

**Strength and environmental properties of cemented paste backfill that
contains sodium silicate**

By

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To my parent and my son

ABSTRACT

Mining is an important industry that plays a significant role in the development of human civilization and economies. However, the underground mining process produces a large volume of mine wastes (e.g., tailings) as well as creates large voids that require filling, typically with an engineering backfill material. Filling the voids with mine waste materials provides an environmental-friendly way of disposing mining waste. It is also an effective way of increasing ore recovery and improving the safety of miners. One of the best technique of mine backfill is called cemented paste backfill (CPB), which is typically a mixture of tailings, binder and water.

The most common binder used in the preparation of CPB is Portland cement (PC). PC is not only a costly binder, but its production is highly energy-intensive and also generates a large amount of CO₂. The cement consumption can represent up to 75% of the cost of CPB. These above mentioned factors have compelled mining companies to seek for cement alternatives that enhance the engineering properties of the CPB, decrease the cement content and reduce the carbon footprint of the mining industry. Sodium silicate is the most recent chemical additive that is proposed to reduce the binder content in CPB. Sodium silicate is an alkaline solution that is used to activate a pozzolanic material, such as cement, slag and Fly ash. However, the effect of sodium silicate on the strength and key environmental properties (permeability or saturated hydraulic conductivity, reactivity) of CPB is not well understood.

The objective of this thesis is to investigate the possibility of using sodium silicate as an activator in cemented paste backfill and obtain an improvement in the aforementioned engineering properties of CPB.

In order to determine the effect of the sodium silicate on backfill properties, some CPB testing methods were developed to fulfill the objectives of this research. Thus, the evolution of hydraulic, mechanical and microstructural properties of CPB samples containing sodium silicate (SS-CPB) have been tested or monitored at different curing ages (1, 3, 7, 28 and 90 days) and different CPB mixtures as well. The results of these studies show that activating CPB with sodium silicate develop CPB strength faster than CPB

samples without sodium silicate. In addition, hydraulic conductivity and reactivity results show a positive change in samples containing sodium silicate compared to free sodium silicate CPB samples. Indeed, this activation leads to decreasing permeability and reactivity due to the formation of cement hydration products and acceleration of the binder hydration process. Moreover, binder type and content in the presence of sodium silicate as an alkali activator in the CPB play a significant role in lowering hydraulic conductivity and reactivity of CPB.

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Chapter 1: General Introduction

1.1. General statement

The mining industry has been significantly impacting the economic and social development of many countries and regions in the world. However, mining activities generate enormous quantities of waste, particularly tailings that must be disposed of. Tailings are byproducts or (toxic) man-made soils (ground rock and process effluents) that remain following the mine extraction and recovery processes of valuable minerals (Fall and Samb, 2009; Ghirian and Fall, 2014). Large volumes of tailings are produced by the mining activities every year. Thus, tailing disposal arise a significant concern of any mining operations. It is difficult to assess the actual volume of tailings produced. However, for example, gold mining in South Africa led to the production of $7.4 \times 10^4 \text{ t a}^{-1}$ of gold tailings from 1997 to 2006 (Letcher and Vallero, 2011).

The tailings have been traditionally disposed on the earth surface in tailings disposal facilities (e.g., dams, ponds and other types of surface impoundments). However, the surface tailings management methods can lead to geotechnical (e.g., failure of tailings dam) and environmental (acid mine drainage, release of heavy metals in the biosphere by the tailings) disasters with major economic and social consequences (Fall et al. 2009; Wang et al. 2016). In order to eliminate or minimize the risks associated with the surface tailings management methods as well as increase the mine productivity, the new technology of mine backfilling has been developed and adopted in the past decades. Mine backfilling consists of recycling the tailings into construction materials to fill the underground voids (stopes) generated by the ore extraction. It is considered as a novel technology to manage mine wastes (Yilmaz and Fall, 2017).

Among the main different types of mine cemented backfills (rockfill, hydraulic backfill, cemented paste backfill), cemented paste backfill (CPB) is the most recent and novel technology of mine backfilling. It has

become important in mining operations around the world due to its technical, economic and environmental advantages (Hassani and Archibald, 1998). CPB is a cementitious material produced by mixing tailings with a solid percentage between 70% and 85%, water that is either fresh or mine processed, and a hydraulic binder, which is usually 3% to 7% by weight. These components are usually combined and mixed on the surface and transported (by gravity and/or pumping) to the underground mine voids (stopes), where the CPB can be used for underground mine support, the maximization of ore recovery, and tailings waste management. The key performance properties of CPB include mechanical stability (usually evaluated by the strength), cost (depends on the binder consumption), environmental (commonly measured by its permeability and reactivity) performance. Cement or a blend of different binders, such as Ordinary Portland cement (OPC), Fly ash (FA) or Ground Blast Furnace Slag (Slag) can be used to provide the paste with the required mechanical strength. However, the cost of CPBs is significantly affected by the binder content. The binder represents up to 75% of the cost of cemented paste backfill (Fall et al., 2005). Moreover, production of OPC is highly energy-intensive and thus generates a large amount of CO₂. These factors mentioned above have compelled mining companies to seek for alternatives to cement that improve the strength of the CPB, decrease the cement content and reduce the carbon footprint of the mining industry. Sodium silicate is one of the most recent chemical additives that is proposed to reduce the binder content in CPB and increase its strength. However, the strength development and environmental properties (permeability, reactivity) of CPB that contains sodium silicate are not well understood.

1.2. Objectives and scope

Recently, there have been some publications (as discussed later) related to applying sodium silicate in mine backfill. Although the results of those studies are valuable, there is still lack in some aspects of using sodium silicate as an alternative for CPB structure. The main goal of this thesis is to address the effect of sodium

silicate on cemented paste backfill and provide better understanding on the specific objectives, as summarized below:

1. To investigate the strength development of CPB that contains sodium silicate and compare it with free sodium silicate CPB.
2. To study the evolution of the hydraulic conductivity (permeability) of CPB containing sodium silicate. Permeability is one of the most important parameters affecting environmental performance and durability of CPBs.
3. To study and evaluate the chemical reactivity as another environmental property of CPB with sodium silicate.
4. To study mechanical and environmental properties of CPB containing sodium silicate with different binder type and content.

1.3. Organization of manuscript

The thesis has been organized into 5 chapters as shown in Figure 1.1. **Chapter 1** is a general introduction, which includes problem statements, objectives, and description of the organization of the thesis. **Chapter 2** presents technical and theoretical background information on mining, backfilling technology (specifically cemented paste backfill and its main component and engineering properties), and reactivity of mine backfill and the different methods to measure it. Moreover, literature review on previous studies that address strength and properties of CPB is also presented in this chapter. **Chapter 3 and 4** are organized into paper-based thesis format so each chapter includes abstract, introduction, experimental methods, result and discussion and at the end conclusion. Therefore, chapter 3 specifically describes the strength development of slag-cemented paste backfill that contains sodium silicate. In this chapter, several comparisons have been done to find out the effect of sodium silicate on CPB strength. Chapter 4 provides information about two main environmental properties, which include reactivity and permeability properties of cemented paste backfill that contains sodium silicate. **Chapter 5** gives the summary, conclusions as well as

recommendations for the future studies.

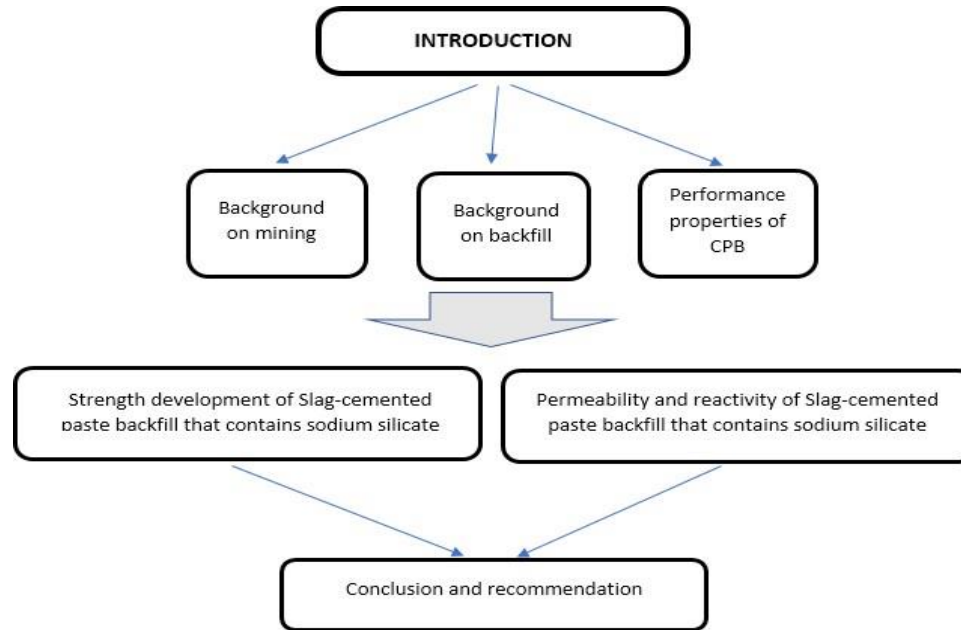


Figure 1. 1: Task and organization of the thesis

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Chapter 2 –Background and Literature review

2.1 Introduction

In order to obtain a small amount of valuable minerals from mining, a large amount of rock and soil is extracted from the ground (Roshani et al. 2017). Not only that the extracted material occupies a very large space on the earth when deposited on the surface, but mining activities also create large cavities in the ground. In the past decades, because of the increasing world population, the demand for mineral products has increased which resulted in a huge rise in the depth of excavations and the volume of mine wastes (waste rock, tailings) generated. The deep excavations create large and unsafe voids in the ground making mining operations more difficult to be conducted. Moreover, there are environmental laws in many countries banning surface disposal of mine wastes since the latter can harm the environment due to various toxic substances (e.g., heavy metals) that may exist in mine wastes, and the potential generation of acid mine drainage (AMD) (Orejarena and Fall, 2008, 2011; Li and Fall, 2016).

One of the effective solutions of managing this large volume of crushed rock (waste rock) and tailings produced by these mining activities as well as improving the stability of the cavities induced by mine excavations is recycling the mine waste into mine construction materials to fill the mine voids instead of disposing it on the surface. Filling voids created by excavation and sub-surface mining with waste material is called backfilling which is an important part of mining operations (Cui and Fall, 2019).

There are several techniques for recycling the mine waste through backfilling. These techniques include, rockfill, hydraulic backfilling and cemented paste backfilling. Cemented paste backfill (CPB) is the most modern and innovative mine backfilling methods. It consists of mix of water, mine tailings and a small percentage of binder (Ghirian and Fall, 2015). The most prevalent binder is Portland cement, which is used

in most mines around the world. Using CPB to fill the underground voids is an efficient method for disposing of a large amount of tailings, which would otherwise be dumped on the ground in predetermined locations (Brackebusch, 1994). Additionally, CPB can also act as a supporting structure that maintains the stability and provides safe environment for the miners.

The properties of the CPB depends on several factors, such as the type of the binder, the additives and the paste density (Fall et al., 2009, 2010). The high cost of cement has encouraged researchers to find other materials or additives that can reduce the cost of CPB while improving these engineering properties (Tariq and Yanful, 2013). For instance, one of the most popular additives is sodium silicate. Not only it has been used in producing concrete, but also recently, the use of this chemical admixture is increasing in backfilling materials (Kermani et al., 2015a).

To facilitate the understanding of the research issues addressed in this thesis and the results obtained the following topics are discussed in the present chapter: (i) mining and its different techniques; (ii) mine backfilling; (iii) Backfill binders; (iv) sodium silicate; (vi) main properties of CPB

2.2. Mining and its different techniques

In mining to extract ore from the bedrock, drilling, blasting, ventilation, loading and haulage, transporting to crushing and mineral process are the trends which is not changed from the 19th century (Ericsson, 2012). However, over centuries and during industrial revolution, the mining technology has gone through considerable progress. These developments including excavating process to waste depositing methods. Beside of it, the number of mines and quarries producing industrial minerals are increasing yearly. Generally, based on the mineral's type, mining mostly can be classified into open-pit and underground mining. According to Ericsson (2012), open-pit mining produce 83% of global metal ore and underground

technology producing 17% of the rest.

2.2.1 Open-pit mining

Open-pit, open-cast or open cut mining (Figure 2.1) is defined as a surface mining for extracting rock or minerals where access to the mineral is a way easier than other type of mining requiring tunneling. In surface mining, minerals are removed without a shaft or a drift. Open-pit mining is more attractive to mining companies due to permitting high recovery of the minerals in a short time and working conditions are safer for miners. However, producing a very large amount of waste makes this form of mining cross the environmental law and regulations.



Figure 2.1: Open-pit mines (Holdstock, 2014)

2.2.2 Sub-surface mining or underground mining

Underground extraction of minerals or sub-surface mining involves digging of tunnels or shafts into the ground to access excavated valuable ore. To process ore and to dispose the waste rock, tunnels and shafts are used to transfer ore and waste rock to the surface too (Hustrulid et al., 2017). Underground mining is necessary when the excavated materials are too deep to be done by open-pit mining or the quality of orebody is high enough to select the costliest mining technique. Variety of techniques can be designed in underground mining in order to select the most appropriate extraction methods and various options like operation challenges, cost and geotechnical interaction should be considered. As shown in Figure 2.2, the various techniques for underground mining are classified based on the nature of support to the unexcavated mass. They can be naturally supported, artificially supported, or completely unsupported.

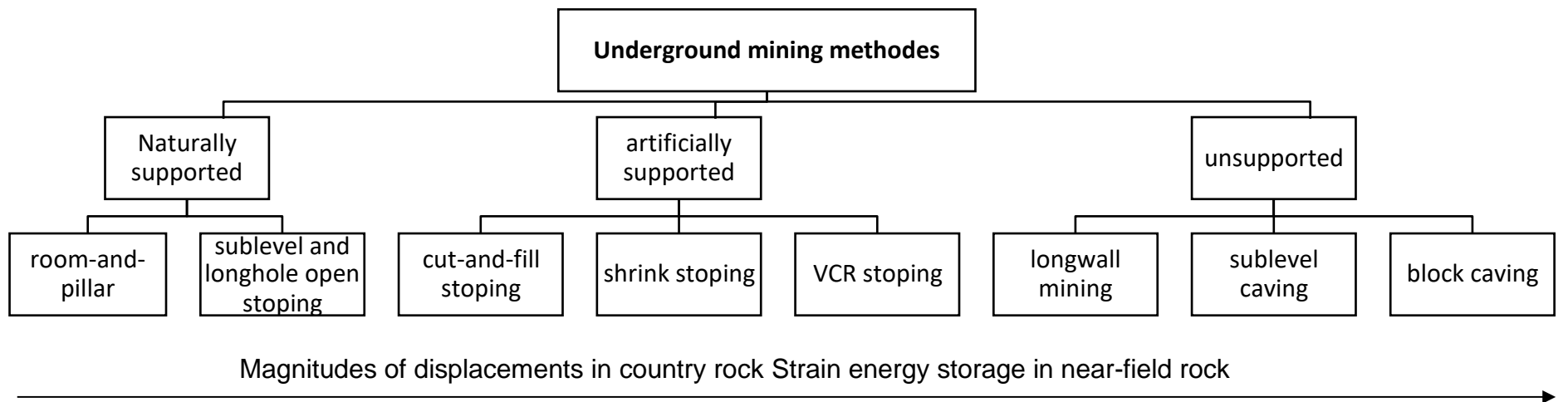


Figure 2.2: Work break-down structure for mining methods (Brady & Brown, 2005)

2.3. Mine Backfill

Mine backfilling refers to filling up underground voids with the waste material. Backfilling in mining dates back to the ancient period of Greek civilization when stone pillars were used for ground support. However, using the mine waste material as a backfill was known for the first time in 1862 at church in the USA by the Philadelphia & Reading Coal & Iron Company (Hassani and Archibald 1998a). Mining activities produce a huge amount of waste while only a small amount of it can be used as a valuable material. Thus, the result of mining practice is a large amount of waste and at same time creating a huge underground's voids. Backfilling is one of the best options with the purpose of disposing or creating a safe working condition as a support for underground mining (Nasir and Fall, 2009). In Canada, using this method is started from the last century to fill cavities, establish supports for poor underground and disposing mine waste materials (Nantel, 1998).

2.3.1 Backfill Types

Backfill can be classified into three main types: rock fill, hydraulic backfill and paste backfill. Rock fills and hydraulic backfills can be cemented or un-cemented, whereas paste backfill is commonly cemented. To select the suitable backfill type for a mine, several factors should be considered. These include grade of the ore bodies, mining depth, stope sequence and size/geometry, available backfill materials and operational costs (Landriault, 2001).

The typical grain size distribution of the three types of backfilling is illustrated in Figure 2.3.

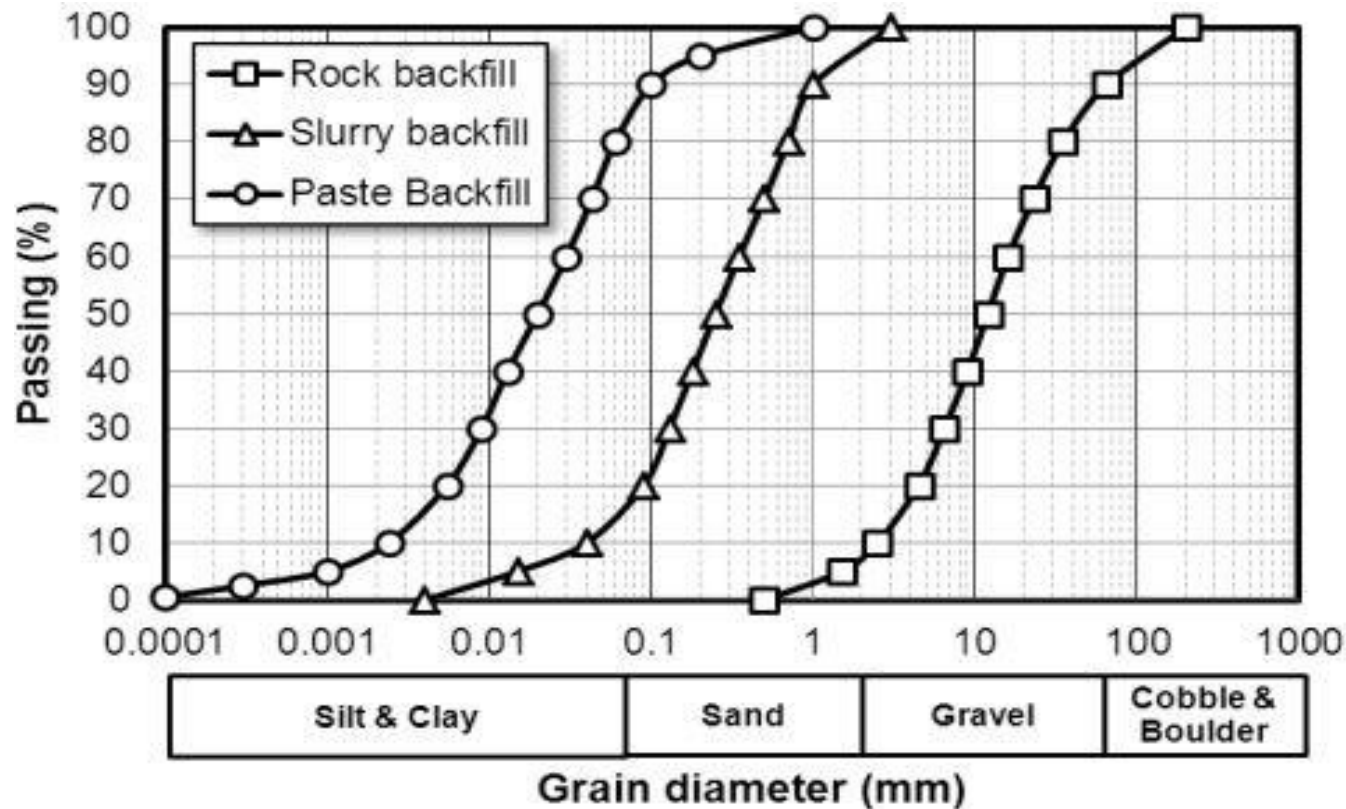


Figure 2.3: Grain size distribution of the three main types of mine backfills (Landriault, 2001)

2.3.1.1 Hydraulic Backfill

Hydraulic backfill or slurry backfill is classified as a material with high permeability and low-density Fig 2.4. Deriving its name from the mode of transportation, hydraulic backfilling describes a method using mainly water to facilitate the transport of the fill materials. It is a mix of aggregate (tailings), water and binder (if cemented) (Potvin et al., 2005). This mixture is usually prepared at the ground level and then transported by pipeline to the underground voids. In this method of backfilling, pumping can be avoided through optimizing the pipeline lay-out. Basically, the milled tailings in the hydraulic backfill are obtained by the desliming process which comprises the circulation the fill material into hydrocyclones to allow fine fraction to be

separated and removed from the milled tailings. Through this process, the fine fraction (particle size ≤ 10 μm) is removed while the residual tailings are mixed with water and binder (for cemented hydraulic backfill) to make slurry fill material, which is then transported to the underground voids (Adhafeeri 2018). The main advantages of hydraulic backfill are:

- The installation and operation are relatively simple.
- Backfill material can be controlled easily.
- Transportation is done by the gravity and no need to be pumped.

However, using extra water is one of the disadvantages associated with hydraulic backfill. Moreover, hydraulic backfill masses are susceptible to liquefaction, which represents a serious threat to the work safety in underground mines.



Figure 2.4: Sample of hydraulic backfill (O'Toole, 2004)

2.3.1.2 Cemented Paste Backfill

Cemented paste backfill (CPB) is one of the broadly used methods in modern mines with the purpose of waste disposal and ground support (Haruna and Fall, 2020). CPB is comprised of full mill tailings, mixed with a small percentage of binder (usually cement), in the order of 3–7% by weight, and water, Fig 2.5. This material is dense and has a toothpaste consistency (Fall et al., 2015). A fresh CPB can be transported through the pipeline into the cavities. In static state, the CPB ingredients are not separated, as the colloidal electrical charges can retain all the water between the particles. In order to create such a state, at least 15% of particles should be smaller than 20 microns (Landriault, 2001; Grice, 1998). As less water is required (compared to other types of backfills) for transporting the CPB through the pipelines, mechanical strength is attained faster since most of the water would be used for cement hydration (Grice, 1998). It should be noted that the chemical and mineralogical properties of the tailings have a significant impact on the engineering properties (strength, permeability, reactivity, etc.) of CPB. Two main issues when implementing CPB are optimization of ore recovery and considering the miners' safety. Moreover, pipelines that transfer CPB materials underground, require frequent inspection (Landriault, 2001; Cui and Fall, 2018). Fig. 2.6 shows a fresh CPB being pumped through a pipeline.

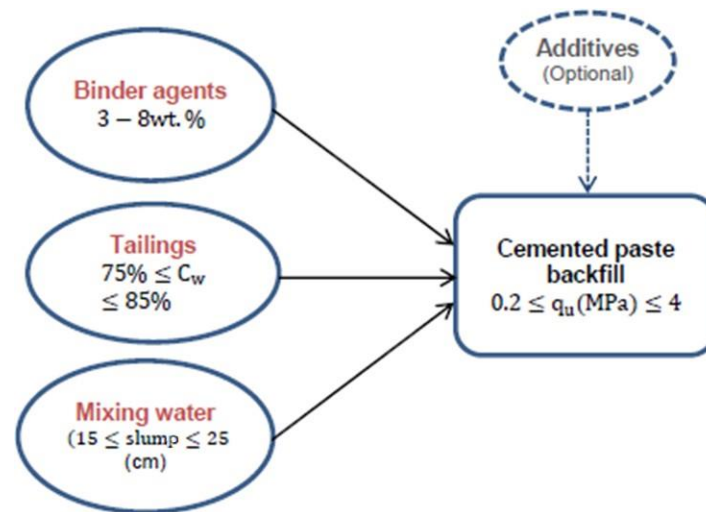


Figure 2.5: Range of various paste backfill components with typical portions (Ghirian, 2016)



Figure 2.6: Cemented paste backfill being pumped (Hosseinzadeh et al., 2005)

2.3.1.3 Rock Backfill

The suitability of rock as a backfill material is highly dependent upon the gradation and hardness of the rock particles. Rock fill and cemented rock fill have been introduced in the 1960s (Potvin et al., 2005). The quantity of hard rock excavated at most subsurface structure sites is relatively small, but selected materials

without cohesion are either difficult or expensive to find. Therefore, excavated hard rock after undergoing a crushing process can be used as selected poor material (Yao et al., 2012). Rock fill can be used as cemented (CRF) or un-cemented (URF) material. This material is a mixture of waste rock, tailings or unclassified sand. The main ingredient is the mineral waste. This type of backfill has many features, namely simple preparation, high strength and no stope dewatering. Usually 4 to 8 percent cement needs to be added in order to increase resistance. Using this method may have a high cost due to the use of the filler, rock crushing and transportation (Landriault, 2001). Factors like grain size distribution, binder content, rock type, placement method, segregation, and water to cement ratio have a significant impact on the strength of the cemented rock fill.

2.3.2 Backfill Binders

Usually, the CPB should be able to support its own weight. This resistance is primarily achieved by the binder. The hydration of the binder increases the strength of CPB with time. The more advanced is the hydration, the higher is the backfill resistance or strength (Jiang and Fall, 2017b). Moreover, in order to obtain the highest resistance in the shortest time, the ratio of water to cement must be properly selected. The most common binder used in backfill operations is Ordinary Portland Cement (OPC). Since the late 1950s, the use of OPC escalated. For example, in 1971, 233,000 tons of cement was used in Canadian mines (Hassani & Archibald, 1998a). However, after the energy crisis in 1970 and with the increasing cost of Portland cement, many materials with cementation properties were introduced (Espley et al., 1970) namely, fly ash (Udd, 1989) and blast furnace slag (Amaratunga and Annor, 1989; Razavi, 2007; Grice, 1998). Fly ash and granulated iron blast-furnace slag (slag) are used to partially replace the cement binder in CPB. Generally, fly ash and slag are used in various percentages as a constituent of blended cement or in combination with normal Portland cement. In either case, the normal proportion of fly ash is 20-40%,

while slag is 30-80% of total cement (Niroshan et al., 2018). Generally, the choice of binder depends on the strength and durability requirements of a particular CPB operation. Therefore, a cost-effective mix design with optimum strength can be achieved by selecting the right binder for given tailings and mixing water (Niroshan et al., 2018). Other materials with cementation properties can also be used to reduce cement consumption and production costs.

2.3.2.1 Ordinary Portland cement

Ordinary Portland cement is the major binder used in CPB which can be produced by either a dry or a wet process, Fig 2.7. The manufacturing process has improved significantly since bricklayer Joseph Aspdin first made Portland cement in his kitchen stove in England in the 19th century. The process of manufacturing cement rely on the technique by which raw material is mixed. Therefore, manufacturing of cement can be classified as dry process or wet process. Portland cement making up from both processes has the same ingredient, lime and silica which is approximately 85% of the cement's mass. In producing cement, more than 30 raw materials are used which can be divided into four categories as: Calcareous, Siliceous, Argillaceous and Ferrifrous. Theses constituents are mixed in the kiln producing a series of complex products which shape into ball called clinker. Primary step is crushing while mixing the raw materials is the second step which can be fed into the kiln system. The main difference in dry and wet process occur in the mixing system. Combining raw materials based on the wet process needs water while introducing raw materials without water into the kiln system happen in the dry state.

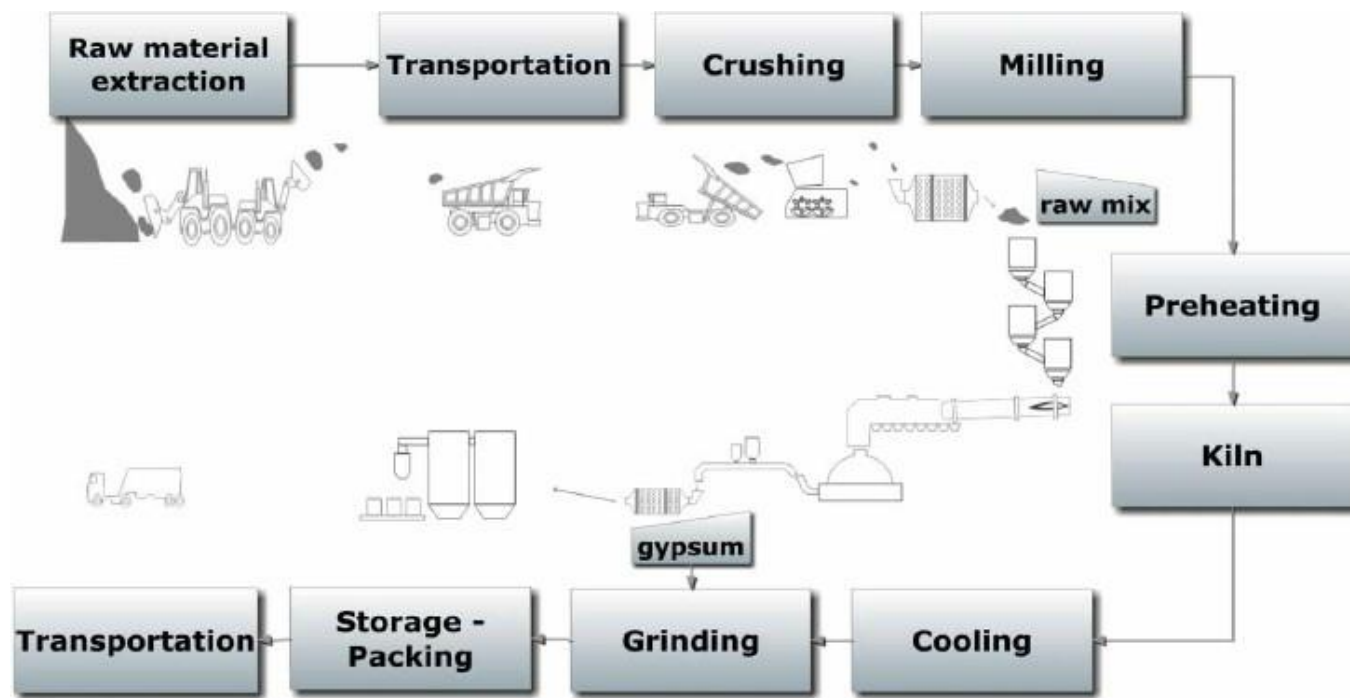


Figure 2.7: Cement production process (Chrysostomou , 2017)

2.3.2.1.1 Portland cement hydration

The major compound of Portland cement clinkers is classified into Silicates and Aluminates. Silicates including tricalcium silicate or alite (C_3S) and dicalcium silicate or belite (C_2S) while Aluminates are: tricalcium aluminate (C_3A) and tetracalcium alumina ferrite (C_4AF) (Hansen et al., 1973). The components in the presence of water can be reacted and produce three main cement hydration products (e.g. Calcium silicate hydrate (C-S-H), calcium hydroxide ($Ca(OH)_2$ or CH), and calcium sulphoaluminate known as ettringite ($3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 31H_2O$) which by passing time produces a firm and hard mass. The main hardening of cement (approximately 60%) occurs by C-S-H formation from the hydration of C_3S and C_2S . Simply put, the short-term strength can be derived by alite while the long-term strength is gained by belite. Figure 2.8 shows schematic diagram of cement hydration process. The chemical reaction is shown as follows (Skalny et al., 2002):

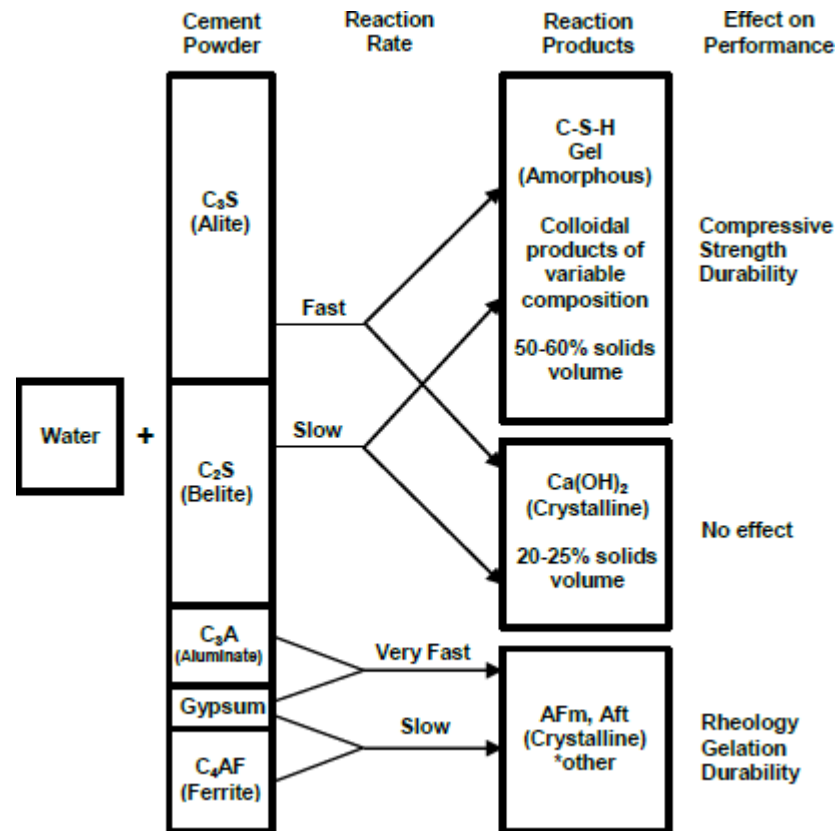
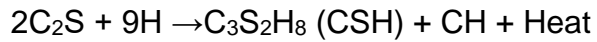
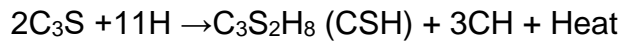


Figure 2.8: Schematic diagram of cement hydration processes

2.3.2.2 Granulated Blast Furnace Slag

One of a nonmetallic by-products of iron production is granulated blast furnace slag (GBFS), which is obtained by cooling directly molten iron mainly comprising crystalline minerals. By drying and grinding this glassy sand-like substance, the cementitious material called GBFS can be obtained, as shown in Fig.2.10, (Eggers 2002). GBFS is used to improve the reactivity of cement. The better the grinding is done, the higher the reactive surface will be. Therefore, better reaction is achieved during hydration (Aldhafeeri et al., 2016). Slag also offers higher resistance to clinker. GBFS consists essentially of silicates and aluminosilicates of

calcium and other bases, CaO, SiO₂, and Al₂O₃. Table.2.1 shows the range of chemical composition of GBFS in the United State and Canada based on ACI233-R.

