EFFECT OF CURING TEMPERATURE ON HYDRAULIC BACKFILL PERFORMANCE WITH CONSIDERATION OF A NEW TYPE OF BINDER

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April 2010

A thesis submitted to Concordia University in partial fulfillment of the requirements of the degree of Master of Science

Your file Votre référence
ISBN: 978-0-494-67270-9
Our file Notre référence
ISBN: 978-0-494-67270-9

ABSTRACT

Mine backfilling has multiple purposes in underground mining operations, such as, improving ground support, increasing production rate and environmental issues. Optimization of such material is of importance to the industry. In order to give appropriate stiffness to the mine tailing to be used as mine backfill, different type of binders have been introduced and investigated in the past. Development of cost-effective mine waste management, including mine backfilling, is a priority for today's mining industry.

Mine backfilling has progressively been integrated into underground mining operations. The high stresses and temperature associated with mining at depth in the Canadian Shield, also requires innovative approaches to mine backfilling to withstand the loading both during and after mining operations. Not only new or modified mine fill systems are required, but also new techniques are needed to increase the speed of the mining cycle for optimizing the mining operation. The backfill must also remain stable chemically to ensure the required long term behaviour.

The major objective is the determination of the role played by sodium silicate with different binders as a new additive in strength acquisition of mixtures. Extensive laboratory testing program has been conducted to investigate the parameters affecting both the long and the short term physical, mechanical properties of such mixtures with special reference to the curing temperatures. It was found that a higher percentage of sodium silicate in binder mixture reduces final setting time as well as a warmer curing condition. A sodium silicate mixture improves strength of samples in both short and long period and also decreases the porosity of the samples. Strength of samples is not always improved by increasing curing temperature and there is an optimum curing temperature.

RÉSUMÉ

Le remblayage est utilisé dans les opérations minières souterraines pour de multiples applications, telles que l'amélioration des conditions environnementales et de soutènement, ainsi que l'augmentation du taux de production. L'optimisation de ce matériel est donc d'une grande importance pour l'industrie. Différents types de liants ont été introduits et étudiés dans le passé afin de donner la rigidité appropriée aux résidus miniers qui seront utilisés comme remblais. Le développement d'un système de gestion des déchets miniers comprenant le remblayage est une priorité pour l'industrie minière d'aujourd'hui.

Le remblayage a été progressivement intégré dans les opérations minières souterraines au fil des années. L'exploitation minière en profondeur dans le Bouclier canadien est associée à des contraintes de stress et de température élevées. Cela exige des approches innovatrices pour le remblayage des mines qui doit être en mesure de supporter les charges imposées pendant et après les opérations minières. Il ne suffit pas de développer ou de modifier des systèmes de remblayage. De nouvelles techniques sont nécessaires pour accélérer le cycle minier et ainsi optimiser l'exploitation minière. Le remblai doit également demeurer chimiquement stable afin d'assurer son comportement à long terme.

L'objectif principal est la détermination du rôle joué par la silice de sodium mélangée à différents liants comme un nouvel additif ayant un rôle dans l'acquisition de force des mélanges. Un vaste programme d'essais en laboratoire a été mené pour étudier les paramètres qui influent sur les propriétés mécaniques, à court et à long termes, de ces mélanges. Une attention particulière a été portée aux températures de curage. Il a été constaté que l'augmentation en pourcentage de la quantité de silice de sodium dans le mélange de liants diminue la force finale des échantillons. La même observation a été faite pour des températures de curage plus chaudes. Un mélange de silice de sodium améliore la force de l'échantillon à court et à long termes, et diminue également la porosité des échantillons. L'augmentation de la température de curage ne se traduit pas

toujours par une augmentation de la force des échantillons, et il a été déterminé qu'il y a une température de curage optimale.

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LIST OF ABBREVIATIONS

AM: Acid Modulus

ASTM: American Society for Testing Materials

Cc: Coefficient of curvature

Cg: Coefficient of gradation

CPB: Cement Paste Backfill

Cu: Coefficient of uniformity

E: Static Modulus of Elasticity

GGBFS: Ground Granulated Blast Furnace Slag

GS: Specific Gravity

LVDT: Linear Variable Differential Transformer

MIP: Mercury Intrusion Porosimetry

NMR: Nuclear Magnetic Resonance

OPC: Ordinary Portland Cement

PCC: Portland Composite Cement

SC: Slag and Cement

SCSS: Slag, Cement and Sodium Silicate

SEM: Scanning Electron Microscope

SRC: Sulphate Resistant Cement

UCS: Uniaxial Compressive Strength

Wt: Weight

XRF: X-Ray Fraction

ACKNOWLEDGEMENTS

I would like to take this opportunity to express my gratitude to my supervisors; Professor Nokken at Concordia University and Professor Hassani at McGill University for their unconditional guidance and life experience training. It was a pleasure for me to work with best colleagues, Mr Mehrdad Fadaeekermani, Mr Shahe Shnohorkian and Mr Mehdi Razavi during the period of this research and their comments made it easier for me to complete this work. I would like to thank my friends at laboratory and office; Mr Pejman Nekovaght, Mr Ali Ghorbani, Mr. Farzan Abbasi and Mr Sameen Ahmad for their friendly help. The support of Vale Inco, NSERC, Lafarge and National Silicate is very much appreciated.

The author wishes to express his love and gratitude to his beloved families; for their understanding and endless love, through the duration of his studies and hard times.

CHAPTER : 1 INTRODUCTION

1.1. BACKFILLING PROCEDURE

Backfill is the process of filling the gaps after ore extraction is carried out. It plays a significant role in the mining industry. Backfill provides mechanical strength and reduces the risk involved at underground slopes. Backfill composition typically consists of cement, slag, tailings, admixtures and other pozzolanic or cementitious materials which act as alternatives for cement. Various cementitious materials have been used in backfill over the years and research is still on going to find the perfect combination which is both economically and mechanically favourable. Due to the high price of cement, various alternatives such as fly ash, slag, tailings and gypsum have been investigated to reduce the costs involved in backfilling. Tailings used in cemented backfill reduce the binder amount and the cost associated with transportation and rehabilitation of waste materials. Backfill enhances the working condition, ore recovery and reduces environmental impacts of waste disposal.

There are three types of backfill as follows: slurry backfill, paste fill and rock fill. Each type has its advantages and disadvantages; hence, the selection of the type of the used backfill depends on several factors. Portland cement is commonly used as a binder in most backfills, but alternatives are being considered for partial replacement for economical and environmental reasons. There are several factors which affect the properties of backfill depending on the materials used and curing conditions.

1.2. BACKFILLING BINDER

Backfill strength depends on various factors. Different binders such as slag silica fume and fly ash enhance the strength of backfill. In general, higher binder consumption gives paste backfill higher strength. The strength of blended Portland cement pastes in which natural pozzolan is partially substituted for Portland cement is considerably less than that of Portland cement pastes at early stages of hydration. The compressive strength

of natural pozzolan blended Portland cement pastes with and without superplasticizer increases with curing time. The development of strength is influenced by the proportions of natural pozzolan and Portland cement. Binder alternatives, such as sodium silicate, also increase the compressive strength of backfill in any curing condition. Admixtures such as calcium nitrate act as setting accelerator with efficiency and this depends on the cement's chemical composition.

Curing temperature and curing age also have significant effect on the strength of backfill. For ordinary plain cement paste of same water to cement ratios, the higher the curing temperature the greater the total porosity. Curing temperature has effect on the strength and durability of backfill mixture, but it varies with the type of cements being used. The curing procedures and conditions play remarkable role on the physical strength of concrete. Increase in early curing temperature causes the hydration rate and concrete strength to increase quickly.

Slag is a by-product of iron industry composed of calcium, magnesium, aluminum silicate and glass. A mixture of slag activation with sodium silicate or water glass and slaked lime result in better reactivity, and higher strength. Several factors influence the alkali activated slag cement properties such as chemical and mineralogical composition, the amount of glassy phase of the slag and the type and concentration of the activator used. Using by-products and producing slag concrete reduce environmental damage and cost of waste disposal. Alkali activated slag not only shows higher early strength but also better corrosion resistance than normal Portland mixtures. It is also a less energy intensive process.

