) (Lin and Meyer 2009, Fall et al. 2010, Wang et al. 2016). Moreover, the higher temperature has greater effect on the reactivity of slag, as compared to other hydraulic binders, and it can also enhance the pozzolanic reaction of FA (Deschner et al. 2013, Ogirigbo and Black 2016). However, the increase in initial temperature will negatively affect the flowability of cemented materials by increasing its rheological properties (Figure 2.10), as it will cause significant reduction in the water content of these materials (Haiqiang et al. 2016, Haruna and Fall 2019). Also, the increase in curing temperature to more than 85°C will significantly reduce the mechanical strength of cementitious material because this range of curing temperature will lead to less uniform microstructure, higher porosity, and coarser pore structure within the soil particles (Maltais and Marchand 1997, Elkhadiri et al. 2009).

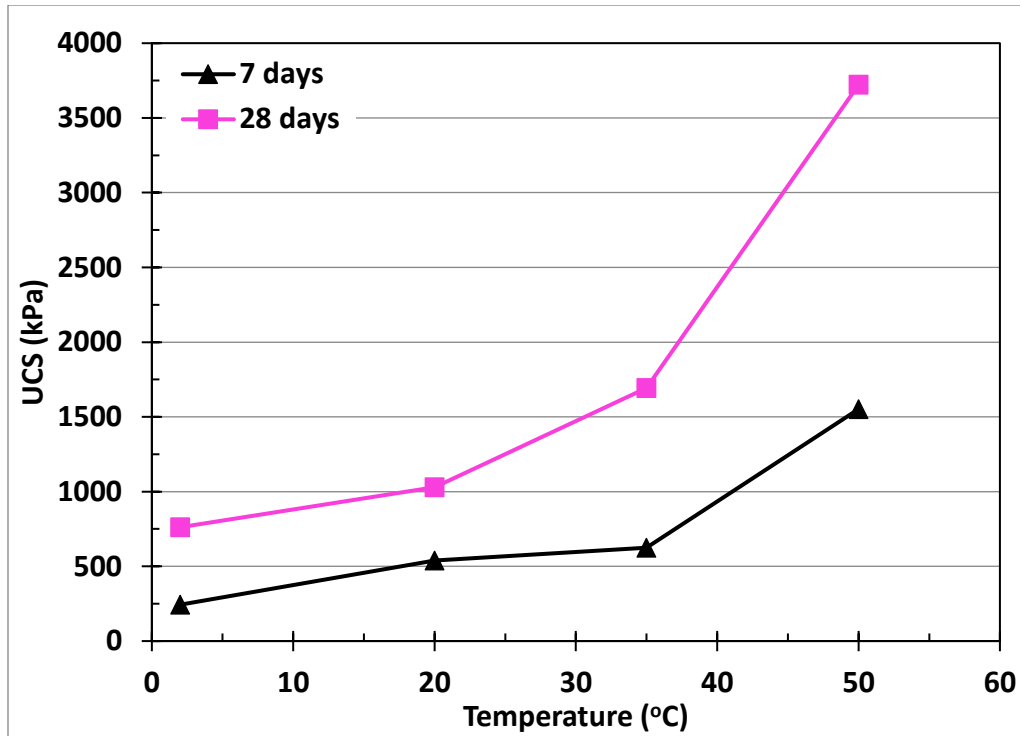


Figure 2.9 Effect of curing temperature on the strength development of cemented Paste Backfill (modified from: Fall et al. 2010).

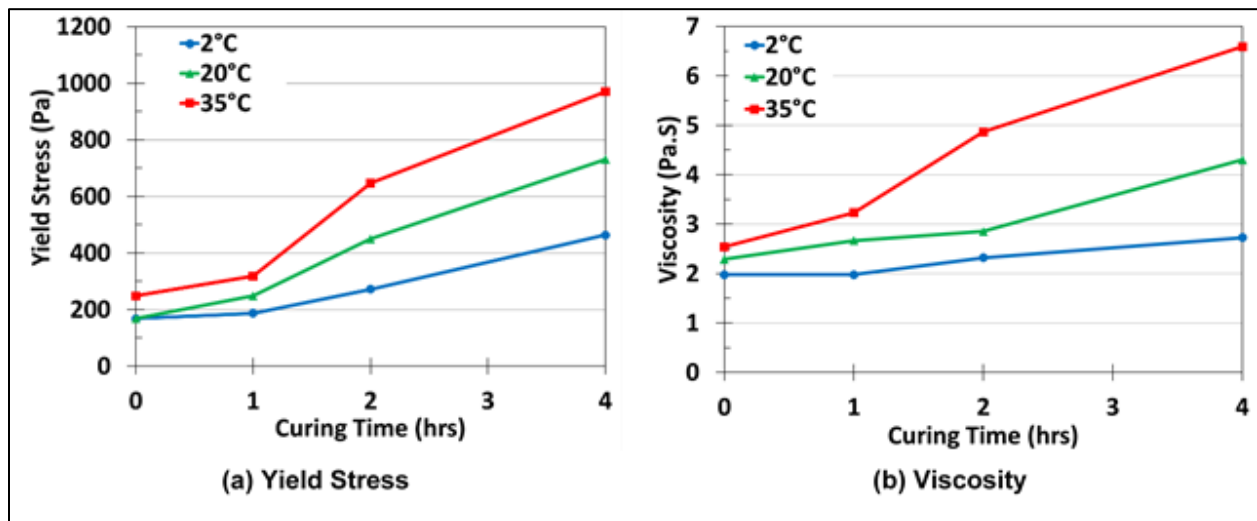


Figure 2.10 Effect of curing temperature on the rheological properties of cemented Paste Backfill: (a) Yield stress, and (b) Viscosity (modified from: Haruna and Fall 2019).

Hydration process of different hydraulic binders was found to be significantly influenced by the chemistry of mixing water. The presence of uncontrolled chemical components (such as sulphate) might slowdown the hydration process because of the reaction between the sulphate anions in the mixture and C_3A grains within the binder which will form a thin coating of anhydrated cement particles and prevent the C_3A from quickly reacting with water (Li and Fall

2016, Alainachi and Fall 2020). On the other hand, the presence of other chemical components (such as sodium silicate) might increase the rate of the cement hydration process and improve the mechanical strength of cementitious soils (Abdul-Hussain and Fall 2012). It was also found that sodium silicate potentially contributes to the activation of pozzolanic binders, such as slag and FA (Jawed and Skalny 1978, Qing-Hua et al. 1992, Palomo et al. 1999, Gruskovnjak et al. 2006, Cihangir et al. 2018).

2.5 Sodium Silicate

The main components of alkaline binders were discovered in 1967. Since then, many studies have been conducted to understand the activation role of these activators (Garcia-Lodeiro et al. 2015). Soluble sodium silicates, also known as water soluble glass, are silicate polymer, which are an inorganic chemical, and a clear, colorless, and viscous liquid. They are classified as Alkali activators that are generally produced by varying proportions of alkali metal and SiO_2 (Razavi and Hassani 2007). The main application of sodium silicate, from geotechnical point of view, is to reduce the water content and/or the w/c ratio of the soil grout and mine tailings backfill. This will reduce the quantity of free water in mine stopes, and thereby improve the strength of the backfill (Abdul-Hussain 2011). Also, adding sodium silicate to cemented material helps in lowering the threat of thermal cracking as it produces lower hydration heat as compared to cement (Hassani et al. 2007). Nevertheless, there is lack of information on the effect of sodium silicate on the rheology and mechanical strength of soils, potentially tailings, which are cemented with different hydraulic binders.

In the following subsection, the effect of sodium silicate on the hydration process of different hydraulic binders is briefly discussed.

2.5.1 Cement-Sodium Silicate Interaction

Mixing cementitious material with alkaline activators (such as sodium silicate) results in alkaline attack on the aluminosilicate of these materials. This will form the alkaline cement, which potentially sets and hardens, and thereby improves the binding properties of these materials (Garcia-Lodeiro et al. 2015). Sodium silicate is widely used as an accelerator and low-density lightning admixture in Portland cement. It is also used as self-healing agent in self-healing cement (Guo et al. 2019). Moreover, it was found that rate of hydration of the cement clinker minerals significantly increases with the presence of sodium silicate, because adding sodium silicate to cemented mixture will result in the reduction of the initial pH of the pore-solution of cement paste and potential generation of hydration products (such as CH) (Brykov et al. 2002).

2.5.2 Slag-Sodium Silicate Interaction

The initial hydration process of slag-water mixture is much slower than the initial hydration process while mixing Portland cement with water. However, the rate of hydration of mixing water with cement-slag blend depends on the dissolution of the glassy structure of slag during the initial hydration of cement (Sajedi and Razak 2010). There is a common agreement that adding sodium

silicate to pozzolans (such as slag) will effectively activate these pozzolans and accelerate its hydration process, because the early hydration process of cement clinker releases alkali and increases temperature, which will provide high energy to activate the alkali-hydroxide attacks on the slag particles (Roy and Idorn 1982, Kolani et al. 2012, Kermani et al. 2015, Azadi et al. 2017). Moreover, Zhao et al. (2020) found that the hydration and mechanical strength of slag are significantly affected by adding sodium silicate, specially at the early age, as it shortens the setting time, increases the production of C-S-H, and enhances the compressive strength. However, it was reported that the hydration process, the production of hydration products and consequently the strength of the slag-cement pastes activated by alkali activators (such as sodium silicate) are influenced by several factors. These factors include the nature of slag and activator, water/slag ratio, fineness of slag, dosage of activator, and curing temperature etc. (Sajedi and Razak 2010).

2.5.3 Fly Ash-Sodium Silicate Interaction

The partial replacement of Portland cement with FA has become common as it lowers the carbon dioxide emission as a result of the manufacturing process of Portland cement. However, the strength development of cement-FA paste, at ambient temperature conditions, was found to be slower, producing material of relatively lower strength. Using sodium silicate as alkali activator was found to enhance the mechanical performance of the cement-FA paste as it promotes the condensation process of the mixture (Palomo et al. 2007, Panias et al. 2007, Rashad 2013, Phoo-ngernkham et al. 2017). However, the cement/FA ratio and/or the type of dosage of the sodium silicate controls the strength development of cement-FA blends activated by sodium silicate, because the products resulted from the reaction between the active silica (from sodium silicate solution) and the calcium (from the cement) are responsible for the strength performance (Morsy et al. 2014, Phoo-ngernkham et al. 2017).

2.5.4 Effect of sodium silicate on CPB

The effect of adding sodium silicate, as alkali activator, on the properties of CPB was studied by only few researchers. For instance, (Kermani et al. 2015) evaluated the effect of sodium silicate on the strength of gelfill, which is the mixture of tailings, hydraulic binder, water, and chemical additives (such as sodium silicate gel). They found that the UCS values of gelfill increase with increase in the dosage of sodium silicate up to 0.3% of the total dry weight (wt%), while these values significantly decrease when the sodium silicate content exceeds 0.5%. Also, they found that CPB materials that contain 0.7% or more of sodium silicate have no measurable strength within the first 14 days of curing (Figure 2.11). They attribute to the increase in the total porosity of samples and the amount of water trapped in the samples.

Abdul-Hussain (2011) experimentally simulated the coupled behavior of the thermal, hydraulic, and mechanical (THM) properties of CPB that contains sodium silicate by building two columns of cemented paste tailings under drained and undrain conditions. Both columns were cured at room temperature for 28 days, and it was found that the strength development (UCS

values) is significantly coupled with the heat development, saturation degree, and suction development within the CPB that contains sodium silicate. They also found that the chemical interaction between the cemented tailings particles, mixing water, and sodium silicate has significant effect on the cement hydration process and rate. So, the strength development is significantly affected by the dosage of sodium silicate (Figure 2.12).

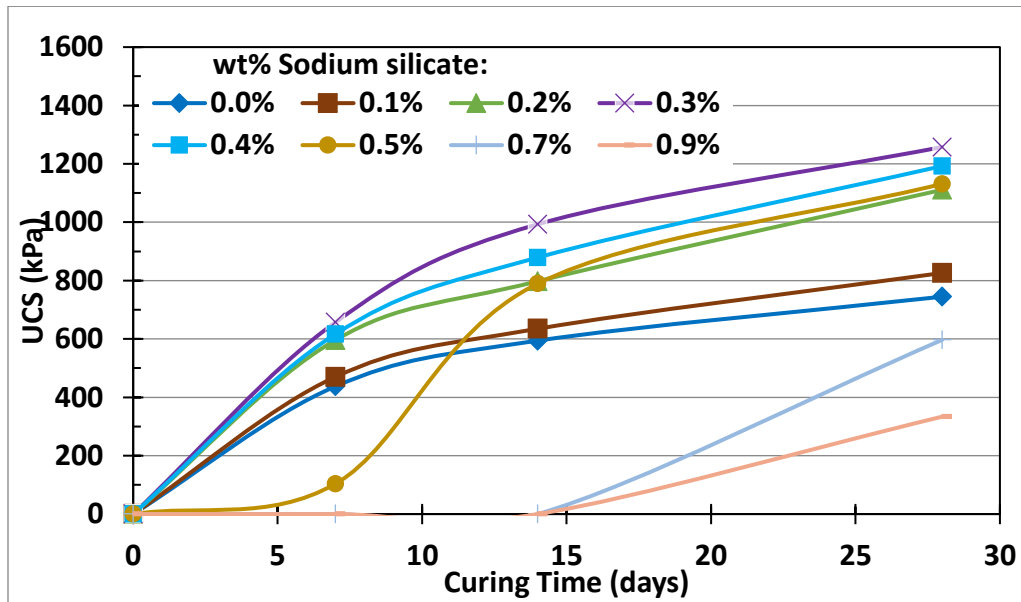


Figure 2.11 Effect of sodium silicate content on the strength development of CPB (modified from:Kermani et al. 2015).

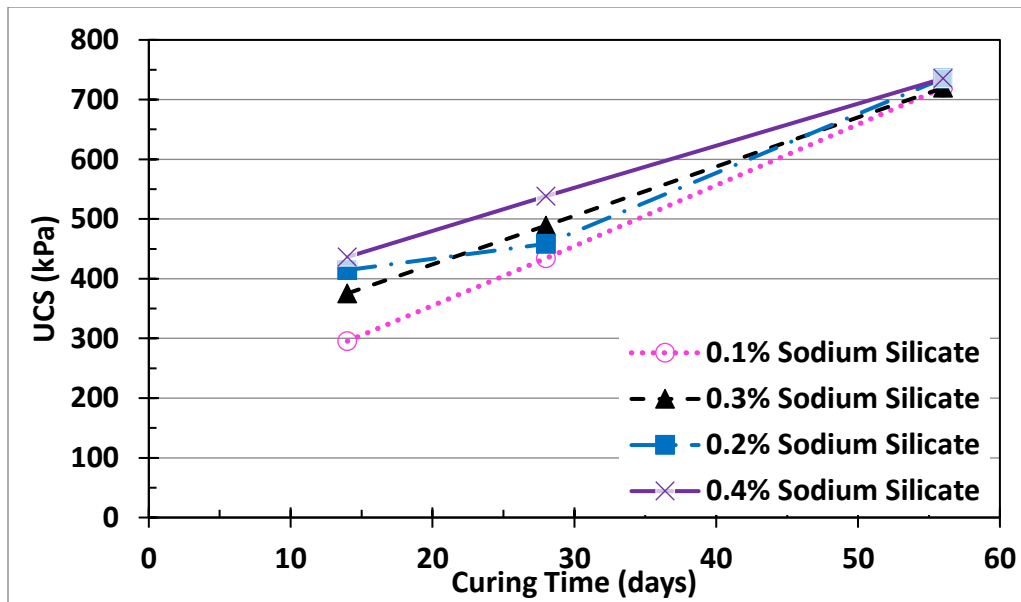


Figure 2.12 Effect of sodium silicate content on the strength development of CPB (modified from: Abdul-Hussain 2011).

2.6 Rheology

Rheology, in general, refers to the deformation and flow of a material in terms of the relationship between stress, strain, and time (Barnes et al. 1989). From civil engineering point of interest, rheology can provide valuable and practical information about the properties of engineered fluids and admixtures (such as concrete, mortars, cement based suspensions, cemented slurries, and cemented paste backfill) that exhibit a time-dependent response to stress (Bannister 1980, Struble et al. 1998, Wallevik and Wallevik 2011). Rheology is also considered as a key factor for determining the flowability of these materials, which is a potential factor in the transportation/delivery systems of cemented slurry. If transported material does not have appropriate flowability, clogging of the pipelines might occur, which will lead to possible flow delays and/or temporary interruption of the progress of these materials as well as possible replacement of the pipelines. Hence, the lack of understanding of the rheological properties of the transported material might lead to significant financial losses (Bannister 1980, Li and Fall 2016, Haruna and Fall 2019). The scientific approach in assessing the rheology, and thus the workability and flowability of cemented slurries, is based on measuring the yield stress and viscosity of these materials (Szecsy 1997).

In the following subsection, an overview of the yield stress and viscosity and a brief discussion on the previous research that studied the rheology of CPB have been presented.

2.6.1 Yield Stress

Many fluids often show plastic behavior, in which they require a certain level of stress to initiate flow. This level of stress is known as “Yield Stress” (Struble et al. 1998). In other words, the term “yield stress” refers to minimum pressure or shear stress that is required to overcome the static friction of the fluid and make it flowable (Sofra 2017). In CPB technology, the yield stress of the backfill should range between 200 and 700 Pa to better work within the operational pipeline transportation system (Figure 2.13) (Potvin et al. 2005, Belem and Benzaazoua 2008).

There are several (indirect and direct) approaches to determine the shear stress of the cemented slurries. The indirect methods, which are based on the interpretation of fundamental shear stress-shear rate data and obtain the shear stress in the limit of zero shear rate, include the direct extrapolation of the rheological shear stress, and shear rate data as well as the extrapolation of the flow curves. While the direct methods rely on measurement of the shear stress, at which flow first begins. These methods are experimentally conducted under the condition of (a) controlled shear stress, at which deformation of material is observed as unctio of time when exposed to constant shear stress, or (b) controlled shear rate, at which the shear stress-time response is measured while exposing the material to low and constant shear rate. The most common direct methods in engineering practice are the stress relaxation method and vane shear method (Dzuy and Boger 1983). In the current study, Vane shear method was adopted to determine the yield stress of CPB.

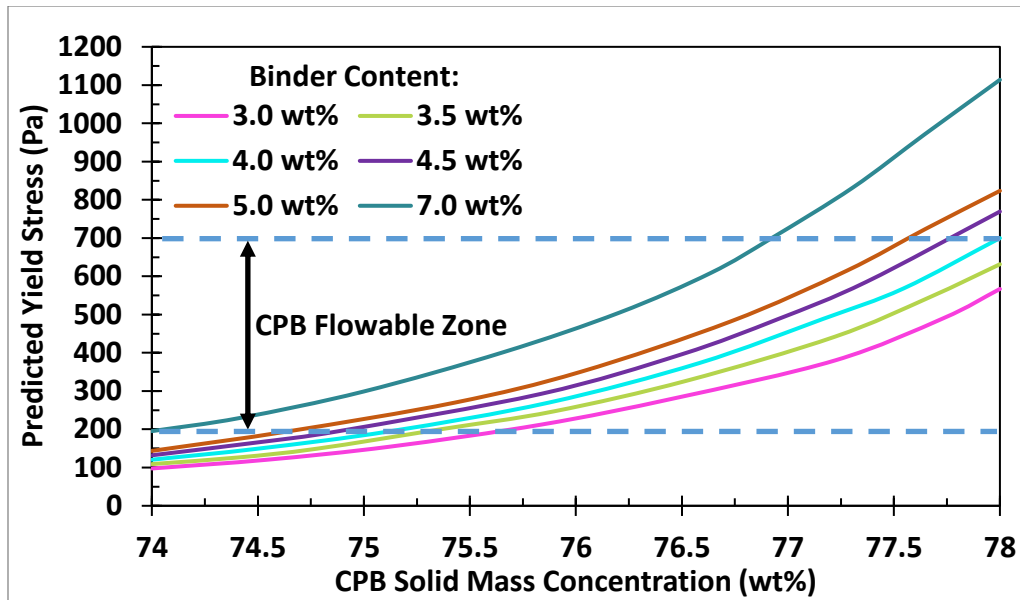


Figure 2.13 Predicted yield stress and flowable zone of CPB of different binder and solid content (modified from: Belem and Benzaazoua 2008).

Vane shear strength testing is considered as one of the most efficient and accurate methods among the direct measuring methods for the yield stress of soil and Newtonian fluids, which can be performed in the field and/or laboratory (Dzuy and Boger 1985, Abd Elaty and Ghazy 2012). The yield stress values provided by vane shear methods were found to be greatly synchronized with the most of the other rheological testing methods. In the recent years, this method has been extensively used for different construction materials, such as the concrete and mortars (Bauer et al. 2007). The vane shear test can be summarized by inserting a four-bladed vane (H:D=2:1) in the end of the remolded cylindrical sample (Figure 2.14). Blades rotation is then applied by a calibrated torque spring (or electrical torque transducer) that is attached directly to the vane (Figure 2.15), and the critical torque that can cause shear in the cylindrical surface of the sample is determined. By converting the torque to a unit shearing resistance, the yield stress can be calculated.

In the current study, a Wykeham-Farrance vane shear (Figure 2.16) has been used to determine the yield stress of the CPB samples. The vane shear consists of an electrical motor that rotates the four-bladed vane with a diameter of 2.5 cm and the height of 2.5 cm, which is connected to the deflection spring and a dial gage to measure the resistance of the sample to the applied torque. The test was performed in accordance with ASTM D4648/D4648M-16 (2015). Sample preparation and the test procedure are discussed in detail in Chapter 3 (Paper I) of this dissertation.

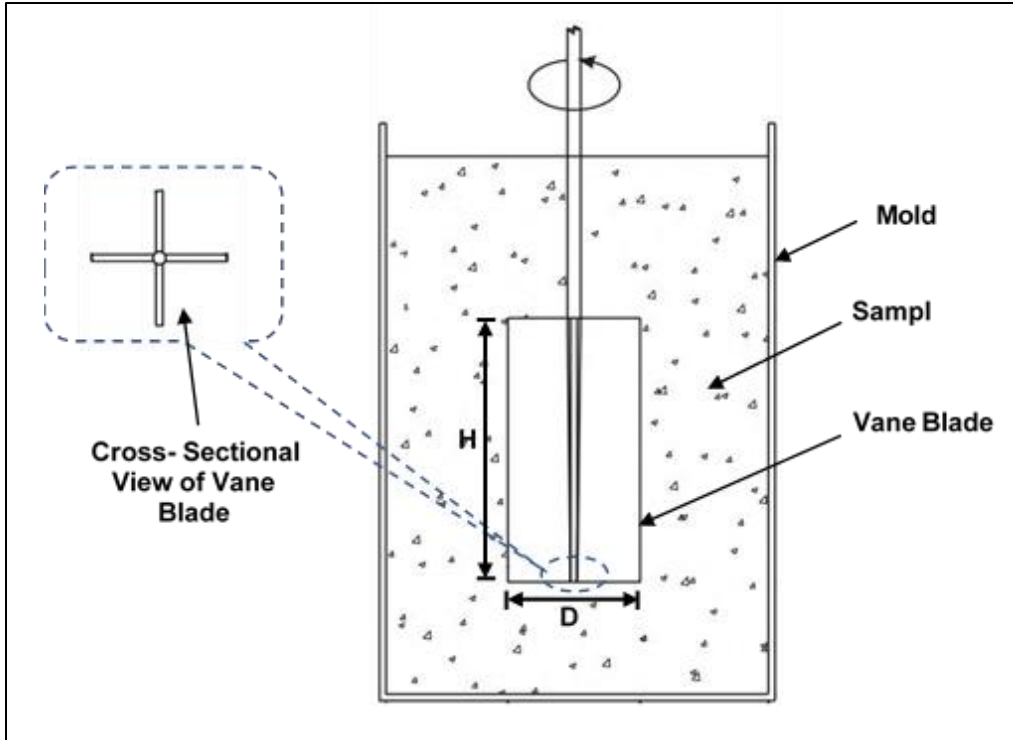


Figure 2.14 Schematic view of the Vane shear test method (modified from: Bauer et al. 2007).

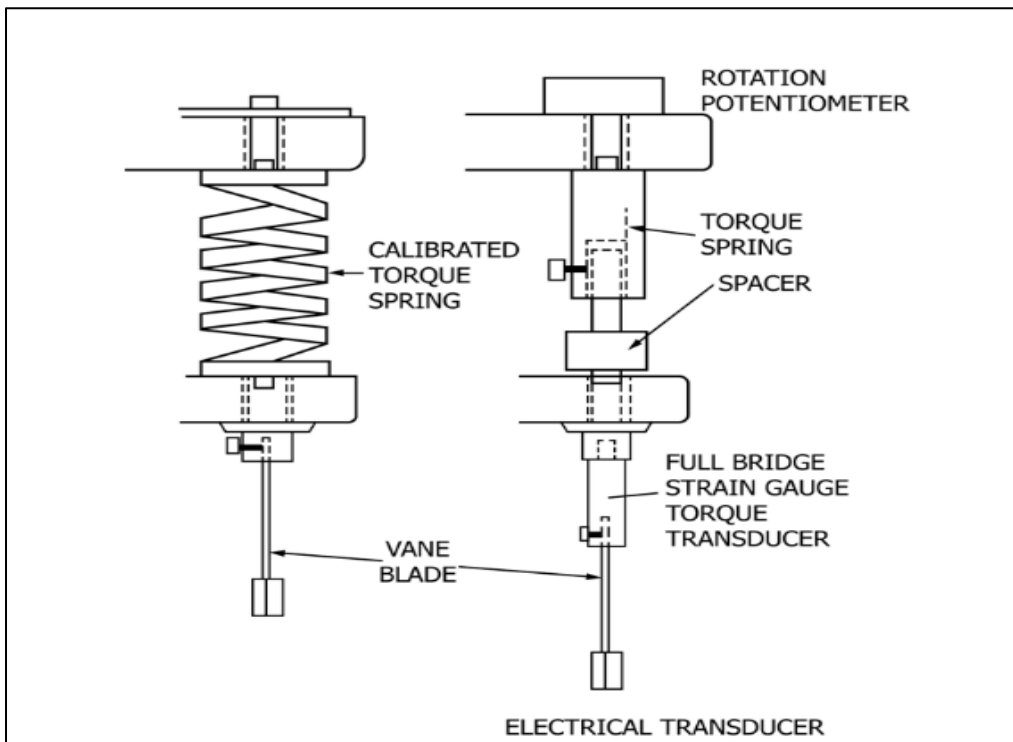


Figure 2.15 Vane torque spring and electrical transducer (ASTM D4648/D4648M-16 2015).