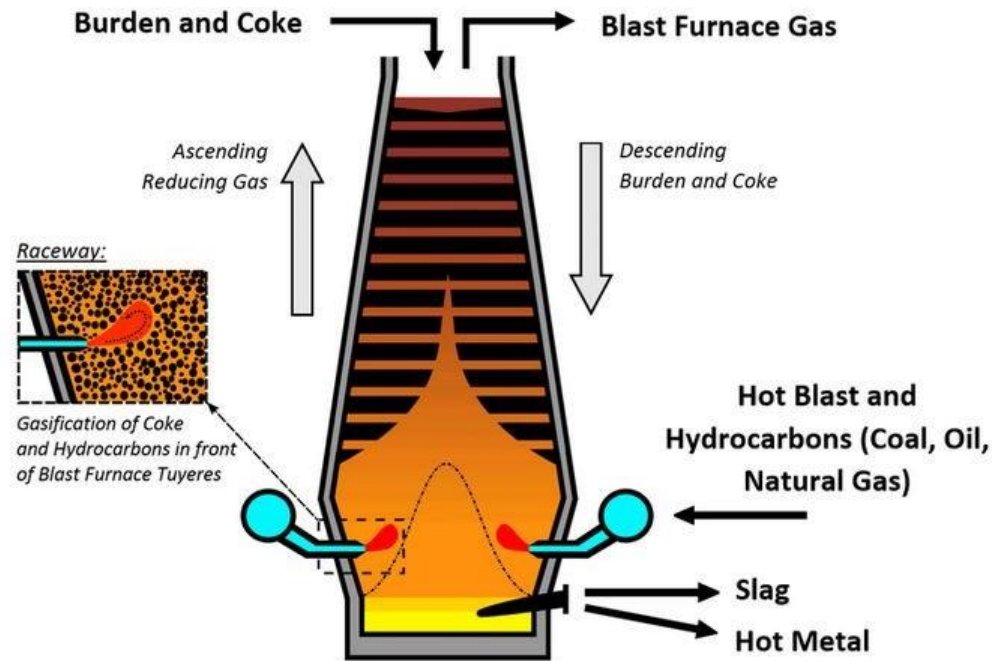


Figure 2.9: Blast furnace slag process overview (Spalang et al., 2016)

Table 2. 1. The range of chemical composition of GBFS (Eggres 2002)

Chemical constituents	Range of composition percent by mass
SiO ₂	32-42
Al ₂ O ₃	7-16
CaO	32-45
MgO	5-15

According to ASTM C989-99 and AASHTO M 302-00, slag can be classified into three groups as grade 120, grade100, and grade 80. The classification is based on the attainable strength of mortar made from

the slag and equal amount of Portland (ASTM International, 2004). These three levels are classified according to the activity index. This index is defined as follows (Eggers 2002):

$$\text{Slag Activity Index (SAI)} = (\text{SP}/\text{P} \times 100)$$

Average compressive strength of slag-referen cecement mortar cubes, psi (SP)

Average compressive strength of reference cement mortar cubes, psi (P)

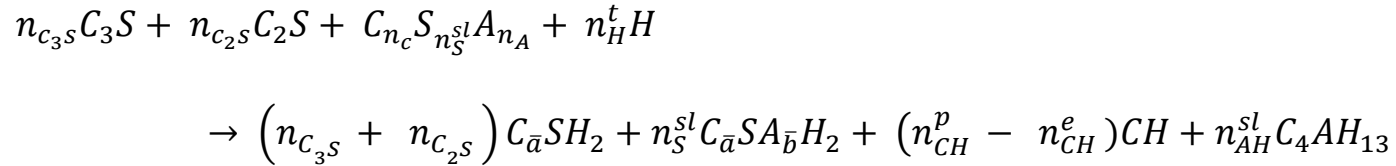
The test results indicate that the grade 120 slag has less strength in the first 3 days compared to the 7 days and beyond. In grade 100 slag, the increase in strength is visible after the 21 days. In grade 80 slag, the strength is low throughout time (Aldhafeeri et al., 2016; Niroshan et al., 2018).



Figure 2.10: Ground Granulated Blast Furnace Slag (Siddique, n.d.)

2.3.2.2.1 Slag hydration process

Cement hydration occurs when Portland cement combined with water and forming the hardened cement paste products. These hydration products are calcium silicate hydrate (CSH) and calcium hydroxide ($\text{Ca}(\text{OH})_2$). By adding slag to Portland cement during the hydration process, combined reaction happens. Indeed, slag can be hydrated with water and form calcium silicate hydrate (C-S-H) and silicates (SiO_2). In addition, the remained slag hydration products (SiO_2) can be reacted by the remained Portland cement hydration products $\text{Ca}(\text{OH})_2$ and producing additional and beneficial calcium silicate hydrate. The slag reactions equation is written as (Wu et al., 2012):



2.3.2.3 Fly Ash

The main ingredients of fly ash are silica (SiO₂), alumina (Al₂O₃), iron oxide (Fe₂O₃), lime (CaO) and magnesium oxide (MgO). Fly ash is the depleted residue from the combustion of ground or powdered coal that is transferred from the boiler by chimney gasses (ACI 116R-90, 2000). Fly ash particles are usually smaller than normal cement, so its hydration is higher than cement. It also has a lower specific gravity approximately between 1.9 and 2.4. According to STM C61 8, it can be divided into two categories according to the type of coal including Class F and Class C. Although both groups have the same physical characteristics, they differ in terms of chemical characteristics. The F-class fly ash, which comes from burning anthracite or bituminous coal, has less than 10% calcium oxide (CaO) which cannot be activated without adding lime. This is while C-class fly ash has 10 to 30 percent calcium. In 1994, the Canadian Energy Board estimated that the national annual production of fly ash is 3.63 million tons including both classes, but Class-F is produced more than Class-C and is therefore more commercialized (Hassani & Archibald, 1998a).

2.3.2.3.1 Fly ash-class F

The burning of harder, older anthracite and bituminous coal typically produces Class F fly ash (Fig.2.10). This fly ash has pozzolanic properties in nature and contains the least lime (CaO). To react and produce cement compounds, the glassy silica and alumina of Class F fly ash requires a cementing agent, such as Portland cement and mixed with water. Alternatively, adding a chemical activator such as sodium silicate (water glass) to a Class F ash can form a geo-polymer.



Figure 2.11: Fly ash class F (Foti, 2019)

2.3.2.3.2 Fly ash-class C

Fly ash produced by burning more recently formed lignite or sub-bituminous coal, in addition to having pozzolanic properties, also has some self-cementing properties. Class C fly ash by presenting of water can get stronger and hardener. It generally contains more than 20% lime (CaO), and unlike Class F, self-cementing Class C fly ash does not require an activator. Alkali and sulfate (SO₄) contents are generally higher in Class C fly ashes, Fig 2.12 (Chusid et al., 2009).



Figure 2.12: Fly ash class c (Chou, 2012)

2.3.3 Sodium silicate and its effect on CPB properties

Sodium silicate was first introduced in 1818. It can be produced by melting sand with sodium at 1200°C. Soluble sodium silicates are a kind of silicate polymer. Different types of silicate polymers exist. Their type depends on the amount of SiO_2 and Na_2O . They range from 3.22 (low alkalinity) to 1.0 (high alkalinity) (Razavi, 2007). The ratio “ $\text{SiO}_2/\text{Na}_2\text{O}$ ” determines the physical properties and chemistry of the product. Changing this ratio allows various uses for sodium silicates (Abdul-Hussain, 2010).

Sodium silicate has many applications in various fields. It has recently been used in cement and construction materials. In the cement and construction industry, it has various applications. For example, it can be used as an alkali activator for slag and fly ash. Also, the sodium silicate increases the setting time and the degree of acceleration (Anderson and Gram, 1998). Silicate is more reactive in solution. For example, in shotcrete, it is used to accelerate the setting time. In addition, it may have effects on compressive strength (Conner, 1990).

The following are the characteristics of liquid sodium silicate (Razavi, 2007).

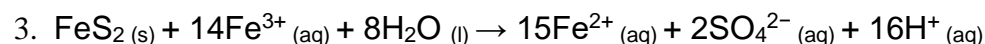
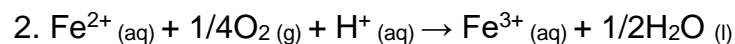
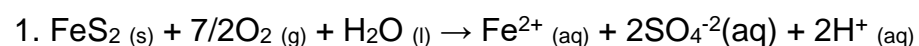
- Producing a stable, environmentally friendly and acid resistant solid.
- Reduced raw material prices by reducing the stabilization process.
- Reducing the volume of materials stabilized.

Compared to the number of studies conducted on the sodium silicate-activation of binders, such as slag and fly ash, there is a lack of studies on the effect of sodium silicate on the hydration and performance of Portland cement. In researches on cement, it has been shown that components of alkali metals (sodium and potassium) at an early age are hydrating stimulants. There is no theory about their effect in the next stages of hydration. However, many believe that alkali metals negatively influence the final strength and durability of concrete.

Studies have shown that the hydration of cement clinker minerals occurs due to the drop in the initial pH of cement vapor solutions and the formation of calcium hydroxide silicate in the presence of sodium silicate (Larichkov et al., 2006). Some studies have reported that depending on the molar ratio of SiO₂ / Na₂O, the soluble sodium silicate affects the liquid pH, the amount of clinker minerals hydration and the C-S-H gel morphology formed as a result of hydration.

2.4 Reactivity of Mine Cemented Backfill and Measurement of Reactivity

The reactivity of sulfidic tailings is one of the main environmental challenges in mining industry due to providing an acidity environment leading to releasing acid mine drainage (AMD). Indeed, sulfidic tailings contain sulfide minerals, such as pyrite and pyrrhotite, between 1% to 70% which in the presence of air and water, they can chemically react and reduce the pH of the leachate, producing a highly solution called AMD (Aldhafeeri, Z, 2018). One of the most common sulphide minerals that can be found in many mine wastes is pyrite (FeS₂). Pyrite oxidation is a complicated process which can be categorized into three main phases as shown in below equations (Dold, 2014):



Several studies show that reactivity of CPB is affected by many factors. These factors play a significant role in sulphide oxidation and AMD production including being exposed to oxidant (especially oxygen), content and type of sulphide mineral, temperature, degree of saturation and initial sulphate content, etc. Each factor influences the reactivity of CPB in a different way. According to Kwong and Ferguson (1990), the most oxidation rate occurs in the CPB with more pyrite (FeS₂) so the reactivity in the CPB with more pyrite than

other type of sulphide minerals is higher as well. Therefore, to measure and predict the effect of chemical reactivity on the CPB, static and kinetic geochemical test can be done. Table 2.2 shows the list of static and kinetic tests used to predict reactivity and AMD generation.

Table 2.2. All static and kinetic tests used to project the reactivity and AMD generation (Aldhafeeri, Z, 2018).

Technique	References
<u>Principal Static Methods</u>	Sobek, et al. (1978) British Columbia AMD Task Force (1989)
- Paste pH	
- Standard Acid Base Accounting	Sobek, et al. (1978)
- Modified Acid Base Accounting	Lawrence et al. (1989)
- British Columbia (B.C.) Research Initial Test	Bruynesteyn & Hackl (1982)
<u>Other Static Methods</u>	
- Net Acid Production	Lawrence, et al. (1989)
- Alkaline Production Potential/Sulphur Ratio	Caruccio, et al. (1981)
<u>Principal Kinetic Prediction Methods</u>	Caruccio, et al. (1981) Lawrence (1990) (Sobek et al., 1978) B. C. AMD Task Force (1989)
- Humidity cells	
- Columns and lysimeters	Ritcey (1989) B. C. AMD Task Force (1989)
- B. C. Research Confirmation test	Bruynesteyn & Hackl (1982)
<u>Other Kinetic Prediction Procedures</u>	Halbert, et al. (1983) Lawrence, et al.(1989)
- Shake Flasks	
- Soxhlet Extraction	Singleton & Lavkulich (1978) Sullivan & Sobek (1982)

In addition to these tests, there are several other methods that can be used, which rely on other factors. David and Nicholson (1995) preferred to practice two other different and independent methods (mass balance and oxygen gradient) to determine the reactivity of sulphide minerals (e.g., pyrrhotite) in mine waste. Therefore, choosing the proper methods to measure the chemical reactivity of mine waste materials

depends on measuring proper parameters. Oxygen consumption is one of these techniques relying on measuring oxygen gas. This test measures the amount of consumed oxygen during oxidation of sulfidic material sample in a short period of time (3-5 hours) (Aldhafeeri, 2018).

2.5. Properties of cemented paste backfill

In this section, the main properties of CPB will be discussed. These include:

- Physical properties of CPB
- Mechanical properties of CPB
- Hydraulic properties of CPB
- Thermal properties and temperature development within CPB
- Chemical properties (reactivity) of CPB

2.5.1. Physical properties of CPB

Physical actors or parameters including porosity, unit weight, water content and degree of saturation affect CPB resistance and other engineering properties significantly. Most of the physical properties of CPB are assessed through laboratory samples, mainly due to the lack of easy access to the stopes. Research in this field has shown that changes in physical properties of CPB are mainly related to factors such as preparation and placement techniques, stress regime, tailings and cement properties. It is reported that at 90 days of curing, the void ratio ranges between 1.10 and 1.40, unit weight from 18.40 to 20.10 kN/m², and degree of saturation between 79% and 100% (Pierce, 1997).

Field studies has shown that void ratio and degree of saturation of CPB are 20% and 10% higher, respectively, than the values obtained in laboratory experimental tests. In a column experiments, carried out

by Benzaazoua et al. (2004) it was shown that 91-day specimens had void ratios between 0.85 and 0.97 and a degree of saturation between 83% and 94%. This is why in the CPB drained column (Figure 2.13), void ratios and the degree of saturation are between 0.77-0.91, 75% and 93%, respectively (Belem et al., 2006a).

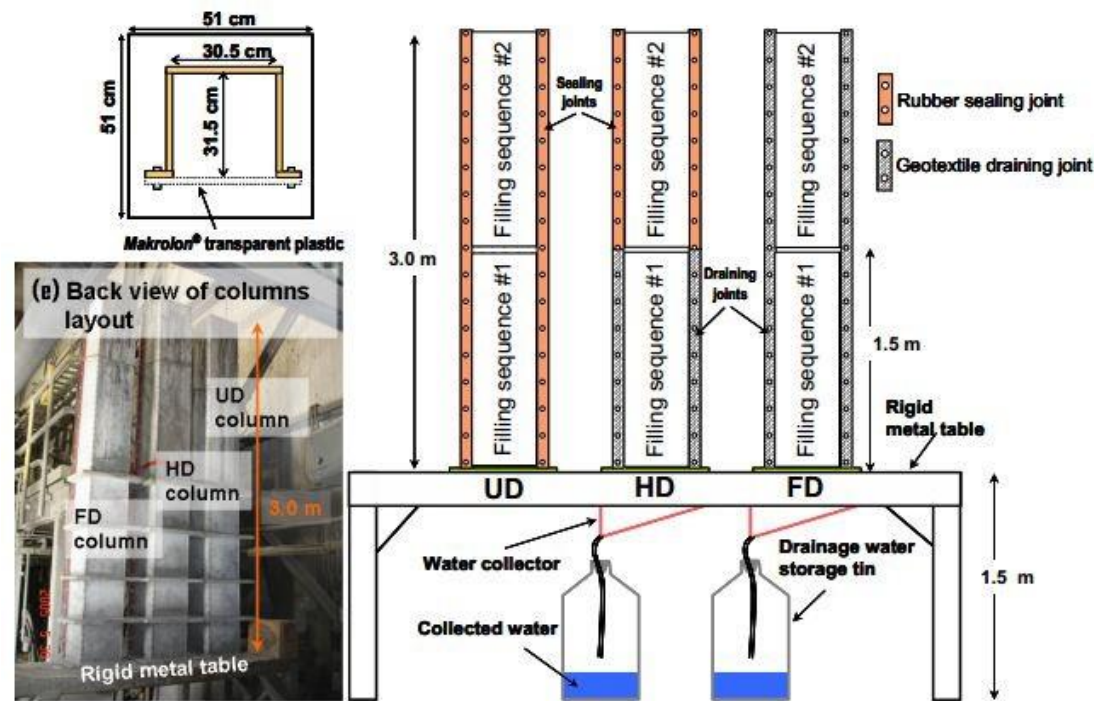


Figure 2.13: Schematic diagram of the experimental set-up showing a photograph of the set-up at mine paste backfill plant (Belem et al., 2006b)

In another study carried out by Yilmaz et al. (2009), the effect of the type of binder on the physical properties of 28-day specimens was investigated. In the report provided for samples prepared with OPC, porosity ranges from 0.42 to 0.47 and a saturation degree of between 90% and 97%, and gravimetric water content between 38% and 46%. However, for samples which are cured under pressure, the porosity varies from 0.41 to 0.45 and the degree of saturation varies between 80% to 94% and gravimetric water content varies from 33 to 42%.

2.5.2. Mechanical properties of CPB

One of the most important design criteria for CPB is mechanical strength (Fall et al., 2007; Cui and Fall, 2016; Ghirian and Fall, 2016; Jiang et al. 2017). Fall et al. (2007) experimentally investigated the stress-compression behavior of CPB under compression. They obtained valuable results on mechanical behavior of CPB. They concluded that the stress-strain properties of CPB are strongly affected by confinement (Figure 2.14) and the CPB mix components, such as the binder type (Figure 2.15) (Fall et al., 2007). The axial stress-strain behaviors of unconfined and confined CPB are significantly different. The strain corresponding to the maximum CPB stress and the ductility capacity of the CPB generally increase with the confining stress (Fall et al., 2007). The results show that by decreasing the ratio of water to cement, the modulus of elasticity and their compressive strength increase in comparison with the higher water to cement ratio. The increase of cement content reduces the ductility of CPB and increases its stiffness (Figure 2.16) (Fall et al., 2007).

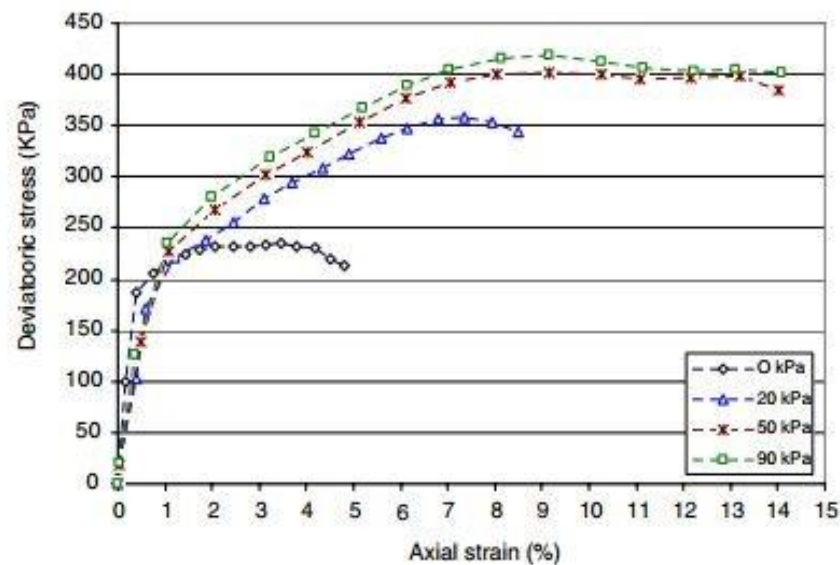


Figure 2.14: Effect of confinement pressure on the stress-strain (Fall et al., 2007).

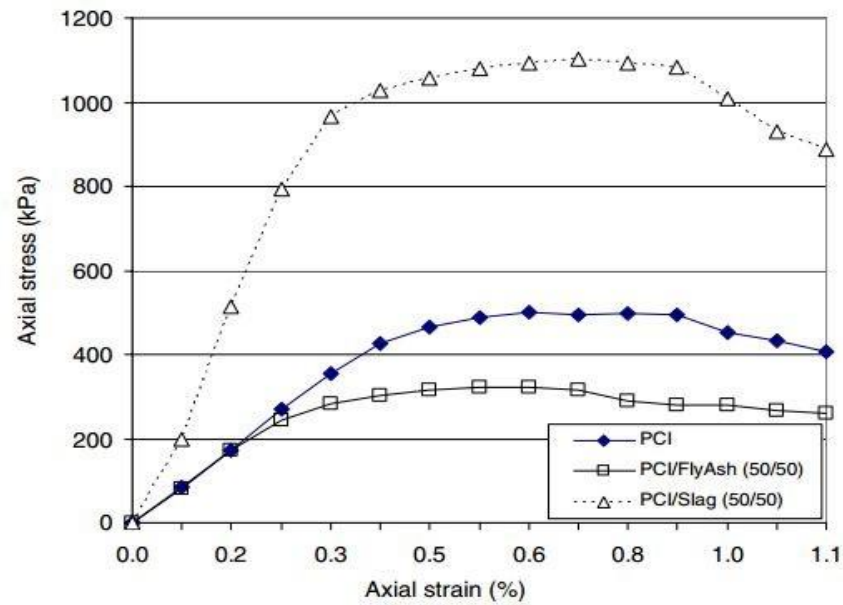


Figure 2.15: Effect of binder type on the stress–strain behavior of CPB (Fall et al., 2007).

It is also found that CPB made with coarse or medium tailings has a higher modulus of elasticity and resistance. Also, the presence of sulfate in the backfill material can lead to a decrease in strength and modulus of elasticity of CPB (Fall et al. 2007).

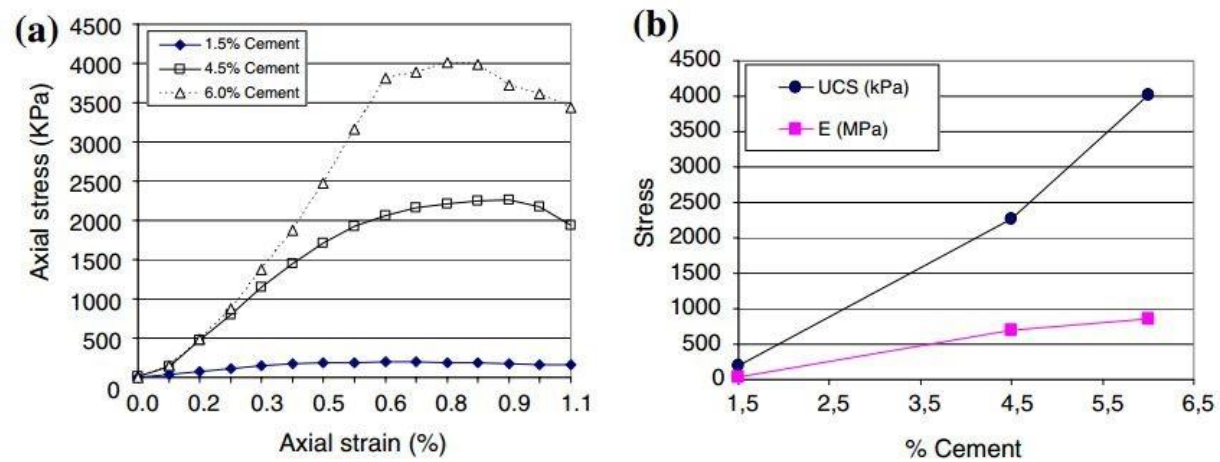


Figure 2.16: Effect of binder proportion on (a) stress-strain behaviour, and (b) peak stress and modulus of elasticity of CPB (Fall et al.2007)

2.5.3. Hydraulic properties of CPB

The most important hydraulic properties of CPB include pore water pressure and (saturated and unsaturated) hydraulic conductivity (Wu et al. 2014). To evaluate mechanical behavior in CPB, the development of pore water pressure should be evaluated. This assessment can also be used to examine the stability and deformation of CPB. Experimental measurements of the evolution of pore water pressure in CPB are rarely considered in the literature. In 2007, Helinski conducted a centrifuge test to investigate the effect of pore water pressure on final stresses in a CPB structure (Helinski, 2007). Simms and Grabinsky (2009) modified a triaxial cell with a miniature tensiometer to measure the evolution of negative pore pressure (suction) during consolidated undrained (CU) triaxial tests. In their study, CPB samples cured at 2 days were subjected to triaxial shear testing, and stress-strain and suction were monitored during shearing. However, suction evaluation and its relationship with curing time and stress were not evaluated. Despite a very limited number of empirical studies on pore pressure measurements, field instrumentation of backfill have increasingly been noticed (Belem et al., 2006b). Researchers have been able to empirically evaluate the pore water pressure development in CPB mass by installing a variety of sensors, including piezometers and pressure cells in different stopes. The major contribution of such studies has been providing an understanding of the fundamental behaviour of CPB with regard to the evolution of pore pressure, effective stress, cement hydration reaction, self-desiccation and hardening process in the field stopes (Ghirian, 2016).

Numerous studies have been performed to assess and understand the saturated hydraulic conductivity of CPB, which also influences the key design parameters of CPB. For instance, Fall et al. (2009) conducted laboratory testing program to investigate the hydraulic conductivity of CPBs and develop a model for predicting its evolution with time. They found that that the saturated hydraulic conductivity of CPB is time-

dependent (Figure 2.17). The permeability of CPB is also affected by the mix components (Figure 2.18), curing temperature (Figure 2.19), and mechanical damage (Figure 2.20).

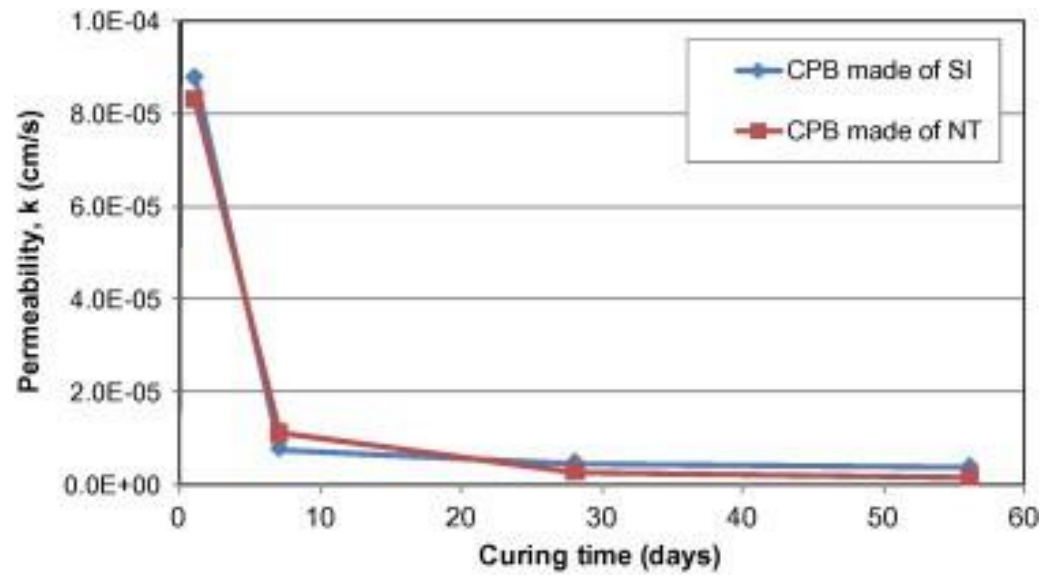
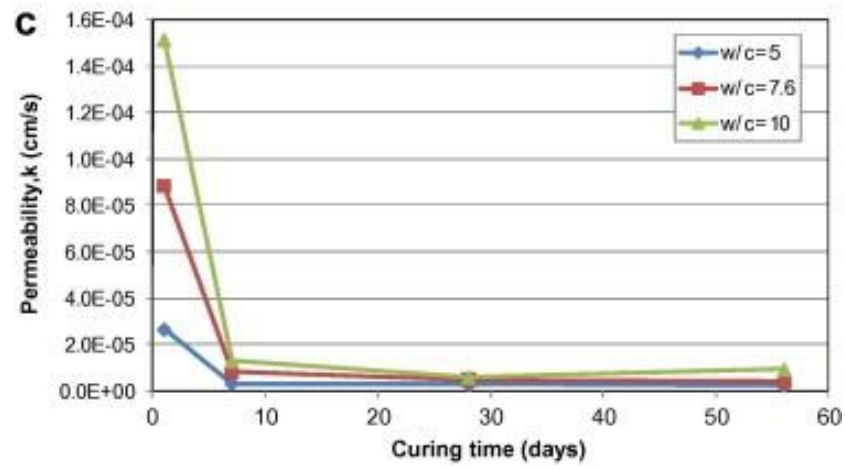
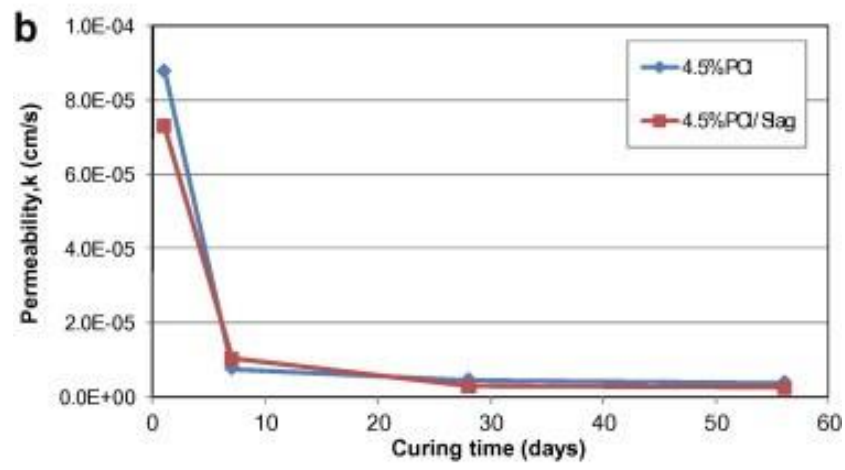
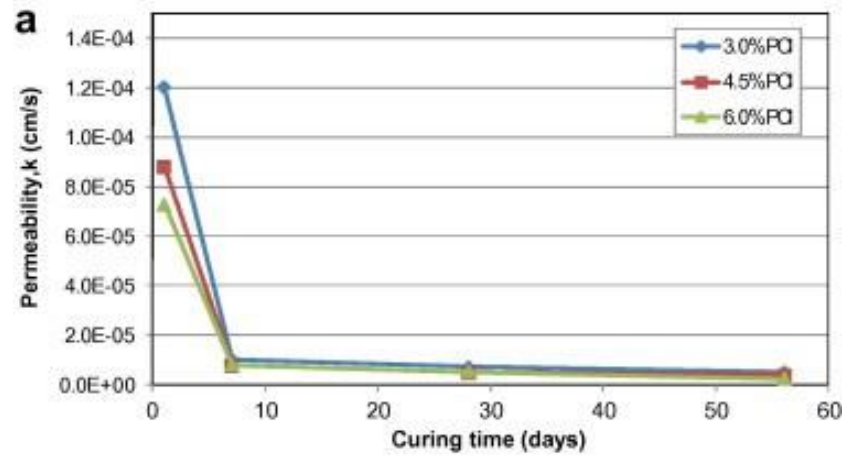


Figure 2.17: Time-dependent change of the permeability of CPBs containing silica tailings (SI) or natural tailings (NT) as tailings materials (4.5% PCI; slump = 18 cm) (Fall et al. 2009)