Over the years, research has been done to determine how to hasten the setting time of slags using different materials. Activators such as sodium silicate are found to accelerate the break up and hydration of slag and hence decrease the setting times. From previous research, it is quite obvious that the increase in curing temperature caused significant changes in setting times and workability of cement mixtures. The nature and dosage of activators play a decisive role in determining the hydration and structure

development of alkali activated cements, and thus the setting time and workability are characteristics which indicate the hardening process in its initial period.

1.3. OBJECTIVES OF RESEARCH

Different contributing factors were determined to reduce the economic costs regarding backfill and also provide the sufficient mechanical strength needed for mine stability and work place safety. The main purpose of the research is to better comprehend the contribution of sodium silicate as well as curing temperature, and curing period. Various tests were done to investigate the strength, and stress-strain properties of backfill mixture and sodium silicate effect on performance of backfill. In other word:

- Investigation of new binder and effect of curing temperatures on it.

1.4. THESIS OUTLINE

Apart from the current introduction, the thesis is divided into the following sections:

- Chapter 2 briefly reviews backfilling in mines and discusses other researchers' work.
- Chapter 3 and 4 introduces the materials that were used in this research and reviews the procedures that followed during research.
- Chapter 5 provides and discusses the results of tests.
- Chapter 6 presents conclusions.
- Chapter 7 suggests future areas to work on them.

CHAPTER : 2 LITERATURE REVIEW

2.1. INTRODUCTION

Mine backfill is a fill material used in assisting the stability of voids created due to mining operations (Potvin et al., 2005). Underground mining creates the voids which need to be filled in order to provide wall support, working surface, slope stabilization, void filler, and a method of tailings disposal. Backfill helps to reduce or eliminate acid rock drainage by making use of the tailings, and to restore the actual landscape to the greatest extent possible (Potvin et al., 2005). It results in an increase in flexibility of ore extraction and improved recovery of ore bodies. Three factors that have been influential in the development of backfill technology are cement addition, environmental pressure, and resource conservation pressure (Hassani and Archibald, 1998). The major components of fill are mill tailings, aggregate or rock, water, and binder. Due to the high cost of cement as a binder, pozzolanic materials are used to reduce the cost, which are cementitious materials such as fly ash, slag, gypsum and others (Hassani and Archibald, 1998).

Mine tailings are waste products of mining and consist mainly of ground rocks which are left over after mineral extraction. Their use in cemented paste backfill is the most innovative, economical and environmental friendly process for their disposal. The utilization of paste backfill reduces cyclical stages of mining, improves surface and underground conditions, speeds up production, and reduces environmental costs (Hassani and Bois, 1992).

2.1.1. IMPORTANCE OF BACKFILL

Stable backfill provides better mine safety due to its local and regional support. Backfill prevents ground motion or uncontrolled deterioration, and workers can perform activities under safer conditions (Amaratunga, 1995; Razavi, 2007; Nasir and Fall, 2008).

In traditional methods, some ore is left behind as pillars to provide ground support for extracted areas. Backfill replaces the need of pillars and all the ore can be extracted. This technique has resulted in increased production and yield, especially in high grade areas and also the use of tailings in backfill reduces the land required for the disposal of waste materials. Thus, it is more environmentally favourable.

2.1.2. TYPES OF BACKFILL

Backfill can usually be classified into three types: hydraulic fill, paste fill, and rock fill. The basic properties that backfill designs require are compressive strength and permeability (Hassani and Archibald, 1998). The fill strength should be designed properly to support pillar recovery as well as the use of heavy equipment. In addition, a proper permeability for the backfill is essential for slurries in order to let the water drain and the material set to gain strength. The fill material can be divided into three categories; inert material, binding agent, and chemical additives. Tailings, waste rock, or coarse slag are used as inert materials in backfill. Portland cement, alkali activated slag, and fly ash work as binding agents required for strength development. Chemical additives such as flocculants, accelerators, and retarders are used to optimize fill permeability and flowability of the slurry (Hassani and Archibald, 1998).

2.1.2.1 Hydraulic backfill

Slurry fill or hydraulic fill includes tailings, sand, and rock which are mixed with a binder and water, and can be produced both above the surface or underground, being easily transported through a network of pipes and boreholes. Without binder addition, hydraulic fill can be used as a bulk filling material. When exposure to void spaces is expected, Portland cement, fly ash, or alkali slag is used to cement the backfill mass. The pulp density in hydraulic fill is typically less than 70% by weight.

The advantages of slurry fill are (Hassani and Archibald, 1998):

I. Simplicity to install infrastructure and function,

II. Fill quality and mixture density controlled at the main fill station,

III. De-sliming is done to increase percolation,

IV. Can be pumped, and reduction of surface waste disposal (Hassani and Archibald, 1998; Potvin et al., 2005).

Some disadvantages include (Hassani and Archibald, 1998):

I. Excess water has to be recovered,

II. Strength can be reduced in stope,

III. Slime produced during stope drainage,

IV. Bulkhead dewatering facilities required,

V. Binder washout may occur.

2.1.2.2 Paste Fill

Paste fill behaves differently from slurry backfill in that its pulp density is higher than the latter, in the range of 75% to 85%. The pulp density is based on the grain size distribution and specific gravity of solids. Paste fill uses some water for cement hydration and the rest remains within its matrix, and is not supposed to carry any bleed water. It has negligible excess water when stationary and remains as a single homogeneous product (Potvin et al., 2005). Paste fill uses total tailings gradation and can sometimes contain sand or waste rock. The fill can be transported from the surface and does not need to be dewatered (Hassani and Archibald, 1998).

Paste fill has numerous advantages (Hassani and Archibald, 1998; Potvin et al., 2005):

I. Laminar flow during pipe transportation and so increased flexibility,

II. The paste material does not accumulate inside the pipeline,

III. Cement hydration process is optimized and cement requirement is minimized while providing the required strength,

IV. Cures relatively faster than slurry fill,

V. Does not create underground mine water handling problems,

VI. Does not generate high pressures on bulkheads,

VII. Tailings are almost completely utilized as backfill.

The disadvantages of paste fill are as follows (Hassani and Archibald, 1998; Potvin et al., 2005):

I. Difficult rheology than lower density slurry fill,

II. When placed in a stope, it has a higher angle of repose,

III. Capital intensive,

IV. Improved dewatering amenities are considered necessary to enable the required concentrations for paste flow without loss of fines,

V. Requires more supervision for modern technology.

2.1.2.3 Rock Backfill

Rock fill consists of waste rock, quarried rocks, or aggregate. The waste rock or surface quarry is distributed by trucks and conveyors to the stopes. The bulking material is often combined with cemented slurry or cemented tailings to produce a cemented rock fill mass (Potvin et al., 2005). Most rock fill systems are simple and have low capital requirements unless an underground conveyor is required.

The advantages of rock fill are (Hassani and Archibald, 1998; Potvin et al., 2005):

I. Normally less capital expenditure,

II. Useful in open pit mines to reduce surface waste rock,

III. Simple preparation system,

IV. High strength can be attained when consolidated with cement,

V. No stope dewatering required.

The disadvantages of rock fill are (Hassani and Archibald, 1998; Potvin et al., 2005):

I. Relies on a combination of fill passes, mobile equipment, and at times conveyors,

II. Fill transportation can be labour intensive and have a higher operation cost,

III. Quarried rock requires crushing, transportation, surface production, and haulage which are cost significant,

IV. Tailings are only partially used and their surface disposal should be considered.

The mine fill system is decided upon depending on the fill material and binder used. They can be classified based on the raw materials used and the production and delivery methods. In this thesis the hydraulic backfill is investigated due to understanding the behaviour of hydraulic backfill and new binder in different curing conditions.