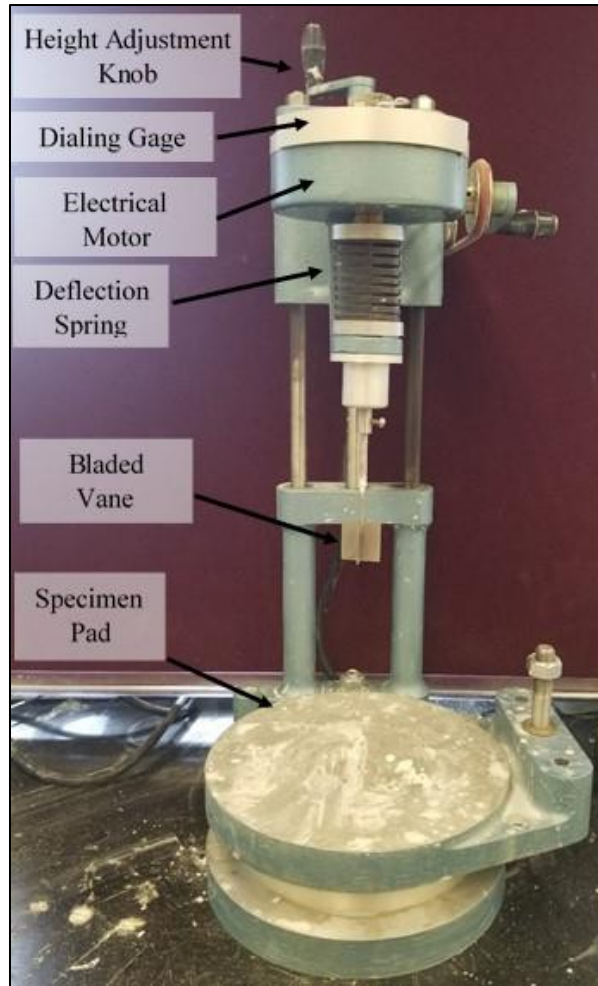


Figure 2.16 Vane shear apparatus used in this study.

2.6.2 Viscosity

Theoretically, viscosity can be defined as the measure of the internal friction of a material and its resistance to flow (Barnes et al. 1989). It might also refer to the force per unit area that produces a motion of a fluid (Szecsy 1997). In CPB technology, the viscosity represents the ratio between the shear stress and the shear rate at a specific shear rate, and it governs the dynamic flow characteristics of the tested material as it measures the resistance of the material against the shear stress-induced flow (Ercikdi et al. 2017, Bian et al. 2019).

There are different empirical methods for determining the viscosity of fluid suspensions, such as the oscillating-vessel method, at which the viscosity is measured by conducting an analytical mathematical model to back calculate the viscosity from the damping of the vessel (Roscoe 1958). However, there are several factors that might result in discrepancy of the results determined by this method, such as the surface tension and slippage at the liquid/vessel interface etc.(Cherne and Deymier 1998), as well as the possible uncertainty of results obtained by these

methods as they are operator-sensitive. Accordingly, there is a great need for adopting new methods that rely on fundamental physical quantities, rather than the methods that are based on the measuring apparatus (Tattersall and Bloomer 1979, Tattersall and Banfill 1983). For this purpose, an advanced viscosity testing apparatus was developed by the community of concrete science, which is named “Viscometer”. In general, the viscometer measures the shear rate by observing the torque as a function of the predetermined rotational speed. Thus, the viscosity is determined as the ratio of the shear stress to the shear rate at the specific shear rate (Sofra 2017). There are several types of viscometer; couette viscometer, coaxial cylinders, and Brookfield digital viscometer (which has been used in the current study). The Brookfield digital viscometer (Figure 2.17) consists of a rotating spindle connected to a calibrated spring. To measure the viscosity, the spindle is immersed into the fluid to be tested and is then rotated in the liquid. The liquid will then resist the rotation, which is known as viscous drag that is determined by the spring deflection. The spring deflection is measured by the rotary transducer (Brookfield 2014). Sample preparation and the test procedure are discussed in detail in Chapter 3 (Paper I) of this dissertation.

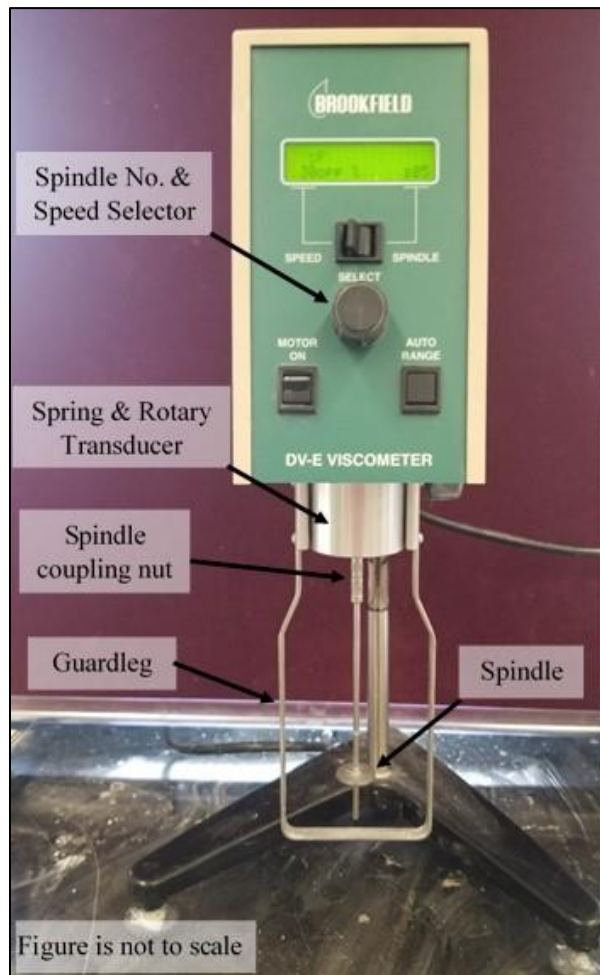


Figure 2.17 Vane shear apparatus used in this study.

2.6.3 Rheology of CPB

The rheology of CPB has received great attention in the literature, as it provides good indication of the workability and flowability of the backfill being transported through the delivery system to the underground mine stops. For instance, Huynh et al. (2006) studied different factors that might affect the yield stress of CPB, and they found that the yield stress of CPB is affected by the percentage of solid concentration. It was found that the CPB mix that is prepared with solid concentration of 70 – 85 wt% produces high yield stress that makes it difficult to transport in real plant operations. It was also found from this study that adding high concentration of chemical additives, such as polyphosphate, is required to disperse the paste to a suitable level of flowability.

Belem and Benzaazoua (2008) developed predictive models to compare different mix designs of CPB, based on the practical experience of the authors and some empirical methods, by determining the rheological properties, binder content, and soil mass concentration. The result of this study predicted that the shear yield stress of CPB material increases with the increase in the solid concentration and/or the increase in the binder content. However, the study found that extensive increase in these components (such as 77 (wt%) of solid mass concentration and 7% of binder content) will lead to a non-flowable CPB mix, as the magnitude of yield stress will fall beyond the CPB flowable zone.

Zhang et al. (2010) discussed the effect of superplasticizer on the workability and initial setting of CPB, and found that the yield stress of CPB increases as the time progresses due to cement hydration. However, it was also noted that by adding superplasticizer, the initial setting time of CPB material will be delayed, that will slow down the increase in yield stress.

Kwan and Chen (2013) discussed the benefits of adding fly ash (FA) to the CPB mixture in terms of improving the packing density, flowability, and strength. The results of this study showed that the cement-FA blends significantly increase the packing density of CPB, which causes large reduction in void ratio. This reduction in void ratio would potentially reduce the amount of water needed to fill these voids that increases the amount of unused water, which can coat the solid particles and act as lubricant. This will thereby reduce the viscosity and yield stress of CPB.

Simon and Grabinsky (2013) assessed the apparent yield stress of CPB material under the effect of the variation of binder type and content, presence of different chemical admixtures (superplasticizer), and pore fluid chemistry (such as pH and zeta potential). It was learnt from this study that the apparent yield stress increases with the partial usage (30%) of FA, as the mixture became denser. Also, the usage of chemical admixtures decreases the apparent yield stress, but the amount of reduction is significantly affected by the type and concentration of these admixtures. Moreover, it was noted that the apparent yield stress decreases at low pH and with the increase in magnitude of zeta potential.

Haiqiang et al. (2016) and Jiang and Fall (2017) studied the development of yield stress of CPB in sub-zero environment. Haiqiang et al. (2016) found that the yield stress of CPB will

significantly decrease when the CPB is prepared and cured at -6°C as compared to the CBSs that are prepared and cured at 23.5°C , and the further decrease in ambient temperature causes more reduction in the yield stress of CPB. Also, the yield stress in sub-zero condition will furtherly decrease with the reduction in binder content, increase in w/c ratio, and/or the partial usage of FA. While the partial usage of slag will increase the yield stress after 1 hour of curing time, Jiang and Fall (2017), on the other hand, found that salinity of CPB mixture (in sub-zero conditions) decreases the yield stress, as the yield stress decreases with the increase in the NaCl concentration.

The yield stress and apparent viscosity of CPB were investigated by Panchal et al. (2018) as a function of hydration age, binder content, and superplasticizer dosage, and it was found that at a given shear rate, the yield stress and apparent viscosity decrease with the increase in hydration age for specific binder content and concentration of superplasticizer.

Haruna and Fall (2019) studied the time and temperature effect on the rheological properties of CPB that contains superplasticizer, and they found that yield stress and viscosity of CPB increase with the increase in the initial (mixing and curing) temperature, and/or the reduction in the superplasticizer concentration. It was also noted that the yield stress and viscosity are also affected by the change in tailing type with different dosages of superplasticizer and different curing temperatures.

Xiapeng et al. (2019) investigated the sulphate-induced changes of the yield stress and viscosity of CPB. The results show that the rheological properties decrease with the increase in sulphate concentration, regardless it was prepared with silica tailings (ST) or natural tailings (NT). Also, the increase in binder content will increase the yield stress and viscosity, regardless of the presence of sulphate.

Kou et al. (2020) experimentally investigated the time-dependent rheological behavior of CPB that contains alkali-activated slag as a binder. It was found that both yield stress and viscosity gradually increase with progress of curing time and the increase in binder content (up to 8%). Further increase in binder content may cause reduction in initial yield stress and viscosity. Moreover, it was found that the increase in the fineness of slag promotes the reactivity of slag, which will produce more cement hydration products and consume more pore-water, and thus increase the rheological properties of CPB that contains alkali activated slag.

Roshani and Fall (2020) found that the addition of non-silica particles to the CPB admixture or increasing the dosage of the non-silica particles will increase the yield stress and viscosity. Also, the increase in the rheological properties of CPB in the presence of non-silica particles becomes more significant with the increase in curing time and temperature. Moreover, the increase in these properties of CPB that contains non-silica particles is dependent on the binder type, as the yield stress of CPB that is made with PCI increases (with the increase in the dosage of non-silica particles) at faster rate as compared to CPBs made of cement-Slag and cement-FA blends.

In the current study, the rheological properties (yield stress and viscosity) of CPB were investigated under the combined effect of curing time, initial (mixing and curing) temperature, different binder (PCI, PCI-Slag, and PCI-FA) blends, and different dosages of alkali activator (sodium silicate). To the best knowledge of the author, the approach of studying the coupled effect of these factors has not yet been addressed in the literature. Further details of the experimental program adopted in this research as well as the methodology, experimental conditions, and instrumentations are briefly described in the Chapter 3 of this dissertation.

2.7 Compressive Strength

2.7.1 Background

The Unconfined Compression Strength (UCS) test, also known as uniaxial compression test, is a simple and effective method to assess the deformability and behavior of a material against compressive loading. UCS is generally determined by subjecting samples to compressive load along a single (usually vertical) axis and recording the compressive displacement (change in dimensions) of the sample along that axis (Wittke 2014, Pan 2019). It was previously found that the unconfined compression of a soil might vary based on the material itself (such as the cemented or non-cemented material and the saturation condition of the material etc.) and the testing characteristics and conditions, such as performing the test under stable or unstable confining stress, drained or undrained conditions, and static or cyclic compressive loading etc. (Mitachi et al. 2001, Chae et al. 2010, Fredlund et al. 2012).

There are several approaches, where the UCS can be conducted. These approaches are mainly based on the type of the tested material. For instance, ASTM D2166/D2166M - 16 is usually used for determining the UCS of cohesive soils, ASTM D5102-09 is used for compacted soil-lime mixture, ASTM D2938-95 is used for intact rock specimens, and ASTM C39/C39M is often used for determining the UCS for cylindrical concrete specimens. In this study, the unconfined compression strength tests of CPB material were conducted according to ASTM C39/C39M (2018), computer-controlled mechanical press of ELE Digital Tritest (Figure 2.18), and the tests were conducted with loading capacity and deformation rate of 50 kN and 1 mm/min respectively. The tested specimen had a height-to-diameter ratio of 2:1. Before conducting the test, the two ends of the samples were first flattened to get their surfaces normal to the plates of the mechanical press. Sample preparation and the detailed test procedure are discussed in Chapter 4 (Paper II) of this dissertation.

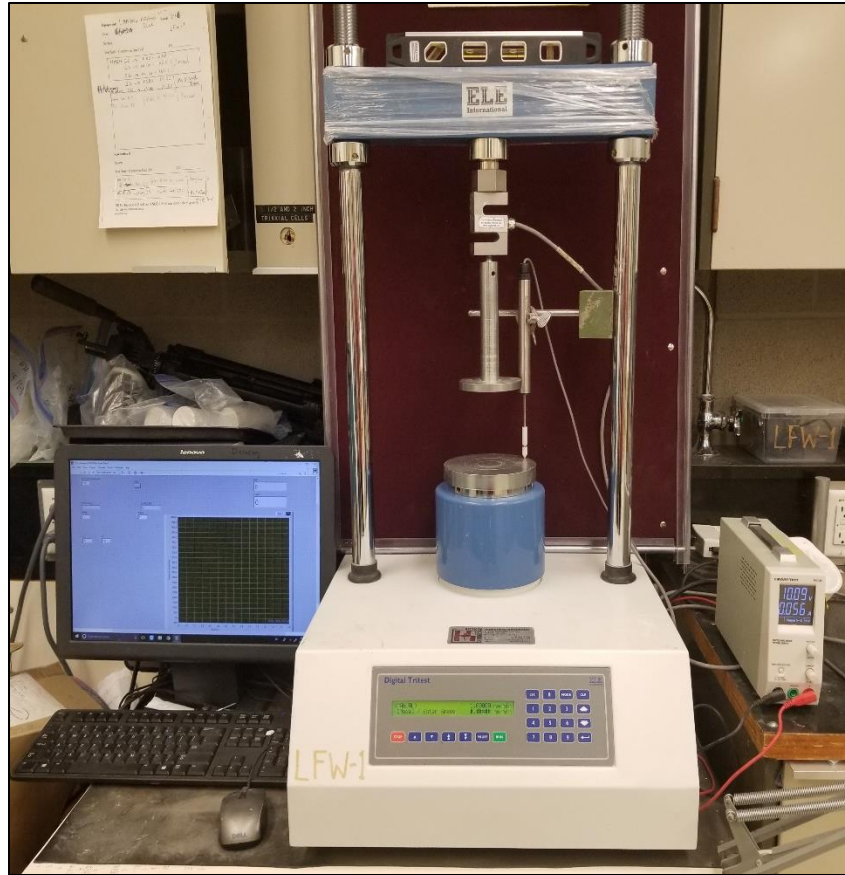


Figure 2.18 Unconfined Compression Strength (UCS) testing apparatus used in this study.

2.7.2 Compressive Strength of CPB

In CPB technology, UCS has become a widely accepted and important property for controlling the quality of CPB (Chen et al. 2019). However, the strength development of CPB is affected by several factors, such as progress of cement hydration process and intensity of cement self-desiccation (Ghirian and Fall 2016). Furthermore, the strength of CPB is significantly governed by the change in backfill temperature, type of hydraulic binder and binder blend ratio used in the CPB mix, and the presence of chemical additives and/or activators (such as sodium silicate) (Fall and Benzaazoua 2005, Fall et al. 2010, Nasir and Fall 2010, Cui and Fall 2016, Li and Fall 2018, Xue et al. 2018).

The UCS of CPB has been extensively discussed in the literature. For instance, Belem and Benzaazoua (2004) mentioned that the UCS of CPB to suitably perform as ground support of underground mines should be at least 5000 kPa.

Benzaazoua et al. (2004) used the UCS test as a tool to better understand the hardening processes of CPB, and they found that the strength development of CPB is effected by the tailings properties, water-to-binder ratio, binder type and content, and the chemistry of the water used in

the CPB mix. The result of this study demonstrated that the grain size distribution of tailings critically influences the hardening process, and thus the strength, of CPB. The increase in water content may affect the hardening process by effecting the precipitation reactions during the cementing phase. Also, different binders were found to give different strength development, and the presence of some chemical components (such as sulphate) may negatively affect the strength of CPB.

Kesimal et al. (2005) conducted the UCS tests on different CPB samples, and it was found that the short-term strength of CPB tends to increase with the decrease in water-to-cement ratio and the long term CPB strength.

Fall et al. (2010) discussed the relationship of UCS development of CPB with the curing temperature, binder type, and curing time. They noted that the UCS values increase with the progress of curing time, and the increase in curing temperature accelerates the cement hydration process and thereby increases the UCS values. They also found that using PCI-slag blends as a binder will yield higher UCS values as compared to PCI only, while the usage of PCI-FA blend will reduce these values.

Nasir and Fall (2010) developed a numerical model for predicting the UCS of undrained CPB. In their model, the strength development was coupled with temperature and degree of hydration, and they found that different mixes of CPB have different strength development with respect to different temperatures.

Abdul-Hussain (2011) used UCS as a tool to determine the right dosage of sodium silicate to prepare a suitable Gelfill (sodium silicate-activated CPB mixtures), and she found that 0.4% wt would be the best proportion of sodium silicate as it results in higher UCS values.

Yilmaz et al. (2011) assessed the UCS of consolidated and unconsolidated CPB materials. The UCS results showed that the consolidated backfills consistently show higher strength than the unconsolidated ones.

Yin et al. (2012) studied the effect of solid components on the mechanical properties of CPB, and found that the magnitude of UCS of CPB samples without slag is almost two times larger than the samples prepared with adding 25% of slag. It was also noted that unground slag would also diminish the mechanical properties of CPB, while the addition of sodium silicate would efficiently help achieve the desired initial strength performance of CPB.

The results of (Ghirian and Fall 2014) showed that the time-dependent increase in UCS of CPB is due to the combined effect of the progress of cement hydration and suction development. Also, the UCS results indicated that the CPB material in the long term (higher curing time) shows higher UCS values and more brittle behavior as compared to those at early age.

Wang et al. (2016) experimentally investigated the influence of initial temperature on the strength development and self-desiccation in cemented paste backfill that contains sodium silicate. In this study, the mechanical strength, particularly UCS, was tested for CPB that contains sodium silicate as the admixture at early age under the effect of various initial temperatures (2°C, 20°C, 35°C, and 50°C). Beside the effect of sodium silicate on activating the hydration of CPB material, this study found that the high initial temperature leads to more intense rapid self-desiccation in the CPB material. It was also found that the rate of strength development at early age of CPB material that contains sodium silicate is significantly enhanced at high initial temperature. However, the overly high initial temperature was found to reduce the compressive strength of CPB that contains sodium silicate after 7 days of curing age (Figure 2.12). Wu et al. (2016), on the other hand, investigated the effect of initial temperature on the deformation (stress-strain) behavior of CPB that contains sodium silicate. They also evaluated modulus of elasticity as a function of the UCS and the degree of hydration. They found that the high initial temperature-induced increase in UCS and hydration degree leads to an increase in the modulus of elasticity, which is not directly affected by the initial temperature.

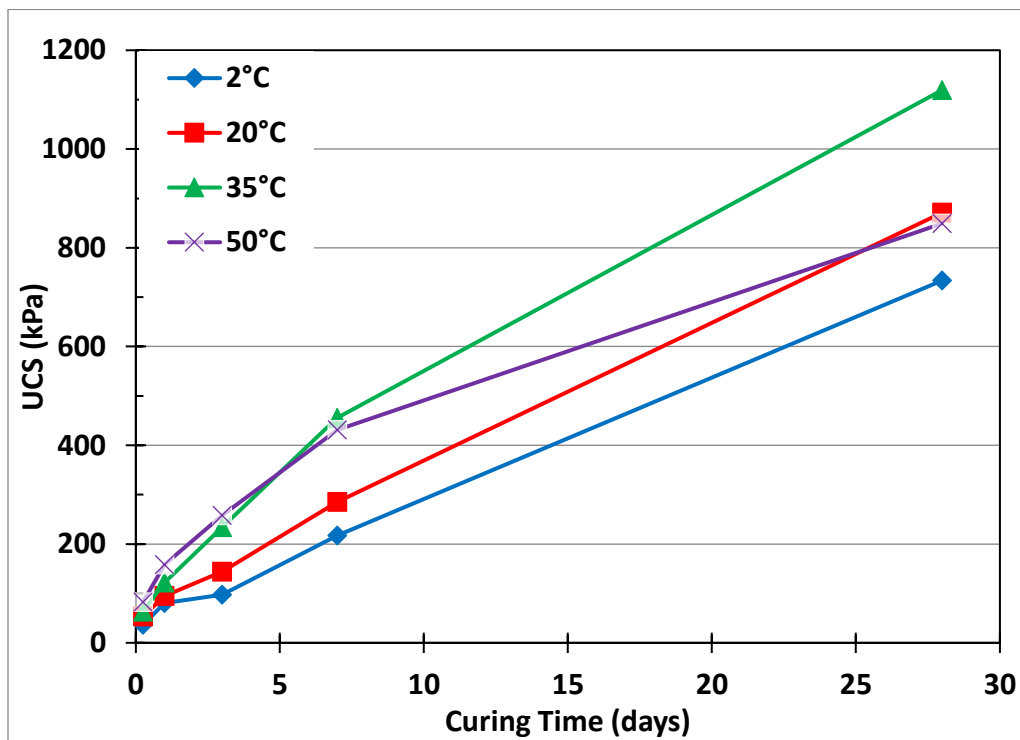


Figure 2.19 Effect of initial temperature on the strength development of CPB that contains sodium silicate at early age (modified from: Wang et al. 2016).

Cihangir et al. (2018) conducted UCS tests to determine the short- and long-term strength of CPB materials that are prepared with different binder and/or dosage of sodium silicate. The results showed that Portland cement-CPBs have higher strength at early age (<14 days) and lower strength at longer age (around 360 days) as compared to sodium silicate activated slag-CPBs.

Jiang et al. (2019) also studied the UCS of CPB that is prepared with different dosages of alkali-activated slag as binder, and they found that the increase in the binder dosage would yield significant increase in the UCS values.

Jafari (2020) conducted UCS test on CPB samples to investigate the change in UCS with curing time and cement content. The UCS results showed that the UCS values of CPB generally increase with the increase in curing time. However, the rate of increase is potentially higher at early ages and it gets lower with the passage of time, which was attributed to the fact that with the passage of time, most of the cement particles have been used to gain strength. It was also found that the UCS increases with the increase in cement content.

In the current study, the UCS of Slag-CPB (CPB that was prepared with PCI-Slag blend) was investigated under the combined effect of curing time, initial (mixing and curing) temperature, and different dosages in alkali activator (sodium silicate). To the best knowledge of the author, the approach of studying the coupled effect of these factors has yet not been addressed in the literature. Further details of the experimental program adopted in this research as well as the methodology, experimental conditions, and instrumentations are briefly described in the Chapter 4 of this dissertation.

2.8 Conclusions

Underground mining operations were found to cause potential geotechnical problems, such as ground subsidence, and produce significant amounts of anti-ecological wastes, such as tailings. Traditionally, these tailings are stocked as surface structures (e.g. tailing dams), but they were found to be exposed to more environmental challenges (such as acid mine drainage) and geotechnical issues (such as tailing dam failure), which can cause significant environmental and economic damages and lead to great amount of casualties in the mine area and the surrounding areas. Over the past few decades, cemented paste backfill (CPB) was found to be a successful modern method of tailing management around the world, as it allows around 60% of the hazardous mining waste to be returned into the underground mine opening and reused as geotechnical support for the underground mine openings (stope). CPB is a mixture of tailings and water (fresh and/or mine processing) that is undergoing cementation, as it contains different types of hydraulic binder (such as Portland cement, slag, and fly ash). CPB mixture is often prepared in paste plants located in the mine area, and the fresh CPB mixture is delivered through transportation pipeline system into the mine stope. So, it is important for the CPB mixture to have suitable flowability in order to avoid pipelines clogging, which might cause production delay and/or possible damages to the transportation system that leads to significant financial losses. In addition, CPB mixtures have to have suitable mechanical strength to achieve its desired performance.

