

Strength and Deformation Behaviour of Cemented Paste Backfill in Sub-Zero Environment

by

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Abstract

Underground mining produces a huge amount of voids and an even larger quantity of mine waste. Overlooking these voids could lead to the possibility of ground subsidence, as well as safety issues during mining operation; while ignoring the waste, could cause environmental pollution and significant suffering. One solution to remedy both (the voids and the waste) is cemented paste backfill (CPB), which is gaining increased recognition in both the mining industry and academic research. Transforming tailings into cemented paste, and transporting this back to underground stopes, not only negates these safety issues to a large degree, but also makes it possible to put waste to good use.

However, most studies involving CPB have been conducted at temperatures above 0°C; knowledge of CPB in sub-zero environments is still lacking. For this reason, this thesis investigates the mechanical behaviour of CPB in a the latter type of environment.

Uniaxial compressive strength tests were carried out on a series of frozen CPB (FCPB) samples to evaluate the mechanical behaviour (e.g. compressive strengths, geotechnical features, and the stress-strain relationships) of FCPB. It has been discovered in this thesis that FCPB exhibits remarkable strength compared to CPB and, has a great resemblance to frozen soil. Factors which may affect the behaviour of FCPB were thoroughly examined. Binder contents and types were found to be irrelevant; water content, in contrast, plays a dominant role, with an optimum value of around 26% by weight. Sulphate was confirmed to have an adverse effect on the strength of FCPB due to the increasing unfrozen water content and the formation of legible ice lenses. Hydraulic conductivity tests, scanning electron microscope observations, thermal gravimetric analyses, and mercury intrusion porosimetry were also performed as subsidiary experiments to understand the geotechnical features of FCPB. This information will be of significant value for numerous practical applications.

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Moreover, I am very grateful about the precious advice given by Pat Parungao; they are very helpful.

A special appreciation goes to St. Joe's supper table, the place that provided me with endless help during the hardest time.

Dedication

This thesis is dedicated to my family: my parents, Chang Xiuhe and Guo Qiuxia, my sister Chang Liang, and my brother Chang Xi. Their love and encouragement are the most important things in my life.

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AMD: acid mine drainage

CPB: cemented paste backfill

DEF: delayed ettringite formation

EC: electrical conductivity

FA: fly ash

FCPB: frozen cemented paste backfill

FPB: frozen paste backfill

HC: hydraulic conductivity

MIP: mercury intrusion porosimetry

PCI: Portland cement type I

RSM: response surface method

SEM: scanning electron microscope

SFCPB: sulphate frozen cemented paste backfill

SG: slag

TGA: thermogravimetric analysis

VWC: volumetric water content

W/C: water-to-cement

Chapter 1

Introduction

1.1 Problem statement

Ever since 1577, when the first mining operation was established by Martin Frobisher on Baffin Island, the mining industry has progressively become prominent across Canada. By the first half of the 20th century, Canada had already emerged as the world's leading producer of a large number of minerals. According to a recent report by the Canadian government, over 50% of the world's publicly listed exploration and mining companies are headquartered in Canada, accounting for more than 30% of global exploration expenditures in 2013 [2]. In terms of domestic contribution, mining and its related industries contributed more than 52 billion dollars to the gross domestic product (GDP) of Canada in the year 2012, ranking fourth out of all 18 Canadian industries. In total, it comprised 20.4% of the value of Canadian goods exported in 2012 [2].

On top of that, the mining and mineral processing industries supply hundreds of thousands of jobs in Canada. It is estimated that approximately 400,000 people were engaged in mining-related work in the country. It is also the largest private sector employer of First Nations people; around 1,200 aboriginal communities were involved in mining in 2012 [2].

However, alongside the significant contribution of the mining industry to the Canadian economy, there is a great concern about environmental disruption. Mining factories gen-

erate numerous adverse by-products, central among is mine waste, which can be roughly grouped into two main types: waste rocks and tailings. Waste rocks refer to any type of soil or rock surrounding host rocks, that have to be removed in order to create the access to ore. Tailings are leftovers or by-products generated during mineral processing. According to the Mining Sector Performance Report (1998-2012) [3], approximately 800,000 tonnes of tailings and 23,000 tonnes of waste rocks were disposed of in 2012. Figure 1.1 depicts the amount of mine waste discarded from 2006 to 2011. A clear and increasing trend can be seen since 2010. How to properly manage this huge amount of waste has been a critical topic since the mining industry was established in Canada. In the early days, with limited knowledge of tailings and their dire consequences for the environment, mining operators simply dumped them into adjacent rivers or ponds [4]. This arbitrary action caused enormous damage to the environs, including surface and underground water contamination, bio-accumulation of heavy metals in wild animals, and so on; these types of damage can take decades to remediate.

Alternative solutions were applied aiming to restrict these impacts. Tailings ponds were developed as a widely used method in modern mining [4] [5]. With an awareness of the consequences of acid mine drainage (AMD), this method was introduced with the aim of capturing tailings to the greatest possible extent. Since the reaction that produces AMD requires the presence of oxygen, the principal idea behind the construction of a tailings pond is to cut off their contact with air, which can be achieved by building tailing dams and using water as a cover.

However, drawbacks to this method soon became apparent. Because of budget shortages, most tailings ponds are challenged by numerous deficiencies, such as uncontrollable leaching of tailings, unpleasant visual pollution, and frequent breaches. Due to the above reasons and accompanying criticisms, engineers have been forced to invent new solutions for the storage of tailings.

Taking into account all the concerns mentioned above, the urgent need for a new technology dealing with mine waste seems to be undeniable and long overdue. Mine filling or mine waste backfilling was one solution that was put into practice from the middle of the

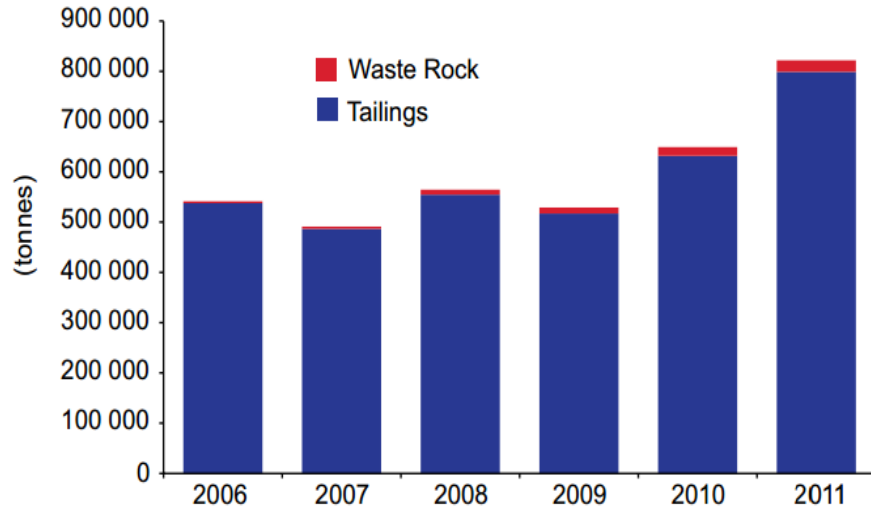


Figure 1.1: Tailings and waste rock disposal, 2006-2011 (Source: Environment Canada, National Pollutant Release Inventory).

20th century. The principal idea is to send mine waste, including rocks and tailings, back underground in the form of mine backfill, in order to fill the voids left by ore extraction. Cemented paste backfill (CPB) is one of the most popular methods of mine backfill; it has been applied to many mines around the world, and is gaining more and more accreditation. CPB is an engineered mixture of thickened and filtered tailings resulting from mineral processing, water, and binders (cement or pozzolans). Its solid content usually ranges from 70% to 85% by weight, depending on different requirements and needs. It is considered superior to other backfilling methods (e.g. dry fill, rock fill, and hydraulic backfill) for its success in minimizing engineering and environmental risks and challenges. More details about CPB will be discussed later in this thesis.

Once transported underground, mine backfill is expected to gain strength as much and as quickly as possible to ensure safe and effective working conditions. An unexpected mine backfill failure could not only lead to financial and environmental ramifications, but also be injuries and fatalities; many such incidents have occurred worldwide. The reasons for failures are various; poor quality of prepared mine backfills, unstable surrounding environments, and inappropriate backfill operations can all result in a mining catastrophic

disaster [5]. Therefore, it is vital to have an awareness of whether a given backfill meets the necessary mechanical stability requirements, as well as other information about its mechanical properties.

Extensive research on CPB (e.g. [6] [7] [8] [9] [10] [11] [12]) has been performed during the last 15 years in order to understand its geotechnical properties and mechanical behaviours. Tremendous progress has been achieved in this area; however, most of these studies were carried out at temperatures above freezing. There is a persistent lack of understanding of the geotechnical behaviour and properties of CPB in sub-zero environmental conditions. In addition, previous research indicates that pre-existing CPB structures are prone to a progressive loss in strength caused by sulphates, which have been found at high concentrations in both tailings and mix water at mining sites. The reaction of CPB to sulphates at temperatures below freezing point is also unknown. Former research related to CPB cured at temperatures above zero provides little help with these two scenarios. Meanwhile, the demand for raw materials for construction and other industrialized activities is increasing (see Figure 1.2); mining companies have started mining activities in Arctic areas, where temperatures remain below freezing (0°C) all year round, to meet this need (see Figure 1.3). Considering the complexity of frozen soil as determined by countless prior studies, and this increase in sub-zero mining activities, a thorough understanding of the geotechnical behaviour of CPB is necessary.

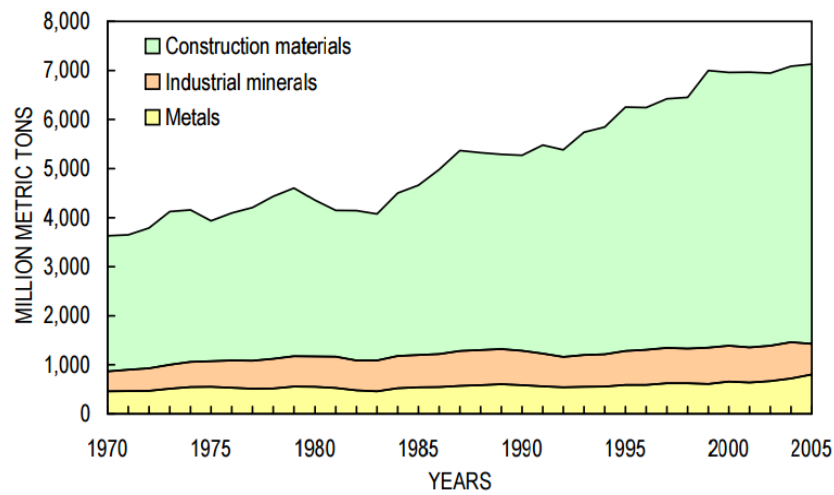


Figure 1.2: Global metal and mineral extraction.



Figure 1.3: Canadian mining industry clusters.

1.2 Objectives of this thesis

The main objectives of this thesis include:

- Studying the strength development of CPB cured in a sub-zero environment;
- Investigating the deformation behaviour of CPB cured in a sub-zero environment;
- Assessing the impact of sulphate on the strength development of FCPB cured in a sub-zero environment.

1.3 Research methodology

A large amount of literature, articles, and papers related to underground mining, mine fill, the physical and mechanical properties and behaviours of frozen soils, the physical and mechanical characteristics of ice, cement hydration, sulphate attack, and pozzolanic reactions, was reviewed in order to gain sufficient knowledge to write this thesis.

All tests and experiments were prepared and conducted with care in the laboratory. Wherever available, the 2004 revision of the American Society for Testing Materials (ASTM standards) was applied to ensure a valid result. In all circumstances, each set was prepared with a minimum of five samples to eliminate experimental error as far as possible.

To investigate the geotechnical behaviour of FCPB, various mixture combinations were examined to find the optimal mix. Uniaxial compressive strength tests were carried out throughout the whole experiment as the primary measure of the behaviour of FCPB. Microstructural analyses, such as mercury intrusion porosimetry and thermogravimetric analysis, were also performed as supplements to assist in the comprehension of FCPB.

A CAT II 600V multimeter was used to monitor the temperature changes within samples of different formulas, and to verify the conclusions of previous researchers that the strength of FCPB decreases at advanced ages due to rising temperatures from cement hydration. 5TE Water Content, Temperature, and Electrical Conductivity sensors were

embedded in FCPB samples to record the evolution of volumetric water content (VWC), temperature, and electrical conductivity, respectively.

1.4 Organization of the thesis

This thesis consists of five chapters. Chapter 1 presents an overall introduction of the thesis, including the problem statement, objectives, and methodology. Chapter 2 contains a review of the relevant literature, including studies on underground mining, mine back-fill, the physical and mechanical properties and behaviours of frozen soils, the physical and mechanical characteristics of ice, cement hydration, sulphate attack, and pozzolanic reactions. This is to provide readers with the necessary knowledge to understand the following chapters and a basic comprehension of the technical background under which the study was carried out. Chapters 3 and 4 are technical papers presenting the essence of this research. The two main objectives of the study were fulfilled by laboratory experiments. New findings obtained from these two papers will benefit future studies of and engineering processes for FCPB. Finally, in Chapter 5 general conclusions for the whole study are made, and expectations and suggestions for future work are discussed. Table 1.1 summarizes the organization of this thesis.

Table 1.1: Organization of the thesis

Chapter	Title	Content
1	General introduction	- Interests and motivation of the study; - Introduction and organization of thesis.
2	Technical background	- Mine tailings backfill; - Physical and mechanical properties of pore ice and frozen soils; - Cement hydration and sulphate attack.
3	Strength development of frozen cemented paste backfill	- Introduction; - Experimental program; - Results and discussion.
4	Study of the impact of sulphates on frozen cemented paste backfill	- Introduction; - Experimental program; - Results and discussion.
5	Conclusion	- Comparison of Papers I and II; - Conclusion and recommendations; - References.

Chapter 2

Background and Literature Review

2.1 Introduction

The successful performance of FCPB activities requires a sufficient knowledge of numerous aspects, such as CPB structural design, the physical and mechanical properties of CPB, ice, soil, frozen soil, and cement hydration. Accordingly, in this chapter a large number of relevant literatures and technical papers are reviewed to provide readers with the necessary knowledge to understand the following chapters, and a basic comprehension of the technical background against which the study was carried out.

2.2 Background on mine tailings backfill

Underground mining produces a huge amount of voids and an even larger quantity of mine waste; neither have any benefits for the industry. Overlooking or mishandling voids not only increases the risks associated with underground mining, but also aggravates the possibility of ground subsidence. Engineers often find themselves caught in a dilemma whereby, on one hand, they must produce the smallest possible amount of waste and voids, but on the other hand, they have to try their best to extract as much ores as possible to achieve the maximum recovery rate to satisfy business interests. The contradiction between these two

objectives is usually resolved via a compromise: walls or pillars are left behind to provide support for upper layers and the ground. However, the decision to leave valuable minerals in these edifices is always made reluctantly, and they often fail to perform as expected. Therefore, a new solution appears necessary.

If anything helped engineers make up their minds to turn things around, it was the growing public awareness of environmental preservation and the resultant criticisms. Since mine waste, in spite of its enormous quantity and problematic nature, is traditionally disposed of into rivers or tailings ponds, mine companies are facing more pressure and criticisms from the public than ever. The idea of mine backfill was mooted to deal with this problem, and has created high expectations. It offers engineers an opportunity to get rid of a huge amount of waste once and for all, and even better, to put it to a good use.

Backfill provides a method for the proper management of mine waste and also assists in facilitating the stability of mining-related voids. Backfill usually uses waste rocks or dewatered tailings as its main materials, sometimes mixed with cement or other binders to achieve higher strength. Fresh backfill is usually made in a plant located not far from a mine on the surface, and is delivered either by truck, pumping, gravity or combined ways to underground cavities. Figures 2.1 and 2.2 illustrate the procedures for cemented paste backfill activity, and the typical mine fill process in a stope.

Mine backfill also plays a multi-faceted role in mining. For example, it provides a working platform for the continuation of mining activities, ensures a stability of adjacent stopes and pillar walls, and maintains an acceptable extent of caving of stope. Furthermore, it reduces the amount of mine waste to be disposed of on the Earth's surface, lessening the disturbance caused by mining. It is also an effective method of eliminating AMD [12]. Potvin et al. [12] summarized the advantages of mine backfill into three points:

- Limiting excavation exposure and ensuring long-term regional stability: large excavations can easily lead to collapse with time. In this case, backfill can be applied as a bulking agent to occupy the voids and thus mitigates the risk of collapsing. For example, if the excavation becomes unstable and loosening material starts falling from the excavation boundary, backfill would prevent further loosening.

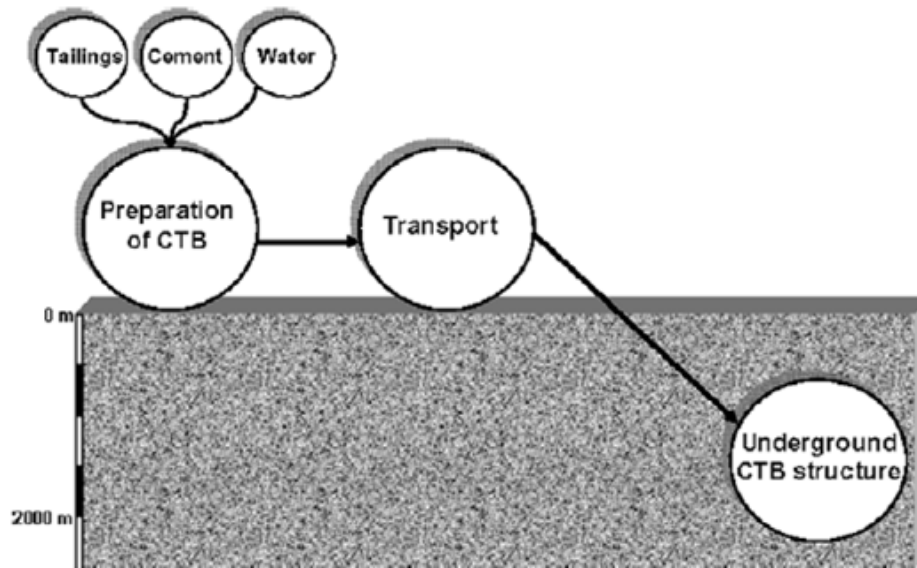


Figure 2.1: Schematic presentation of the different phases of CPB technology: preparation, transport and underground placing of the CPB, where it is used to build a CPB structure (Fall et al., 2008).

- Waste disposal: mine backfill is increasingly perceived as an environmentally friendly option to manage mine waste. Underground storage allows a reduction of surface storage of mine waste up to 60%. Studies also show that once captured underground, chemicals within mine waste tends to remain in an inert state, and most reactions are inhibited.
- Improving recovery rate of minerals: in most underground mining systems, pillars or walls have to be left behind to support the roof. However, with the application of mine backfill to supply the extra support, these areas can be exploited, thereby allowing a maximum recovery rate.

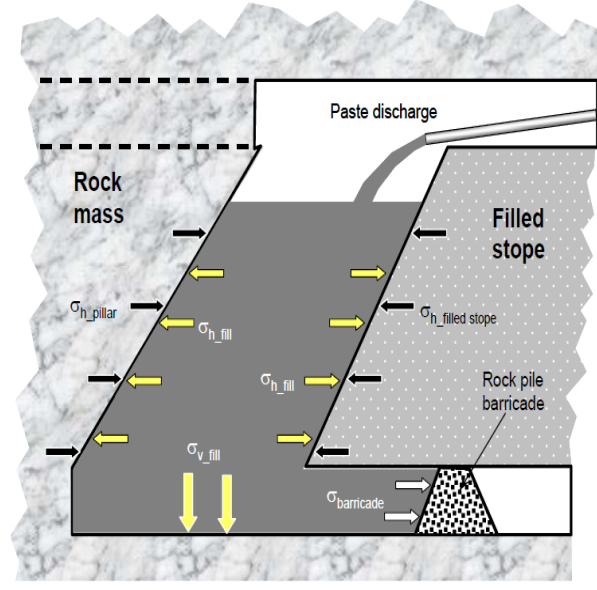


Figure 2.2: Schematic showing the components of a backfilled stope and the stress field (Belem and Benzaazoua, 2004)

2.2.1 Geotechnical design of CPB

A successful CPB operation can benefit mining activities in several ways: by offering a stable platform for miners to work on; providing extra ground support for the wall of the adjacent adits; preventing cave-ins and roof falls; and enhancing pillar recovery. It also makes mining extremely flexible and allows it to cope with changes in the geometry of the ore body [13]. However, designing CPB is complicated. It relies very much on the specific in-situ placement of each individual mine, as well as its practical operation processes. A thorough analysis should take into account: 1. backfill design parameters (e.g. internal pressure development, required strength, and mix optimization); 2. pumping qualities (consistency and rheological); 3. delivery to an underground operation through pipelines; and 4. work safety and expense [14].

Design of the horizontal pressure on the filled stope sidewalls

Since self-support stresses of CPB play a decisive role in backfill design, the traditional approach to design a backfill can be reduced to one simple rule: making sure the uniaxial compressive strength (UCS) of a free-standing wall equals at least the overburden stress at the bottom of the filled stope [14]. However, in practice, the adjacent walls are able to take part of the responsibility for supporting the backfill by arching effects and boundary shear [15]. Therefore, determining the correct the amount of extra support coming from rock walls is the first step in any CPB design. By definition, arching effects refer to pressure transfer from the vertical direction of the filled stope to the horizontal direction of the rock walls; this makes the pressure at the bottom of the filled stope less than the weight of the overlying fill [16]. The magnitude of pressure transferred to the rock walls can be evaluated by Martson's model and its modified version.

Martson's cohesionless model:

$$\alpha_h = \frac{\gamma B}{2\mu'} \left[1 - \exp\left(\frac{2k_a\mu' H}{B}\right) \right] \quad (2.1)$$

$$\sigma_v = \frac{\sigma_h}{K_a} \quad (2.2)$$

$$K_a = \tan^2(45^\circ - \phi/2) \quad (2.3)$$

where:

γ = fill bulk unit weight (kN/m³);

B = width of stope (m);

H = total height of filled stope (m);

$\mu' = \tan\sigma' =$ coefficient of sliding friction between fill and sidewalls;

$\sigma'(^{\circ}) =$ angle of wall friction;

ϕ = angle of internal friction of fill (degree);

σ_v = vertical pressure at the floor of the stope (kPa);

K_a = coefficient of active earth pressure.

Design for the required strength of a CPB

The required strength of a CPB depends on its intended function. According to Glasser et al. [17] and Kesimal et al. [18], this expected strength can be best represented in terms of uniaxial compressive strength (UCS) with an intended value between 0.7 and 2MPa. Other researchers [7] suggest a wider range, from 0.2 to 4 Mpa. Donovan [19] came up with equations to estimate the UCS for the simplest situation, where the only vertical support of the backfill need to be considered. Based on the assumption that any vertical loading will be a result of roof deformation, see Figure 2.3:

$$UCS_{design} = E_p \left(\frac{\delta H_p}{H_p} \right) FS \quad (2.4)$$

where:

E_p = the modulus of elasticity of the pillar;

H_p = the initial height of the strata;

δH_p = the deformation of the strata;

FS = factor of safety.

However, when pillar recovery is conducted after primary ore recovery, paste backfill will be prone to a large exposure in the vertical dimension (Figure 2.4). In this case, backfill must have sufficient strength to withstand blast effects and remain free-standing, or the already settled backfill block may fail [14]. Specific simulations were made to cope with various exposure situations (one exposed face, two exposed faces, narrowly exposed fill face, etc.). Based on the concept of a confined fill block surrounded by the wall rock, pseudo-2D

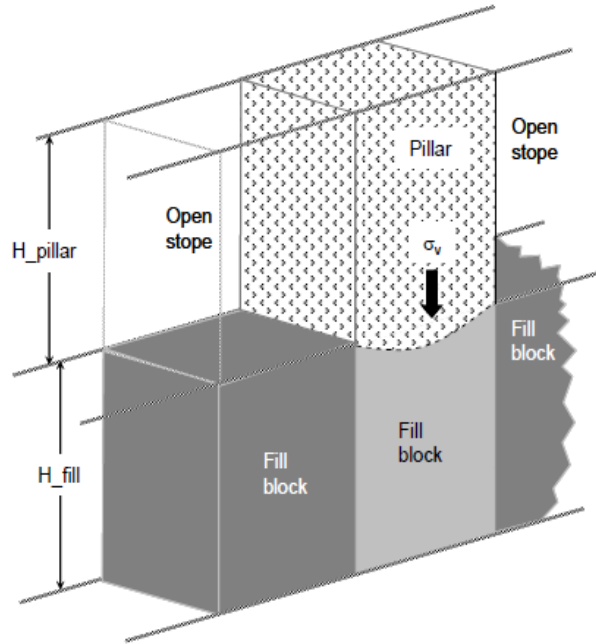


Figure 2.3: Schematic representation of the vertical loading of a pillar into the fill mass (Belem and Benzaazoua, 2004).

and -3D empirical models, developed in recent years, are able to take into account many factors such as arching effects, cohesion, and friction along sidewalls [14]. Ground support and working platform were also studied as follow-up procedures to ensure that backfilling operations run smoothly. With respect to cyclic backfilling operations such as the cut-and-fill mining method, which requires platforms for mining equipment and personnel, short-term strength is necessary.

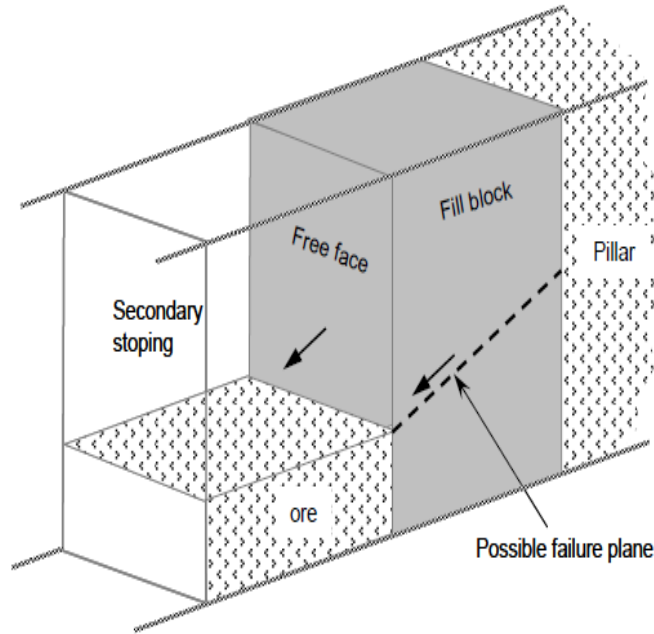


Figure 2.4: Fill block failure mechanism during secondary stoping (Belem and Benzazoua, 2004).

Optimization of paste backfill mix designs

As soon as the required strength is determined, optimization of the paste backfill mix can begin. The aim of this section is to find the most cost-effective method of mixing that does not upset the target strength. Factors for consideration include binder content and type, solids concentration, tailings grain size distribution and mineralogy, and water-to-cement ratio [9]. Fall et al. [8] combined the response surface method (RSM) with the desirability approach to develop a comprehensive method of determining the optimal mix composition. By taking cement, W/C ratio, fine-grains proportion, and density of the tailings as inputs, and RSM-based modeling as the tool, the potential cost, slump, and UCS can be determined. According to the intended design, all inputs can be adjusted to achieve the most desirable outputs (Figure 2.5).

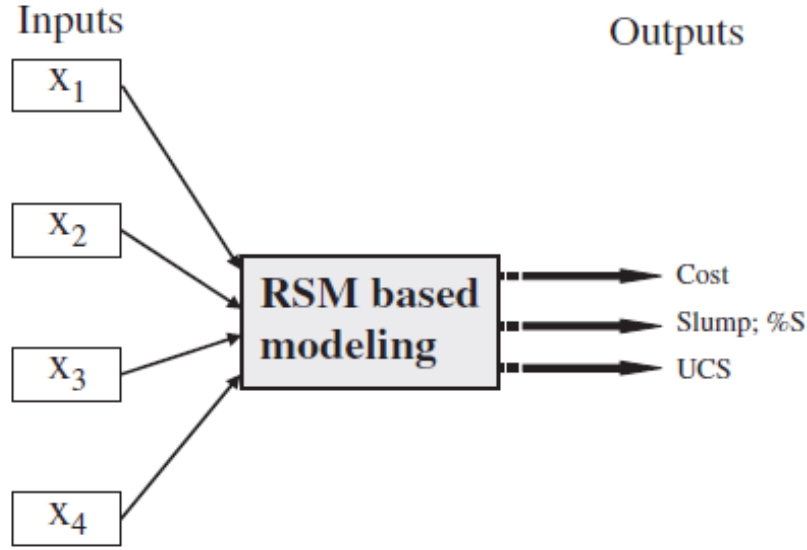


Figure 2.5: Schematic presentation of the developed models: cost in Canadian dollars per ton; slump in cm; percentage of solids (%S); and UCS in kPa (Fall et al., 2007).

CPB transportation and its rheological properties

Paste backfill is a slurry of high density that exhibits a non-Newtonian-fluid character and plug flow. With such high viscosity, it requires an applied force to maintain its flow in pipelines, or it will become stuck (Figure 2.6). The main flow mode for paste backfill in a pipeline is described as full-fall; that is, a continual flow with no air or any discontinuities existing in it. Its rheological properties such as viscosity, yield stress, and shear rate govern its flow behaviour, and can be determined through a standard slump test. The influence of rheological properties on a non-Newtonian fluid can be fundamentally illustrated by the relationship between its shear rate and pipe wall shear stress. Once this relationship is defined, the behaviour of a given non-Newtonian fluid can be deduced by the Herschel-Bulkley model (Equation 2.5), given by:

$$\tau_w = \tau_0 + K \left(\frac{dV}{dr} \right)^2 = \tau_0 + K \dot{\gamma}^n \quad (2.5)$$

Where:

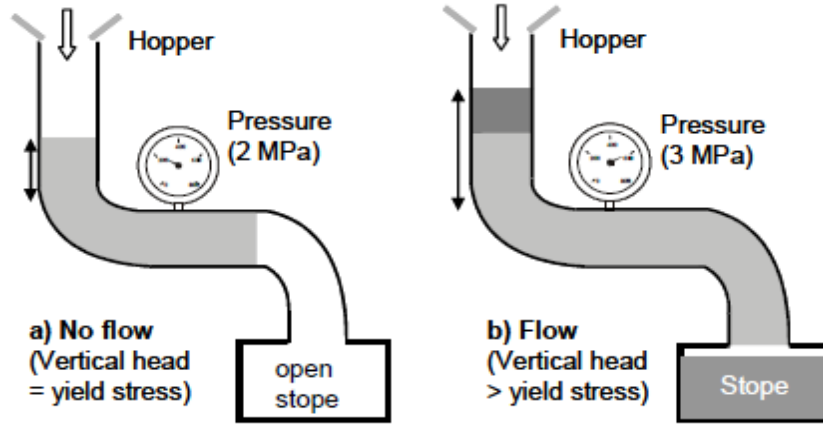


Figure 2.6: Schematic illustrating the yield stress of paste backfill flowing in pipelines (after Benzaazoua, 2004).