2.2. COMMON HYDRAULIC BACKFILLING MATERIALS

2.2.1. TAILINGS

Mine tailings are waste products of ore processing and consist of ground rock from which valuable minerals have been extracted (Ouellet et al., 2007). The addition of cementing materials to tailings provides stability against possible fill liquefaction,

mucking floors, and impermeable layers (Wong and Mitchell, 1982). The use of tailings in cemented backfill reduces environmental impacts and surface disposal costs.

The particle size of tailings and their shape have significant effects on the compressive strength, porosity, pumpability, and permeability of cemented backfill (Hassani and Archibald, 1998; Kesimal et al., 2004; Razavi, 2007). The finer the tailings materials, the greater the overall density of cement pastes becomes due to requirement of more cement usage (Fall et al., 2004). Tailings fineness has a significant effect on the backfill water to cement ratio. Coarse and medium tailings are more favourable for higher strength in cemented paste backfill (Fall et al., 2004).

2.2.2. BINDERS

In mining, tailings are usually used for backfill purposes since they could cause environmental pollution such as acid mine drainage. The tailings are combined with slag and cement mixtures and transformed into cemented paste backfill, lowering the cost when compared to hydraulic backfill binders such as Portland cement (Benzaazoua et al., 2005). Adding the binders to backfill helps improve the mechanical properties, which also develops mining methods in different areas, such as reducing mining cycles plus increased safety and performance for the mining industry (Geller and Udd, 1990; Hassani and Archibald, 1998).

2.2.2.1 Cement

Portland cement is by far the most important binder in terms of amount manufactured (Singh et al., 2002), and involves the grinding of raw materials (a mixture of limestone, clay, silica and iron-oxide) at 1500°C, followed by grinding of cement clinker and gypsum. It is an energy intensive process. The industry is constantly looking for better alternatives to effectively reduce the high energy and environmental costs of cement manufacture (Puertas et al., 2008). Thus, alternative materials are being tested for partial replacement for fuel, raw materials, or clinker. The process also consumes a wide variety of waste raw materials and fuels, and provides the industry with important

opportunities to mutually utilize the large quantities of by-products of other industries (Henrik et al., 2003). In order to reduce cost and improve the production of quality blended cements, blends of Portland cement clinker with mineral admixtures are being made (Singh et al., 2002). The most common blended hydraulic cement is currently the Portland cement in combination with pozzolanic additives. A pozzolan is a siliceous material that develops hydraulic cementitious properties when it reacts with calcium hydroxide and water (Henrik et al., 2003).

2.2.2.2 Slag

The utilization of waste is an attractive alternative to its disposal since it reduces both costs and environmental pollution problems. Rapidly cooled iron blast furnace slag, steel slag, phosphorus slag, copper slag, and lead slag all have cementitious properties which contribute to lower cost of production for binders due to lower energy consumption (Shi and Qian, 2000).

The most common type is the blast furnace slag that comes from the iron and steel industry. Glassy granular slag materials from molten blast furnace slag cools rapidly when immersed in water. The fast cooling causes a reduction in crystallization and converts the molten slag into small aggregate particles (Atis et al., 2007). Slag is composed of calcium, magnesium, aluminum silicate, and glass. Ground granulated blast furnace slag possesses hydraulic properties that enable its activation with alkaline reagents. A combination of slag activated with sodium silicate or water glass and slaked lime was been found to result in better reactivity, high strength, and unusual rheology (Qing-Hua and Sarkar, 1994). This yields equivalent or higher compressive strengths than with Portland cement alone (Douglas et al., 1991), which may lead to the formation of slag concretes without Portland cement and with improved properties such as higher resistance to sulphate attack.

Alkali activated slag is a new attractive binder for the cement manufacturing industry. It uses an industrial by-product, emits less carbon dioxide, and requires less energy than ordinary Portland cement. In fact, it has superior qualities such as low

hydration heat, high early strength, and better durability in adverse environments (Chang, 2003). Several factors influence the alkali activated slag cement properties such as the chemical properties of slag, mineral composition, the amount of glassy phases, and the type and concentration of the activator used (Zivica, 2006).

2.2.2.3 Sodium Silicate

Soda ash is heated with sand at high temperatures – such as 1200°C – to form various sodium silicates. Their uses in today's industry include soap and detergents, silica gel, pulp and paper, paints, agriculture, and rubber (McDonald and Thompson, 2009). Dissolved or liquid silicates are the most popular commercial forms of sodium silicate, and their solid form is used less. Sodium silicate undergoes four distinct chemical reactions: hydration/dehydration, gelation, precipitation, and surface charge modification. It acts as a film binder, matrix binder, and chemical binder (McDonald and Thompson, 2009). Sodium silicate has the diverse chemistry of bonding by forming a film and reacting with water to form a matrix (McDonald and Thompson, 2009). In the research project, sodium silicate gel was used as a binder with the other materials.

The most common problem associated and reported with the use of sodium silicate is achieving sufficient bond strength. It is sometimes beneficial to include small amount of silicate compatible surfactant (McDonald and Thompson, 2009), and Sodium silicate has been used as a slag hydration accelerator. The benefits of using pozzolanic cements as matrix binders for agglomeration is that they tend to be economical, environmentally friendly, more absorbent of liquids, and produce a highly durable product. The disadvantages of pozzolans are slower setting time and their reduced tolerance to impurities (McDonald and Thompson, 2009), which is why other necessary additives are combined to it in order to provide early strength. Pozzolans must be activated either by heat or chemically by alkali such as sodium silicate. The alkali activates the siliceous material and the silica portion contributes to the formation of calcium silicate hydrate. (McDonald and Thompson, 2009)

2.3. TEST METHODS FOR BACKFILL

2.3.1. DIRECT SHEAR TEST

The shear strength of geological materials is often represented by the Coulomb or the Mohr-Coulomb theory. Shear strength shows a linear relation with applied stress and two shear parameters are found from the graph, which are known as the cohesion intercept and the angle of shearing friction.

Different properties, such as tailings fineness and particle size, also play a role in determining the shear properties of a specimen. Under constant volume, cyclic, direct shear loading it was found that tailings with finer grains generally show a cumulative decrease in effective strength with progressive degradation of shear stiffness (Wijewickreme et al., 2005). In the direct shear test, the soils containing lower amounts of fine aggregates showed that the shear strength is reduced after reaching the peak value. On the other hand, higher fine aggregate content samples showed that the shear strength remained constant after reaching the peak strength. It was concluded that lower fine aggregate content helps the soil in exhibiting sand characteristics rather than those of clays (Kim et al., 2005). In the direct shear test, with decreasing fine aggregate content, hardening-softening behaviour takes place with low residual strength (Kim et al., 2005).

The shear stress-strain properties and shear strength parameters of the interaction between the cement paste backfill (CPB) and rock are significant in designing a safe and optimal CPB structure (Nasir and Fall, 2008). It is essential for static and dynamic stability and for building high performance cement backfill structures. The stability of CPB structures depends on many factors including the mechanical properties of the CPB and also the interface shear strength between the CPB and rock mass surrounding or near the CPB structure (Nasir and Fall, 2008). The direct shear test is conducted on specimens to investigate the shearing behaviour of materials under varying normal stresses. Knowledge of the shear strength is required by engineers whenever the structure is dependent on the soils' shearing resistance (Reddy, 1997). The direct shear test can be performed on soils under consolidated drained or undrained conditions. A predetermined

normal stress is applied on a test specimen, which is sheared by displacing one plate tangentially relative to the other at a constant rate of displacement and measuring the resulting shear force (ASTM D 6528, 2006). The test can be done on all soil materials and undisturbed, remoulded, or compacted materials (ASTM D 3080, 2006). The shear compression failure plane is controlled by the orientation of the maximum shear stress in the compression field (Wong et al., 2007). The direct shear test is used mostly to measure the flow of properties or the shear strength of granular materials (Zhang and Thornton, 2007).