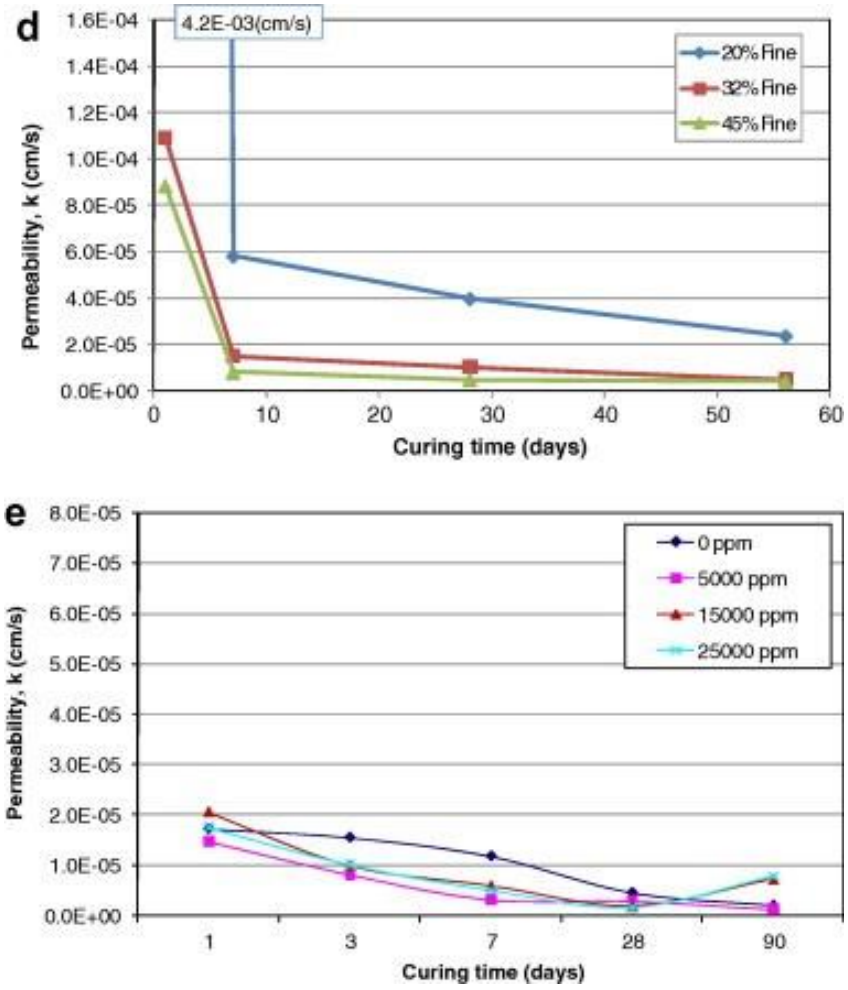


Figure 2.18: Effect of CPB's mix components on its permeability: Effect of (a) binder content (PCI; slump = 18 cm; SI used); (b) binder type (4.5% cement; slump = 18 cm; SI used); (c) w/c ratio (4.5% PCI; SI used); (d) tailings fineness (4.5% PCI; slump = 18 cm; SI used); and (e) sulphate content (4.5% PCI; slump = 18 cm; SI used).(Fall et al. 2009)

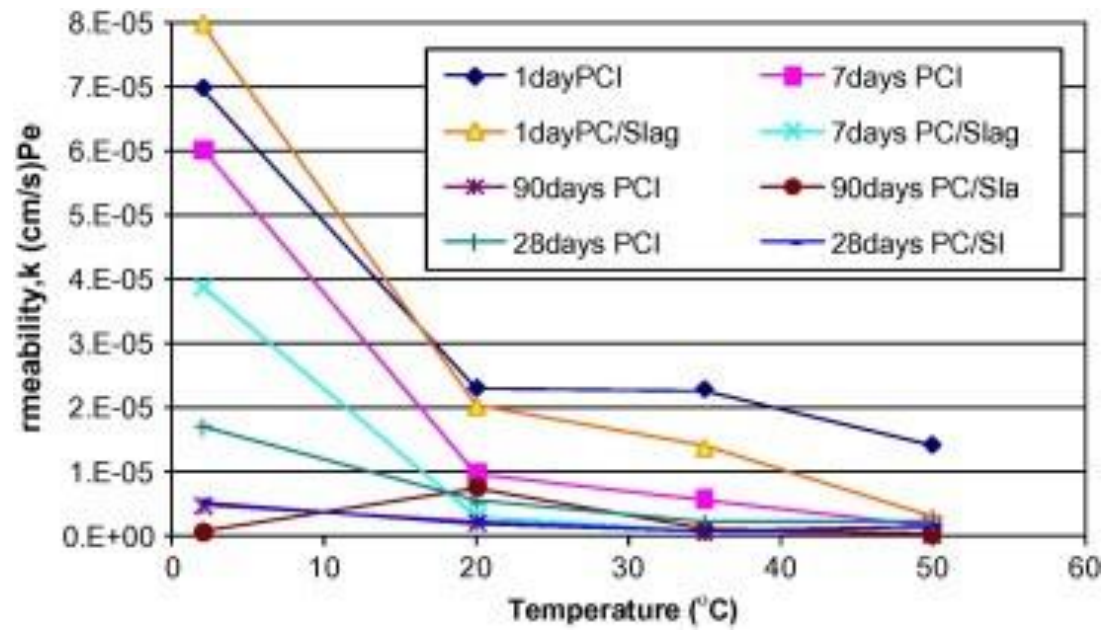


Figure 2.19: Coupled effect of temperature and binder on the hydraulic conductivity of CPB at various curing temperatures (4.5% binder; slump = 18 cm; SI used) (Fall et al. 2009)

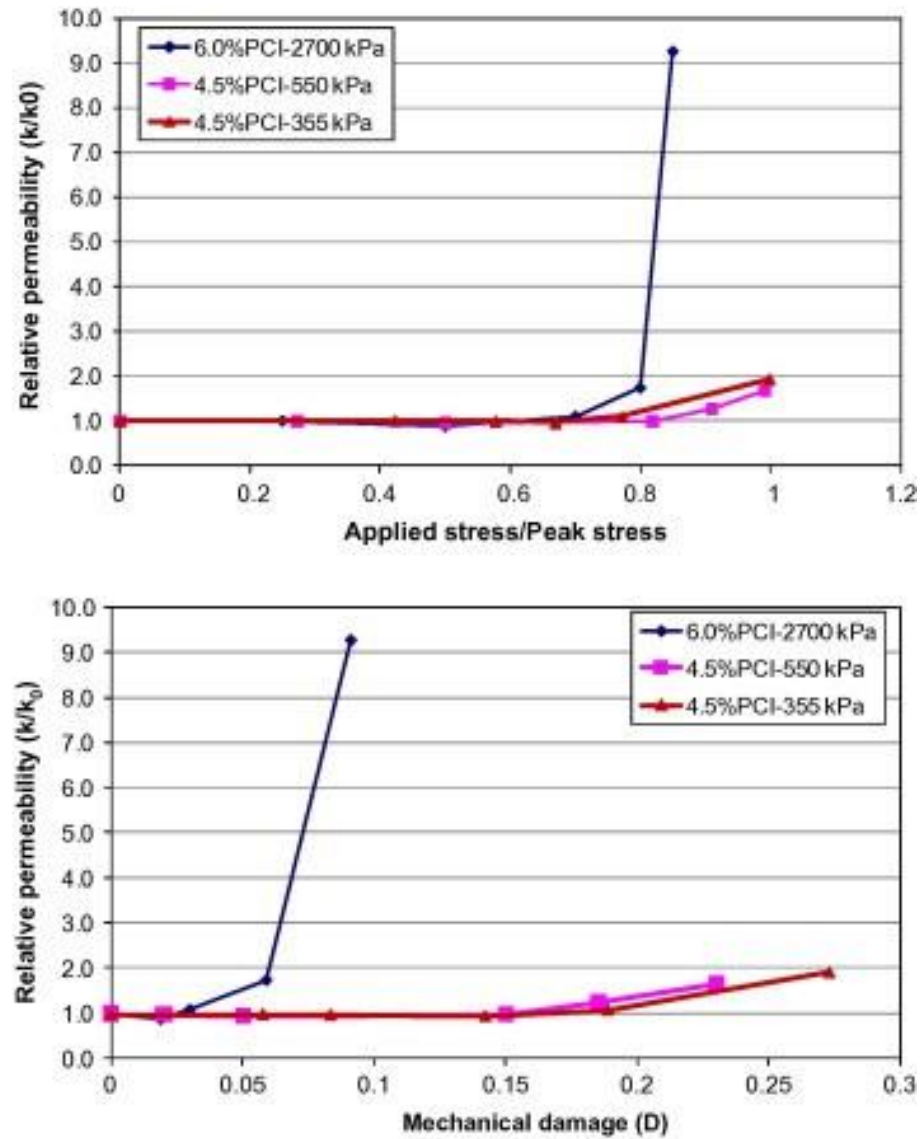


Figure 2.20: Evolution of the relative hydraulic conductivity (k/k_0) of various CPBs with the (a) ratio of the maximum applied stress to peak stress (applied stress/UCS) and (b) mechanical damage (D) (Fall et al. 2009)

However, little studies were conducted on the unsaturated hydraulic conductivity of cemented paste backfill. Therefore, the unsaturated hydraulic properties of CPB is poorly understood. An understanding of the unsaturated hydraulic properties of the CPB is essential to designing a cost-effective, environmentally friendly and durable backfill structure.

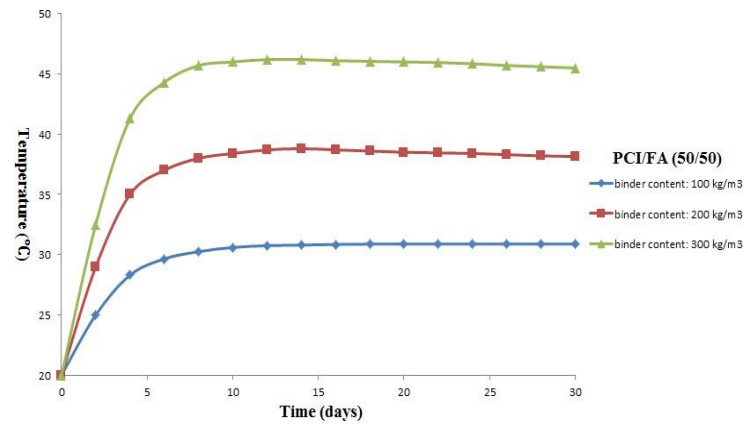
2.5.4. Thermal properties and temperature development within CPB

The most important thermal properties of CPB include:

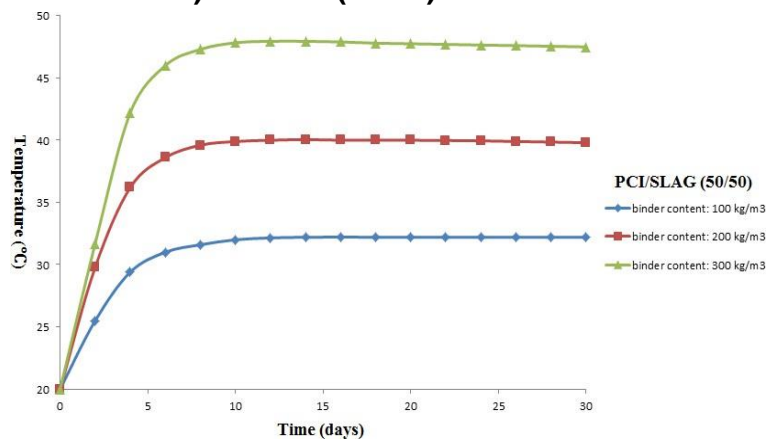
- Thermal conductivity.
- External and internal temperatures to which the CPB will be exposed, including initial CPB temperature, the heat resulting from binder hydration, and the temperature of the rock mass surrounding the CPB structure.

Both factors are required in order to understand the thermal behavior of CPB. For thermal analysis of CPB and heat transfer between CPB and the environment, it is necessary to understand the thermal conductivity. Several factors can influence the thermal conductivity of the CPB, such as mineralogy of the tailings, temperature, porosity and degree of saturation (Célestin and Fall, 2009). For example, conductive mineral materials (such as quartz) have a higher thermal conductivity. Moreover, some factors including w/c ratio, cement type, binder content, curing time and sulphate content have minor effects on thermal conductivity (Célestin and Fall, 2009).

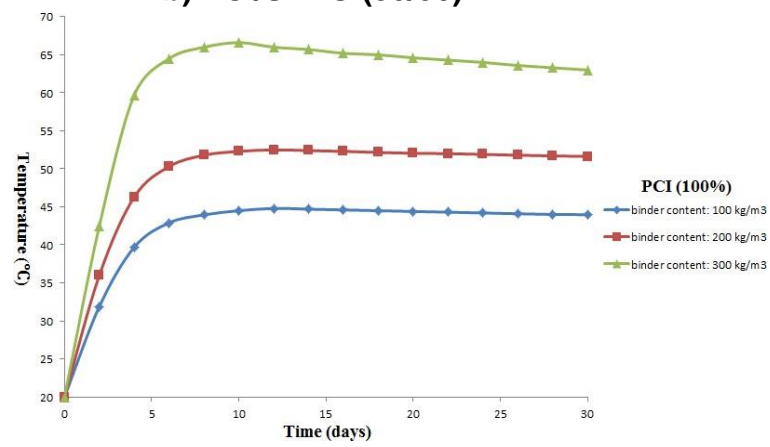
In another study by Wu et al. (2012), it has been shown that high temperatures can be developed within CPB mass. The temperatures in the CPB mass is affected by several factors, such as curing time (Figure 2.20), binder type (Figure 2.21), rockmass temperature (Figure 2.22). This backfill temperature significantly affects its engineering properties and behavior. For example, Wu et al. 2016 has concluded that the stress-stress relationship (Figure 2.23) and the elasticity modulus (Figure 2.24) of CPB are significantly affected by the curing time and the initial temperature of the CPB (Figure 2.25)



a) PCI/FA (50/50)



b) PCI/SLAG (50/50)



c) PCI (100%)

Figure 2.21: Effect of binder content on the heat development within CPB with: a) PCI/FA (50/50) b) PCI/SLAG (50/50) c) PCI (100%) (Wu et al. 2012)

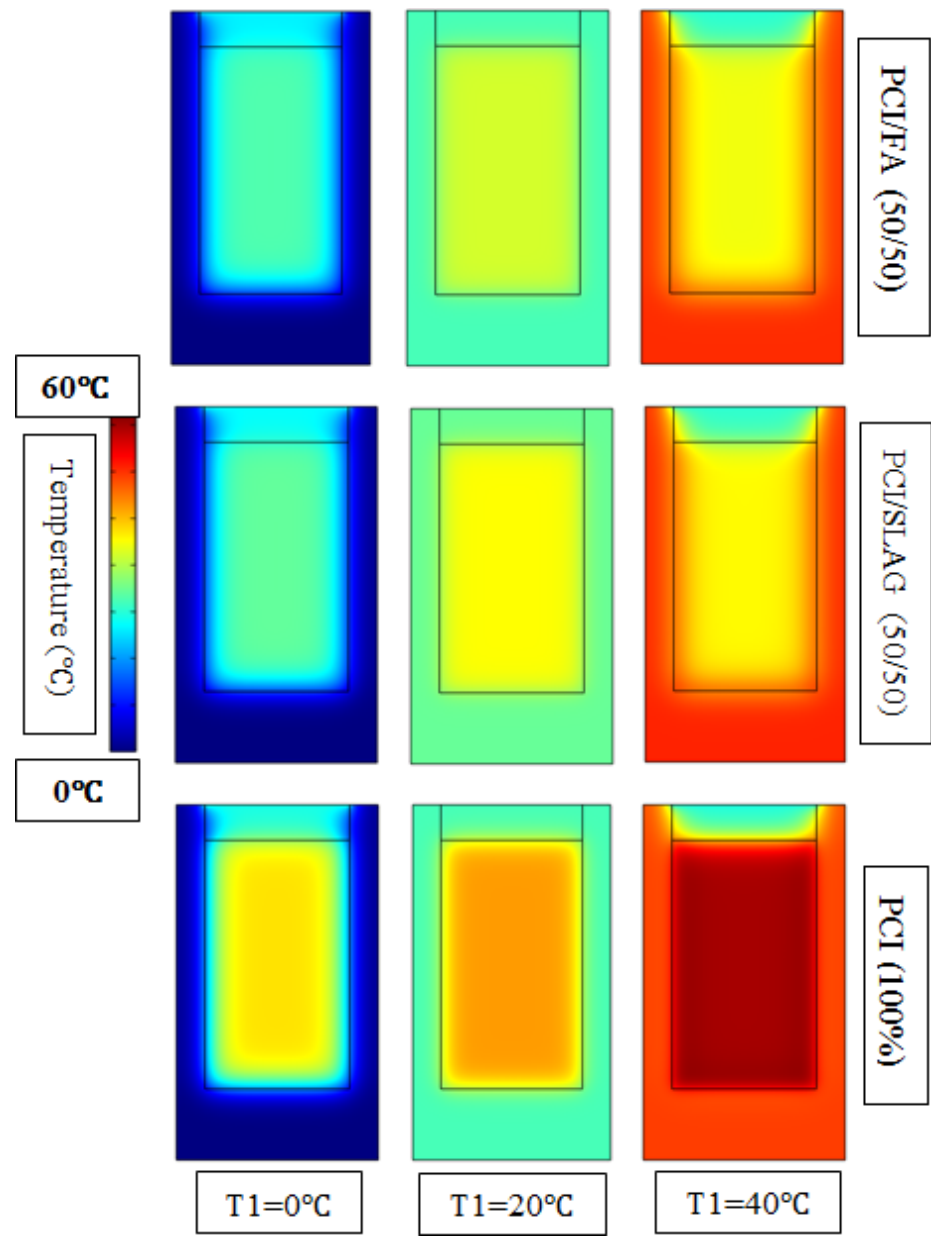


Figure 2.22: Temperature distribution in CPB and the surrounding rock mass with different initial temperatures after 7 days (Wu et al. 2012)

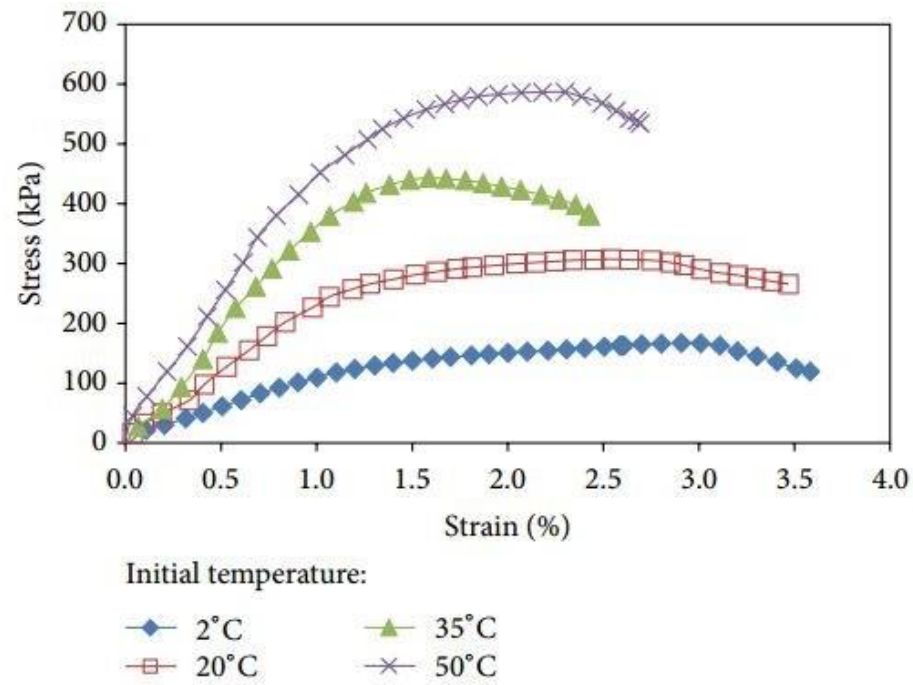


Figure 2.23: Effect of initial temperature on the stress-strain shape of CPB in 7 day samples (Wu et al., 2016)

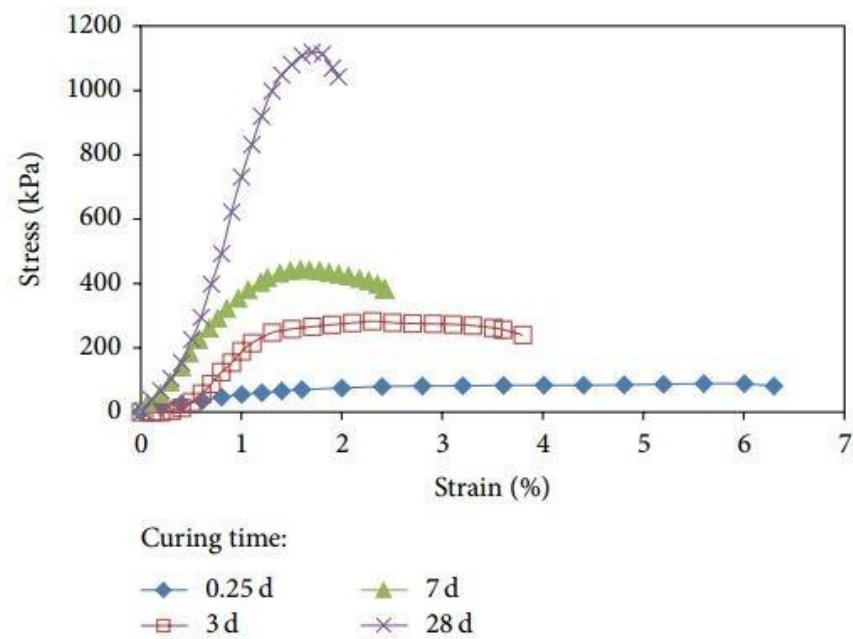


Figure 2.24: Effect of curing time on the stress-strain curve of CPB at initial temperature (35 deg)(Wu et al., 2016)

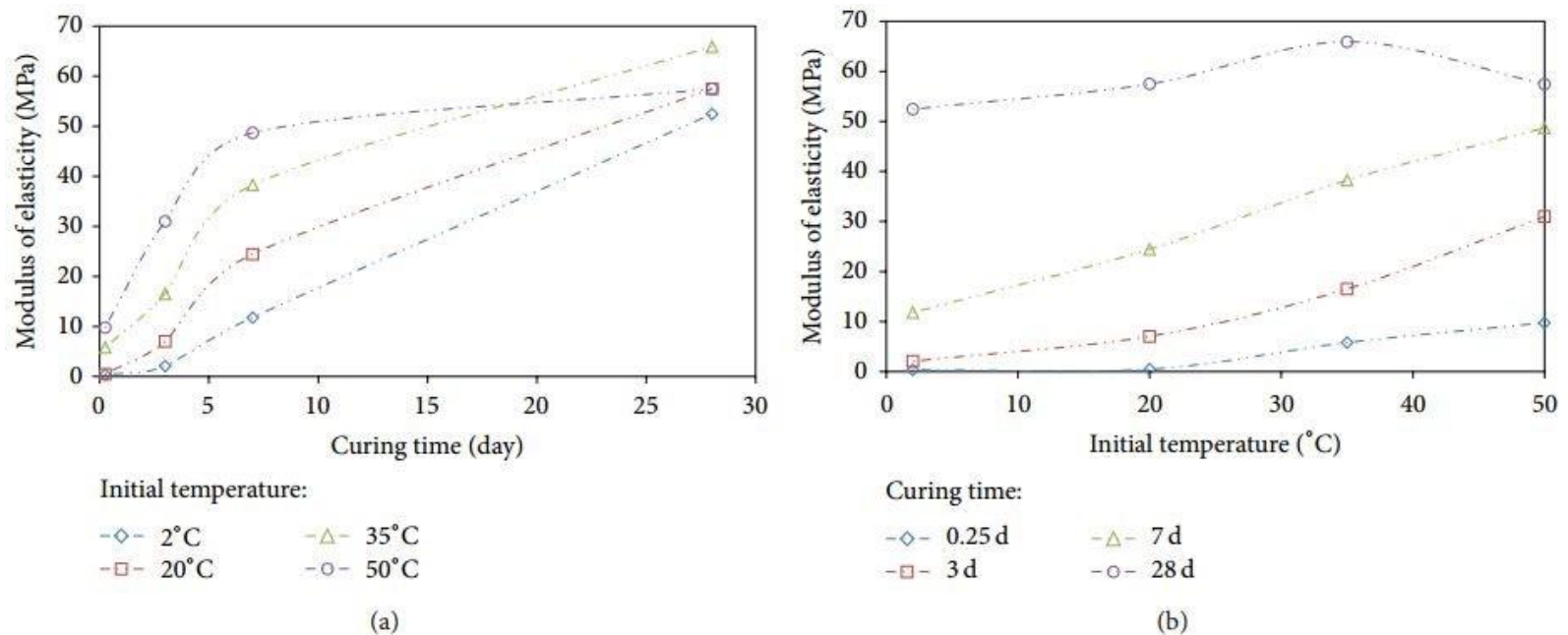


Figure 2. 25: Effect of initial temperature and curing time on the evolution of CPB ! (a) effect of curing time and (b) effect of initial temperature (Wu et al., 2016).

2.5.5 Chemical properties (reactivity) of CPB

The CPB may exhibit chemical behavior based on the chemistry and mineralogy of its constituents, namely tailings, binder, mixing water and admixtures, where applicable (Cihangir et al., 2015; Ghirian & Fall, 2017). This property plays a significant role on the environmental performance of the CPB and may also influence its strength development (Ghirian & Fall 2013; Aldhafeeri & Fall, 2017). Reactive CPB material may produce chemical compounds that could be harmful to the environment. For example, acid mine drainage (AMD) can lead to the contamination of both surface and ground water bodies. Moreover, the mechanical strength of CPB, which largely depends on binder hydration has been observed to be affected by sulphate content in the tailings (Benzaazoua et al., 2002; Kesimal et al., 2004; Fall & Benzaazoua, 2005). For these reasons, several studies have been conducted to understand the chemical properties (e.g., reactivity) of the CPB.

Figure 2.25 shows the range of observed reactivity values for CPB and tailings investigated by different researchers.

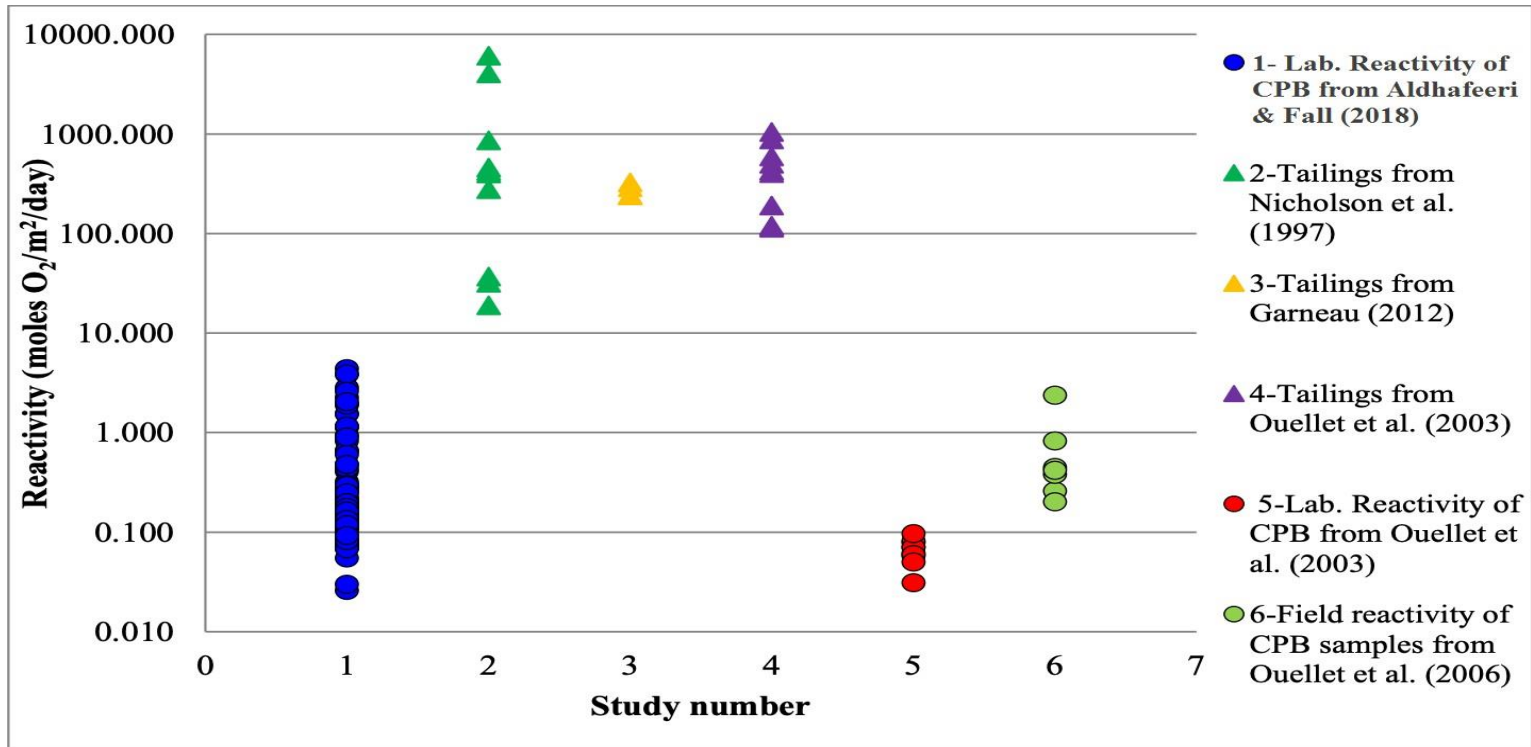


Figure 2. 26: Typical reactivity values of CPB and tailings from the literature (Aldhafeeri & Fall, 2018)

Certain factors have been observed to affect the reactivity of CPB. A study by Aldhafeeri & Fall (2016) revealed that the chemical reactivity of CPB generally decreases with curing time as shown in Figure 2.26. Although the CPB with the highest pyrite content is the most reactive, the decreasing trend is true regardless of the pyrite percentage. Another factor identified to influence the reactivity is the initial sulphate content. As the sulphate content increases the reactivity of the CPB also increases as indicated in Figure 2.27 (Aldhafeeri & Fall, 2017). Furthermore, the reactivity of the CPB was found to decrease appreciably as the curing temperature increases (Figure 2.28). Aldhafeeri and Fall (2016) attributed this to the increase of oxygen diffusion coefficient as well as pyrite oxidation rate at high temperatures.

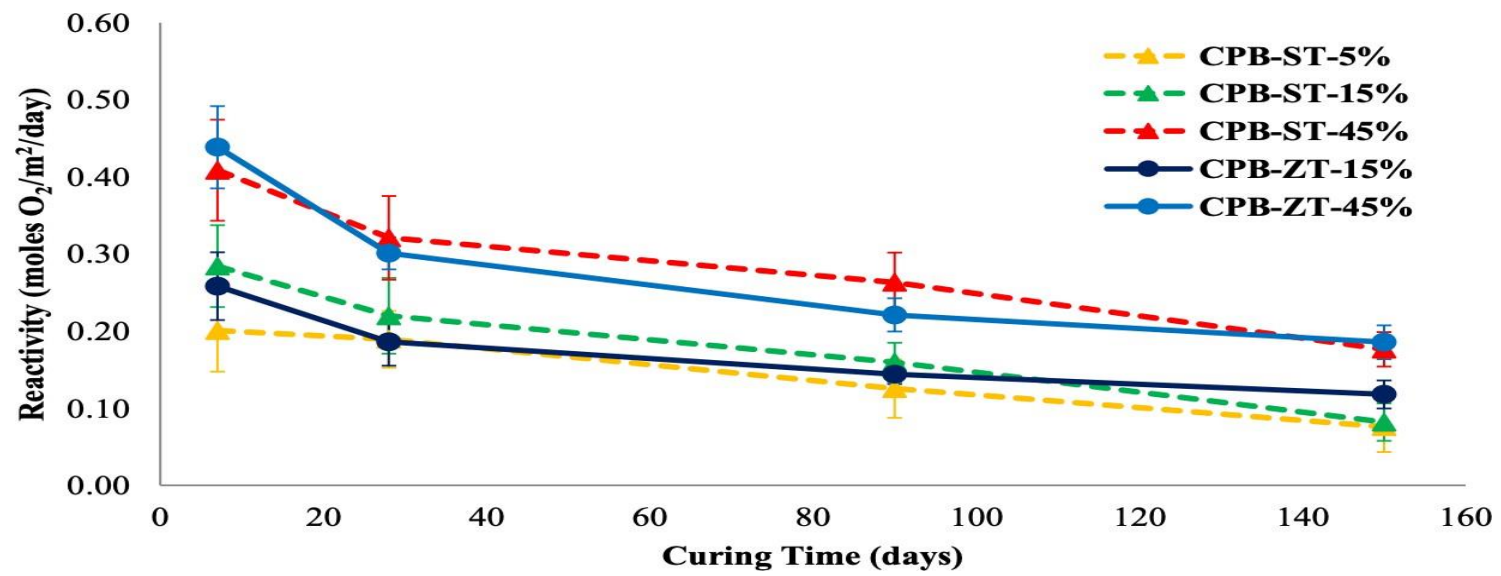


Figure 2.27: Effect of curing time on the reactivity of CPB samples containing various pyrite contents (Aldhafeeri & Fall, 2016A)

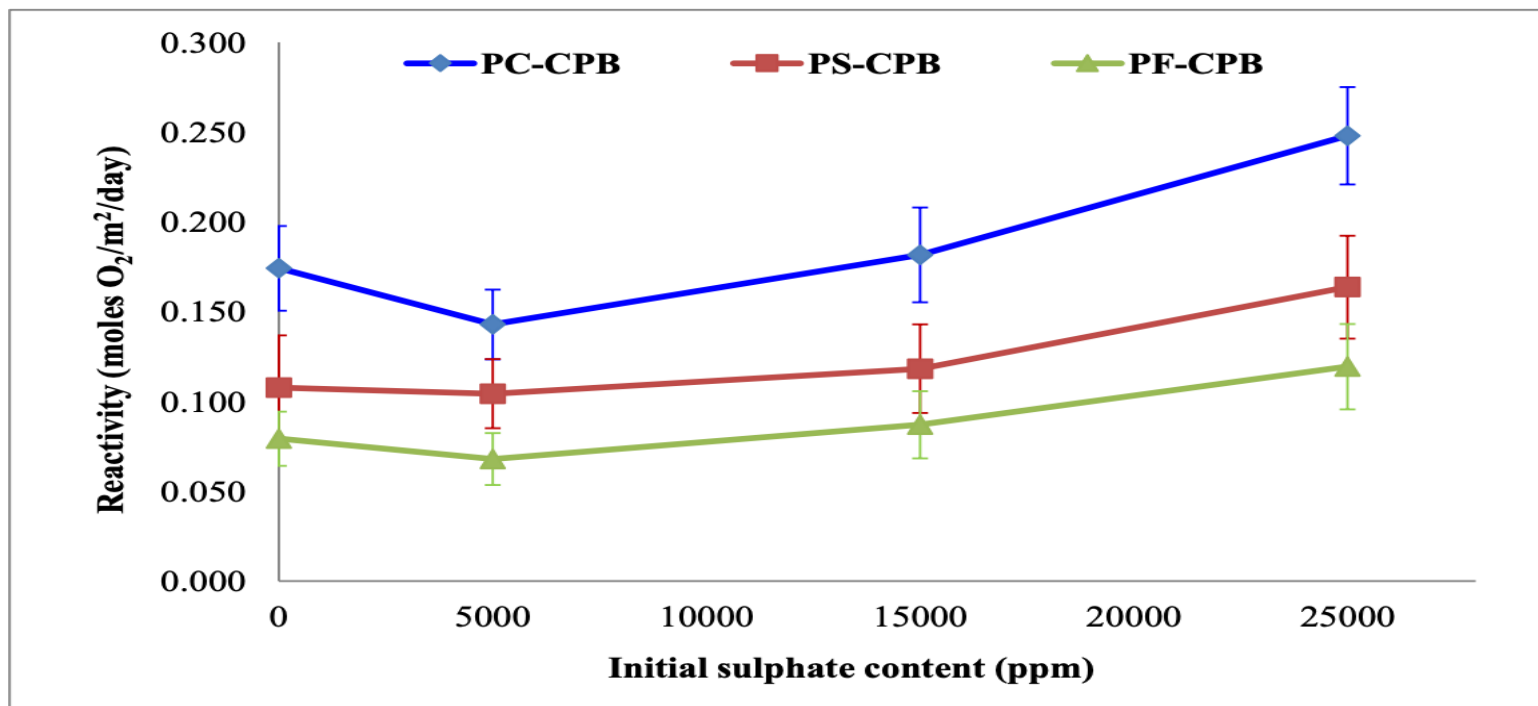


Figure 2.28: Effect of initial sulphate content on the reactivity of CPB made of various types of binder (Aldhafeeri & Fall, 2017).