τ_0 = yield stress (Pa);

K = consistency parameter or viscosity (Pa.s);

$\frac{dV}{dr}$ = paste angular velocity or shear rate (s^{-1});

r = point of velocity profile (m);

R = radius of the pipe (m);

V = paste linear velocity (m/s);

n = flow parameter.

Three possible configurations for CPB transportation are widely used, as shown below (Figure 2.7); each has its own merits.

- The gravity/pump system is entirely located underground, causing minimal disruption to surface activities. In addition, little or no pumping energy is required, due to its favourable vertical to horizontal distance.
- The gravity system is granted, as it manages to convert vertical head to horizontal pressure progressively, and hence shorter and lighter pipes are qualified.

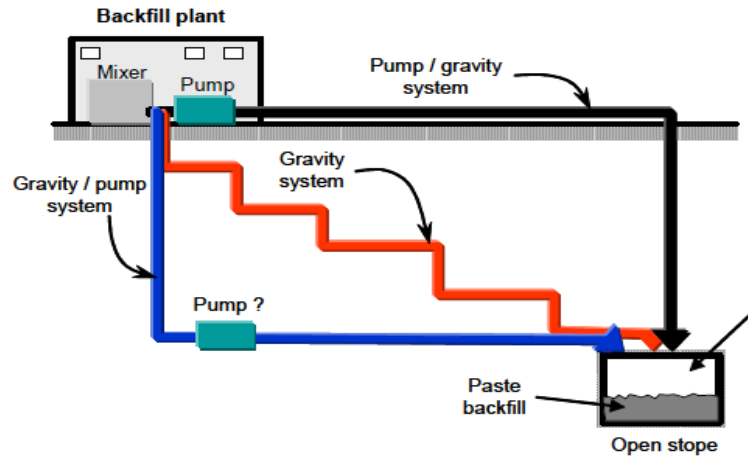


Figure 2.7: Basic configurations for paste backfill distribution systems (after Thomas et al., 1979).

- The pump/gravity system, meanwhile is easy to install, inspect, and maintain. It needs no additional preparation for underground level placement and causes little disruption of the main shaft.

2.2.2 Main sources of heat in underground mine and backfill operations

Temperature within an underground mine or backfill opening is complex and unique to each location. Fall et al. [10] summarized five main heat resources that can alter the temperature in such situations:

- (1) Depth of the mine: though mine stopes located at the same depth may be subjected to different geological conditions and thus have very different temperatures, there is a general correlation of higher temperatures with greater depths. Away from tectonic plate boundaries, it is estimated that temperature rises by 25°C per km of depth in most land areas of the world (see Figure 2.8).

- (2) Geographical location of the mine: this mainly relates location refers mostly to latitudes. A relatively shallow mine is significantly influenced by the climate of the region. For example, in a permafrost area such as northern Canada, the average ambient temperature under the ground surface is about -20°C in winter time and -5°C in summer time [20]. With respect to deeper mining, local rocks can remain consistently frozen down to a depth of 1000 metres [21].
- (3) Cement hydration and backfill transportation: cement hydration can produce a non-negligible amount of heat that can alter the ambient underground temperature significantly. Because most cemented backfill structures are characterized by a large size (e.g. a couple of metres in height and width), heat produced from exothermic cement hydration is unable to dissipate quickly enough, and will accumulate with time, especially in the central part of the structure. Previous studies [11] [13] demonstrated that cemented backfill temperatures reached 50°C after four days of curing. Aside from cement hydration, heat produced during transportation also plays an important role. Friction loss is the main factor governing this temperature increase.
- (4) Self-heating mechanisms: Bernier and Li [22] observed unusually high temperatures ($>400^{\circ}\text{C}$) in some Canadian underground mines. The reason for this was later found to be the exothermic reactions of sulphide minerals. Sulphides such as pyrrhotite are abundant in many Canadian mines, existing in the rock mass or cracked backfill structures. However, it should be mentioned that such extraordinarily high temperatures are not common and not yet well understood.
- (5) Other human-induced temperature variations: other than the four main heat resources mentioned above, some mining operations can also cause temperature increases as well. Machinery, blasting operations, lighting ventilation, fires, and so on can give rise to a certain amount of heat production in a relatively small area.

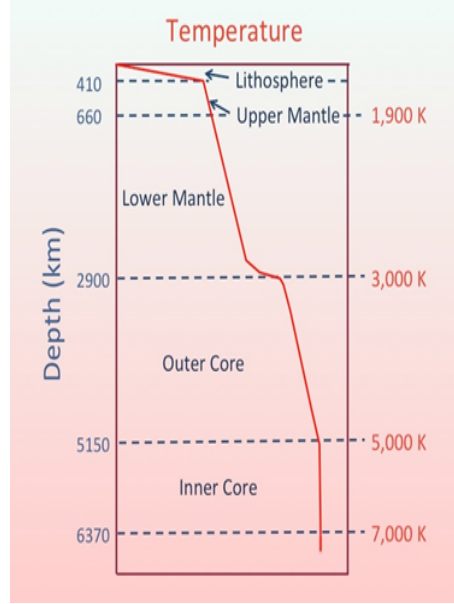


Figure 2.8: Temperature profile of the inner Earth, schematic view. (Source: Wikipedia)

2.2.3 Compressive strength of mine backfills and factors affecting it

Over the past few decades, the study and application of CPB has progressed considerably. Factors that play a role in the performance of CPB have been defined and precisely quantified. These achievements not only guarantee the spread of CPB techniques in the near future, but also provide a strong theoretical basis for the study of FCPB. Indeed, with little knowledge of FCPB and the enormous similarities between it and CPB, there is no harm in adopting the methodologies used in the research into CPB to develop the application of FCPB. In this thesis, the design and engineering principles used for FCPB followed the footprint of CPB. Potential obstacles and concerns were taken into account in advance from the perspective of CPB, so as to shorten the study period as much as possible. In this case, a brief elaboration of factors affecting the behavior of CPB is necessary.

Effects of tailings' properties on the strength of CPB

The most important property of tailings, in regards to their mechanical behaviour, is fineness content. Fineness dictates the boundary affinity among cement grains and tailings particles, and thus influences a series of factors such as overall porosity, cement matrix pores, and capillary porosity of the interfacial transition zones [6] [7] [23]. Fall et al. [8] conducted experiments investigating the proportion of fine materials in CPB that results in optimal strength. He suggested that CPB with 45% of fine materials by weight has the potential to develop the maximum strength. This claim was later verified afterwards by Mercury intrusion porosimetry. It was shown that at this proportion of fine materials, CPB displays the lowest void ratio, which indicates a high strength (see Figure 2.9). Any level of fineness content beyond this value would lead to a higher porosity, coupled with a growing water-to-cement (W/C) ratio.

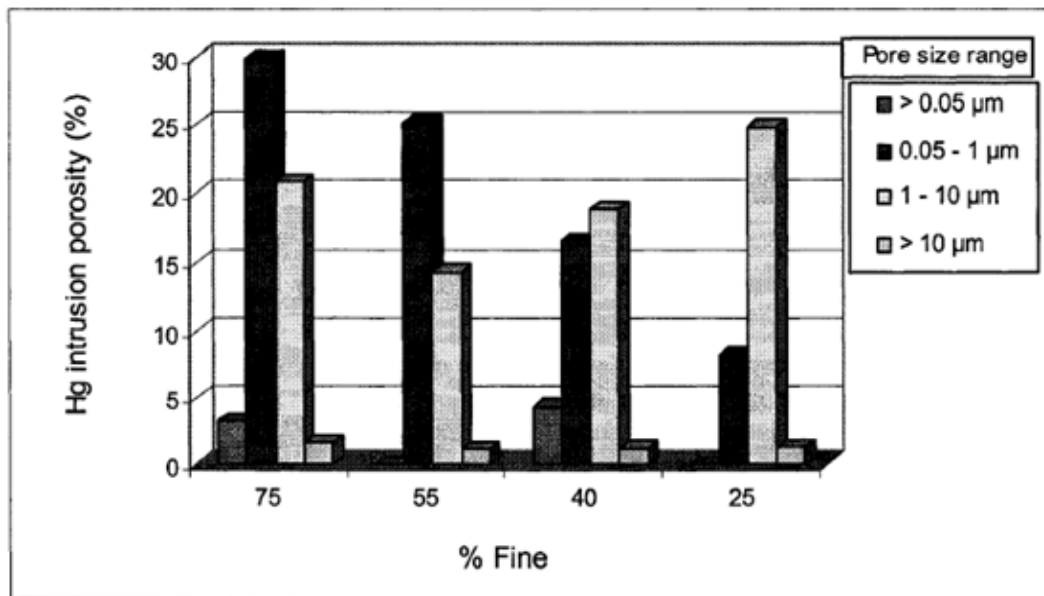


Figure 2.9: Mercury intrusion porosimetry showing large numbers of pores in the range of 0.05 to 1 micrometre in mixes formed from tailings whose content of fine materials is more than 45%. It also shows a large intrusion porosity for pores in the range of 1 to 10 microns in mixes formed from tailings containing less than 45% fine materials (Fall et al., 2008).

Effects of binder on the strength of CPB

The role of binder in CPB should be considered from two perspectives: its economic performance and its mechanical behaviour. From the first perspective, binder accounts for up to 75% of the total cost of CPB [24]. Therefore, finding a formula that requires the minimum usage of binder, and yet does not compromise the necessary strength of CPB, is one of the top priorities. As for the mechanical behaviour, it is primarily dependent on the degree of cement hydration, and which relies mainly on factors such as curing temperature, W/C ratio, binder types, and binder content. Commonly used binders in CPB are Portland cements (PC), fly ash (FA), and blast furnace slag (SG).

Fall et al. [10] summarized the behaviour of binder in terms of multiple factors. High temperature accelerates the rate of hydration of cement, and allows a higher strength of CPB. Excessive water, although it facilitates cement hydration, also increases the pore water content. As a result, the overall strength of CPB declines. Larger binder content undoubtedly favours the hardening process of CPB, since more hydration products form in it. The effect of binder types or composition on CPB, however, is rather complex and depends on curing temperature and age. In the early stage (<7 days), when the temperature is relatively low ($<20^{\circ}\text{C}$), CPB mixed only with PCI (Portland cement type I) exhibits a higher strength than others. This is because at a low temperature, the pozzolanic reaction is initially significantly inhibited and slag contributes little to the strength. However, as curing proceeds, CPB mixed with both PCI and SG presents a higher strength due to the pozzolanic reaction. As regards high curing temperatures, at early stages, CPB mixed with both PCI and SG develops a stronger strength. This is, again, due to the added binding effect from the pozzolanic reaction. However, after 150 days, CPB mixed with PCI and SG experiences a drastic reduction in strength. This is done by the “cross-over” effect, common in concrete materials, which results from a coarse pore structure caused by high initial curing temperatures.

Effects of the W/C ratio

For any fixed binder contents, the W/C ratio conclusively determines two important things in CPB: 1) slump and 2) void ratio. Unlike concrete casting projects, whose W/C ratio can be as low as 0.4, the W/C ratio of CPB is generally higher than 5 [22]. This distinctly excessive use of water is to ensure that a desirable slump and flowability will be achieved in the preparation. A desirable slump can be interpreted as: an appropriate level of workability of the mix that allows a cost-effective transportation from the surface to the disposal site, without causing any damage to the pipelines. A common value of slump in Canadian mines is around 18 cm. The W/C ratio also strong controls the porosity of the mix. As cement hydration and water drainage proceed within CPB paste, pores that were previously saturated with water become partial or total voids filled by air.

Curing temperature

Fall et al. [10] investigated the effect of curing temperatures on the strength development and deformation behaviour of CPB at four temperature levels. When comparing the results of samples with different binder contents, binder types, grain size distribution, and W/C ratios, they noticed that the curing temperature plays a significant role in the mechanical properties of CPB. A high temperature benefits the hardening process of CPB and, in particular, exhibits a more prevalent effect over other parameters. CPB displays a drastic increase in strength with the acceleration of curing temperature due to the high ratio of cement hydration. In addition, a high curing temperature ($<35^{\circ}\text{C}$) favours binder consumption, which in turn contributes to cost savings to a great extent.

Effect of sulphate content

Sulphates in CPB are known for sulphate attack, which manifests as hardened paste deteriorating and losing strength due to expansion. This expansion is caused by the formation of ettringite and gypsum. It is safe to say that the degree of deterioration increases with higher sulphate concentration.

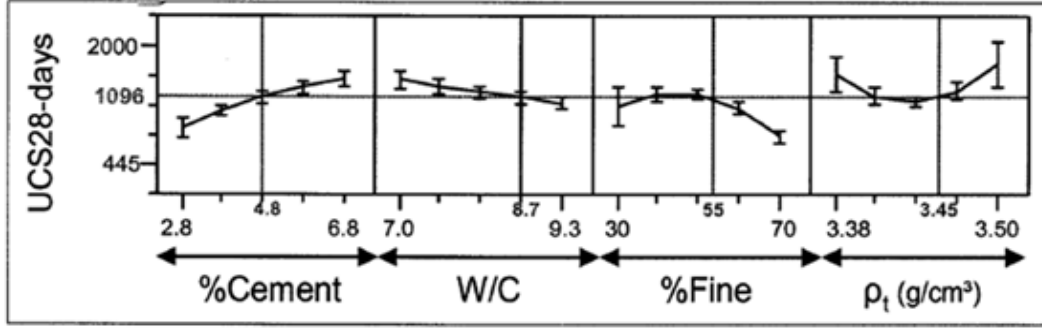


Figure 2.10: Effect of binder content, W/C ratio, tailing fineness, and tailings density on UCS (kPa) of CPB (after Fall et al., 2008).

Figure 2.10 illustrates the main factors related to the application of CPB, along with their effects. One issue that must be brought into attention is that these factors influence the behavior of CPB in a combined way.

2.3 Background on Cement Hydration

2.3.1 Cement definition and composition

Generally speaking, cement refers to a kind of building material made by grinding calcinated limestone and clay to a fine powder, which can be mixed with water and poured to set as a solid mass or used as an ingredient in making mortar or concrete [25]. Portland cement, developed from hydraulic limes, is one of the most common types of cement used around the world. It is also a basic ingredient of concrete, mortar, stucco, and most non-specialty grout [26]. According to ASTM C150, there are up to five types of Portland cement commonly applied today, known as type I types I, II, III, IV, and V. In this paper, all mentions of Portland cement specifically refer to type I. The compositions of Portland cement vary from plant to plant as a result of different mother materials (limestone and clay). However, it is generally composed of four primary types of minerals, as follows: tricalcium silicate (Ca_3SiO_5), dicalcium silicate (Ca_2SiO_4), tricalcium aluminate ($\text{Ca}_3\text{Al}_2\text{O}_5$), and calcium aluminogerrite ($\text{Ca}_4\text{Al}_n\text{Fe}_{2-n}\text{O}_7$). Table 2.1 shows an abbrevia-

Table 2.1: Chemical formula and cement nomenclature for major constituents of Portland cement (Source: <http://cnx.org/content/m16445/latest>)

Mineral	Chemical formula	Oxide composition	Abbreviation
Tricalcium silicate (alite)	Ca_3SiO_5	$3\text{CaO}.\text{SiO}_2$	C_3S
Dicalcium silicate (belite)	Ca_2SiO_4	$2\text{CaO}.\text{SiO}_2$	C_2S
Tricalcium aluminate	$\text{Ca}_3\text{Al}_2\text{O}_4$	$3\text{CaO}.\text{Al}_2\text{O}_3$	C_3A
Tetracalcium aluminoferrite	$\text{Ca}_4\text{Al}_n\text{Fe}_{2-n}\text{O}_7$	$4\text{CaO}.\text{Al}_n\text{Fe}_{2-n}\text{O}_3$	C_4AF
Abbreviation notation: C=CaO, S=SiO ₂ , A=Al ₂ O ₃ , F=Fe ₂ O ₃			

tions of the nomenclature of these four materials, based on the oxides of various elements. In most conditions, gypsum is also included in the production process to avoid flash set caused by a high rate of aluminate hydration. Taylor [27] summarized the portion of each component into a typical clinker, in which CaO, SiO₂, Al₂O₃, Fe₂O₃, and others account for 67%, 22%, 5%, 3%, and 3%, respectively.

A more thorough understanding of Portland cement can be gained through the cross-section view in Figure 2.11, which illustrates the composition of a cement grain.

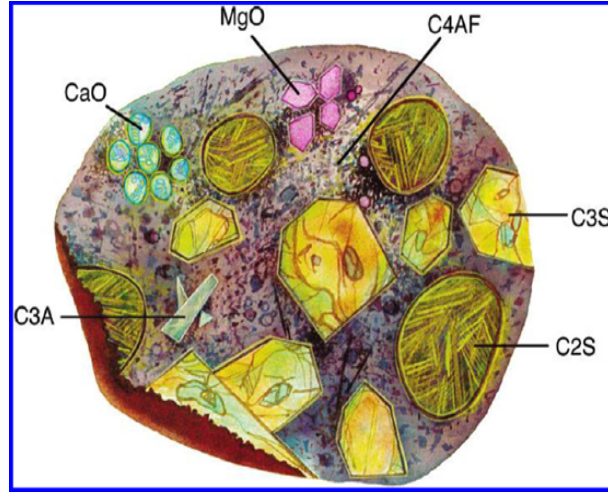


Figure 2.11: A pictorial representation of the cross-section of a cement grain. (Source: <http://cnx.org/content/m16445/latest>)

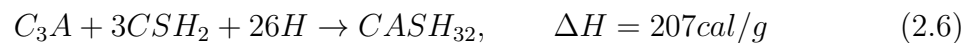
2.3.2 Cement Hydration

To sum up, cement hydration refers to a process in which cement derives its strength from a series of chemical reactions between cement and water. It generally contains two steps, dissolution and precipitation. In the first step, cement dissolves in water, releasing ionic constituents to the solution. This process continues until the solution is supersaturated, which means no more ions can be dissolved. At this point, the second step begins wherein the excess ions combine into new solid phase and precipitate out as hydration products rather than remaining dissolved. In the meantime, precipitation gives rise to a further dissolution of cement in water as the concentration of the solution decreases.

This process can be summarized in a series of chemical reactions below:

- The tricalcium aluminate reacts with the gypsum in the presence of water to generate ettringite and heat:

Tricalcium aluminate + gypsum + water $\rightarrow$ ettringite + heat



(Ettringite is comprised of long crystals that are known to remain stable only in a

gypsum solution. The compound contributes little to the strength of the cement glue.)

- The tricalcium silicate is hydrated to produce calcium silicate hydrates and lime, as well as heat:

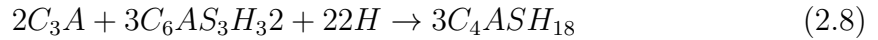
Tricalcium silicate + water $\rightarrow$ calcium silicate hydrate + lime + heat



(The CSH consists of a short-networked fibre structure that contributes greatly to strength of the cement glue, especially the initial strength.)

- Once all the gypsum is used, the ettringite will become unstable and react with the remaining tricalcium aluminate to produce monosulphate aluminate hydrate crystals:

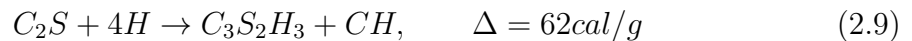
Tricalcium aluminate + ettringite + water $\rightarrow$ monosulphate aluminate hydrate



(The monosulphate crystals are only stable in a sulphate-deficient solution. So in the presence of sulphates, the crystals revert back ettringite with crystals that are two-and-a-half times the size of the monosulfate. This increase in size is the main reason for cracking and is known as sulphate attack.)

- The dicalcium silicate also hydrates to form calcium silicate hydrates and heat:

Dicalcium silicates + water $\rightarrow$ calcium silicate hydrate + lime

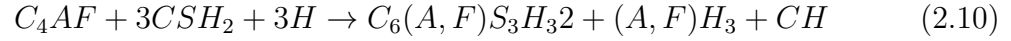


(The calcium silicate hydrates also make a contribution to the strength of the cement paste. This reaction produces less heat and proceeds at a relatively slower rate, which means that the contribute of C₂S to the strength of the cement paste will initially be slow. However, this compound is responsible for the long-term strength of Portland cement concrete.)

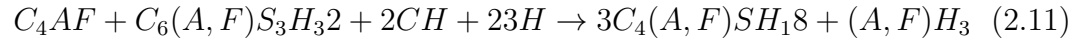
- The ferrite goes through two progressive reactions with the gypsum:

1. In the first of the reactions, the ettringite reacts with gypsum:

Ferrite + gypsum + water $\rightarrow$ ettringite + ferric aluminum hydroxide + lime:



2. Ferrite + ettringite + lime + water $\rightarrow$ garnets:



The garnets contribute little to the strength of the cement paste.

2.3.3 Properties of hydrated cement compounds

Figure 2.12 and Table 2.2 summarize the properties of each hydrated cement compound, along with their contribution to the strength at different time intervals [28].

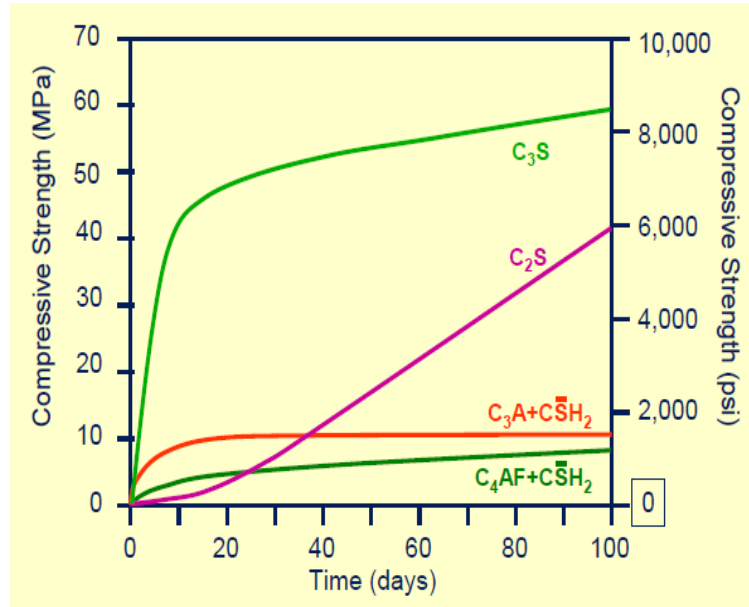


Figure 2.12: Compressive strength development in pastes of pure cement compounds.

(Source: Mindess et al., 2003; Kurtis, 2007)

Table 2.2: Properties of hydrated cement compounds [1]

C_3S	Tricalcium silicate (alite)	- Hydration and hardening occur rapidly - Responsible for initial early strength on long term strengths
C_2S	Dicalcium silicate (belite)	- Hydration and hardening occur slowly - Contributes to the strength of later age (after 7 days)
C_3A	Tricalcium aluminate	- Releases a huge amount of heat in the first few days - Contributes slightly to the early strength - Responsible for initial set
C_4AF	Tetracalcium aluminoferrite (ferrite)	- Hydration occurs intensively but does not contribute in any way to the strength - Ferrite hydration causes the color to change to grey

It is clear from the Table 2.2 and Figure 2.12 that the short-term strength of cemented materials derives from the hydration products of C_3S . Long-term strength, however, is provided by the hydration products of C_2S [27] [29].

The hydration rate is dependent on a number of factors including curing temperature and additives [30].

2.4 Effect of sulphate on cementitious materials

2.4.1 Sulphate attack

When cement-built structures, such as dams, bridges, and piers, are located in a sulphate-rich environment, it is often seen that they tend to deteriorate with time. Visible expansion and cracks can often be discovered in such structures at a certain point, and eventual failures seem to be inevitable [31]. This deterioration process of strength in cementitious structures, due to exposure to sulphate salts, is known as sulphate attack. Based on the mechanism behind this process, sulphate attacks can be classified into two types: external and internal. External attacks are more common and usually take place where water containing dissolved sulphate penetrates the structure. Internal attacks only occur when the amount of sulphate exceeds that of gypsum, which is also observed in CPB. However, no matter what the type, sulphate attack is considered one of the most severe threats to modern structures that contain a large amount of concrete [32].

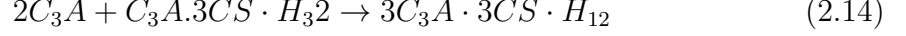
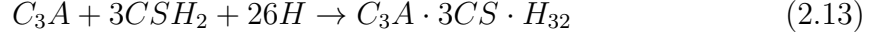
According to Bing and Cohen [33], two main chemical reactions are expected to happen in a sulphate attack:

- 1) the formation of gypsum derived from the reaction between sulphate and CH:



- 2) the formation of ettringite as a result of the reaction between sulphate ions and tricalcium aluminate (C_3A); when the concentration of sulphate ions is low, the

ettringite will break into monosulphates to attain a stable state:



The formation of ettringite and gypsum affects the durability of hardened cementitious materials [32]. When ettringite develops within such a material, its volume expands significantly, and thus needs sufficient room to grow. If this demand is not met, the ever-growing ettringite will exert force on the adjacent environment until it exceeds the tensile strength of the surrounding structure and causes cracking and [7] [33] [34]. However, the role of ettringite in sulphate attack is still a subject of controversy, and researchers cannot agree whether the formation of ettringite in concrete gives rise to expansion [33]. Nevertheless, despite the doubts that have been cast on ettringite, more researchers are now focussed on the adverse effect it may cause. The formation of gypsum, meanwhile is explicit. As a result of cation exchange, gypsum causes concrete or other cementitious materials to experience loss of stiffness and strength, followed by expansion and cracking, eventually turning into a soft or non-cohesive mass.

Since sulphate attack is a rather complex process, its odds and degree cannot be predicted and defined by limited factors. Cement type and content, sulphate type and concentration, chloride ions, soil porosity, the form of the construction, sulphate concretion, and so on all play a part in the outcome of a sulphate [31] [34] [35] [36]. Sulphate concentration is the first factor that should be considered; there is no doubt that as the concentration of sulphate increases, sulphate attack becomes more intense. With regards to cement type and content, an emphasis has been placed on its mineralogical phases (e.g. C_3A , C_3S/C_2S ratio, and C_4AF). In most cases, cement with low C_3A content exhibits a lower vulnerability to sulphate attack. However, when low C_3A content is coupled with a higher C_3S/C_2S level, it will lead to the opposite result. This is because the increased C_3S content has the ability to generate a larger amount of calcium hydroxide ($Ca(OH)_2$), which is able to react with sulphate ions to produce gypsum. As mentioned above, gypsum is harmful to the durability of concrete [32].

Many measures have been taken to prevent or minimize sulphate attack in concrete materials. The ACI Building Code 318-83, which was issued by the American Concrete Institute (ACI) to specify the requirements for four classes of sulphate concentration in concrete.

- Negligible attack: When the sulphate content is less than 0.1% in soil, or less than 150 ppm (mg/l) in water, there shall be no restriction on the cement type and W/C ratio.
- Moderate attack: When the sulphate content is 0.1 to 0.2% in soil, or 150 to 1,500 ppm in water, ASTM Type II Portland cement, Portland pozzolan, or Portland slag cement shall be used, with less than a 0.5 W/C ratio for normal-weight concrete.
- Severe attack: When the sulphate content is 0.2 to 2.0% in soil, or 1,500 to 10,000 ppm in water, ASTM Type V Portland cement, with less than a 0.45 W/C ratio, shall be used.
- Very severe attack: When the sulphate content is over 2.0% in soil, or over 10,000 ppm in water, ASTM Type V cement plus a pozzolanic admixture shall be used, with less than a 0.5 W/C ratio.

Blending cements with pozzolanic materials, such as fly ash, blast furnace slag, and silica fume, has been widely adopted as an effective method of controlling sulphate attack. Salah and Al-Dulaijan [36] concluded from their research that Portland cement mixed with pozzolanic binders shows a stronger resistance to sulphate attacks than Portland cement used alone. The reason for this is that in the presence of water, pozzolanic materials will consume a certain amount of CH during pozzolanic reaction, and produce secondary CSH. This has two benefits. First, as CH decreases, the amount of gypsum produced also falls, inhibiting concrete expansion. Second, the formation of secondary CSH in the cement paste alters the inner structure of concrete, making it less impermeable and stronger. As a result, the ingress of sulphate is significantly limited [37]. Aside from the pozzolanic reaction, adding pozzolanic materials also reduces the portion of C_3A . This, in turn, lowers

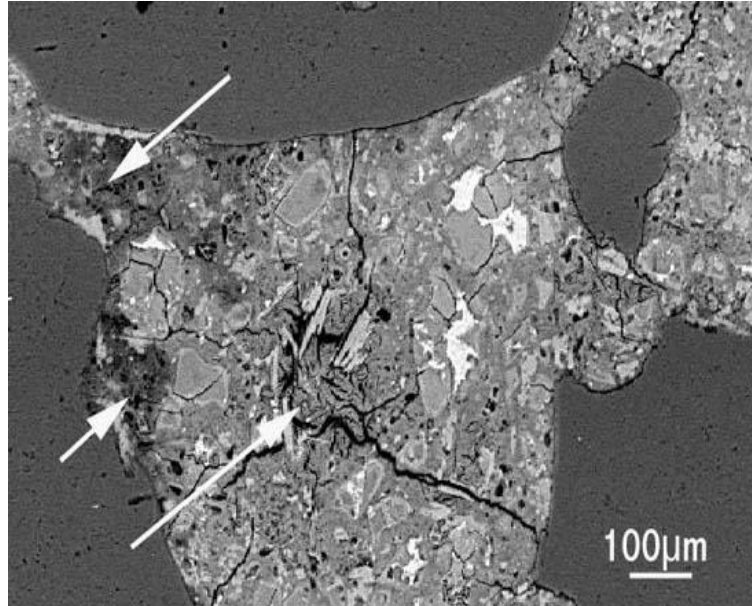


Figure 2.13: Scanning electron microscope image of sulphate attack in concrete. (Source: <http://www.understanding-cement.com/sulfate.html>)

the potential for ettringite formation [37]. Sahmaran et al. [32] reached the aforementioned conclusions by conducting SEM testing. This shows that even after prolonged exposure to a sulphate-bearing environment, hydration products from blended cement exhibit little formation of ettringite or gypsum.

In Figure 2.13, it can be seen very clearly that ettringite (arrowed) has encroached into parts of the calcium silicate hydrate (the white areas are alternating with dark areas). As a result of these replacements, this cement paste has become weaker than it initially was. Although most parts of the cement paste seem to be intact at this point, the ettringite will eventually spread throughout the entire paste and damage it significantly.