2.3.2. HYDRAULIC SETTING TIME TEST

In practice, a binder should have a reasonable setting time depending on its application since the concrete or mortar made should have enough time to permit transport of the material (Chang, 2003). It is evident based on previous research that an increase in temperature causes significant changes in both setting and workability (Zivica, 2006). The partial replacement of ordinary Portland cement with Silica Fume (SF) blended cement extended the initial and final setting times at 20°C (Heikal et al., 2004). Sometimes different admixtures are added in order to solve problems associated with setting time. Problems of low early strength and slow setting of phosphorous slag cement cannot be solved until appropriate admixtures such as calcined gypsum and sodium sulphate are used (Dongxu et al., 2000).

Industrial slags have cement-like or pozzolanic properties and can be used as partial or full replacement for high-cost Portland cement. Metallurgical slags are used to partially replace Portland cement, which results in lower early strengths and longer setting times. Certain activators such as sodium silicate can accelerate the break-up of the structure and hydration of slags. Slag mortar activated with sodium carbonate has comparable setting times, strength, and shrinkage properties to Portland cement mortars (Atis et al., 2007). Sodium silicate based activators (sodium silicate and sodium hydroxide) are found to have the best performance in terms of strength development (Chang, 2003).

2.3.3. UNIAXIAL COMPRESSIVE STRENGTH (UCS) TEST

Mechanical stability is one of the most important qualities for the hardened paste backfill. The uniaxial compressive strength (UCS) of backfill is often used to investigate stability as it is a relatively inexpensive test and could be used for routine quality control programs at the mine (Fall et al., 2004). The UCS value differs for each underground mine depending on the application or function of the backfill (Fall et al., 2004). The test has been the main quantitative evaluation method for determining strength parameters such as unconfined compressive strength and static modulus of elasticity (E) for several years (Tiryaki, 2008). A strain-controlled axial load is applied on cohesive soil to determine the strength at which an unconfined cylindrical specimen of soil will fail in a simple compression test. With this method, an approximate value of the strength of cohesive soil is measured in terms of total stresses (ASTM D 2216, 2005). It is only applicable to cohesive materials which do not expel water due to compression or deformation when loading is applied. During the test, the maximum load attained per unit area at 15% axial strain is designated as the unconfined compressive strength. The shear stress is calculated to be half of the compressive stress at failure (ASTM D 2216, 2005).

2.3.4. POROSITY DETERMINATION

Mercury Intrusion Porosimetry (MIP) is used extensively in cement paste and concrete research to evaluate pore size distribution (Diamond, 2000). The most important properties such as compressive strength and durability of hardened concrete are associated with its pore structure (Kumar and Rao, 2002). The test involves the squeezing of a non-wetting fluid, i.e. mercury, into the pore structures of hardened concrete. A pressure inversely proportional to the diameter of the pore must be applied (Radim et al., 2000). MIP uses the non-wetting property and high surface tension of liquid mercury since it does not flow into small cavities, pores, or cracks spontaneously until pressure is applied. During the test process, the volume versus pressure path traversed during intrusion is not the same as that during extrusion. At any given pressure, the volume on the extrusion curve is greater than the corresponding volume on the intrusion curve (Lowell and Shields, 1984). Precise explanation of mercury intrusion data is only

possible for cylindrical pores (With and Glass, 1997). The use of mercury porosimetry is often limited to the evaluation of pore volumes or pore size distributions (Leon and Carlos, 1998). Three important parameters of MIP are porosity, mean distribution radius, and retention factor (Kumar and Rao, 2002).

Mercury intrusion porosimetry is a technique used to evaluate the microstructural pore properties of cement mixtures, the size and distribution of which help optimize the cemented backfill (Oullet et al., 2007). There are only a few other methods to find the microstructural pore properties, such as helium pycnometry, methanol pycnometry (Day, 1988), and nitrogen absorption porosimetry (Midgley and Illston, 1983), but mercury intrusion porosimetry has a reliable accuracy under normal conditions. Mercury intrusion porosimetry (MIP) is commonly used to evaluate the total porosity and pore-size distribution of different geo-materials (Oullet et al., 2007). Differences between porosity measured by MIP and that by helium pycnometry was shown only when there was proof of significant pozzolanic reaction (Day and Marsh, 1988). Porosity by helium pycnometry was able to measure only accessible porosity which was related to permeability of pastes. Direct oven drying of specimens tends to break the microstructure and remove some of the chemically bound water, and freezing-drying and solvent exchange are the two drying methods that reduce the amount of structural collapse that takes place. Measuring porosity by saturation of oven dried sample paste with methanol or propan-2-ol can be used as an alternative for MIP (Day and Marsh, 1988). High pressure mercury can damage the pore structure of blended pastes. The alcohol exchange process which is followed by a removal of alcohol is a preferable drying method if accurate pore size distributions are found (Day and Marsh, 1988).

2.4. CONSIDERING RELATIONS BETWEEN TESTS AND PARAMETERS

2.4.1. EFFECT ON THE SETTING TIME

Dongxu et al. (2000) studied the influence of admixtures on the properties of phosphorous slag cement, and they were found to lengthen the setting time of cement.

The main reason for the delay in early strength development of the phosphorus slag cement is due to phosphorus decreasing the setting time. Calcium gypsum, calcined alumstone, and sodium sulphate were used as admixtures and they were found to improve the strength and the pore structure of phosphorus slag cement (Dongxu et al., 2000).

Based on a number of studies, it has been found that sodium silicate has a considerable effect on the setting characteristics of alkali activated slag cement paste. Chang (2003) performed a study on the setting characteristics of alkali activated slag pastes with sodium silicate. Several factors like pH, acid modulus (AM), and alkali activator dosage had effects on the properties of the slag paste. It was concluded that although the pH value and the acid modulus have a strong influence, they do not affect the overall setting time. In fact, the activator dosage was the factor that interfered significantly with the setting time and over dosage may effect negatively. The results also showed that SiO_2 had more influence on the setting time than Na_2O. Phosphorous acid which was used as a retarder was found to have a strong retarding effect as well (Chang, 2003).

Goberis and Antonovich (2004) investigated the influence of the amount of sodium silicate on the setting time using a complex binder made up of high aluminate cement, liquid glass, and metallurgical slag. It was concluded that the first and second exothermal stages of hydration largely depend on the amount of sodium silicate.

2.4.2. CORRELATION BETWEEN SETTING TIME AND UCS VALUE

The Vicat and Unconfined Compressive Strength tests are useful tools to study strength development in cemented backfills (Klein and Simon, 2006). Cemented paste with sufficient strength at a very early age is vital in most situations. Concrete with the above attributes can be obtained by using admixtures, and setting and hardening accelerators (Aggoun et al., 2008). The strength development in cemented backfill is sensitive to paste composition. Higher strengths can be obtained at early stages with the use of silica fume, and the acceleration observed in Portland cement hydration is due to the ultra fine particles in this additive (Colak, 2002).

An increase in the setting times of mixtures can be observed with an increase in the natural pozzolan content, and the effect is noticeable when large amounts are used (Colak, 2002). The strength of blended Portland cement pastes in which natural pozzolans are partially substituted is considerably less than that of Portland cement pastes at early stages of hydration. The compressive strength of Portland cement pastes blended with pozzolans – with and without super plasticizer – increases with curing time. The development of strength is influenced by the proportions of natural pozzolans and Portland cement. Admixtures such as calcium nitrate act efficiently as a setting accelerator which depends on the cement's chemical composition. Its effect on the long term strength increase was not significant and therefore, it is not considered a hardening accelerator (Aggoun et al., 2008). Triethanolamine and tri-isopropanolamine perform well as hardening accelerators at all ages regardless of the type of cement. A combination of calcium nitrate with either triethanolamine or tri-isopropanolamine reduces the initial and final setting times, and increases the strength of cement pastes at all ages (Aggoun et al., 2008). For strength increase purposes, tri-isopropanolamine is more efficient than triethanolamine.