From the technical background information, it was learnt that there are several sources of heat that can affect the temperature of CPB in the delivery pipelines when placed in the mine stopes. This heat potentially influences the rheological properties (and hence the flowability) of transported CPB as well as the mechanical behavior of CPB in the mine stope. Also, it was

understood that using different binder blends in the CPB mixtures can significantly change the rheological properties and the mechanical response of CPB. In addition, using chemical additives, such as sodium silicate, has potential influence on the behavior of CPB at early age.

From the reviewed studies, it can be observed that many researchers studied the rheological properties, potentially yield stress and viscosity, of CPB. These studies investigated the effect of initial temperature, binder content, or the usage of chemical additives on the rheological properties of CPB. However, these studies only investigated the individual or coupled effect of some of these conditions. Since no study till now (as per the knowledge of the author) has assessed the combined effect of all these conditions on the rheological properties of CPB, so there is a great need to assess the rheological properties of fresh CPB under the effect of initial temperature, binder type, and the presence and initial content of sodium silicate. Furthermore, similar to the rheological properties of the fresh CPB, the mechanical strength of CPB might also be affected by some parameters (such as the initial temperature and type and content of chemical additives.). There is, therefore, a great need to understand the combined effect of these parameters on the mechanical strength of fresh CPB in order to fill the gap of previous studies. Also, a better understanding will contribute to more safe and efficient design of CPB structures at early age.

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CHAPTER 3

Paper I: Time- and Temperature-Dependence of Rheological Properties of Cemented Tailings Backfill with Sodium Silicate

Paper I: Time- and Temperature-Dependence of Rheological Properties of Cemented Tailings Backfill with Sodium Silicate

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Ghada Ali, Mamadou Fall and Imad Alainachi

3.1 Abstract

Cemented paste backfilling is a novel tailings management method that is widely used in the mining industry to minimize the environmental and geotechnical risks associated with traditional mine waste management techniques, ensure safe work conditions in underground mines, and increase mine productivity. To enhance the early age strength of cement paste backfill (CPB), sodium silicate has been proposed and adopted as an admixture in CPB systems due to its activation ability. However, no studies have been done to gain insight into the effect of temperature on the rheological properties (yield stress and viscosity) of CPB with sodium silicate and different types of binders, although a CPB with sufficient flowability is critical for its successful application, and CPB is subjected to various temperatures in the field during its transport. Thus, this study aims to investigate the effect of temperature (2°C, 20°C, and 35°C) and time (0 to 4 hrs) on the rheological properties (yield stress and viscosity) of CPB samples with different amounts of sodium silicate (0, 0.1%, 0.3%, and 0.5%) and different binders. The results show that the yield stress and viscosity of CPB with sodium silicate increase with time and temperature. It is also found that the partial replacement of Portland cement with blast furnace slag or fly ash enhances the flowability (reduces the rheological properties) of CPB, regardless of the temperature. Also, an increase in the sodium silicate dosage will increase the yield stress and viscosity of CPB at any temperature. The findings of this research will contribute to a more efficient design of CPB transportation systems.

Keywords: Cemented paste backfill; Tailings; Cement; Rheology, Yield Stress, Viscosity, Sodium Silicate, Temperature

3.2 Introduction

Mining is an important industry around the world and has been making significant contributions to the economy in many regions and countries. In Ontario (Canada), for instance, mining is the source of around \$10 billion CAD of revenue every year (Dungan and Murphy 2012, Ontario Mining Association 2017). However, during ore rock extraction processes, an immense quantity of mine wastes (e.g., tailings, which are human-made soils generated by mining activities) are produced (Aydın and Kızıltepe 2019, Esmaili et al. 2020). Improper disposal of such wastes has negative impacts on the environment (Dimitrova and Yanful 2011, Cihangir et al. 2012, Farkish and Fall 2013, Ahmari et al. 2015, Alainachi and Fall 2019a, Chen et al. 2019). To manage tailings in a more environmentally friendly and safer way, a novel technology of tailings disposal has been developed by mixing tailings with water and binders. This disposal technology is called cemented

paste backfilling (Fall et al. 2005, Tariq and Nehdi 2007, Abdul-Hussain and Fall 2012, Niroshan et al. 2017, Cihangir et al. 2018). Recently, this technology has been extensively used in underground mining operations because the process allows for a large volume of tailings to be reused and returned to the mine stopes, and thus, reduce the volume of such wastes that need to be managed on the ground surface. Also, cemented paste backfill (CPB) can be produced and delivered to the mine stopes in a short period of time. This facilitates the completion of the stope backfilling process (stope cycles) in a matter of only days, while weeks or months might be required with older backfilling methods. Reducing the stope cycle time allows for additional revenue to be generated for the mine (Fall and Benzaazoua 2005, Fall et al. 2005, Kesimal et al. 2005, Ercikdi et al. 2009a, Ercikdi et al. 2009b, Thompson et al. 2009, Nasir and Fall 2010, Yin et al. 2012, Wu et al. 2013, Haiqiang et al. 2016). In addition, using CPB can increase the stability of the underground mine openings or rock mass which, otherwise, might be susceptible to several geotechnical problems, such as ground subsidence, rockburst and rock mass failure (Kesimal et al. 2005). The aforementioned stability increase due to CPB is mainly a function of the mechanical strength performance of CPB. The later can be significantly affected by several conditions or factors, such as static and dynamic loading conditions, potential chemical interaction of the ingredients within the CPB materials and curing temperatures (Cao et al. 2018, Xue et al. 2018, Alainachi and Fall 2019a).

Typically, CPB mixtures consist of around 70% - 85% tailings, fresh or mine processed water, and often 2% - 9% (by total weight of solid) of hydraulic binder (usually cement), based on the primary and tertiary requirements of the mine stope (Alakangas et al. 2013, Yilmaz et al. 2013, Aldhafeeri and Fall 2016, Haiqiang et al. 2016, Koohestani et al. 2018). The CPB is mixed and prepared in a paste backfilling plant usually located on the mine surface and then delivered into the mine stopes through pipelines either through gravity and/or pumping. Therefore, the flowability of CPB is a very important factor in transporting CPB to the mine stopes. If the transported material does not have suitable flowability, clogging of the pipelines might occur which will lead to significant financial losses due to the possible flow delays and/or temporary interruption of the progress of CPB production as well as possible replacement of the pipelines (Li and Fall 2016, Haruna and Fall 2019).

It is a well known fact that the flowability of cemented slurries depends on their rheological properties (Bannister 1980). Hence, to ensure the better flowability of fresh CPB in the pipelines, it is important to study the rheological properties (such as yield stress and viscosity) of the CPB material.

Yield stress and viscosity are the most important rheological parameters of paste flow through pipelines. Yield stress represents the minimum shear stress that is required to make the material flowable. In order to flow in the pipeline transport systems, the yield stress of CPB has to be within its flowable range, which is usually between 200 and 700 Pa (Potvin et al. 2005, Yilmaz and Fall 2017). The viscosity, on the other hand, is defined as the ratio between the shear stress and the shear rate at a specific shear rate. The viscosity of CPB is a measure of the resistance of

the CPB material towards the flow induced by the shear stress (Yilmaz and Fall 2017, Bian et al. 2019).

Past studies have shown that regardless of the method of delivery in CPB transport systems, it is evident that CPB material transported from the above ground paste plant to the underground mine stopes is subjected to several different temperatures from various sources (Figure 3.1) which might significantly affect its flowability. These sources include: (i) the temperature generated from the friction between the interior sidewall of the pipeline and the fresh CPB (Fall et al. 2010); (ii) the heat generated from the rock mass that is surrounding the pipeline pathway and the mine stopes, especially in deep mines. The thermal interaction between the CPB within the pipelines and the temperature of these rocks will increase the temperature of the transported CPB (Wang et al. 2016); (iii) the geographic location of the mine itself, which greatly affects the temperature of the transported CPB, especially in the extremely hot regions and/or the permafrost regions (Wang et al. 2016, Alainachi and Fall 2019b); and (iv) the internal heat generated due to the cement hydration of the fresh CPB, which might change the temperature of the CPB in the field. Indeed, the hydration process and thermal increase start immediately after the CPB components are mixed (Aldhafeeri et al. 2016, Alainachi and Fall 2019b). This heat will affect the flowability of the CPB in the pipelines, especially if the CPB is placed into a stope that is in a deep mine as the transport distance (in some cases) can be extremely long.

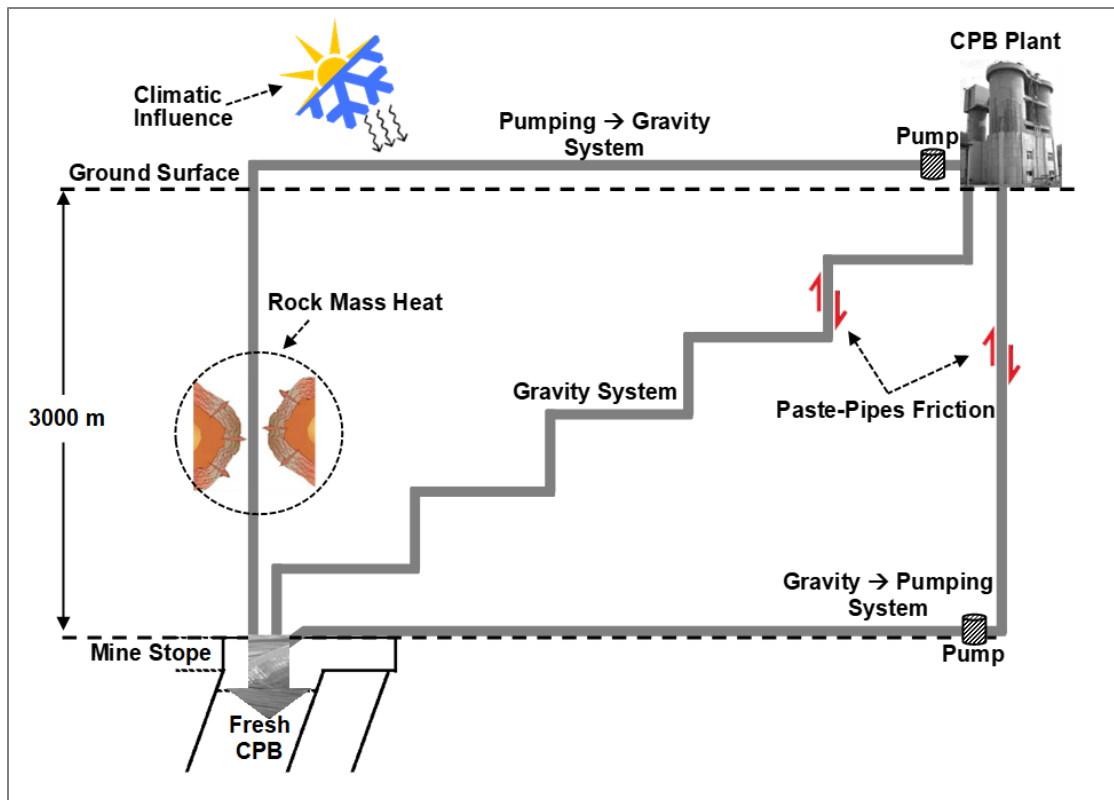


Figure 3.1 CPB transport system and sources of temperature change.

Other than the temperature, there are other external and internal factors that might also affect the rheological characteristics of fresh CPB. However, most of the previous studies (e.g. Wang et al. 2004, Huynh et al. 2006, Yin et al. 2012, Wang et al. 2018) have studied the rheology of CPB under the effect of an individual factor (e.g. binder content, type of binder, and pH value).

Some other studies have been conducted recently to understand the coupled effect of time and temperature on the rheological properties of different mixtures of CPB. For instance, Wu et al. (2013) developed a mathematical model to predict and assess the changes in the rheological properties of CPB under the coupled effect of curing time and temperature. The simulation showed that the temperature and progression of cement hydration (curing time) considerably influence the rheological behavior of CPB. In line with the work in (Wu et al. 2013), Haiqiang et al. (2016) investigated the combined effect of time and sub-zero temperatures on the yield stress of CPB. Their study found that the yield stress of CPB samples studied in sub-zero temperatures is much lower than that of the sample tested in room temperature. Jiang and Fall (2017), on the other hand, investigated the effect of the salinity of CPB on the time-temperature coupled behavior of CPB. It was found that an increase in salinity reduces the yield stress of CPB in subzero temperatures. Moreover, Haruna and Fall (2019) added different dosages of superplasticizer to a CPB mixture to determine the temperature-time induced changes in the rheological properties of CPB. Their results are in agreement with those of previous studies in that an increase in time and temperature will jointly increase the yield stress and viscosity of CPB. Also, they found that adding superplasticizer will reduce the yield stress and viscosity of CPB in high temperatures. Cheng et al. (2020) further analyzed the effect of time and temperature on CPB rheology by establishing a calculation model implemented with numerical software, and established a 3D simulation model to determine the effect of the velocity distribution of CPB with time and temperature. They concluded that the rheological behavior of CPB has a time-temperature equivalent effect. It was also noted that, along with the decrease in yield stress and viscosity, the flow velocity also decreases with an increase in time and temperature.

On the other hand, some studies (e.g. Brough and Atkinson 2002, Chang 2003, Abdul-Hussain and Fall 2012, Kermani et al. 2015a, Kermani et al. 2015b, Cihangir et al. 2018, Jiang et al. 2019, Kou et al. 2020) have used sodium silicate as a chemical additive in different CPB mixtures. They used sodium silicate as an activator in different types of CPB binders (such as Portland cement type I (PCI), blast furnace slag (Slag) and Fly ash (FA)), and also examined the effect of sodium silicate on the mechanical strength of CPB in the long term. However, no study to date has investigated the effect of sodium silicate on the rheological properties of CPB at the very early ages (up to 4 hours). Also, no study has evaluated the combined effects of time, temperature, binder content, and sodium silicate content on the rheological properties of CPB. Accordingly, the goal of this study is to experimentally examine the combined effects of temperature (mixing and curing), type of binder (PCI, Slag, and FA), chemical additive (sodium silicate), and progression of cement hydration (curing time) on the rheological properties of fresh CPB mixtures.

3.3 Materials and Experimental Program

3.3.1 Materials

3.3.1.1 Tailings

The tailings material used in this study is ground silica tailings (ST) which are a synthetic tailings material (manufactured by U.S. Silica Co.). The grain size distribution of the ST is similar to the average grain size distribution of the mine tailings extracted from nine different mines (9MT) in eastern Canada (Figure 3.2). ST are used in this study because they comprise mostly quartz, which is the predominant mineral in Canadian hard rock mine tailings. Also, ST have a high percentage of silica (SiO₂; 99.8%) which makes them a chemically inter material. The use of ST will allow the mineralogical and chemical compositions of the tailings to be controlled and the uncertainties related to the use of natural tailings to be minimized (Aldhafeeri and Fall 2016, Alainachi and Fall 2019a). The primary physical properties and main chemical elements of the ST are listed in Tables 3.1 and 3.2.

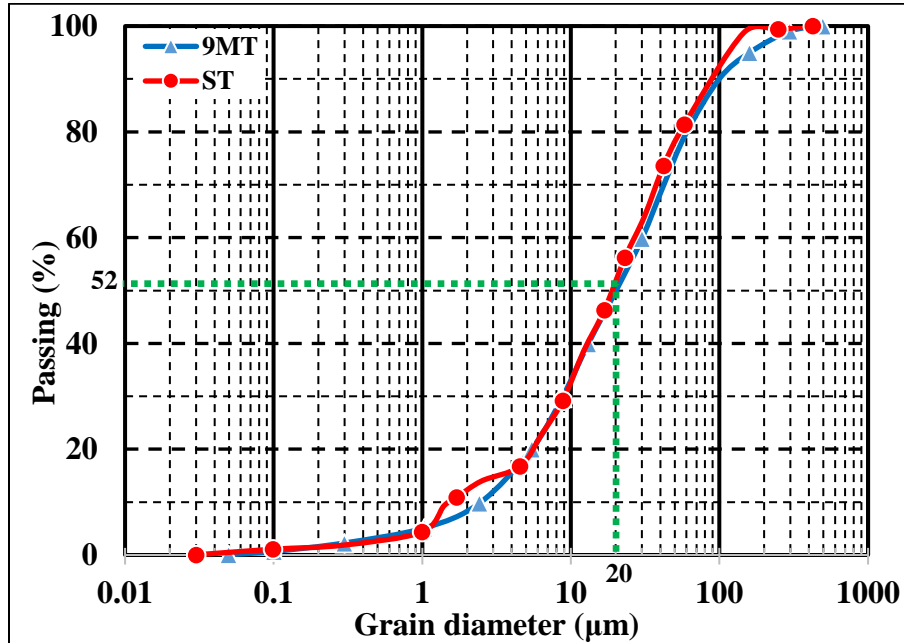


Figure 3.2 Grain size distribution of silica tailings used in this study vs. average grain size distribution of tailings from nine Canadian mines.

Table 3.1. Primary physical properties of tailings used in this study

Element	Gs	D ₁₀ (µm)	D ₃₀ (µm)	D ₅₀ (µm)	D ₆₀ (µm)
ST	2.7	1.9	9.0	22.5	31.5

Table 3.2. Main chemical elements of tailings used in this study

Element	Al wt. %	Ca wt. %	Si wt. %	Fe wt. %	Na wt. %	Pb wt. %	S wt. %	K wt. %
ST	0.10	<0.01	99.80	<0.01	<0.01	0.00	0.00	0.00

3.3.1.2 Water and binders

To enhance the mechanical properties of CPB, a sufficient amount of binder is often used in the mixture. In this study, the binder agent used in the preparation of the CPB mixtures is Portland cement type I (PCI), Fly Ash (FA), and blast furnace slag (Slag). These binders are the most frequently used in preparing CPB worldwide (Haiqiang et al. 2016). The properties of these binders are listed in Table 3.3. PCI is used alone or blended with Slag or FA. The blending ratio of PCI and Slag or FA is 50/50. This ratio is selected based on: (i) the need of the industrial partner in this project; (ii) cost savings; (iii) previous studies on performance of this ratio in CPB systems (e.g., Fall et al. 2010). Tap water is used in this study to mix the binders with the tailings.

Table 3.3. Primary physical and chemical properties of binders used in this study

Binder	SSA* (m ² /g)	R.D.**	S (wt%)	Ca (wt%)	Si (wt%)	Al (wt%)	Mg (wt%)	Fe (wt%)
PCI	1.32	3.2	1.5	44.9	8.4	2.4	1.6	1.9
FA	2.20	2.6	1.3	13.3	15.2	9.2	2.6	4.1
Slag	2.10	2.8	12	26.6	18.9	3.9	6.9	0.5

* Specific surface area and ** Relative density

3.3.1.3 Sodium silicate

Soluble sodium silicates are silicate polymers which are an inorganic chemical, and a clear, colourless, and viscous liquid. Sodium silicate are also known as water soluble glasses which are generally produced from varying proportions of an alkali metal and SiO₂. Sodium silicates have many applications; for instance, they are used in the geotechnical engineering field in soil grout and mine tailings backfill, and as an admixture for cement products. In this study, the primary reason for using sodium silicates is to evaluate their effect on the rheological properties of CPB. In this regard, a commercial solution of sodium silicate (Type N) was added in the liquid form to the CPB mixture as the admixture for the binders. The properties of the sodium silicate used in this study are listed in Table 3.4.

Table 3.4. Properties of sodium silicate (National Silicate Ltd.)

Property	Value
Na ₂ O% by weight	8.90
SiO ₂ % by weight	28.66
Weight ratio, %SiO ₂ /%Na ₂ O	3.22
Specific gravity @ 20°C	1.39
Solids %	37.56

3.3.2 Sample Preparation

The CPB ingredients (tailings, water, and binder) were first stored in a temperature controlled chamber (refrigerator or oven, as the case may be) for a minimum of 24 hours or until they reached the desired temperature (2°C, 20°C, and 35°C). Then, 125 CPB samples were prepared by mixing a given amount of tailings, binder (4.5 wt%), and water (w/c ratio =7.8) at the testing temperature (2°C, 20°C, and 35°C). During the mixing of these ingredients, sodium silicate (SS) was added to each mixture (each sample) with different concentrations (0, 0.1, 0.3, and 0.5% by weight solid components; solid components = tailings + binder; %SS = (mass of SS/mass of Solids)x100). The sodium silicate gel used in these mixtures contains 62.44% of water. This water content was considered when determining the amount of mixing water required for the preparation of the CPB samples. The samples were mixed for 7 min to obtain a homogenous mixture (Bian et al. 2019). Then, the prepared CPB mixtures were poured into a 600 ml low-form Griffin beaker and vibrated to remove the entrapped air. They were subsequently poured into plastic cylindrical containers (10 cm in diameter × 10 cm in height). To prevent the evaporation of water, each sample was covered with plastic film. They were then placed aside for curing at the same mixing temperatures. To reflect the range of temperature that the CPB might be subjected to during its transport through the pipeline system, the CPB samples were prepared, cured and tested at 2°C, 20°C, and 35°C. Moreover, to reflect the different transportation times in backfill practice from the CPB plant to the underground stope, the prepared CPB was cured for 0, 0.25, 1, 2, and 4 h before being tested. Selecting a curing time up to 4 hrs enables to consider the long transportation time that can be required in deep or ultra depth mines. To ensure that the CPB remained homogeneous, the samples were agitated for 1 min before each test. This activity also experimentally models the shearing process of the backfill during transport and prevents the self-weight settlement of the soil particles during curing (Haiqiang et al. 2016). Moreover, some of the samples were used for microstructural analyses or monitoring tests (pH and zeta potential, and electrical conductivity (EC)). Table 3.5 summarizes the experimental program and the mix proportion of CPB for each stage of the program.

Table 3.5. Summary of CPB mix proportion and stages of experimental program

Sample Label	Binder Content (%)	%PCI in the binder	%Slag in the binder	%FA in the binder	W/C ratio	SS content (%)	Mix/Curing Temp. (°C)	Curing Time (h)
A. Effect of temperature on the time-dependent changes in rheological properties of CPB								
SS-CPB-2°C	4.5	100	0	0	7.8	0.3	2	0, 0.25, 1, 2, 4
SS-CPB-20°C	4.5	100	0	0	7.8	0.3	20	0, 0.25, 1, 2, 4
SS-CPB-35°C	4.5	100	0	0	7.8	0.3	35	0, 0.25, 1, 2, 4
B. Effect of binder content and curing temperature on rheological properties of CPB								
PCI-SS-CPB	4.5	100	0	0	7.8	0.3	2, 20, 35	2
PCI/Slag-SS-CPB	4.5	50	50	0	7.8	0.3	2, 20, 35	2
PCI/FA-SS-CPB	4.5	50	0	50	7.8	0.3	2, 20, 35	2
C. Effect of sodium silicate content and curing temperature on rheological properties of CPB								
0.0-SS-CPB	4.5	100	0	0	7.8	0.0	2, 20, 35	2
0.1-SS-CPB	4.5	100	0	0	7.8	0.1	2, 20, 35	2
0.3-SS-CPB	4.5	100	0	0	7.8	0.3	2, 20, 35	2
0.5-SS-CPB	4.5	100	0	0	7.8	0.5	2, 20, 35	2

PCI: Portland cement I, FA: Fly ash, SS: Sodium silicate.

3.3.3 Test Methods

3.3.3.1 Viscosity test

A Brookfield digital viscometer (DV-E model) was used to determine the viscosity of the CPB samples under the conditions provided above. This viscometer consists of a rotating spindle connected to a calibrated spring. To measure the viscosity, the spindle is immersed into the fluid to be tested and then rotates in the liquid. The liquid will then resist the rotation which is known as viscous drag, and determined by the spring deflection. The spring deflection is measured by the rotary transducer (Brookfield 2014). In this study, an RV5 spindle is used and a specific speed is applied to ensure that the readings are between 10 and 100% of scale. The rotation speed of the rotor is changed until obtaining an acceptable viscosity reading, as described in (Xiapeng et al. 2019)). The rotation speeds, 30 revolution per seconds, is used. Before testing, the guard leg was mounted onto the viscometer to ensure the stability of the sample during testing. In order to prevent the trapping of air, the spindle was gently inserted into the center of the CPB samples to be tested in their titled position. Afterwards, the spindle was attached to the spindle coupling nut of the

viscometer, which was subsequently turned on and allowed to run for a specific length of time until reaching a relatively constant reading. After reaching stabilization, the test was carried out on each CPB sample (according to the aforesaid experimental program). To ensure the repeatability and accuracy of the obtained results, each test was repeated at least twice. Further details on the procedure of viscosity measurements of CPB using this viscometer are given in Brookfield (2014).