2.4.2 Delayed ettringite formation

Delayed ettringite formation (DEF) is another type of internal sulphate attack that we need to be aware of. It has been a significant problem in many countries and can occur without the necessity for excessive sulphate. The main cause of DEF is believed to be

elevated temperatures. For example, in a pre-cast and massive concreting project where the temperature within the concrete is constantly maintained at over 70°C, the formation of ettringite is severely inhibited, and a considerable amount of sulphate is adsorbed by CSH gel [38] [39]. However, the concrete structure will eventually cool down and the sulphate adsorbed at the earlier stage will be gradually released, so that the production of ettringite will begin in the already-hardened concrete, causing cracks. Other than a high temperature, the presence of water and alkali-silica reactions are also considered necessary conditions for the occurrence of DEF. Yet, despite this limited knowledge, the mechanism of DEF is still not fully understood.

2.4.3 Source of sulphate in backfill operations

The sulphate involved a sulphate attack on concrete can either come from an external source or be inherent within the cementitious materials. For most concrete structures located in the ground, the potential source of sulphate is most likely the outside; for example, from ground water or soil [40]. Possible methods by which these sulphates to get can infiltrate concrete structures include diffusion, sorptivity, and capillary suction [41]. In CPB, however, the main source of sulphate is from tailings. Elberling et al. [42] claimed that most tailings applied in CPB have a high chance of containing ferrous sulphide minerals in the form of pyrite or pyrrhotite. The concentration of sulphide in such tailings ranges from 2 to 6%, depending on the composition of the mother ores [7]. These sulphides become oxidized in the presence of oxygen and moisture, and then produce an effluent with a high concentration of sulphate and a low pH value [42]. In addition, the application of mine process water can also result in increased sulphate content in CPB materials.

2.5 Background of physical and mechanical properties of pore ice

2.5.1 The structure of ice

It has been verified that ice can exhibit in fifteen different phases [43]. (See Figure 2.14). The most common type of ice in daily life is I_h , which dominates Earth's surface and atmosphere. This type features an ordered structure with a six-fold symmetry, containing an oxygen atom at each end [43]. This hexagonal crystal structure led to it being named hexagonal ice or I_h . I_h has a density of around $9.17 \times 10^2 \text{ kg/m}^3$ at 0°C , which is a little lower than that of water. However, one thing that must be noted is that this value is not constant: it increases as temperature goes down. Other physical properties of I_h can be found in many related papers (e.g [44]), including that its latent heat of melting is 5987 J/mol.

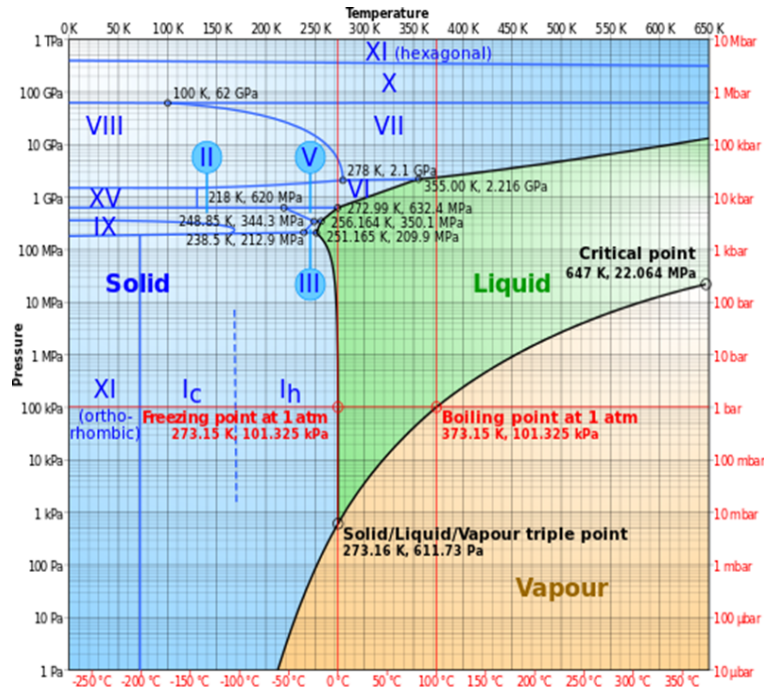


Figure 2.14: Phase diagram of water. (Source: <http://en.wikipedia.org/wiki/Ice>)

It is believed that only I_h exists within frozen soil [43]. Dutch [45] and Chaplin [46]

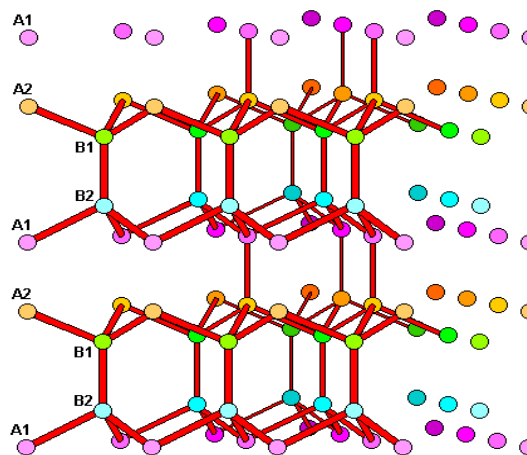


Figure 2.15: Structure of hexagonal ice. (Source: <http://www.uwgb.edu>)

further specified hexagonal ice's characteristic: the crystals can be described as consecutive sheets lying on top of each other. Ordinary ice is composed of a stacking arrangement of two lattices with close-packed hexagonals that interpenetrate each other. Although the hexagonals are closely packed, the molecules themselves do not face any constraints. A tetrahedron of equal spheres is achieved by each vertex of the tetrahedron being fixed 3.27 radii apart. A front and a top view (Figures 2.15 and 2.16) of I_h briefly show the relationship between the two layers. In Figure 2.15, the red lines represent O-H-O lines (hydrogen bonds), and darker colours mean the direction is toward the rear of the structure.

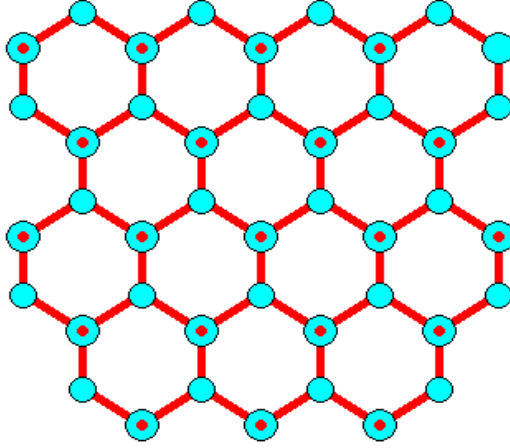


Figure 2.16: Top view of the I_h structure. (Source: <http://www.uwgb.edu>)

In Figure 2.16, the red dots represent two B layers joined by an O-H-O link. The ones without the red dots indicate a joint between two A layers. The distance between two adjacent oxygen atoms is around 275pm.

The microstructure of natural ice is mainly determined by its thermo-mechanical history. Typical ice grains vary in size from 1 mm to 20 mm, and typical shapes range from equiaxed to elongated. For example, glacial ice develops from snow being sintered together under pressure, so its microstructure features an equiaxed crystals and randomly oriented grains close to the top. However, when it flows down mountains, creep deformation and dynamic recrystallization may lead to reorganization, making its structure more complex. Arctic sea ice, meanwhile, develops from unidirectional solidification of salt water, characterized by column-shaped grains that extend the growth direction.

Ice is able to exhibit both brittle and ductile behavior in an UCS test, depending on what stress is used and how it is applied. Temperature is eliminated in this scenario due to the fact that the melting point diffusivity of I_h is less than 10-15 m/s, leading to relatively slow diffusion-assisted stress relaxation [47]. Strain rate, however, has been proven to be the dominant factor in determining the mechanical behaviour of ice. At low (less than 10^{-7}s^{-1}) and intermediate (10^{-7}s^{-1} to 10^{-3}s^{-1}) strain rates, where cracks do not form or are tolerable, ice exhibits ductile behaviour. At high (more than 10^{-3}s^{-1}) strain rates, ice shows brittle behaviour [47].

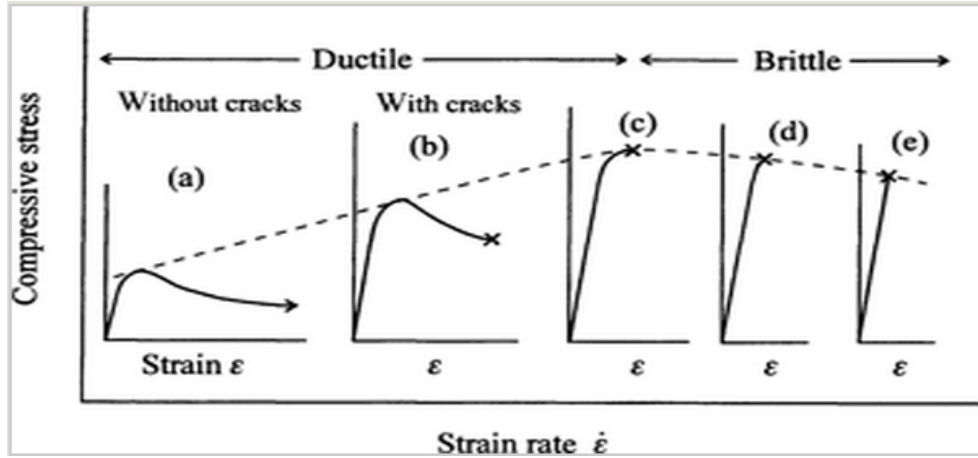


Figure 2.17: Series of stress-strain curves illustrating the ductile-to-brittle transition with increasing strain rate; crosses indicate the point of fracture, but at the lowest strain rates creep can continue indefinitely (after Petrenko and Whitworth, 2002).

Figure 2.17 is an example of the mechanical behaviour of ice under five different strain rates. Curve A and curve B characterize low strain rates; both show ductile behaviour. Curve D and curve E represent high strain rates, indicating brittle behaviour. Brittle behaviour sets in at a high strain rate, breaking up after achieving less than 0.5% deformation [47].

Studies on the mechanisms of the deformation of ice have been mostly based on laboratory-scale experiments, which are generally broken into two separate and yet also overlapping subjects: deformation of single ice crystals, and deformation of polycrystalline ice. Single crystals are highly anisotropic. They creep in a form of glide and multiplication of dislocations on the basal plane [48]. On a polycrystalline level, a large amount of stress could be built up among individual grains under this constant load, and if it is not relieved by diffusion, cracking would occur to dissipate the stress. This fracture theory involves both propagation and nucleation, and will be further discussed in the following sections. The way polycrystalline ice deforms can be described as follows: when a constant load is applied, the ice's creep rate first accelerates, then becomes steady, and eventually accelerates again.

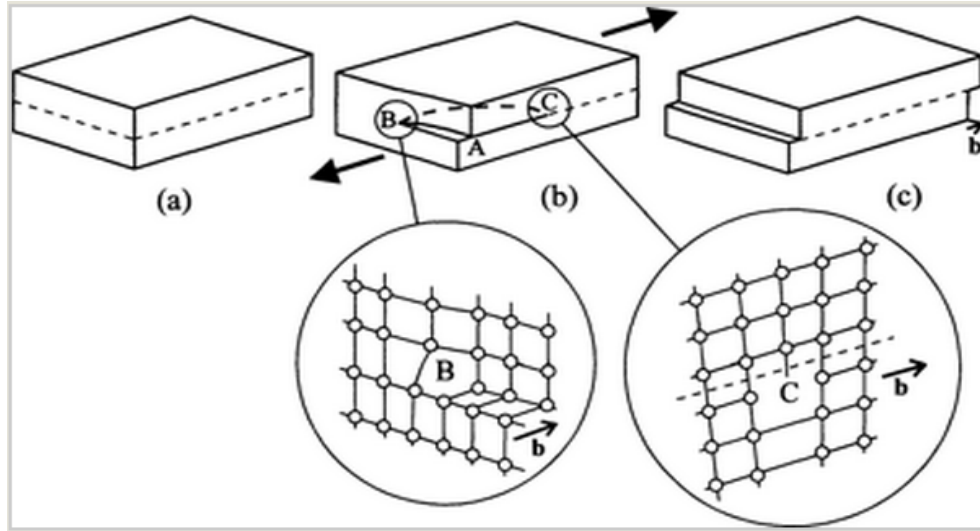


Figure 2.18: The production of a slip in a crystalline material by the glide of the dislocation BC across the slip plane, starting at A. The amount of slip is given by the Burger's vector b . The magnified portions show the arrangement of atoms around the screw component at B and the edge component at C (after Petrenko and Robert, 2002).

Plastic deformation of single crystals

Single crystals, as mentioned above, are known for their high anisotropy; this characteristic makes them very ductile. An experiment conducted by Glen and Perutz [49] showed that a rod was extended into a ribbon with a length six times its original length. This could be a result of a special motion of ice known as basal slip. This refers to dislocations (line defects in crystals) of single crystals gliding through the lattice, and causes one layer of molecules to slip over another (see Figure 2.18).

In practice, an entire layer slipping over the plane simultaneously is impossible. Usually, a slip initiates at one corner, then proceeds to the rest of the plane. Figure 2.18 illustrates a slip process. In this figure, (a) is an intact crystal of two planes, while (b) presents an intermediate stage where a slip occurs at the corner, and proceeds in close proximity to lines B and C. Lines B and C are about to split and act as boundary lines. These boundary lines can also be interpreted as dislocation lines, or dislocations for short, which are the induce elastic distortion. The lattice Vector b that makes slips occur is known as the

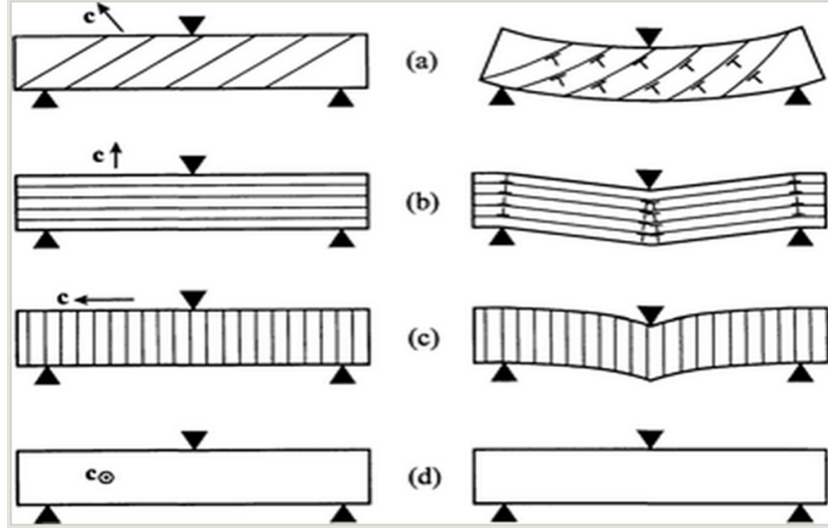


Figure 2.19: Modes of plastic deformation in three-point bending of single crystals of ice with differently oriented c-axes (after Petrenko and Robert, 2002).

Burger's vector of the dislocation. Finally, (c) is the configuration after a total basal slip. However, not all dislocations happen in the form of a basal slip. When the direction of the basal Burger's vector changes, the deformation will change with it, even though it takes more energy. This kind of dislocation is known as a non-basal dislocation (see Figure 2.19).

Plastic deformation of polycrystalline ice

Now, let us take a step back and look at the picture on a larger scale. Since single crystals of ice are highly anisotropic, when grains of different orientation aggregate into a polycrystalline structure they cannot easily be deformed in a mutually compatible way [48]. As a result, when applying force to polycrystalline ice, a basal slip will start to occur for those grains that are favourably oriented. Yet for those grains oriented otherwise, they may stand still, leading to a buildup of stresses within polycrystalline ice. All these stresses play a role in the deformation of ice and must be relieved in some way. Petrenko and Robert [48] summarized seven rules of physical processes in relation to the deformation of polycrystalline ice:

- 1) Non-uniform deformation caused by various Burger's vectors resulting in different

bonds and tilt boundaries as shown in Figure 2.19; this is referred to as polygonization.

- 2) Slips do occur on prismatic planes with significantly high stress concentrations.
- 3) Grains can slide over one another. Partial sliding may also take place when two adjacent grains deform differently.
- 4) Migration of grains' boundaries occurs, resulting in some grains growing at the expense of others. The principle behind this is that the rearrangement of molecules on one side of the boundary merges into the structure of those on the other side.
- 5) Diffusion-controlled recovery can occur within the deforming grains.
- 6) Dynamic recrystallization can be found in regions subjected to highly deformed recrystallization. This process is rather fast in contrast to annealing.
- 7) Cracks may be initiated when the stress becomes too high. Their formation facilitates the stability of ice for a certain time due to the relief of stress, but eventually brittle fractures are inevitable because of the growth of such cracks.

Creep curves

The creep behaviour of ice has been extensively studied; however, it is still not fully understood. Ice exhibits non-viscous behaviour under constant stress or an incessant strain rate, which implies that the strain rate is not dependent on the stress alone, but also on the time. In a case where an ice specimen is under constant stress, deformation begins, and the strain rate decreases with time to a minimum degree. However, from this point onwards, the strain rate starts to increase again and reaches an asymptotic limit. Figure 2.20 shows a typical creep curve for polycrystalline ice. It is very obvious from the figures that three regions are exhibited, corresponding to the progress just mentioned. Curve B-C is known as primary creep. It is highly important from an engineering point of view, since most engineering projects dealing with cold regions experience it. Curve C-D is known as secondary creep, which is followed by tertiary creep (curve D-E).

Models have been developed to predict the creep behaviour of ice based mainly on the creep law proposed by Hult [50], which is written as follows:

$$\epsilon_c = K \sigma^\alpha t^b \quad (2.15)$$

The famous strain-hardening formulation is derived from this one, and shown as:

$$\frac{d\epsilon_c}{dt} = \dot{\epsilon}_c = b K^{1/b} \sigma^{a/b} [\epsilon_c]^{(b-1)/b} \quad (2.16)$$

Where:

ϵ_c = creep strain;

σ = stress applied to the ice;

K = temperature-dependent constant;

a = independent constant;

b = independent constant.

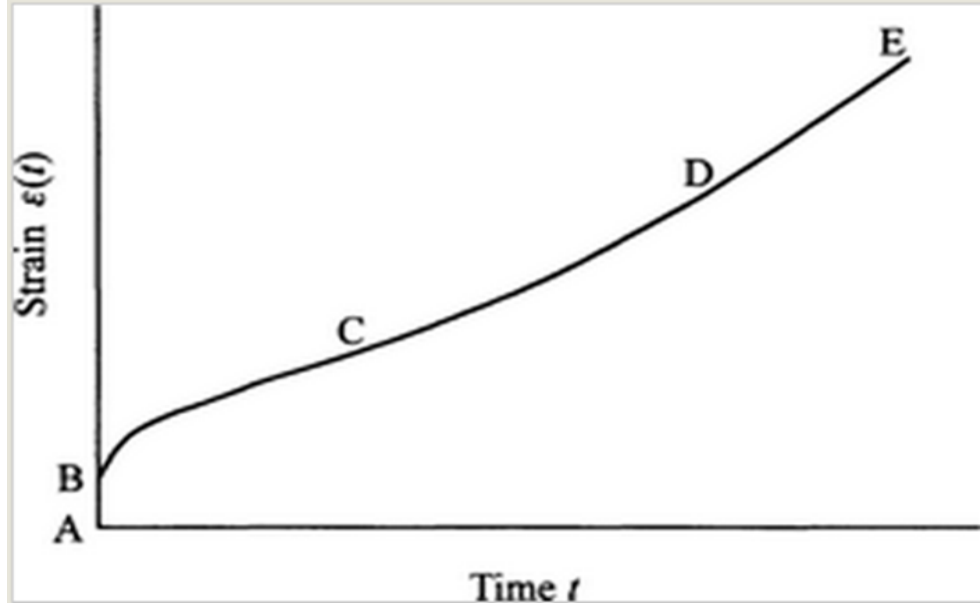


Figure 2.20: Schematic creep curve for polycrystalline ice under constant load (after Petrenko and Robert, 2002).

2.6 Background of physical and mechanical properties of frozen soils

Frozen soil is introduced to the study of FCPB in this section due to the similarities between the two. A thorough understanding of the properties and mechanical behaviour of frozen soil is believed to facilitate the comprehension of the behaviour of FCPB.

2.6.1 Physical properties of frozen soil

The definition of frozen soil is any type of soil or rock whose temperature is below the freezing point of water [51]. Frozen soil is recognized as a natural multi-component system comprised of mineral (and occasionally organic) solid particles, ice, unfrozen water, and gases of various chemical composition [52]. An ideal model of frozen soil may contain unfrozen water, solids, air, and ice in a total four-phase composition (Figure 2.21). In practice, a given frozen soil can be identified by its geotechnical characteristics, including

parameters such as temperature (θ), porosity (n), void ratio (e), total water content (w), ice content (w_i), unfrozen water content (w_u), density (ρ), specific gravity (G_s), and so on. The ASTM has issued two more detailed standards (ASTM-D7099-04, ASTM-D4083-89, and so on.) for a thorough description of frozen soil.

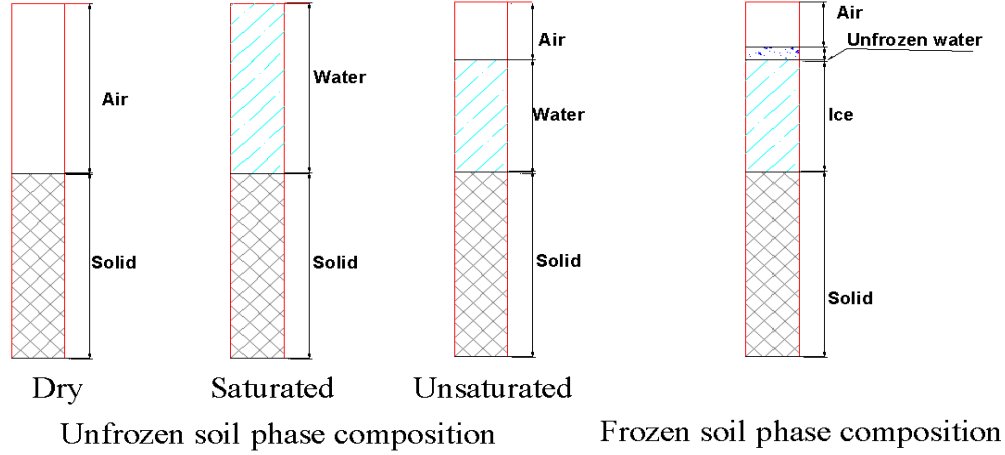


Figure 2.21: Schematic diagrams of the phase compositions of unfrozen and frozen soils (Source: Han, 2011).

2.6.2 Unfrozen water content in frozen soil

There is no doubt that unfrozen water can significantly affect frozen soil's behaviour in various ways. For example, the physical (thermal and hydraulic) and mechanical (strength and strain behaviour) characteristics will all change with different amounts of unfrozen water existing in a given soil. Knowledge of the influence of unfrozen water on frozen soil enables people to predict its behaviour and thus not only be able to avoid unnecessary engineering disasters, but also to facilitate the cost-effectiveness of projects.

Burt and Williams [53] stated in their research that water flow in a soil presents a linear relationship in response to hydraulic gradient. However, in addition to a pressure-induced hydraulic gradient such as gravity, and soil type, the movement of water is also subject to another force caused by different temperatures of unfrozen water content in a frozen soil. This newly-created gradient provides motivation for water to flow towards colder

regions of a soil. The aforementioned phenomenon is also known as frost heave, which is a big issue in many engineering projects such as highway construction, slope stabilization, and pipeline design. Hivon and Sego [54] discovered when investigating the correlation between unfrozen water content and strength that a reduction in strength was exhibited as the unfrozen water content rose.

Speculation on the causes of unfrozen water in frozen soil occurred throughout most of the last century. Temperature was first believed to be responsible for the existence of unfrozen water in frozen soil. As shown in Figure 2.22, despite of the different soil types, unfrozen water content decreases drastically as the temperature drops below the freezing point. However, Bouyoucos [55] observed in his experiments that up to 40% of the total water within certain samples can remain unfrozen even when the temperature is as low as -78°C . Bourbonnais [56] [57] also discovered that that water in frozen clay did not freeze even when the temperature dropped as low as -110°C .

This seemingly unlikely phenomenon has been found in many cases, from ever-growing nanotechnology to the application of radiators fluid; it has occurred in every corner of our society. Numerous investigations have been carried out to attempt to unravel this mystery. Schofield [58] noticed in his experiment that as the water content of a sample decreases, the degree of suction necessary for water in contact with it to enter must increase. He later related this pressure deficiency or suction of the soil water to the freezing point depression, and discovered that there was a relationship between the suction of soil water and the freezing point.

Anderson and Morgenstern [43] classified water within soil into two categories: strongly bound water films that surround mineral particles and are held by strong intermolecular forces, and weakly-bounded water absorbed in the pores. The former category suppresses freezing even at very low temperatures; the latter can freeze easily [43]. More recently, new theories have interpreted this depression of freezing point into two mechanisms: one is the melting-point depression and the freezing point depression. The melting-pointing depression refers to the phenomenon where the melting point of a given material decreases along with its size reduction towards the atomic scale. In nano-scale materials or in a very

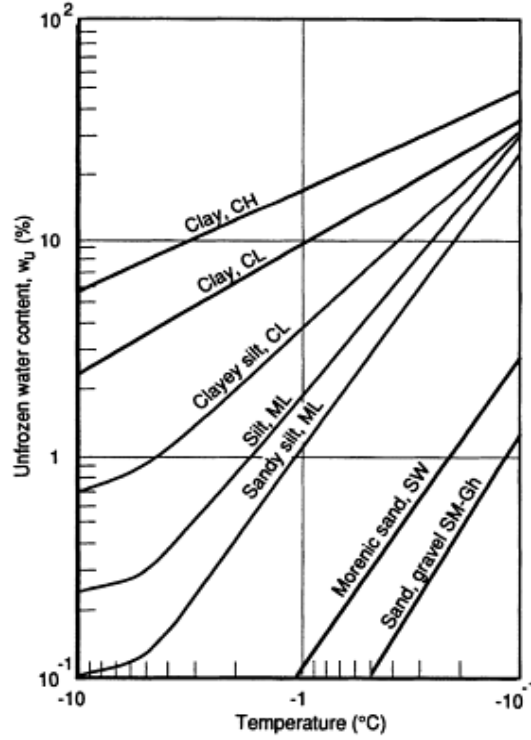


Figure 2.22: Typical unfrozen water content-temperature relationships for various soils (from Rowe, 2001).

fine and porous media, this phenomenon is rather common. Watanabe and Mizoguchi [59] attributed it to these materials tending to have huge surface-to-volume ratios which gives them a very different adsorption force and curvature than bulk materials; as a result, this changes their thermodynamic and thermal properties dramatically. Freezing-point depression, on the other hand, is a process in which the freezing point of a solvent is reduced after a solute or solutes are added. This process can be best explained by the theory of Gibb's free energy, combined with the Van't Hoff equation. In a water-ice closed system where the temperature is constantly controlled at the freezing point, both water and ice exist. In this situation, the tendencies of the ice becoming water and, in turn, changing back to ice reach an equilibrium, which means that both water and ice have the same amount of Gibb's free energy or the same chemical potential (see Equation 2.27).

$$\mu_{water} = \mu_{ice} \quad (2.17)$$

Where:

R is the gas law constant (8.314 J/mol K);

T is the temperature;

Q is the reaction quotient.

As we know that particles have a tendency to move from a higher to a lower chemical potential, in this case, ice particles will melt to water particles, which lowers the freezing point.

Approaches for determining the unfrozen water content of frozen soil vary. Dilatometry [60], adiabatic calorimetry [61], heat capacity, isothermal calorimetry [62], time domain reflectometry [63], differential thermal analysis, and the nuclear magnetic resonance technique [59] have all been developed in the last fifty years to accomplish this objective, and each one is able to provide a sufficiently accurate result.

2.6.3 Deformation behaviour of frozen soils

Designing any structure in a cold region requires a good knowledge of the strength and strain characteristics of frozen soil. It is commonly accepted that the most distinct geotechnical property of frozen soil is creep behaviour throughout the whole deformation process under a sustained loading. Consideration of the creep characteristics of frozen soil is important and necessary when constructing retaining walls in FCPB, as discussed in this thesis. Substantial effort has been made in the last few decades to attempt to determine the causes of creep. Many theories have been developed, but none can be universally applied to any situation that may be encountered. So far, most of the existing theories of creep boil down to two viewpoints: micro-mechanistic and macro-analytical [64]. The former focuses on the atomic level, aiming to identify the factors that control creep. Typical examples of

such theories were postulated by Mitchell et al. [65] and Andersland and Akili [66]. The second viewpoint, which involves a large amount of lab experiments, aims to extend the classic plasticity theory to account for temperature and time effects [64]. Representative examples include Vialov’s theory of creep of frozen soils [67] [68] and Odquist and Hult’s theory of creep in metals [50].

In most cases, the creep behaviour of frozen soil is believed to relate to the co-existence of ice and viscous unfrozen water in the frozen soil. Vialov and Tsytovich [69] demonstrated a pattern of creep in frozen soil when a constant load was added to a given frozen soil sample. This constant load increased the pressure between soil particles, as well as their interface between ice crystals, and thus gave rise to a pressure-melting phenomenon of ice. Water released from ice then created a gradient of water surface tension in the adjacent environment, which impelled unfrozen water to move towards cooler regions with lower stress. This water refroze and settled down at the new location until pressure was transmitted there, causing another pressure-melting phenomenon. Along with the process of ice melting, water moving, and water re-freezing is the breakdown and reconstruction of the matrix of ice and grains, the deformation of pore ice, and the readjustment of particles’ location. This series of time-dependent processes, which is known as ice creep, results in two outcomes: strain-hardening and strain-softening. The former occurs because the structural deformation serves to compact frozen soil, leading to an increasing number of firm contacts among soil grains, and therefore inducing a growth of internal friction between particles [70]. The latter takes place due to the weakening of the structural cohesion and the increasing amount of unfrozen water content in frozen soil [71]. All of these actions feature a highly time-dependent process and compensate for each other. For example, if the added load does not surpass the long-term strength of the given frozen soil, then the strain-softening process will be upset by the strain-hardening process, which means the deformation will decline with time. On the contrary, if the added loads surpass the long-term strength of the frozen soil, the collapse of internal bonds plays a more important role, and thus the deformation rate grows with time [71].

Many models have been developed to depict and predict the deformation behaviour of

frozen soil. The most widely acknowledged idea so far can be summarized as follows: the failure characteristics of frozen soil show both ductile and brittle traits under a compressive load [57] [72] [73]. In addition, the strain on frozen soil under a constant loading contains an instantaneous portion and a time-dependent portion; both of these portions contains a reversible and an irreversible part [57]. After discovering the similarities between the creep curves of frozen soil and metals from experimental results, Vialov and others [68] proposed a famous mechanical rheological models to describe the total strain of frozen soil. However, it does not take into account the initial plastic deformation or third stage of creep (visco-plastic flow). Based on this model, Hivon [74] came up with a modified version (see Equation 2.28):

$$\epsilon = \epsilon_0 + \epsilon_1(t) + \epsilon_2(t) + \epsilon_3(t) \quad (2.18)$$

where:

ϵ = total strain;

ϵ_0 = instantaneous elastic strain represented by a spring;

ϵ_1 = visco-elastic strain represented by a spring in parallel with a dashpot;

ϵ_2 = visco-plastic strain represented by a braking element in series with a dashpot;

ϵ_3 = failure strain.