The setting time of cement backfill is lengthened with a decrease in the cement content of the specimens as found by some researchers, and this is due to the fact that smaller amounts of cement hydrates are formed as its percentage decreases (Klein and Simon, 2006). Calcium chloride and sodium chloride decrease the initial time of setting as compared with that of the controlled paste without any additives. The strength of cement backfill containing calcium and sodium chloride was found to be greater at 28 days of curing, but had little effect on long term strength development (Klein and Simon, 2006). Superplasticizers affect the setting time and unconfined compressive strengths (UCS) of cemented backfill depending on their type. Backfill containing polycarboxylated acrylic acid based polymer was observed to develop the highest UCS value after a year and had the most rapid stiffness development compared to specimens without the admixtures (Klein and Simon, 2006). Other author found that the addition of fly ash increases the UCS value by a small margin after a year of curing when compared to the controlled paste, but the effect of fly ash addition on strength development is most remarkable during the advanced stages of hydration (Colak, 2002).

2.4.2.1 Considering Setting Time and Strength on Concrete Samples

The setting time of concrete increases with increasing sulphate concentration until it reaches an optimum point (Kumar and Rao, 1994). The behaviour is more obvious for the initial setting time than for the final setting time for the same ion concentrations. The cations of the sulphate solution influence the degree of sulphate attack and the effect of sodium sulphate on the setting time of concrete was found to be the most influential among four sulphate solutions. Compressive strength decreases as a function of sulphate concentration and period of exposure due to sulphate attack. The effect of magnesium sulphate on the compressive strength was found to be most severe (Kumar and Rao, 1994), and highly concentrated sodium sulphate solution was seen to be very effective in reducing the strength of blended Portland cement pastes (Colak, 2002).

An investigation on setting time was carried out to determine the effect of metal oxides of iron (III), zinc, chromium (III) and lead (Olmo and Irabien, 2001). Zinc oxide was found to strongly retard the initial and final setting times compared to normal cement. It also decreases the UCS of final product at short sample ages and the effect of it decreases as the sample age increases (Olmo and Irabien, 2001). Iron (III) oxide decreases the long term UCS values but does not affect the setting time and UCS of short term samples. Chromium (III) oxide does not affect the setting time or the UCS values significantly at all. Lead oxide retards the setting time with respect to the cement but does not affect the UCS of cementitious products at long term samples like chromium (III) oxide (Olmo and Irabien, 2001).

2.4.3. PARAMETERS AFFECTING THE STRENGTH OF BACKFILL

The parameters that affect the strength of backfill can be classified into two categories:

I. Macroscopic parameters- surrounding rock mass, crack within cement paste

II. Intrinsic parameters- which include tailings, binder, water.

The strength of cemented backfill depends on various parameters such as water content, tailings, mineralogy, pozzolan use, particle size distribution, and curing temperature. For the last three decades, research has been done to optimize backfill performance and reduce the cost.

In underground mines, the bulk of cemented tailings are used as ground control backfill. Strength as found to increase with increased cement content, increased pulp density, decreased porosity, and decreased curing humidity (Mitchell and Wong, 1982). From the work done, it was found that the unconfined compressive strength of cemented tailings backfill increases with curing time and that cement curing rates are temperature-dependent (Thomas, 1979). Ideally, sand backfill should be of sufficient quantity to fill all mine openings in sequence while maintaining adequate drainage and strength properties. The inclusion of too much fines causes a reduced permeability and underground water handling problems arise. Porous cement is formed at low temperature which results in higher porosity in the backfill. The cement content has a lesser effect on sample porosity than pulp density. Higher uniaxial strengths result from lower porosities due to greater particle interlocking and the fact that more cement is available per unit volume of backfill. Since cement gel grows rapidly and cement is normally added in the backfill plant with up to 5 min of pipeline transport time, the in-place porosity decreased and strength is likely to be increased significantly by maintaining a high delivery pulp density (Fall et al., 2004). Finer particles mean that there is more surface area to be wetted, which in turn yields both higher moisture levels and lower densities for a given consistency. Additionally, coarse tailings require less water than medium or fine tailings to reach the same consistency or pulp density (Fall et al., 2004).

2.4.4. EFFECT OF TAILINGS' PROPERTIES ON THE STRENGTH OF BACKFILL MIXTURE

Fall et al. (2004) studied the effects of tailings properties on the performance of paste backfill. The mechanical strength of the backfill was measured using uniaxial compressive strength (UCS) and it was found that an increase in tailings density provides the cemented backfill with a higher strength for the same binder proportion (Fall et al.,

2004). The results also showed that tailings fineness plays an important role on the water to cement ratio and pulp density.

Kesimal et al. (2005) studied the effects of tailings and binder properties on the short and long term strength and stability of cemented paste backfill. The experiment was carried out on two different sulphide tailings and two different Portland cement-based binders, and their UCS values were measured in the short and long terms by testing the samples at 28, 180, and 360 days. The results indicated that the physical, chemical, and mineralogical properties of tailings and binders play a significant role in strength development. For example, samples with higher pulp densities were seen to have higher strengths and those with mineral additives were better in short and long term performance. They also indicated that samples under sulphate and acid attacks for a long time lost strength (Kesimal et al., 2005).

The use of mine tailings reduces the binder amount and other costs associated with their transportation and rehabilitation. The use of the correct dosage and type of binder results in the maximum optimization of the paste backfill. Various cementitious materials have been used as alternatives or as partial or complete replacements for Portland cement, which was initially used as the only binder. These include blast furnace slag (Douglas et al., 1991), fly ash (Maltais and Marchand, 1997), gypsum (Amaratunga, 1995), and other alkali activated slag mixtures (Qing-Hua and Sarkar, 1994).

Fall et al. (2004) investigated the mix proportioning of cemented tailings backfill. In the experiment, all the performance properties such as UCS, slump, solid concentration, and cost were optimized. The modeling results were similar to those performed by previous authors (Amaratunga, 1995; Hassani and Archibald, 1998; Fall et al., 2004). To optimize the performance, the factors affecting CPB properties and main components such as cement, binder, tailings, grain size, density, mixing water, and water to cement ratio was investigated. The results showed that the cement content, tailings fineness, water to cement ratio, and tailings density significantly affected the performance properties of CPB.

An investigation by Kesimal et al. (2009) showed the influence of binder type and dosage on the mechanical and microstructural properties of cemented paste backfill (CPB). In an investigation the tailings used contained a high percentage of sulphate and the binders used were ordinary Portland cement (OPC), Portland composite cement (PCC), and sulphate resistant cement (SRC). The CPB samples of OPC and PCC decreased in strength after 56 days. This might be due to attack on hydration products by sulphate and acid generated internally by the oxidation due to the presence of pyrite. The long term mechanical strength was attained only when sulphate resistant cements were used or SRC was blended with OPC (Kesimal et al., 2009). Increasing the binder dosage improved the UCS of CPB samples up to a certain percentage without any loss in strength. Decreasing the water to cement ratio benefitted the UCS of CPB sample. The investigations provided a practical importance of binder type, dosage and water to cement ratio for the short and long term mechanical performance of CPB (Kesimal et al., 2009).

2.4.5. EFFECT OF DIFFERENT BINDERS ON STRENGTH

Generally speaking, a higher binder content results in higher strengths in cemented backfill (Fall et al., 2004).The compressive strength of composite granulated blast furnace slag and fly ash is found to be much higher than ordinary Portland cement (Singh et al., 2002). Industrial wastes such as slag, fly ash, silica fume and the like are increasingly used for concrete manufacturing. This is not only to improve the major characteristics of cement and concrete, but also to solve environmental problems (Goberis and Antonovich, 2004).

Different industrial slags can be used as partial or complete replacement of cement in backfill. Douglas et al. (1991) studied the behaviour of alkali activated ground granulated blast-furnace slag concrete. Five mixtures were studied using sodium silicate as the activator without the addition of Portland cement. The results suggest that ground granulated blast furnace slag activated with sodium silicate is ideal for manufacturing slag concretes and has satisfactory workability and strength.