Note: PC = 100% Portland cement; PS = Blend of PC and blast furnace slag; PF = Blend of PC and Fly ash

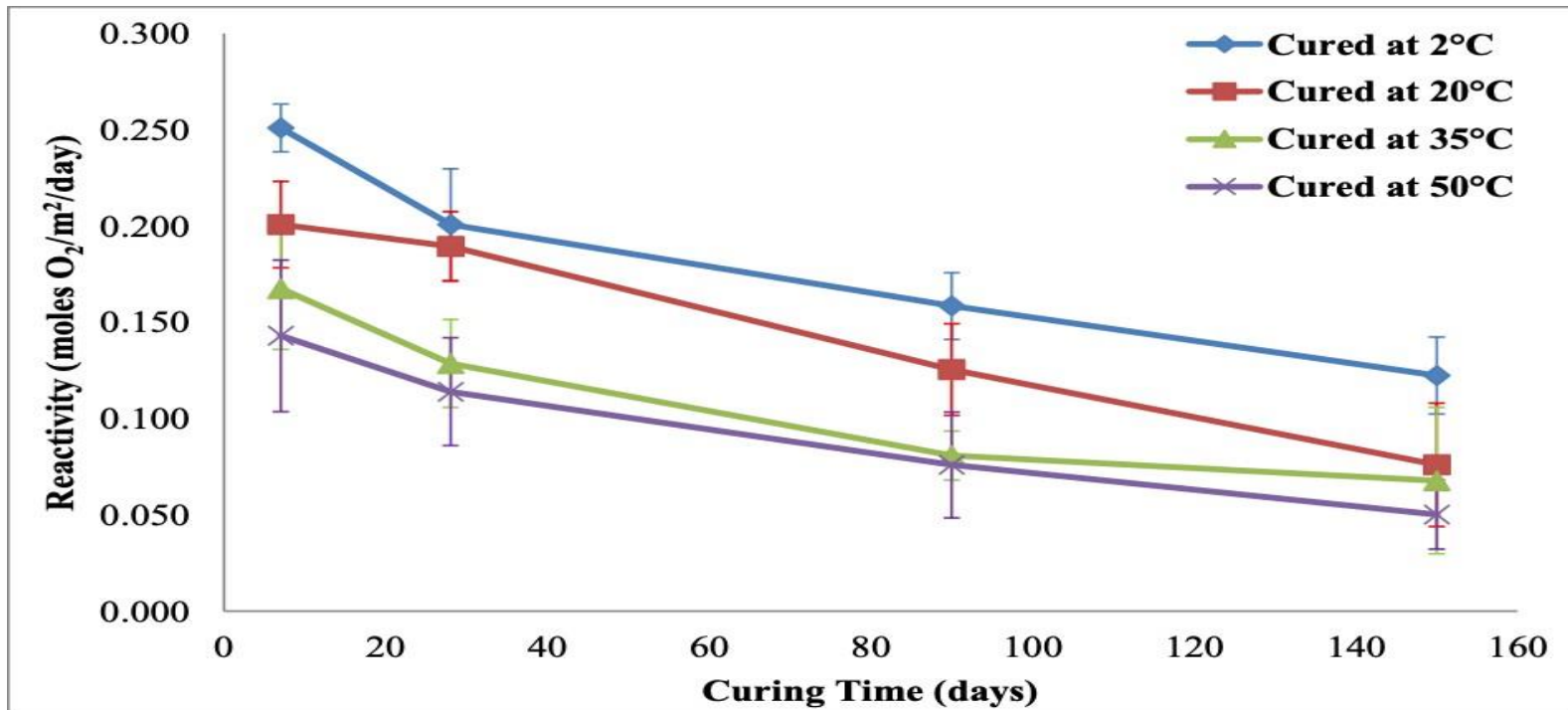


Figure 2.29: Effect of curing temperature on the reactivity off CPB (Aldhafeeri & Fall, 2016B)

2.6 Previous studies on strength and environmental properties of sodium silicate-cemented backfill

There is paucity of data on the strength development and key environmental properties (permeability, reactivity) of CPB that contains sodium silicates (S-CPB). In 2015, Kermani and colleagues conducted a series of lab tests on strength and water retention of mine cemented backfill with sodium silicates. These experiments were performed on two configurations including Gelfill and cemented hydraulic fill (CHF). Gelfill is constituted of water, tailings and binder, also contains sodium silicate. A large number of Gelfill (GF) and cemented hydraulic fill (CHF) were prepared with different mixing designs (Table. 2.3).

Table 2.3. Binder mixtures characteristics of backfill samples (Kermani et al., 2015a)

Sample ID	NPC (wt%)	BFS (wt%)	Sodium silicate(wt5)	Binder (wt%)	Mixing time (min)	Curing temperature(0C)
5 min-CHF	0.5	4.5	0	5	5	20
10 min-CHF	0.5	0	0	5	10	20
15 min-CHF	0.5	4.5	0	5	15	20
20 min-CHF	0.5	4.5	0	5	20	20
30 min-CHF	0.5	4.5	0	5	30	20
60 min-CHF	0.5	4.5	0	5	60	20
5 min-Gelfill	0.5	4.5	0.3	5	5	20
10 min-Gelfill	0.5	4.5	0.3	5	10	20
15 min-Gelfill	0.5	4.5	0.3	5	15	20
20 min-Gelfill	0.5	4.5	0.3	5	20	20
30 min-Gelfill	0.5	4.5	0.3	5	30	20
60 min-Gelfill	0.5	4.5	0.3	5	60	20
CHF/5 °C	0.5	4.5	0	5	5	5
CHF/15 °C	0.5	4.5	0	5	5	15
CHF/25 °C	0.5	4.5	0	5	5	25
CHF/35 °C	0.5	4.5	0	5	5	35
CHF/50 °C	0.5	4.5	0	5	5	50
Gelfill/5 °C	0.5	4.5	0.3	5	5	5
Gelfill/15 °C	0.5	4.5	0.3	5	5	15
Gelfill/25 °C	0.5	4.5	0.3	5	5	25
Gelfill/35 °C	0.5	4.5	0.3	5	5	35
Gelfill/50 °C	0.5	4.5	0.3	5	5	50

Samples were cured for up to 28 days (Kermani et al., 2015a). The 28-day axial resistance of the specimen was obtained and compared with a resistance test specimen without sodium silicate. The test results showed that adding an appropriate amount of an alkaline activator, such as sodium silicate, can increase mechanical (one-way compressive strength) and physical (water retention) properties of the Gelfill. The study and analysis of the pore structure of the GF by mercury intrusion porosimetry (MIP) showed that the addition of sodium silicate could refine the particle size distribution and reduce the total porosity of GF, resulting in the improvement of its mechanical properties of (Figure 2.30). It has also been shown that the time and rate of drainage in the Gelfill sample are lower than those of CHF without sodium silicate (Figure

2.31). This study concluded that the addition of sodium silicate can lead to an increase in mines production with respect to mine safety (Kermani et al., 2015a).

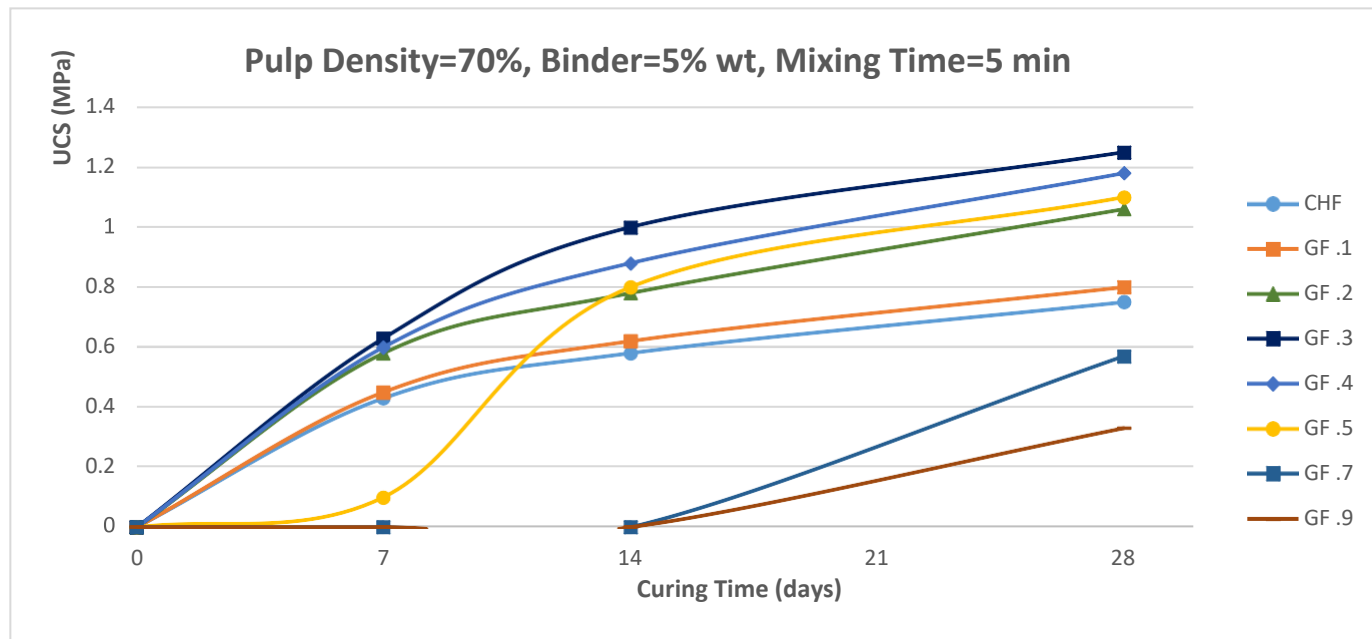


Figure 2.30: Effect of sodium silicate dosage on the uniaxial compressive strength (UCS) of CHF and Gelfill (Kermani et al., 2015a)

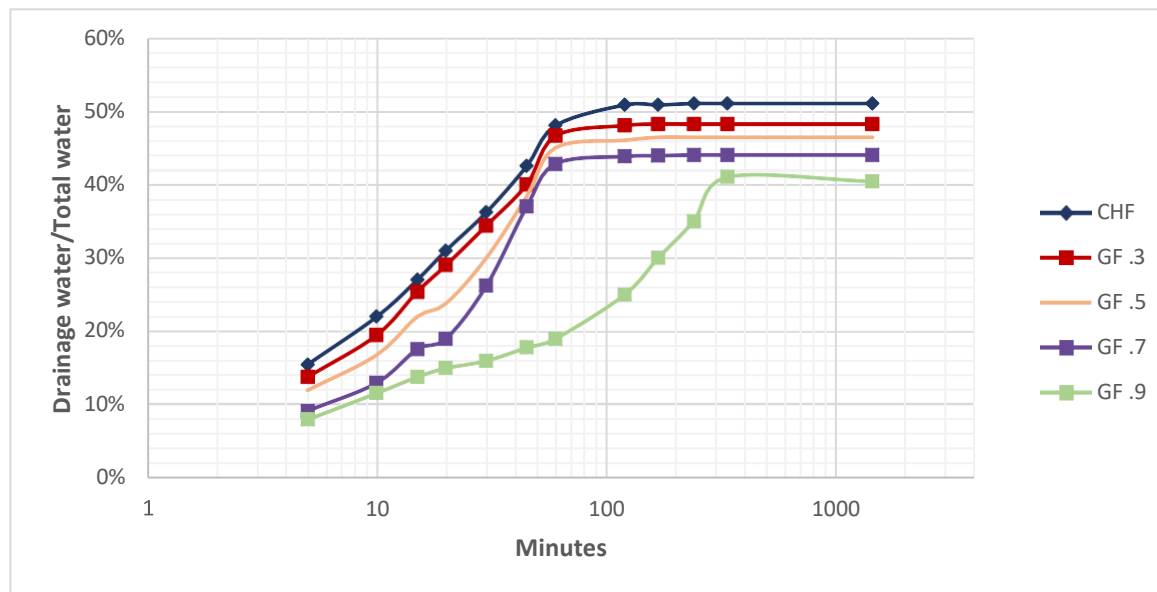


Figure 2.31: The effect of sodium silicate concentration on the drainage of Gelfill and CHF(Kermani et al., 2015)

In another study by Kermani et al. (2015b), the effects of mixing time and curing temperature on the uniaxial compressive strength (UCS) and the microstructure of cement hydraulic filler (CHF) and GF were studied in the laboratory (Kermani et al., 2015b). Table 2.5 shows a set of samples of CHF and GF that were mixed for a duration of 5 minutes to 60 minutes and maintained at 7, 14, and 28 days at 5 to 50 degrees Celsius. It was shown that, increasing mixing time had a negative effect on UCS of GF samples, but had no clear effect on CHF samples. However, curing temperature had a significant positive effect on UCSs of both GF and CHF.

Studies conducted by Guo et al., (2018) present empirical results that focus on the effect of sodium silicate gel (SS) on cemented tailings backfill (CTB) that contains lead-zinc melting slag. It has been found that SS can increase the activation effect and strength of CTB (Figure 2.32) consisting of Portland cement and lead melting slag. Activation involves refining the CTB pore structure, forming more cement hydration products in CTBs, and the acceleration of the binding hydration process, which is confirmed by the results of microstructure analysis and electrical conductivity (EC) (Figure 2.33), respectively. It can be concluded that SS can have an active effect on CTBs that contain lead and zinc melting slag. The activation effect of sodium silicate gel on cement-bonded cavities, which includes lead and zinc melting, can be observed in the development of early-onset CTBs. Figure 2.32 shows the evolution of the UCS with the curing time of the CTBs prepared with SS and without SS. It can be seen in Fig. 2.32 that for CTB, containing lead melting slag, the 28-day UCS is greater when including SS. This higher UCS value can be attributed to the fact that SS acts as an alkali active agent of molten smelting slag, which in turn leads to the induction of hydration of the lead melting slag. It contributes to the progression of CTB's strength. In summary, the results presented

above show that the activation of lead-based melting slag by SS, significantly increases UCS of CTB.

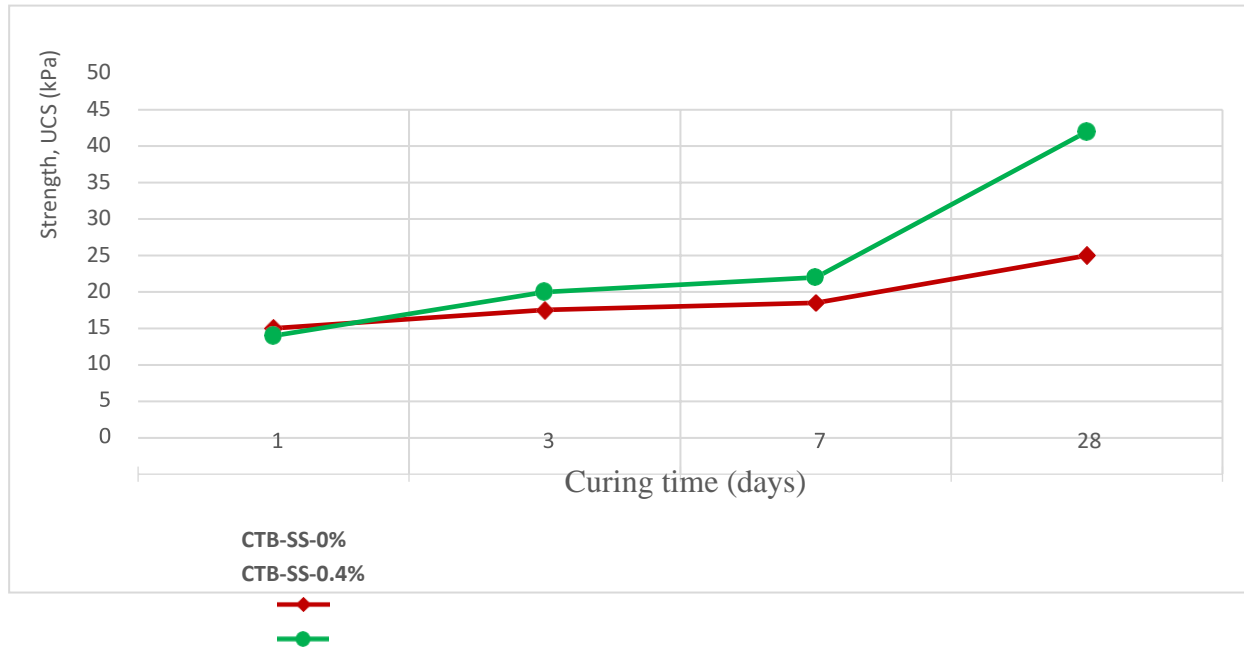


Figure 2.32: The evolution UCS of CTB for different SS content at an early age (Guo et al. 2018).

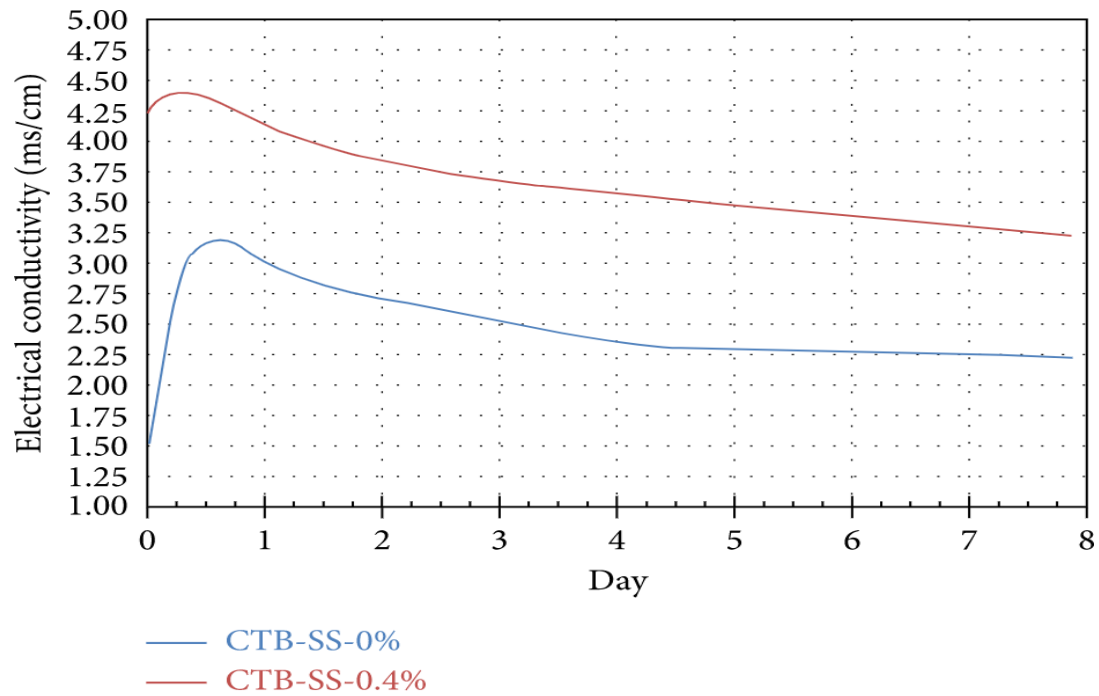


Figure 2.33: Results of electrical conductivity monitoring of CTB that contains (NFS) with SS and without SS (Guo et al. 2018)

Table 2.4. Binder mixtures characteristics of backfill samples (Kermani et al., 2015b)

sample ID	NPC (wt%)	BFS (wt%)	Sodium silicate(wt%)	Binder (wt%)	Mixing time (min)	Curing temperature (°C)
5 min-CHF	0.5	4.5	0	5	5	20
10 min-CHF	0.5	0	0	5	10	20
15 min-CHF	0.5	4.5	0	5	15	20
20 min-CHF	0.5	4.5	0	5	20	20
30 min-CHF	0.5	4.5	0	5	30	20
60 min-CHF	0.5	4.5	0	5	60	20
5 min-Gelfill	0.5	4.5	0.3	5	5	20
10 min-Gelfill	0.5	4.5	0.3	5	10	20
15 min-Gelfill	0.5	4.5	0.3	5	15	20
20 min-Gelfill	0.5	4.5	0.3	5	20	20
30 min-Gelfill	0.5	4.5	0.3	5	30	20
60 min-Gelfill	0.5	4.5	0.3	5	60	20
CHF/5 °C	0.5	4.5	0	5	5	5
CHF/15 °C	0.5	4.5	0	5	5	15
CHF/25 °C	0.5	4.5	0	5	5	25
CHF/35 °C	0.5	4.5	0	5	5	35
CHF/50 °C	0.5	4.5	0	5	5	50
Gelfill/5 °C	0.5	4.5	0.3	5	5	5
Gelfill/15 °C	0.5	4.5	0.3	5	5	15
Gelfill/25 °C	0.5	4.5	0.3	5	5	25
Gelfill/35 °C	0.5	4.5	0.3	5	5	35
Gelfill/50 °C	0.5	4.5	0.3	5	5	50

With increasing the mixing time, the UCS of Gelfill samples decreased, whereas the UCS of CHF samples did not change. The UCS of Gelfill was almost 30% greater than that of CHF for the samples mixed for 5 min. However, the difference between Gelfill and CHF UCSs decreased with increasing the mixing time (Fig. 2.34). Sodium silicate was added to Gelfill in different density (0 wt %, 2 wt%, 6 wt%, 10 wt% and 15 wt %). The initial and final setup time for the NPC / BFS was reduced dramatically by adding sodium silicate (Figure 2.35)

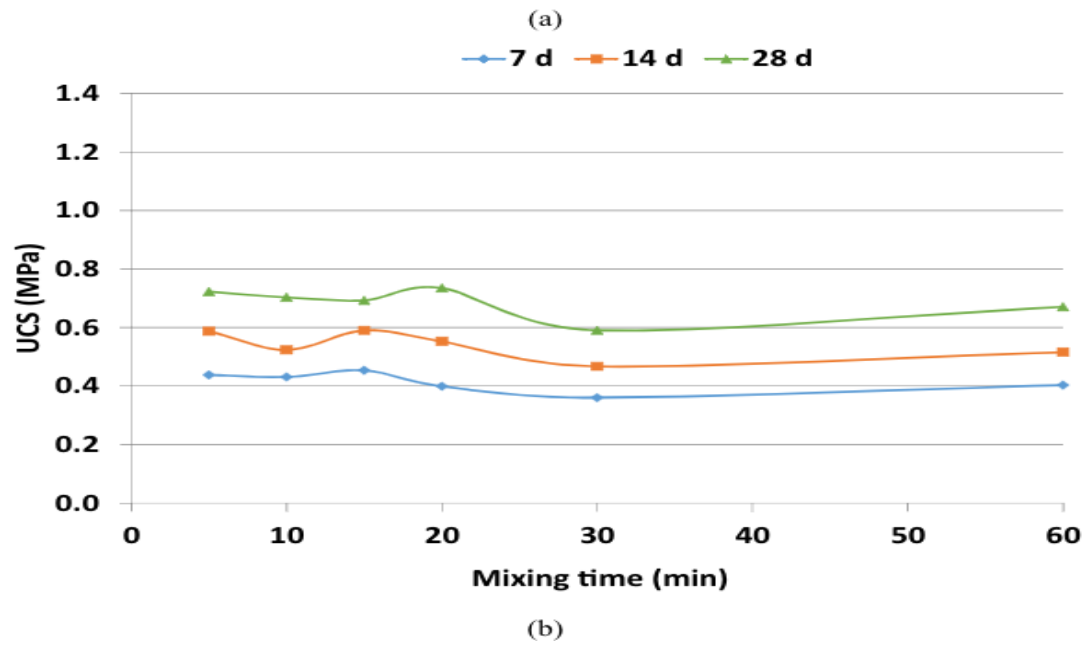
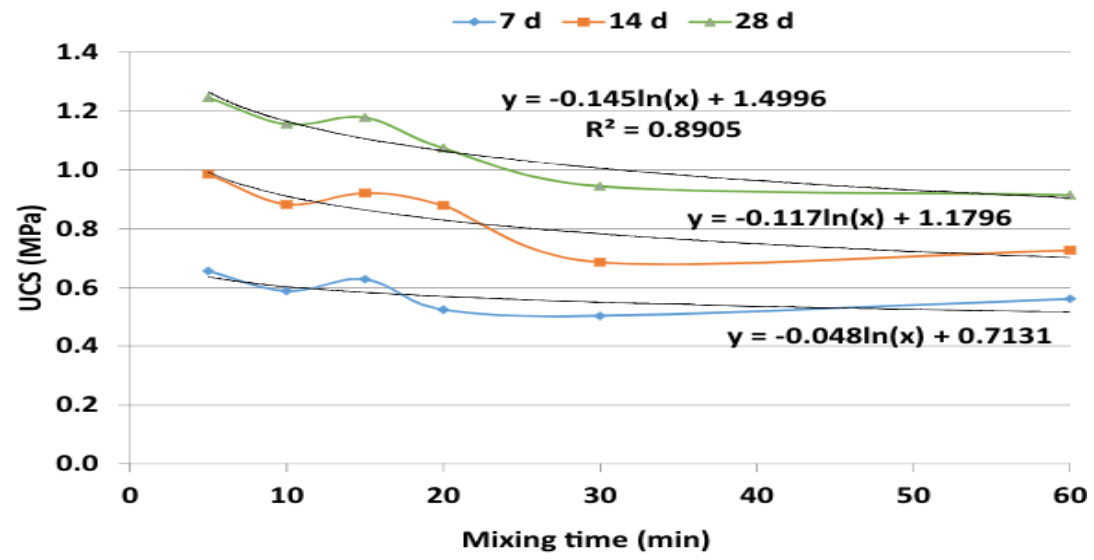


Figure 2.34: Effects of mixing time on the UCSs of (a) Gelfill and (b) CHF samples after three specific days (Kermani et al., 2015).

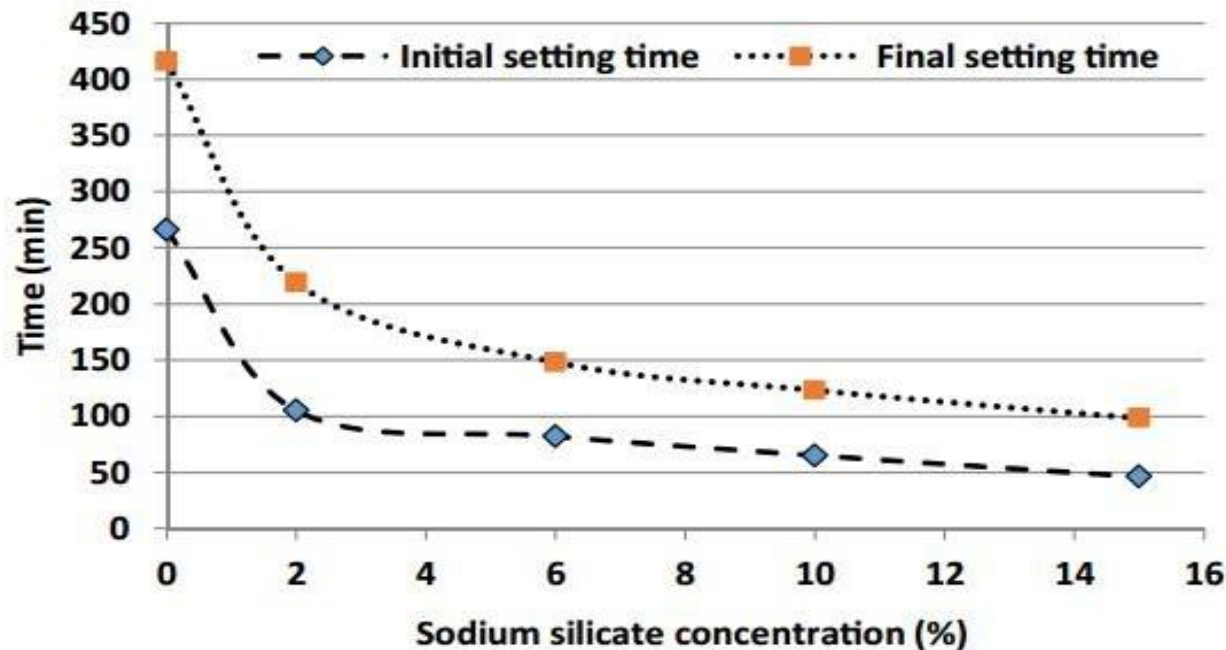


Figure 2.35: Effects of sodium silicate concentration on the initial and final setting times of binder made with NPC and BFS (Kermani et al., 2015b).

In 2011, Abdul Hussain and Fall studied and evaluated the unsaturated hydraulic properties (water retention curve (WRC) or characteristic water curve, AEV, remaining water content, unsaturated hydraulic conductivity) of GF. Various backfill specimens were kept and examined at room temperature at different ages of (3, 7, 28, and 90 days), with different components (Abdul-Hussain and Fall, 2011). Saturated hydraulic conductivity and microstructural tests were conducted; WRCs were measured by using a WP4-T dewpoint potential meter and the saline solution method. Using the equation of van Genuchten (1980), the authors predicted unsaturated hydraulic conductivity of the backfill. Volumetric water content and suction for each GF mix and curing time were presented by the water retention curve (WRC) (Figures 2.36). According to the results obtained by Abdul-Hussain and Fall (2011), the unsaturated hydraulic conductivity of GF decreases when suction, binder content, and degree of hydration are increased (Figure 2.37). Also, in the low suction range, the effect of binder and the amount of hydration was very clear (Abdul-Hussain and Fall, 2011).

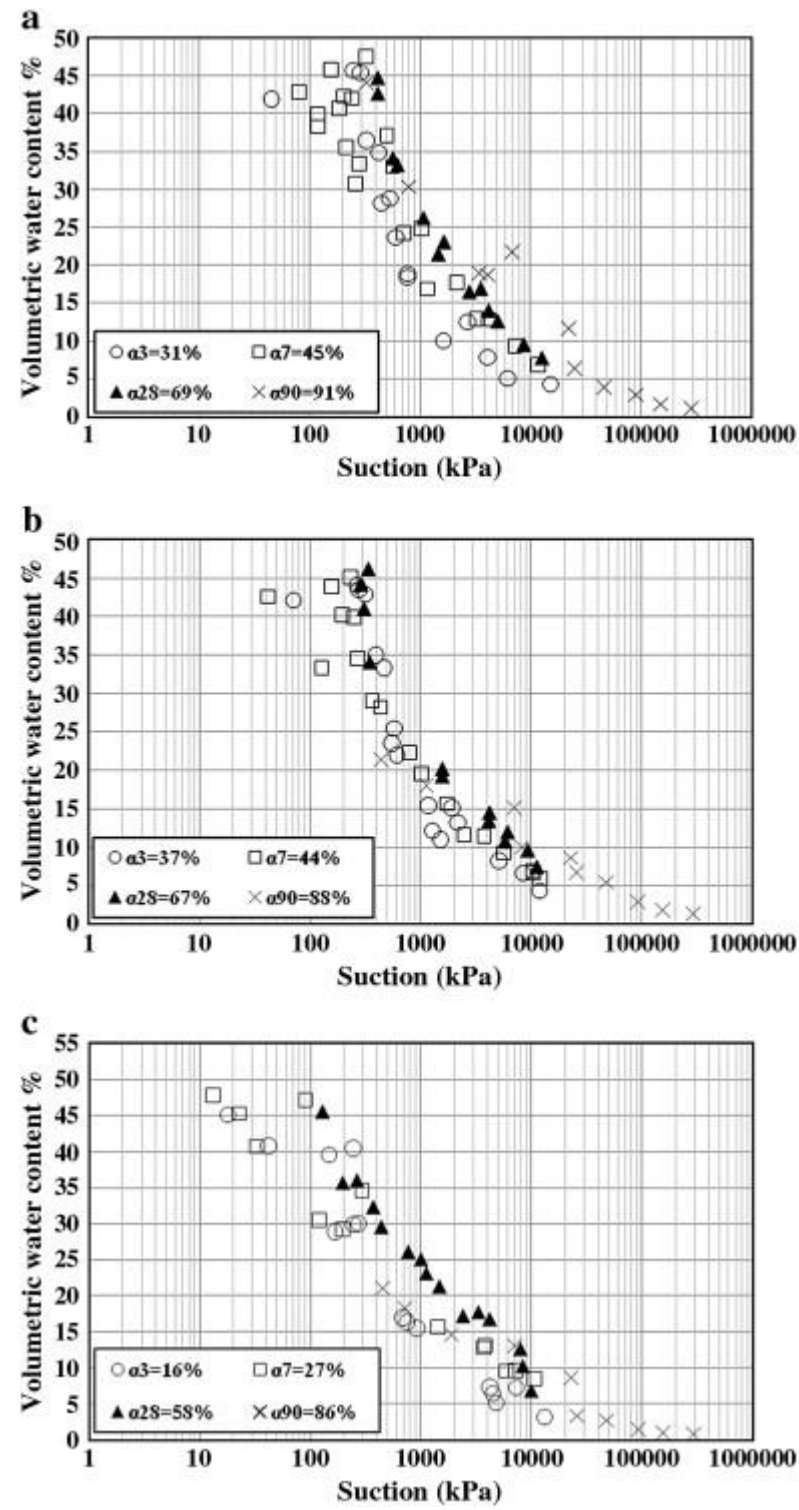


Figure 2.36: The evolution of water retention curve with degree of hydration for GF samples with (a) 6% PCI, (b) 4.5% PCI, and (c) 2% PCI (Abdul-Hussain & Fall, 2011).