3.3.3.2 Yield stress test

Vane shear testing is considered as one of the most efficient, direct and accurate single-point methods for measuring the yield stress of soil and non-Newtonian fluids (Bauer et al. 2007, Abd Elaty and Ghazy 2012, Kondraivendhan and Bhattacharjee 2015). In this study, a Wykeham-Farrance vane shear is used to determine the yield stress of the CPB samples. The vane shear consists of an electrical motor that rotates the four-bladed vane with a diameter of 2.5 cm and height of 2.5 cm which is connected to the deflection spring, and a dial gage to measure the resistance of the sample to the applied torque. Before testing, the vane was slowly inserted into the middle of the containers (10 cm in diameter \times 10 cm in height) of CPB samples to be tested. A waiting period of 30 seconds was provided to allow the material to stabilize. Afterwards, the motor was turned on to rotate the vanes at a constant rate of 0.18 rpm until the maximum torque was reached. The test was carried out in accordance with ASTM D4648/D4648M-16 (2015) and the readings obtained from the dial gage were subsequently calculated into the corresponding yield stress values. Each test was repeated at least twice to ensure the repeatability and accuracy of the obtained results.

3.3.3.3 Microstructural analysis

To understand the effect of the microstructural changes in the CPB on its flowability when subjected to the different testing conditions, several microstructural analyses were conducted on selected CPB samples at different curing times. The microstructural analyses were carried out by using X-ray diffraction (XRD) to determine the changes in the mineralogical composition of the different CPB samples, and thermal analyses (differential thermogravimetry (DTG) and thermal gravimetry (TG)) to monitor the changes in the weight, heat flow, and temperature changes in each CPB sample. Before conducting these analyses, the samples were first dried at 45°C in a vacuum oven until mass stabilization. The XRD process was conducted with a Scintag XDS2000 x-ray diffractometer. The thermal analyses, in the other hand, were undertaken with a TGA Q5000 thermal analyzer (TA Instruments). The various (dried) samples (about 20 mg each) were heated in an inert nitrogen atmosphere at the rate of 10°C per minute up to a temperature of 1000°C.

3.3.3.4 pH and zeta potential measurements

Zeta potential is often determined by measuring the electrophoretic mobility of suspended particles in samples and applying the Henry equation on the measured values (Clogston and Patri 2011). In this study, a Zetasizer Nano series was used to determine the difference in the zeta potential of two CPB samples that were prepared and cured at 2°C, and 35°C. Both samples were prepared by using

PCI as the binder, consisted of 0.3% of sodium silicate and cured for 4 h. All specimens of this test were prepared with 0.1g/L solid to water ratio, and the basic water of these suspensions was distilled water (Jiang and Fall 2017). As with all the testing, each test was repeated at least three times to ensure accuracy and repeatability.

On the other hand, measuring the pH of the different samples provides an idea of the changes in the pore solution volume and alkalinity of the tested samples in line with the changes of the testing conditions (Ghirian and Fall 2014). The pH changes were determined by again using CPB samples with PCI (PCI-CPB) as the binder and 0.3% of sodium silicate. They were prepared and cured at 2°C, 20°C, and 35°C for 0.25 hr (15 min), 1 h, 2 h, and 4 h. One of the samples was also not cured for any amount of time. The variations in the pH of the tested CPB samples show the time-temperature effect on the volume of the pore solution of SS-CPB. An Oakton pH Tester 10 Waterproof BNC Pocket pH Tester with a pH range of -1.00 to 15.00 and accuracy of ± 0.01 pH was used in this regard.

3.3.3.5 Electrical conductivity monitoring of samples

The changes in the electrical conductivity (EC) reflect the rate of ion movement from the chemical reactions that take place between cement and water. Monitoring the EC is an effective way to assess the progression of cement hydration and the related structural changes (Li and Fall 2016). This part of the experimental program aims to monitor the changes in the EC under the combined effects of type of binder and sodium silicate content. Accordingly, two sets of EC tests were conducted. To determine the effect of the binder content, the EC was monitored for three samples with different binder contents as follows: (i) 100% PCI, (ii) 50% PCI and 50% Slag, and (iii) 50% PCI and 50% FA. All three samples consisted of 0.3% of sodium silicate, and their initial and curing temperatures were both 35°C. On the other hand, to determine the effect of the sodium silicate content, the EC was monitored on two PCI-CPB samples and consisted of: (i) no sodium silicate content, and (ii) 0.3% sodium silicate. The initial and curing temperatures of both samples were 35°C. The EC monitoring of all of the samples was carried out while the samples were cured for 48 h. An ECH2O 5TE sensor that measures the EC in the range of 0-23 dS/m with an accuracy of ± 0.1 was used in this regard.

3.4 Results and Discussion

3.4.1 Effect of temperature on time-dependent changes of rheological properties of CPB

The effect of temperature on the changes in the yield stress and viscosity of the PCI-CPB samples with 0.3% sodium silicate as a function of time is illustrated in Figures 3.3 and 3.4, respectively.

As expected, for a given temperature, there is a gradual enhancement of the rheological properties with time (yield stress and viscosity). This is related to the growth of more cement hydration products, and process of reduction of free water that occurred in the CPB material due to self-desiccation with time (Bullard et al. 2011, Haiqiang et al. 2016, Bian et al. 2019). This behavior is consistent with the findings of previous studies (e.g. Roussel 2007, Simon and

Grabinsky 2013, Wu et al. 2013, Haiqiang et al. 2016, Panchal et al. 2018, Haruna and Fall 2019, Xiapeng et al. 2019) which indicated that the rheological properties of fresh CPB or other cementitious materials (e.g., fresh concrete) can significantly increase in the initial stage of cement hydration (30 minutes or less) and beyond, as a result of the progress of cement hydration. It is also evident from results of Figures 3.3 and 3.4 that, for a given curing time, the initial and curing temperatures have a direct relationship with both the yield stress and viscosity. Samples which are subjected to a temperature of 2°C have the lowest yield stress and viscosity, while those subjected to a temperature of 35°C have the highest. This phenomenon is attributed to the acceleration of the cement hydration process under high temperatures (Aldhafeeri et al. 2016, Cui and Fall 2016). This acceleration of the cement hydration due to high temperatures will result in the formation of more cement hydration products (such as calcium silicate hydrate (C-S-H), ettringite, and calcium hydroxide (CH)) as well as increase the self-desiccation of the backfill material (Fall et al., 2010). This is beneficial for yield stress increases of the CPB because more of these products will result in increased cohesion between the tailings particles (the free CPB particles are progressively bonded to the hydration products to form a 3D network structure, or a gel structure, which leads to increased cohesion between particles) as well as reduce the size of the pores between the CPB particles, which increases the yield stress (Wang et al. 2016, Alainachi and Fall 2019b). Furthermore, the consumption of capillary water by the cement hydration (self-desiccation) will lead to the reduction of the space between the CPB particles and increased particle inter-friction (Wang et al., 2016). Accordingly, the interaction and the cementitious bond between the particles rise, which in turn, leads to higher yield stress values. Moreover, this temperature-induced formation of more hydration products and higher consumption of water molecules in the capillary pores mean that the solid volume fraction of the backfill material will increase and the CPB will become more viscous (Xiapeng et al. 2019), which is obviously associated with an increase of the viscosity of the CPB. This rationale is supported by the results of the thermal analysis in this study, and Figure 3.5 provides the TG/DTG diagrams for two PCI-CPB samples (with 0.3% of sodium silicate) prepared and cured (up to 4 h) at 20°C and 35°C. These diagrams confirm that more hydration products are produced within the CPB at higher initial and curing temperatures. The first two peaks (sudden change in weight) show a higher weight loss within the CPB prepared and cured at 35°C. The first peak is due to the destruction of the C-S-H, ettringite, and gypsum (e.g. Taylor 1990, Fall et al. 2010), while the second peak is associated with the degradation of CH (e.g. Aldhafeeri et al. 2016). The third peak represents the decomposition of the calcite in the cement (Taylor 1990, Mehta and Monteiro 2001), and is lower for the CPB prepared at 35°C compared to the CPB prepared at 20°C.

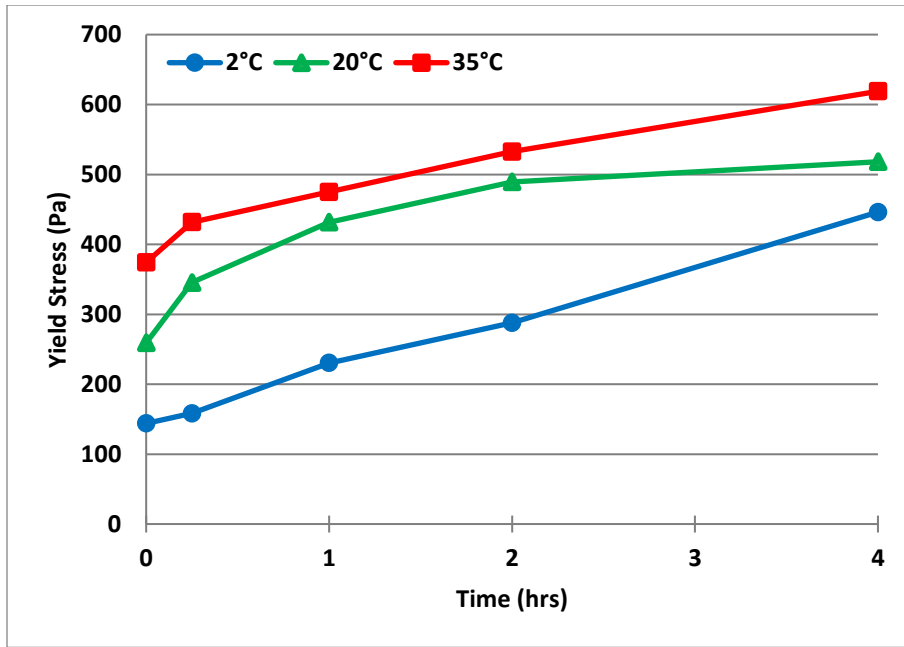


Figure 3.3 Temperature vs. yield stress of PCI-CPB samples with 0.3% sodium silicate.

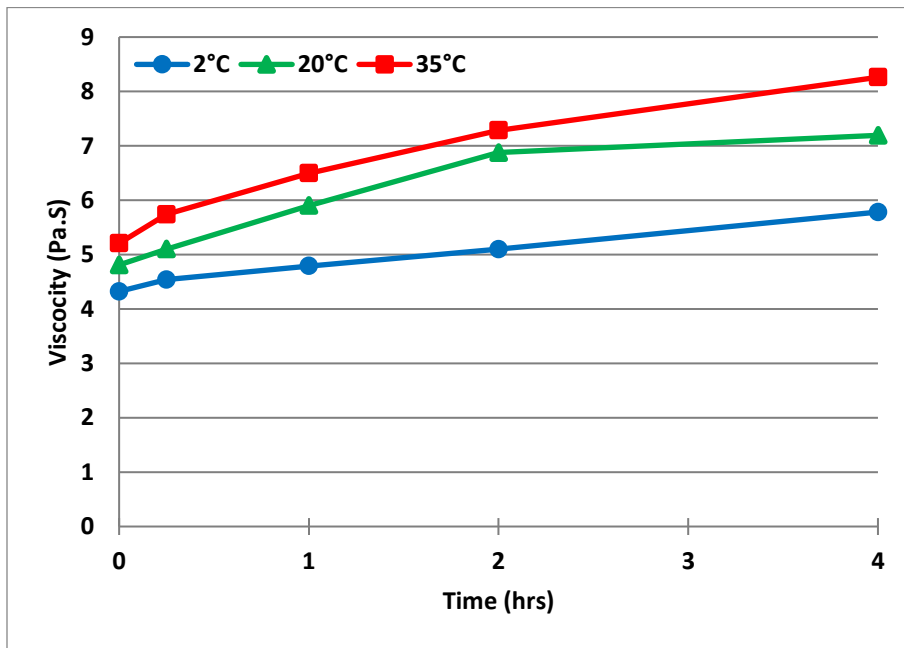


Figure 3.4 Temperature vs. viscosity of PCI-CPB samples with 0.3% sodium silicate.

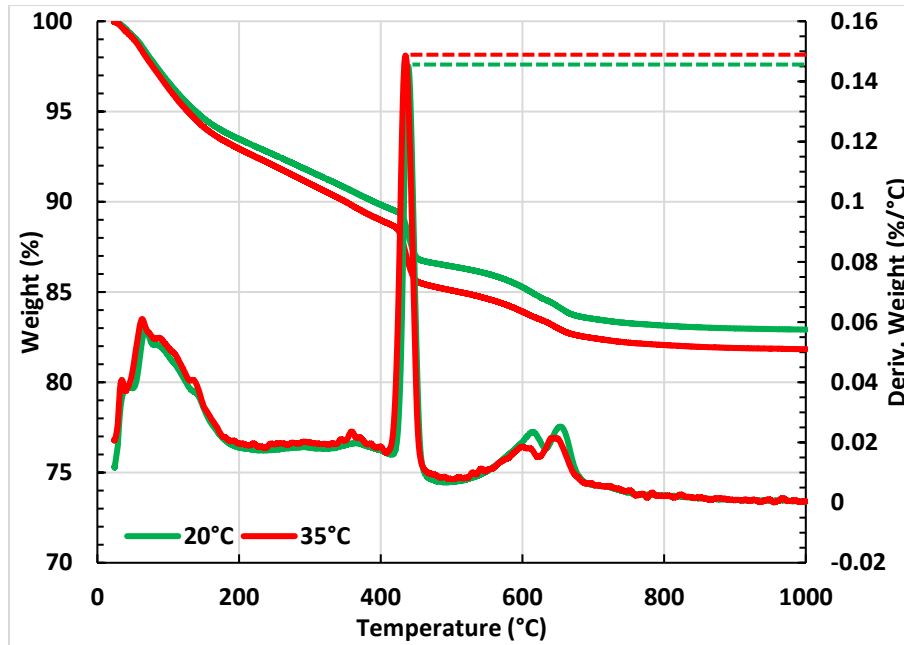


Figure 3.5 DT/DTG diagrams for PCI-CPB samples with 0.3% sodium silicate cured for 4 h vs. different curing temperatures.

The increase in the viscosity of the backfill material at higher temperatures is consistent with the results of the zeta potential measurements of the PCI-CPB samples (0.3% of sodium silicate) cured up to 4 h at 2°C and 35°C, as shown in Figure 3.6. The CPB samples prepared and cured at a temperature of 2°C has the highest absolute zeta potential value compared to those prepared and cured at a temperature of 35°C. This higher zeta potential value at lower curing temperatures suggest that stronger repulsive forces are found between the particles in CPB subjected to lower temperatures, since the zeta potential is the electrostatic surface charge or magnitude of the electric repulsion or attraction between colloidal particles (Elakneswaran et al. 2009, Jiang and Fall 2017). In other words, the particle dispersion within the CPB is higher in low temperatures, and thus results in better flowability (Huynh et al. 2006, Haruna and Fall 2019). These zeta potential measurement results and the resultant conclusion made are in good agreement with the results of the pH measurements that were conducted on the CPB samples at different temperatures and shown in Figure 3.7. The pH values are higher for the CPB samples that are prepared and cured at higher temperatures compared to those at lower temperatures. This is related to the fact that higher temperatures increase cement hydration, and thereby leads to higher electrolyte concentration and reduced water content in the CPB system. It is well acknowledged that increasing the electrolyte concentration or decreasing the water content reduces the thickness of the diffuse double layers (DDLs) (Mojid 2011), and results in an increase in the pH of the solution which compresses the diffuse layer due to the well-known concentration effect (Nagele 1986).

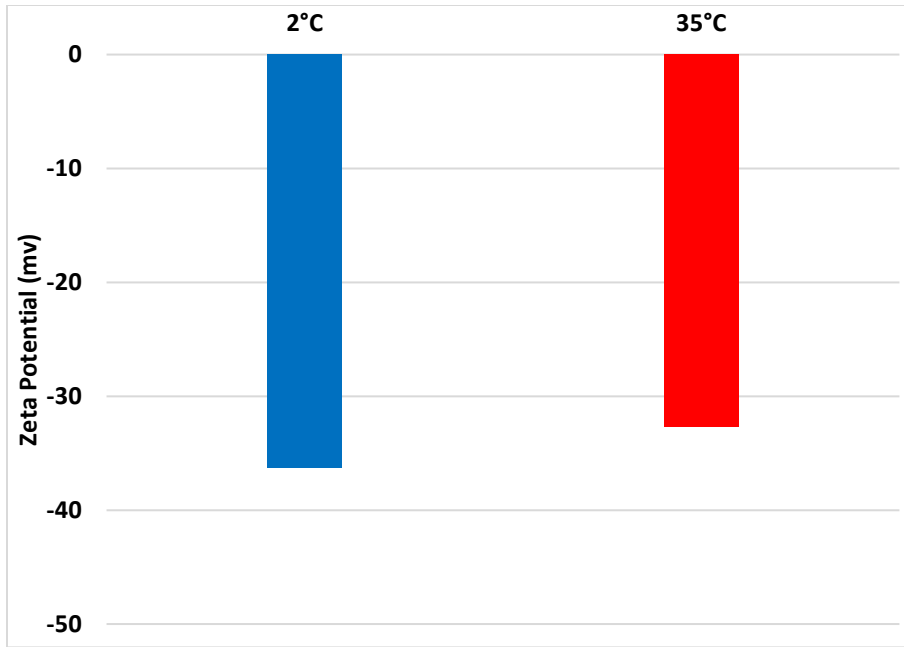


Figure 3.6 Zeta potential of PCI-CPB samples with 0.3% of sodium silicate prepared and cured at different temperatures for 4 h.

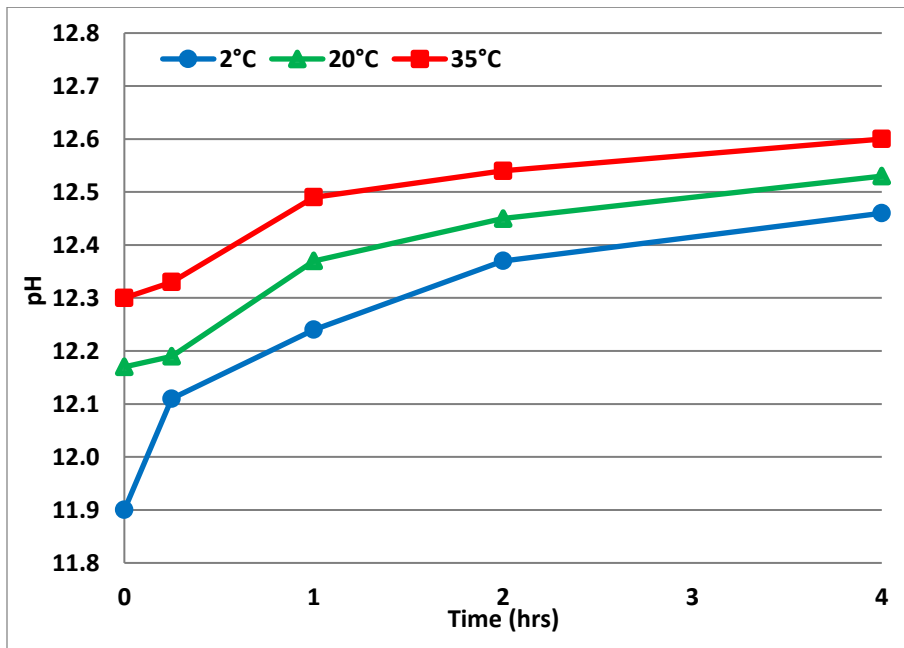


Figure 3.7 Changes in pH of PCI-CPB samples with 0.3% sodium silicate prepared and cured at different temperatures for 4 h.

3.4.2 Combined effect of type of binder and curing temperature on rheological properties of CPB

Pozzolanic materials, such as Slag and FA, are often used in paste backfill mixes as a partial replacement for Portland cement to reduce the cost due to cement consumption, while maintaining the desired strength (Cui 2017). There is also consensus in the literature that pozzolans can be effectively activated by adding alkali activators, such as sodium silicate (Kermani et al. 2015a, Azadi et al. 2017). During the early hydration process of cement-slag blends, the hydration of the clinker releases alkali and increases the temperature. This provides the energy to activate alkali-hydroxide attacks on the Slag particles, which activates the Slag and accelerates its hydration process (Roy and Idorn 1982, Kolani et al. 2012). Several research studies (e.g. Qing-Hua et al. 1992, Brough and Atkinson 2002) have suggested that CH reacts with sodium silicate and leads to the consumption of its silica components which leaves the sodium oxide (Na_2O) relatively free around the Slag particles, thus activating/accelerating the Slag hydration and producing higher amounts of C-S-H. In other words, the reaction between CH and sodium silicate increases the concentration of alkali ions around the Slag particles and produces a high pH (e.g. Qing-Hua et al. 1992), which is indicative of the start of the Slag hydration and strength build-up of the mixture (e.g. Qing-Hua et al. 1992, Brough and Atkinson 2002). Cement-FA blends, on the other hand, have an initial reaction that is similar to the cement-Slag blends. However, the exothermic process between sodium silicate and the FA activates the alkali reaction of the FA, during which the FA structure is destroyed. This results in the progressive accumulation of reaction products that are produced during the destruction of the FA structure. Subsequently, condensation occurs on the surface of the FA particles, which contributes to creating a cementitious material with high mechanical strength (Palomo et al. 1999). In other words, the presence of sodium silicate increases the alkali reaction rate, and consequently accelerates the development of the mechanical strength of the mixture with the blended binders (Palomo et al. 1999, Cihangir et al. 2012). Moreover, it is widely accepted that compounds of alkali metals (sodium and potassium) stimulate the hydration of the key phases of Portland cement, at least in the early stages of the hydration process, and accelerate the setting of cement paste (e.g. Jawed and Skalny 1978, Taylor 1990). For example, Brykov et al. (2002) indicated that the hydration of Portland cement clinker minerals happens at a higher rate in the presence of sodium silicates because of a reduction in the initial pH of the pore solutions of cement pastes and intensive production of CH.

Previous studies (e.g. Simon and Grabinsky 2013, Haiqiang et al. 2016, Jiang and Fall 2017) have observed that addition of Slag or FA to the binder of CPB will significantly affect the rheological properties of CPB. However, no studies have investigated the rheological properties of CPB samples with Slag or FA activated with sodium silicate. This knowledge gap is addressed in the following section.

In this part of the study, the effect of type of binder on the rheological properties of CPB samples with sodium silicate is assessed by determining the yield stress and viscosity of samples cured for 2 h with different types of binders: (i) 100% PCI, (ii) 50% PCI and 50% Slag, and (iii)

50% PCI and 50% FA. All of the samples contain 0.3% of sodium silicate. The effects of the type of binder on the yield stress and viscosity with respect to the mixing and curing temperatures are shown in Figures 3.8 and 3.9, respectively.

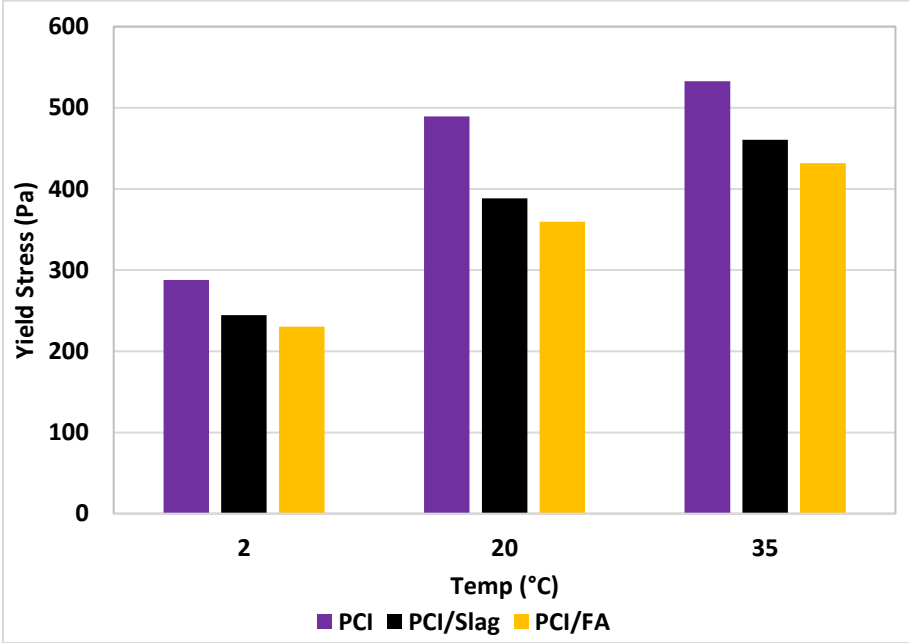


Figure 3.8 Effect of temperature and binder type on yield stress of CPB samples with 0.3% sodium silicate cured for 2 hrs.

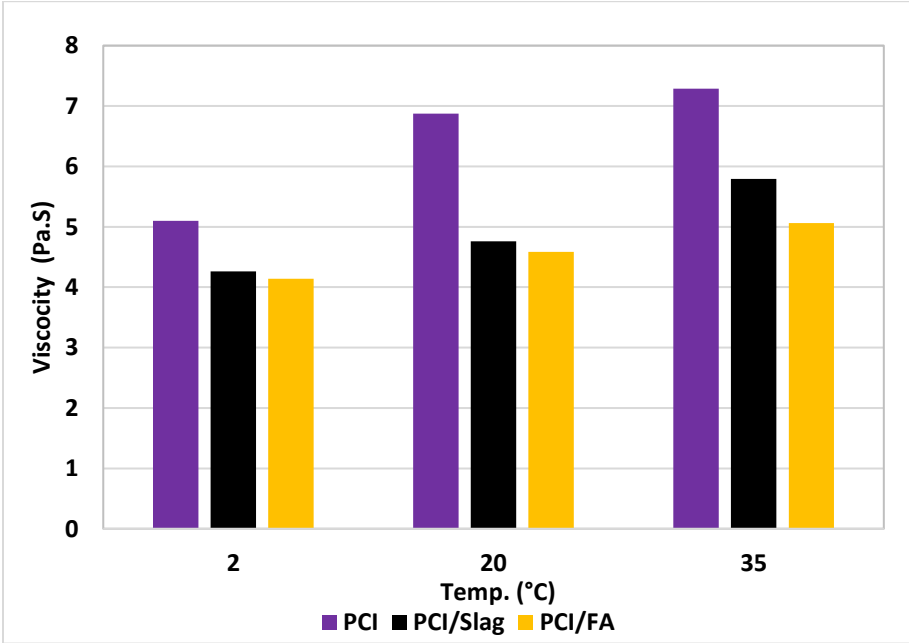


Figure 3.9 Effect of temperature and binder type on viscosity of CPB samples with 0.3% sodium silicate cured for 2 hrs.