Shi and Qian (2000) investigated the development of high performance cementing materials based on activated slag such as blast furnace slag, steel slag, copper slag, and phosphorus slag. They concluded that alkali activated slag not only shows higher early strength but also better corrosion resistance than normal Portland process. Furthermore, it requires a less energy intensive process for manufacturing.

The compressive strength of a composite of ordinary Portland cement, granulated blast furnace slag, and rice husk ash was found to be much higher than ordinary Portland cement (Singh et al., 2002).

2.4.6. EFFECT OF SODIUM SILICATE ON CEMENTED BACKFILL

The use of sodium silicate is growing as a replacement for phosphates in detergents as a component that does not pollute rivers and lakes. Dissolved or liquid silicates are the most popular commercial form of sodium silicate. This reagent undergoes four distinct chemical reactions: hydration/dehydration, gelation, precipitation, and surface charge modification. Sodium silicates act as film binders, matrix binders, and chemical binders (McDonald and Thompson, 2009).

The most common problem is achieving sufficient bond strength (McDonald and Thompson, 2009). Sodium silicate is used as a Portland cement accelerator. Finely divided siliceous or siliceous and aluminous materials can be defined as pozzolanic cement. The benefits of using pozzolanic cement as a binder for agglomeration are that they tend to be economical, environmentally friendly, more absorbent of liquids, and produce a highly durable product. Thus, additives are required to be added in order to give the strength required. Pozzolans must be activated either by heat or by chemical reactions with alkalis such as sodium silicate, which activates the siliceous material, and the silica portion contributes to the formation of calcium silicate hydrate. (McDonald and Thompson, 2009)

Research by McDonald and Thompson (2009) concluded that sodium silicate is used as a Portland cement accelerator. The benefits of using pozzolanic cement as matrix binder for agglomeration is that they tend to be economical, environmentally friendly,

and more absorbent of liquid and for producing a highly durable product. The disadvantages of pozzolans are: slower setting time and low tolerance to impurities like clay, silt and etc". Thus, necessary additives are added in order to give strength. Pozzolans must be activated either by heat or by chemical interaction through alkalis such as sodium silicate (Chang, 2003), sodium hydroxide, or sodium carbonate.

2.4.6.1 Considering Affect on Concrete Samples

High temperature water and steam curing are sometimes used to accelerate the rate of strength development, and the effect of these curing methods on foamed concrete has been investigated. Regardless of fly ash content, it was found that higher curing temperatures resulted in lower ultimate strengths (Kearsley and Mostert, 2005). There was a significant benefit in curing the foamed concrete at lower temperatures for more than 7 days. On the other hand, exposure of normal concrete to high early temperature has an effect on the formation of micro cracks because of the thermal expansion of air bubbles in cement pastes (Neville, 1995).

There is a higher rate of strength development in a binder when cured at 27°C in 90% humidity than other conditions (Singh and Garg, 1991). In general, at higher curing temperatures, better compressive strengths, tensile splitting strengths, and flexural strengths are obtained from pulverized fuel ash concrete, but regular Portland cement does not behave in a similar way (Balendran and Martin-Buades, 1999). Since high performance concrete has fewer pores than ordinary concrete, its strength decrease ratio is also less. Low temperature affects the high performance concrete less than ordinary concrete (Husem and Gozutok, 2004).

In previous work, it was found that despite reducing Portland cement content with increasing amounts of additives, compressive and flexural strengths remain the same after curing at high temperatures when compared to Portland cement concrete, as the active SiO_2 in the additives reacts at high temperatures with the $Ca(OH)_2$ of cement in the initial period (Ujhelyi and Ibrahim, 1990). The strength of the ground granulated blast furnace slag (GGBFS) was found to be higher than that of ordinary Portland cement control

concrete at 7, 14 and 28 days of curing. The initial rate of hydration is slower for slag cements but their strength development is eventually greater. In addition, GGBFS concretes were found to be more prone to poor curing than ordinary Portland cement concretes (Austin et al., 1992). In an investigation on sulphur concretes, it was found that when they were continuously immersed in water at different temperatures, they developed lower strengths than samples exposed to normal air conditions (Abdel-Jawad, 1993).

Researchers indicated that an elevation of the curing temperature contributes to the reduction of long term compressive strength of ordinary Portland cement mixture (Maltais and Marchand, 1997). The same work also concluded that an increase in curing temperature is much less detrimental for fly ash mixtures.

The effects of poor curing procedures were observed for both silica fume and ordinary Portland cement concrete. They were found to be equally detrimental for both in terms of strength and skin properties as drying would not be uniform and would occur more readily on the outer layers of concrete, which would also possess moisture. This could facilitate reasonable strength development, but it may not develop the dense microstructure obtained in the core. The skin properties of the concrete were determined by the depth of carbonation under accelerated conditions (Bentur and Goldman, 1989). The increase in temperature of mixtures of alkali activated slag cement and Portland cement showed acceleration of setting time and decrease of workability (Zivica, 2006).

Curing sulphate resistant cement at higher temperatures raised the early age compressive strength but had adverse effects after 15 days (Elkhadiri and Puertas, 2008) whereas cement cured below 22°C had a more gradual development of strength. On the other hand, concrete specimens made with 10% silica fume had the highest compressive strength values at all temperatures of thermal treatment with stronger binding forces and sufficient thermal stability (Saad et al., 1996).

According to other work, the rate of evaporation from freshly placed concrete surfaces depends on the time of casting, ambient conditions, the temperature of concrete, and moisture condition (Hasanain et al., 1989).

The compressive strength of samples cured by wrapping at the early stages is greater than those that are cured by dry air. Concrete specimens with mineral admixtures produced greater strengths after 7 days of curing. The acceleration of pozzolanic reactions due to the presence of admixtures such as silica fume at medium temperatures was seen to increase strength (Mohd Zain and Radin, 2000).

2.4.7. EFFECT OF CURING TEMPERATURE ON STRENGTH OF CEMENTED BACKFILL

Maltais and Marchand (1997) performed a study to investigate the influence of fly ash and curing temperature on cement hydration and compressive strength development. Two different types of fly ash were tested by changing the cement percentage and the curing temperature. It was found that fly ash increases the rate of early hydration and the elevation of curing temperature reduces the long term compressive strength. Fall and Benzaazoua (2005) analyzed the strength development and cost of underground backfill containing different amounts of sulphate. Their study demonstrated that sulphate significantly influences cemented backfill strength; in the short term sulphate helped increase strength by filling fine porosities but in the long term sulphate caused cracks and reduction of strength.

Fall and Samb (2008) investigated the effect of high temperature on strength and microstructural properties of cemented backfill. Different types of cement backfill were exposed to different high temperatures; the strength, porosity, pore size distribution, and water absorption were analyzed. It was concluded that temperatures above 200°C reduced the strength of the backfill mixture, but as it exceeded 400°C the strength decrease was dramatic.

The physical properties of concretes such as strength and durability are based on several factors which include curing temperature and the use of supplementary cementing materials (Fall and Samb, 2008). Poor curing procedures also play a role in the strength and physical properties (Bentur and Goldman, 1989). The presence of supplementary cementing materials does play major role in the hydration kinetics of cement (Maltais and

Marchand, 1997). Elkhadiri and Puertas (2008) investigated and found that the total porosity in pastes hydrated at temperatures up to 40°C decreased, and then increased at temperatures higher than 85°C. This is attributed to lower strength of mixtures cured at higher temperatures.

In another work, ordinary Portland cement and a blended silica fume cement was investigated by changing temperature parameters during the early hydration. As silica fume was added into the cement paste, it led to an increase in shear stress values. The decrease in the shear stress of cement pastes made with silica fume was found to be larger. As the temperature increased, the shear stresses decreased with an increase in the shear rate (Heikal et al., 2004).