Figures 2.23 and 2.24 show a modified version of Vialov's rheological model and its corresponding creep curve under a constant load with time. It was expanded to include instantaneous plastic and visco-plastic deformation. As shown in Figure 2.23, different elements of the model are tagged to imitate different characteristics of the frozen soil and, in turn, to represent the appropriate sections of the resulting creep curve. Element ① is a spring, which represents an elastic deformation; element ② is an air gap with an uncertain span. Its deformation is considered plastic; element ③ is a Kelvin-Voigt element whose function is similar to a spring-dashpot. It characterizes a visco-elastic deformation.

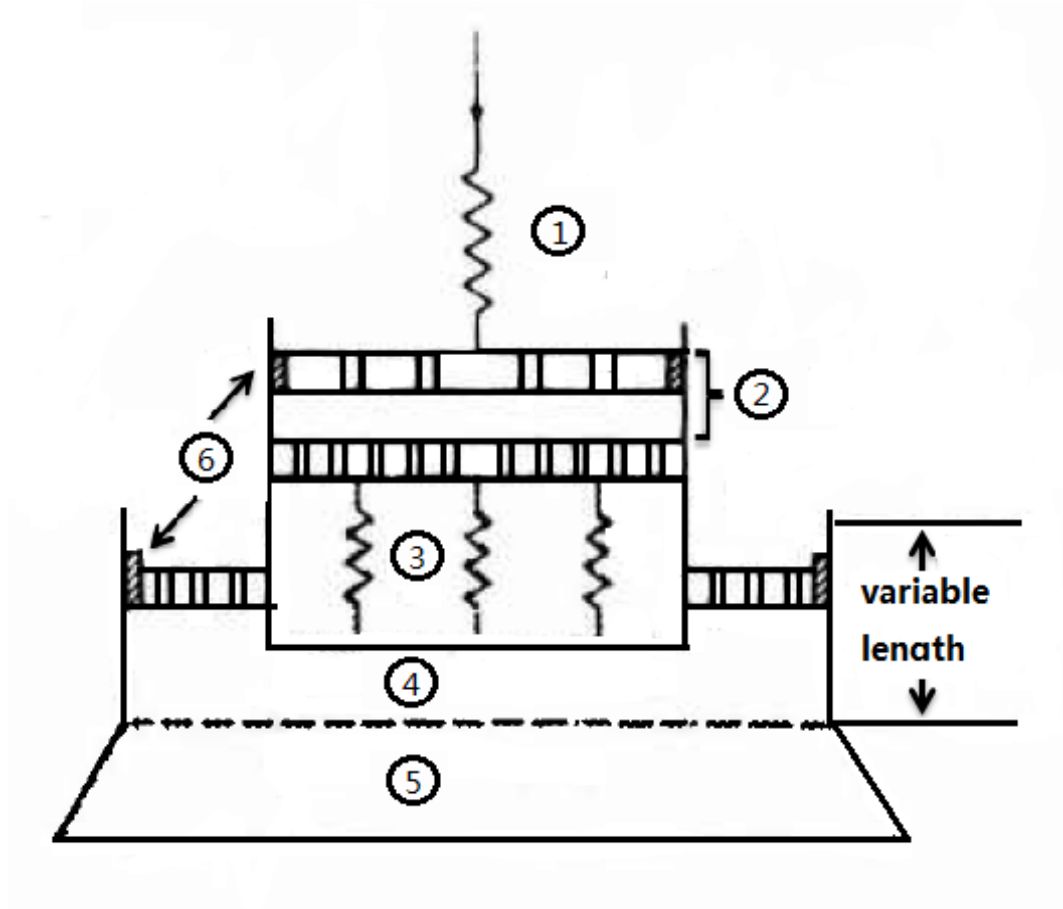


Figure 2.23: A modified rheological model of frozen soil under a constant load (after Sayles, 1968).

Element ④ is a viscous dashpot representing the viscous portion of the resulting curve; element ⑤ is a visco-plastic dashpot, representing the plastic deformation at the very late strains; element ⑥ is a blocking device, representing the initial plastic deformation. The mechanism of this model can be described as follows: the initial plastic deformation is considered to be a displacement taking place as a result of frictional slide. An air gap exists between the blocking device (slide) and Kelvin-Voigt elements, which has a finite value up to zero. These two characteristics, frictional slide and air gap, together guarantee that the initial plastic deformation mentioned above occurs before viscoelastic movement begins. To activate the friction element (blocking device) to slide, a certain magnitude of force is necessary. If the load applied to the frozen soil exceeds its initial yield point,

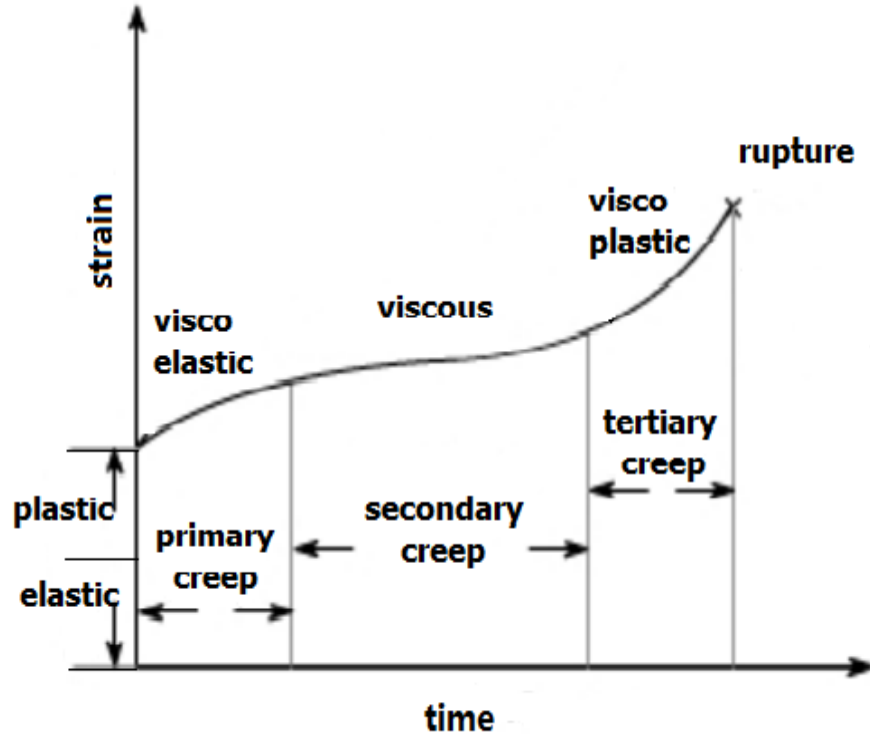


Figure 2.24: The resulting creep curve for the modified rheological model of frozen soil by Sayles (after Sayles, 1968).

the slide will move downwards, and thus reduce or close the gap. As a consequence, an instantaneous plastic deformation will happen, which cannot be recovered when the load is removed. Sometimes, although the applied load does not reach the plastic limit of the frozen soil, deformation still takes place. In this case, it is a sheer elastic deformation of the Kelvin-Voigt element which contributes a postponed elastic effect. Stage II (the viscous portion of the curve) with a variable length is due to the presence of element 4. As the material approaches failure, an increasing rate of visco-plastic flow will be observed [71].

2.6.4 Strength of frozen soils

For any engineering designs related to foundation construction in cold regions, the strength of frozen soil is the most important property, and is generally determined by its compressive

strength. Factors that govern this mechanical property are classified into two categories: external and internal. External factors, including temperature and loading rate, dominate the overall compressive strength of frozen soil. Internal factors, such as soil type and structure, mineral composition, water content, dry density, and so on, while not having a decisive influence on the compressive strength, still affect it to a certain degree and cannot be ignored.

The internal factors alter the strength of frozen soils by changing their internal friction and cohesion. According to Vialov and Tsytoich [69], the cohesion component in frozen soils depends on (1) the molecular forces of attraction between soil grains; (2) the physical or chemical cementing effects of particles; and (3) the cementing effect of pore ice formation on soil particles. The cementing effect by pore ice is often considered to come from the bonds between the ice crystals and the soil particles, despite the fact that there is actually a film of unfrozen water surrounding soil particles. The reason for this is because this unfrozen water film complies with the molecular forces of the soil particles to a great degree, so it is safe to assume it is fully capable of transmitting both normal and shear forces between ice crystals and soil grains. The degree of ice cohesion lies in the amount and strength (temperature) of the ice, and the way the ice contacts the soil particles. The internal friction on the other hand depends on a number of soil characteristics including soil grains's sizes, distribution, shape, arrangement, and so on. One thing that must be mentioned is that ice cohesion is the most dominant strength factor in frozen soil after internal friction.

The effect of water content and dry unit weight

Shusherina and Bobkov [75] conducted a series of UCS experiments studying the influence of total water content on the peak strength of frozen silty sand, clay-like silt, and clay, at temperatures ranging from -10°C to -55°C . According to the results from samples with a total water content ranging from 15% to 125%, Shusherina and Bobkov suggested [75] that frozen soils with very low total water content tend to exhibit a higher strength than that of polycrystalline ice. However, as total water content increases the strength of frozen

soil reduces and eventually drops below that of ice. In contrast, at very high levels of total water content, the strength of frozen soil increases correspondingly, until it reaches the strength of ice. The reason behind these two different tendencies is that when it comes to low water content, the adhesion (internal friction and cohesion) of soil particles dominates the strength of frozen soil. Yet in soils with high water content, the ice matrix plays a more crucial role [73].

Sayles and Carbee [76] introduced ice content and dry unit weight when studying the yield stress of frozen silts. Within a water content range from 28% to 54%, they observed that the yield stress increased with growing ice content and declining dry unit weight. They later explained this discovery by attributing the rise to the increasing cohesion of the ice matrix, coupled with the frictional resistance of soil grains and fractured ice crystals.

The effect of fine content

Neuber and Wotlers [77] concluded, after conducting a number of triaxial compression tests involving six different types of soil with varying proportions of fine contents, that the compressive strength of frozen soil reduces as fine grain content increases. This is in keeping with early findings by Tsytoich and Sumgin [78]. Sayles and Haines [79] also suggest that the larger grain size and bulky shape of particles would lead to a higher frictional resistance, and thus a higher compressive strength.

The effect of temperature and strain rate

Bragg and Andersland [80] claimed that a low temperature and high strain rate facilitate a high initial yield stress (y), peak strength, and initial tangent modulus of frozen soils. They found that when the strain rate is less than 10^{-5}s^{-1} , an apparent region of a plastic strain-hardening curve will appear without any visible cracking nor shear planes. Yet, once subjected to a strain rate higher than $4 \times 10^{-4}\text{s}^{-1}$, the sample exhibits a linear relationship between strain and stress, followed by brittle failure.

He et al. [81] proposed a constitutive model incorporating temperature and strain rate

to predict the behaviour of saturated frozen silt. Different combinations of temperatures (-2°C , -5°C , -10°C , and -15°C) and strain rates ($1.1 \times 10^6 \text{s}^{-1}$, $9.3 \times 10^{-6} \text{s}^{-1}$, and $7.1 \times 10^{-4} \text{s}^{-1}$) were used in the uniaxial compression test for this investigation. The final equation is given as Equation 2.28 below.

$$\sigma = 1.123 \epsilon^{0.089} \left(\frac{\theta}{\theta_0} \right)^{0.0887} \left(\frac{\epsilon}{\epsilon_y} \right)^{0.524 \epsilon^{0.055} \left(\frac{\theta}{\theta_0} \right)^{-0.387}} \quad (2.19)$$

Where:

σ = the stress, MPa;

ϵ = strain rate, s^{-1} ;

θ = the negative temperature of the frozen soil, $^{\circ}\text{C}$;

θ_0 = a reference temperature, -1°C ;

ϵ = strain;

ϵ_y = yield strain, independent to the strain rate.

The effect of confining pressure

When submitted to a load, frozen soils can develop several types of stress-strain curves. The determining factor of this has something to do with the phenomenon is known as confining pressure. It has been found that the strength of frozen soil increases as confining pressure goes up, until it reaches a peak value, and starts to decline [82] [83]. Indeed, a high level of confining pressure can not only increase the friction between soil particles, facilitating high strength and low deformation, but can also lead to the decline of cohesion between ice interfaces and grains, as well as an increasing amount of unfrozen water. The latter factors will undoubtedly compromise the strength of frozen soils [81]. Zhang et al. [84] divided the stress-strain behaviour of frozen soil, caused by applied confining pressure, into five types (see Figure 2.25): a) strong strain-hardening, b) weak strain-hardening, c) strong

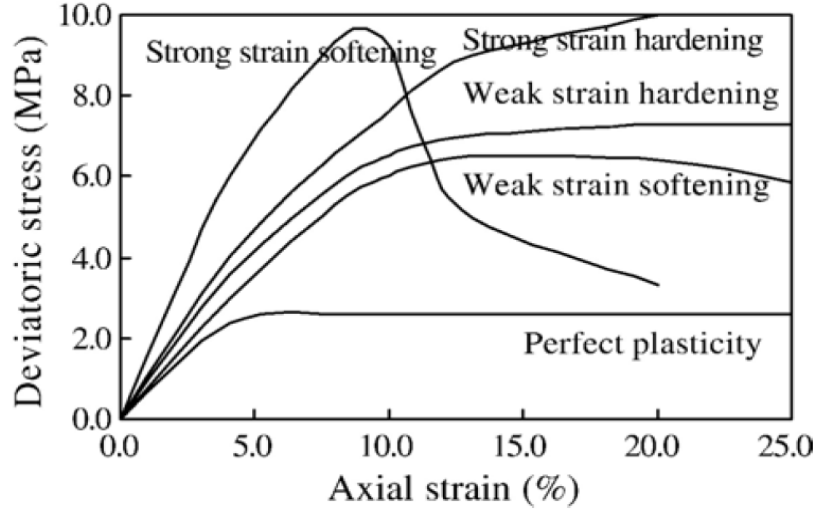


Figure 2.25: Schematic stress-strain curves for frozen soils (Source: Zhang et al., 2006).

strain-softening, d) weak strain-softening, and e) perfect plasticity. They further specified these with regards to different soil types:

- (1) Sandy soil exhibits a strong strain-softening type under a confining pressure less than around 0.6 MPa ($\sigma_3 < 0.6$). However, when increasing the confining pressure to a range of 0.6-3.0 MPa ($0.6 < \sigma_3 < 3.0$), it switches to a weak strain-hardening mode. Above this level, frozen soil shows strong strain-hardening behaviour.
- (2) For silt clay, the stress-strain behaviour starts as a strong strain-softening type when subjected to a confining pressure lower than 0.3 MPa, but will grade into a weak strain-softening pattern and then a strain-hardening pattern when the confining pressure rises up to 0.3 MPa $< \sigma_3 < 1.0$ MPa and $\sigma_3 < 1.0$ MPa, respectively.

Effect of internal confinement on the compression strength of frozen soil

In addition to the confining pressure added outside the frozen soil, the mobility of soil grains is also constricted by the internal confinement generated during deformation. Landanyi and Morel [85] suggest that any dense granular material with a viscoelastic matrix has the

ability to result in extra strength when submitted to a compression. This extra strength is due to the internal confinement produced by tensile stresses when the ice matrix opposes the dilation. Ting et al. [86] specifically mentioned in one paper that the strength of frozen soil consists of (1) pore ice strength, (2) soil strength, (3) increase in the effective stress due to the adhesive ice bonds resisting dilation, and (4) synergistic strengthening effects between the soil and ice matrix. Ting et al. [86] also proposed a mechanism map (Figure 2.26) to illustrate these four components.

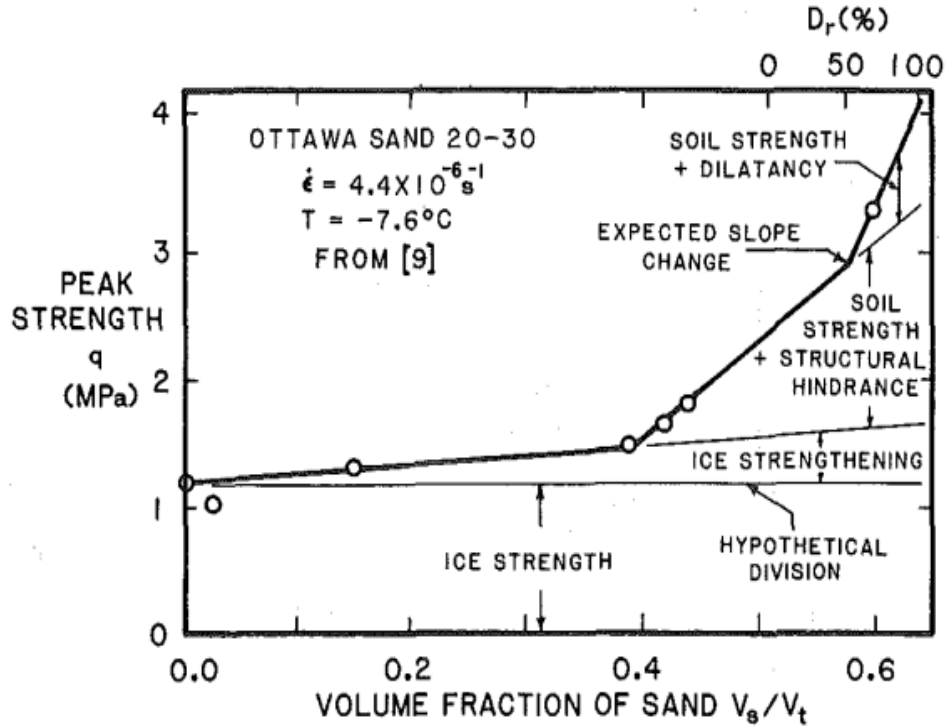


Figure 2.26: Failure mechanism map for unconfined compressive strength of frozen Ottawa sand at $T = -7^\circ\text{C}$ and at an axial strain rate of $4.4 \times 10^{-4} \text{ s}^{-1}$ (after Goughnour and Anderland 1968; Ting et al., 1983).

It is clear from this map that as the fraction of soil grains increases, the soil's peak strength grows. After the soil fraction reaches about 60%, a dilatancy effect occurs. Sayles [79] suggested that this dilatancy-hardening effect only takes place in a strain range of 1-2%, after which pore ice would start to behave in a brittle manner.

2.7 Review of frozen saline soil

A review of frozen saline soil is presented in this section for a thorough comprehension of FCPB behaviour when subjected to a saline-rich environment. Wilson and Vinson [87] explained the effect of salts on the formation of ice. Ice formation is initiated at a relatively lower temperature in a saline solution than in a pure water solution, due to the freezing point depression. As this process goes on, more and more unfrozen water turns into ice and the concentration of salts goes up, resulting in a further depression of the freezing point that makes it more difficult for water to freeze. Eventually, depending on the salinity and ambient temperature, part of the saline water will get frozen, but the rest of it would remain unfrozen; some salts may also be excluded because of the low solubility. Sheeran and Yong [88] proposed a sketch map of saline frozen soil (Figure 2.27) to illustrate its structure and unfrozen water distribution. It is easy to see from this map that soil particles, pore ice, high salinity brine, brine inclusion, and adsorbed water co-exist in a saline frozen soil system.

2.7.1 Strength of frozen saline soil

Mellor [85] reported that the factors governing the mechanical behaviour of saline frozen soil are the same as those governing non-saline frozen soil: temperature, strain rate, grain size, structure, bulk density, and unfrozen water content. However, the unfrozen water content or brine volume, as well as the shape of the brine columns, has a much bigger impact on the soil's strength owing to their relatively larger quantity. According to various research [89] [90], the unfrozen water content increases with rising temperature and salinity, resulting in a decrease in strength. Figure 2.28 shows the relationships between unfrozen water content and temperature of samples of different salinity.

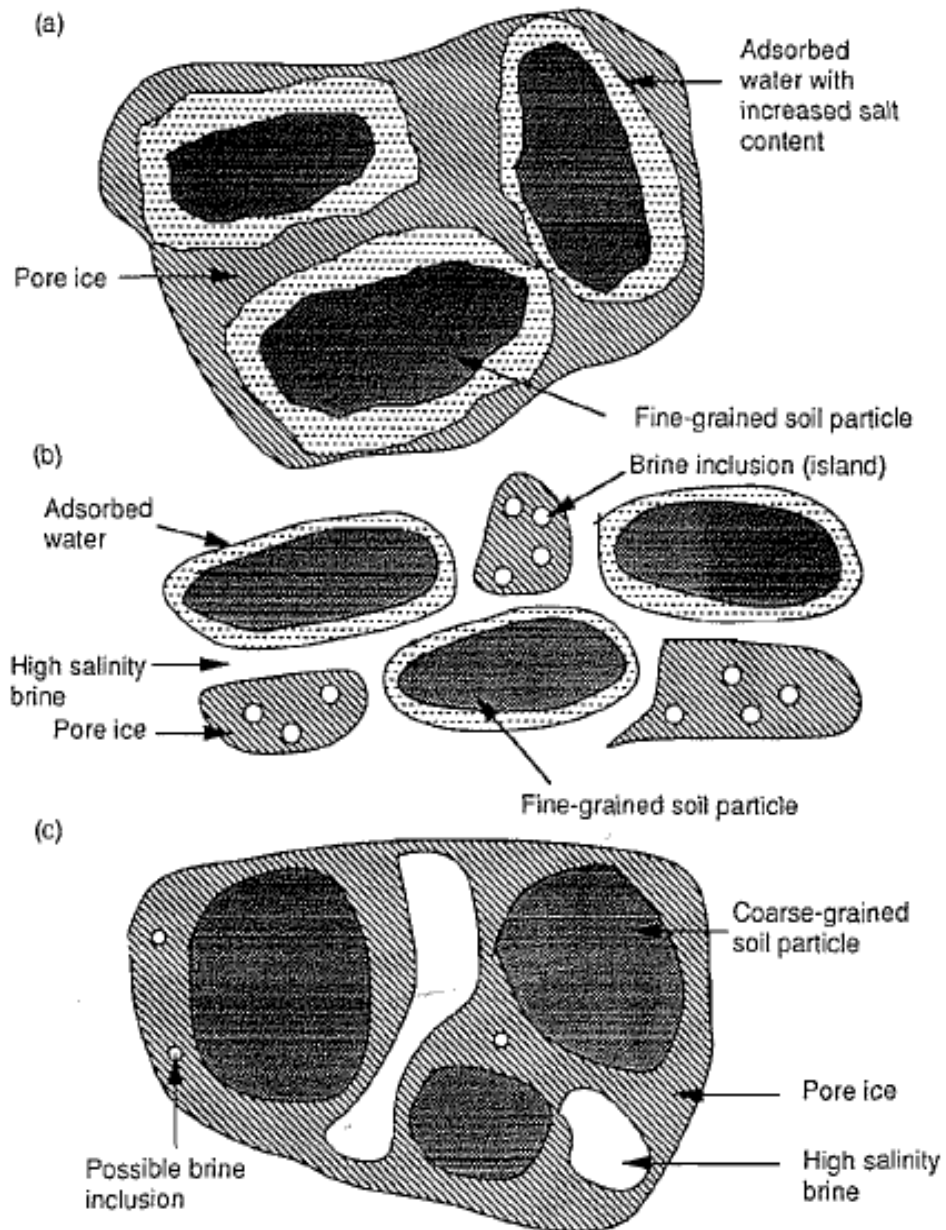


Figure 2.27: Sketch map of a frozen saline soil system and unfrozen water distribution (after Sheeran and Yong, 1975).

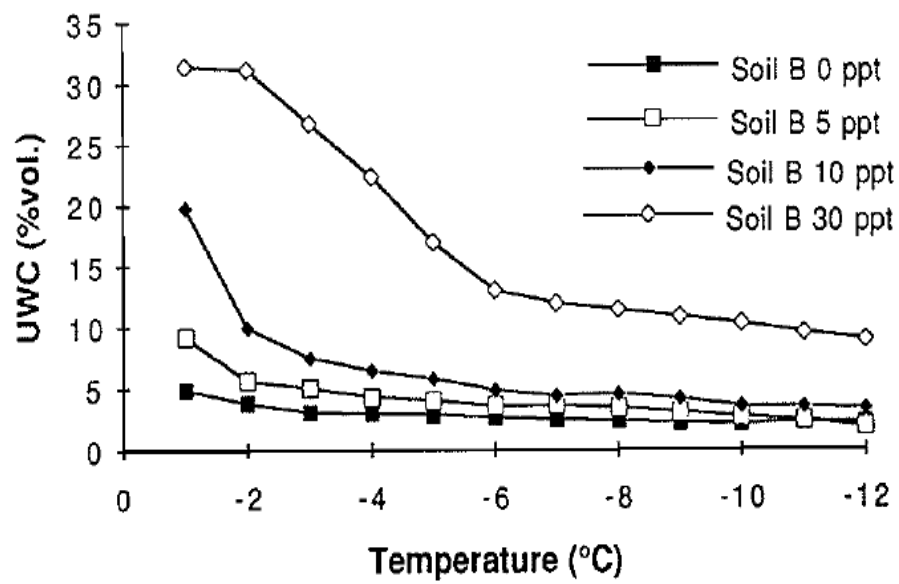


Figure 2.28: Volumetric unfrozen water content (UWC) and distribution of fine silty sand (after Hivon and Sego, 1995).

Chapter 3

Strength and Deformation Behavior of Frozen Cemented Paste Backfill

3.1 Introduction

Ever since it was introduced by German companies in the 1970s, cemented paste backfill (CPB) has been gaining greater recognition over the last few decades. It is considered not only a rising solution to mine waste management, but also a cost-effective construction material [91] [92] [93] [94]. Its economic and environmental benefits altogether can facilitate and promote a success for operation in the near future.

Fresh CPB requires a slump characteristic generally between 150 mm to 250 mm, and a solid concentration around 70-85% by weight in order to have the necessary workability and technical consistency [93]. However, once settled underground, CPB is expected to achieve a sufficient strength as soon as possible, so that it would be able to withstand the pressure resulting from later backfilling as the operation continues. A desirable strength is estimated to be in a range from 0.7 to 2.0 MPa [92] [95] [96]. Other than that, parameters such as stiffness and stress-strain behavior are also big concerns in CPB designs [8].

Extensive laboratory research has been carried out in recent years to investigate the strength development and deformation behavior of CPB, with immense success [8] [10] [93].

Nevertheless, most of those studies have been focused on room or higher temperature, research related to sub-zero environment is far from complete. Given the fact that many companies have launched plans for exploiting mines in Arctic areas due to the increasing demand for mine products, such knowledge of CPB at the temperature below freezing is beneficial for achieving better performance.

Towards this end, the objectives of this chapter are:

- Acquiring an overall understanding of the strength development of frozen CPB (FCPB);
- Studying the deformation behavior (stress-strain behavior) of FCPB;
- And discovering the influential factors and optimal mixing portions for binders of FCPB.

3.2 Experimental Program

3.2.1 Materials used, sample preparation and curing

3.2.1.1 Materials used

Materials that were chosen for this study include tailings (silica tailings), water, and binders (Portland cement I, slag, and fly ash).

Tailings

Silica tailings (SILT-CO-SIL) were used to prepare all samples for this study. They are commercially available, and share a great deal of resemblance with natural tailings from nine mine sites in eastern Canada [93]. Derived directly from ground silica, these tailings contain as high as 99.8% SiO₂ (quartz). One benefit of using silica tailings rather than natural tailings for this research is that Silica tailings are able to eliminate potential interference caused by chemicals found inherently in natural tailings. For instance, many

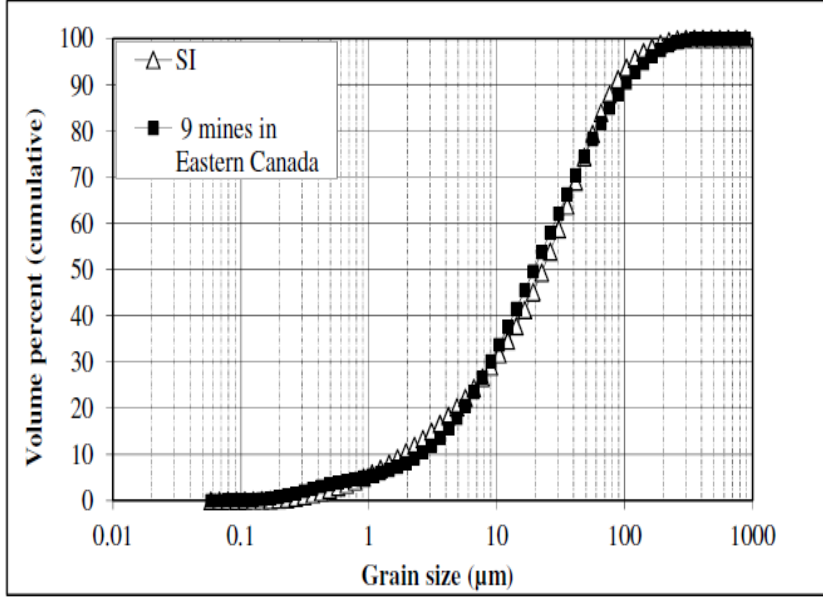


Figure 3.1: Grain size distribution curves for tailings used and the average of natural tailings from 9 mines in eastern Canada (Fall and Samb, 2006).

natural tailings contain sulphate, which is known for its huge impact on cement hydration. By contrast, silica tailings can provide an accurate control over the mineralogy and chemical compositions of the CPBs. Figure 3.1 shows the average grain-size distribution of silica tailings in comparison to that of natural tailings from nine Canadian mines. In this picture, both curves present a fine portion (particles with diameters smaller than 20 μm) around 45%, which can be classified as medium tailings. In addition, by studying the index properties (e.g., liquid and plastic limits) of silica tailings, Fall [97] indicates that silica tailings fit the standards of non-plastic sandy silts. Tables 3.1 and 3.2 give a more direct information about the physical properties and main chemical elements of silica tailings.

Table 3.1: Physical properties of the tailings (SI).

Element Unit	Gs -	D ₁₀ μm	D ₃₀ μm	D ₅₀ μm	D ₆₀ μm	D ₉₀ μm	Cu -	Cc -
	2.7	1.9	9.0	22.5	31.5	88.9	16.2	1.3

Table 3.2: Chemical properties of the tailings by percentage (Source: US Silica Company).

	SiO ₂	FeO ₃	Al ₂ O ₃	TiO ₂	CaO	MgO	Na ₂ O	K ₂ O	LOI	pH
ST	99.8	0.035	0.05	0.02	0.01	<0.01	<0.01	0.02	0.1	7

Binders

Three types of binders of different composition were used in this study: PCI (Portland cement type I), PCI/Slag (Portland cement type I mixed with blast furnace slag (Slag) in a proportion of 50/50), and PCI/FA (Portland cement type I mixed with fly ash in a proportion of 50/50). The main composition and properties of PCI and Slag are outlined in Table 3.3.