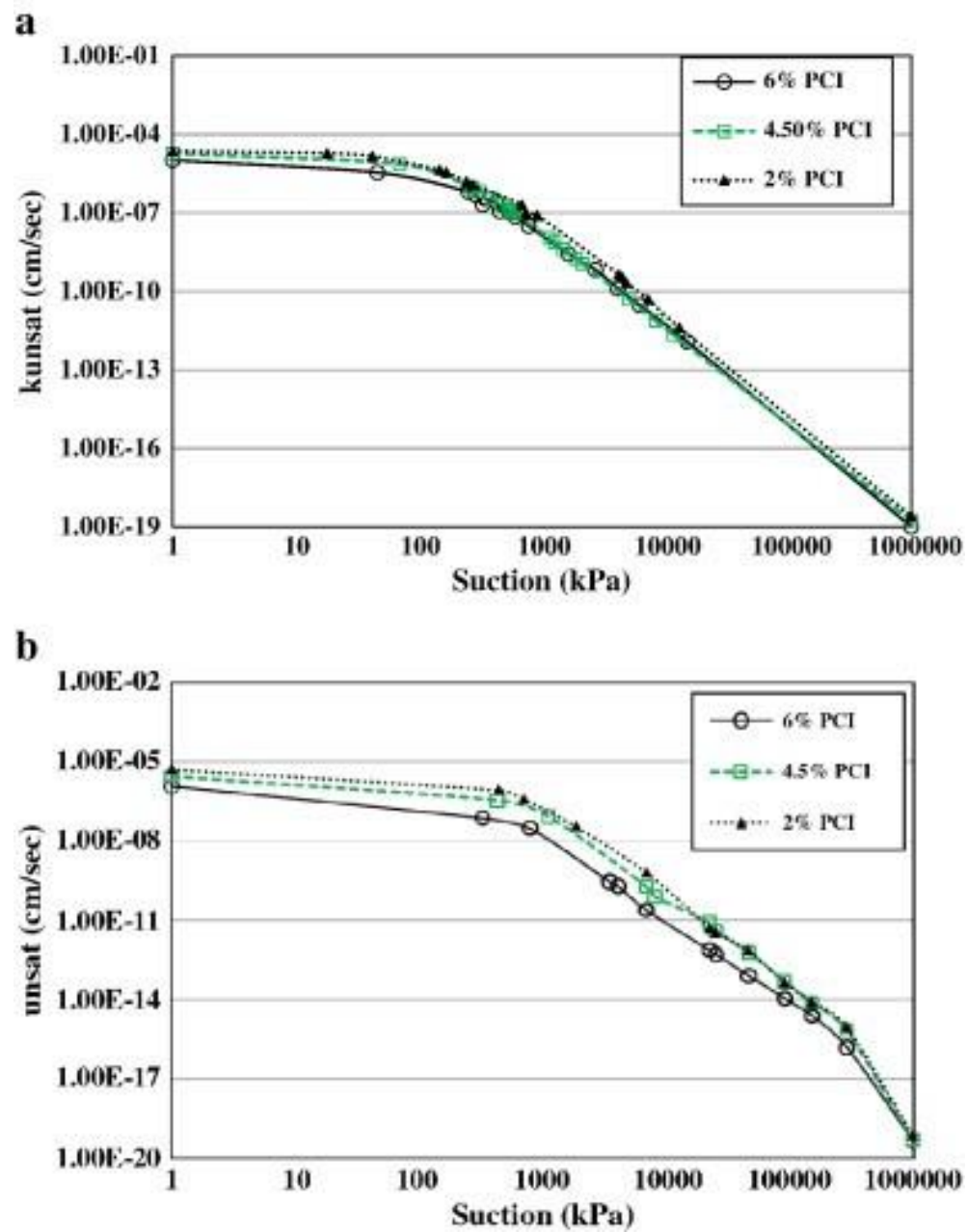


Figure 2.37: Unsaturated hydraulic conductivity of GF vs. suction for various binder contents and curing time of (a) 3 days and (b) 90 days. (Abdul-Hussain & Fall, 2011).

2.7 Conclusions

From the theoretical and technical background information provided above, it can be concluded that CPB with or without sodium silicate, which is a novel ground support and tailings management method, is a complex system due its ingredients and their variability in quantity and characteristics. Moreover, key properties of CPB include physical, mechanical, hydraulic, thermal and chemical properties, which can be affected by several factors (mix composition, temperature, etc.). Several previous studies were conducted to understand the aforementioned properties of CPB without sodium silicate. However, there is a paucity of investigations on the (physical, mechanical, hydraulic, chemical) properties of CPB with sodium silicate. Our understanding of these properties, particularly mechanical and chemical (reactivity), of CPB with sodium silicate is limited.

From the results of the review of previous studies on the strength development and environmental properties of mine cemented backfill the following conclusions can be drawn:

- Despite the significant contribution of the previous studies on the strength development of backfill with sodium silicates to understand the impact of sodium silicate on the strength development of mine cemented backfill, the strength of backfill with sodium silicate at key curing times at the early ages (e.g., 1 day, 3 days) were ignored in many investigations, although these curing times are essential in the assessment of the liquefaction potential of CPB structures and their stability at early ages as well as to determine when the barricades can be opened, which, in turn, has a significant effect on the mining cycle, in other words, on the mine productivity. There is a need to address this knowledge gap. Moreover, the previous studies mentioned above did not provide sufficient insight into the sodium silicate-induced microstructural changes in cemented backfill and their links to backfill strength development. In addition, how the type of binder

and sodium silicate content will change the mechanical response of cemented backfill with sodium silicate was not addressed in these previous studies. These issues should be addressed to develop a deep comprehension of the strength development of mine cemented backfill that contains sodium silicate, which will enable a cost-effective design of backfill structures.

- No previous studies have been performed to assess the saturated hydraulic conductivity and reactivity of cemented paste backfill containing sodium silicate with various types of binders. Thus, there is a need to address this knowledge gap since permeability and reactivity are critical parameters to assess the environmental performance of cemented backfill.

2.8. References

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Chapter 3– Technical Paper I - Strength development of Slag-cemented paste backfill that contains sodium silicate

Abstract

This paper focuses on the strength development of cemented paste backfill containing sodium silicate. A series of laboratory experiments was performed to investigate the effect of sodium silicate as an alkali-activator in a paste backfill with different binder type and content, mineral additive (e.g., fly ash) on the mechanical property (strength). According to the uniaxial compression test results, the addition of an appropriate amount of sodium silicate (0.3%) can enhance the mechanical property of CPB. Meanwhile, testing results reveal that combination of slag and PCI as the binders, raise the strength more than using only PCI in the backfill. Beside this, the microstructure analysis conducted by mercury intrusion porosimeter (MIP) showed that using sodium silicate as an additive to the backfill, can affect the pore size distribution and total porosity which can result in improving the mechanical property of backfill. The results presented in this manuscript will contribute to design cost-effective CPB structures that contain sodium silicate.

3.1 Introduction

Mining industries provide substantial financial revenue in many countries around the world, such as Canada. The economy of some countries mainly depends on these revenues. However, there are some risks associated with mining activities. For instance, beside the extracted valuable materials, large quantities of mining soil wastes (tailings) are also produced. Mining waste can be categorized in three different types such as tailing, waste rock, and water treatment sludge. To control the destructive impact of mining waste on the environment, the effective and environmentally friendly disposal ways should be chosen.

Among all practical disposal techniques, cemented paste backfilling is known as a technology that effectively reduces the environmental impacts of mining soil wastes through recycling the tailings in the form of cemented paste backfill (CPB). CPB technology is practiced in several mine sites around the world (Fall et al., 2008). Besides allowing the mining industry to extract more ores, CPB considerably reduces the quantity of mine waste to be deposited on the surface (Cheng et al., 2009). Typically, CPB is a mixture of dewatered tailings (ranges from 70% to 85%), hydraulic binder (usually cement), often between 3% to 7% to meet the backfill stability requirements, and adequate water to reach the desired consistency for transporting the paste to the mine backfilled stopes (Yilmaz et al., 2009). The CPB must show satisfactory mechanical properties (e.g., strength) to ensure the stability of the cavities and ground in underground mines (Abdul Hussain, 2011). It is known that underground mining creates large underground voids, causing risk of mine collapse in deeper extraction phases. Therefore, the key mechanical design criterion of CPB includes strength (uniaxial compressive strength, UCS). Moreover, a high rate of early CPB strength gain achieved in an economical way is a goal for all in the mining industry. Early strength gain is of special importance for opening of the barricades, scheduling the extraction of adjacent stopes and thus, reduction of the mining cycle time, hence increasing mining efficiency and production. This clearly leads to economic benefit for mines. Cement or a blend of different binders, such as Ordinary Portland cement (OPC), Ground Blast Furnace Slag (Slag) can be used to provide the CPB with the needed strength. However, the cost of CPBs is considerably affected by the cement content. The cement represents up to 75% of the cost of CPB (Fall et al., 2005). Furthermore, fabrication of OPC is highly energy-intensive and thus produces a big quantity of CO₂. These factors have compelled mining companies to look for alternatives to cement that enhance the strength of the CPB, increase the CPB strength gain rate, decrease the cement content and reduce the carbon footprint of the mining industry.

One of the best alternative options is the chemical additives, such as alkali activator, to activate pozzolanic material like binder to quickly reach to the desired mechanical properties or strength (Kermani et al., 2011). One of the alkali activators is sodium silicate or water glass which by adding it to the CPB, the strength of cemented paste backfill can increase. Alkali activators have the ability to absorb a high quantity of water which results in the enhancement of the strength (Hassani et al., 2007; Doucet et al., 2007). Few studies have been carried out to evaluate the mechanical properties of CPB with presence of sodium silicate. For example, Abdul Hussain (2011), Kermani et al. (2015) and Razavi (2007) conducted UCS tests in laboratory on CPB with sodium silicate. The results revealed that the mechanical properties of CPB are significantly influenced by adding sodium silicate to the mixture. However, despite the significant contribution of these previous studies to understand the impact of sodium silicate on the strength development of mine cemented backfill, the strength of backfill with sodium silicate at key curing times at the early ages (e.g., 1 day, 3 days) were ignored in many investigations, although these curing times are essential in the assessment of the liquefaction potential of CPB structures and their stability at the early ages as well as to determine when the barricades can be opened, which, in turn, has a significant effect on the mining cycle, in other words, on the mine productivity. Moreover, the previous studies mentioned above did not provide sufficient insight into the sodium silicate-induced microstructural changes in CPB and their links to backfill strength development. In addition, how the type of binder and sodium silicate content will change the mechanical response of CPB with sodium silicate was not addressed in these previous studies. These issues are addressed in this manuscript to develop a deep comprehension of the strength development of CPB that contains sodium silicate, which will enable a cost-effective design of backfill structures.

3.2 Materials and Experimental Program

3.2.1. Materials

3.2.1.1 Tailings

Two different tailing types (Artificial tailing ST, Natural tailing NT) were used in this study. Typically, tailings represent between 70 and 85% of the weight of the cemented paste backfill. Artificial tailings, called silica tailings (ST), consist of pure ground silica (99.8% SiO₂, manufactured by U.S. Silica Co.). ST are chemically inert material used for accurately controlling the mineralogical and chemical composition of the tailings which can lead to maintaining the level of uncertainties at a minimum level. Also, ST have a particle-size distribution similar to the average grain size distribution of tailings from nine different mines in eastern Canada (Figure 3.1). The particle size of approximately 50% of ST is smaller than 20µm. The second tailing is natural tailings obtained from a Canadian hard rock mine and is called NT. The primary physical properties and mineral composition of the two tailings are shown in Tables 3.1 and 3.2. The particle size distribution of ST and NT is shown in Figure 3.1.

Table 3.1. Primary physical properties of used tailings

Tailings	Gs	D ₁₀ (µm)	D ₃₀ (µm)	D ₅₀ (µm)	D ₆₀ (µm)	C _u	C _c
ST	2.7	1.9	9.0	22.5	31.5	16.6	1.3
NT	2.78	1.6	10.6	29.5	37.8	23.6	1.9

Table 3.2. Mineral composition of used tailings

Tailings/ Mineral (Wt. %)	Quartz	Albite	Dolomite	Calcite	Chlorite	Magnetite	Pyrite	Talc	Magnesite	Pyrrhotite	Spinel	Others	Total
ST	99.8	-	-	-	-	-	-	-	-	-	-	0.2	100
NT	15	32.8	15	4.2	16.1	2.4	1	7	1.8	0.3	1.8	2.6	100

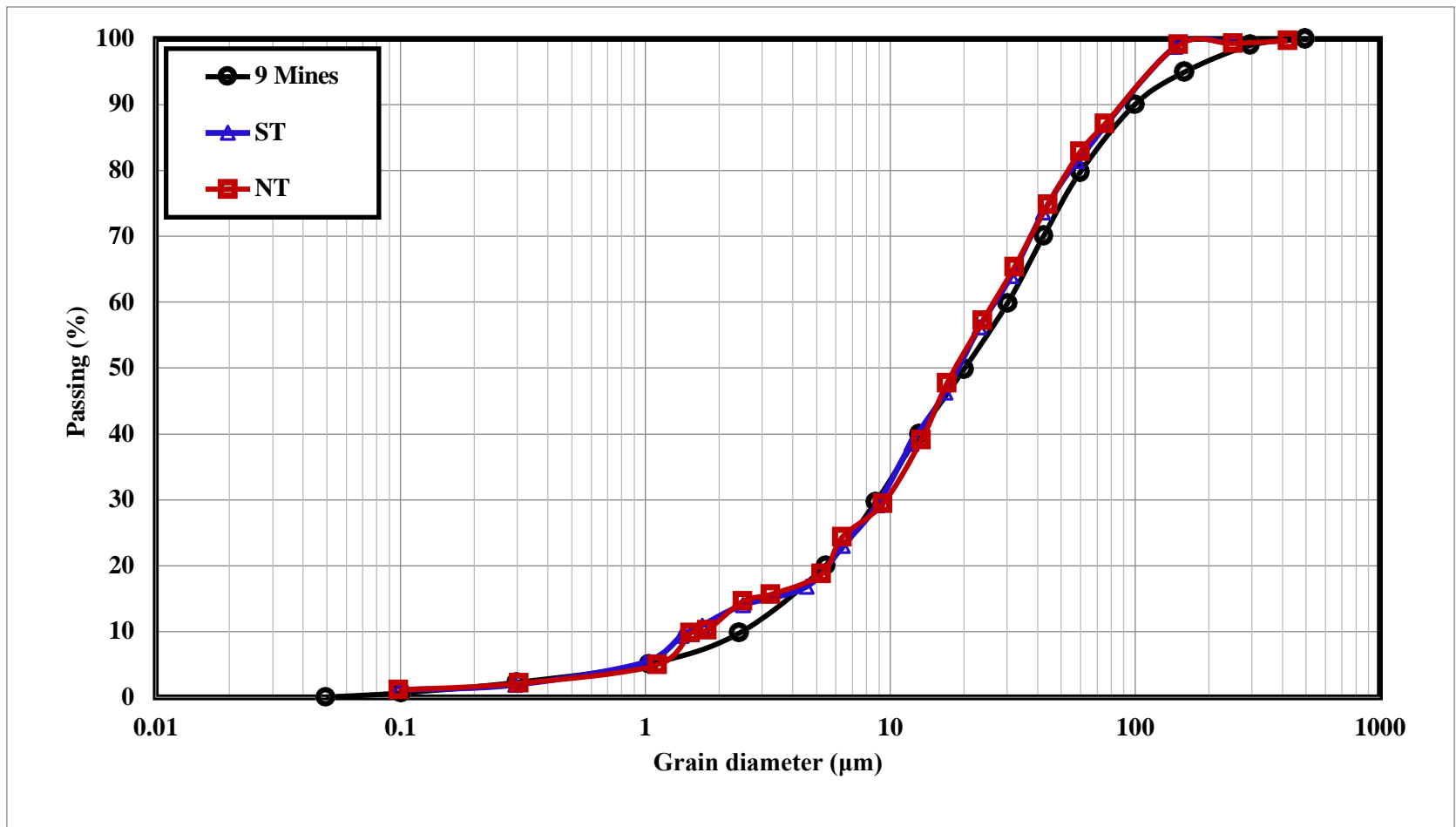


Figure 3.1: Particle size distribution of ST and NT used and average grain size distribution of tailings from nine Canadian mines.

3.2.1.2. Binder

Binder is a key parameter for backfill design optimization in order for the CPB to develop sufficient mechanical strength. The physical and chemical properties of the binder are very important in this regard. In addition, up to 75% of the total mine backfill cost is dependent on the binder cost (Benzaazoua et al.,2004). Therefore, binder is one of the cost-controlling factors at mine backfill method. Portland cement type I (PCI), and blast furnace slag (Slag) were employed as binders in this study. The different combination or blending ratio of these binders is provided in Section 3.3.2. Table 3.3 presents the physical and chemical properties of PCI and Slag.

Table 3.3. Characteristics of Portland cement type I and Slag.

Binder	MgO (wt.%)	CaO (wt.%)	SiO ₂ (wt.%)	Al ₂ O ₃ (wt.%)	Fe ₂ O ₃ (wt.%)	SO ₃ (wt.%)	Rel. Density (%)	Specific surface (m ² /g)
PCI	2.65	62.82	18.03	4.53	2.70	3.82	3.82	1.3
Slag	10.98	41.14	34.23	9.54	-	3.87	3.87	2.2

3.2.1.3. Sodium silicate

Sodium silicate (SS) in reaction acts as an alkaline and accelerator for binders (Hurley & Thornburn, 1972). Sodium silicate type N in the form of liquid is used in this study. It is produced by smelting sand (SiO₂) with sodium carbonate (Na₂CO₃) at high temperature (~1200°C). The ratio of N type is between 1.6 and 3.85 for sodium silicate materials so as a result it is the most efficient activator for ground blast furnace slag. Table 3.4 shows the properties of the sodium silicate used.

Table 3.4. Sodium silicate properties (National Silicates Ltd.)

Characteristics	Values
Na ₂ O (wt. %)	8.90
SiO ₂ % (wt. %)	28.66
Weight ratio, %SiO ₂ /%Na ₂ O	3.22
Specific gravity @ 20 °C	1.39
Solids (wt. %)	37.56

3.2.1.4. Mixing water

To hydrate the binder and achieve acceptable consistence of CPB with respect to transportability, tap water was used in this study.

3.3. Sample preparation and mix proportions

3.3.1. Sample preparation

In order to investigate the effect of sodium silicate on the strength development of CPB, around 100 CPB specimens were prepared by mixing the desired amounts of tailings (ST or NT), binders, sodium silicate and water. The ingredients were mixed for 10 minutes by using a B20F food mixer to produce a homogenous paste. After producing the CPB mixtures, the paste backfills were poured into cylindrical and polyvinyl moulds, 10 cm in height and 5 cm in diameter. The CPBs samples were then cured at 1d, 3d, 7d, 28d and 90 days, respectively. The curing conditions were kept at 20 ±1 °C and 85 ±1% humidity.

3.3.2. Mix proportions

Table 3.5 shows the mix proportions. Also, cement pastes (CP) of CPB were prepared to study the microstructure, as shown in Table 3.6.

Table 3.5. Prepared CPB samples to study the strength (UCS) development of CPB that contains sodium silicate

CPB Sample	Binder	Binder content (wt%)	Binder ratio	Tailings Type	Sodium Silicate (wt%)	w/c ratio	Curing times (Day)
ST-Slag-CPB-0.33SS	PCI/Slag	4.5	50/50	Silica Tailing	0.3%	7.6	1, 3, 7, 28, 90
NT-Slag-CPB-0.33SS	PCI/Slag	4.5	50/50	Natural Tailing	0.3%	7.6	1, 3, 7, 28, 90
ST-Slag-CPB	PCI/Slag	4.5	50/50	Silica Tailing	0.0%	7.6	1, 3, 7, 28, 90
ST-Slag-CPB	PCI/Slag	4.5	50/50	Silica Tailing	0.1%	7.6	1, 3, 7, 28, 90
ST-Slag-CPB	PCI/Slag	4.5	50/50	Silica Tailing	0.5%	7.6	1, 3, 7, 28, 90
NT-Slag-CPB	PCI/Slag	4.5	50/50	Natural Tailing	0.0%	7.6	1, 3, 7, 28, 90
ST-Slag-CPB-SS-25	PCI/Slag	4.5	25/75	Silica Tailing	0.3%	7.6	1, 3, 7, 28, 90
ST-Slag-CPB-SS-75	PCI/Slag	4.5	75/25	Silica Tailing	0.3%	7.6	1, 3, 7, 28, 90

Table 3.6. Prepared cement paste of CPB to study the microstructure (TG/DTG; XRD)

CPB Sample	Binder	Binder content	Binder ratio	Tailings Type	Sodium Silicate (wt%)	w/c ratio	Curing times (Day)
ST-Slag-CPB-0.33SS-CP	PCI/Slag	4.5	50/50	Silica Tailing	0.3%	1	1, 3, 7, 28, 90
ST-Slag-CPB-0.0SS-CP	PCI/Slag	4.5	50/50	Silica Tailing	0.0%	1	1, 3, 7, 28, 90
ST-Slag-CPB-SS-25-R	PCI/Slag	4.5	25/75	Silica Tailing	0.3%	1	1, 3, 7, 28, 90
ST-Slag-CPB-SS-75-R	PCI/Slag	4.5	75/25	Silica Tailing	0.3%	1	1, 3, 7, 28, 90

3.3.3. Testing of specimens

3.3.3.1. Unconfined compression strength test

Unconfined compression tests (UCS) were performed on the specimens at 1, 3, 7, 28, 90 days. The specimens were tested according to the ASTM C39 by means of a computer controlled mechanical press. The press has a normal loading capacity of 50 kN. All samples were tested at a constant deformation rate of 0.8 mm/min. All test data were collected using a computerized data logging system. Finally, the computer software Lab-View used to analyze the collected results. To obtain reliable result, at least two samples were tested for each age.

3.3.3.2. Microstructural analyses

Thermogravimetry (TG) and Differential Thermogravimetry (DTG) technique was used to experimentally investigate the effect of sodium silicate on the evolution of binder hydration products formed in the CPB. TG/DTG tests were performed on cement pastes of CPB with and without sodium silicate. Before testing, all of the samples were dried at 45°C up to 4 days to be prepared for the test. TG/DTG tests were performed on 10 mg (± 0.5 mg) of powdered samples (oven dried at 45 °C) by using an Ulvac TGD 9600 thermal analysis system at a heating rate of 10 °C/min from an ambient temperature up to 1000 °C in an atmosphere of Nitrogen gas (rate of flow: 100 cm³ min⁻¹). The tests were done on samples made of binder and water with a water/binder ratio of 1. The main reason behind choosing this ratio is to simulate the high water content of CPB.

X-ray diffraction (XRD) analysis tests were used to assess the effect of sodium silicate on the mineralogical composition of the cemented paste backfill and progression of cement hydration on samples cured at room temperature for 90 days. The XRD tests were conducted by using a Scintag XDS-2000

diffractometer on dried and grounded samples.

Mercury Intrusion Porosimetry (MIP) technique was used with a Micromeritics Auto-Pore III 9420 mercury porosimeter on CPB samples to characterize the effect of sodium silicate on the pore structure of cementitious materials. Before conducting a MIP test, each sample was first dried at 50 °C until reaching a constant mass. Drying at this temperature can prevent the formation of cracks (Fall et al., 2010).

3.4. Results and discussion

3.4.1. Evolution of mechanical properties

Figures 3.2 and 3.3 illustrate the effect of sodium silicate (0.3%wt) on the relationship between uniaxial compressive strength and curing time of the paste backfill samples made with silica tailing (ST) and natural tailing (NT), respectively, whereas Figure 3.4 shows the strength development of CPB that incorporates different amounts of sodium silicate. Each compressive strength represents an average value gained from two tests at least.

From these figures, it obvious that, as expected, the strength of the backfill samples increases as the curing time increases (expect the 90 days-CPB without sodium silicate). This is because longer curing results in a higher degree of cement hydration – that is, more cement hydration products (C-S-H, CH) will be generated within the CPB system (Fall et al., 2010). More hydration products are associated with an enhancement of the cohesion of the CPB and refinement of its pore thereby leading to the strength development of CPB (Fall et al., 2010). C-S-H is the main strength giving component in a cementitious material (Zhou and Glasser, 2001). Lower strength observed at 90 days for the CPB made with NT and sodium silicate may be attributed to the presence of sulphate ions in NT, which may lead to sulphate attack and/or an interaction between the sulphate ions and the sodium silicate.

Future studies should elucidate the interactions between sulphate ions and sodium silicate in CPB systems, and their effect on CPB strength. From Figures 3.2 and 3.3, it can be also clearly observed that, regardless of the type of tailings used, the strength development of the Slag-CPB samples is significantly influenced by the sodium silicate, particularly at the early ages (up to 28 days). It can be seen that the CPB samples with sodium silicate acquired consistently higher strength than those without any alkali activator. For example, the 28 day-strength of the CPBs with sodium silicate is approximately 50% higher than those without sodium. The 28 day-UCS values of the CPBs with sodium silicate are higher than 1.0 MPa, while the values of the samples without sodium silicates are lower than 1.0 MPa. In CPB practice, a UCS of ≥ 1.0 MPa at 28 days is often regarded by mine operations to be the threshold for the self-supporting stopes and the mined stopes adjacent to ore extraction (Stone, 1993). These figures also show the sodium silicate significantly affects the rate of gain in early age strength of CPB. This has important practical applications. A high rate of early CPB strength gain achieved in an economical way is a goal for all in the mining industry. Early strength gain is of special importance for opening of the barricades, scheduling the extraction of adjacent stopes and thus, reduction of the mining cycle time, hence increasing mining efficiency and productivity. This is clearly associated with profits for mines. Furthermore, this high early age strength gain may play a significant role in reducing the potential for CPB liquefaction at an early age of cure and thus decreasing barricade failure risk. This is because the binder hydration product is able to create bonds between individual tailings particles of the CPB and provides strength (Fall et al. 2010). This observed higher strength and strength gain rate of the specimens with sodium silicate can be attributed as the result of the fact that sodium silicate acts as an alkaline activator of the Slag which is correlated with the stimulation of the hydration of PCI-Slag binder and thus the production of a larger quantity of cement hydration products (e.g., C-S-H, CH), as supported by the XRD and TG/DTG analyses presented below. More hydration products (e.g., C-S-H) are associated with an enhancement of the cohesion of the CPB and refinement of

its pore structure (as supported by the MIP results below), thereby leading to the strength development of CPB. Indeed, it is generally agreed that soluble sodium silicate doubly contributes to strength development, both by acting as an alkaline activator and giving rise to the formation of a C-S-H of low C/S ratio (Fernandes-Jiménez et al., 2009). This activation results in an acceleration of the binder hydration. Combined with the formation of additional C-S-H gel due to the reaction of SiO_2 (formed in the hydrolysis of sodium silicate solution) with CH (generated by the hydration of the PC), this activation contributes to the increase of SS-CPB (CPB with sodium silicate) strength. This scenario is not possible with sodium silicate-free CPB due to the absence of sodium silicate within this backfill material.

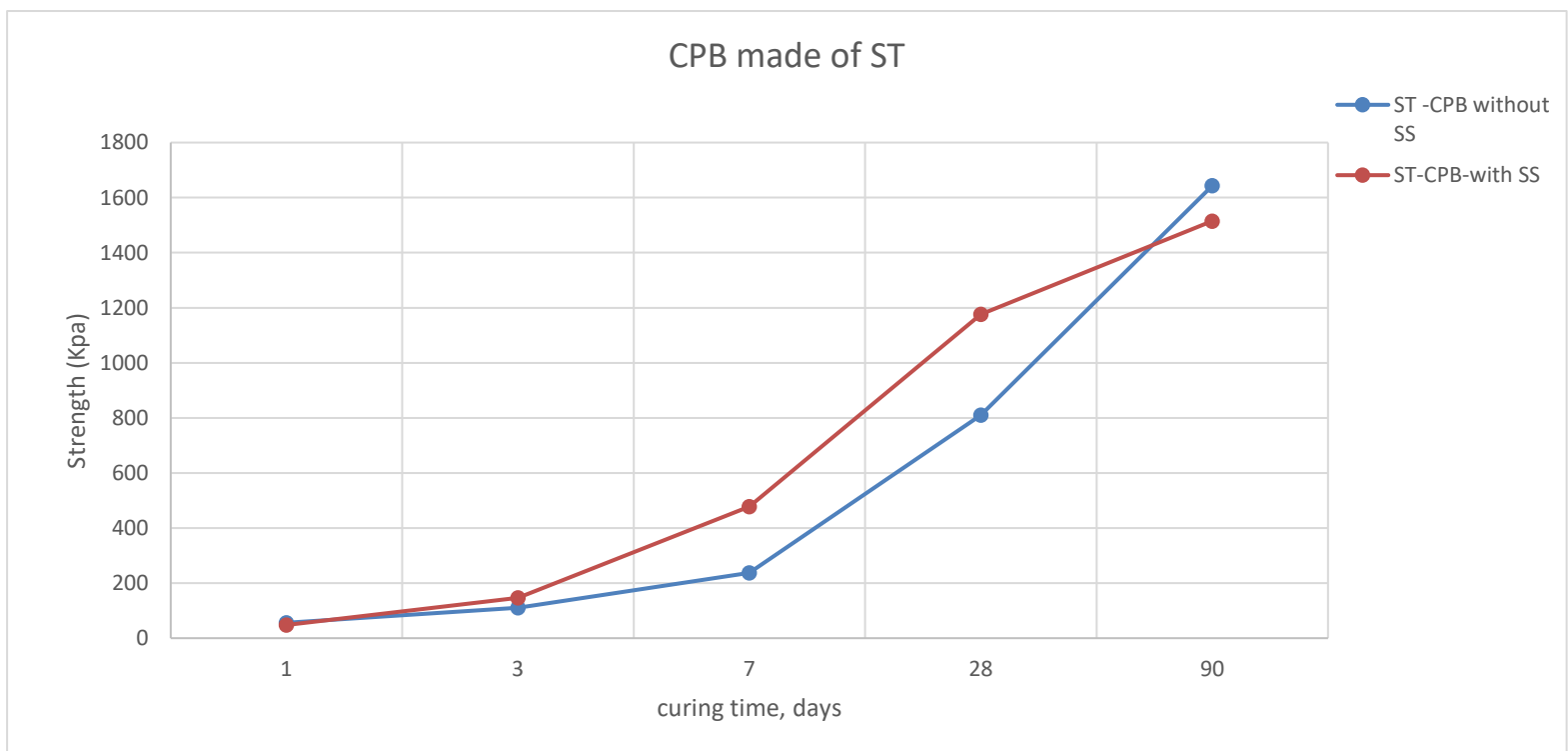


Figure 3.2: The strength development of ST-CPB (made of ST) samples containing sodium silicate

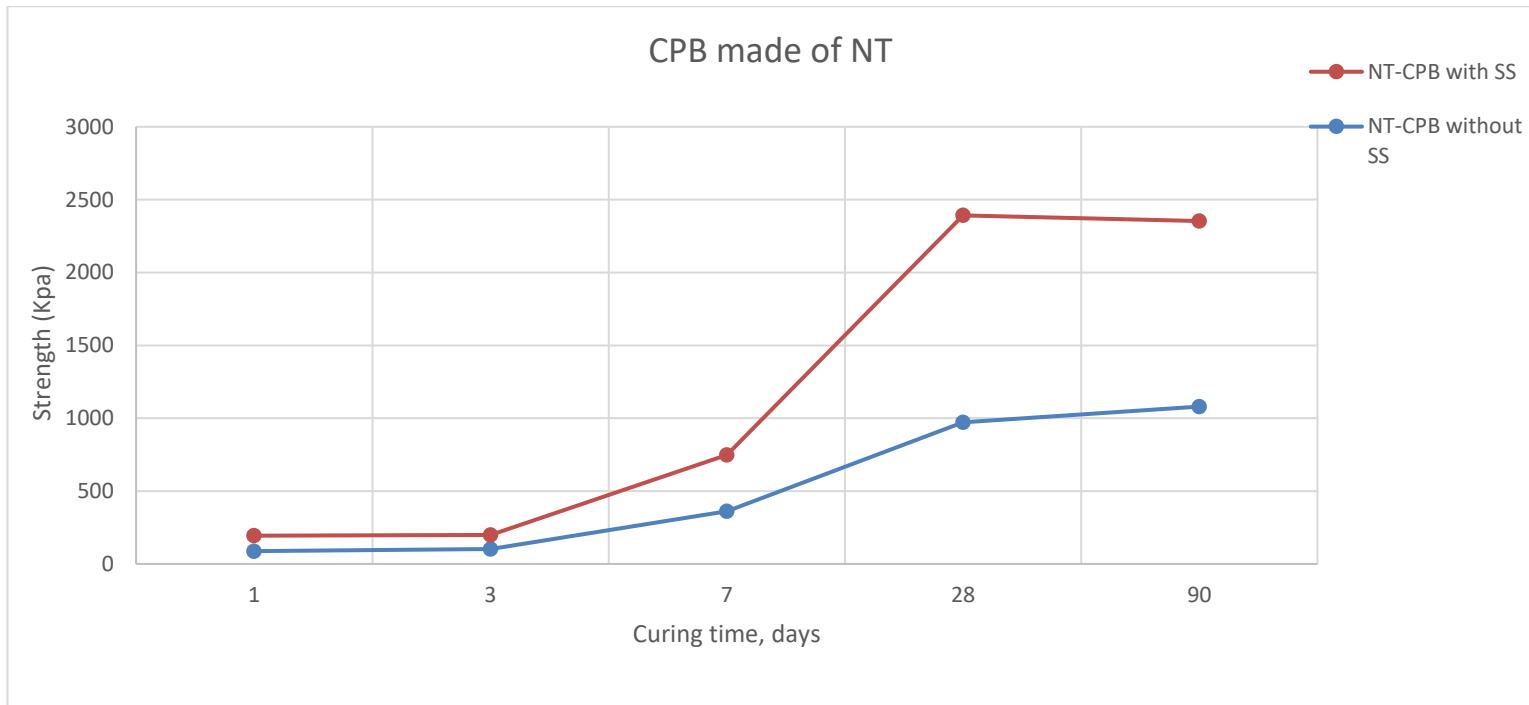


Figure 3.3: The strength development of the NT-CPB samples (made of Natural Tailing) containing sodium silicate

However, the results presented in Figure 3.4 indicates that the UCS value and sodium silicate content is not a simple linear relationship. Indeed, there is a critical sodium silicate content (0.3%wt in this study) beyond which the UCS decreases as the sodium silicate content is increased. This increase of UCS with the sodium silicate content (up to 0.3%) could be attributed to higher Na_2O in the backfill mixture, which also impacts the alkali content of the CPB system (Cengiz et al. 2009). More work is needed to understand this decrease of UCS as the sodium silicate content goes beyond 0.3%wt.