2.4.8. MATURITY METHOD

Various factors such as temperature, relative humidity, wind speed, and solar radiation affect the development of concrete material properties. Among the above factors, the curing temperature plays the most dominant role in affecting the strength development of concrete. At any given age from the time of mixing, the strength of the concrete depends on the concrete thermal history (Topcu et al., 2007). The maturity concept is mainly used to study the combined effect of temperature and elapsed time. It was developed in 1950s and still continues to evolve because of the inherent limitations it has (Kim and Rens, 2008); these limitations are:

- Concrete must be retained in a condition that permits cement hydration.
- The method does not take into account the effects of early age concrete temperature on the long term final strength.
- The method needs to be supplemented by other indicators of the potential strength of the concrete mixture.

An accurate investigation of the early age properties of high performance concrete is important in constructions such as bridges and buildings where early cracking is a

major concern in low water to cement ratio high performance concrete, having been caused by restrained autogenous shrinkage which develops at early ages (Zhang et al., 2008). The factors which influence the early age strength gain are material properties, concrete composition, concrete replacement and compaction factor, geometric dimensions of building element, cure cycle, and curing conditions (Topcu and Topark, 2005). Several types of fine aggregates are used in concrete mixtures and each one changes the geometric properties of the cement paste and affects both the shell formation during heat treatment and the properties of concrete (Topcu and Topark, 2005). The maturity method suggests that if the same specimens are kept under different curing conditions, strength estimations can be done with strength-maturity relationships regarding the temperature history of the concrete specimens. Tensile strength and modulus of elasticity are two important properties that influence the early age cracking (Zhang et al., 2008).

At an early age, thermal deformations and autogenous shrinkage occur together and may lead to cracking if not restrained. The thermal deformations mentioned occur due to the temperature rise caused by the hydration reactions and are proportional to the thermal dilation coefficient of the cement paste (Turcry, 2002). Autogenous shrinkage is the result of chemical shrinkage since the hydration products are denser than the unhydrated compounds from which they are produced. Hydration reactions occur through the consumption of capillary water, leading to an increase in the tensile stress in pore water. This leads to a global compressive stress on the solid skeleton, which results in deformation (Turcry et al., 2002). In autogenous conditions, early age volume changes of cementitious materials are assumed to include two components (Mounanga et al., 2006), which are:

- Autogenous deformation related to hydration process and progressive hardening of the material.
- The thermal dilation governed by the coefficient of thermal expansion.

An increase in early curing temperature causes a rapid increase in hydration rate and concrete strength. Nevertheless, because of the non homogeneous diffusion of the

hydration products and the difference in the thermal expansion coefficients of the constituents, the porosity of the cement paste increases and leads to the formation of micro cracks. This results in a decreased strength of the product at latter stages (Topcu and Toprak, 2007).

2.4.9. DIFFERENT VARIABLE EFFECT ON POROSITY AND STRENGTH OF MIXTURES

The microstructural evolution of various cement backfill samples with silica fume was investigated using MIP by Ouellet et al. (2007) Three types of binders were used; ordinary Portland cement (OPC), OPC with fly ash, and OPC with blast furnace slag. Three types of water were added to the mixture and their effect on the microstructural properties was examined. After curing for certain periods, the MIP test was performed on the samples, and the results showed that the main peak intensity of the MIP decreased with curing time. The MIP test is sensitive enough to detect differences between porosity evolutions of various cemented backfill mixtures with 5% binder weight to total tailings weight (Ouellet et al., 2007).

Ouellet et al. (2007) performed an investigation on the microstructural evolution of different cemented paste backfill samples and evaluated it using MIP. Three different binders were used; ordinary Portland cement, ordinary Portland cement with fly ash, and ordinary Portland cement with blast furnace slag. Three types of water – one de-ionized and two sulphated – were used and their curing times were studied. UCS tests were performed to relate them with the MIP results, which showed that the slag based binder with high sulphate mixing water showed the highest percentage of fine pores and highest strength (Ouellet et al., 2007).

Mineral additives have positive effect on strength increase and porosity reduction; what is really remarkable is that higher percentage of fly ash cannot be activated with cement and this binder's sample has lower strength and higher porosity even after 90 days curing (Li et al., 2006).

The threshold diameter decreases with curing time for all mixtures, and indicates a decrease in pore size in the main pore network. For different types of water used during the curing period, the cemented paste samples made with sulphated water showed higher UCS results and had smaller threshold diameter. Cemented backfills which were made with binder a mix 20:80 of T10 and blast furnace slag (T10SL) showed the highest percentage of fine pores and the highest UCS values (Ouellet et al., 2007). The high w/c ratio and the low binder percentages and the hydration/precipitation phenomena do not significantly affect the total porosity of cemented backfill (Ouellet et al., 2007). Despite a constant MIP total porosity, the MIP pore size distribution is affected by different factors such as curing time, binder type, proportion, and water quality (Ouellet et al., 2007). The water to cement ratio of hardened cement paste affects the microstructural properties and also changes them during maturing (Midgley and Illston, 1983). At high water to cement ratios it was found that very large pores are formed at early ages. Cook and Hover (1999) concluded after their research of MIP on hardened cement paste that the longer curing times and higher water to cement ratios gave greater degrees of hydration. It was also found that longer curing times and lower water to cement ratios resulted in lower porosity values (Cook and Hover, 1999). Longer curing times and lower water to cement ratios also result in lower threshold pore width values.

The curing temperature has a significant role in determining the pore structure of hydrated cement paste. Plain cement of equal water to cement ratio, for the same degree of hydration but with higher curing temperature has a greater total porosity (Kjellsen et al., 1990). In research conducted by Escalante-Garcia and Sharp (1998), hydration of two Portland cements at five different temperatures was investigated. X-ray diffraction, thermogravimetry, scanning electron microscopy, and compressive strengths were applied for all the samples. As the temperature increased, the hydration initially accelerated. However, on the long run, the degree of hydration was reduced and the compressive strength decreased with increased porosity (Escalante-Garcia and Sharp, 1998).

Recently, Fall and Samb (2008) investigated the influence of high temperatures on the strength and microstructure of cemented backfill. The results showed that high

temperatures play a significant role on the properties of cemented backfill. In general, up to 200°C, increasing the temperature gives higher strength in most backfill mixtures. Above 200°C, temperature increases reduce the strength of the CPBs, and at 400°C, the change is the most drastic and strength decreases notably. This decrease in strength is accompanied by changes in porosity, pore size distribution, and mineral phases. The water to cement ratio and the tailings type also play vital roles on the strength and microstructure at high temperatures (Fall and Samb, 2008).

For hardened Portland cement, it was found that at elevated temperatures there is a change in the hardened cement microstructure. MIP and SEM were performed on samples and it was observed that as temperature increased, the pore structure coarsening is intensified (Peng and Huang, 2008).

Elkhadiri and Puertas (2007) used several techniques to study the effect of curing temperature on sulphate resistant cement. In order to study the hydration process, they looked at the following parameters; compressive strength, thermogravimetry, x-ray diffraction data, MIP, electron imaging data, and nuclear magnetic resonance (NMR) data. The test results showed that higher temperatures accelerated the hydration reactions. The total porosity in pastes cured at higher temperatures was low during the early curing ages. Research showed that curing sulphate resistant cement at higher temperatures of about 85°C increased the early age compressive strengths, and the total porosity of the pastes cured at higher temperatures was small in the beginning. However, total porosity decreased with time in pastes hydrated at temperatures until 40°C and for materials cured at 85°C, the total porosity rose about 20% after 28 days of hydration (Elkhadiri and Puertas, 2007).

The long term compressive strength and degree of hydration was seen to decrease for Portland cement as the temperature increased, but the porosity increased (Escalante-Garcia and Sharp, 1998).

Recently, limestone is widely being used in blended cements to reduce energy and raw material consumption. One of the most important cement properties is the pore structure. In a recent research it was found that limestone addition affects the pore

structure of the cement paste as it increases the size of capillary pores linearly when the maximum amount of limestone is used (Pipilikaki and Beazi-Katsioto, 2009). The threshold diameter decreases exponentially and limestone also diminishes the size of gel pores due to higher hydration rates (Pipilikaki and Beazi-Katsioto, 2009). It was concluded that limestone can be used in cementitious materials and is structurally adequate for constructions.