As shown in Figures 3.8 and 3.9, high temperatures significantly increase the yield stress and viscosity of CPB with sodium silicate, regardless of the type of binder. The key process responsible for this behaviour is the temperature-induced increase of the binder hydration products, as discussed earlier. However, it should be noted that increasing the temperature will also enhance the activation of the clinker, FA and Slag particles, which is obviously associated with the production of more binder hydration products in comparison to the samples without sodium silicate. The latter is an additional factor that contributes to explaining the significant increase in the yield stress and viscosity of the CPB samples with sodium silicate and Slag or FA with temperature (Figures 3.8 and 3.9), as will be discussed later.

Moreover, it can be observed in Figures 8 and 9 that the partial replacement of PCI with Slag or FA reduces the yield stress and viscosity of the sodium silicate rich CPB, regardless of the temperature. It is evident that at all testing temperatures, the yield stress and viscosity of CPB made with PCI/Slag as the binder (and containing sodium silicate) are lower than those of the PCI-CPB samples (and containing sodium silicate). Furthermore, CPB samples made with PCI/FA as the binder (and containing sodium silicate) have the lowest yield stress and viscosity.

The reduction of the yield stress and viscosity when FA and PCI are used as the binder can be explained by the combined effect of three processes: (i) the spherical shape of the FA particles reduces the friction forces between the particles (Siddique 2004, Park et al. 2005); (ii) the partial replacement of PCI with FA increases the packing density of the CPB particles because the FA particles are much finer than the PCI particles, which will reduce the volume of pore-water required to fill the voids between particles. This will allow the water to act as a lubricant for the particles (Lee et al. 2003, Kwan and Chen 2013), and reduce the stickiness and consequently reduce the yield stress and viscosity; and (iii) the internal heat within the cemented mixture due to the cement hydration process will be significantly reduced by using FA (Le et al. 2019). Hence, the hydration process will slow down and thus the yield stress and viscosity will decrease at the very early ages of hydration. On the other hand, the higher values of yield stress and viscosity of CPB when Slag and PCI are used as the binder (compared with PCI/FA as the binder) can be attributed to the fact that the combination of Slag and PCI increases the volume of cement hydration products produced as a result of the exothermic reaction between Slag and CH that is facilitated from the early hydration of cement (Fall et al. 2010, Tariq and Yanful 2013). However, it is evident that the yield stress and viscosity of CPB made with PCI/Slag are lower than those of the PCI-CPB samples. This phenomenon is due to the initial setting time, which significantly affects the rheology of cement paste as the initial setting time is when the cement paste gradually loses its plasticity (Sarda et al. 2001), and is associated with the cement hydration process (Zheng et al. 2015), which occurs at longer curing times for PCI/Slag systems (Brough and Atkinson 2002). Moreover, using Slag will reduce the heat released during the hydration process (Kolani et al. 2012), and thus slow down the hydration process and reduce the volume of cement hydration products at the very early ages.

This assertion is experimentally validated by the results of the thermal analyses (TG/DTG and XRD) performed on the CPB samples with different types of binders and 0.3% of sodium silicate, and cured for 2 hours at 35°C. The TG/DTG graphs in Figure 3.10 confirm that the sodium-silicate rich CPB sample prepared with only PCI as the binder has a larger volume of hydration products, while there are less (e.g., C-S-H, ettringite) within the samples with PCI/Slag compared to the sample with only PCI, but higher than in the sample with PCI/FA. The graphs in Figure 10 show that from the first weight loss and peak in the TG and DTG curves, respectively, which occurred at about 100°C, the volume of cement hydration products, such C-S-H and ettringite, is more in the PCI-CPB samples, followed by PCI/Slag and finally PCI/FA. The lower peak and weight loss at 450°C of the PCI/Slag sample compared to the PCI/FA sample is the result of the consumption of CH during the activation of Slag, which obviously results in the generation of additional C-S-H.

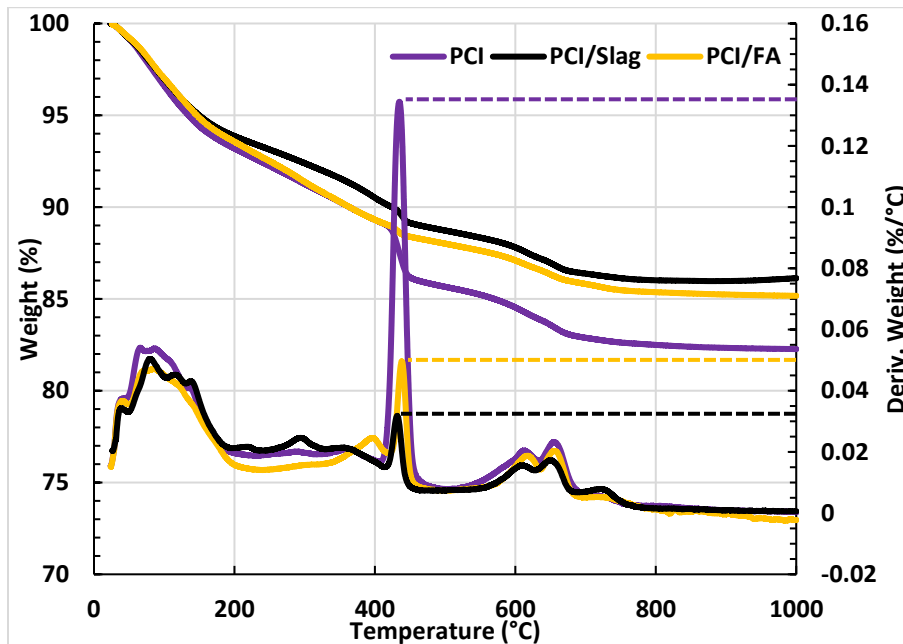


Figure 3.10 DT/DTG diagrams for CPB samples with 0.3% of sodium silicate and cured for 2 h with different binder type at 35°C.

Moreover, the plotted information in the TG/DTG graphs is consistent with the results of the XRD results (Figure 3.11), in which the type and relative volume of cement hydration products formed within these CPB samples are illustrated. It is apparent that some of these hydration products show a higher intensity for the CPB sample with only PCI compared to the samples with PCI/Slag, while the intensity is the lowest for the CPB samples with PCI/FA. In other words, more hydration products formed in the PCI-CPB samples than the PCI/Slag samples, with the lowest volume in the samples with PCI/FA. For example, the intensity of portlandite at 18° - 2θ is 621 CPS and 196 CPS for the CPB samples with PCI and PCI/FA, respectively. Similarly, the intensity of

ettringite at 41° - 2θ is 117 CPS, 99 CPS, and 96 CPS for the PCI-CPB, PCI/Slag and PCI/FA samples, respectively.

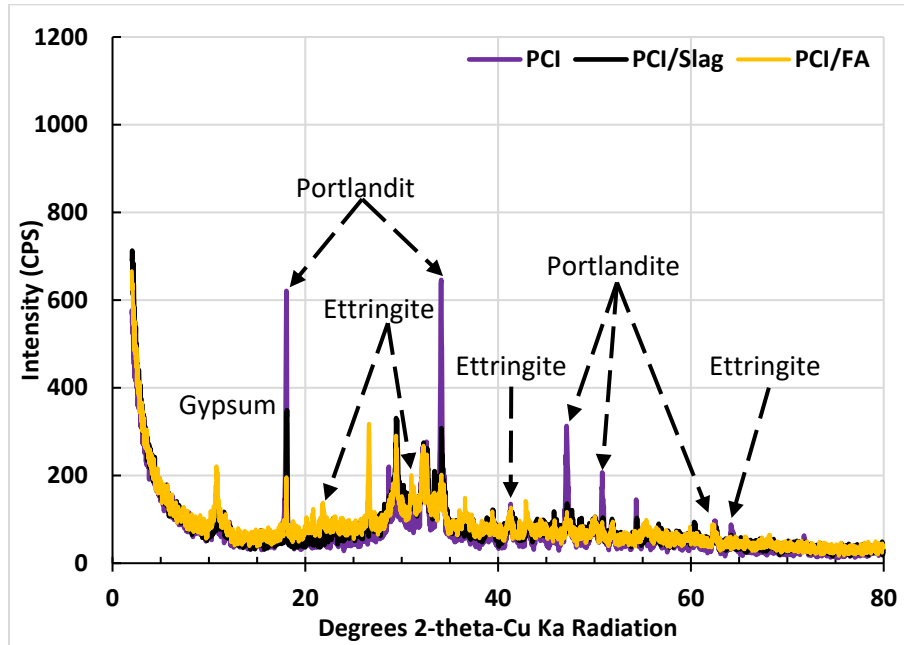


Figure 3.11 XRD intensities for CPB samples with 0.3% of sodium silicate cured for 2 h with different binder type at 35°C .

Furthermore, the EC monitoring results in Figure 3.12 support this result. It can be observed from Figure 3.12 that the CPB sample with only PCI has the highest EC over a curing time of 4 h. This indicates that the concentration and mobility of ions are the highest when only PCI is used, which would also suggest that the hydration reaction within the PCI-CPB samples consumes more free pore-water, so that the CPB becomes more viscous (Figure 3.9). Moreover, the EC of the PCI-CPB peaks earlier than that of the PCI/FA or PCI/Slag samples. In other words, the cement hydration is more rapid in the PCI-CPB samples, which is consistent with the results presented in Figures 3.8 and 3.9. On the other hand, the PCI/FA samples show a higher EC as opposed to the PCI/Slag samples for the first 1.6 hours (100 minutes), and then the EC of the PCI/Slag samples increases and then exceeds the EC of the PCI/FA samples. The EC continues to be high for the rest of the monitored period during which the PCI/FA samples show a gradual reduction in EC. This observation shows that the partial use of FA increases the mobility and concentration of ions at the beginning (compared with the partial use of Slag) and then decreases after 100 minutes.

The results obtained here suggest that the use of PCI/FA as the binder in CPB with sodium silicate will impart better flowability in the CPB material even in high temperatures.

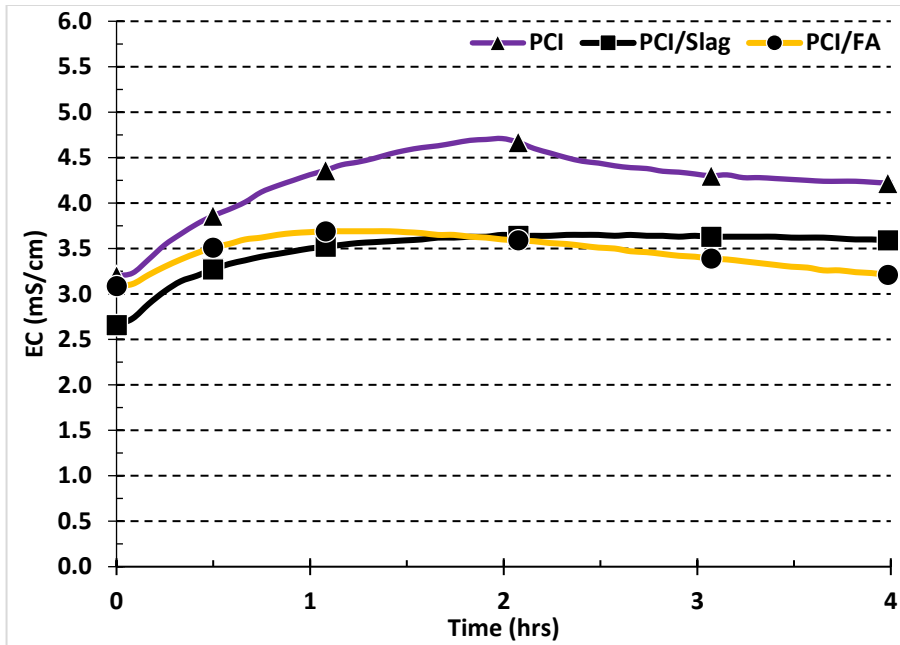


Figure 3.12 Development of EC in CPB samples with 0.3% of sodium silicate and different binder type at 35°C.

3.4.3 Combined effect of amount of sodium silicate and curing temperature

The effect of the amount of sodium silicate on the rheological properties of CPB was established by testing the yield stress and viscosity of the PCI-CPB samples cured for 2 hours with different amounts of sodium silicate (0.0%, 0.1%, 0.3%, and 0.5%). The effect of the binder content on the yield stress and viscosity of the PCI-CPB samples with respect to the mixing and curing temperatures are shown in Figures 3.13 and 3.14, respectively.

It is evident in Figures 3.13 and 3.14 that, for a given temperature, an increase in the sodium silicate content increases the yield stress and viscosity of the PCI-CPB. The yield stress and viscosity are found to be directly correlated with the sodium silicate content. Moreover, it can be also observed that PCI-CPB with 0.3% and 0.5% of sodium silicate show a higher relative increase in yield stress and viscosity at higher temperatures (20°C and 35°C) compared to a lower temperature (2°C). This phenomenon is explained as follows. On the one hand, an increase in the initial and curing temperatures of PCI-CPB samples with sodium silicate enhances the sodium silicate as an alkali activator (Živica 2007). The enhancement in activation energy of sodium silicate at high temperature is associated with the increase of absorption ability due to the stress-induced sodium ion migration (Souquet et al. 2010). An increase (albeit not an excessive one) in sodium content leads to the activation of more Slag and FA particles as well as the clinker phases (Taylor 1990, Abdul-Hussain and Fall 2012). Consequently, these two factors lead to the growth of more cement hydration products, thereby changing the rheological properties of the CPB. On the other hand, high dosages of an alkali activator also significantly decrease the setting time of

CPB (Azadi et al. 2017), and subsequently increase the yield stress and viscosity of CPB (Warner 2004, Azadi et al. 2017). This is also in agreement with the findings in (Kermani et al. 2015b), who examined the initial setting time of CPB with different amounts of sodium silicate, and found that the initial setting time of CPB without any sodium silicate is approximately 4.5 hours (270 minutes) but about 3.8 hours (230 minutes) and 3.5 hours (210 minutes) for CPB samples with 0.3% and 0.5% of sodium silicate, respectively. This argument which is related to the formation of more cement hydration products with higher amounts of sodium silicate is consistent with the results of the thermal analyses (TG/DTG) carried out on the CPB samples with different amounts of sodium silicate (0.0%, 0.3% and 0.5%), and cured for 2 hours at 35°C in this study, as shown in Figure 3.15. For example, it is evident from the first peak in the DTG curves, which occurred at about 100°C, that the volume of cement hydration products (mainly C-S-H, ettringite and gypsum) is higher in the samples with 0.5% of sodium silicate, followed by the CPB with 0.3% of sodium silicate and finally the samples made without sodium silicate. Moreover, the second peak which occurs at about 450°C shows an almost similar volume of CH produced within the CPB samples with 0.3% and 0.5% of sodium silicate, and less within the CPB samples with no sodium silicate.

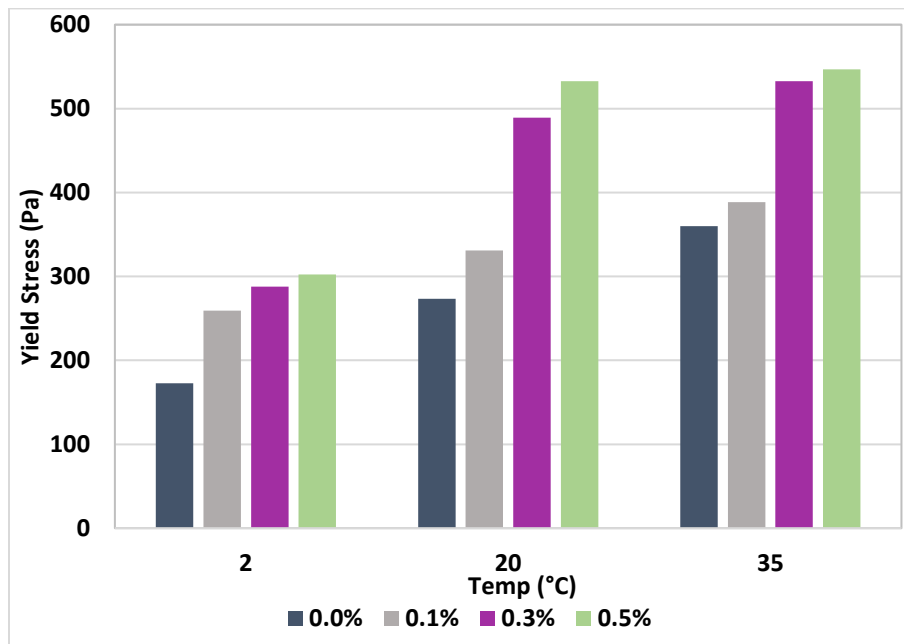


Figure 3.13 Effect of temperature and sodium silicate content on yield stress of PCI-CPB cured for 2 h.

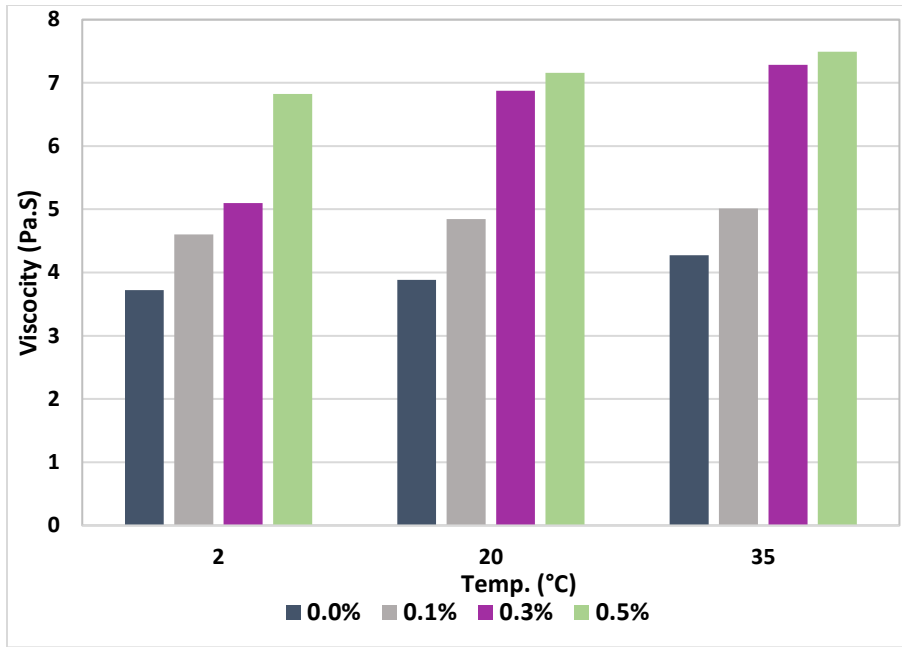


Figure 3.14 Effect of temperature and sodium silicate content on viscosity of PCI-CPB cured for 2 h.

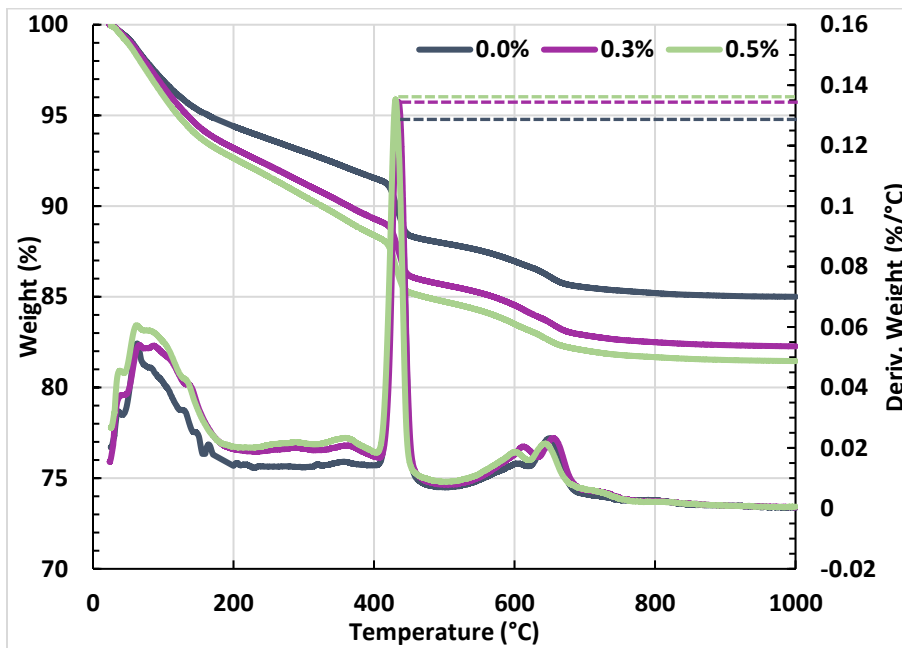


Figure 3.15 DT/DTG of PCI-CPB-SS with different sodium silicate contents prepared and cured for 4 h at 35°C.

Furthermore, the EC monitoring results in Figure 16 support this finding. In the figure, the initial EC values of CPB with 0.5% sodium silicate are higher than those of CPB with 0.3% sodium silicate and no sodium silicate mainly due to their initial differences in the sodium silicate, particularly in the volume of free Na⁺ ions. Moreover, it is also obvious from these curves that the

plotted EC values of the CPB with 0.5% of sodium silicate peak first (at 1.75 hrs), followed by the CPB with 0.3% of sodium silicate (2 hrs), and finally, the CPB without sodium silicate (2.3 hrs). This suggests that the rate of binder hydration increases as the sodium silicate content is increased. It can also be observed from Figure 16 that the CPB with 0.5% of sodium silicate shows the highest rate of increase in EC and a significantly higher EC persists over the 4 hours of curing, which indicates that the concentration and mobility of ions are higher with 0.5% of sodium silicate. This also means that the PCI-CPB consumes more free pore-water, so that the CPB become more viscous (Figure 3.14). On the other hand, the samples with 0.3% of sodium silicate show a lower rate of increase in the EC compared to those with 0.5% sodium silicate, but higher rate of increase than the CPB samples with no sodium silicate. This observation further validates that the addition of sodium silicate to CPB mixtures accelerates the mobility and increases the concentration of ions within the CPB, thus resulting in more rapid binder hydration and consumption of more capillary water, and thereby increasing the yield stress (Figure 3.13) and viscosity (Figure 3.14) of the backfill material.

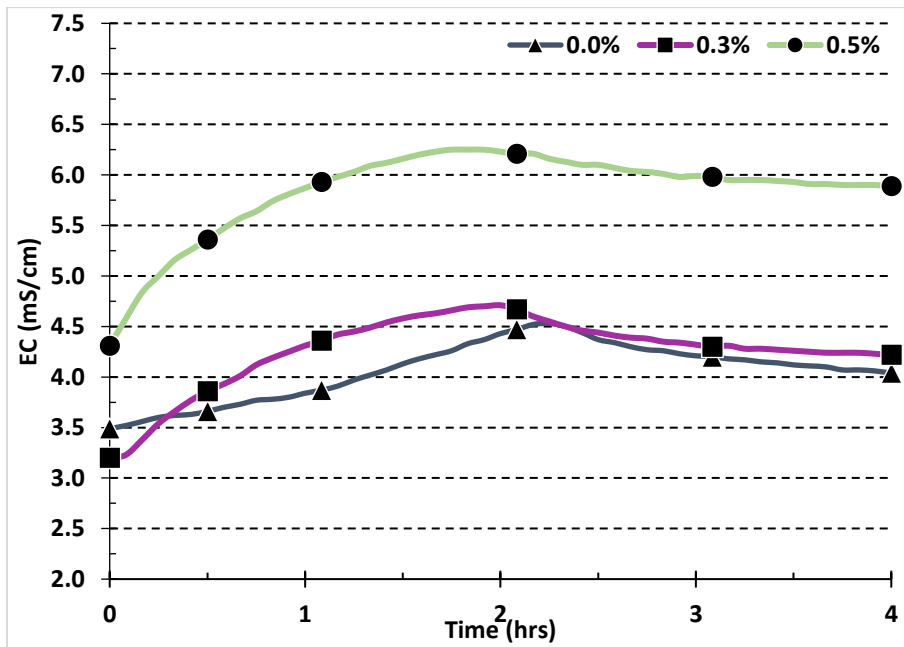


Figure 3.16 Time-dependent changes of electric conductivity of PCI-CPB-SS with different sodium silicate contents cured at 35°C.