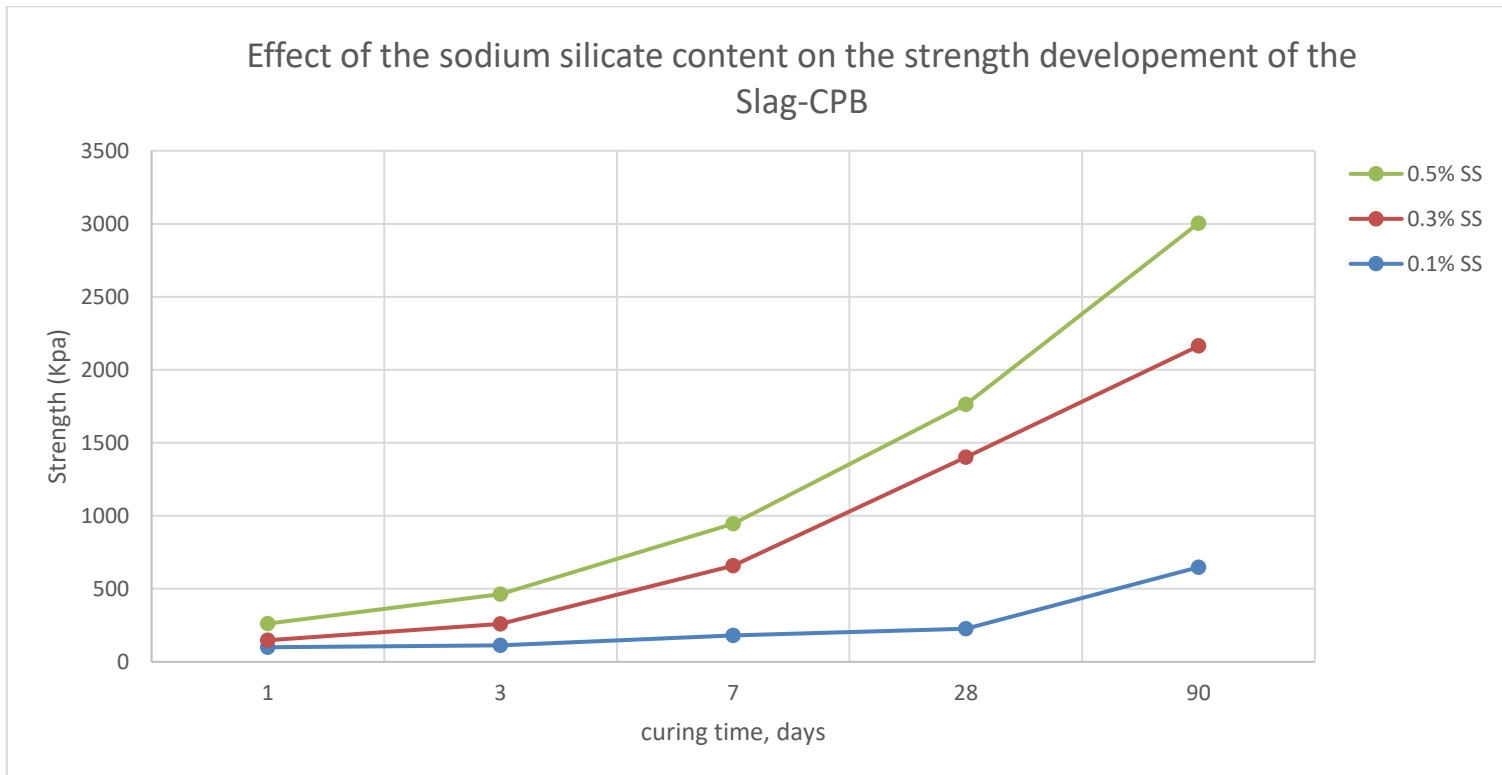


Figure 3.4: The effect of various sodium silicate contents on the strength of CPB samples

Figure 3.5 highlights the effect of sodium silicate (0.3%wt) on the strength development of CPB with PCI/Slag (25/75), PCI/Slag (50/50), and PCI/Slag (75/25). From this figure, it can be observed that the PCI/Slag ratio or binder type has a significant impact on the strength development of CPB with sodium silicate. The SS-CPBs with PCI/Slag (50/50) and PCI/Slag (75/25) show the highest strength. The UCS of the PCI/Slag (50/50)-CPB with sodium silicate is only slightly lower than (or close to) that of the PCI/Slag (75/25) with sodium silicate. The slightly lower value of UCS of PCI/Slag (50/50) or significantly lower UCS value of PCI/Slag (25/75) can be mainly attributed to their lower amounts of C_2S and C_3S (Pokharel and Fall, 2010).

The partial (50%) or (75%) replacement of PCI with slag provides these blended cements with only 50% of 75% of C_2S and C_3S , respectively, in comparison to PCI. Hence, the quantity of C_3S and C_2S is reduced at advanced ages and as curing time increases, owing to the hydration reaction in the cement matrix, and thus the blended cements produce smaller amounts of C-S-H and CH. This means that fewer CH will be available in the backfill system to activate the slag grains in the PCI/Slag (50/50) or PCI/Slag (25/75) backfill systems. Thus, less secondary C-S-H will be formed.

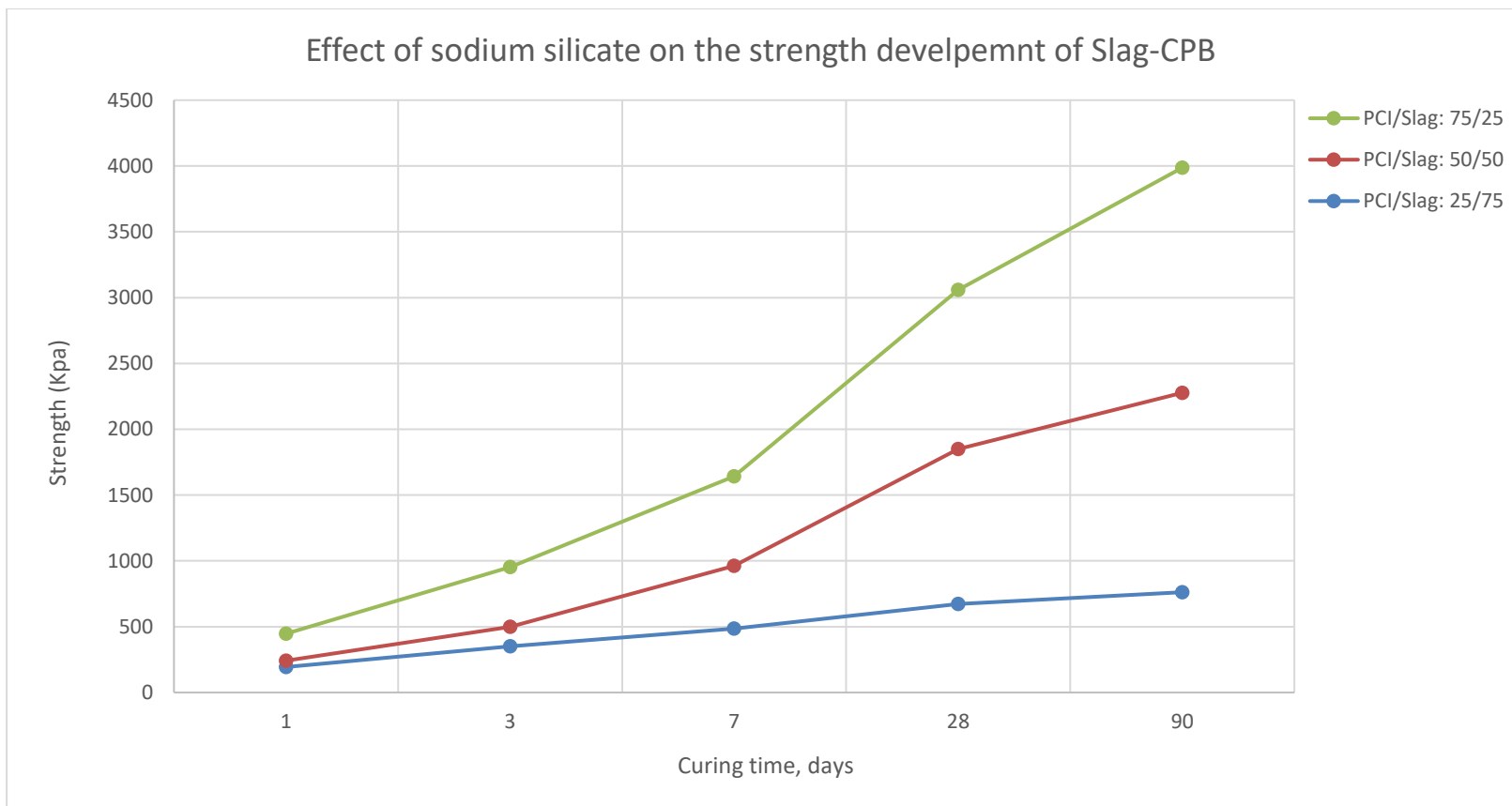
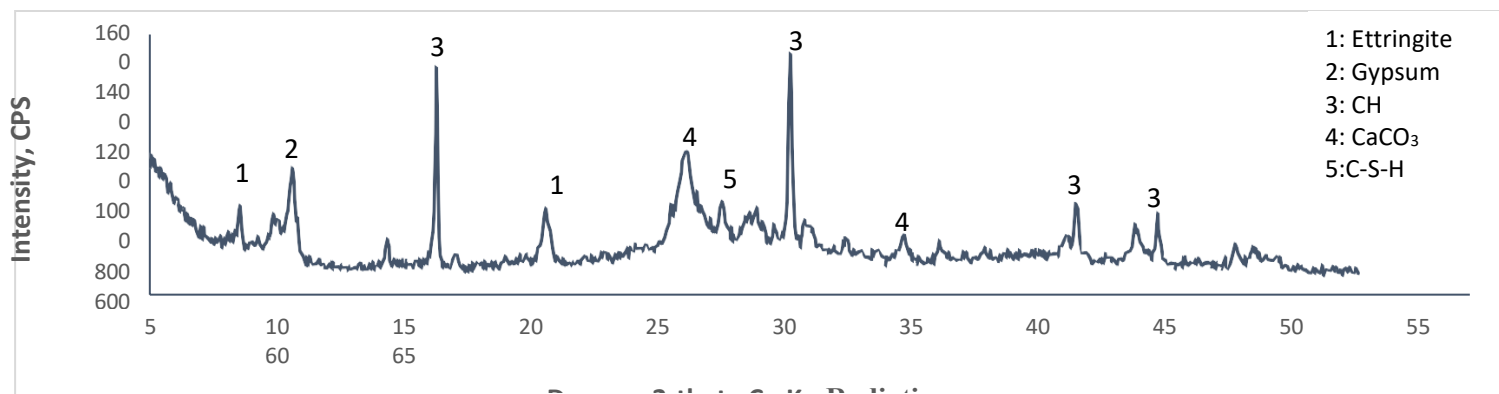


Figure 3.5: The effect of various PCI/Slag ratios on the strength CPB samples with 0.3% sodium silicate

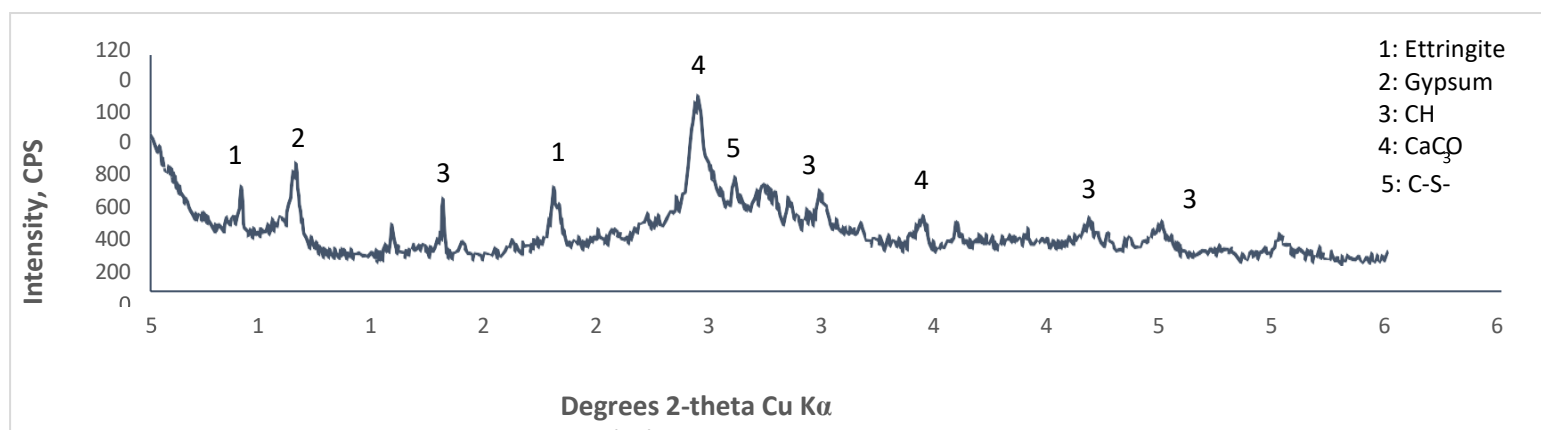
3.4.2. Evolution of microstructural properties

3.4.2.1. XRD Results

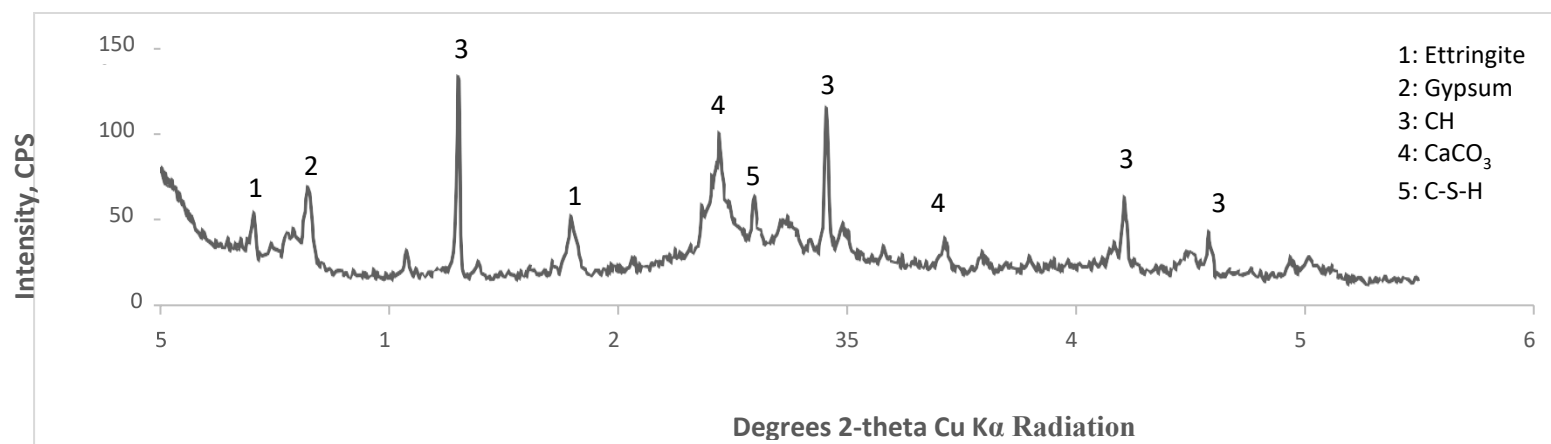
Figure 3.6 presents the XRD results of CPB samples activated by sodium silicate (SS) and without SS with different binder ratio (PCI/Slag: 50/50; 25/75) and cured at 20°C for 28 days. These results show clearly that adding activator like SS to the CPB samples leads to the production of more binder hydration products. From this figure, it can be observed that, the intensity of calcium hydroxide (CH) at 18, 34 and 47 degree 2-theta is comparatively higher in the cement paste with sodium silicate than in the sodium silicate free samples. This indicates that less CH are formed in the sodium silicate-free samples. This increased formation of CH has a positive effect on the strength gain of CPB-SS since CH is the main binding phase in hardened cement. Also, by comparing XRD patterns of CPB samples containing SS with different binder ratio (PCI/Slag: 50/50; 25/75), the results clearly show that both XRD patterns are similar, however the intensities of the peaks for CH are higher in the sample with PCI/Slag (50/50) than in the PCI/Slag (25/75) specimen, which is consistent with the UCS results presented and discussed previously.



a-CPB sample with 0.3%SS and PCI/Slag (50/50)



b-CPB sample without SS and PCI/Slag (25/75)



c-CPB sample with 0.3%SS and PCI/Slag (25/75)

Figure 3.6: XRD plots of cured CPB samples at 20°C for 28 days, (a) CPB sample with 0.3% of SS and with PCI/Slag (50/50) (b) CPB sample without SS (c) CPB sample with 0.3%SS and PCI/Slag (25/75)

3.4.2.2 TG/DTG Results

Fig. 3.7 presents TG-DTG diagrams of the cemented pastes with and without sodium silicate and cured for 28 days. Indeed, the contribution of sodium silicate to the formation of more hydration products is also confirmed by the results of TG/DTG tests. This test presents weight loss due to the decomposition of different phases in the paste. As can be seen in the Figure 3.7, three main endothermic peaks occurred in the ranges of 50-200 °C, 400-450 °C and 600 -700 °C which is related to rapid weight loss and transformation of hydration products. The first peak or weight loss is located between 50°C and 200°C. This major phase transformation results from the dehydration reactions of ettringite, gypsum, C-S-H and calcium aluminate. Based on several studies, the decomposition of gypsum, ettringite and loss of water from part of the carboaluminate hydrates happen in the temperature range of 110-170°C while previous researches showed that losing of bound water from the decomposition of the C-S-H at 170-300°C temperature is the main reason of the weight loss at the mentioned temperature (Anderberg, 1997; Noumowe, 1995; Zhou and Glasser; 2001; Khoury, 1992). The second weight loss occurs at 400-500°C, resulting from the dehydroxylation of CH. The third main peak observed at 600-700°C, which is attributing to the decomposition of amorphous carbonated phases and decomposition of calcite (CaCO_3) (Andeerberg, 1997; Noumowe, 1995). By comparing the three TG-DTGs diagrams of PCI-CPB free sodium silicate and PCI-CPB containing sodium silicate with different PCI ratio, the endothermic peaks or weight losses are higher for cement paste with sodium silicate. In other words, the activation of Slag by sodium silicate leads to the formation of more cement hydration products (C-S-H, CH) in the SS-CPB specimens. and higher PCI. This is positive for the SS-CPB strength development, since the cement hydration products will enhance the cementation of the tailings particles as well as decrease the capillary porosity of the SS-CPB. This refinement of the pore structure of SS-CPB with sodium silicate is confirmed by the results of mercury intrusion porosity (MIP), as discussed below. Moreover, the comparison of the TG/DTG diagrams of the cement pastes of PCI/Slag

(50/50)-CPB with SS and PCI/Slag (25/75)-CPB with SS shows that the endothermic peaks or weight losses are higher for PCI/Slag (50/50)-CPB with SS. These experimental observations indicate that the amount of hydration products formed in the PCI/Slag (50/50)-CPB with SS is higher, which is agreement with the UCS results presented in Figure 3.5.

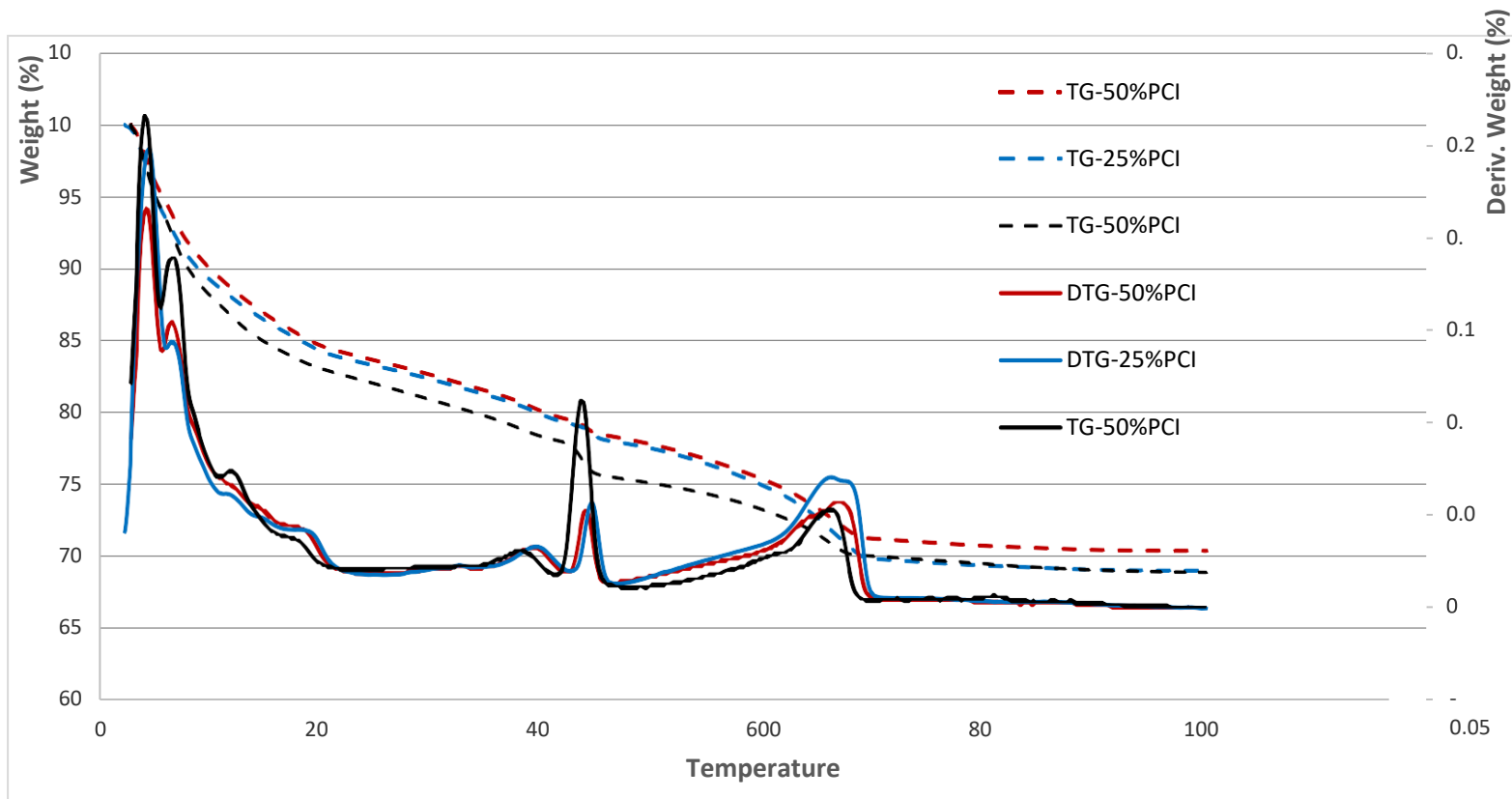


Figure 3.7: TG and DTG diagrams for cemented paste samples containing sodium silicate and sodium silicate-free CPB samples

3.4.2.3. MIP Results

Figure 3.8 shows the results of the MIP tests performed on 90-day cemented paste backfill samples with 0.3%SS and without SS made of different ratio of PCI/Slag (50/50; 25/75). From this figure, it can be observed that the sample without sodium silicate and with 50% PCI has a larger proportion of macropores (30-100 μ m) than the samples with sodium silicate. In fact, more macro-porosity results in decreasing UCS

value of CPB (Li and Aubertin, 2003; Fall et al. 2010). Figure 3.8 also indicates that the SS-CPB with lower amount of PCI (PCI/Slag: 25/75) has a higher percentage of macropores (30-100 μm) than the SS-CPB with higher proportion of PCI (PCI/Slag: 50/50). This finding is agreement with the UCS presented in Figure 3.5 and discussed previously.

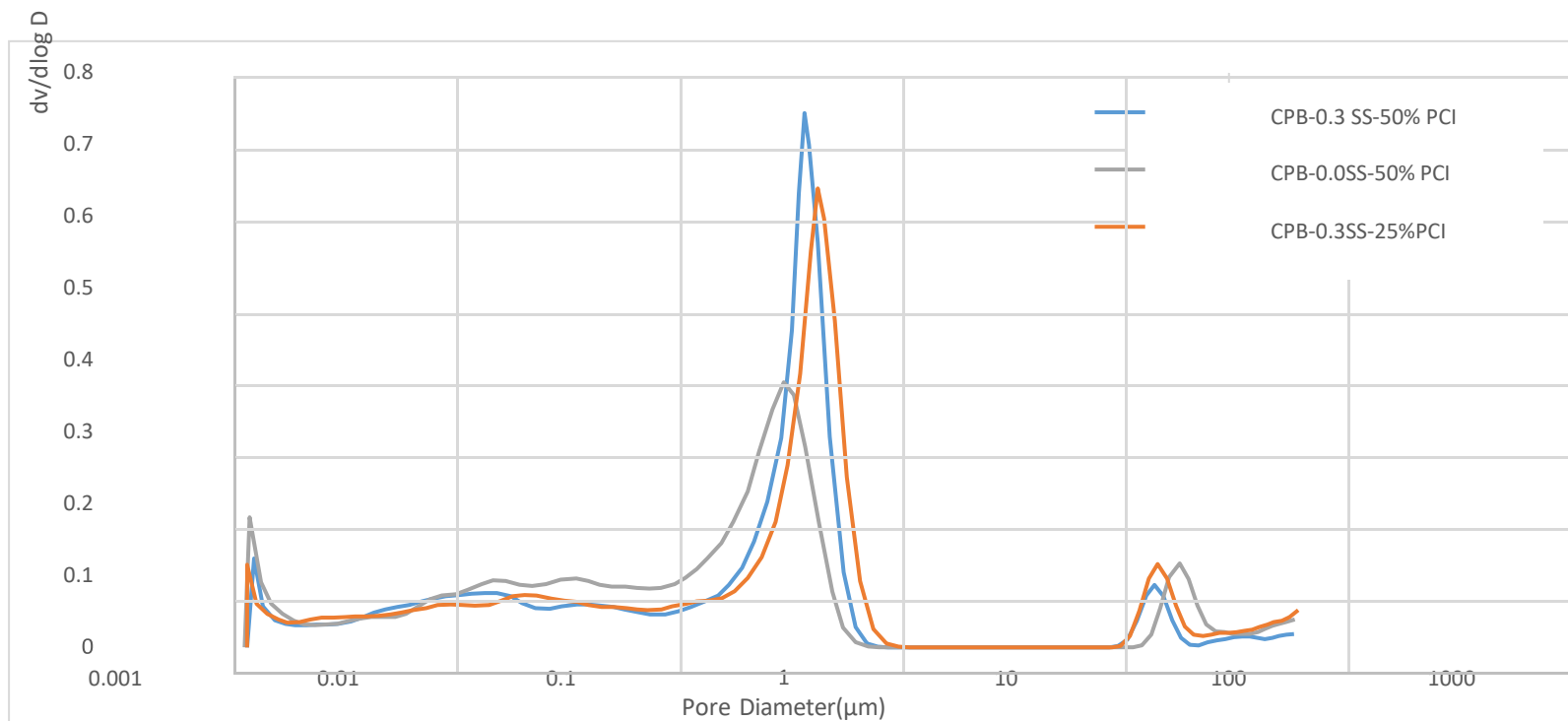


Figure 3.8: Differential pore size distribution curves of 90 days CPB specimens (w/c= 7.6) cured with sodium Silicate with variety binder ratio

3.5. Conclusions

The current paper presents the experimental results of the effect of sodium silicate on the strength development and microstructural properties of CPB. The obtained results show that sodium silicate has an influential effect on the mechanical (strength) and microstructural properties of CPB. Sodium silicate can significantly increase the strength of Slag-CPB and the early age strength gain. This has significant

implications with respect to increasing mining cycles and productivity. This increase is due to the activation of the Slag by the sodium silicate, which results in the formation of more cement hydration products. The results obtained have also shown the blending ratio of PCI and Slag has a significant influence on the effect of sodium silicate on the strength development of CPB.

Using sodium silicate allowed to produce CPBs with decreased total volume of macroporosity through reducing the number of capillary pores which refine the microstructure. This refinement is the result of more cement hydration in the CPB containing SS and the acceleration of the binder hydration process, which has been confirmed by the results of microstructural analyses (XRD, MIP and TG/DTG).

This study provides essential information for understanding the evolution of mechanical and microstructural properties of CPB with sodium silicate. The findings of this paper will help mining operators and engineers who work with SS-CPB materials to take into consideration, the geotechnical aspects, and stability assessment of SS-CPB structures.

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Chapter 4 – Technical Paper II – Permeability and reactivity of Slag-cemented paste backfill that contains sodium silicate

Abstract

Key environmental properties of sodium silicate-bearing cemented paste backfill (SS-CPB) are investigated in this study. These properties include the permeability and reactivity of SS-CPB. Cemented paste backfills containing sodium silicate consists of tailings, water and binder, along with the addition of sodium silicate, which is an alkali activator. The SS-CPB samples are prepared and cured for 90 days. The reactivity of the S-CPB samples with sulphidic-tailings (45% wt. pyrite) is investigated by performing oxygen consumption (OC) tests, whereas the permeability of the S-CPB is evaluated through saturated hydraulic conductivity tests. The results of the OC tests show that the addition of 0.3% wt. of SS to CPB can reduce its reactivity due to an increase in the formation of cement hydration products in the CPB. Furthermore, the results show that SS reduces the hydraulic conductivity of the S-CPB by enhancing or refining its microstructure. The results obtained suggest that the addition of SS to CPB would enhance its environmental properties or performance.

4.1. Introduction

Mining is a multibillion-dollar industry in many countries around the world. Although mining economically is known as a crucial part in the world, the extraction of ore from an underground mining creates significant and severe problems such as underground voids and waste materials at the ground level for disposal (Hudson-Edwards et al., 2011). Mining industry produces different kinds of waste such as waste rock, mill tailings and water treatment sludge (Benzaazoua et al., 2004). These wastes are generated during the process of extraction, beneficiation and processing of minerals. Once the ore is brought to the surface, disposal of waste rocks becomes a considerable challenge for mining industry because of containing

contaminative metal elements in the large amount of waste. For example, the quantity of mine solid waste produced yearly in Canada is estimated at around 500 million tonnes (Amaratunga & Yaschyshyn, 1999). In addition to this, to extract a small amount of valuable minerals, millions of tons of ore are removed from the ground. Consequently, this huge volume of produced waste in mining is a great threat to human lives and ecosystem due to containing dangerous substances like heavy metals which can create environmental problems as a result of leakage, instability, liquefaction and erosion. On the other hand, ore removal creates very large voids in different shapes which are at the risk of failure during mine operations (Yilmaz & Fall, 2017). Therefore, a safe way to successfully solve or reduce these problems is sending tailings to where they came from which is called backfilling.