Table 3.3: Chemical and physical properties of the binders PCI and Slag used.

Binder	MgO	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SiO ₃	Relative Density	Specific Surface (m ² /g)
PCI	2.65	68.82	18.03	4.53	2.7	3.82	3.1	1.3
Slag	10.98	41.14	34.23	9.54	—	3.87	3.3	2.2

Mixing water

Tap water was used in mixing binders and tailings.

3.2.1.2 Sample preparation

The process of making one batch of specimens of the same composition usually took about half an hour. Tailings materials, binders and tap water were properly weighed and mixed together using a mixer until a homogenous paste resulted. This mixing procedure usually lasted seven minutes. It is worth noting that, because the setting time of cement is rather prompt, tailings and binder were blended in advance to achieve a uniform powder before adding any water. Following the guidelines from previous research, trial slump tests were performed beforehand using the slump test cone [10] in accordance with ASTM C-143, maintaining all specimens a consistent slump of 18 cm.

After the desired paste consistency was achieved, the paste was poured manually into curing cylinders, which were 5 cm in diameter and 10 cm in height. With the filling of cylinders, the process would be separated into three steps, and by the end of each filling the sample would be gently tapped on the counter, to ensure that no air bubbles were trapped within. Usually, performing this process should not require more than 7 minutes [8]. The samples were then sealed with impervious tape to avoid evaporation of the water.

In addition to backfill samples, cement pastes (with $W/C = 1$ to simulate the cement matrix of CPB) were also prepared and cured in the same conditions as the backfill samples.

3.2.1.3 Curing samples

A custom-made, walk-in freezer was used for the curing process for all specimens that were cured in sub-zero environments. The temperature and humidity within the freezer were precisely controlled by an Omega iSeries Humidity and Temperature Controller (CNiTH and DPiTH Series), with $\pm 0.5^{\circ}\text{C}$ and $\pm 2\%$ RH. The curing temperature was set at -6°C , with a relative humidity of 67%. Freezing-time periods were 7, 28, and 90 days, respectively.

3.2.2 Testing program

FCPB specimens with different binder types and contents were prepared at a temperature of -6°C , and later were tested for uniaxial compression strength (UCS). Curing times were set at 7, 28, and 90 days in order to obtain a thorough strength development of FCPB. The temperature within the curing samples was monitored for up to 90 days to collect data about the evolution of the temperature. The information can be useful in interpreting ice formation, cement hydration, and strength development. Thermal gravimetric analyses (TGA), coupled with mercury intrusion porosimetry (MIP), and X-ray diffraction analysis (XRD) have also been applied to provide supporting data for this study. Additional experiments, including specimens with different water content, were conducted as supplements.

3.2.2.1 Uniaxial compressive strength test

The strength of a material can be interpreted broadly as its ability to resist imposed forces, thus indicating the maximum stress the material can sustain under specified loading and boundary conditions [98]. In a case of frozen soil or FCPB, the uniaxial compressive strength (UCS) test is widely adopted as a main approach for evaluating the strength of a given specimen. It is conducted by compressing a right circular cylinder of the material between two platens of a testing machine. The maximum load necessary to crush the specimen is defined as the peak uniaxial compressive strength [98] (see Figure 3.2). In this chapter, UCS of each sample was evaluated by laboratory tests.

The UCS tests were conducted in accordance with ASTM D7300-06, and repeated at least four times for each individual type of sample, to ensure valid values. The UCS machine was modified with an isolated box mounted onto the platen to create a closed chamber for the testing specimens. The isolated box itself was made from an 80×80 cm refrigerator, so that, once connected to the power, it had the ability to regulate the temperature down to 0°C . Pre-frozen ice cubes from another freezer were introduced to help bring temperature down further to -6°C . Internal temperature was monitored by a CAT II 600V multimeter to ensure favorable experimental conditions. Before each specimen was

tested, plastic plates used for adjustment and isolation were frozen and transported in a food cooler, along with the specimen.

The deformation rate was set at 0.15 mm/min. Applied load, together with deformation information, were automatically logged by LabVIEW and stored in the computer. A stress-strain curve was developed and presented as the test proceeded. Peak strength of each sample was recorded as objective strength for this study. If no peak strength appeared due to the strain-hardening process, objective strength corresponding to a strain of 15% was selected as the peak strength of the FCPB.

FCPB specimens with various binder contents (0%, 3%, 4.5%, and 6%) correspond with water-to-cement ratio of 0, 11.1, 7.6, and 5.9 respectively, and frozen paste backfill (FPB) specimens with various water content (5%, 10%, 20%, 30%, and 50%) were selected as studying subjects. Slumps for each paste were maintained at 18 cm, which is the most popular value in practice. Specimens were trimmed evenly on the upper end (to get rid of volumetric dilation due to ice formation), and then were extracted out by air pump one day before completion of the curing. This was done to limit temperature interference. The obtained specimens were then sealed in zip-locks bags (to avoid evaporation) and sent back to the curing freezer for one more day of curing. The entire operation lasted no more than two minutes, and the specimens remained intact and unharmed. More information about the mix composition for each sample can be seen in Table 3.4 and Table 3.5.

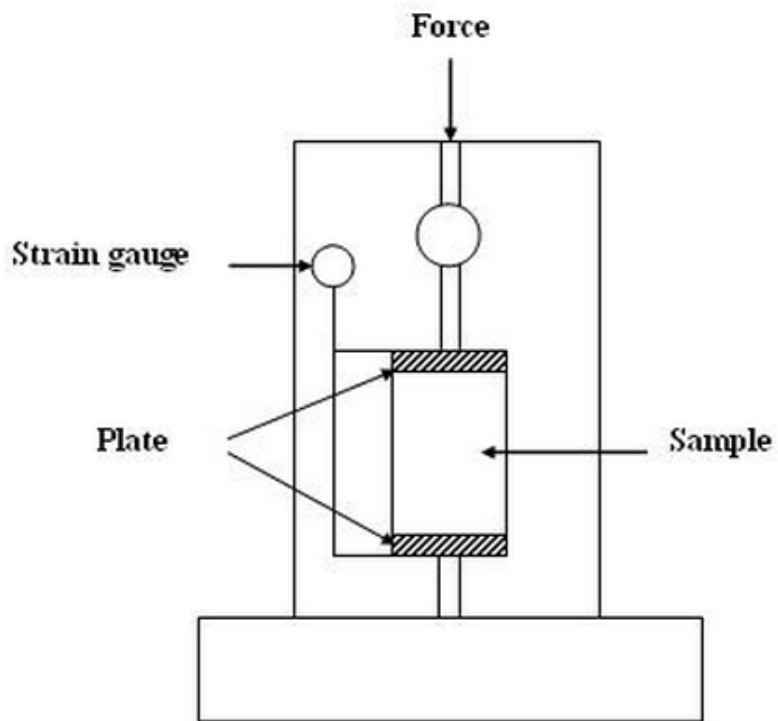


Figure 3.2: Schematic configuration of an uniaxial compressive strength testing machine.

Table 3.4: Mix design for the study of strength development of FCPB.

Sample name	Binder content	Binder combination	Blending ratio	W/C ratio	Curing age (days)
PCI-3.0-7	3%	PCI	100	11.1	7
PCI-4.5-7	4.5%	PCI	100	7.6	7
PCI-6.0-7	6.0%	PCI	100	5.9	7
SPCI-4.5-7	4.5%	PCI/Slag	100	7.6	7
FPCI-4.5-7	4.5%	PCI/FA	100	7.6	7
FPB-0-7	0	/	/	/	7
PCI-3.0-28	3%	PCI	100	11.1	28
PCI-4.5-28	4.5%	PCI	100	7.6	28
PCI-6.0-28	6.0%	PCI	100	5.9	28
SPCI-4.5-28	4.5%	PCI/Slag	100	7.6	28
FPCI-4.5-28	4.5%	PCI/FA	100	7.6	28
FPB-0-28	0	/	/	/	28
PCI-3.0-90	3%	PCI	100	11.1	90
PCI-4.5-90	4.5%	PCI	100	7.6	90
PCI-6.0-90	6.0%	PCI	100	5.9	90
SPCI-4.5-90	4.5%	PCI/Slag	100	7.6	90
FPCI-4.5-90	4.5%	PCI/FA	100	7.6	90
FPB-0-90	0	/	/	/	90

Table 3.5: Mix design for the study of Strength development of frozen paste backfill (FPB).

Sample name	Water content (%)	Binder combination	Blending ratio	W/C ratio	Curing age (days)
FPB-5-7	5	/	/	/	7
FPB-10-7	10	/	/	/	7
FPB-20-7	20	/	/	/	7
FPB-30-7	30	/	/	/	7
FPB-50-7	50	/	/	/	7
FPB-0-28	26	/	/	/	28
FPB-5-28	5	/	/	/	28
FPB-10-28	10	/	/	/	28
FPB-20-28	20	/	/	/	28
FPB-30-28	30	/	/	/	28
FPB-50-28	50	/	/	/	28

3.2.2.2 Mercury intrusion porosimetry (MIP) test

Of all the engineering properties of cement and concrete, or cementitious materials (e.g., cemented backfill), the governing factors involved with durability and strength are related in some way to the porosity or pore structures [99]. Many studies [99] [100] indicate that the characterization of the pore system is often a better predictor of durability than other parameters. Factors that influence microstructure and pore characteristics not only involve mix design, but also curing conditions. For this reason, mercury intrusion porosimetry (MIP) tests were conducted to further assess the strength and durability of FCPB. The word “porosimetry” refers to an analytical technique used to determine various quantifiable aspects of a material’s porous nature, such as pore diameter, total pore volume, surface area, and bulk and absolute densities [101]. A micromeritics AutoPore III 9420 mercury porosimeter was used to perform this task. Specimens with different binder types (PCI, PCI/Slag, PCI/FA), different curing times (7 days and 90 days), and different tailing types (Ground Sicilia Tailings and Natural Zinc Tailings) were prepared for the study. After 7 to 90 days of curing in the walk-in freezer, specimens were taken out of their curing tubes, put into a 50°C oven, and dried for five days. Drying at this temperature can largely prevent the formation of cracks [8]. Once the drying procedure was finished, each specimen was trimmed (e.g., cutting off both ends of the sample) to avoid the interruptions from frost-heaving phenomena and disturbances caused by the settling of solid grains, and to ensure maximum homogeneity. More details may be seen in Table 3.6.

Table 3.6: Mix design for the study of porosity of FCPB.

Sample name	Binder content	Binder combination	Blending ratio	W/C ratio	Curing age (days)
PCI-4.5-7	4.5%	PCI	100	7.6	7
SPCI-4.5-7	4.5%	PCI/SG	50/50	7.6	7
FPCI-4.5-7	4.5%	PCI/FA	50/50	7.6	7
PCI-4.5-7	4.5%	PCI	100	7.6	90
SPCI-4.5-7	4.5%	PCI/SG	50/50	7.6	90
FPCI-4.5-7	4.5%	PCI/FA	50/50	7.6	90

3.2.2.3 Thermogravimetric analysis (TGA)

Thermogravimetric Analysis (TGA) is a method of thermal analysis in which the amount of weight change, either as a function of increasing temperature or as a function of time [102]. TGA can provide valuable information about materials, including vaporization, sublimation, absorption, and desolvation, especially dehydration. It is especially useful when it comes to the determination of selected characteristics of materials that exhibit either mass loss or gain due to decomposition, oxidation, or loss of volatiles (such as moisture). Taking into account the factors above, TGA was performed in this study to determine the degree of cement hydration for each sample through the analysis of characteristic decomposition patterns. Up to eight different types of specimen were prepared with different curing conditions and curing times. Once specimens were prepared, they were put in the walk-in freezer with a constant temperature of -6°C for a given period of time, and then were taken out and dried in the oven for five days at a temperature of 50°C . After the curing process, samples were ground into powders and filtered by screens. The size of the screen was a Canadian Standard sieve series NO.100 ($150\text{ }\mu\text{m}$, or 0.009 inches). The thermal analyses were carried out by using a simultaneous thermogravimetric analyzer and differential scanning calorimeter (SDT) from TA Instruments, available at the University of Ottawa. Testing conditions were set up as; 1) atmosphere: Nitrogen; 2) desired heating

ramp: 10°C/min; 3) temperature range: from room temperature to 1000°C. More detail is given in Table 3.7.

Table 3.7: Mix design for the preparation of TGA samples.

Sample name	Binder combination	Blending ratio	W/C ratio	Curing age (days)
DG-PCI-7	PCI	100	2	7
DG-SG-7	PCI/SG	100	2	7
DG-FA-7	PCI/FA	100	2	7
DG-20-7	PCI	100	2	7
DG-PCI-7	PCI	100	2	90
DG-SG-7	PCI/SG	100	2	90
DG-FA-7	PCI/FA	100	2	90
DG-20-7	PCI	100	2	90

3.2.2.4 Temperature monitoring

In his study, Han [103] discovered a slight drop in the strength of FCPB samples at advanced age (>90 days), and attributed to the heat produced during cement hydration. Yet, because of the limitations on the experiments, he could not provide any convincing evidence to support this hypothesis. In this study, temperature monitoring of a series of FCPB samples was performed to verify his hypothesis or argument. Table 3.8 shows the samples that had been monitored during curing (up to 90 days). A CAT II 600V multi-meter was chosen to monitor the temperature changes. The frequency of monitoring was once every three hours during three days, then once a day for the remaining 87 days.

Table 3.8: Types of FCPB samples being monitored for temperature changes.

Sample name	Binder content	Binder combination	Blending ratio	W/C ratio
TPCI-3.0	3.0%	PCI	100	11.1
TPCI-4.5	4.5%	PCI	100	7.6
TPCI-6.0	6.0%	PCI	100	5.9
TSPCI-4.5	4.5%	PCI/Slag	50/50	7.6
TFPCI-4.5	4.5%	PCI/FA	50/50	7.6
TFPB	0%	/	/	/
PCI-20	4.5%	PCI	100	7.6

3.3 Results and Discussion

3.3.1 Uniaxial compressive strength of frozen soil

In this study, the peak compressive strength σ_m of a specimen is defined by the maximum stress of the sample during the compression; if the stress continuously increases with the strain, the corresponding strength at a strain rate of 15% was selected as an alternative peak strength. The derived strength developments of FCPB samples are summarized in Figure 3.3.

Putting aside all other details, FCPB specimens in general show rather desirable (higher) strength compared to CPB. CPB samples, which cured at room temperature (20°C), reached a strength of around 300 kPa at the age of 7 days, then continued to grow stronger with time, eventually achieving a value of around 1200 kPa after 90 days of curing. The strength development of CPB has been explained in many articles [8] [10] [96], and is due to the binding effect produced by cement hydration. Via the hydration process, cement compounds (C_3S , C_2S , C_3A , and C_4AF) in CPB react with water (hydrates), producing C-S-H, CH and other hydration products that bond individual grains together to form a

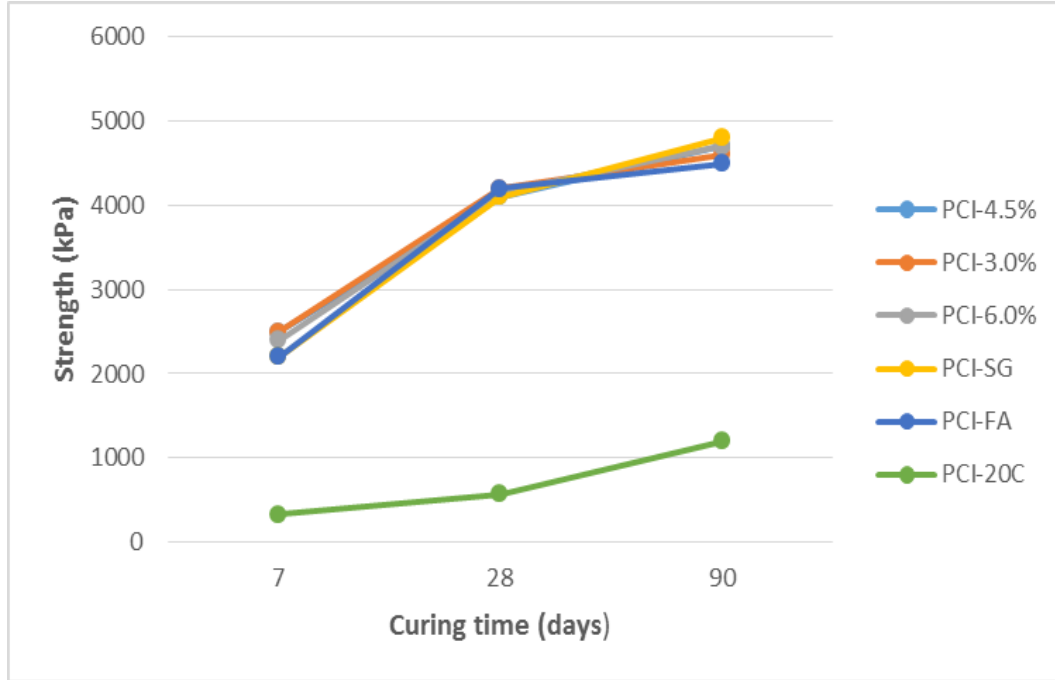


Figure 3.3: Strength development for different binder types, binder content, and curing temperature.

united solid mass. Whereas, for FCPB specimens that cured at sub-zero environmental conditions (-6°C), a precipitous growth of strength can be seen clearly from 7 to 28 days, and a relatively stable growth of strength followed until 90 days. The obtained strength for merely 7 days of curing was as high as 2500 kPa. At 28 days, this value increased to 4200 kPa, and then slowly kept increasing until 90 days, where it reached 4500 kPa. By rough calculation, the acquired strength of FCPB specimens was about 5 times that of CPB specimens. The reason for such a remarkable increase in strength may be credited to the formation of ice within the FCPB. Indeed, once soil freezes, its strength, along with its other mechanical properties, changes dramatically.

Sayles [71] suggested that the strength of frozen soils, as with unfrozen cohesive soils, depends upon both cohesion and internal friction of the component materials. Since CPB and FCPB are made of the same materials, the friction within both can be assumed to be the same in this case, and so is not a part of this study. As for cohesion, according to Vialov and Tsytoich [69], it can be attributed to 1) the molecular forces of attraction between

solid particles; 2) physical or chemical cementing of particles together; 3) cementing of the soil particles by ice formation in the soil voids. And if conditions are favorable where temperature is low enough to allow moisture content to freeze, the cementing effect by ice contributes the most strength to the frozen soil. In the case of FCPB, the molecular forces of attraction between soil particles are assumed to be the same as those that cause friction. The physical or chemical cementing of particles resulting from the cementing effect by CH and C-S-H, on the other hand, is largely dependent on cement hydration. Fall et al. [8] have shown in their research that low temperature does not favor cement hydration, and leads to a decrease in strength. From this point of view, FCPB should have a much smaller cementing effect than CPB. This argument is supported by the results of thermal analysis presented in Figure 3.11 (will be discussed later). However, the cementing effect caused by ice formation is zero for CPB, whereas for FCPB, it is enormous and is responsible for the strength enhancement found in FCPB. In order to highlight the role of the ice matrix in frozen soils, Ting et al. [86] straightforwardly separated strength composition of frozen soil into three parts: ice strength; soil strength; and interaction between ice and soil. In his statement, the ice strength plays the most fundamental role and is known as a function of many factors, including applied strain rates, temperature, confining pressure, stress state, grain size, shape and orientation, and deformation mechanism (cracking, easy or hard glide).

It is also interesting to learn from Figure 3.4 that the strength-gaining rate is faster in the first 4 weeks (28 days) than during the next 9 weeks. This can be explained by the fact that the strength development of FCPB follows the process of ice formation rather than cement hydration, which continues gradually throughout the whole curing time. After 28 days of curing, most water has frozen, and so ceases to provide any ice content to increase its strength.

Effect of binder content

Peak strength versus curing time for FCPB samples with four different cement contents (0%, 3%, 4.5%, and 6%) is plotted on Figure 3.4. It is clear that all three types of

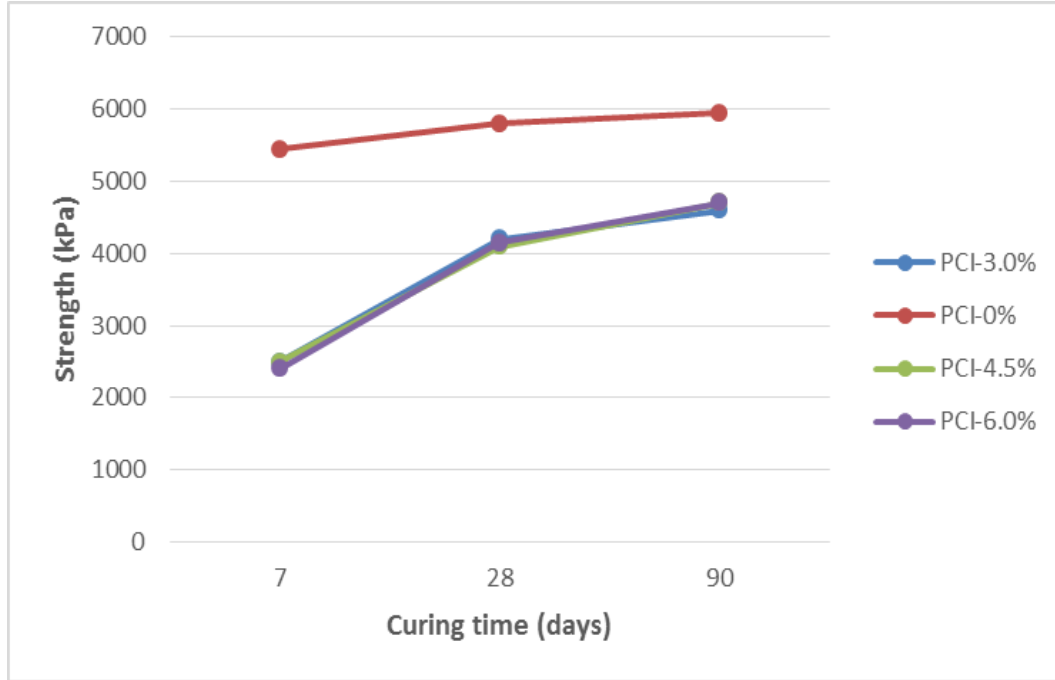


Figure 3.4: Strength development of FCPB of different cement content (0%, 3%, 4.5% and 6%) cured up to 90 days at -6°C .

FCPB using binders developed an extraordinarily uniform strength growth under sub-zero conditions. This finding implies that cement content has no significant effect on the general behaviour of the strength development for FCPB.

It is of little surprise to learn that cement failed to provide any significant strength for FCPB. After all, cement hydration to a great extent relies on the curing temperature. As curing temperature declines, the hydration process is inhibited. As soon as curing temperature drops below the freezing point (0°C), capillary water within the paste starts to freeze, and therefore, the hydration process is suspended almost completely. Husem and Gozutok [104] discovered in their research that casting concrete at a temperature of -5°C could result in a loss of strength of up to 80%. Moreover, the freezing water has a potential to lead to an expansion within the concrete matrix, giving rise to cracks and thus a further loss in strength [105]. At this point, it seems safe to say that cement makes little difference to the mechanical behaviour of the frozen paste backfill due to the limited hydration ratio. Having said that, specimens with 0% cement (FPB) show a different story. Without any

application of cement, FPB samples exhibited a greater and more distinctive compression strength than any of the other specimens at any time. In light of the influential factors on frozen soil suggested in Chapter 2, for a number of similar frozen soil specimens, the strength is usually an interplay among three parameters: 1) unfrozen water content; 2) ice content; 3) frictional resistance.

Compared with silica tailings, Portland cement characterizes a relatively finer grain-size distribution. According to Ferraris et al. [106], the particle size of cement grains varies from less than $1\text{ }\mu\text{m}$ to $1000\text{ }\mu\text{m}$, and nearly 50% of all cement grains have a size lower than $10\text{ }\mu\text{m}$. By contrast, the particle size distribution with respect to silica tailings varies from $1\text{ }\mu\text{m}$ to $1000\text{ }\mu\text{m}$, and only about 30% of the grains are smaller than $10\text{ }\mu\text{m}$, around 20% of the grains have a size close to $1000\text{ }\mu\text{m}$. This is to say that, once mixed with cement, CPB samples will possess a higher portion of fine material than they usually do. This has an adverse effect. Overwhelming evidence suggests that a fine ratio increment within frozen soil will fortify the phenomenon of melting-point depression, and thus promote the unfrozen water content [107]. And increasing the unfrozen water content has a negative effect on strength.

Moreover, large grain size and bulky particle shape are credited with increasing the resistance that contributes to friction, which in turn increases the strength of frozen soil [73]. Tsytoich and Sumgin [78] realized in their experiment that frozen sand had about four times the strength of Suffield clay at the same temperature. After examining all potential influences, they later put forward a claim that, apart from reduction in unfrozen water content, the presence of fine grains results in a smaller friction angle and reduced strength. More recently, concepts such as internal confinement, soil dilatancy and structural hindrance have served to highlight the significance of grain size on frozen soil [73] [86].

Other than this, the difference in water content remains an issue. All the FCPB specimens containing cement have water contents ranging from 24.98% to 25.24%. However, for FPB, this figure is 26.14%. On first thought, this 0.9% difference in water content hardly seems to be worth mentioning, but the data gathered from the study of water content indicated otherwise. Results show an extraordinarily sensitive response of the compres-

sive strength to changes in water content. Such differences in water content are capable of altering the mechanical behaviour of frozen soil. A rough calculation predicted that a increase of 1% in water content would correspond to a 5% growth in total compressive strength.

Speculations once were made about whether heat produced by the cement hydration process, coupled with water consumed at the same time would account for part of the decrease in strength. Han [103] and Thiessen [108] both observed a slight decrease in compressive strength of FCPB of advanced age (90 days), and they attributed this decrease to the “heat generated form delayed cement hydration”. These explanations seem far-fetched; while it is true that cement hydration is an exothermic chemical reaction, in which heat will start to be released as soon as two components are mixed together, this is only worth considering in large-scale applications. At the laboratory scale the amount of heat produced by cement hydration is negligible, to say nothing of a sub-zero environment, where the hydration process itself is largely hindered and heat dissipates very quickly because of the huge thermal gradient. The temperature monitoring experiment also provided strong support for the previous statement (Figure 3.10; will be discussed later). Results revealed that once the specimens were put into the freezer, the temperature of each dropped very rapidly. As a result, within 24 hours they reached thermal equilibrium with the freezer temperature (Figure 3.10).

As for the volume of water consumed by cement hydration, it is too small to be considered. In the study of the minimum water-to-cement (W/C) ratio for cement hydration, Xiong and Wang [109] illustrated the theoretical amount of water required to achieve a complete reaction between cement compounds and water. In such an ideal scenario, where all cement grains reacted and there was no water left, the total water content was only around 21.9% of the cement content by weight. The derivation process is given below:

Assume a most common composition of Portland cement type I (see Table 3.9)

So chemical reaction for each compound during hydration process:

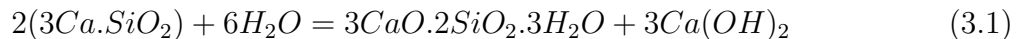
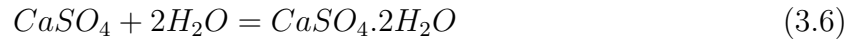
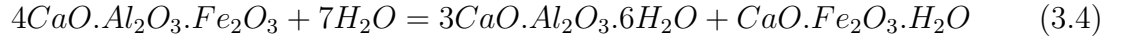
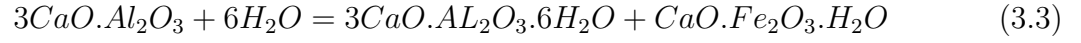
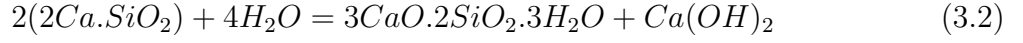


Table 3.9: Composition of Portland cement type I

C ₂ S	C ₃ S	C ₃ A	C ₄ AF	MgO	CaSO ₄	SUM
49.9%	22.1%	9.1%	12.2%	2%	4.25%	100%



So the minimum W/C ratio for the individual compound is:

$$W/C_{(C_3S)} = (6 \times 18)/(2 \times 228) = 0.237 \quad (3.7)$$

$$W/C_{(C_2S)} = (4 \times 18)/(2 \times 172) = 0.209 \quad (3.8)$$

$$W/C_{(C_3A)} = (6 \times 18)/(3 \times 56 + 102) = 0.4 \quad (3.9)$$

$$W/C_{(C_4AF)} = (7 \times 18)/(4 \times 56 + 102 + 160) = 0.259 \quad (3.10)$$

$$W/C_{(MgO)} = 18/40 = 0.45 \quad (3.11)$$

$$W/C_{(CaSO_4)} = 2 \times 18/136 = 0.265 \quad (3.12)$$

So the total W/C ratio is:

$$0.449 \times 0.237 + 0.221 \times 0.209 + 0.091 \times 0.4 + 0.122 \times 0.259 + 0.02 \times 0.45 + 0.0425 \times 0.265 = 0.219 \quad (3.13)$$

So technically speaking, the W/C ratio required to fulfill a complete cement hydration is merely 0.219. Compared with most W/C ratios applied in this study (which are 5.9, 7.6, and 11.1), this value is too small to be taken into account, not to mention that cement hydration is largely crippled in a frozen condition.

Effect of binder types

Blending pozzolans with cement in CPB to achieve a cost reduction has gained considerable attention over the past decades. It has been demonstrated that combining pozzolans such as slag and fly ash does not compromise the strength at all. Instead, it improves the durability in the long run [8]. However, previous studies on such combinations have focused only on temperatures above zero, with little effort devoted to conditions below freezing. Therefore investigations to test the effects of different binder types on FCPB were made in this section.