3.5 Summary and Conclusion

This study mainly examines the combined effects of temperature, type of binder and sodium silicate content on the time-dependent changes in the rheological properties (yield stress and viscosity) of CPB material. The primary conclusions based on the results of this research are summarized as follows:

1. An increase in the initial and curing temperatures of CPB with sodium silicate increases cement hydration reactions, which consequently increases cement hydration and reduce the water content in the CPB, and results in increased yield stress and viscosity of the CPB material. The temperature-dependent increase holds, regardless whether the CPB sample is made with only PCI, PCI/Slag, or PCI/FA.
2. Adding sodium silicate to the CPB significantly reduces its flowability by increasing its yield stress and viscosity, regardless of the exposed temperature. This reduced flowability is attributed to the activation to the pozzolanic compounds (Slag particles, FA) and acceleration of the hydration of the clinker and binder, which result in stronger and more viscous CPB. However, the magnitude of this effect of the sodium silicate on the rheological properties of CPB is a function of the sodium silicate content and the temperature.
3. The yield stress and viscosity increments are directly proportional to the increase in the dosage of sodium silicate regardless of the change in temperature due to more complete activation of the studied pozzolans and more rapid cement hydration. However, the CPB samples with sodium silicate show a higher relative increase in yield stress and viscosity at higher temperatures (20°C and 35°C) compared to a lower temperature (2°C). The behaviour described above can be explained by the: (i) temperature-induced enhancement of sodium silicate as an alkali activator, and (ii) acceleration of the setting time of CPB due to high dosages of sodium silicate.
4. The partial use of Slag or FA as a binder with PCI reduces the yield stress and viscosity (enhances the flowability) of the CPB samples with sodium silicate. However, the partial use of FA enhances the flowability more compared with the partial use of Slag, as CPB samples with PCI/FA show a lower yield stress and viscosity than those with PCI/Slag.

The findings of this investigation demonstrate the significance of temperature and its effects on the time-dependent rheological behavior or flow ability of CPB with various contents of sodium silicate and provide significant technical information for the designing of cost-effective transport systems of CPB with sodium silicate as well as for the optimal assessment of the flowability of CPB under various mine thermal conditions. Further studies are required to gain insight into the flow ability of CPB with sodium silicate in sub-zero environments as increasingly number of mining activities are being conducted in permafrost or cold regions

3.6 Data Availability Statement

All data, models, and code generated or used during the study appear in the submitted article.

3.7 Acknowledgements

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CHAPTER 4

Paper II: Temperature Dependency of the Strength Development of Slag-Cemented Paste Backfill that Contains Sodium Silicate

Paper II: Temperature Dependency of the Strength Development of Slag-Cemented Paste Backfill that Contains Sodium Silicate

(to be Submitted)

Ghada Ali, Mamadou Fall and Imad Alainachi

4.1 Abstract

Cemented paste backfill (CPB) is a novel backfill technology that is used in mining industry in order to minimize the geotechnical and environmental hazards associated with the traditional mine waste management. Also, CPB is used as a backfill to enhance the stability of the mine stope (underground opening) and the adjacent area as well as to minimize the possible geotechnical risks, such as ground subsidence. CPB consists of tailings, water, and hydraulic binder (such as cement and blast furnace slag). Often, CPB is prepared and mixed in the paste plants located in the mine area, and the fresh CPB is delivered into the mine stope through pipelines by gravity and/or pumping. Recently, sodium silicate has been used as chemical activator for the hydraulic binders used in CPB mixture to improve mechanical behavior of CPB. However, in order to achieve the desired performance of CPB placed in the mine stope, it is necessary to study the strength of early aged (up to 28 days) CPB. To do so, this study aimed to assess the strength of sodium silicate activated slag-CPB under diverse conditions. Unconfined compression strength (UCS) of slag-CPB was experimentally tested in this research for slag-CPB samples that contain sodium silicate and were cured (up to 28 days) at different curing temperature (2°C, 20°C, and 35°C). Also, in order to examine the effect of sodium silicate on the strength of CPB cured at different temperatures, the slag-CPB mixtures that contained different content of sodium silicate (0.0%, 0.3%, and 0.5%) were studied in this research. The result of this study found that the strength development of CPB increases with time and temperature. It was also found that the increase in the sodium silicate dosage will enhance the mechanical strength of CPB at any temperature. The findings of this research will effectively contribute to the safer design of CPB structures.

Keywords: Cemented paste backfill; Tailings; Cement; Strength, Sodium Silicate, Temperature; Mine

4.2 Introduction

Mining industry has significantly improved the economy of several countries around the world. For instance, the total mining production in 2017 was around 19 billion USD in Canada, and around 600 billion USD around the globe (Alainachi and Fall 2019a, Statista 2019). However, mining activities are associated with several challenges, such as generation of huge quantities of mine wastes (e.g., tailings). These wastes may negatively impact the environment if they are improperly disposed of (Farkish and Fall 2013, Alainachi and Fall 2019b). In the past decades, a novel method called cemented paste backfill (CPB) was proposed in order to better manage the disposal of the tailings. This technology is characterized by reusing these tailings and returning them back to the underground mine stopes as a paste after mixing them with water and binder

materials (such as cement) (Belem and Benzaazoua 2004, Fall et al. 2005, Abdul-Hussain and Fall 2012). Using CPB method reduces the time needed to complete the mine stope backfilling, which decreases the stope cycle (stope backfill process) and consequently improves the mine productivity. Also, using tailings as main component of CPB reduces the amount of these environmentally challenging wastes. Moreover, using CPB can also improve the stability of the mine stope in short and long term, which will minimize the geotechnical risks (such as ground subsidence and mine stope instability) that might endanger the safety of the workers at the mine workplace and the surrounding area (Ghirian and Fall 2015, Alainachi and Fall 2019a). Therefore, CPB has become a common technology in mining industry around the world (Brakebusch 1995, Belem and Benzaazoua 2004, Aldhafeeri et al. 2016, Alainachi and Fall 2019c). However, the mechanical strength or performance of CPB is significantly affected by several conditions, such as exposure to variation in static loading, dynamic (seismic) loading as well as the potential chemical interaction of the chemical elements within the CPB components (Cao et al. 2018, Xue et al. 2018, Alainachi and Fall 2019b).

The typical components of CPB mixture are tailings with a content of around 70%-85%, hydraulic binder of around 2%-9% of content (based on the stope requirements), and fresh or mine processed water. The CPB is generally mixed and prepared in paste backfill plant usually located on the mine surface and is then delivered into the mine stope through pipelines by gravity and/or pumping (Yilmaz et al. 2004, Fall et al. 2005, Jamali 2012, Haiqiang et al. 2016, Koohestani et al. 2018).

In order to have safe and cost effective CPB structures, CPB mixtures must demonstrate an appropriate stability performance as soon as it is placed in the mine stope. Mechanical strength properties are significant factors in this regard. These properties can be evaluated by determining the unconfined compression strength (UCS) of early aged CPB (Ghirian and Fall 2013, Cui and Fall 2016).

It was learnt from previous studies, such as (Fall et al. 2010, Wu et al. 2013), that the strength and pore-structure of fresh CPB placed in the mine stope are significantly affected by temperature variation. There are several sources of the temperature variation (Figure 4.1), which include (i) internal heat generated during cement hydration process (Fall et al. 2010, Alainachi and Fall 2019c), (ii) temperature variation based on the geographic location of the mine, especially in the permafrost regions and/or extremely hot regions (Wang et al. 2016), and (iii) heat generated from the rock masses surrounding the mine stope that varies with the depth of the mine stope and the geological condition of the mine area (Fall et al. 2007, Aldhafeeri et al. 2016). In addition, the temperature of CPB in the mine stope might also be affected by other human-induced temperature variations, such as the heat coming from the mine machinery, lighting, and ventilation, as well as the heat generated due to the blasting operations (Fall et al. 2010). Anyways, the temperature variation of CPB in the mine stope, regardless of its source, was found to be of significant influence on the mechanical behavior (mechanical strength) of CPB (Fall et al. 2010, Fall and Pokharel 2010).

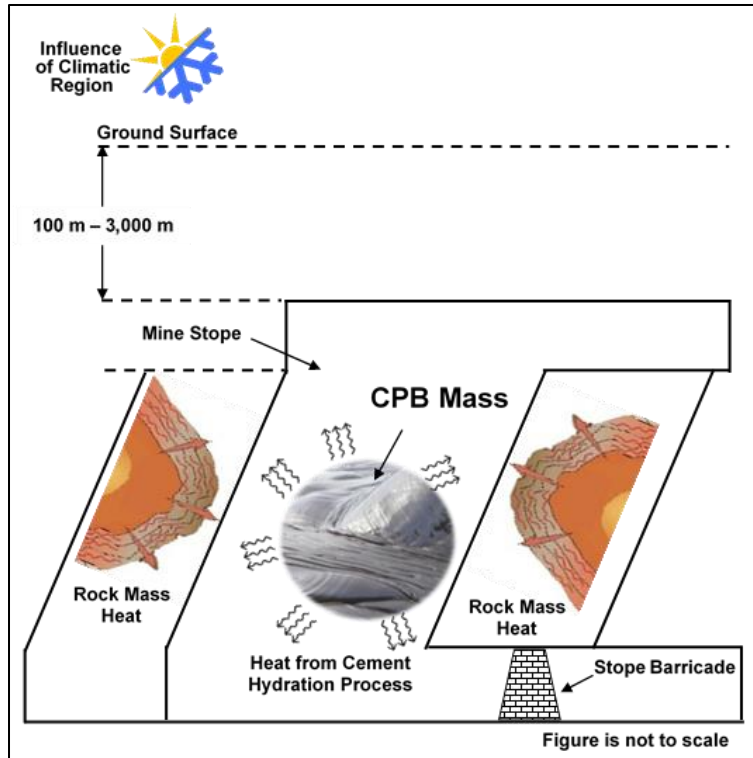


Figure 4.1 Sources of temperature change in CPB placed in mine stope.

Other than the temperature, there are other factors (external and internal) that might also affect the mechanical strength of CPB, such as the density and concentration of CPB (binder type and water content), and the availability of water-reducing admixture and/or chemical additives (Yilmaz et al. 2004, Kesimal et al. 2005, Bentz 2006, Ercikdi et al. 2009, Ercikdi et al. 2010, Abdul-Hussain and Fall 2012, Cihangir et al. 2012, Ghirian and Fall 2014, Kermani et al. 2015a, Cihangir et al. 2018). However, some of these studies have addressed the mechanical strength of CPB under the effect of individual factor. For instance, Kesimal et al. (2005), Ercikdi et al. (2009), and Li and Fall (2018) studied the effect of different types and dosages of binders on the short-term and long-term strength of CPB. Some other studies have been conducted recently in order to understand the coupled effect of temperature on the mechanical strength of CPB that is made with different binder contents. For instance, Fall et al. (2010) studied the impact of different curing temperatures (2°C, 20°C, 35°C, and 50°C) on the strength of CPB made with different binders, such as Portland cement (PCI), and blast furnace slag (Slag). This study revealed that the strength of CPB increases as the curing temperature increases. Moreover, it was also noted that the partial usage of slag (as a binder) has a significant impact on the temperature induced increase in CPB strength. Nasir and Fall (2010) developed a numerical model to predict the coupled effect of temperature and degree of hydration on the unconfined compression strength (UCS) of CPB. This study found that increasing the initial temperature of CPB from 0°C to 20°C increases the strength of CPB by 60%. Also, the early strength development of CPB is significantly affected by the binder content. Furthermore, Pokharel and Fall (2011) compared the effect of curing temperature on the

strength development of CPB containing only PCI and CPB containing PCI and slag as binders. It was revealed that at temperatures of 2°C and 35°C, the UCS of CPB containing PCI and slag is higher than the UCS of CPB containing PCI only, while at the temperatures of 20°C and 50°C, the UCS of CPB containing PCI and slag is lower than the UCS of CPB containing PCI only.

On the other hand, some studies (e.g. Brough and Atkinson 2002, Chang 2003, Živica 2007, Kermani et al. 2015a, Kermani et al. 2015b, Cihangir et al. 2018, Jiang et al. 2019) have used sodium silicate as chemical additive to different mixtures of CPB. These studies addressed the role of sodium silicate as activator for different binders of CPB (such as PCI and Slag) as well as the effect of sodium silicate on the mechanical strength of CPB in the long term.

Up to date, there is a paucity of information and technical data on the effect of curing temperature on the strength development of CPB that is made of PCI and Slag as binders and contains sodium silicate of different dosages. In other words, no study has evaluated the combined effect of curing time, temperature, binder content, and sodium silicate content on the strength of CPB. Accordingly, the goal of this study is to experimentally examine the combined effect of different curing temperatures (2°C, 20°C, and 35°C), dosages of sodium silicate (0.0%, 0.3% and 0.5%), and progress of cement hydration (curing time) on the strength of slag-cemented paste backfill (Slag-CPB) mixtures.

4.3 Materials and Equipment Used in the Experiment

4.3.1 Materials

4.3.1.1 Tailings

Ground Silica Tailings (ST), which are synthetic tailings material (manufactured by the U.S. Silica Co.), were used in this study. The physical properties of ST (Table 4.1) are similar to those of Canadian mine tailings. For instance, ST are essentially made of quartz, which is the predominant mineral in Canadian hard rock mine tailings. Also, the grain size distribution of ST is similar to the average grain size distribution of nine main tailings (9MT) extracted from nine different mines in eastern Canada (Figure 4.2). Thus, ST was selected in this study to be the foremost component of CPB mixture. Moreover, ST has high percentage of silica (99.8% SiO₂), which makes it a chemically inter material (Table 4.2). It will control the mineralogical and chemical compositions of the tailings and minimize the uncertainties related to the use of natural tailings (Aldhafeeri and Fall 2016). The primary physical properties and main chemical elements of ST are illustrated in Tables 4.1 and 4.2.

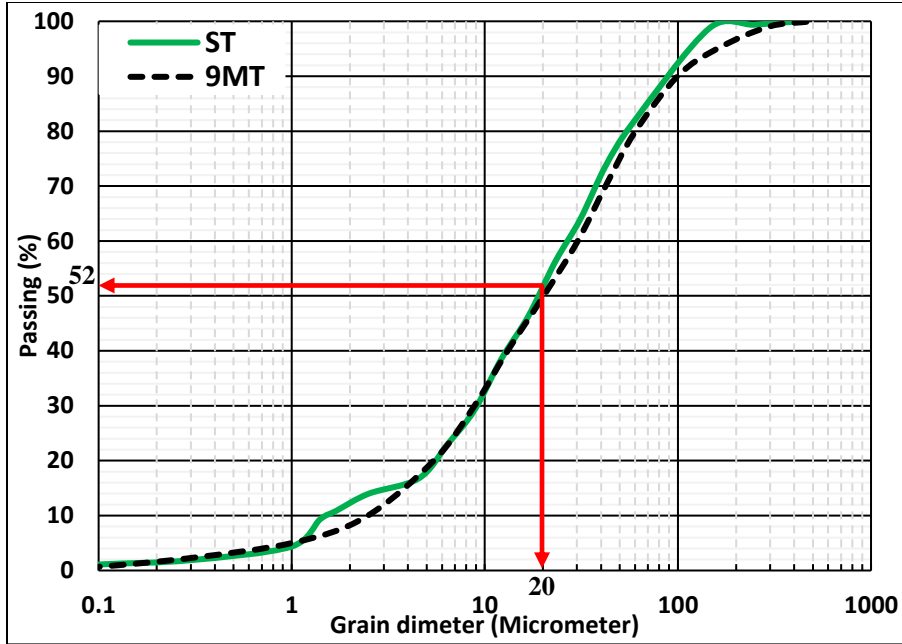


Figure 4.2 Grain size distribution of silica tailings used in this study and the average grain size distribution of tailings from nine Canadian mines.

Table 4.1. Primary physical properties of tailings used in this study

Element	Gs	D ₁₀ (μm)	D ₃₀ (μm)	D ₅₀ (μm)	D ₆₀ (μm)	Cu	Cc
ST	2.7	1.9	9.0	22.5	31.5	16.6	1.3

Table 4.2. Main chemical elements of tailings used in this study

Element	K wt.%	Pb wt.%	S wt.%	Ca wt.%	Fe wt.%	Na wt.%	Al wt.%	Si wt.%
ST	0.00	0.00	0.00	<0.01	<0.01	<0.01	0.10	99.80

4.3.1.2 Water and Binder

To enhance the mechanical properties of CPB, a sufficient amount of binder is often used in the mixture. In this study, the binder agent used in the preparation of the CPB mixtures is Portland cement type I (PCI), Fly Ash (FA), and blast furnace slag (Slag). These binders are the most frequently used in preparing CPB worldwide (Haiqiang et al. 2016). The properties of these binders are listed in Table 3.3. PCI is used alone or blended with Slag or FA. The blending ratio of PCI and Slag or FA is 50/50. This ratio is selected based on: (i) the need of the industrial partner in this project; (ii) cost savings; (iii) previous studies on performance of this ratio in CPB systems (e.g., Fall et al. 2010). Tap water is used in this study to mix the binders with the tailings.

Table 4.3. Primary physical and chemical properties of binders used in this study

Binder	SSA ⁽¹⁾ (m ² /g)	R.D ⁽²⁾	S (wt%)	Ca (wt%)	Si (wt%)	Al (wt%)	Mg (wt%)	Fe (wt%)
PCI	1.32	3.2	1.5	44.9	8.4	2.4	1.6	1.9
Slag	2.10	2.8	12	26.6	18.9	3.9	6.9	0.5

⁽¹⁾ Specific surface area and

⁽²⁾ Relative density

4.3.1.3 Sodium silicate

Soluble sodium silicates (SS) are silicate polymers, which are inorganic chemical, clear, colorless, and viscous liquid. SS are also known as water soluble glasses, which are generally produced from varied proportions of an alkali metal and silicon dioxide (SiO₂). There are several applications, wherein SS are often used. For instance, SS are used in geotechnical engineering field in soil grouting and mine tailing backfill. They are also used as an admixture for cement products. In this study, the main objective of using SS was to evaluate its effect on the strength of CPB. In this regard, commercial solution SS (Type N) was added in liquid form to the CPB mixture to be used as an admixture for the binders. Properties of SS used in this study are shown in Table 4.4.

Table 4.4. Properties of the sodium silicate used in this study (national Silicate Ltd.).

Properties	Values
Specific gravity @ 20 °C	1.39
Weight ratio, %SiO ₂ /%Na ₂ O	3.22
Na ₂ O% by weight	8.90
SiO ₂ % by weight	28.66
Solids %	37.56

4.3.2 Sample Preparation

For the UCS tests, approximately 72 CPB specimens were prepared by mixing given amount of tailings, binder (4.5 wt%), and water (w/c ratio =7.8). During the mixing of these ingredients, sodium silicate was added to each mixture (each sample) with different concentrations of 0, 0.3, and 0.5% by weight solid components. The sodium silicate gel, which is used in these mixtures, consists of 62.44% of water. However, this water content was considered while measuring the amount of mixing water of these samples. Samples were mixed for 7 min in order to obtain homogenous mixture (Bian et al. 2019). Afterwards, the prepared CPB mixtures were poured into a 600 ml low-form Griffin beaker and vibrated to remove the entrapped air. Samples were then poured into plastic cylindrical containers (50 mm diameter × 100 mm height). To prevent water evaporation, each sample was covered with plastic film. Samples were then securely cured at different temperatures of 2°C, 20°C, and 35°C and curing times of 1, 3, 7, and 28 days in an environmentally controlled chamber. Moreover, some cement paste samples of CPB with w/c = 1 (to mimic the high water content of CPB) and additional CPB samples were also prepared in the

similar manner as the aforementioned CPB specimens in order to conduct microstructural analyses or monitoring tests. Table 4.5 below summarizes the testing program and the CPB mixing composition for each phase of this program.

Table 4.5. Summary of CPB mix proportion and stages of experimental program

Sample ID	Binder Content (%)	%PCI in the binder	%Slag in the binder	W/C ratio	SS content (%)	Curing Temp. (°C)	Curing Time (days)
A. Effect of curing temperature on the time-dependent evolution of strength of Slag-CPB that contain sodium silicate							
SS-CPB-2°C	4.5	50	50	7.8	0.3	2	1, 3, 7, 28
SS-CPB-20°C	4.5	50	50	7.8	0.3	20	1, 3, 7, 28
SS-CPB-35°C	4.5	50	50	7.8	0.3	35	1, 3, 7, 28
B. Effect of Sodium silicate content, temperature and curing time of CPB's strength							
0.0-SS-CPB	4.5	50	50	7.8	0.0	2, 35	1, 3, 7, 28
0.3-SS-CPB	4.5	50	50	7.8	0.3	2, 35	1, 3, 7, 28
0.5-SS-CPB	4.5	50	50	7.8	0.5	2, 35	1, 3, 7, 28

PCI: Portland cement I, SS: Sodium silicate.

4.3.3 Test Methods

4.3.3.1 Unconfined Compression Test (UCS) test

Unconfined Compressive tests (UCS) were conducted on the CPB specimens that were prepared with different sodium silicate content under different temperatures and were cured under same temperatures to different lengths of time. In this research, ASTM C39/C39M (2018) was followed to conduct the UCS tests by using computer-controlled mechanical press of ELE Digital Tritest 50 load frame. The loading capacity and deformation rate of this test was defined as 50 kN and 1 mm/min respectively (Li and Fall 2018). To maintain reputability as well as to ensure accuracy of gathered results, each test was repeated at least twice.

4.3.3.2 Microstructural analysis

To understand the effect of microstructural evolution of CPB on its strength development under the influence of test conditions, several series of microstructural analyses were conducted on selected CPB samples at several testing ages (curing time). Microstructural analyses encompass thermal analysis (differential thermogravimetry (DTG), thermal gravimetry (TG), and Mercury Intrusion Porosimetry (MIP) tests. Before conducting microstructural analysis, testing samples

were first dried at 45°C in a vacuum oven up to mass stabilization. Thermal analyses, on the other hand, were undertaken using a TGA Q 5000 IR from TA Instruments. The various (dried) samples (about 20 mg each) were heated in an inert nitrogen atmosphere at the rate of 10°C per minute up to a temperature of 1000°C. MIP measurements were performed using Micromeretics AutoPore III 9420 mercury porosimeter.

4.3.3.3 Monitoring of the specimens

To better understand the coupled effect of curing temperature and sodium silicate content, CPB samples were prepared and poured into plastic cylinders (diameter of 100 mm and height of 200 mm), and placed in the controlled temperature chambers to be cured at the different curing temperatures of 20°C and 35°C for 28 days. During curing period, CPB samples undergone continuous monitoring in terms of the evolution of temperature, electrical conductivity (EC), volumetric water content (VWC), and suction. Changes in EC reflect the rate of ion movement due to the chemical reactions between cement and water. Monitoring EC is an effective way to assess the cement hydration progress and the related structural changes (Li and Fall 2016). On the other hand, monitoring the VWC enables to assess the self-desiccation of CPB (capillary water consumed by the cement hydration) as well as the water flow within the CPB mass. The monitoring of the temperature and suction enables to understand the progress of the cement hydration within the tested specimens.

In this regard, the ECH2-5TE sensor was used to monitor EC, VWC, and temperature as this sensor measures EC in the range of 0-23 dS/m with the accuracy of ± 0.1 , measures VWC in the range of 0-80% with the accuracy of ± 0.01 from 1-40% and the accuracy of ± 0.15 from 40-80%, whereas the temperature measurement accuracy is $\pm 1^\circ\text{C}$. While dielectric water potential sensor (ECH2-MPS6 sensor) was used to monitor the suction evolution. This sensor is designed to measure soil water potential in the range of -9 to -100,000 kPa with a resolution of 0.1 kPa and accuracy of ($\pm 10\%$ of reading + 2 kPa, from -9 to -100 kPa). These sensors were installed within the CPB specimens and connected to the Em50 series logger in order to record the data.

4.4 Results and Discussion

4.4.1 Effect of temperature on the strength development of Slag-CPB that contains Sodium Silicate.

Figure 4.3 illustrates the effect of temperature on the strength of slag-CPB samples that contain sodium silicate. As expected, regardless of the temperature conditions, there is a continuous improvement in the mechanical strength of slag-CPB samples that contain sodium silicate with 1 day to 28 days curing time. This is mainly attributed to the progress of cement hydration, and the mechanism of self-desiccation, which is involved in lowering the free water in the CPB material (Bullard et al. 2011). It is also agreed that the partial usage of slag will increase the generation of cement hydration products that will contribute to the strength (7 days and more of hydration) of material cemented with PCI (Mehta and Monteiro 2001). This is because slag will be activated by

the CH released by cement clinker hydration and produce additional calcium silicates hydrate (secondary C-S-H) (Li and Ding 2003, Tariq and Yanful 2013). In addition, it will trigger the role of the alkali activators (such as sodium silicate) in activating the pozzolans binders (slag) leading to the acceleration of its hydration process and the formation of more CH (Roy and Idorn 1982, Kolani et al. 2012).

It can also be observed from figure 4.3 that the increase in curing temperature has a direct relationship with strength development. Samples cured at the temperature of 35°C showed higher values of UCS strength, while the samples cured at the temperatures of 2°C showed the lowest values. This is because curing CPB samples at high temperatures accelerates the cement hydration process (Aldhafeeri et al. 2016, Cui and Fall 2016) and produces more cement hydration products (Wang et al. 2016). The accelerated cement hydration process will lead to more consumption of the CPB pore-water, and the higher production of cement hydration products will result in the refinement of the pores between CPB particles as well as an enhancement in the cohesion between the CPB particles. Thus, the strength of CPB material will significantly increase (Alainachi and Fall 2019c, Haruna and Fall 2019). Moreover, C-S-H was found to be the major binding phase in hardened cement (Taylor 1990), so the more production of C-S-H, the more development of CPB strength (Fall et al. 2010).