Backfilling technology has increased in recent years because it is technically suitable, economically viable, environmentally sustainable, and socially responsible. This technology not only offers an environmentally friendly way of mining, but it also can provide better safety factors for underground mining (Yilmaz & Fall, 2017). Meanwhile, according to economic factors and other goals, such as development of mine or abandoning the mine, backfilling is a cost-effective option. Backfilling is a cost-effective mine-waste disposal by converting tailings to the paste form. Consequently, through filling mined-out voids, the risk of collapsing decreases; the backfill provides ground support to the pillars and walls (Yilmaz & Fall, 2017). It is mainly classified into rock backfilling, hydraulic backfilling and cemented paste backfilling methods (Ghirian, 2016). Cemented Paste Backfill (CPB) has recently become a standard practice for use in many mines around the world. Paste backfill consists of milled tailings, hydraulic binders, which are typically Portland cement and some forms of supplementary cementing materials, and water. Although paste backfilling in some ways is expensive technology, this technology provides a safe mine for operating. The cost of CPB is mostly related to cement consumption (Grice, 1998). Therefore, to meet cost-effective CPB mix designs, it is important to minimize cement consumption by adding different materials while meeting the required strength (Ghirian,

2016). One of the relatively new additives is sodium silicate which is used as an alkali activator in CPB system (Kermani et al., 2015).

Sodium silicate or water glass can be produced by smelting silica with sodium carbonate at 1110 to 1200°C temperature and its formula is as $\text{Na}_2\text{O}\cdot n\text{SiO}_2$. By adding sodium silicate as an alkali activator to binder content and mixing with other mine backfill materials, sodium silicate-CPB (or gel-fill), a new mine backfill material can be created. Using of sodium silicate in mine backfill as an alkali activator is relatively new, so not enough studies have evaluated the environmental performance of sodium silicate-CPB (SS-CPB). Key environmental properties of SS-CPB is reactivity (oxygen consumption) and permeability properties. However, no studies have been carried out on the reactivity and hydraulic conductivity of SS-CPB.

Therefore, the chemical reactivity of SS-CPB should be investigated to understand the ability of SS-CPB to produce acid mine drainage which is consider as an environmental issue. One of the methods to measure and evaluate the reactivity of CPB is oxygen consumption (OC) test. This technique is the most common used method to assess the reactivity of mine waste due to its relatively low cost, its high accuracy and simplicity (Aldhafeeri 2016; Aldhafeeri and Fall, 2017; Aldhafeeri and Fall, 2016; Pokharel, 2008; Ouellet et al., 2003, 2006).

The objectives of the present manuscript are:

- To assess the chemical reactivity of SS-CPB by conducting oxygen consumption tests
- To evaluate the hydraulic conductivity of SS-CPB by performing saturated hydraulic conductivity tests.
- to develop a basic understanding of the effect of sodium on key environmental properties of CPB

4.2. Materials and Experimental Program

4.2.1. Materials

4.2.1.1. Tailings

Typically, tailings represent between 70 and 85% of the weight of the cemented paste backfill. In this study, two types of tailings were used. The first one is synthetic tailings and called silica tailings (ST). ST consist of pure ground silica (99.8% SiO₂, manufactured by U.S. Silica Co.). ST are chemically inert material used for accurately controlling the mineralogical and chemical composition of the tailings which can lead to minimizing the level of uncertainties. Also, ST have a particle-size distribution similar to the average grain size distribution of tailings from nine different mines in eastern Canada. The second one is natural tailings (NT) obtained from a Canadian hard rock mine. The primary physical properties and mineralogical composition of the two tailings are shown in Tables 4.1 and 4.2, respectively. The particle size distribution of ST and NT is shown in Figure 4.1.

Table 4.1. Primary physical properties of used tailings

Tailings	Gs	D ₁₀ (µm)	D ₃₀ (µm)	D ₅₀ (µm)	D ₆₀ (µm)	C _u	C _c
ST	2.7	1.9	9.0	22.5	31.5	16.6	1.3
NT	2.78	1.6	10.6	29.5	37.8		

Table 4.2. Mineralogical composition of used tailings

Tailings/ Mineral (Wt. %)	Quartz	Albite	Dolomite	Calcite	Chlorite	Magnetite	Pyrite	Talc	Magnesite	Pyrrhotite	Spinel	Others	Total
ST	99.8	-	-	-	-	-	-	-	-	-	-	0.2	100
NT	15	32.8	15	4.2	16.1	2.4	1	7	1.8	0.3	1.8	2.6	100

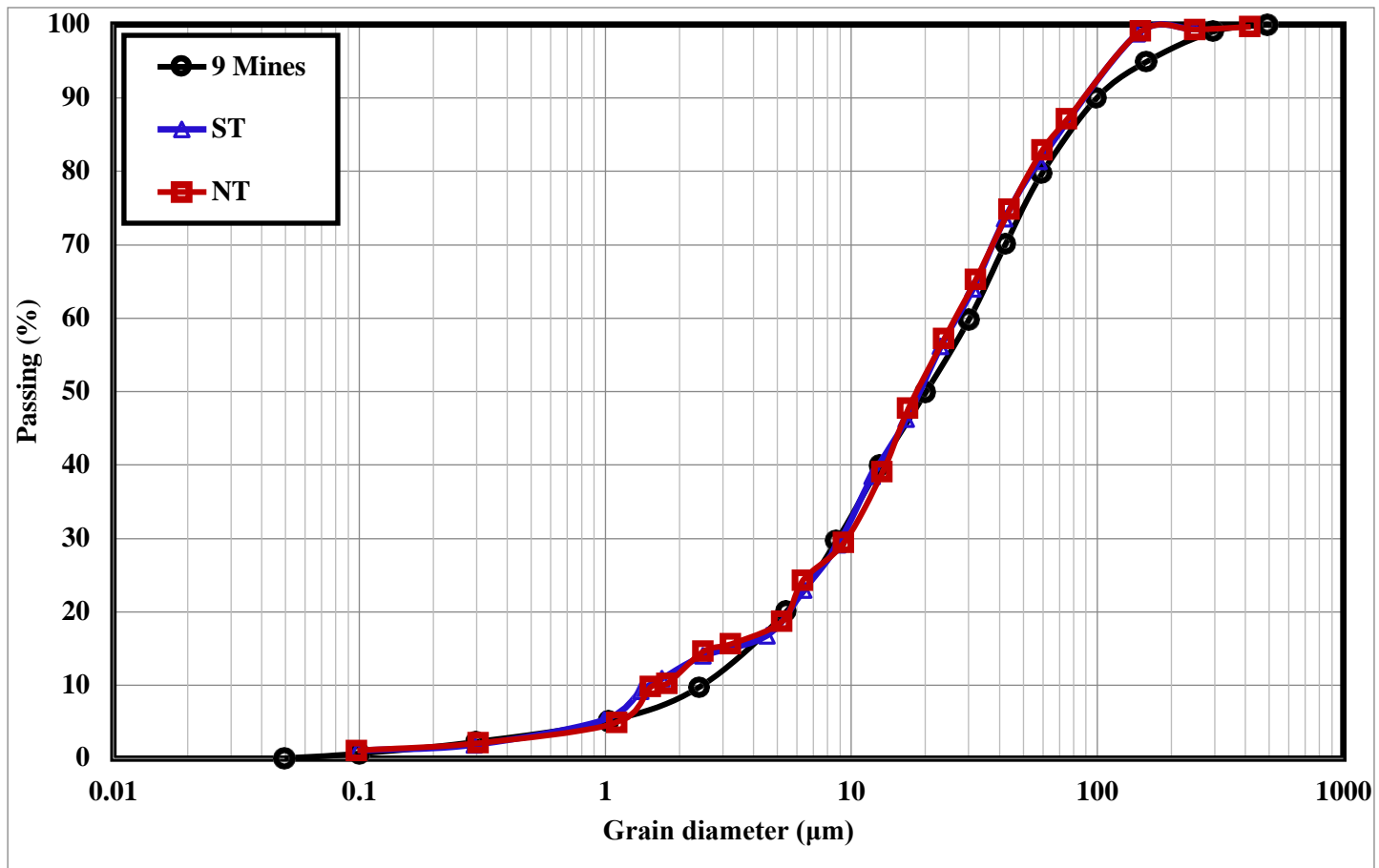


Figure 4.1. Particle size distribution of ST and NT used and average grain size distribution of tailings from nine Canadian mines

4.2.1.2. Binder

Binder is a key parameter for backfill design optimization in order to develop sufficient mechanical strength in the CPB, and it is highly dependent on physical and chemical properties of the binder used. Portland cement type I (PCI) and blast furnace slag (Slag) were employed as binders in this study; they are often used in Canadian CPB operations. Table 4.3 presents the physical and chemical properties of the PCI and Slag used.

Table 4.3. Characteristic of Portland cement type I and Slag

Binder	MgO (wt.%)	CaO (wt.%)	SiO ₂ (wt.%)	Al ₂ O ₃ (wt.%)	Fe ₂ O ₃ (wt.%)	SO ₃ (wt.%)	Rel. Density (%)	Specific surface (m ² /g)
PCI	2.65	62.82	18.03	4.53	2.70	3.82	3.82	1.3
Slag	10.98	41.14	34.23	9.54	-	3.87	3.87	2.2

4.2.1.3. Sodium silicate

Sodium silicate (SS) in reaction act as an alkaline (Hurley & Thornburn, 1972). Sodium silicate type N in the form of liquid is added to the CPB mix. The ratio of N type is between 1.6 and 3.85 for sodium silicate materials so as a result it is the most efficient activator for ground blast furnace slag. Table 4.4 shows the properties of the sodium silicate.

Table 4.4. Sodium silicate properties

(National Silicates Ltd.)

Characteristics	Values
Na ₂ O (wt. %)	8.90
SiO ₂ % (wt. %)	28.66
Weight ratio, %SiO ₂ /%Na ₂ O	3.22

Specific gravity @ 20 °C	1.39
Solids (wt. %)	37.56

4.2.1.4. Mixing water and Pyrite

Tap water was used as mixing water in this study. In addition, to synthesize sulphide mineral-bearing tailings, a commercial pyrite powder (FeS₂; M.W. = 119.98) was used (Table 4.5). This pyrite powder has grains with a size similar to that of pyrite minerals often encountered in the natural tailings of hard rock mines. Pyrite-bearing tailings with a pyrite content of 45% wt. were prepared by mixing ST with the appropriate amounts of pyrite powder.

Table 4.5. Physical properties of pyrite (Source: Washington Mills North Grafton, Inc.)

Bulk density (g/cm ³)	Density at 20°C (g/cm ³)	Specific gravity	pH	Melting point (°C)
2.35	4.7	4.6	4.0- 6.0	~ 1193

4.2.2. Sample preparation and mix proportions

4.2.2.1. Preparation of CPB samples

To test hydraulic and chemical properties of CPB with sodium silicate, around (55) CPB specimens were prepared by mixing the two different types of tailing with the binder (PCI/slag in different blending ratio) and sodium silicate. The ingredients of the CPB mixture were blended by using a B20F food mixer to gain homogenous samples. The mixing time was 10 min. After the CPB mixtures were produced, cylindrical and polyvinyl moulds, 10 cm deep and 5 cm in diameter, were used to pour the fresh CPB samples in. The prepared samples were sealed and then cured at room temperature. The curing times for the specimens were 90 days. Tables 4.6 and 4.7 show the mix compositions of the backfill specimens prepared for the hydraulic conductivity tests and measurement of reactivity, respectively, while Table 4.8 and 4.9 display the samples prepared for the microstructural analyses.

Table 4.6. Prepared samples to study the permeability of CPB that contains sodium silicate

CPB Sample	Binder	Binder content (wt%)	Binder ratio (PC/Slag)	Tailings Type	Sodium Silicate (wt%)	w/c ratio	Curing times (day)
ST-Slag-CPB-0.3SS-P	PCI/Slag	4.5	50/50	ST	0.3%	7.6	90
NT-Slag-CPB-0.3SS-P	PCI/Slag	4.5	50/50	NT	0.3%	7.6	90
ST-Slag-CPB-0.0SS-P	PCI/Slag	4.5	50/50	ST	0.0%	7.6	90
NT-Slag-CPB-0.0SS-P	PCI/Slag	4.5	50/50	NT	0.0%	7.6	90
ST-Slag-CPB-SS-25-P	PCI/Slag	4.5	25/75	ST	0.3%	7.6	90
ST-Slag-CPB-SS-75-P	PCI/Slag	4.5	75/25	ST	0.3%	7.6	90
ST-Slag-CPB-SS-100-P	PCI/Slag	4.5	100/0	ST	0.3%	7.6	90

Table 4.7. Preparation of CPB samples to study the reactivity of CPB that contains sodium silicate

CPB Sample	Binder	Binder content (wt%)	Binder ratio (PC/Slag)	Tailing Type	Sodium Silicate (wt%)	w/c ratio	Pyrite	Curing times (day)
ST-Slag-CPB-0.3SS-R	PCI/Slag	4.5	50/50	ST	0.3%	7.6	45	90
NT-Slag-CPB-0.3SS-R	PCI/Slag	4.5	50/50	NT	0.3%	7.6	45	90
ST-Slag-CPB-0.0SS-R	PCI/Slag	4.5	50/50	ST	0.0%	7.6	45	90
NT-Slag-CPB-0.0SS-R	PCI/Slag	4.5	50/50	NT	0.0%	7.6	45	90
ST-Slag-CPB-SS-25-R	PCI/Slag	4.5	25/75	ST	0.3%	7.6	45	90
ST-Slag-CPB-SS-75-R	PCI/Slag	4.5	75/25	ST	0.3%	7.6	45	90
ST-Slag-CPB-SS-100-R	PCI/Slag	4.5	100/0	ST	0.3%	7.6	45	90

Table 4.8. Prepared cement pastes (CP) of samples to study the microstructure (TG/DTG; XRD)

CPB Sample	Binder	Binder content (wt%)	Binder ratio (PC/Slag)	Tailings Type	Sodium Silicate (wt%)	w/c ratio	Curing times (day)
ST-Slag-CPB-0.3SS-P	PCI/Slag	4.5	50/50	ST	0.3%	1	90
ST-Slag-CPB-0.0SS-P	PCI/Slag	4.5	50/50	ST	0.0%	1	90

Table 4.9. Prepared CPB samples to study the MIP-pore structure of CPB that contains sodium silicate

CPB Sample	Binder	Binder content (wt%)	Binder ratio (PC/Slag)	Tailings Type	Sodium Silicate (wt%)	w/c ratio	Curing times (day)
ST-Slag-CPB-0.3SS-P	PCI/Slag	4.5	50/50	ST	0.3%	7.6	90
ST-Slag-CPB-0.0SS-P	PCI/Slag	4.5	50/50	ST	0.0%	7.6	90

4.2.3. Testing of specimens

4.2.3.1. Reactivity: oxygen consumption test

OC test shows the rate of oxygen consumption through the oxidation of sulphide bearing material. Therefore, to evaluate the effect of sodium silicate on the oxidation rate of CPBs, oxygen consumption (OC) tests were performed. The OC test consists of measuring the quantity of consumed oxygen in a sealed air chamber for a short period of time about 3 to 5 h (Schmieder et al 2012). It is assumed that the steady state condition will be maintained from the beginning to the end of the test. By using the galvanic cell (model GC33-200, GC Industries) as an oxygen sensor with the accuracy of 0.1% O₂, the reactivity of SS-CPB is measured. The voltage produced, the oxygen sensor connects to a voltmeter (Hantek 365A USB Data Logger Recorder Digital Multimeter Voltage Current PC Base) and measures the oxygen reaction that happens in the sealed chamber, Fig 4.2. The recorded voltage is exactly the partial pressure of oxygen in the gas phase. As can be seen in the Fig.4.2, the cylinders are covered with an airtight lid which was equipped with an oxygen sensor (Aldhafeeri, 2018).

To evaluate the reactivity of SS-CPB specimens, oxygen flux (F_L) under pseudo-steady state condition is used (Elberling & Nicholson, 1996). F_L is determined based on the two following equations:

$$F_L = C_o (K_r D_e)^{1/2} \quad (1)$$

where K_r is the first-order reaction rate coefficient, D_e is the effective diffusion coefficient and C_o is the initial (atmospheric) concentration of oxygen.

$$\ln (C/C_o) = (K_r D_e)^{1/2} (A/V) \quad (2)$$

where A and V are the area and the volume of the source reservoir, respectively.

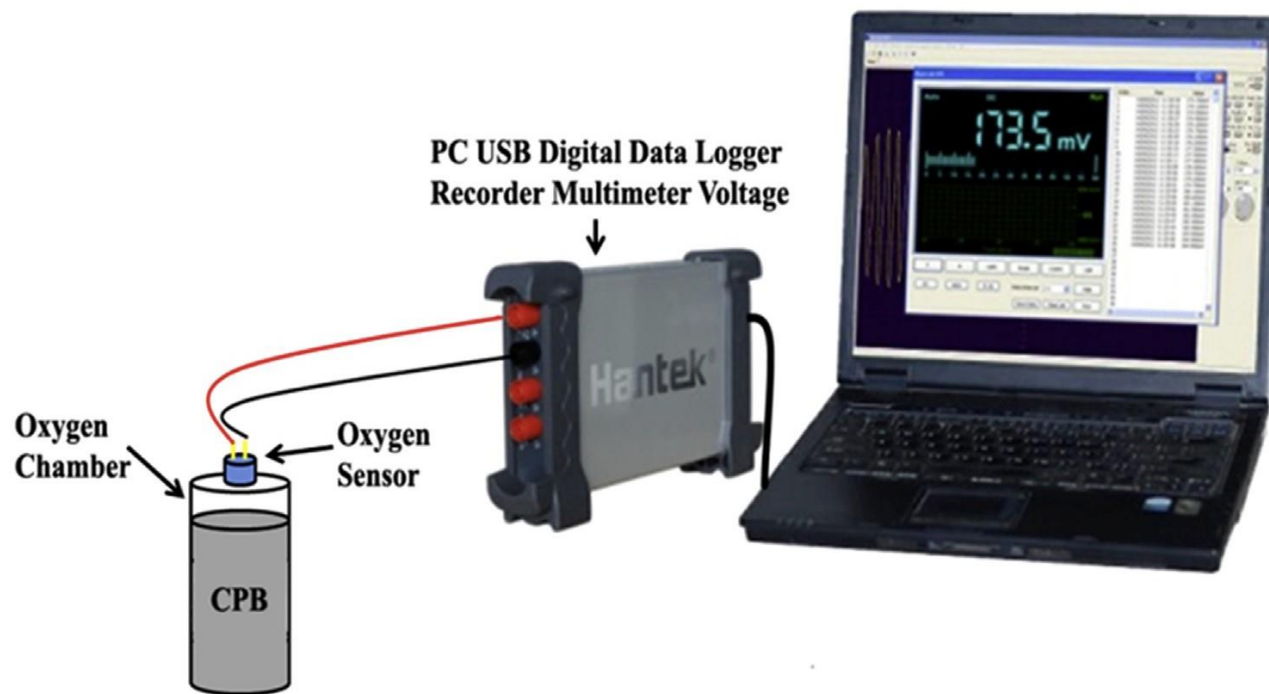


Figure 4.2. Schematic of experimental setup of OC testing (Aldhafeeri, 2018)

4.2.3.2. Hydraulic conductivity test

In this study, hydraulic conductivity study is required to assess the fluid transportability of backfill which affects the environmental performance of CPB containing sodium silicate. In addition, the knowledge of the hydraulic conductivity is necessary to determine the leakage potential of contaminants from the backfill into the groundwater (Levens et al., 1996). To experimentally clarify the impact of sodium silicate on hydraulic conductivity of SS-CPB, saturated hydraulic conductivity tests are carried out according to ASTM D5084-00. The hydraulic conductivity was determined using a flexible wall permeameter with a constant head. Based on this method, the pressure difference between the inflow and outflow through the test specimen was kept at 69 kPa (10 Psi) to achieve a desired hydraulic gradient. The samples were saturated in water for 24 hours prior to the testing. The specimen was fully saturated by bridging the inflow and outflow burettes and

then applying backpressure until the flow through both is constant. After this stage where full saturation was achieved, the CPB samples were subjected to permeability tests. Each hydraulic conductivity test was performed twice, and the average of obtained values was taken as the saturated hydraulic conductivity (k_{sat}) of the sample tested.

4.2.3.3. Microstructural analysis

Three different microstructural testing/analysis techniques were used to experimentally investigate the effect of sodium silicate on the pore structure and evolution of binder hydration products of CPB. Before testing, all the samples were dried at 45°C up to 4 days to remove the capillary water (dry samples). Mercury Intrusion Porosimetry (MIP) tests were conducted on SS-CPB samples to assess their pore structure or pore size distribution. The MIP tests were carried out by using a Micromeritics Auto Pore III 9420 mercury porosimeter in accordance with ASTM D4404-10.

Thermal Gravimetric (TG) and Differential Thermal Gravimetric (DTG) analyses were done on powdered and dried specimens of cement pastes of SS-CPB by using a thermogravimetric/differential thermal analyzer (TGD-9600, ULVAC) to assess the effect of sodium silicate on the cement hydration products that develop in the backfill samples. The tests were done on cement paste samples of CPB with a water/binder ratio of 1. The main reason of choosing this ratio is to simulate the high-water content of CPB. The last microstructural analysis is x-ray diffraction (XRD). This technique was conducted to examine the impact of sodium silicate on the mineralogical composition of the hydration products of CPB samples. The XRD was conducted by using a Scintag XDS 2000 x-ray diffractometer on dried and grounded samples of cement pastes of CPB. In addition, by using a Scintag XDS 2000 XRD, the XRD patterns of the prepared samples can be obtained.

4.3. Results and discussion

4.3.1. Permeability of Slag-cemented paste backfill containing sodium silicate

The effect of sodium silicate on the saturated hydraulic conductivity (k_{sat}) of CPB with different type of tailings (NT or ST) is depicted in Fig 4.3. The CPB samples were prepared with a total binder content of 4.5% and cured at room temperature for period of 90 days, Table 4.6. From Fig 4.3, it can be observed that, regardless of the type of tailing (NT or ST), the sodium silicate affects the hydraulic conductivity of CPBs. As expected, by adding sodium silicate, the permeability decreases. The reason for this behavior is the increase in the amount of cement hydration products (e.g., CSH, CH, ettringite) due to the sodium silicate induced activation of slag, which results in enhancing refinement of the pore structure and reduction of the porosity of the CPB materials. Simply put, decreasing the porosity leads to filling the pores in the cemented matrix of the CPBs with the aforementioned hydration products and blocking the interconnected pores. This result is in perfect agreement with the results of the MIP test (Fig 4.9) performed on the SS-CPB samples and CPB free sodium silicate samples with the same binder content (4.5%) and w/c ratio of 7.6. It can be seen that the pore size distribution of the sodium silicate free CPB samples is coarser than that of the specimen with sodium silicate. The aforementioned increase in binder hydration products because of the activation of the Slag hydration by sodium silicate is experimentally supported by the results of XRD and DT/DTG analyses presented in Figures 4.5 and 4.6, respectively. The XRD result in Figure 4.5 shows the presence of a higher quantity of hydration products in the sample with SS. It is observed that the intensity of CH at 18° 2-theta is 1399 CPS in the case of the SS-sample whereas for CPB without SS, this intensity is approximately 398 CPS. In the same way, the intensity of CH at 34° , 47° , and 51° 2-theta for the sample with SS is a higher than that without SS.

This higher amount of binder hydration products (e.g., C–S–H, CH) formed within the SS-CPB samples is also demonstrated by the results of thermal analyses performed on cement pastes specimens of CPB with and without SS (Figure 4.6). It can be observed that weight losses and endothermic peaks in the 50– 150 °C and 400-450 °C temperature ranges are higher for the specimen with SS than for the sample without SS. This indicates that higher amounts of CSH, ettringite and CH formed with SS-CPB.

Moreover, Figure 4.3 indicates that the permeability values of CPB specimens made of NT are higher than those with ST. This can be attributed to the sulphate attack on CPB caused by sulphate ions in NT (~7000 ppm), which in turn, results in the formation of more porous cemented matrix due to the physical damage of the matrix of the CPB by the excessive pressure generated by expansive minerals (ettringite, gypsum) formed in the CPB (Fall and Benzaazoua, 2005). It is known that sulphate attack has damaging effects on the quality (e.g., strength deterioration) of cement-based products (concrete, mortars, cemented paste backfill, etc.) (Fall and Benzaazou, 2005). Sulphate attack on cementitious materials leads to the formation of expansive minerals such as ettringite, gypsum and other phases. Sulfate attacks can not only decrease strength, i.e., the mechanical stability of CPB structures, but they can also contribute to the development of cracks in the CPBs, and thus increase of the permeability of CPB (Pokharel and Fall, 2010).

Figure 4.4 shows the effect of different binder proportion on the permeability of CPB samples with sodium silicate. From this figure, it appears that the proportion of PCI has a large effect on the hydraulic conductivity of SS-CPBs. It can be noted that the permeability of the SS-CPB decreases as the Slag content increases (Taylor, 1990; Fall et al., 2008). This lower permeability associated with Slag concentration can be explained by the fact that by increasing Portland cement in the binder content, more precipitation of hydration products may occur. Therefore, refinement in the pore structure leads to the decrease of SS-CPB porosity.

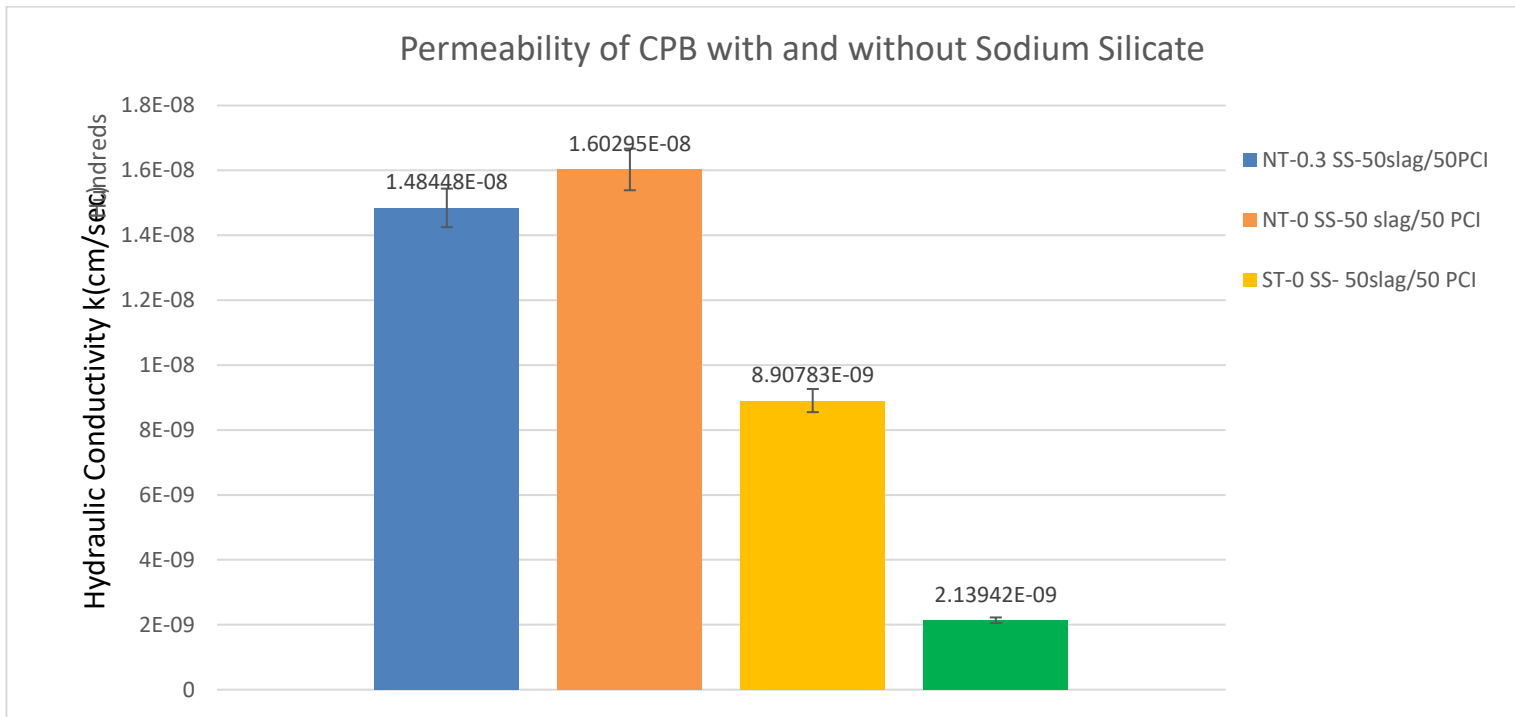


Figure 4. 3. Effect of sodium silicate on the saturated hydraulic conductivity of CPB

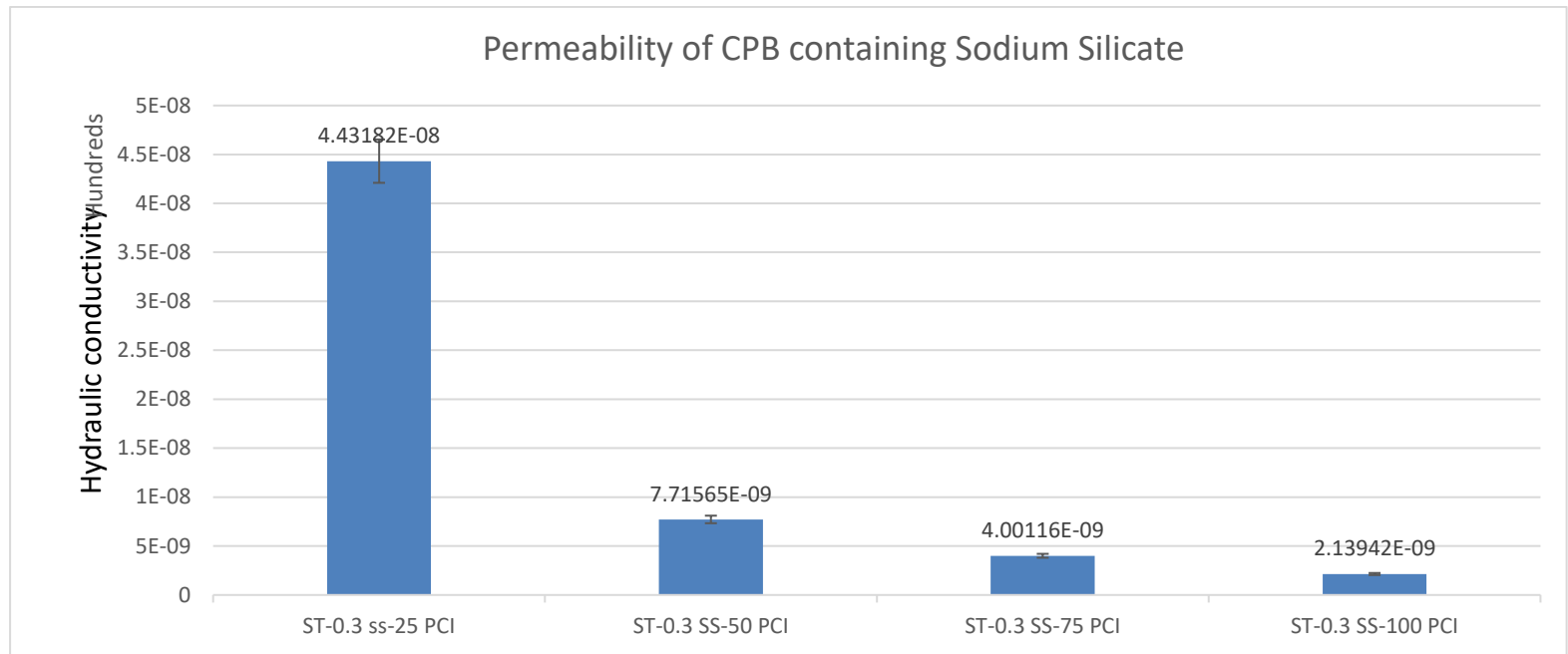
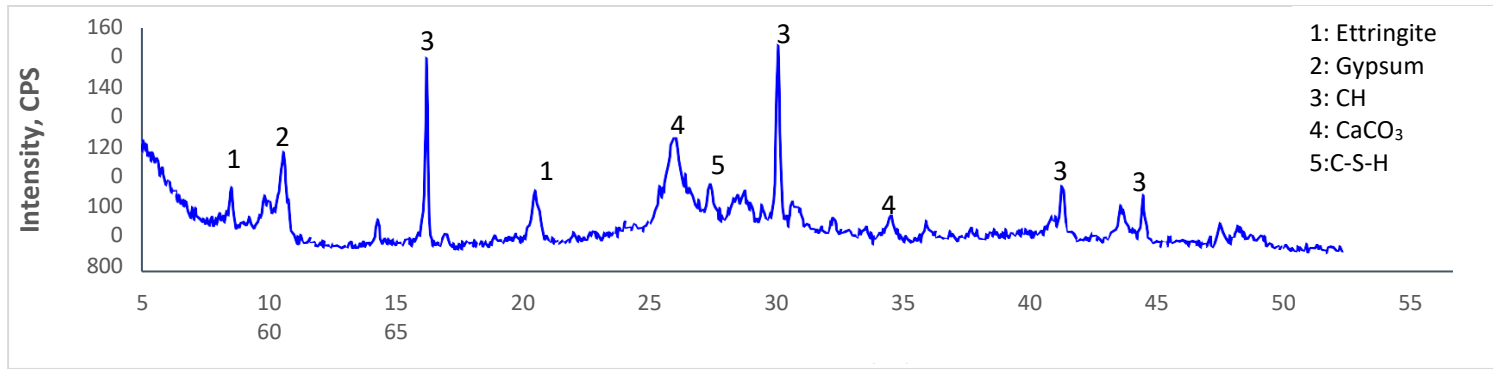
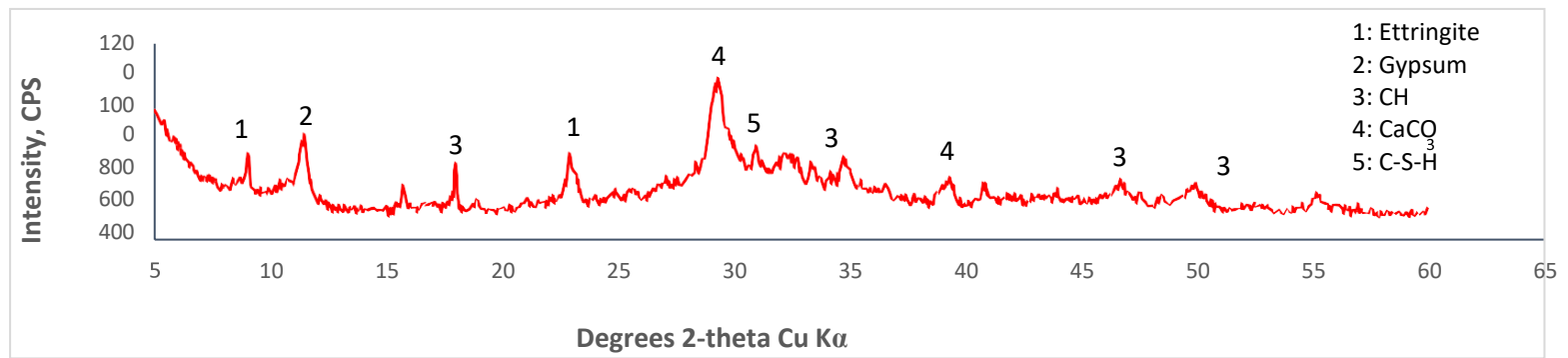