The overall measurement results are summarized in Figure 3.5. The graph indicates that, no matter which type of binder was used, FCPB samples exhibited an unanimous strength development up to 90 days. It is of little surprise to see that mixing pozzolans as new binders with cement had no influence on the mechanical behaviour of FCPB. As explained before, unlike CPB samples that are cured at room temperature and whose strength comes mainly from cement hydration products, FCPB samples express their strength by

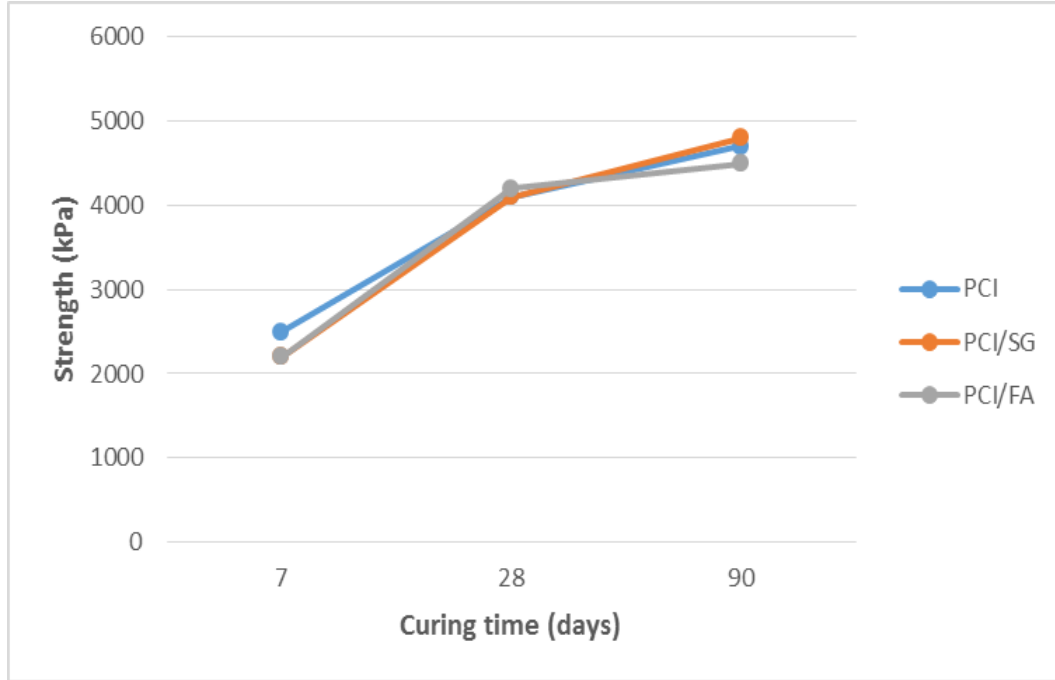


Figure 3.5: Strength development of FCPB of different binder types (PCI, PCI/SG, PCI/FA) cured at -6°C .

means of an ice matrix. That is to say that, as long as the ice content is the same and internal friction is similar, the strength of FCPB is basically determined. The pozzolanic reaction observed in CPB at room temperature was not expected to take place under such circumstance. This is because the pozzolanic reaction occurs between calcium hydroxide (CH) and silicic acid (SH), and CH can only be provided from cement hydration, which has been inhibited for the most part due to the low curing temperature as shown by Figure 3.12.

Effect of water content

Figure 3.6 and Figure 3.7 show the variations of peak compressive strength of FPB specimens with different water content cured at a temperature of -6°C . The peak compressive strength is found to be very sensitive to the change of water content. As shown in those two figures, at low total water contents (5%–26%) peak strength increases sharply with water content, but beyond that point (26%) the compressive peak strength declines dramatically, and eventually comes close to the strength of ice. Similar observations have also

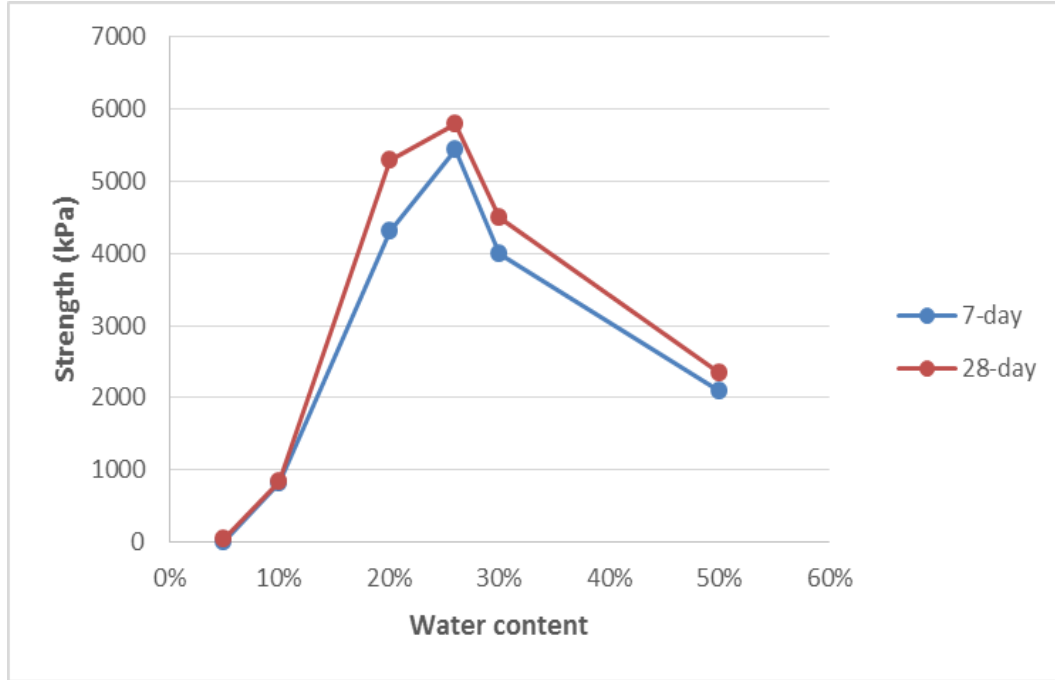


Figure 3.6: Strength developments of PT samples with different water content up to 28 days.

been made by Wijeweera and Joshi [73], and other researchers. The explanation for the above outcomes is given below:

- 1) When water content varies in the range from 5% to 26%: as temperature drops and pore water freezes, the formation of ice provides an extra binding effect in addition to soil cohesion, which results in an increase in soil strength. Within this range, soil grains and ice matrix together regulate the strength of FCPB. However, between the water contents of 5% and 10%, FPB specimens show little trace of ductile behaviour under the compression; instead both of them fail at earlier strains (see Figure 3.9). This indicates that the amount of ice content within these two types of FPB is not sufficient enough to enable ductile behaviour. As can be seen from Figure 3.8 during the UCS test, FPB samples with 5% water content were crushed into powders. In FPB samples with 10% water content, even though they remained intact, severe cracks could be found on the bearing body. When the water content reached 20%, clear ductile deformation appeared and no distinctive failure or cracks were found

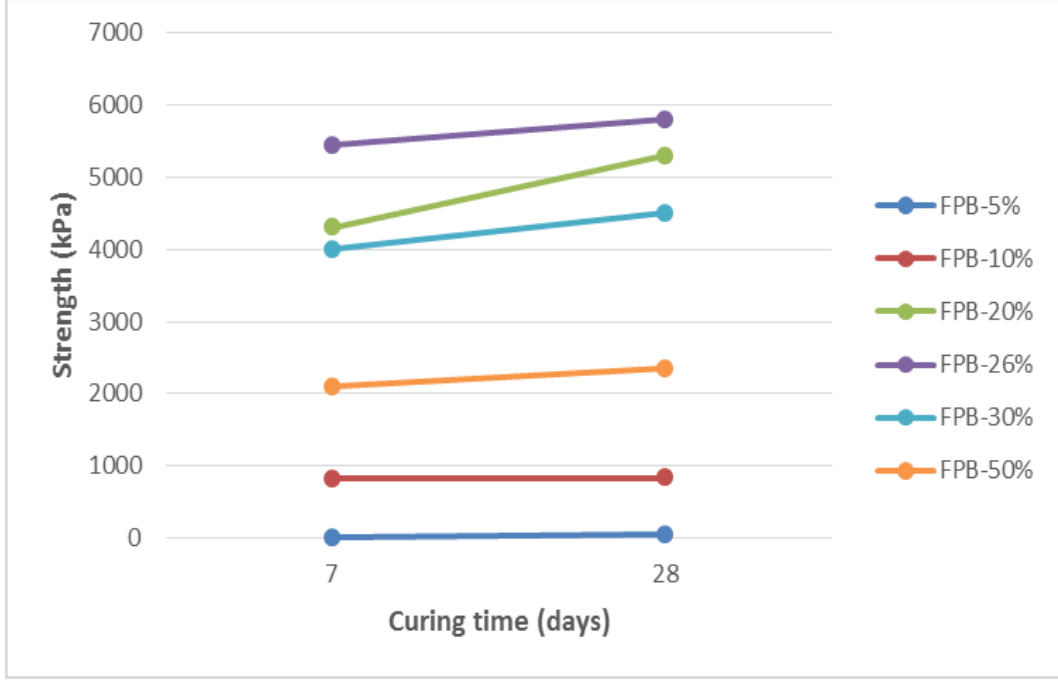


Figure 3.7: Strength variations of FPB specimens in terms of water content.

during the experiments (see Figure 3.9). At water content of 26%, the FPB specimens displayed the highest compressive stress with a standard strain-hardening process (see Figure 3.8).

- 2) Once water content is beyond the point of 26%: as water content continues to grow, the ice matrix starts to take on the overall contribution to compressive strength. In other words, the soil cohesion and internal friction effect are impaired because of the over-saturated ice content, and FCPB samples behave more and more like ice cylinders. One drawback from this transition is that the ductile behaviour of FCPB gradually turns brittle under the same strain rate, and causes a loss of strength. Figure 3.8 and 3.9 give an example of such transition. At water content of about 30% or more, cracks occur during compression, and strength drops correspondingly. Similar observations have also been made by Wijeweera and Joshi [73].

Another key point to which attention needs to be paid is that, FPB samples show a far more rapid growth of strength compared to other FCPB samples (see Figure 3.7).

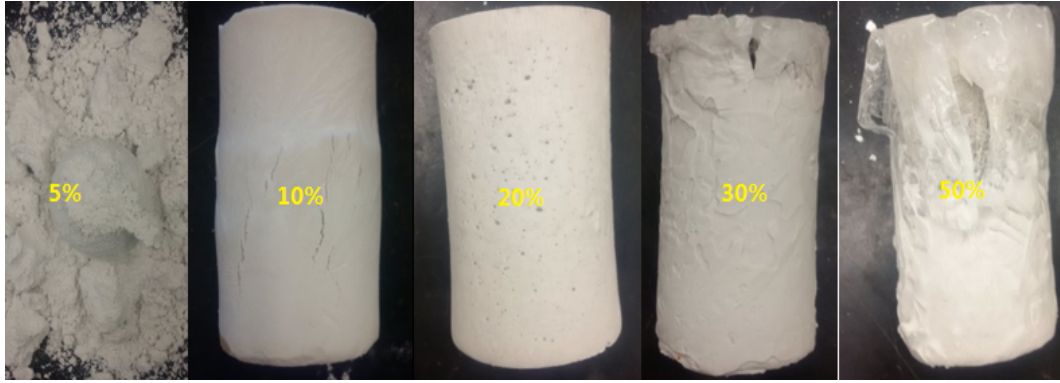


Figure 3.8: Failure modes of FPB samples with different water content.

Unlike FCPB samples in which there is a rapid growth in strength between 7 and 28 days, all FPB specimens achieved most of their strength in the first 7 days, and made slightly improvements during the following 3 weeks. This suggests that water freezes faster in FPB samples than it does in FCPB samples. Unfortunately, the graph does not enable us to determine the causes of such acceleration in freezing. But considering the fact that all monitored samples demonstrated a consistent temperature (which will be explained in later sections), the most likely explanation for it is that abandoning the usage of binder somehow reduces the ability for FCPB samples to remain as much supercooled water as FPB does. Moreover, ice has a thermal conductivity that is more than four times greater than water [110], which means the already formed ice has an effect that further speeds up the formation of ice.

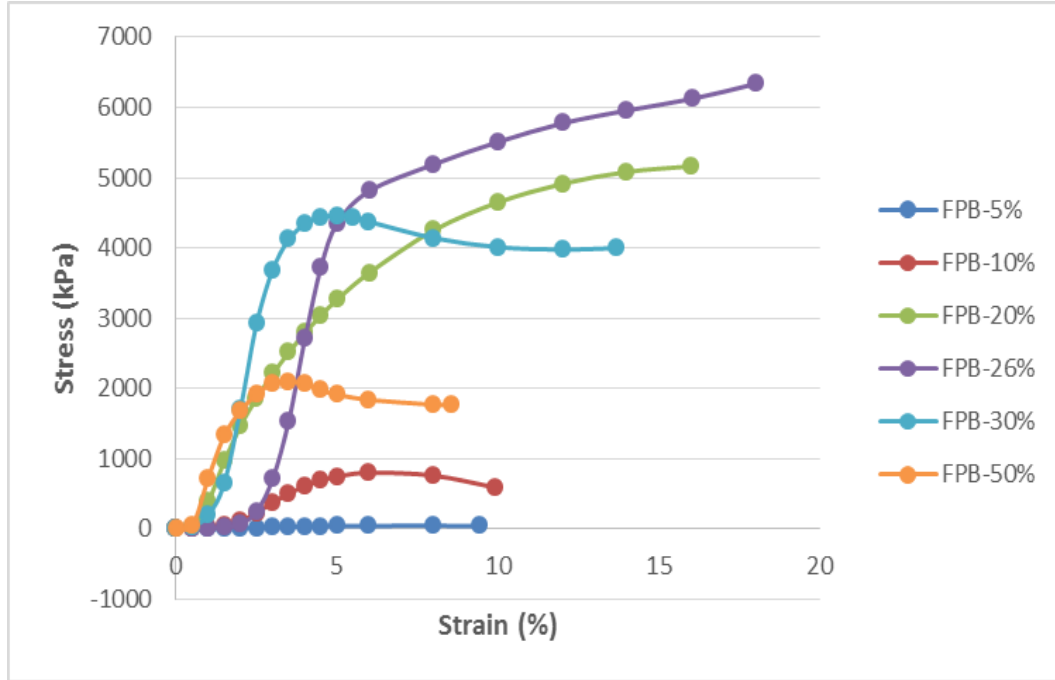


Figure 3.9: Stress-strain curves of FPB samples with different water content after 7 and 28 days of curing.

3.3.2 Temperature monitoring

Temperature monitoring was incorporated into this study for the purpose of understanding ice formation, cement hydration, and strength development. A CAT II 600V multimeter was chosen for monitoring the temperature changes. The frequency of monitoring was once every three hours for the first three days, then once a day for the remaining 87 days. Figure 3.10 is a brief summary of temperature development tendencies. For orderliness, only part of the data are presented here.

It may be noted that all specimens, followed almost the same path of thermal development, regardless of different mix composition adopted. No noteworthy temperature rise was observed during the monitoring, which rules out the possibility of the negative effect of softening caused by hydration-induced heat at advanced ages.

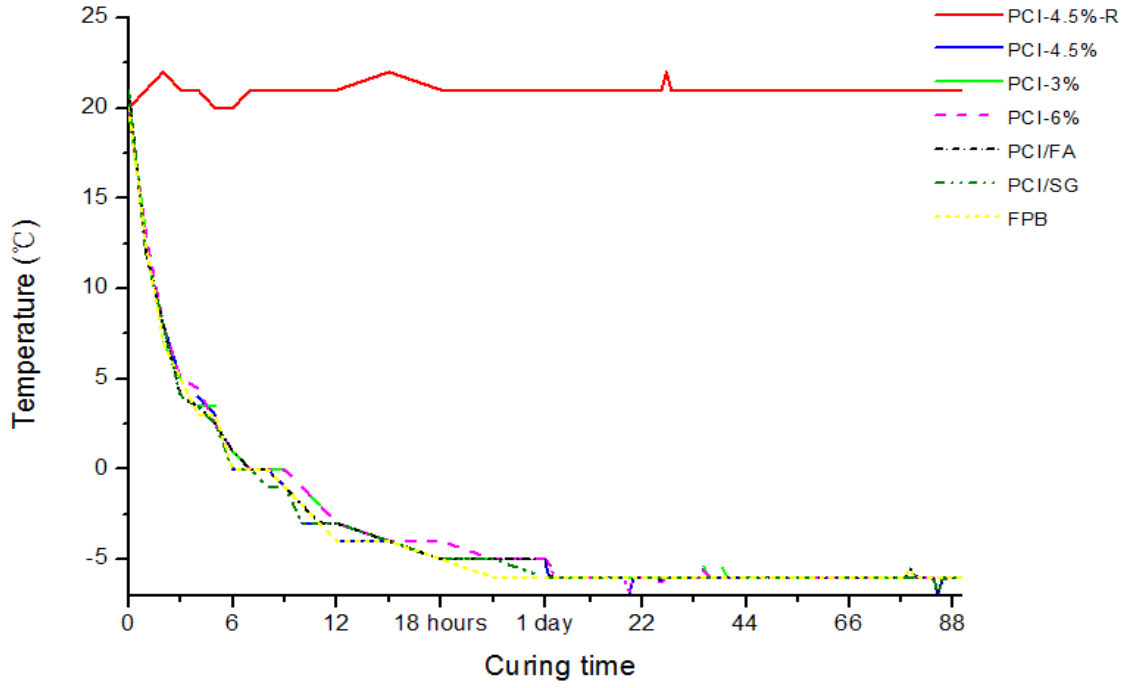


Figure 3.10: Temperature monitoring of seven different FCPB samples; PCI-4.5%-R (FCPB with 4.5% cement ratio cured at room temperature); PCI-4.5% (FCPB with 4.5% cement ratio cured at -6°C); PCI-3% (FCPB with 3% cement ratio cured at -6°C); PCI-6% (FCPB with 6% cement ratio cured at -6°C); PCI/FA (FCPB mixed with fly ash cured at -6°C); PCI/SG (FCPB mixed with slag cured at -6°C); FPB (FCPB with 0% cement ratio cured at -6°C).

3.3.3 Thermogravimetric analysis

Results derived from thermogravimetric analysis are in great agreement with the findings in earlier sections. Figure 3.11 presents the DTG/DTA diagrams of three different CPB and FCPB specimens. In this graph, the DTA pinpoints the temperature at which thermal decompositions of different phases occur in paste, while DTG simultaneously reflects the weight loss as a result of these decompositions [10]. Three main decomposition peaks associated with rapid weight loss and major phase transformations were noticed for each specimen in this figure. According to Zhou and Glasser [111] and Fall et al. [10], the first peak, which is in the range of 110 to 200°C , is involved with the dehydration of hydrates,

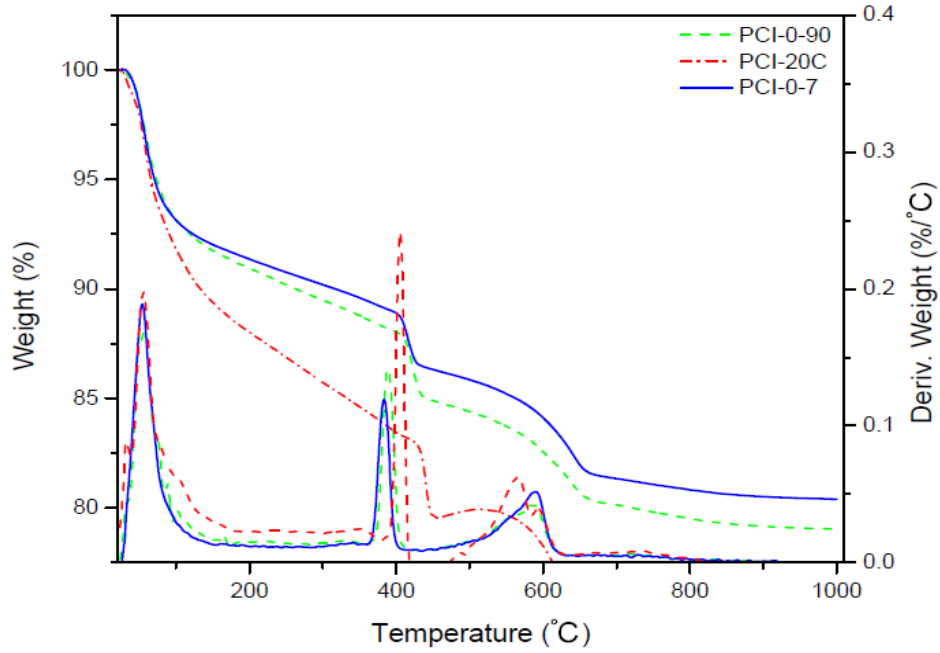


Figure 3.11: TG/DTA diagrams for 7- and 90-day old CPB and FCPB cured at 20°C and -6°C , respectively.

such as C-S-H, carboaluminates, ettringite, and gypsum. The second peak, located between 400 and 500°C, is due to the dehydroxylation of CH [10]. The last peak, between 650 to 750°C, is the result of the decomposition of calcite (CaCO_3) [10] [112].

In light of the information above, let us take a closer look at this figure. The red dash-dot line (PCI-20C) represents samples that were cured at room temperature for 90 days. It displays the most weight loss of all three curves, indicating the highest degree of hydration. On the other hand, the other two samples, which were cured under frozen condition (-6°C), exhibit clearly lower weight loss, implying insufficient hydration. This demonstrates that low temperature has a huge adverse impact on cement hydration. Moreover, PCI-0-7 (blue line) and PCI-0-90 (green dash-dot line) are identical samples except for different curing times. PCI-0-7 was cured for only 7 days in the freezer, while PCI-0-90 spent up to 90 days at -6°C . However, in comparing those two curves, either on weight loss or decomposition rate, both of them display nearly identical graphs, suggesting that no notable changes happened after the samples became frozen. This is consistent with the results from the

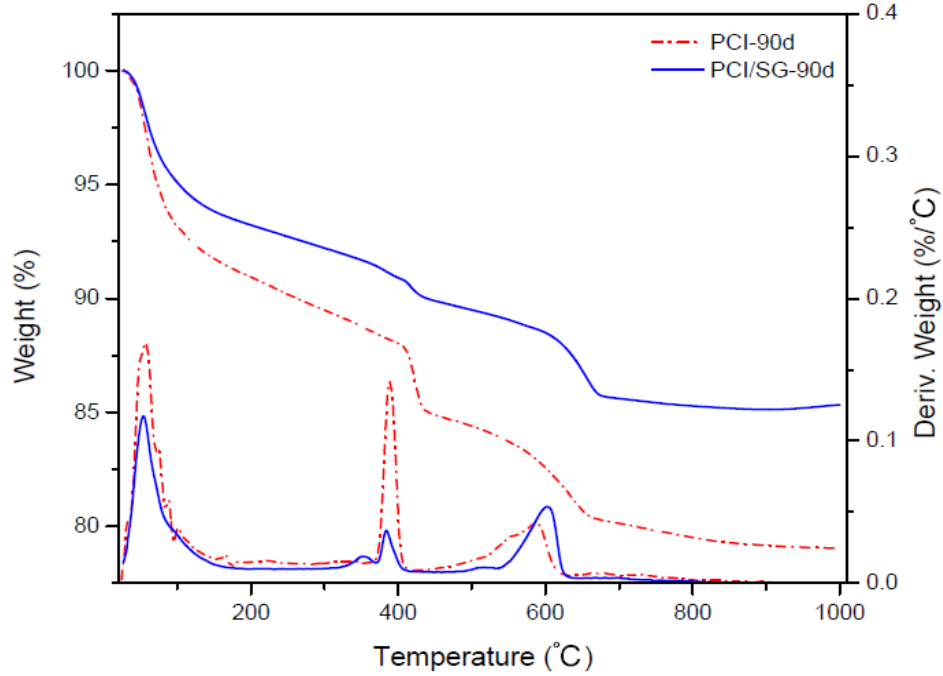


Figure 3.12: TG/DTA diagrams for 90-day old frozen cement pastes of PCI and PCI-Slag, cured at -6°C .

UCS tests, in which all samples show similar compression strengths despite different binder contents or binder types. A similar observation was found for FCPB containing a slag and PCI mix (with a weight ratio of 50/50) and FCPB containing PCI alone (See Figure 3.12). According to Fall et al. [10], the weight loss for each of these two is supposed to be in a similar range at high temperature. Yet, Figure 3.12 shows a nearly 50% weight difference between these two samples. That is, the pozzolanic reaction is significantly inhibited under freeing conditions.

3.3.4 Mercury intrusion porosimetry

Mercury intrusion porosimetry measurements on FCPB and CPB samples are shown in Figure 3.13 and Figure 3.14. FCPB samples, which were cured at -6°C , displayed higher pore volume in total than CPB samples that were cured at 20°C . Especially in the range from 0.003 to $0.7\mu\text{m}$, FCPB exceeded CPB by nearly 10% in terms of total pore

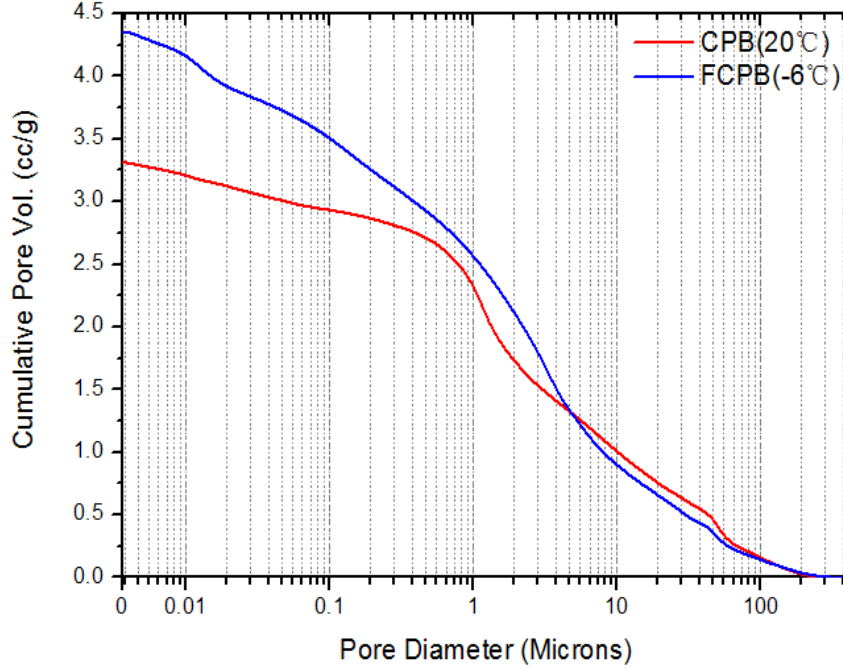


Figure 3.13: MIP pore size distribution of CPB cured at 20°C and -6°C .

space. This difference perhaps came from the premelted films that were enclosed within small pores or around the surfaces of tiny particles. Rempel et al. [113] reported that, although the thickness of premelted films depends upon many factors, they generally are on the order of 10^{-8} m. Additionally, both types of sample contain the largest fraction of pore volume at the scale of 10^{-6} m, which fits the pore size distribution of most hardened cement paste and mortar materials. Then again, FCPB exhibits coarser diameters; approximately four times as great as those of CPB samples. This can be explained by ice lenses, which form within paste and disturb the original arrangement by pushing soil grains away from each other.

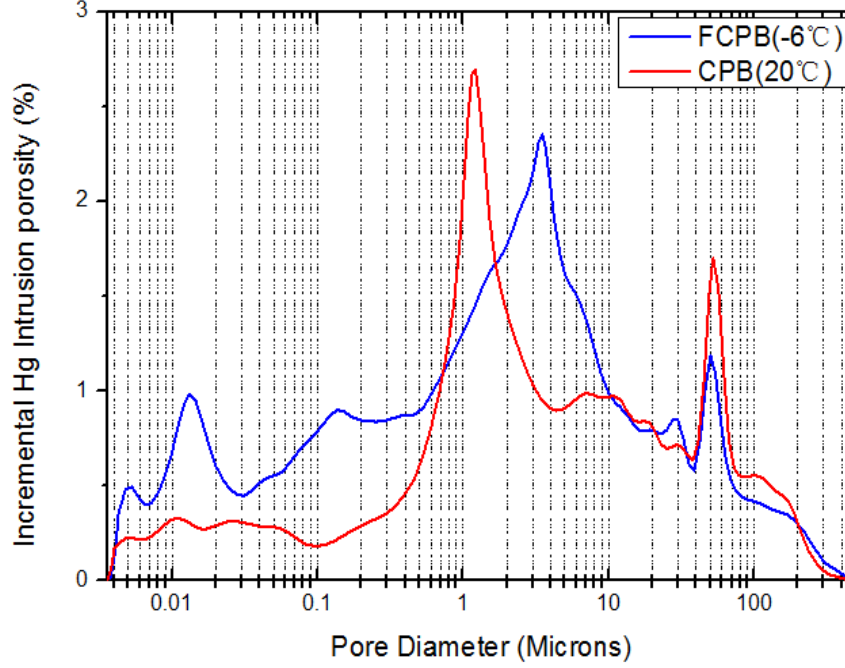


Figure 3.14: MIP pore size distribution of CPB cured at 20°C and -6°C .

3.3.5 Characteristics of stress-strain curve

On account of the fact that mechanical characteristics of FCPB fit well into frozen soils, the stress-strain curve of FCPB is presented particularly in this sector in an effort to further explore the mechanical properties of FCPB. To that end, it seems appropriate to recall some of the basic characteristics before going into a more detailed discussion.

Prior to 1952, the study of frozen soil was generally incomplete in all aspects and was based solely on empirical equations. Since 1952, the Arctic Construction and Frost Effects Laboratory started to issue a series of technical reports involving deformation properties of frozen soils, coupled with intact experimental data. In one of the reports, Vialov and Tsytovich [69] explained the physical process of creep in frozen soil and later came up with a classic rheological model to simulate it. The explanation of the physical process of creep in frozen soil was depicted as follows: pressure-melting of the ice happens at points of contact between grains of soil; migration of unfrozen water is then initiated and water move to regions of lower stress. Unfrozen water freezes again at new regions and there is

a readjustment in the particle arrangement. During this process, both strengthening and weakening effect occur. The strengthening is a result of denser packing of soil particles (i.e., increased number of firm contacts between soil particles), and the weakening is due to the reduction in cohesion and the increase in unfrozen water content.