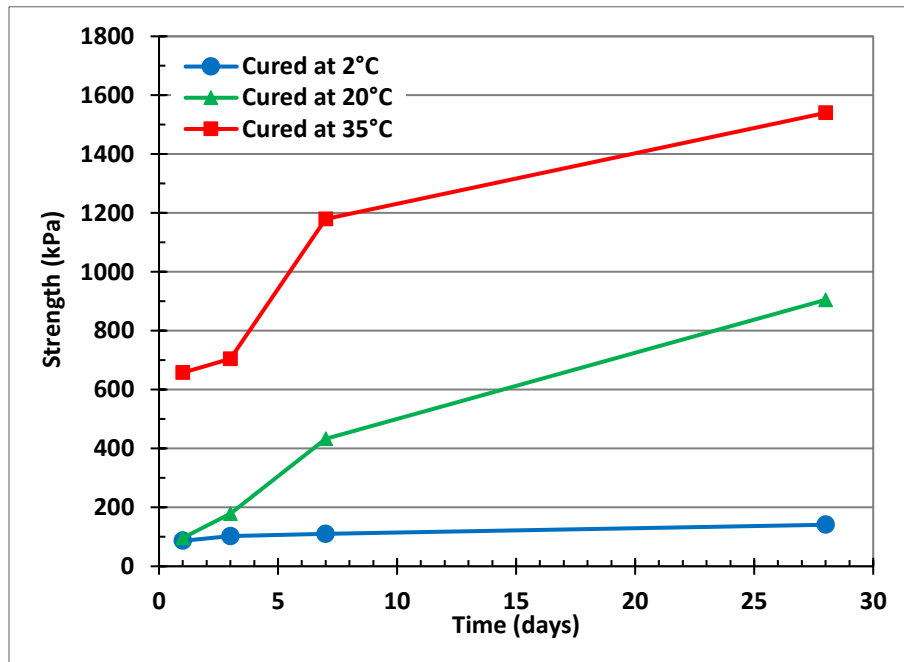


Figure 4.3 Effect of temperature on the strength development of Slag-CPB that contain 0.3% sodium silicate.

This argument is experimentally supported by the results of the microstructural analysis, including thermal analysis (Figure 4.4) and MIP analysis (Figure 4.5). Figure 4.4 illustrates the TG/DTG diagram for two slag-CPB samples that contain 0.3% of sodium silicate, which were

cured (up to 7 days) at different temperatures of 20°C and 35°C. It is evident from these diagrams that more hydration products are produced within the slag-CPB that was cured at higher temperature. The first peak (DTG) or change in weight (TG), which is found between 50°C and 150°C and is the resultant of the dehydration reactions of hydrates (such as C-S-H, ettringite, and gypsum), and the second peak, which is associated with generation of CH and is observed at 400-500°C, are much higher for the CPB cured at 35°C as compared to the CPB cured at 20°C. While the third peak and weight loss, which represents the decomposition of the calcite in the cement and is found between 600°C and 700°C, was found to be lower within the CPB cured at 35°C as compared to the CPB cured at 20°C. Figure 4.5, on the other hand, illustrates the differential pore size distribution of 7-days slag-CPB samples that contain sodium silicate, which were cured at the temperatures of 20°C and 35°C. It is observed that the threshold pore diameter decreases with the increase in curing temperature. It indicates a refinement in the pore structure of the cementitious material cured at high temperature as a result of the large amount of hydration products, and thus higher mechanical strength (Aldhafeeri et al. 2016, Haruna and Fall 2020).

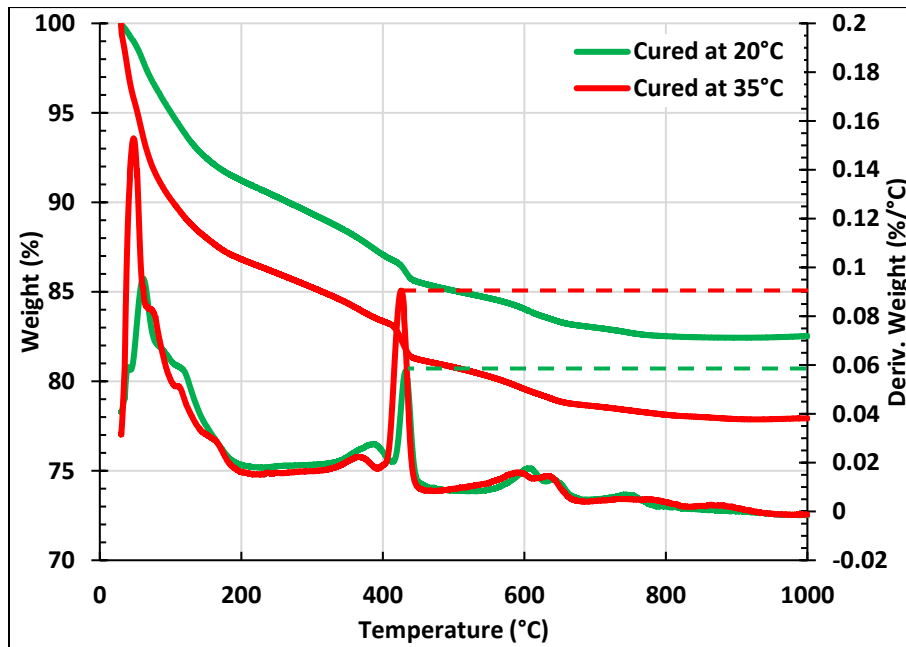


Figure 4.4 DT/DTG diagrams for 7 days Slag-CPB specimens that contain 0.3 % sodium silicate, cured at various temperatures (20°C and 35°C).

Moreover, the acceleration of cement hydration rates due to high curing temperature is also evident in the EC monitoring results shown in Figure 4.6, which demonstrates the changes in EC of slag-CPB samples that contain sodium silicate and were cured (up to 28 days) at different temperatures of 20°C and 35°C. It can be observed from this figure that the time required for EC within the CPB samples cured at 35°C to reach its maximum value is much less than those of CPB samples cured at 20°C. In addition, the peak EC values of CPB samples cured at higher temperature were much higher than those for samples cured at lower temperature. The CPB samples cured at

35°C reached the peak of around 4.0 mS/cm within around 3 hours, while it needed around 11 hours for CPB samples cured at 20°C to reach the peak EC of 3.2 mS/cm. It provides a clear indication of the rapid increase in the rate of cement hydration due to a higher curing temperature, and the higher concentration and higher mobility of ions within the CPB samples cured at higher temperature (Li and Fall 2016, Fang and Fall 2018).

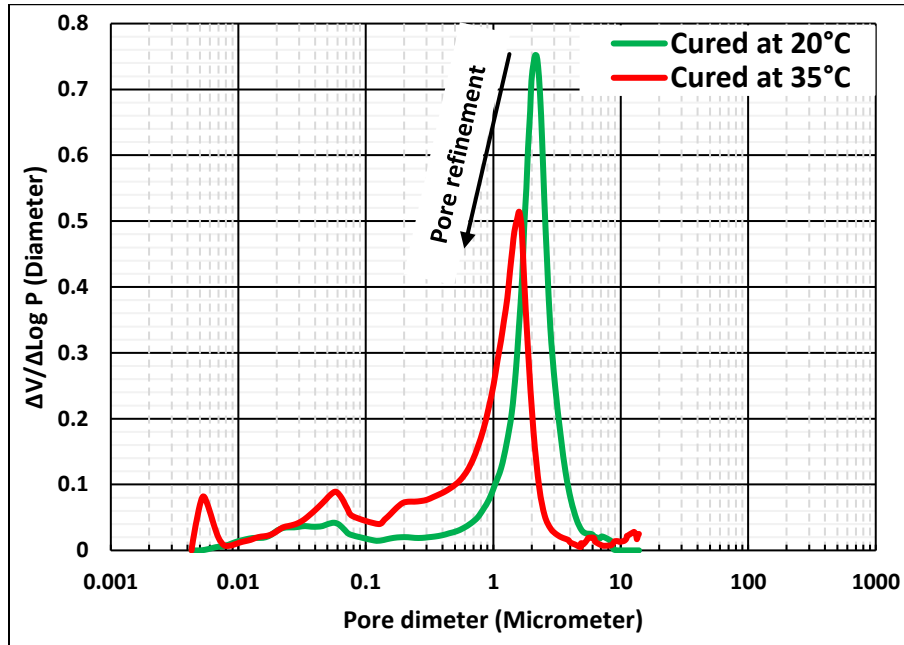


Figure 4.5 Differential pore size distribution curves of 7 days CPB specimens that contain 0.3% sodium silicate, cured at various temperatures (20°C and 35°C).

Furthermore, there is a common consent that high curing temperature of CPB material intensifies the cement self-desiccation of the material (Alainachi and Fall 2019c). Self-desiccation is the shrinkage of pores due to the progress of cement hydration process, which will lead to a net reduction in the total volume of the solids and water within the cementitious materials (Bentz 2008). This will consequently reduce the volumetric water content within the CPB pores, and thereby decrease the pore-water pressure (PWP) and could lead to generation of negative pore-water pressure (suction). Hence, the temperature-induced increase in self-desiccation will enhance the mechanical strength of CPB (Abdul-Hussain and Fall 2012, Ghirian and Fall 2013). This argument can also be supported by the results presented in Figure 4.7, which illustrate the evolution of suction and volumetric water content (VWC) of slag-CPB samples that contain sodium silicate and were cured (up to 28 days) at the temperatures of 20°C and 35°C. These results showed that after 1 day of curing, there was a rapid decrease in VWC and rapid increase in suction values (higher negative PWP) within the CPB sample cured at 35°C as compared to those cured at 20°C. This will help in understanding the effect of curing temperature in accelerating the cement hydration process and thereby enhancing the mechanical strength of CPB samples.

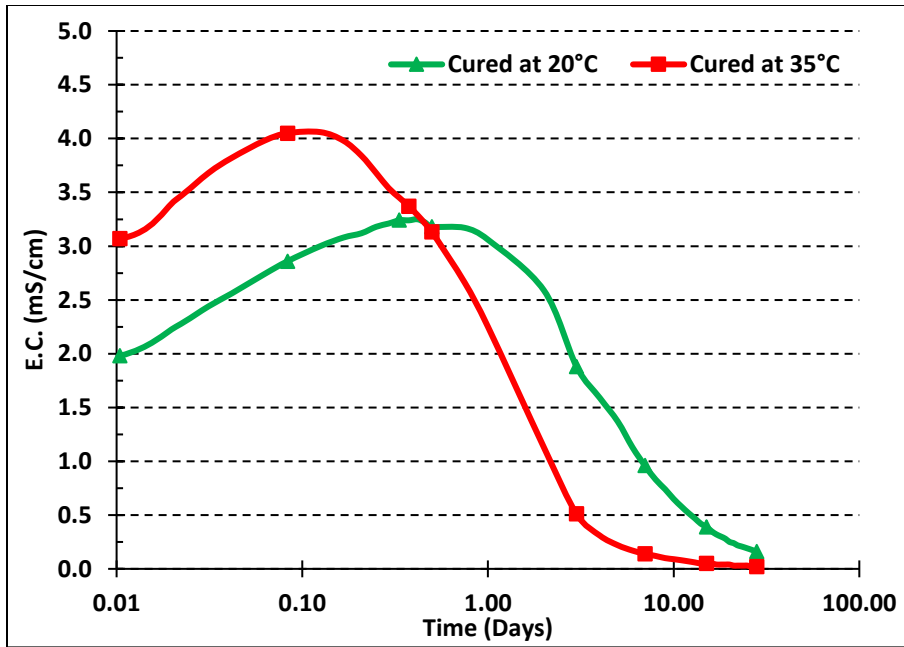


Figure 4.6 EC of the 28 days Slag-CPB samples that contain 0.3% sodium silicate, cured at different temperatures of 20°C and 35°C.

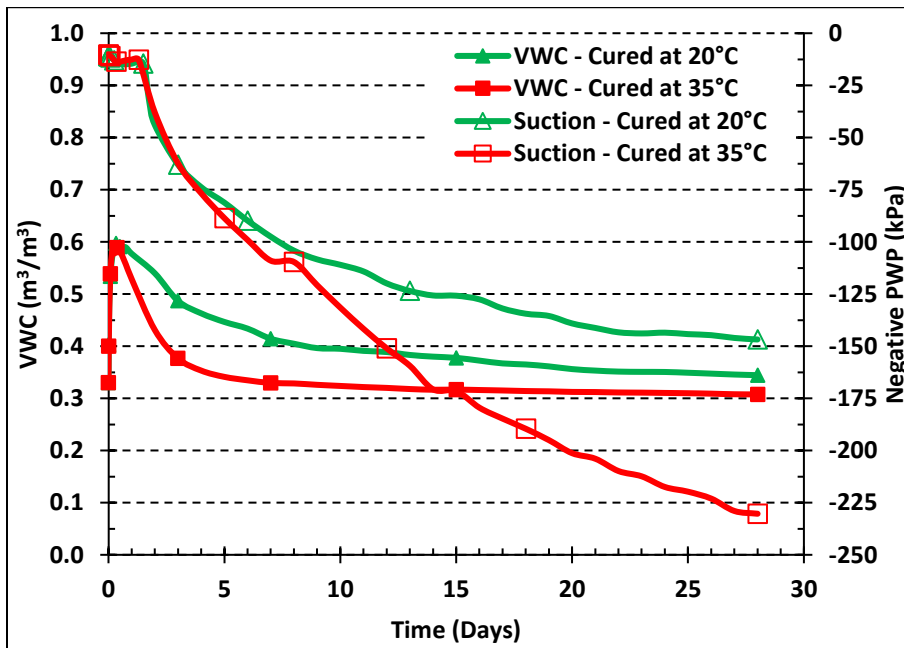


Figure 4.7 Coupled development of VWC and suction (negative PWP) of the 28 days Slag-CPB samples that contain 0.3% sodium silicate, cured at different temperatures of 20°C and 35°C.

4.4.2 Coupled effect of sodium silicate content and curing temperature on CPB strength.

The effect of sodium silicate content on the strength development of slag-CPB samples with respect to different curing temperatures is shown in Figure 4.8 (a and b). The increase in temperature has significantly increased the UCS of CPB samples regardless of the sodium silicate content, as shown in figure 8 (a and b). As discussed earlier, this is because of the high temperature-induced acceleration of cement hydration processes, which leads to more production of cement hydration products (Fall et al. 2010) and higher intensity of the self-desiccation within the CPB samples (Alainachi and Fall 2019c).

Moreover, it is evident from Figure 4.8 (a and b) that the increase in sodium silicate content increases the strength of CPB regardless of the temperature conditions. It is evident that at both testing temperatures, the UCS of CPB that does not contain sodium silicate (0.0% of sodium silicate) is lower than that of those samples that contain sodium silicate. Furthermore, it was found that the CPB strength increases with the increase in the sodium silicate content. Indeed, the increase in curing temperature of CPB that contains sodium silicate accelerates the role of sodium silicate as alkali activator (Živica 2007). The high temperature-induced increase in activation energy of sodium silicate is associated with the increase in absorption ability as a result of the stress-induced sodium ion migration (Souquet et al. 2010). Accordingly, it is clearly observed from Figure 8 (a and b) that the increase in UCS of CPB samples that contain sodium silicate was relatively higher when cured at high temperature (35°C) as compared to those that were cured at low temperature (2°C). It means that the sample with higher content of sodium silicate was more affected (activated) at higher temperature, and thereby showed higher mechanical strength. This finding can be attributed to two factors: (i) the effect of high temperature on enhancing the role of sodium silicate as an activator (as discussed above) (Živica 2007); and (ii) the setting time of slag pastes becomes relatively faster when exposed to high dosage of alkali activator (such as sodium silicate) (Azadi et al. 2017), which enhances the mechanical strength of CPB (Warner 2004, Azadi et al. 2017). This is also consistent with the findings of Kermani et al. (2015b), who found that the final setting time of sodium silicate-free CPB is around 7.0 hours (420 minutes), while it is around 6.3 hours (375 minutes) and 5.8 hours (350 minutes) for CPB samples that contain 0.3% and 0.5% of sodium silicate respectively.

This argument was experimentally confirmed by conducting microstructural analysis, including thermal analysis (Figure 4.9) and MIP analysis (Figure 4.10). In Figure 4.9, the TG/DTG diagrams of two slag-CPB samples, which contain 0.0% and 0.3% of sodium silicate, and were cured (up to 7 days) at the temperature of 35°C, are presented. These diagrams confirm that the presence of sodium silicate in CPB samples increases the cement hydration and generates more amounts of hydration products.

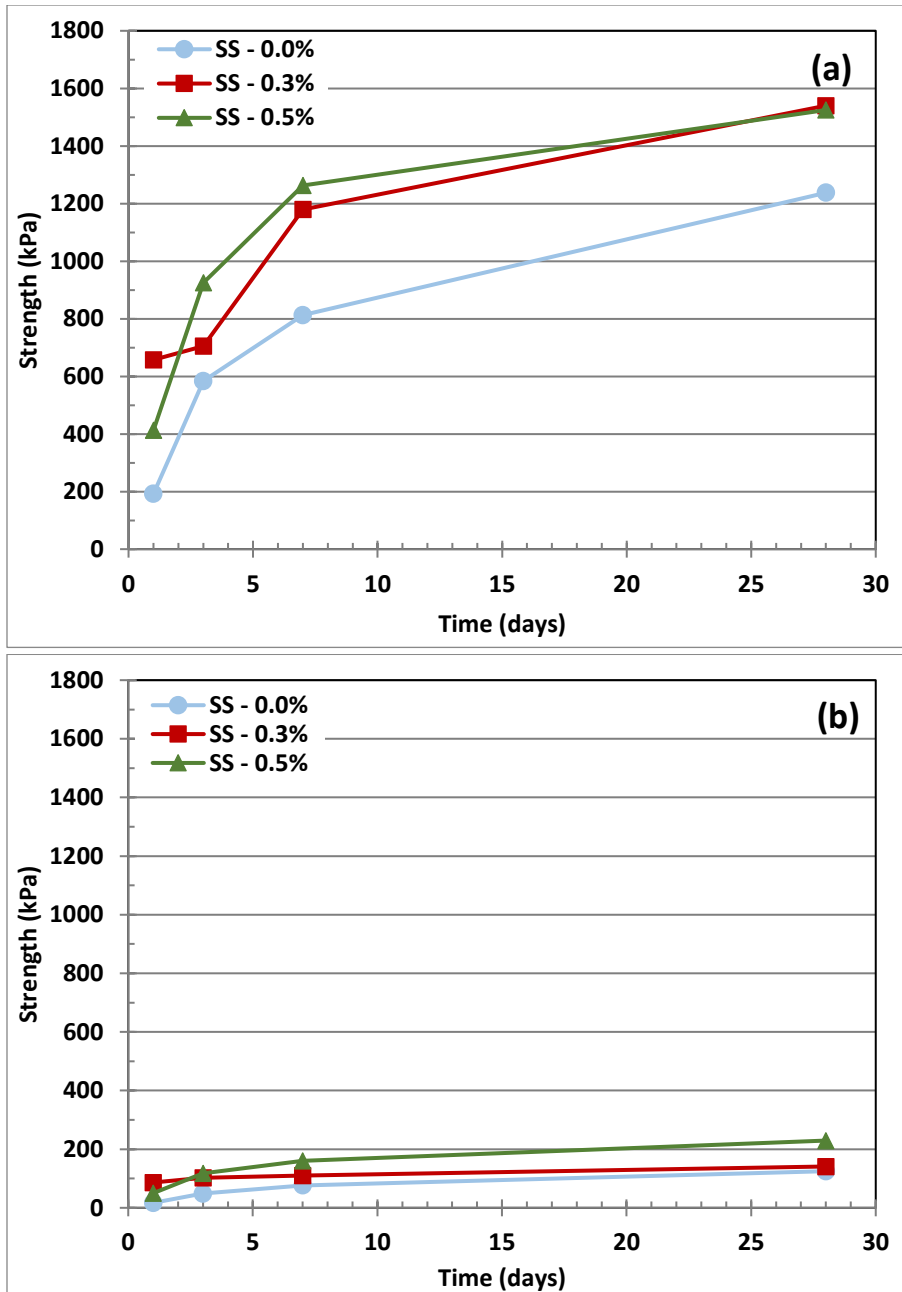


Figure 4.8 Effect of temperature and sodium silicate content on the strength development of Slag-CPB that contain sodium silicate and cured at: (a) 35°C; (b) 2°C.

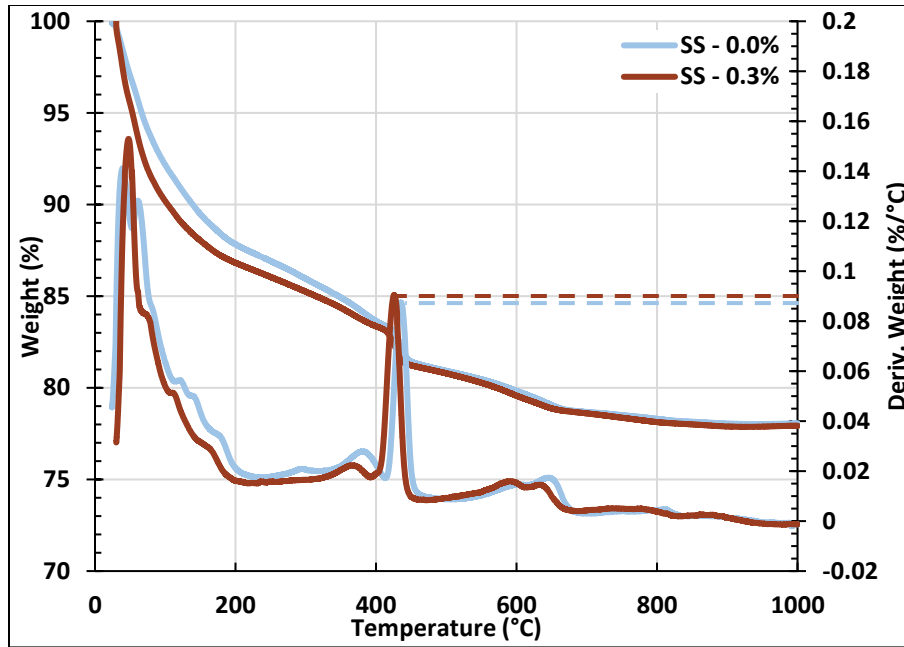


Figure 4.9 Effect of sodium silicate content on TG/DTG diagrams for 7 days Slag-CPB specimens that cured at 35°C.

It can be seen from the graphs in figure 4.9 that the sudden change in weight (TG curves) indicates highest weight loss from the CPB sample that contains 0.3% of sodium silicate as compared to the sodium silicate-free samples. In contrast, it is evident from the first peak in the DTG curves, which occurred at about 100°C, that the amount of cement hydration products (mainly C-S-H, ettringite, and gypsum) was higher in the samples that contain sodium silicate as compared to the sample that was prepared without sodium silicate. Moreover, the second peak, which occurred at about 450°C, shows more production of CH within the slag-CPB samples that contain 0.3% sodium silicate as compared to the slag-CPB samples of 0.0% of sodium silicate.

Figure 4.10, on the other hand, shows the differential pore size distribution of two slag-CPB samples that were prepared with and without sodium silicate, and were cured (up to 7 days) at the temperature of 35°C. It is observed that due to the presence of sodium silicate, the threshold pore diameter slightly decreases. In other words, the presence of sodium silicate will lead to a slight refinement in the pore structure of the cementitious material as a result of the larger amount of hydration products, which will improve the mechanical strength of CPB (Aldhafeeri et al. 2016, Haruna and Fall 2020).

Furthermore, the EC monitoring results in Figure 4.11 support this assertion. It can be observed from this figure that the EC within the CPB samples prepared with sodium silicate and cured (up to 28 days) at 35°C reached its maximum value faster than the sodium silicate-free CPB samples. In addition, the peak EC values of CPB samples that contain sodium silicate were much higher than the peak EC values of samples prepared without adding sodium silicate. The CPB samples that contain sodium silicate reached the peak of around 4.0 mS/cm within around 3.0

hours, while the one that does not contain sodium silicate reached the peak of 3.6 mS/m in around 4.0 hours. It provides a clear indication of the rapid increase in the rate of cement hydration due to the presence of sodium silicate and the lower concentration and lower mobility of ions within the sodium silicate-free CPB samples (Li and Fall 2016, Fang and Fall 2018).

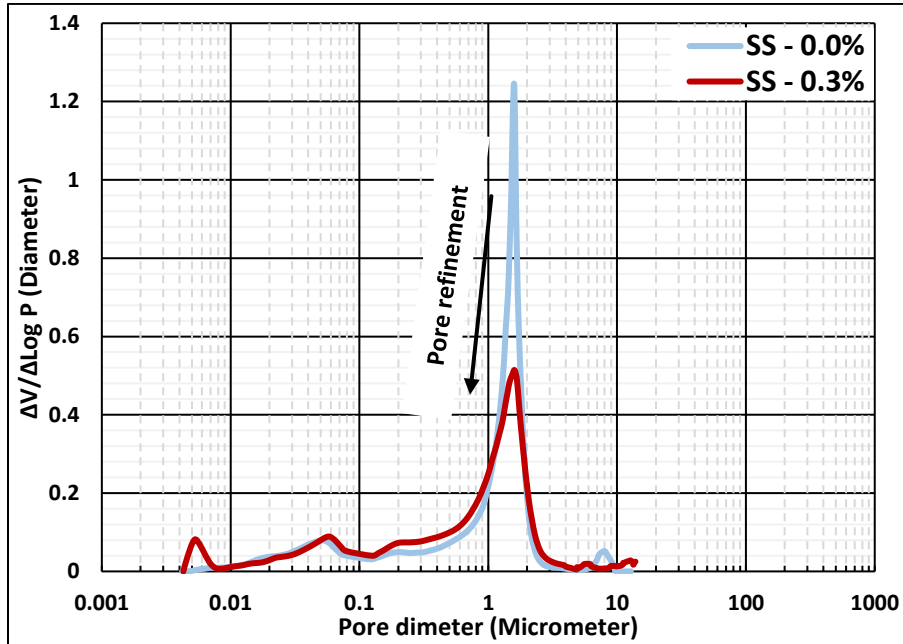


Figure 4.10 Effect of sodium silicate content on the differential pore size distribution for 7 days Slag-CPB specimens that cured at 35°C.