Figure 4.4: Effect of sodium silicate on the saturated hydraulic conductivity of CPB made of PCI/Slag with different blending ratios (25/75, 50/50, 75/25, 100/0)



a-CPB sample with 0.3%SS and PCI/Slag (50/50)



b-CPB sample without SS and PCI/Slag (25/75)

Figure 4.5. XRD plots of cured CPB samples, (a) CPB sample with 0.3% of SS and with PCI/Slag (50/50)
(b) CPB sample without SS

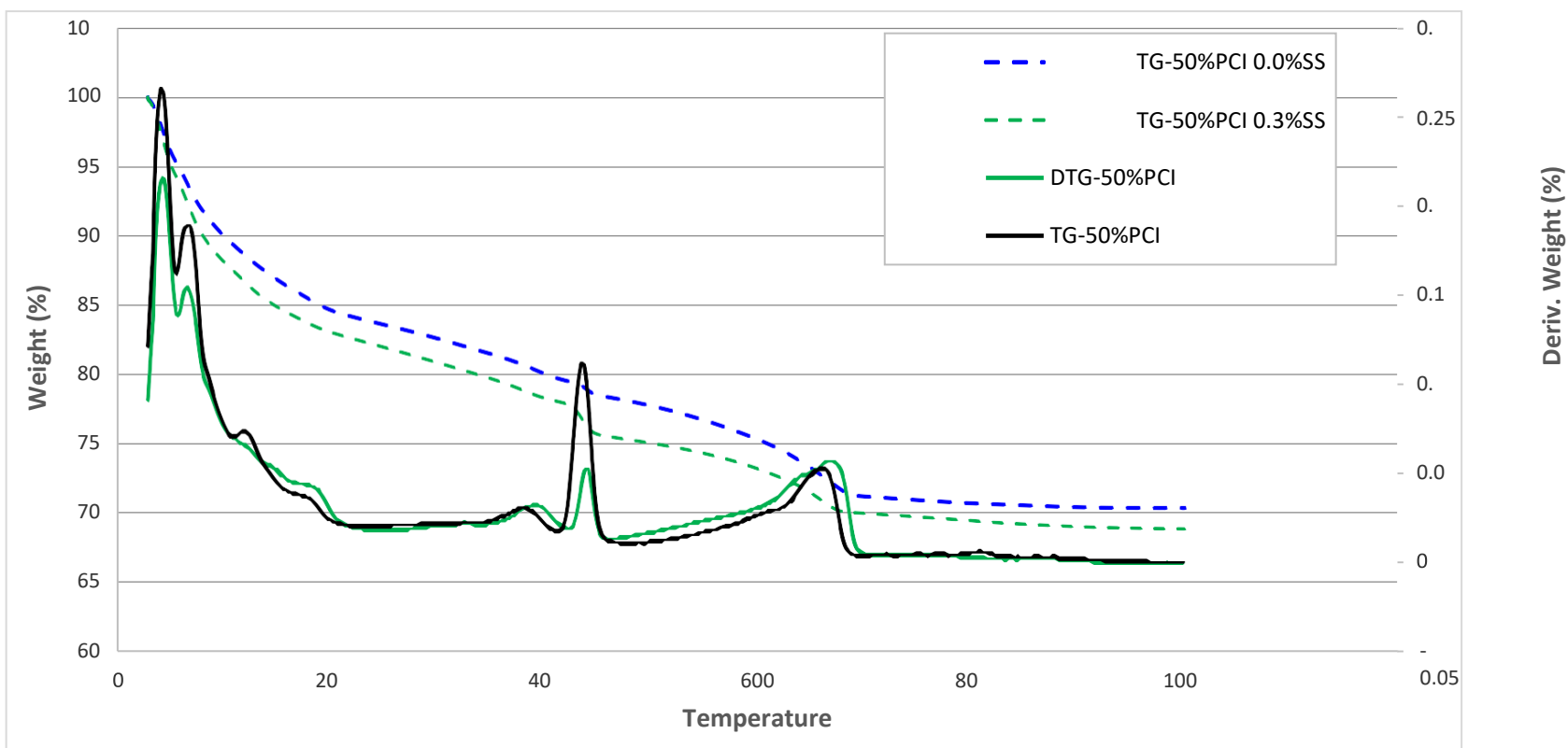


Figure 4.6. TG and DTG diagrams for cemented paste samples containing sodium silicate and sodium silicate-free CPB samples

4.3.2. Reactivity of Slag-cemented paste backfill that contains sodium silicate

Figure 4.7 shows the influence of sodium silicate on the reactivity of CPB specimens (with pyrite content of 45%) cured at 90 days. The results presented in Fig 4.7 show that in general, the reactivity of the CPB specimens decrease with adding sodium silicate regardless of the type of tailings. Indeed, the lower oxidation rate measured in the SS-CPB samples is attributed to the fact that sodium silicate accelerates binder hydration, which results in precipitation of higher amount of hydration products, thereby decreasing the pore structure (Figure 4.9). For that reason, the reactivity of the CPB specimens will decrease due to the reduction in the effective diffusion coefficient of oxygen (D_e) (see Eqs. (1) and (2)), which in turn decreases

the oxidation rate of pyrite. This reduction in reactivity is fully supported by MIP results of CPB samples with and without sodium silicate.

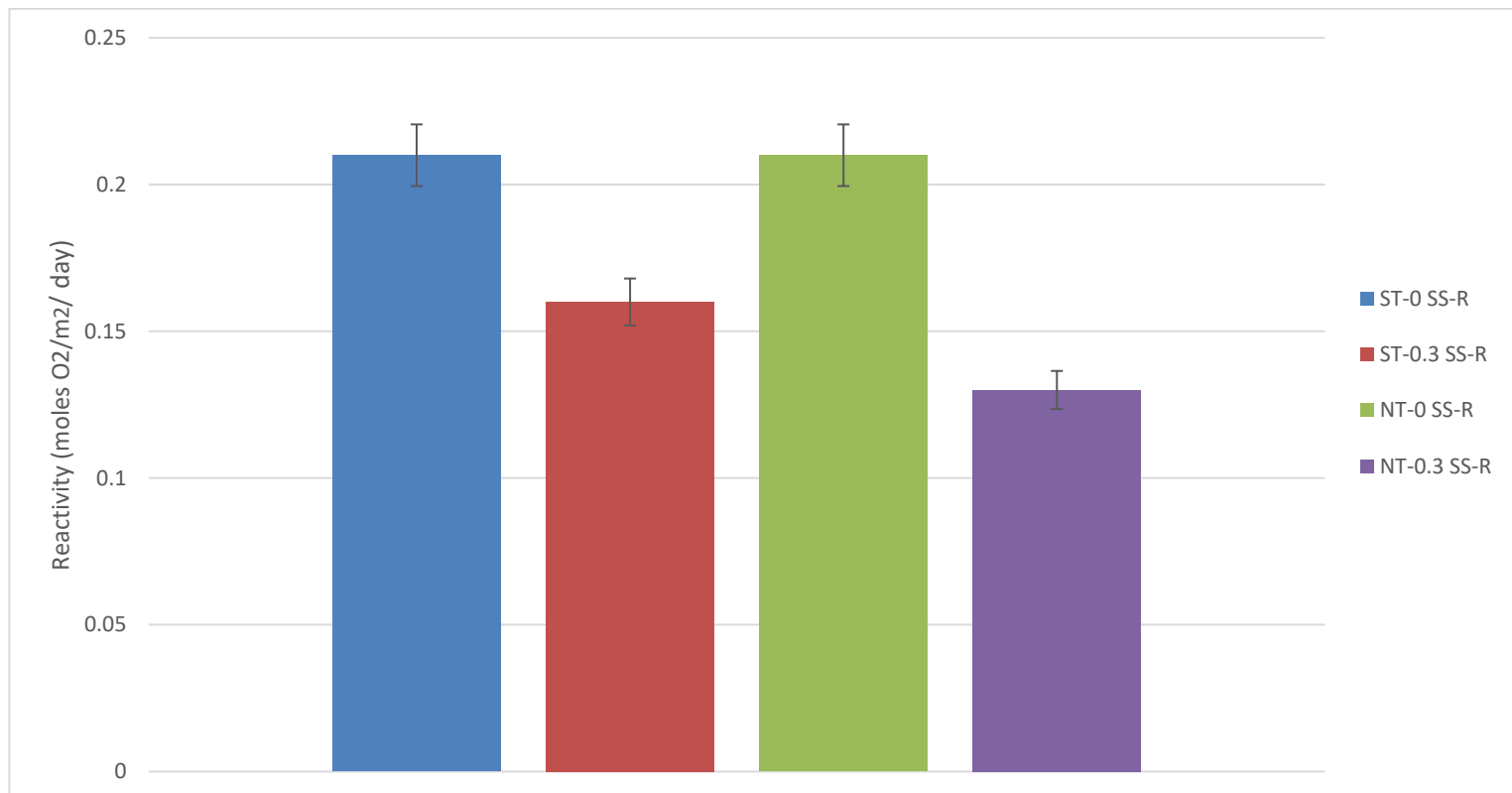


Figure 4.7. Effect of sodium silicate on the reactivity of CPB made with different tailing type
Note: Each value is the average of two samples.

Furthermore, Figure 4.8 clearly shows that adding sodium silicate in CPB specimens with different binder ratio can have a remarkable effect on the reactivity of CPB samples. As mentioned in the previous chapters, the addition of any alkali activator leads to increasing hydration process. Existing alkali activator in CPB specimens increase cement hydration and thereby more hydration products are generated which fill the pores in the CPB-SS specimens. By filling the CPB pores, oxygen consumption decreases and then the reactivity gets less. The result revealed that the CPB containing 25% PCI and 75% slag is the most reactive

(0.18 mol/m²/day), followed by the CPB containing 50% PCI and 50% slag (0.165 mol/m²/day), and then the CPB containing 75% PCI and 25% slag. However, the reactivity of the CPB containing 100% PCI is slightly higher than that containing 75%. This is an indication that the combination of slag and PCI is beneficial in achieving a less reactive backfill material.

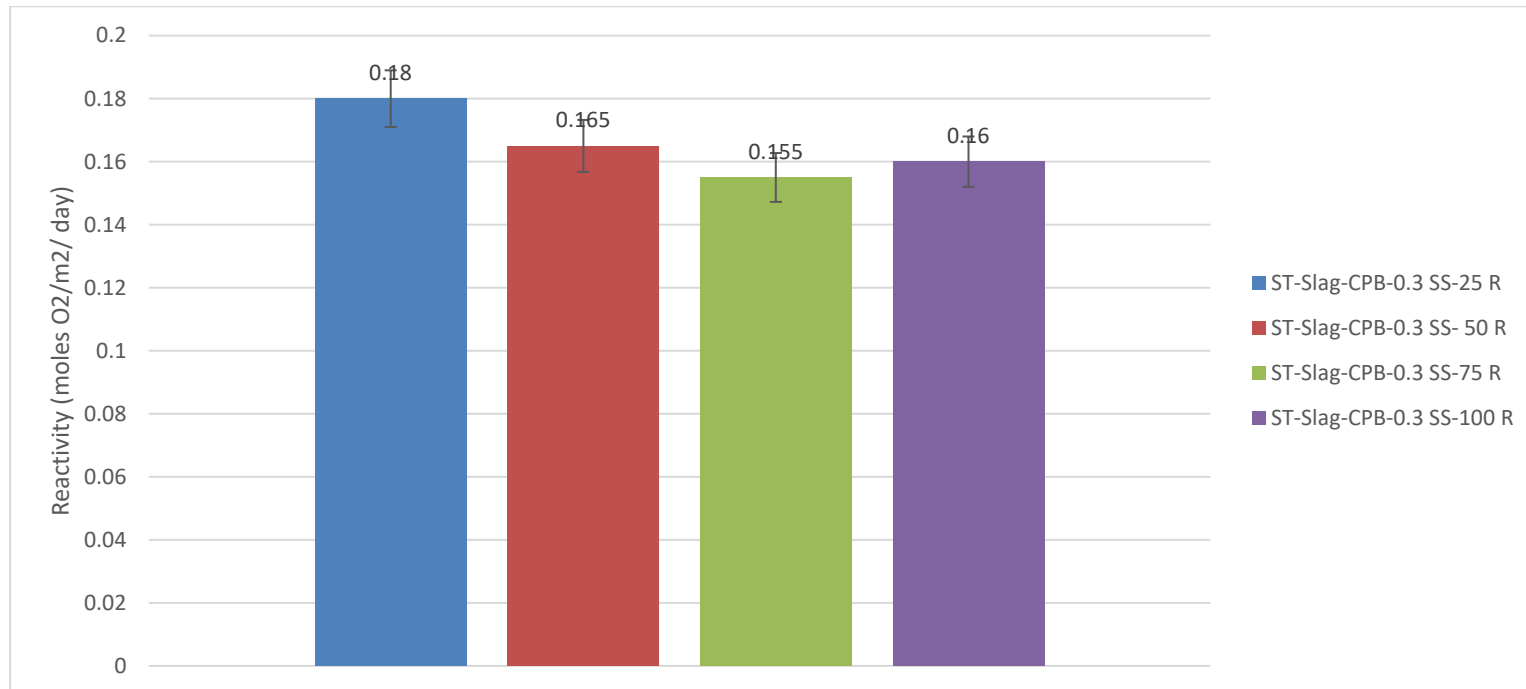


Figure 4.8: Reactivity of CPB-SS with different binder ratio

4.3.3. MIP microstructural analysis of CPB specimens containing sodium silicate

Figure 4.9 depicts the results of MIP tests performed on 90 days CPB samples made of PCI/Slag (50/50) with and without sodium silicate. From this figure, it appears that CPBs without sodium silicate have a coarse pore structure. The CPB-SS has a finer pore size distribution than that without the SS because of the Slag activation by SS. This finer pore structure is obviously associated with a decrease of permeability and reactivity of SS-CPBs. In other words, their environmental performance would increase.

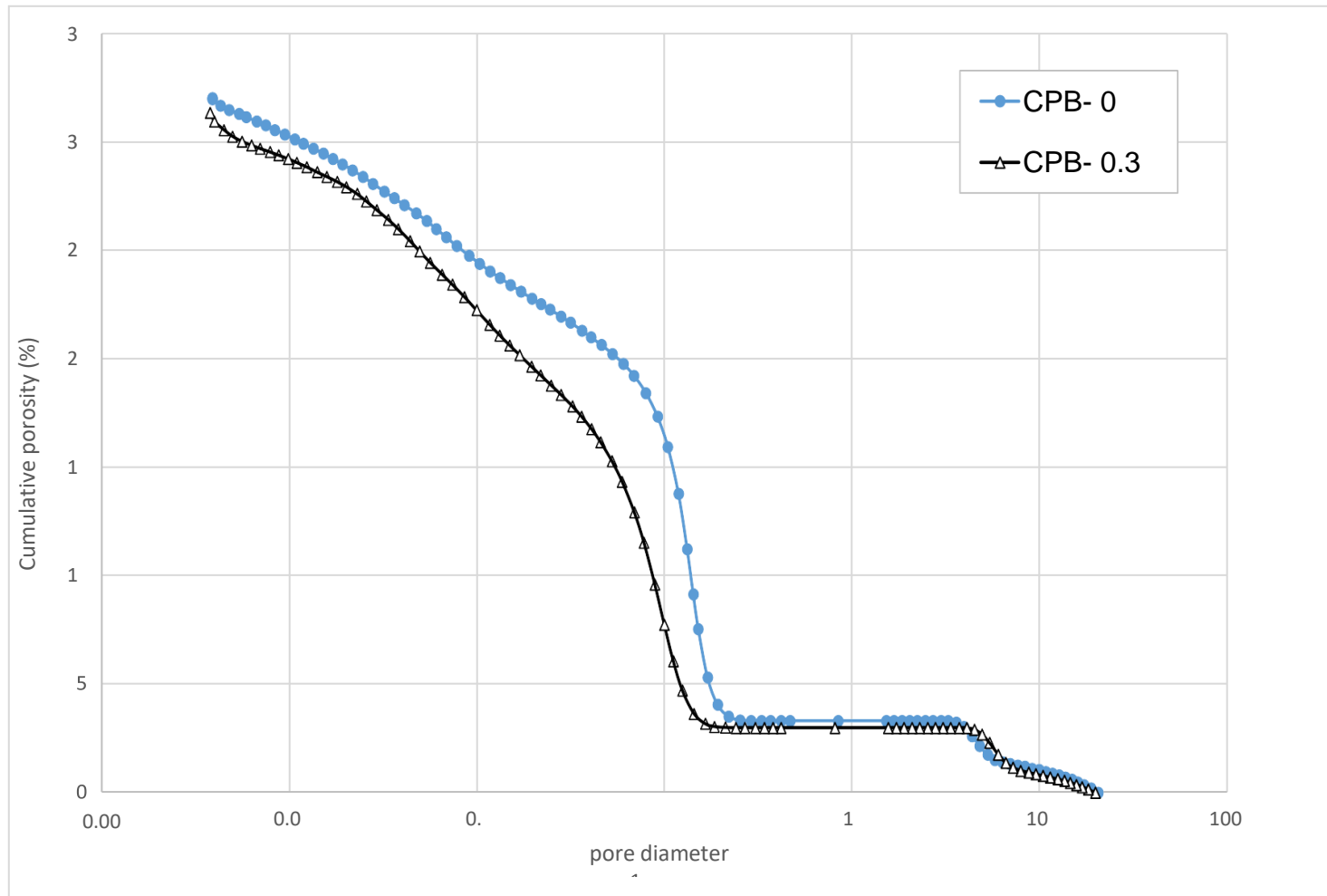


Figure 4.9. Trend of pore diameter with cumulative porosity in the presence and absence of sodium silicate

Figure 4.10 shows the MIP pore distribution of the SS-CPBs made of PCI/Slag (50/50) and PCI/Slag (75/25). It is shown in this figure that the pore structure of the SS-CPB with only 25%PCI is slightly coarser than that with 50% PCI. This observation is consistent with the reactivity and permeability results discussed previously.

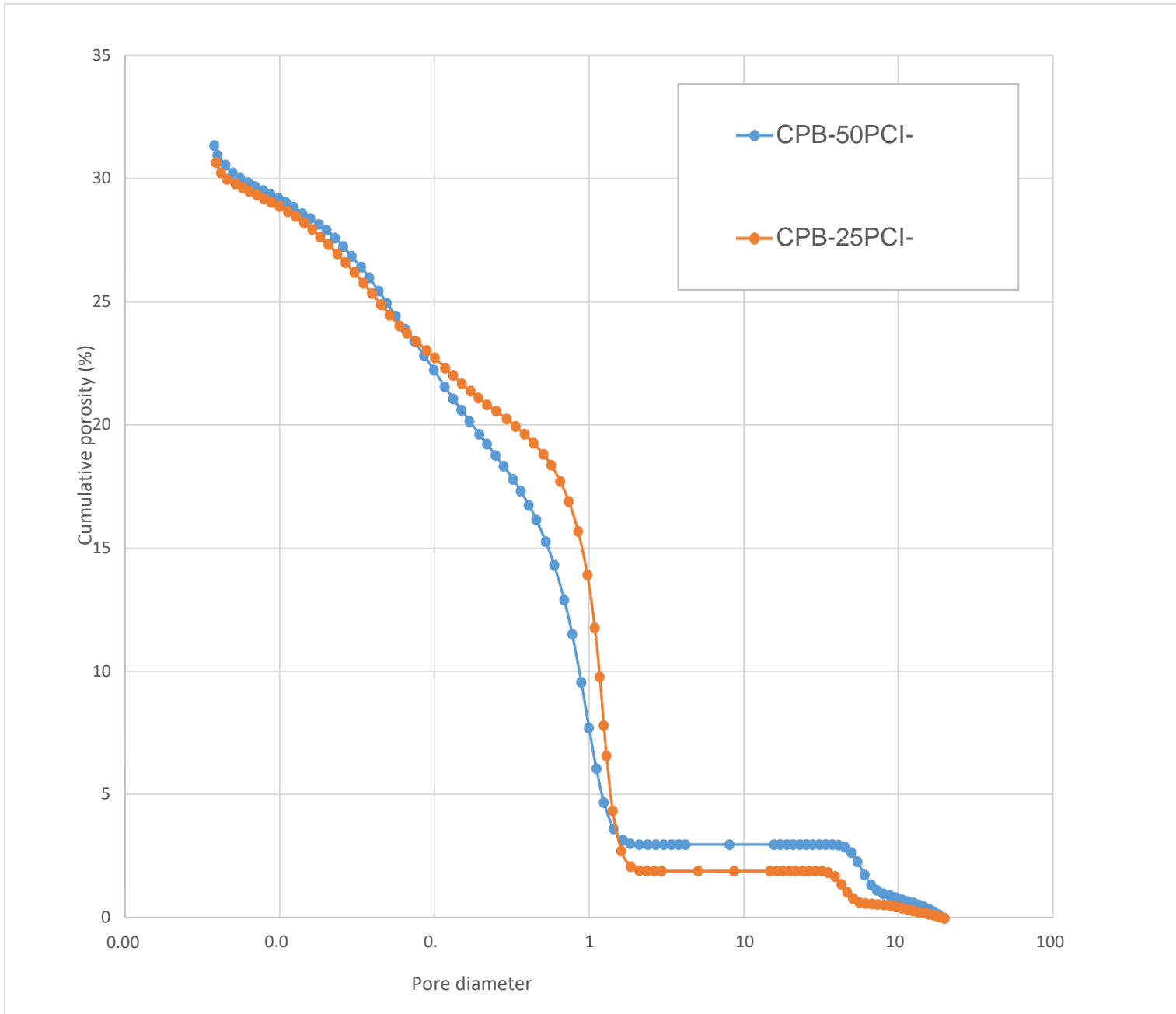


Figure 4.10: Trend of pore diameter with cumulative porosity in the CPBs containing SS and different PCI/Slag ratios (50/50; 75/25) ratio

4.4. Summary and Conclusions

This study has presented results of the effect of sodium silicate on the saturated hydraulic conductivity and reactivity of CPB. Hydraulic conductivity and OC tests were conducted to determine the permeability and reactivity of the backfill material. Valuable results have been obtained.

- It is found that the addition of sodium silicate to CPB reduces its coefficient of permeability. This is due to the fact that the sodium silicate activates the hydration of Slag, thereby leading to a production of larger amount hydration products, which, in turn, decreases the porosity and fluid transportability of CPB. This precipitation of more hydration products and refinement of the pore structure of CPB have been confirmed by the results of DG/DTG analyses and MIP tests, respectively. This sodium silicate induced-decrease of the permeability of CPB would enhance the environmental performance of CPB. Indeed, one of the most important parameters affecting environmental performance and durability of CPBs is permeability. Susceptibility to acid mine and ability to release contaminants into the mine areas and/or groundwater (after mine flooding) are relevant environmental design criteria for CPB structures. Permeability is one of the key factors controlling the groundwater flow rate through the CPB mass once it is flooded. In other words, it significantly impacts the leaching potential and transport of contaminants through the backfill mass to groundwater.
- The OC test results revealed that the addition of sodium silicate to CPB reduces its coefficient of reactivity due to changes in its microstructure (induced by the activation of Slag hydration by sodium silicate). This decrease in reactivity means that CPB with sodium silicate would be less susceptible to develop acid mine drainage. It is also found that the combination of slag and PCI is beneficial in achieving a less reactive backfill material. However, the optimal PCI/Slag blending ratio should be defined and selected for each CPB system.

4.5. References

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Chapter 5- Conclusion and recommendations

Conclusion

The main goal of this manuscript was to investigate the behavior of cemented paste backfill with the presence of sodium silicate to a better assessment of strength and environmental properties of CPB. Therefore, UCS test is performed to assess the effect of sodium silicate on the strength development of SS-CPB samples. Meanwhile, microstructural analyses provide technical support about the influence of sodium silicate on the microstructural evolution of SS-CPB. In addition, saturated hydraulic conductivity test is performed and presented for better understanding of the fluid transportability of the CPB samples with sodium silicate. Finally, by conducting OC tests on CPB samples that contain sodium silicate, useful information is provided to understand the reactivity process of SS-CPB.

The outcomes of these tests are drawn as below:

- 1- The mechanical strength of CPB increases by using alkali activator such as sodium silicate. Sodium silicate can also significantly increase the strength gain rate of Slag-CPB and its early age strength. This is attributed to the fact that using sodium silicate in the cemented paste backfill activates the slag. This activation results in the formation of more hydration products and the refinement of the pore structure of CPB. The results obtained have also shown that the blending ratio of PCI and Slag has a significant influence on the effect of sodium silicate on the strength development of CPB. These results suggest that the use of sodium silicate can enable to open the barricades earlier which can lead to significant profits for the mine.
- 2- The hydraulic property of CPBs is affected by the addition of sodium silicate in the CPB samples. Hydraulic conductivity results show a positive change in samples containing sodium silicate compared to free sodium silicate CPB samples. The activation of Slag by sodium silicate leads to the decrease of permeability due to the formation of cement hydration products and acceleration of the binder hydration process. Moreover, binder type and content in the presence of sodium silicate as an alkali activator in the CPB play a significant

role in lowering hydraulic conductivity of CPB. This sodium silicate induced-decrease in hydraulic conductivity enhances the environmental performance of CPB.

3- In terms of reactivity, this study has shown that the reactivity of CPB is influenced by sodium silicate due to changes in its microstructure. The results indicate that the addition of sodium silicate to CPB decreases the coefficient of reactivity of CPB due to changes in its microstructure. This means that CPB with sodium silicate would be less susceptible to develop acid mine drainage. It is also established that the combination of slag and PCI is beneficial in achieving a less reactive backfill material. However, the optimal PCI/Slag blending ratio should be defined and selected for each CPB system.

The lower oxidation rate measured in SS-CPB is attributed to the fact that mixing sodium silicate in the CPB creates a thin oxidized layer having a less porosity in the surface due to precipitation of the secondary minerals. This microstructural test showed that pyrite reactivity is decreased by mixing sodium silicate with CPB which is resulting in the formation of more hydration products, low porosity, and low oxygen consumption. Therefore, combination of these factors reduces further oxygen consumption by the SS-CPB.

This study provides essential information for understanding the evolution mechanical, microstructural and hydraulic properties as well as reactivity of CPB with sodium silicate. The findings of this thesis will help mining operators and engineers who work with SS-CPB materials to take into consideration, the geotechnical and environmental aspects of SS-CPB structures.

Recommendations

Based on the observations from this study, the following recommendations are suggested.

1- In the future, more studies need to be carried on a field scale on the CPB containing sodium silicate to assess the environmental behaviour of SS-CPB in situ condition.

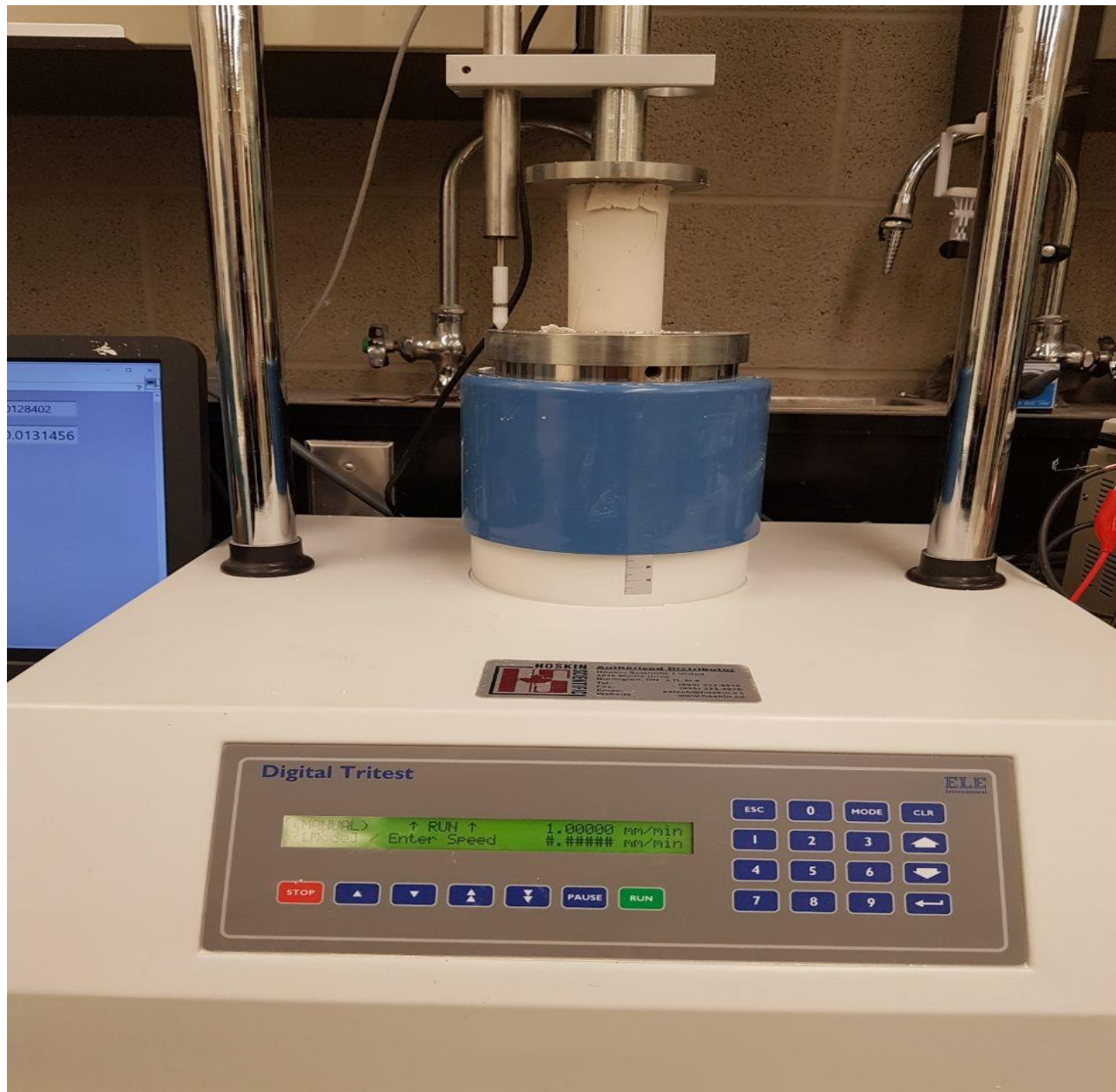
2- Further studies need to be done on the reactivity of SS-CPB at longer curing time to understand the long-term effect of sodium silicate.

3- Several other microstructural tests should be introduced to investigate the effect of sodium silicate on the pore structure of CPB in more detail. Alkali activator is considered as a significant advantage for activating cement hydration. Therefore, more research can be done to investigate new and similar activators.

4- Combined thermal, hydraulic, mechanical, and chemical (THMC) processes in the CPB containing SS should be studied to have a better understanding of its effects.

5- The obtained results can be used to validate a mathematical model in better understanding of THMC behavior of CPB.

Appendix Photos from the study



UCS test



OC test



Mixture