With this in mind, let us take a closer look at the results. Independent of binder content and binder types, the stress-strain curves of FCPB specimens are characterized by ductile behaviour with strain softening at 7 days of curing, and strain hardening after 28 days of curing, but either way there is no apparent peak strength for FCPB. Judging by Vialov's rheological theory, it is possible to conclude that the competition between the strengthening effect caused by denser packing of soil particles and the weakening effect due to the reduction in cohesion, coupled with the increase in unfrozen water content, were influenced by curing time. At early ages, when there was still quite an amount of unfrozen water in the FCPB, the weakening effect stood out and dominated. Nevertheless, as the curing process proceeded this advantage gradually vanished, and eventually samples started to exhibit strain hardening. In addition to the difference in strain behaviour, the initial yield point is another variance worth discussion. The initial yield point refers to the point at which the initial slope of the stress-strain curve starts to decrease noticeably [73]. Sayles and Carbee [76] and Zhu and Carbee [114] suggested that the yielding is mainly attributable to the initial failure of the ice matrix. This is in strong agreement with the data derived from this study. Figure 3.15 shows that the yield strength increases with curing time. At 7 days, the yield strength is about 2000 kPa for all FCPB samples; at 28 days, it climbs to 3800 kPa; and at 90 days, it reaches a point around 4200 kPa. Considering that the only significant change to have happened within the FCPB is the ice formation, the increase in initial strength is easy to explain as a result of the growing ice content. Besides, Zhang et al. [115] and Yang et al. [116] claimed that severe volumetric strain of frozen soil happened during compression, and played an important role in its mechanical behaviour. On the condition that there is no confining pressure applied to frozen soil samples during the UCS test, sandy frozen soil samples were found to yield a series of relationships between volumetric and axial strain. They have demonstrated that within the first 5% of axial strain, volumetric deformation of sandy frozen soil is relatively

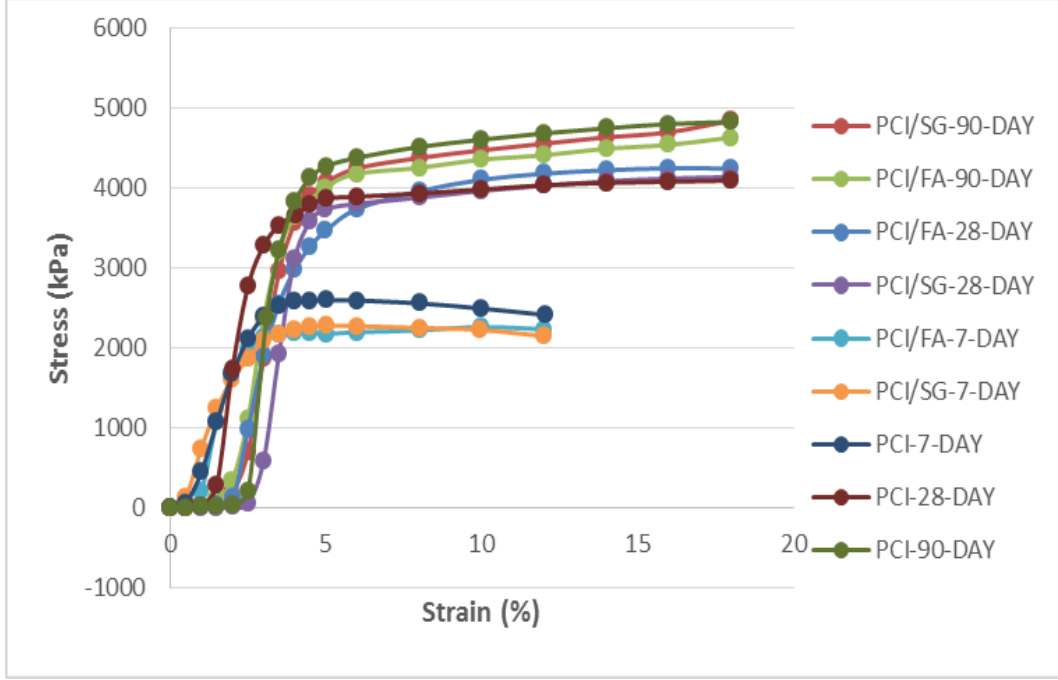


Figure 3.15: Stress-strain curve of FCPB samples with different binder types at different curing times.

small and safely can be assumed to be zero. However, as soon as axial strain passes 5%, volumetric deformation takes place and presents an almost linear relationship with axial strain. This indicates that the first 5% axial strains are mainly contributed by the tensile deformation of the ice matrix. Once the ice matrix broke, samples readily became compressible and volumetric deformation commenced.

Another interesting discovery is that, regardless of what stress path FCPB samples adopted towards the yield point, they eventually displayed a number of stress-strain curves with similar characteristics near the end. To put it another way, there seems to be a disordered-to-ordered transformation taking place during deformation. It cannot be denied that this disorder during early strain could be to some extent, the result of inconsistent device stiffness or the uneven surface of specimens. That said, we cannot exclude the possibility that there is a rearrangement of soil grains during deformation.

3.4 Conclusion

The following conclusions may be drawn from this chapter:

- FCPB exhibits remarkable strength at advanced ages (> 90 days), which is nearly three to four times stronger as CPB during the same curing time. Furthermore, it gains strength rather quickly. Within less than one month since casting, FCPB achieved 90% of its total strength. These two features are considered of tremendous significance in terms of the practical operation of shortening the interval time between filling two stopes.
- It is recognized that no noteworthy failure surface was observed from FCPB samples during compression, and strain hardening phenomena were commonly seen after the initial yield point. The mechanical behaviour and properties of FCPB were found to deviate from concrete or CPB, but were in an excellent agreement for frozen soil. Stress-strain relationships among FCPB samples also displayed a type of ductile behaviour that is normally discovered in frozen soil.
- Neither binder content nor binder type showed any evidential influence on the strength development of FCPB samples. This could be explained by the fact that cement hydration and pozzolanic reactions were severely inhibited at such a low temperature, and thus provided little contribution to overall strength. It was also confirmed by the TGA that 7-day and 90-day FCPB yielded almost identical curves, indicating an inert attitude among cement compounds toward any chemical reactions. On the other hand, initial water content plays a key role. At a relatively low water content ($< 26\%$ by weight), compressive strength goes up sharply as water content increases; yet above this level, strength declines gradually to ice strength.
- It is hypothesized that the growth of compressive strength of FCPB with time is due to the increasing amount of ice. Although temperature monitoring of six different kinds of FCPB samples demonstrated that temperature within the samples became constant after only one day of curing, formation of the ice matrix would still develop progressively afterwards.

Chapter 4

Impact of Sulphates on the Properties of Frozen Cemented Paste Backfill

4.1 Introduction

The significance of the mining industry to the Canadians' life cannot be overestimated. Mining and its related industries are not only credited with the fourth largest contribution to Canadian economy, but also provide the number one employment opportunity for aboriginal peoples. Nevertheless, all benefits set aside, mining activities pose many challenges, central among is mine waste. According to Statistics Canada [2], nearly 500 million tonnes of mine waste is produced each year. This mine waste, which includes waste rock and tailings, requires and awaits management. Due to their high mobility, tailings in particular draw attention from the public. Currently, the best way to deal with tailings is believed to be backfilling.

The application of CPB first occurred in the 1970s, and is beneficial in many aspects. For example, it increases the safety factor for mine operations; it promotes a higher rate of ore recovery; it limits environmental impact associated with tailings management; and it reduces overall costs [7] [117]. However, to ensure successful performance, a complex

assessment of mine conditions, coupled with any anticipated variability at mine sites with time, is always necessary. Numerous investigations have been conducted on the strength of CPB, as well as on its long-term durability. Results indicate that CPB is prone to a number of progressive losses in strength, one of the principal causes of which is sulphate attack. Sulphates have been found at high concentrations in both tailings and mix water at mining sites. Observations of concrete structures and cemented paste backfill at mine sites show severe damage in terms of the loss of durability and strength, and increased permeability [7] [9] [118]. Extensive research has been conducted regarding the effect of sulphate attack on CPB, and has made great progress. Yet, most research has focused only on temperatures above freezing, studies of the effects of sulphates under sub-zero conditions still are lacking. Therefore, research related to the effect of sulphates on the properties of frozen cemented paste backfill (FCPB) has been carried out in this study. The main objectives of this research are:

- Examining the long-term strength of FCPB samples with various sulphate concentrations;
- Studying the pore structure and mineralogical composition of FCPB samples in the presence of sulphates;
- Identifying potential factors that influence the mechanical behaviour of sulphate frozen cemented paste backfill (SFCPB).

The experimental program and the main results are presented and discussed in the following sections.

4.2 Experimental Program

4.2.1 Materials used, sample preparation and curing

4.2.1.1 Materials used

Silica tailings, Portland cement type I, slag, distilled water, and ferrous sulphate were used in preparing specimens for the study. The compositions of tailings and binder are the same as they were in Chapter 3. Distilled water was used instead of tap water to mix all specimens. The purpose for this is to maintain a desired and accurate concentration of sulphate in each sample; it is also to avoid any chemical interference that tap water might introduce. Desired concentrations of sulphate were achieved by adding calculated amounts of ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) to the distilled water. A more detailed description of the binders and tailings used in this chapter are given in Section 3.2.1.

4.2.1.2 Sample preparation and curing

The making process for SFCPB specimens for UCS tests is described in detail in Section 3.2.1; therefore, only a brief description will be presented here.

SFCPB specimens with various sulphate concentrations (0 ppm, 5,000 ppm, 15,000 ppm, and 25,000 ppm) were prepared. Two types of binder were used: Portland cement type I (PCI) and Portland cement type I mixed with slag in a proportion of 50/50 (PCI/SG). Slumps for each paste were maintained at 18 cm, which is the most popular value in practice. The desired concentrations of sulphate solution were prepared first by adding certain amount of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ to distilled water. Four different concentrations were obtained, as indicated in Table 4.1. Tailings, binders and sulphate solution were then properly weighed and mixed together in a mixer to form a homogenous paste. As soon as the desired consistency was reached, the paste was poured manually into curing cylinders, 5 cm in diameter and 10 cm in height. Once each cylinder mold was filled, it was tapped gently against the counter to make sure no air bubbles were trapped in the paste. The samples were then sealed with impervious tape to avoid evaporation of the water.

Table 4.1: Formula for desired concentrations of sulphate solution.

Distilled water volume	Sulphate concentration	Salt mass ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$)
1 L	0 ppm = 0 g/L	0 g
1 L	5,000 ppm = 5 g/L	14.48 g
1 L	15,000 ppm = 15 g/L	43.44 g
1 L	25,000 ppm = 25 g/L	72.40 g

For the freezing facility in the curing phase of the experiment, the same walk-in, custom-made freezer used in Chapter 3 was chosen. The temperature and humidity within the freezer were precisely controlled by an Omega iSeries Humidity and Temperature Controller (CNiTH and DPiTH Series), with errors of $\pm 5^\circ\text{C}$ and $\pm 2\%$ RH. The curing temperature was set at -6°C , with a relative humidity of 67%. The curing period for all samples was 90 days.

In addition to backfill samples, cement pastes (with $\text{W/C} = 1$ to simulate the matrix of CPB) with various initial sulphate contents were also prepared and cured in the same conditions as the backfill samples.

4.2.2 Testing program

The uniaxial compressive strength test was chosen as the primary test of long-term strength for this investigation because of its simplicity, suitability, and comparability with previous tests on other frozen soils. In addition to the compression tests, hydraulic conductivity tests and scanning electron microscope (SEM) were performed. Both of these tests were to explore the pore structure as well as the permeability of sulphated frozen cemented paste backfill. Volumetric unfrozen water content of certain samples was monitored. Such knowledge is conducive to understanding the strength development and ice formation in sulphated frozen cemented paste backfill.

Table 4.2: Mix design for the study of strength development of sulphate SFCPB.

Sample name	Binder content	Binder combination	Blending ratio	W/C ratio	Sulphate concentration (ppm)	Curing age (days)
PCI-0	4.5%	PCI	100	7.6	0	90
PCI-5	4.5%	PCI	100	7.6	5,000	90
PCI-15	4.5%	PCI	100	7.6	15,000	90
PCI-25	4.5%	PCI	100	7.6	25,000	90
SPCI-0	4.5%	PCI/Slag	50/50	7.6	0	90
SPCI-5	4.5%	PCI/Slag	50/50	7.6	5,000	90
SPCI-15	4.5%	PCI/Slag	50/50	7.6	15,000	90
SPCI-25	4.5%	PCI/Slag	50/50	7.6	25,000	90

4.2.2.1 Uniaxial compressive strength test

UCS were conducted on the samples (sulphated frozen cemented paste backfill, SFCPB) shown in Table 4.1. Testing of SFCPB specimens strictly followed the same procedure as that for FCPB samples. Specimens were trimmed one day before testing to minimize any potential influence from trimming. UCS tests were conducted in accordance with ASTM D7300-06. The deformation rate was set at 0.15 mm/min, and results were automatically logged into a computer by LabVIEW. If no peak strength was available due to the strain-hardening effect, the strength corresponding to a strain of 15% was selected as the peak strength instead.

4.2.2.2 Monitoring volumetric unfrozen water content by 5TE soil sensors

It is commonly accepted that frozen soils consist of soil grains, ice, unfrozen water, and air [54]. The amount of unfrozen water in frozen soils is dependent on several factors, such as temperature, applied pressure, specific surface area, and pore fluid chemistry. In

Table 4.3: Samples for monitoring the volumetric unfrozen water content.

Sample name	Binder content	Binder combination	Blending ratio	W/C ratio	Sulphate concentration (ppm)	Monitoring span (days)
VUW-PT	0%	/	/	/	0	90
VUW-PCI	4.5%	PCI	100	7.6	0	90
VUW-PCI-S	4.5%	PCI	100	7.6	25,000	90

fine-grained soils, unfrozen water closely depends on the soil particles themselves because of adsorption forces. The presence of unfrozen water can significantly influence the mechanical properties of frozen soils. For this reason, the monitoring of volumetric unfrozen water content in SFCPB samples was added to this study. Volumetric unfrozen water content is defined as the ratio of the volume of unfrozen water to the total sample volume, normally expressed as a percentage [119]. 5TE soil sensors were embedded in the SFCPB samples to record the evolution of the volumetric water content (VWC) during curing. The 5TE soil sensors use an electromagnetic field to measure the dielectric permittivity of the surrounding medium. The sensors supply a 70 MHz oscillating wave to the prongs, which charge according to the dielectric properties of the material. The stored charge is proportional to the soil dielectric permittivity and soil volumetric water content. The 5TE microprocessor measures the charge and computes a value for the dielectric permittivity from the sensor. An Em50 data logger was used to collect transient moisture values from the 5TE soil sensors at a frequency of once every 30 minutes. The results were downloaded via ECG20 software.

Three types of FCPB and SFCPB samples were cast to study the evolution of unfrozen water content; these are summarized in Table 4.3. Samples were molded in bigger cylinders (20 × 10 cm) in order to accommodate the size of the 5TE sensors.

4.2.2.3 Hydraulic conductivity

By definition, hydraulic conductivity, symbolically represented as K , is a property of vascular plants, soils, and rock that describes the ease with which a fluid (usually water) can move through pore spaces or fractures [120]. Therefore, the hydraulic conductivity test is able to reveal information about pore structure, cracking, pore interconnectivity, and water retention characteristics of given samples. Such data is commonly seen as key index for assessing the durability of concrete, especially when concrete is to be exposed to an aggressive environment [117] [97].

A Hydraulic conductivity (HC) test was conducted using the flexible wall permeameter principle. After reaching the requested curing time, samples were dried in an oven at a temperature of 50°C for 5 days. The samples were cut into 4-cm-long cylinders and the tests were completed in constant head mode with a head gradation of 1 kPa across the sample. Saturation of the samples was achieved by bridging the influent and effluent lines and applying backpressure for up to 1 day. Saturation was deemed complete when the influent volume equaled the effluent volume. Furthermore, after each HC test the saturation degree of the sample should be determined. The saturation degree was higher than 97%. Each HC test was carried out at least twice. Up to eight types of FCPB sample were tested; the same as with UCS tests.

4.2.2.4 Scanning electron microscope observation

It has long been demonstrated that freezing modifies the geotechnical properties of soils. As water freezes, pore characteristic of the soil will change, and these changes will inevitably lead to further changes of the soil skeleton characteristics. Alteration in microstructure, in turn, can have an effect on macroscopic phenomena. For this study, scanning electron microscope (SEM) observations were implemented on SFCPB samples to determine the effects of freezing and the associated formation of ice lenses on the microstructure of CPB.

An SEM is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample,

Table 4.4: Samples for SEM tests.

Sample name	Binder content (%)	Binder combination	W/C ratio	Sulphate concentration (ppm)	Curing temperature (°C)	Curing time (days)
PCI-R-90	4.5	PCI	7.6	0	20	90
PCI-4.5-90	4.5	PCI	7.6	0	−6	90
PCI-R-90	4.5	PCI	7.6	25,000	−6	90

producing various signals that contain information about the sample’s surface topography and composition that can be easily detected [121]. It produces images with resolution better than 1 nanometer [121].

FCPB samples PCI-R-90, PCI-4.5-90, and PCI-25 were selected to undergo SEM tests. Samples were cured under the same conditions as UCS test samples, but all were dried before testing. Even though there is a potential closing of the space of previous ice segregation sites as water drains away, and a possibility that terminated cement hydration has restarted in the meantime, the sites do not close extensively. Thus, it is safe to assume that there is no significant change in the intra-aggregate porosity during the thaw and consolidation. More information about testing samples can be found in Table 4.4.

4.3 Results and discussion

4.3.1 Uniaxial compressive strength test of SFCPB

Effect of sulphate concentration on long-term strength of SFCPB

The resulting peak compressive strengths of eight samples with various sulphate concentrations are summarized in Figure 4.1. It is clear that the strength is influenced by the sulphate concentration. Evidence explicitly points to a declining trend in strength with

increasing sulphate concentration, regardless of the type of binder used. It shows that, after 90 days of curing, the greatest strength (4800 kPa) was found in SFCPB samples with zero sulphate concentration; as sulphates concentration increased from 5,000 ppm to 15,000 ppm and 25,000 ppm, compressive strength of FCPB steadily dropped from 4100 kPa to 3700 kPa and 3500 kPa, respectively.

Reasons accounting for the loss of strength in SFCPB due to the addition of sulphates can be considered from two aspects, according to Ting et al. [86]: 1) the weakening of the soil matrix; 2) the weakening of the ice matrix. For the first of these, the biggest concern is with regard to sulphate attack on cement hydration products. Yet, as was demonstrated in the previous chapter, cement hydration, which is the basis for sulphate attack, was inhibited tremendously at such a low temperature (-6°C). Therefore, it is safe to ignore the influence of sulphate on soil or cement. On the other hand, the weakening of the ice matrix is worth discussing. The strain vs. stress curves in Figure 4.2 indicate a pronounced decreasing tendency in initial yield strength with sulphate concentration. Considering that initial yield strength is mainly attributed to the ice [76] [114], this leads automatically to the conclusion that the addition of sulphate causes a weakening effect on the ice matrix, and thus an overall decrease in the strength of the SFCPB samples. This raises the question as to what are the reasons that cause this decrease in strength with added sulphate? According to current knowledge, this can be boiled down to two mechanisms: the increase of unfrozen water content and the formation of ice lenses. More details about these are given in Sections 4.2.5 and 4.2.6.

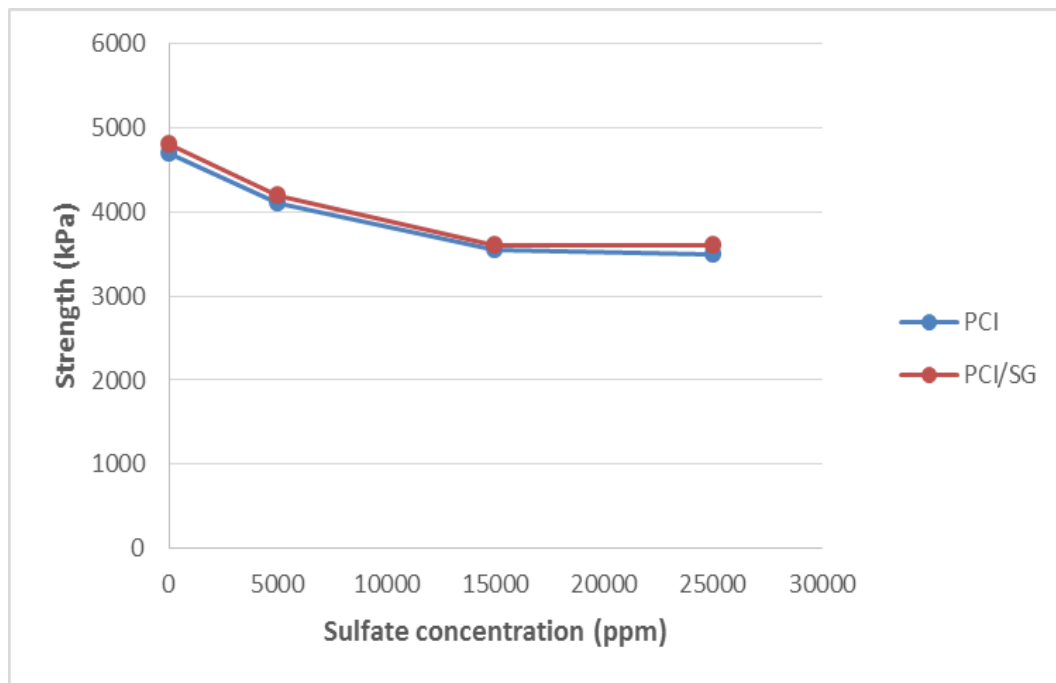


Figure 4.1: Peak compression strengths of SFCPB samples with different binder types and varying sulphate concentration.

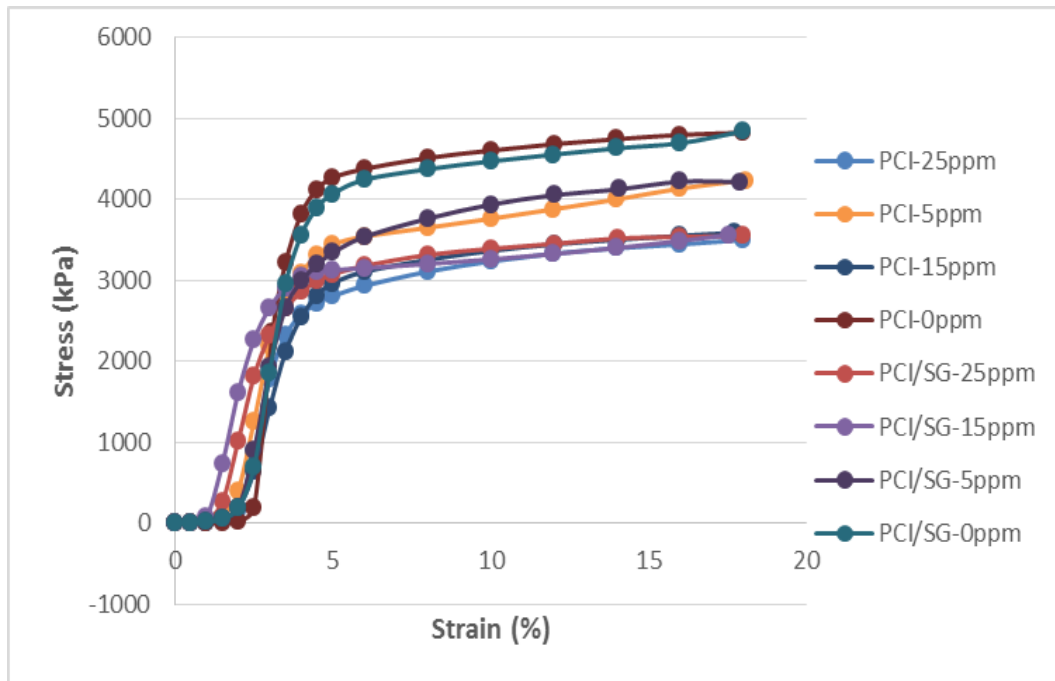


Figure 4.2: Stress vs. Strain curves of SFCPB samples with different binder types and varying sulphate concentration.

Effect of binder types

Partially replacing cement with pozzolans such as slag is not only a common way to reduce the cost of binders, but also an effective approach for limiting sulphate attack in sulphate-rich backfill. In the current thesis, the effect of binder types on SFCPB was investigated in order to evaluate their performance in a sulphate-rich environment.

As expected, in comparisons of the obtained peak compressive strengths (Figure 4.1) as well as the deformation curves (Figure 4.2) of SFCPB samples between PCI-SFCPB and Slag-SFCPB, no noticeable differences were found. That is to say, binder types have no effect on sulphate SFCPB. This finding is in agreement with other observations involving cement hydration. Despite being credited with high reliability in impeding sulphate attacks, pozzolans, in fact, do not provide any differences when there are no attacks, or at least none to a noticeable degree, actually taking place.

4.3.2 Effect of sulphate concentration and binder type on the hydraulic conductivity of SFCPB

The values obtained for the hydraulic conductivity (HC) of the 8 SFCPB samples, as well as the corresponding sulphate concentrations, are shown in Figure 4.3. It can be seen that the HC coefficient varies with the different samples in a relatively large range between $1.83 \times 10^{-4} \text{ cm/s}$ and $3.15 \times 10^{-5} \text{ cm/s}$.

SFCPB samples mixed with both Portland cement and slag exhibit an incontrovertible growing HC trend as the sulphate concentration increases. The lowest HC value of $3.15 \times 10^{-5} \text{ cm/s}$ is presented when the sulphate concentration is zero, and higher HC values are noted as the sulphate concentration rises. When the sulphate concentration reaches 25,000 ppm, the HC is as high as $1.60 \times 10^{-4} \text{ cm/s}$. This tendency of HC to increase along with sulphate concentration can be attributed to sulphate attack. Even though sulphate attack was declared nonexistent in the study of the compressive strength of FCBP, it does occur and should be considered in the HC tests. This is because the samples prepared for HC tests were dried in an oven for five days to eliminate water content, and during

this process, the once-postponed cement hydration was reactivated as a result of the high temperature and the transient presence of available water. Consequently, sulphate attack occurred, giving rise to a number of cracks in the SFCPB sample. These cracks, either inside or on the surface, created short cuts for water to flow through, and significantly boosted the HC.

SFCPB samples mixed with PCI only display a major fluctuation in response to sulphate content. The initial HC coefficient of $3.54 \times 10^{-5} \text{cm/s}$ jumped to $1.83 \times 10^{-4} \text{cm/s}$ as the sulphate concentration reached 15,000 ppm and then dropped suddenly when the sulphate concentration hit 25,000 ppm. The reason that sulphate exerts a more severe influence on SFCPB with PCI than it does on SFCPB with PCI/SG can be explained by the fact that pozzolans have the ability to inhibit sulphate attack. In the cement industry, some cement is commonly partially replaced with pozzolans to reduce the amount of C_3A , which is believed to be associated with sulphate attack. In the case of SFCPB with only PCI, cracks formed more often without this inhibiting effect, resulting in a larger HC. The sudden drop at the 25,000 ppm point can be explained by the consolidation of SFCPB during the drying process. Because the sulphate applied in this study was ferrous sulphate, a common retarder in concrete casting, cement hydration was, to a large degree, interrupted in the oven. The ice matrix disappeared faster than the hydration matrix could form, so that the SFCPB samples could not withstand their own weight and collapsed during drying. This collapse and closing induced a refinement on the pore structure of SFCPB, which decreases its HC.

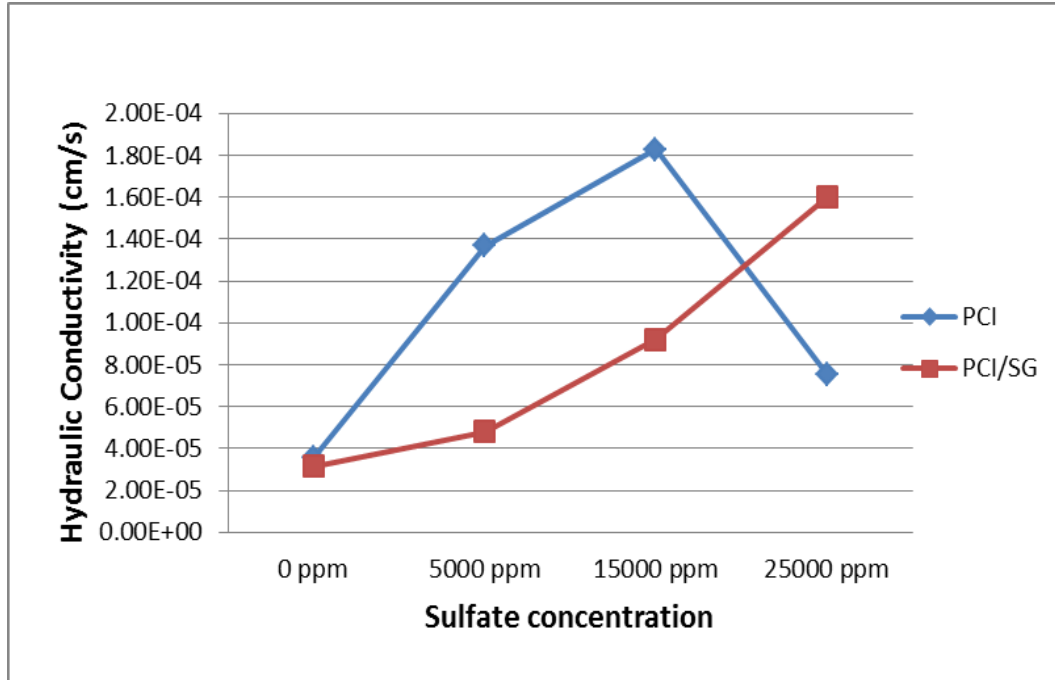


Figure 4.3: Hydraulic conductivity results for SFCPB samples with different binder types cured at sub-zero temperatures (90-day curing time at -6°C , 4.5% cement by total weight, $w/c = 7.6$).

4.3.3 Effect of freezing and the associated formation of ice lenses on the microstructure of SFCPB

The effects of freezing on the microstructure of SFCPB were investigated, using samples that were taken after they had been thawed in an oven for at least five days. The results of the scanning electron microscopy (SEM) research presented below show that total pore space and its distribution of CPB, FCPB, and SFCPB are significantly distinct in appearance. On an order of $10\ \mu\text{m}$, soil skeletons appeared in the images depicted in the figures below.

The images (Figure 4.4), obtained by SEM, are of 90-day CPB samples cured at room temperature. At a magnification of 1500, rather small pore sizes are noted, and there is dense but well-proportioned pore distribution compared with the other two samples. Additionally, all soil grains were closely bonded with each other, implying a strong soil

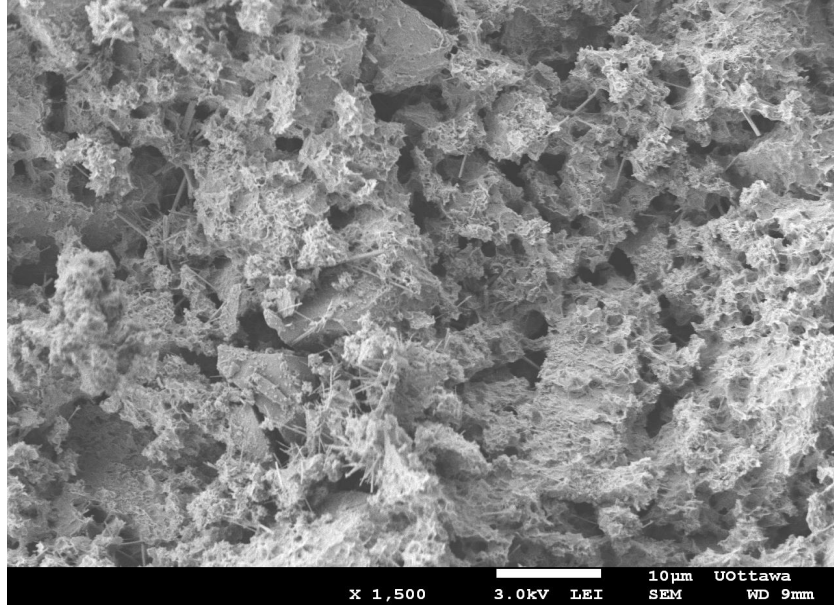


Figure 4.4: SEM image of the microstructure of a 90-day CPB sample cured at room temperature (1500 times magnification).

structure. Clusters of needle-like units and plate-like structures can be found in the image; these represent cement hydration products, ettringites and CH, which indicates a well-developed cement hydration process.