Additionally, Figure 4.12 presents the coupled evolution of suction and volumetric water content (VWC) of two slag-CPB samples that were prepared with and without sodium silicate and were cured (up to 28 days) at 35°C. The results presented in Figure 4.12 advocate these findings. These results showed that there was a rapid decrease in VWC of both samples. However, the reduction in VWC was higher within the CPB sample that contains sodium silicate. On the other hand, evolution of the negative PWP (suction) was dramatically higher within the CPB sample that contains sodium silicate as compared to the one that does not contain sodium silicate. It can be explained as the role of sodium silicate in activating the slag. Hence, the cement hydration process will be accelerated and will speed up the cement self-desiccation. It will consequently reduce the volumetric water content within the CPB pores and decrease the water content and pore-water pressure (PWP), and will consequently generate the negative pore-water pressure (suction) and increase the mechanical strength of CPB (Bentz 2008, Abdul-Hussain and Fall 2012). Accordingly, this fact provides an additional support to the idea of the effect of the presence of sodium silicate in accelerating the cement hydration process, and consequently improving the mechanical strength of CPB samples.

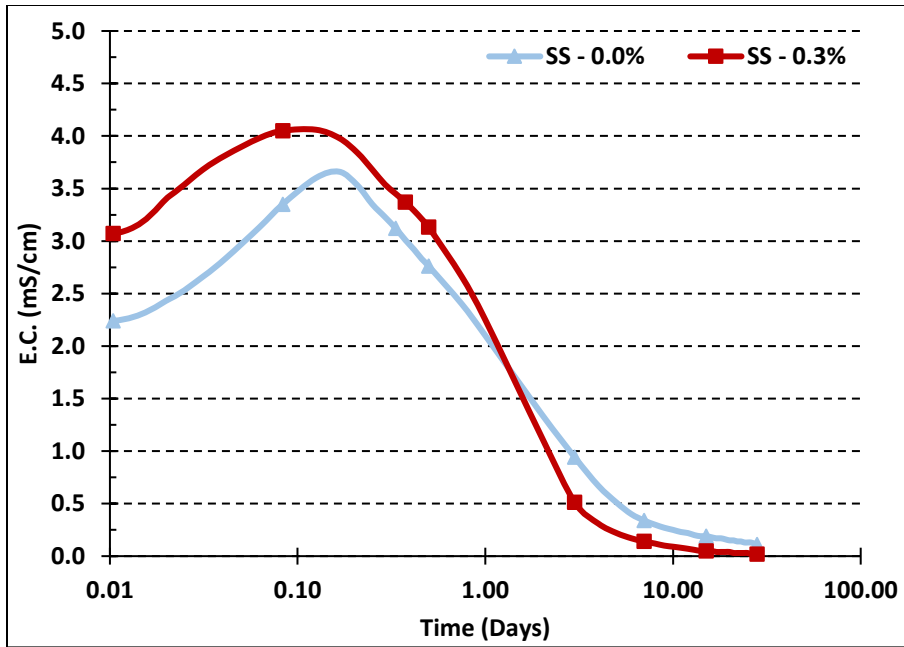


Figure 4.11 Effect of sodium silicate content on the EC of 7 days Slag-CPB specimens that cured at 35°C.

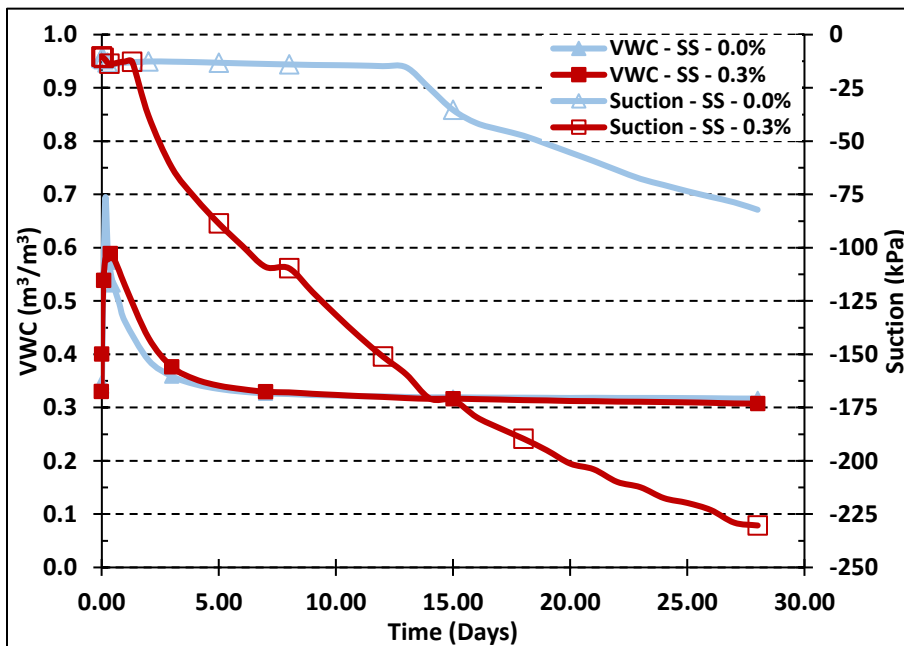


Figure 4.12 Effect of sodium silicate content on the VWC and suction of 7 days Slag-CPB specimens that cured at 35°C.

4.5 Summary and Conclusion

This manuscript mainly studied the combined effect of temperature and sodium silicate content on the time-dependent development of mechanical strength (UCS) of CPB material at early ages (up to 28 days). Thus, the study revolves around two main axes:

- a. The effect of temperature on the strength of Slag-CPB material that contains 0.3% of sodium silicate.
- b. The effect of temperature on the strength of Slag-CPB material that contains different dosages of sodium silicate (0.0%, 0.3%, and 0.5% of the total dry weight).

A total of 72 CPB specimens were prepared at different temperature (2°C, 20°C, 35°C), and were subjected to UCS tests. To better understand the reason behind the nature of the results observed, these samples were also subjected to microstructural analysis (including thermal analysis, and MIP), monitoring of electrical conductivity, volumetric water content, and suction. The major conclusions, based on the results of this research, can be summarized as:

1. Generally, the strength of slag-CPB, regardless of temperature and/or content of sodium silicate, increases with the progress of curing time. However, the rate of increase differs with the change in any of the other conditions.
2. Increase in curing temperature accelerates the cement hydration progress and consequently increases the strength of slag-CPB material. The temperature-dependent increase is evident regardless of the content of sodium silicate. However, the amount of increase in the mechanical strength with temperature is affected by the change in sodium silicate content.
3. Adding sodium silicate to the CPB significantly increases the strength of CPB at any temperature.
4. The UCS increment is directly proportional to the increase in the dosage of sodium silicate regardless of the change in temperature. However, at high temperature (35°C), there is a significant increase in strength of slag-CPB samples that contain high dosage of sodium silicate (0.3% and 0.5%) as compared to those that do not contain sodium silicate.
5. The results of this research will contribute to more efficient and safer design of CPB structures in mining areas.

4.6 Data Availability Statement

All data, models, and code generated or used during the study appear in the submitted article.

4.7 Acknowledgements

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CHAPTER 5

Synthesis and Integration of Results

5.1 Introduction

The results obtained from the two technical papers, presented in Chapters 3 and 4 of this thesis, have been synthesized in this Chapter. Each of these papers has investigated the combined effect of several factors on the rheological properties and mechanical strength of fresh CPB material as shown in Table 5.1. Table 5.2 summarizes the experimental tests that were conducted in each paper.

Table 5.1 Summary of effective factors that were investigated in this thesis

Chapter	Technical Paper	Initial Temperature			Curing Time								Binder Blend			Sodium Silicate Content					
		2°C	20°C	35°C	0.0 hrs.	0.25 hrs.	1.0 hrs.	2.0 hrs.	4.0 hrs.	1 days	3 days	7 days	28 days	PCI	PCI/Slag	PCI/FA	0%	0.1%	0.3%	0.5%	
3	1	×	×	×	×	×	×	×	×					×	×	×	×	×	×	×	
4	2	×	×	×						×	×	×	×		×			×		×	×

PCI: Portland Cement Type I;

Slag: Blast Furnace Slag

FA: Fly Ash

Table 5.2 Summary of experimental tests conducted in this thesis

Chapter	Technical Paper	Rheological Properties		UCS	pH	Zeta Potential	Microstructural Analysis			Monitoring Tests		
		Yield Stress	Viscosity				TG/DTG	XRD	MIP	EC	Suction	VWC
3	1	×	×		×	×	×	×		×		
4	2			×			×		×	×	×	×

UCS: Unconfined Compression Strength; TG/DTG: Thermal Analysis; XRD: X-ray Diffraction;

MIP: Mercury Intrusion Porosimetry; EC: Electrical Conductivity; VWC: Volumetric Water Content.

Section 5.2 of this chapter presents the representative results from Chapter 3 to show the combined effect of the curing time, initial temperature, binder type, and sodium silicate dosage on the rheological properties (yield stress and viscosity) of CPB samples that were: (i) cured for different curing time (0 hrs, 0.25 hrs, 1 hr, 2 hrs, and 4 hrs), (ii) mixed and cured at different temperatures (2°C, 20°C, and 35°C), (iii) prepared with binder blends of 100% PCI, 50%PCI-50%Slag, and 50%PCI-50%FA, and (iv) contained different dosages of sodium silicate (0%, 0.1%, 0.3% and 0.5%). Afterwards, Section 5.2 presents the main results obtained from Chapter 4 which investigated the combined effect of curing time, curing temperature, and content of sodium silicate on the mechanical strength of fresh CPB by conducting the UCS tests on Slag-CPB samples that were cured for 1 day, 3 days, 7 days, and 28 days under different curing temperatures of 2°C, 20°C, and 35°C), and contained 0%, 0.3% and 0.5% of sodium silicate.

5.2 Effect of Time and Temperature on the Rheological Properties of Cemented Paste Backfill that Constrains Sodium Silicate

In Chapter 3, the effect of cement hydration progress (curing time), initial (mixing and curing) temperature, binder type, and dosage of sodium silicate on the rheological properties (yield stress and viscosity) of fresh CPB samples were assessed. Yield stress and viscosity tests, pH and zeta potential tests, microstructural analysis (TG/DTG and XRD), and electrical conductivity monitoring tests were conducted on 125 CPB samples that were prepared with different binder blends (PCI, PCI/Slag and PCI/FA) and contained different dosages of sodium silicate. These samples were prepared and cured under different temperatures of 2°C, 20°C, and 35°C and were cured for 0 hrs, 0.25 hrs, 1 hr, 2 hrs, and 4 hrs of curing time.

It has been observed that the rheological properties of CPB increase with the increase in curing time owing to the progress of cement hydration process with time. They also increase with the increase in initial temperature because the high mixing and curing temperature accelerates the cement hydration process and produces more cement hydration products. In addition, they increase with the increase in sodium silicate content due to the increase of sodium silicate role in activating the pozzolanic compounds (slag and FA) and accelerating the hydration of the clinker and binder. It was also found that the partial usage of slag or FA as a binder with PCI reduces the yield stress and viscosity (enhances the flowability) of the CPB samples with sodium silicate. The enhancement in the flowability of CPB with the partial usage of FA was more evident than the case of the partial usage of slag. These understandings of different behavior of the rheology of CPB can help in designing CPB mixtures with better flowability and workability, and thus increase the efficiency of these designs.

It is commonly known that 10% - 20% of the total cost of the mining operations in underground mines are related to the cost of the backfilling processes. However, 70% of the backfill system failures were found to be attributed to the failure in backfill delivery system caused by the transportation of backfill material that does not have appropriate flowability, leading to possible flow delays and/or temporary interruption of the progress of CPB production as well as

possible replacement of the pipelines (Grice 1998, Fehrsen and Cooke 2006, Haruna and Fall 2019). So, understanding the flow behavior of backfill can help in saving large amount of the possible related financial losses. In addition, shortening the time required to deliver the backfill into the mine stope can reduce the cost of the backfilling process by allowing the mine operations to be conducted at faster rate. Therefore, many mines have implemented several approaches to accelerate the backfill transportation, such as increasing the flow rate, increasing the fill slump by adding water, and using pipelines of high diameters, and/or using high-pressure pumps. Nevertheless, these approaches might have negative impacts on the backfill process. For instance, increasing the fill flow rate has a great influence on the pressure gradient and causes higher stresses to be applied to the walls of pipelines and/or the stope barricade, which might exceed their capacities and cause damages to the pipelines, and/or failure of the stope barricade. Also, adding water may result in increasing the water-cement ratio of the fill mix, and thereby compromising the desired strength. Despite the advantage of using pipes of higher diameter and/or using the high-pressure pumps, such as accelerating the delivery process and avoiding the laminar flow setting (which occurs while transporting viscous material), this approach may have significant cost implication in some situations, especially while mining small size mines (Clark et al. 1995, Fehrsen and Cooke 2006, Paterson 2012).

Accordingly, the results obtained in this study show some technical implications in the practice of the backfill technology by understanding the factors that influence the flow behavior of the backfill, and thus reducing the risk of failure in backfill delivery system in addition to using cement-slag or cement-FA binder blends that reduce the yield stress and viscosity of the fill mixture, which will accelerate the backfill process without damaging the pipes and/or risking the stability of the stope barricade, and without compromising the desired strength by adding sodium silicate to the mixture. Moreover, these implementations can also help in avoiding laminar flow setting without having to afford the high cost of using high-diameter pipes and/or high-pressure pumps.

5.3 Effect of Temperature on the Strength Development of Cemented Paste Backfill that Constrains Sodium Silicate

In Chapter 4, the combined effect of curing time and temperature on the mechanical behavior of slag-CPB that contains sodium silicate has been studied by conducting UCS tests, microstructural analysis (TG/DTG and MIP), and monitoring tests (electrical conductivity, suction and volumetric water content) on 72 CPB samples that were prepared with 50%PCI-50%Slag blend, cured for 1, 3, 7, and 28 days under different curing temperatures (2°C, 20°C, and 35°C), and contained sodium silicate of different content (0%, 0.3% and 0.5%).

It has been found that the strength of slag-CPB samples increases with time regardless of the variation of temperature and/or sodium silicate content because of the progress of cement hydration process with time. However, the rate of increase in the strength of CPB increases with the increase in curing temperature and the increase in the dosage of sodium silicate. This is

attributed to the acceleration of cement hydration process and increase in the generation of the cement hydration products with the increase in curing temperature as well as the role of sodium silicate in activating the slag and thus accelerating the binder hydration, which will then increase the bonds between the CPB particles. These results may help in designing better performing and safer CPB structures.

In addition, the obtained results may have some practical implications in the CPB technology that have been described briefly below:

5.3.1 Mechanical Stability of CPB Structures

One of the most important goals of the CPB technology is to provide an adequate ground support against the mining-induced geotechnical issues, such as the ground subsidence and the instability of the mine stope, which might danger safety of the mine workers and the surrounding area. To achieve the desired mechanical performance of CPB, it is imperative to maintain the mechanical stability or strength of the CPB structure in high standards. For instance, it is indicated that the required UCS for the CPB in a common underground mining operation is 0.7–2 MPa (Brackebusch 1995, Fall et al. 2010). However, this UCS widely changes, depending on the sizes of the CPB mass, application or role of the backfill structure and mine characteristics. Therefore, assessing the factors that affect the mechanical strength of CPB, potentially the UCS, is of high importance. These factors, as described in this thesis, include the combined effect of temperature, and the usage of sodium silicate. Several researchers have studied the UCS as a function of the stope size, which is also called the critical compression strength (UCS_c). The determination of UCS_c can help evaluate the mechanical stability in faster and less expensive way. Mitchell (1983) determined the critical UCS for a free standing 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. Figure 5.1 shows the variation of the critical strength of CPB with respect to the stope size.

As shown in Figure 5.1, the UCS_c of a stope size of 60 m (H) \times 40 m (L) is around 500 kPa. By analyzing these values with the UCS values of CPB obtained from this study (Figures 4.3 and 4.8 in Chapter 4 of this study), it can be seen that the UCS of 28 days-aged CPB material that does not contain sodium silicate and was cured at low temperature (2°C) was around 100 kPa, while by increasing the curing temperature to 35°C, the 28 days-UCS reached a value of around 1200 kPa. Moreover, by adding 0.3% of sodium silicate to the mixture and curing it at 35°C, the 28 days-UCS value was more than 1500 kPa. In other words, high curing temperature or adding sodium silicate will enhance the strength of CPB, and combining the high curing temperature and sodium silicate will significantly increase the strength of CPB. Owing to these findings, CPB that contains sodium silicate and was cured at high temperature will have higher mechanical strength as compared to the CPB that does not contain sodium silicate and was cured at low temperatures.

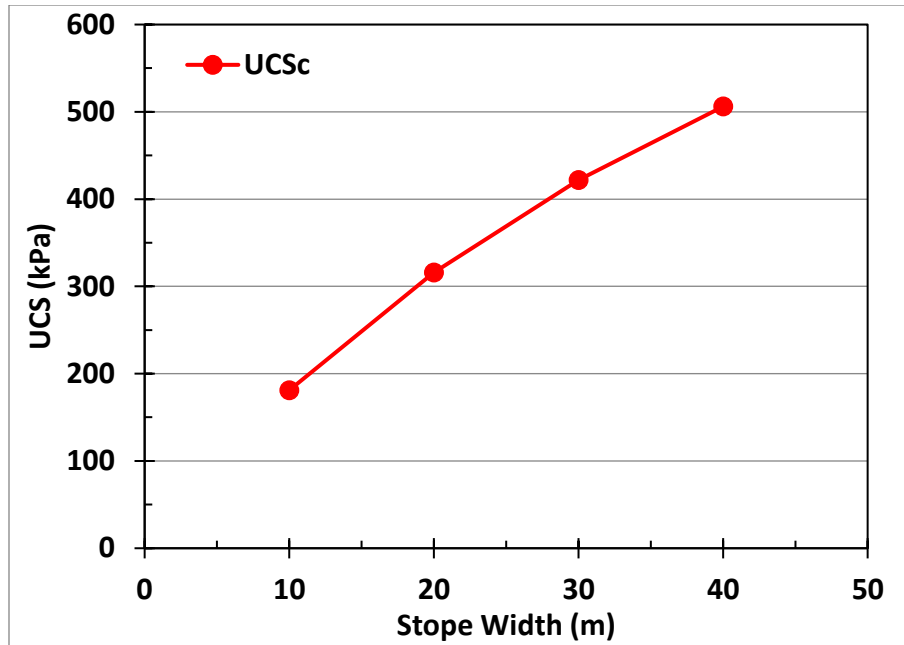


Figure 5.1 Critical UCS of CPB with respect to the width variation of a 60 m height stope.

5.3.2 Barricade Stability

Before completing the filling process of CPB, retaining wall structure (named stope barricade) must be constructed at the bottom of the mine stope (at the entrance of the mine drift) in order to hold the CPB in place. To ensure the safety of the workers of the mine as well as the equipment, it is imperative to ensure that the stresses applied by the fresh backfill does not exceed the barricade capacity (strength). The applied stresses on the barricade was found to be directly associated to the horizontal total stress and PWP of the backfill material. It was also noted that these stresses are significantly influenced by temperature increase due to cement hydration reaction, as this increase in temperature will accelerate the cement hydration process and reduce the PWP acting on the barricade (Ghirian 2016).

The result obtained from the current study revealed that the high curing temperature will accelerate the cement hydration process, produce more cement hydration products, and reduce the water content (and consequently reduce the PWP and total stress) of the CPB material. It was also found that using sodium silicate as a chemical additive will activate the hydraulic binder, particularly the Slag, and release more of the hydration heat. This will significantly increase the hydration temperature, accelerate the hydration process, and consequently reduce the PWP and total stress applied to the barricade. These findings may be taken into consideration during the backfill design to help mine operators work beside a stable barricade, and thereby in safe work environment.

5.3.3 Mining Cycles

From financial point of view, the total cost of the mining operation highly depends on the mining cycle time (stope cycle), which represents the time required to complete the whole process in a single mine stope. Longer mining cycle of a mine stope causes a delay in mining the adjacent ore reserves. Shortening the mining cycle time, on the other hand, will allow the adjacent stope to be mined, so the mining productivity will be enhanced, more cost savings, and consequently more financial revenues will be profited from the mine. Rather than accelerating the filling rate which might affect the stability of the stope barricade, continuous pouring of backfill into the mine stope can help in reducing the operation cost and mining cycle time without risking the stability of the stope barricade. Thus, the mine operators will be able to develop optimal stope cycle for opening barricades and proceed with mining sequences. However, adopting continuous pour process necessitates significant evolution of backfill strength, especially at the early hours (or days) of curing to avoid the risk of backfill and barricade failure.

The enhancement in strength evolution of CPB at early age by adding sodium silicate and curing at high curing temperature, shown in the current study, can be correlated with the critical UCS (UCS_c) as mentioned in section 5.3.1. For instance, the UCS_c of a stope size of 60 m (H) \times 40 m (L), as shown in Figure 5.1, is around 500 kPa, and by analyzing the UCS values of CPB obtained from this study (Figures 4.3 and 4.8 in Chapter 4 of this study), it can be seen that the UCS of CPB material that does not contain sodium silicate and was cured at 2°C did not reach more than 100 kPa in 28 days of curing time, while by curing the same material at 35°C, the UCS reached the value of 500 kPa in only 3 days of curing time. This is because the high curing temperature was known to accelerate the cement hydration process. Moreover, by adding 0.3% of sodium silicate to the mixture and curing it at 35°C, the UCS value exceeded the critical UCS of 500 kPa and reached a UCS of around 650 kPa in less than one day of curing time. This is related to the fact that the presence of sodium silicate will shorten the setting time of cemented material. In other words, high curing temperature or adding sodium silicate will enhance the strength of CPB, and combining the high curing temperature and sodium silicate will reduce the mine cycle time by significantly increasing the UCS of CPB in very short time, and thereby the mine productivity will potentially be improved.

5.3.4 Binder Consumption

Despite the fact that the binder used in CPB preparation might typically cost around 75% of the backfilling process, it is common practice in many underground mines to use high binder content within the fill in order to accelerate its strength development, especially while placing the first 2-3 m thick layer (named Plug fill) (Grice 1998, Yilmaz et al. 2015). Accordingly, an obvious cost saving can be significantly achieved by optimizing the binder consumption. In this study, it was revealed that the UCS of CPB cured at high curing temperature with the presence of sodium silicate is shown to result in significant improvement in the mechanical strength of the backfill. This approach can help reduce the cost of mining operation by reducing the binder usage, and thus improve the mine productivity.

5.4 References

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CHAPTER 6

Conclusions and Recommendations

6.1 Conclusions

Following findings have been drawn from the results obtained in this study: -

- There was a gradual increase in rheological properties (yield stress and viscosity) of CPB with progress of curing time, regardless of the type of the hydraulic binder, initial temperature, and/or dosage of sodium silicate.
- The increase in initial temperature will significantly increase the rheological properties, and thus reduce the flowability of CPB.
- Yield stress and viscosity of CPB increase with the increase in the sodium silicate content.
- The combination of high initial temperature and high sodium silicate content significantly accelerates the rate of increase in the rheological properties of CPB.
- The partial usage of Slag or FA enhances the flowability of CPB by reducing the yield stress and viscosity.
- Using PCI-FA blend as binder in CPB that contains sodium silicate better enhances the flowability of CPB as compared to CPB using the PCI-Slag blend.
- The mechanical strength of CPB that is prepared with PCI-Slag blend as binder increases with progress of curing time, regardless of the variation of curing temperature and/or dosage of sodium silicate.
- Slag-CPB samples that contain sodium silicate showed higher UCS values, and thus higher strength, when cured at high temperature (e.g. 35oC), and significantly lower UCS values when cured at low temperature (2oC).
- Adding sodium silicate to the slag-CPB potentially increases the strength of slag-CPB at any temperature, as the sodium silicate activates the pozzolanic binder (slag).
- High dosage of sodium silicate significantly increases the UCS of slag-CPB, especially when cured at high temperature.
- The results obtained in this research can contribute to efficient, cost effective, and safer design of CPB structures in mining area.
- The obtained results can reduce the financial losses associated with unsuitable flowability of CPB transported in the CPB delivering system.

6.2 Recommendations

Taking into consideration the time limitations of the experimental tests conducted in this study, the following recommendations are proposed for future works.

- Investigating the combined effect of curing time, initial temperature, binder type, and dosage of sodium silicate on the rheological properties and strength development of CPB material that contains different chemical elements, such as sulphate, sodium chloride.
- Investigating the combined effect of curing time, binder type, and dosage of sodium silicate on the rheological properties and strength development of CPB material that is prepared and cured at sub-zero temperatures.
- Investigating the combined effect of the tested conditions on the rheological properties and strength development of CPB prepared with different mix design than those applied in this study, such as:
 - ✓ Using natural tailings instead of silica tailings
 - ✓ Using mine processing water instead of fresh (tap) water
 - ✓ Water-to-cement ratio that is different than the one used in this study
 - ✓ Different binder blends and/or content than those used in this study
- Studying the combined effect of curing time, temperature, and binder type/content of CPB that contains different chemical additives or activators, such as superplasticizer, or sodium hydroxide etc. on the rheological properties and/or strength development of CPB.