In contrast, the SEM images in Figures 4.5 and 4.6 depict SFCPB and FCPB samples with a number of discontinuities and enlarged pores. These large voids and spaces were occupied by ice segregation, however, after the water drained away, the spaces remained. In general, both the FCPB and SFCPB samples exhibit loose and coarse structures. Soil grains appear individually with no clear bonds between them. Fault-like structures, boulders, and debris are found. All these features are evidence of a significant interruption of the cement hydration caused by ice formation within the soil grains. A great deal of space between individual soil grains coupled with small pieces of debris illustrate that ice lenses not only separated the soil particles from each other, preventing them from developing a united, compact structure, but also damaged the bonds that had already formed.

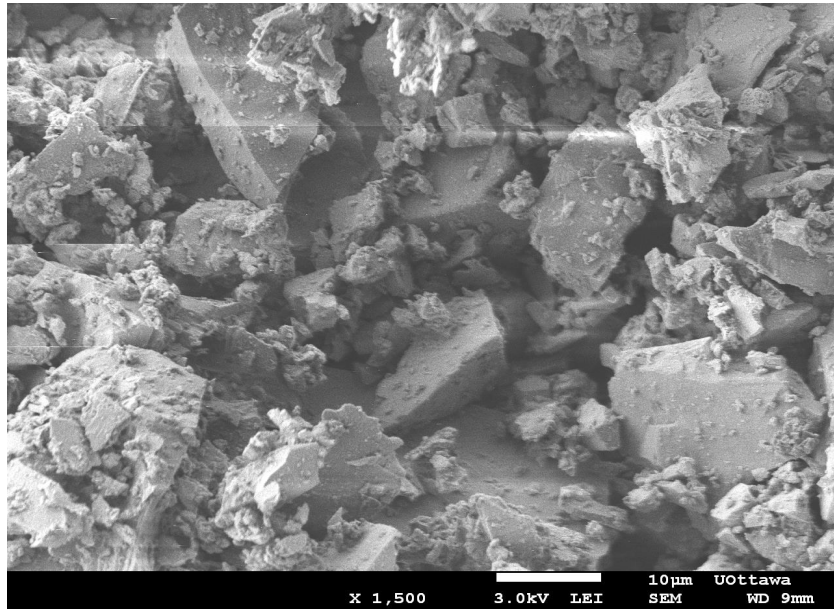


Figure 4.5: SEM image of the microstructure of a 90-day FCPB sample cured at -6°C (1500 times magnification).

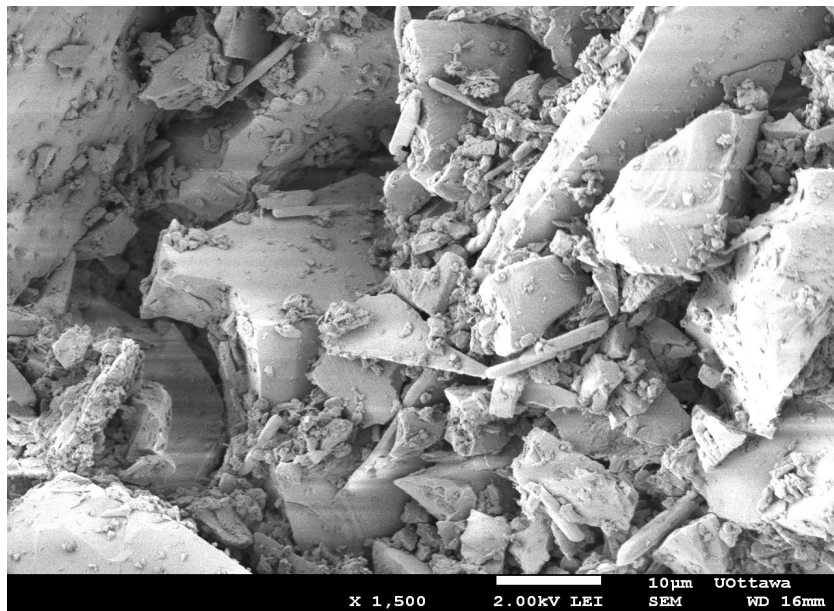


Figure 4.6: SEM image of the microstructure of a 90-day SFCPB sample cured at -6°C (1500 times magnification).

4.3.4 Unfrozen water content evolution monitoring of FCPB samples by 5 TE soil sensors.

Figure 4.7 shows data for three samples that were monitored for unfrozen water content at -6°C . The results indicate that the total unfrozen water contents were in the range of 3% to 5% cm^3/cm^3 , which corresponds with values obtained for silty soils at around the same temperature [122].

FPB exhibits the lowest volumetric unfrozen water content of all three samples. As soon as the temperature dropped below 0°C , the unfrozen water content decreased sharply from 34.2% cm^3/cm^3 to one-tenth the total amount of water, and subsequently decreased gradually. After 36 hours of curing, the value stabilized around 3.5% cm^3/cm^3 . FCPB follows a similar dropping pattern. Starting out with a volumetric water content (VWC) of 51.2% cm^3/cm^3 , FCPB displays a dramatic drop in VWC as approaching 36 hours of curing, and a stable VWC thereafter. Eventually, the amount is fixed at around 5.78% cm^3/cm^3 . The results are similar for the VWC of SFCPB, in which there is a severe fall from 44.54% cm^3/cm^3 to 5.18 cm^3/cm^3 at the beginning and a stabilizing trend towards the end.

However, in the analysis of these three curves, a number of suspicious points cannot be ignored. First, the initial water contents for the three samples, which are supposed to be in a similar range, turned out otherwise. It can be seen clearly from Figure 4.7 that initial water content values vary from 34.2% to 51.2% cm^3/cm^3 . Considering that the eventual water contents fluctuated merely between 3% and 6% cm^3/cm^3 , this difference at the beginning could cause a huge interruption in the picture of the development of ice formation. Second, the unfrozen water content for all three curves were approaching stabilized after 36 hours of curing or less, which did not correspond with the expectation that the strength of the FCPB increases gradually accompanied by a reduction in unfrozen water content. Earlier in this thesis, it was observed in UCS tests that the FCPB samples exhibited an increasing trend in terms of compressive strength with curing time. Further studies on stress-strain behaviour, microstructure, and material identification generated the realization that strength is mainly attributed to the growth of ice formations. This

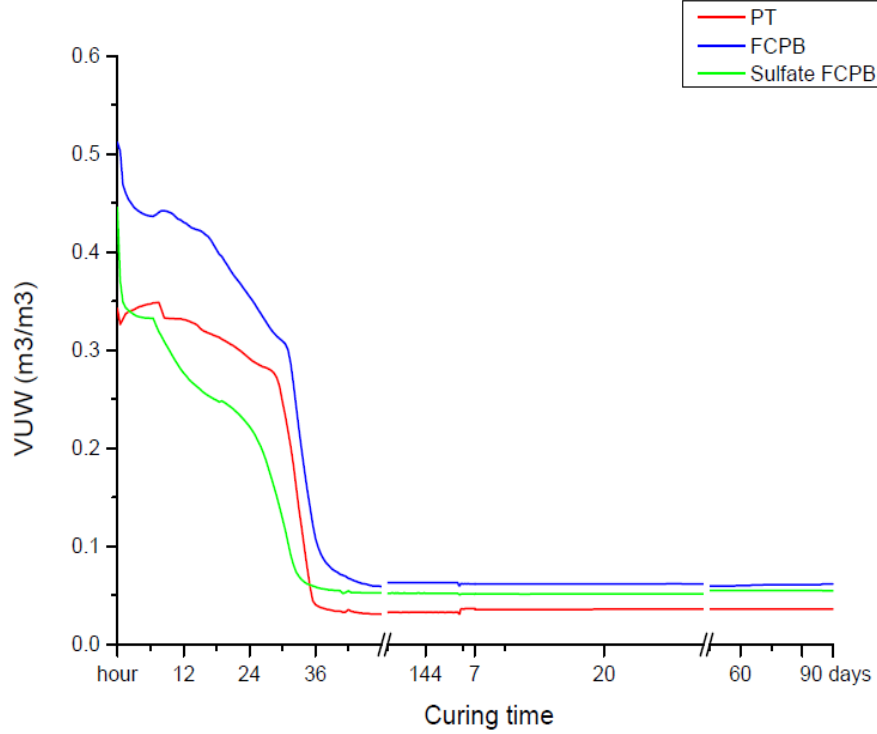


Figure 4.7: Unfrozen water evolution for the three FCPB samples (PT, FCPB, and SFCPB) at -6°C up to 90 days.

conclusion is consistent with most studies dealing with frozen soil or FCPB, but was not adopted for use in this study.

Having established that fact, a background check using 5TE sensors was conducted; it has been found that 5TE sensors have an accuracy roughly between $\pm 3\% \text{ cm}^3/\text{cm}^3$ for volumetric water content. As the value is under $5\% \text{ cm}^3/\text{cm}^3$, it is reasonable to believe that results based on a $\pm 3\%$ accuracy are not suitable and could be misleading. In addition, dissolved solids and pore fluid chemistry are found interferential for the performance of 5TE sensors. Even though 5TE sensors claim viability as long as long as the electrical conductivity (EC) of the soil smaller than $10\text{Sd}/\text{cm}^3$, this was not the case in the present study. A subsequent parallel experiment suggested that initial water content has a keen response to the EC value; as the EC value rose, the initial water content increased, even though the EC value was maintained under $10\text{Sd}/\text{cm}^3$. This is achieved by adding varying amounts of sodium chloride to create an EC gradient. Apart from that, the 5TE sensors

might have damaged the ambient environment around their probes, and hence miscalculated the unfrozen water content. The unfrozen water content remaining in frozen soil is called super-cooled water, which only exists in the capillary pores or the periphery of small particles [123] due to surface tension and other phenomena. The problem is that inserting 5TE probes into samples drastically changes the ambient atmosphere, and therefore, destroys the habitat for super-cooled water.

Though we do so rather reluctant, it is necessary to point out that 5TE sensors seem not to have been the best option for monitoring unfrozen water content in this study. More reliable techniques are recommended, such as Time Domain Reflectometry, Nuclear Magnetic Resonance, and Frequency Domain Reflectometry, which have been widely used and are commonly accepted as measurement methods for unfrozen water content.

4.3.5 Sulphate-induced formation of visible ice lenses in the SFCPB sample and its potential effects

The purpose of adding ferrous sulphates into SFCPB was originally to study sulphate attack on cement hydration products. However, as it has been demonstrated earlier in this study that cement hydration was tremendously inhibited due to the low temperature (-6°C), the effect of sulphate attack is considered inconsequential since it only occurs on the condition that the former reaction takes place. Yet, it is important to note that there is a decrease of uniaxial compressive strength corresponding to the increase of ferrous sulphates solute. This means that regardless of sulphate attack, there were some changes during the curing process that impaired the strength of the FCPB. The most distinct difference between the specimens with and without sulphates that can be perceived with the naked eye is the formation of ice lenses (see Figure 4.8). Chances are that the specimens with ferrous sulphate tended to develop visible ice lenses throughout their entire body; the more sulphates added, the more pronounced this will be. Interests and effort were devoted to finding the cause and consequence of this phenomenon. It turns out that this phenomenon is more than common, predictable, and plays an important role.



Figure 4.8: Comparison between SFCPB samples (blue and green) and FCPB samples (white).

Soil water potential and water migration in frozen soil

In order to fully understand the process of lenses formation in frozen soil, it is worth a brief digression to discuss soil water potential. The term “soil water potential” has been introduced in many studies aiming to depict water migration within frozen soil. Since water usually does not move rapidly in soil, its kinetic energy can be neglected. In this situation, water’s tendency of movement can be indicated by its soil water potential (ψ), which is defined as the amount of work water would do as it moves from its present state to the reference state [124]. Like many other physical phenomena that are subject to a high potential for change along with a potential gradient to achieve a more stable state that is of lower potential, water also moves constantly in the direction of its potential energy to decrease its soil water potential.

There are three important factors governing the soil water potential (ψ_t): gravitational soil water potential ψ_g , osmotic soil water potential ψ_o , and matric soil water ψ_m [124].

Thus, the total soil water potential can be expressed as:

$$\psi_t = \psi_g + \psi_o + \psi_m \quad (4.1)$$

The force of gravity acts on soil water as it does on all other bodies. In a soil profile, the gravitational potential (ψ_g) of water near the soil surface is always higher than ψ_g in the subsoil [124]. The osmotic potential (ψ_o) is attributable to the attraction between a water molecule and various ions (e.g., cations) and solutes (e.g., soluble salts) in the soil solution. The presence of large amounts of soluble salts results in osmotic potentials (ψ_o) that reduce soil water potential. Matric soil water potential(ψ_m), in other words, the adhesion or attraction of water to the soil matrix, varies with different soil types and external conditions. It is a function of multiple factors, such as water content, soil type, dry density, and temperature. As for frozen soil, emphasis is usually placed on the osmotic soil potentials and matric soil water potential.

The formation of ice lenses

Due to the Gibbs-Thomson effect of the confinement of liquids in pores, as well as very its high curvature, water in soil can remain liquid at a temperature that is several tens of degrees below the bulk freezing point of the liquid [125]. In addition, because water has a lower thermodynamic free energy when in bulk ice than when in the super-cooled liquid state, there is a continuous replenishment of water flowing from the warm side to the cold side of the particle. As a result, a collective amount of unfrozen water will build up and concentrate in a small area, and eventually become big enough to break the confinement and start freezing. As soon as ice segregation occurs, intermolecular forces will cause premelted fluid to migrate via capillary action and supply segregated ice growth [126], and thus bigger ice lenses will form.

The role of sulphate

When conditions allow a lens to form, water is drawn toward it. If the fluid supply is sufficient and rapid, the lens grows faster and develops well, otherwise, it is hindered [126]. Sulphate added to FCPB facilitates the formation of ice lenses in two ways:

1. It assures a sufficient water supply for ice by increasing the unfrozen water content due to freezing point depression.
2. It nurtures a favourable environment for water migration within frozen soil by strengthening osmotic soil water potential.

The growth of ice lenses requires a sustained water supply to the freezing front via capillary action. Such amounts of unfrozen water can be easily obtained from FCPB in the presence of ferrous sulphates. This is because ferrous sulphate, as one of salts once dissolved in the solute, is able to lower the chemical potential of water without much changing the chemical potential of the ice [127]. That means that in an ice-water co-existence system, water molecules are more stable in the liquid phase than in the solid phase. This phenomenon can best be explained by Gibbs free energy equation:

$$\mu_{solutionwater} = \mu_{purewater} + RT \ln X_{solutionwater} \quad (4.2)$$

Where R is the gas law constant (8.314 J/mol K), T is the temperature, and $x_{solutionwater}$ is the mole fraction of water in the solution. As salt is added to the water, the concentration of water in the solution decreases, making $x_{solutionwater}$ less than one. The natural log of a number less than one is negative, so the chemical potential of the water will drop. This mechanism is also known as freezing point depression.

A favorable environment for water migration created by ferrous sulphate comes from the enhancement of the osmotic soil water potential (ψ_o). In the case of soil containing dissolved salts in pore water, osmotic pressure and a corresponding osmotic suction will occur. The total soil suction for water containing dissolved salts is the sum of the matric suction h_m

related only to pure water with osmotic suction h_{osm} . Generally, the osmotic potential gradient is not considered a significant driving force for water migration in a saturated soil with plentiful water content, since it is easy to achieve an equilibrium condition after a period of time elapses. However, this all changes once water starts freezing and salts separate from it. The concentration of solutes increases dramatically in the vicinity of the ice, leading to a severe gradient of osmotic soil water potential (ψ_o). Water with a lower salt concentration is sucked toward the ice to equalize the osmotic soil water potential.

4.3.6 Effect of the formation of ice lenses on SFCPB

In view of the role of sulphate in FCPB, we question whether ice lenses formed due to the presence of sulphate have any effect on the mechanical behavior of SFCPB—and if so, what are the consequences? One change that could be readily detected by visual inspection was the formation of cracks on the SFCPB samples with visible ice lenses. Figure 4.9 shows a comparison of two samples after UCS testing. A is a sample without any ice lenses. After being subjected to a 15% strain, it exhibits perfect ductile deformation with no cracks detected. Sample B has legible ice lenses; it is obvious that cracks appeared along the ice lenses after the same degree of compression. The implication of these two utterly different responses is that large ice lenses are capable of turning a ductile failure into a brittle one.

Previous studies offer a large number of examples of similar transitions associated with frozen soil. In Section 3.3.1 of the current thesis, it has been well documented in the study of “the effect of water content on the strength of FCPB”. To illustrate, let us use the extreme example of frozen soil and an ice cube under compression. Even though creep behaviour is found rather prominently on both cases, it is expressed differently. For frozen soil in a homogeneous condition, a discontinuous ice matrix often goes through processes such as breaking, melting, moving, and reforming. The point is that as long as internal pressure is allowed to be released in time, crack-free creep behaviour will appear [48]. This is easy to achieve because frozen soil is characterized as a Kelvin-Voigt element that has a buffer effect against sudden stress. However, this does not apply to ice aggregates. With respect to ice aggregates, which are highly anisotropic, a brittle failure is more likely to be found

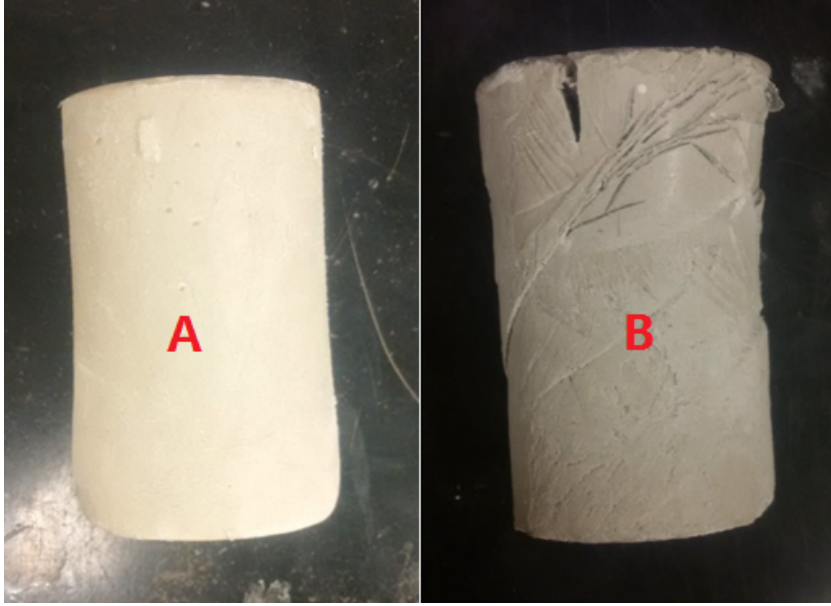


Figure 4.9: Comparison of appearance between two samples after UCS test (A: sample without ice lenses; B: sample with ice lenses).

during compression. This is because the deformation of ice aggregates can be accomplished in two ways: basal slip and non-basal slip. Basal slip refers to the displacement of ice grains along basal planes (0001). It has been reported that the critical resolved shear stress for non-basal slip is 60 times or more greater than that for basal slip [48]. This means that anisotropy will lead to a build-up of internal stresses on the scale of the grain size for ice aggregates when they are under stress. Unless time is allowed for the internal stresses to relax, plastic flow will initiate cracks. If the cracks are tolerated, the ice will exhibit macroscopically ductile behaviour. If not, the material will exhibit macroscopically brittle behavior [48]. In a scenario where both cases co-exist in one system, that is, an SFCPB with large ice lenses, what is more than likely to happen is that Kelvin-Voigt element-like SFCPB addresses stress very well by buffering it with a “damper ” and absorbing it with a “spring ”. On the other hand, ice lenses break easily due to their high anisotropy.

Even though cracks were discovered in certain samples, especially with high frequency in those using sulphate, most of them were not found to cause any mentionable strength loss and thus were excluded as being responsible for the lower compression strength

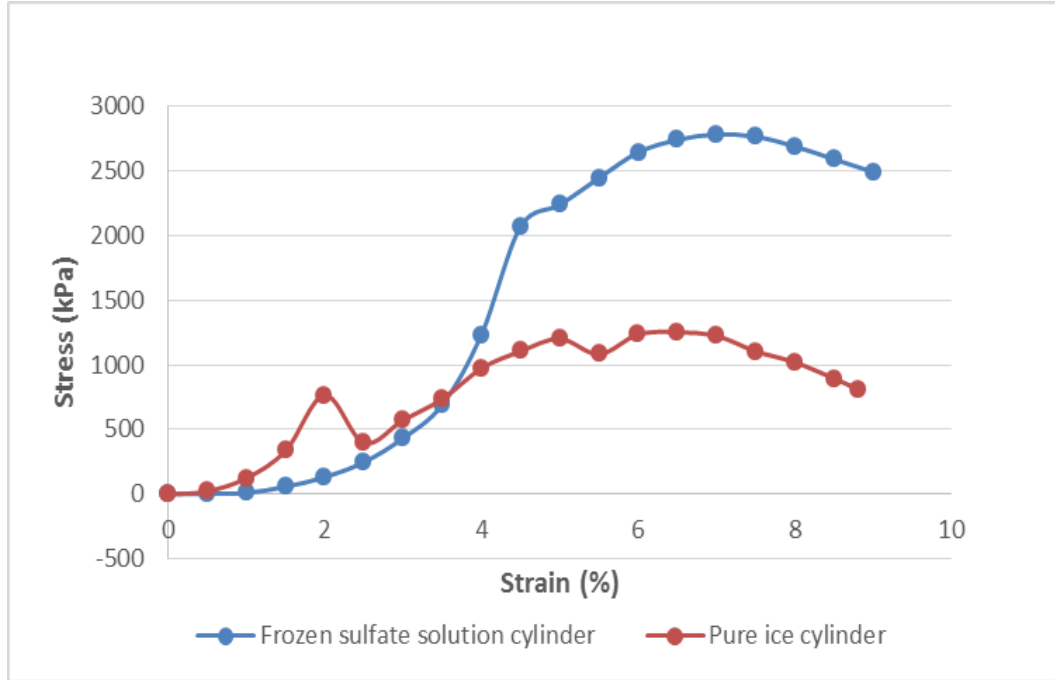


Figure 4.10: Stress-strain behavior of pure ice and frozen ferrous sulphate solution cured for 40 days.

found in SFCPB. Then again, sulphate's influence on SFCPB cannot be absolutely ruled out. There is reason to believe that as the deformation rate accelerates, or at higher strains, the effect of ice lenses on SFCPB will manifest strongly.

Figure 4.10 presents the UCS results of a set of complementary parallel experiments. Specimens made with distilled water and specimens made with ferrous sulphates solution (25,000 ppm) were tested after 40 days of curing. The differences in the stress-strain behaviour between sample A and sample B are obvious. The deformation curve of sample A exhibits multiple peak waves followed by sudden drops. This is due to the crack-caused brittle failure of the ice and the overall weakening effect. Curve B shows a smoother deformation development and a single strength because only one crack occurred during compression. Setting aside the factors causing the pure ice cylinder to be more brittle, it is easy to determine that the appearance of cracks on the pure ice sample during compression significantly compromised the strength of the samples tested.



Figure 4.11: A cylinder of pure ice and a cylinder of ferrous sulphate solution samples after UCS testing.

4.4 Conclusion

In this chapter, research was conducted on the effect of sulphate on FCPB. The parameters investigated represented conditions that are likely to be encountered at mining facilities. The derived laboratory results can be utilized to understand the behaviour of FCPB in sulphate-rich environments. These results are presented below:

- UCS tests were conducted on SFCPB samples with different sulphate concentrations (0 ppm, 5,000 ppm, 15,000 ppm, and 25,000 ppm). It was found that the presence of sulphate can lead to a direct loss of initial and peak strength. The reasons for this loss are believed to have not much to do with sulphate attack, but increasing water content caused by melting point depression. The influence of binder type is considered inconsequential as cement hydration was largely inhibited.
- Results of HC tests on sulphate FCPB showed that the formation of ice in SFCPB severely altered its micro-structure, making it extremely coarse and loose. Thus, water can easily flow through it. Moreover, sulphate attack will begin once the

temperature becomes favorable and aggravate the situation by creating short-cuts via cracks.

- SEM images provided an inside look into how the freezing process modifies the geotechnical properties of CPB. Substantial discontinuities, enlarged pores, and cracks were noted in the cross-sections of the FCPB and SFCPB samples, readily differentiating them from the CPB samples that were cured at room temperature. All these features are evidential changes brought about by ice formation in the CPB, which correspond with the characteristics of high HC. The resemblance between the samples with and without sulphate, as well as the appearance of debris, on the other hand, confirmed that cement contributes very little to overall strength.
- The evolution of unfrozen water content, as monitored by the 5TE sensors, showed that ice forms quickly once the temperature drops below 0°C. Eventually, the unfrozen water content came to rest in the range of 3-6% cm^3/cm^3 after mere 2.5 days of curing. However, this finding did not correspond with the strength composition of the FCPB, nor its development with time. This was most likely due to the use of 5TE sensors that did not meet the degree precision requirements, and were not suitable for working at high EC conditions.
- Legible ice lenses were commonly found on the SFCPB samples due to an alteration in the soil water potential and an abundant supply of unfrozen water. Such features induced a ductile-to-brittle failure mode transition in the SFCPB samples, and may have lowered the strength at higher strain rates or strains.

Chapter 5

Summary and Conclusion

5.1 Comparison between FCPB and SFPCPB

The mechanical behaviour of FCPB and SFPCPB and their relative influential factors were studied in technical papers I and II. As expected, the two studies share a great deal of resemblance and similarities; yet, they also vary. For a better understanding of both FCPB and SFPCPB, in the ensuing discussion, a brief comparison between the two papers is given to highlight the similarities and bridge the gap between the two studies.

It is understood that both FCPB and SFPCPB display extraordinarily high compressive strength compared to that of CPB, and both FCPB and SFPCPB characterize strain-hardening stress-strain ductile behaviour. This is because the governing mechanism for the frozen paste is an ice-strengthening effect, which depends only on temperature and water content. Conversely, binder type and binder content were found to exert little effect on strength development due to the disturbed cement hydration. Results from microstructure experiments such as MIP, SEM, and HC are consistent with the findings above. HC tests performed on both FCPB and SFPCPB proved that the formation of ice grains enlarged the pore space, creating a coarse structure. SEM conducted for technical paper II including all three types of samples (CPB, FCPB and SFPCPB) offers a depiction of the re-arrangement of soil particles caused by the formation of ice and traces of cement products. However, in both the FCPB and SFPCPB samples, the soil particles were pushed away from each

other by ice aggregate, leaving similar vacancy. Evidence of cement hydration products was lacking. MIP conducted in technical paper I shows a significant increase in the pore volume of FCPB between 0.003 and 0.7 μm , which fits the size of the pre-melted films that were enclosed within the small pores or around the surfaces of tiny particles. Though it was only performed on the FCPB sample, it is reasonable to assume the same applies to SFCPB.

As for dissimilitude, the biggest concern regards the legible ice lenses forming within SFCPB samples. The reasons for this are involved with the change in soil-to-water potential and increasing unfrozen water content for sufficient water supply.

5.2 Summary and conclusion

In this thesis, investigations on FCPB and SFCPB were conducted to test the viability of performing CPB in sub-zero environments. UCS tests were used to approximate the strength development of FCPB and SFCPB. Parameters such as binder type, binder content, water content, and sulphate concentration were studied, which are likely to be encountered in practical situations. Various techniques were applied, including TGA, MIP, HC, and SEM, to not only find the mechanisms responsible for the derived results from UCS tests, but also to obtain a thorough comprehension about the mechanical behaviours and properties of FCPB and SFCPB. The summary is presented below:

- Regardless of the various mix compositions adopted, both FCPB and SFCPB exhibit remarkably stronger strengths than CPB in general. It was also discovered that they gain strength directly from ice formation rather than cement hydration, making the process rapid. For the same reason, the mechanical behaviours of FCPB and SFCPB were very much identical to frozen soil rather than concrete materials, and ductile behaviour represents most of the deformation.
- Because of the low curing temperature, cement hydration and pozzolanic reaction were severely inhibited. Therefore, the influence of factors such as cement content,

binder type, and sulphate concentration were eliminated. This is further proved by TGA. Results show that, despite curing age, samples contained very similar compositions, implying there was no change or reaction during curing time. However, water content played an important role in the strength development of FCPB. As a governing factor, it is capable of changing the failure mode of FCPB from brittle to ductile based on volume and temperature.

- Although sulphate attack was not discovered during the curing period, the presence of sulfates is proven to have an adverse impact on FCPB. The loss of initial strength and peak strength indicate a softening effect on the ice matrix caused by the increasing amount of unfrozen water. Sulphates are also believed to be responsible for the formation of large ice lenses that could give rise to a sudden drop of strength at a late strain.
- The temperature monitoring of six different samples demonstrated that the temperature dropped quickly and the ice initiated on the very first day of curing. This fits the observed acceleration of strength on strength growth and inertness among the chemical reactions during curing. However, it also shatters the hypothesis that cement hydration produces an amount of heat that lowers the strength of the ice matrix.
- SEM, MIP, and HC tests confirmed that geotechnical changes occurred within back-fill matrixes as a result of the formation of ice aggregates. Unlike CPB or concrete materials, FCPB and SFCPB exhibit much coarser micro-structures. Such a change does not compromise the strength but otherwises. However, when a cycle of freezing and thawing is initiated, the durability of FCPB or SFCPB would dramatically decrease.
- 5TE sensors provided additional information about the evolution of unfrozen water content. However, they were found to not perfectly match the expectations of the current study. Further investigation suggests that 5TE sensors are not suitable in this case due to their low accuracy and high EC value. Therefore, a better approach

is suggested.

- Vialov's rheological model can be successfully utilized to depict the physical process of the stress-strain behavior of FCPB and SFCPB. Thus, it is suggested to be extended for FCPB and SFCPB.

5.3 Recommendations for further research

Now that it has been classified as a type of frozen soil, it seems necessary for FCPB to go through certain routine tests related to the study of frozen soils. For example, the creep test should be conducted first to gain a more elaborate understanding of the deformation and rheological characteristics of FCPB. Moreover, testing conditions such as temperature, strain rate, and confining pressure ought to be considered as main factors governing the behaviour of FCPB. Previous research has already proved that the behaviours of frozen soils are very dependent on those testing conditions. In addition, a thorough understanding of the thermal properties of FCPB is beneficial to improving FCPB performance in practical applications not only in strength development but also in scheduling and shift arrangement. However, it is no harm in using the improvements determined by a few experiments. For instance, by stripping off the remaining water content within testing specimens before them drying in the oven, the credibility of the TGA, SEM, and HC tests largely increase. New methods are being suggested for unfrozen water content monitoring, and more information is expected to be revealed from them. A volumetric stress-strain relationship and the cross-section deformation of FCPB is able to provide helpful parameters in modeling that may not be available otherwise. It is also a good way to combine Vialov's rheological theory for further exploration of the mechanical behaviours and properties of FCPB and SFCPB.

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