It should be emphasized that drainage plays important role in case of porosity, as Fall et al. (2005) were investigated this fact in their experiment. They found out the sample with drainage possibility in their curing period have lower cumulative porosity and also smaller porous diameter. This phenomenon is concluded as letting the material settle down during drainage of samples (Fall et al., 2005).

2.4.9.1 Considering MIP on Concrete Samples

Li et al. (2006) performed MIP studies on ordinary Portland cement pastes with 10% to 40% mineral additives, which included steel-making slag, granulated blast furnace slag, and fly ash. The porosity and compressive strength for all the samples were determined at 3, 7, 28, 90 and 180 days. It was found that the development of micro pore structure of OPC was delayed with the use of mineral additives (Li et al., 2006).

Collins and Sanjayan (2001) investigated the micro cracking of alkali activated slag concrete. The level of micro cracking was measured using crack-detection microscope, water sorptivity test, and mercury intrusion porosimetry (MIP). The results illustrated that the lack of moist curing of alkali activated slag cement is responsible for an increased level of micro cracking. The strength development is also reduced due to the lack of moist curing (Collins and Sanjayan, 2001).

The addition of silica fume to normal concrete leads to the use of calcium hydroxide obtained during cement hydration. Specimens containing 10% silica fume have lower porosity values at all temperatures of thermal treatment (Saad et al., 1996). Concrete with 10% silica fume also has the highest compressive strength values at all temperatures of thermal treatment.

For monitoring changes in microstructure, x-ray diffraction, MIP, and scanning electron microscope (SEM) are used. Peng and Huang (2008) used all of the above procedures to study the changes in the microstructure of hardened cement paste at elevated temperatures. The calculated hardened cement paste pore size distribution showed a coarsening effect at elevated temperatures on the pore structure, and the SEM observation confirmed it. It was concluded that the pore structure coarsening is due to the formation of equivalent cracks which are responsible for the reduction in the mechanical properties of hardened cement paste (Peng and Huang, 2008).

CHAPTER : 3 MATERIALS

3.1. INTRODUCTION

This chapter identifies the characteristics of the material used in the experiments. A series of soil and rock mechanics laboratory tests was performed using ASTM and other valid standards. Furthermore, concrete technology methods are referred to where they were applied.

3.2. MINE TAILINGS

3.2.1. PARTICLE SIZE

Most tailings particles are clay or silt sized (Wong and Mitchell, 1982), and contain different minerals depending on the mine geology. The methods of determining the particle size distribution of tailings are sieving, microscopy, sedimentation, permeametery, electrozone sensing, and laser diffraction (Rhodes, 1998). Two methods were used to determine the tailings particle sizes for this project; sieving and laser light diffraction. Based on the M.I.T. Classification System (Means and Parcher, 1964; Razavi, 2007), the tailings were classified as silt-sand. Figure 3-1 shows the particle size distribution of the material used in this investigation from the Creighton mine near Sudbury, Ontario. Different useful parameters can be obtained from this graph. The effective size, D10, is the size of amount of particles less than 10% of the total mass. The median size, D50, means that 50% of mass are smaller than that particular diameter. D50 is commonly used in the mining industry to classify tailings (Thomas, 1947), and its value for the tailings used was 45μm, meaning that 50% of the particles were smaller in diameter than this value. Also, D30 and D60 show 30% and 60% of mass passes the diameter values assigned to them from the plot.

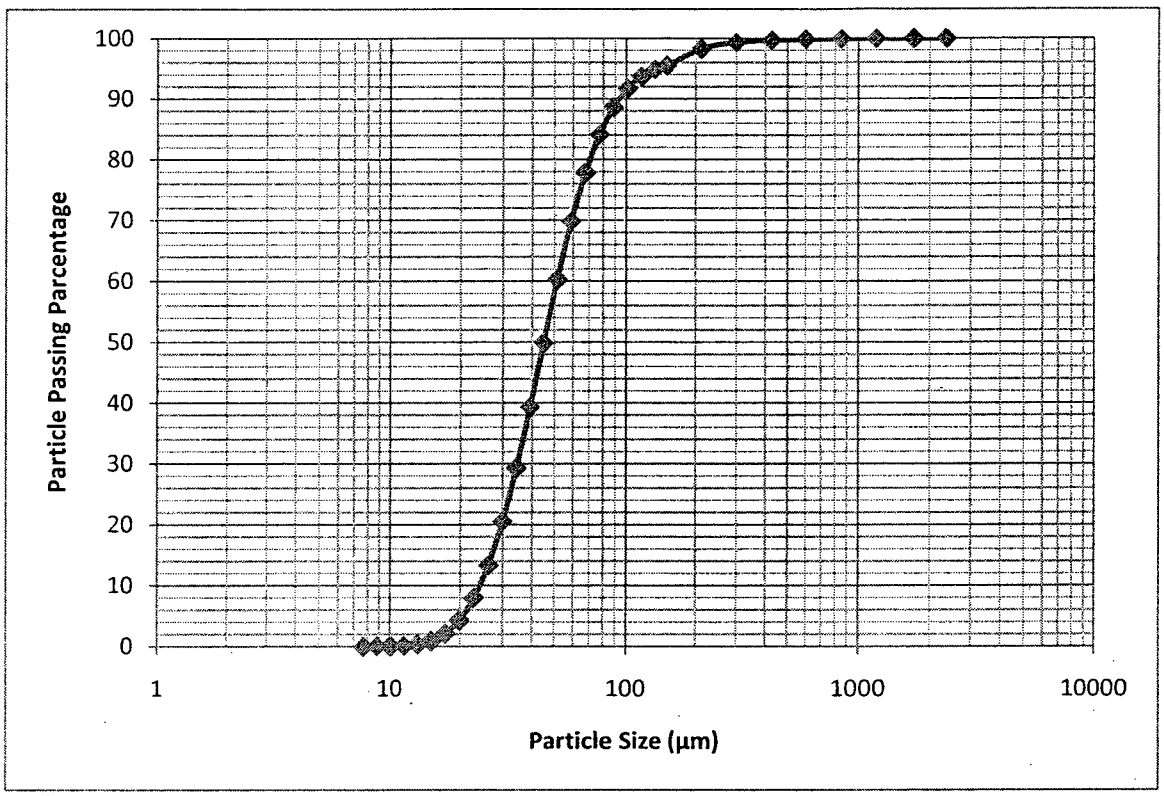

Figure 3. 1: Particle Size Distribution of tailings (sieve and laser results merged together).

Knowing these parameters helps to calculate the coefficient of uniformity (Cu) and the coefficient of curvature (Cc) or coefficient of gradation (Cg). These values are defined in Equations 3.1 and 3.2.

$$\text{Equation 3.1: } Cu = \frac{D60}{D10}$$

$$\text{Equation 3.2: } Cc = \frac{(D30)^2}{D60 \times D10}$$

In the Unified Soil Classification System (Bowles, 1998), if Cu is larger than 6 and Cc is between 1 and 3, the soil is classified as well graded. According to the calculations for the tailings used, Cu=2.52 and Cc=0.88; therefore, it can be classified as poorly graded sand.

3.2.2. THE SPECIFIC GRAVITY OF SOIL PARTICLES (GS)

It is not common to use the specific gravity (GS) value to classify soil particles but for purpose of design and construction it is measured in laboratory, and its value for the tailings used was GS=2.94; to obtain this value, ASTM C 128 (2004) was followed.

3.2.3. SHAPE AND TEXTURE

As Figure 3.2 illustrates the shape of dry tailing at normal scale, this material does not have coarse particle. Scanning Electron Microscopy (SEM) was used by Razavi (2007) to investigate the particle shape and surface texture of the tailings, which were sprinkled upon an aluminum stub and covered with double sided adhesive tape. Then to enhance the visualization of the particles, the samples were coated with carbon. The photomicrograph obtained is shown below in Figures 3.3 and 3.4. Based on the images and using the British Standard Classification System, the shape and surface texture of the particles were determined.

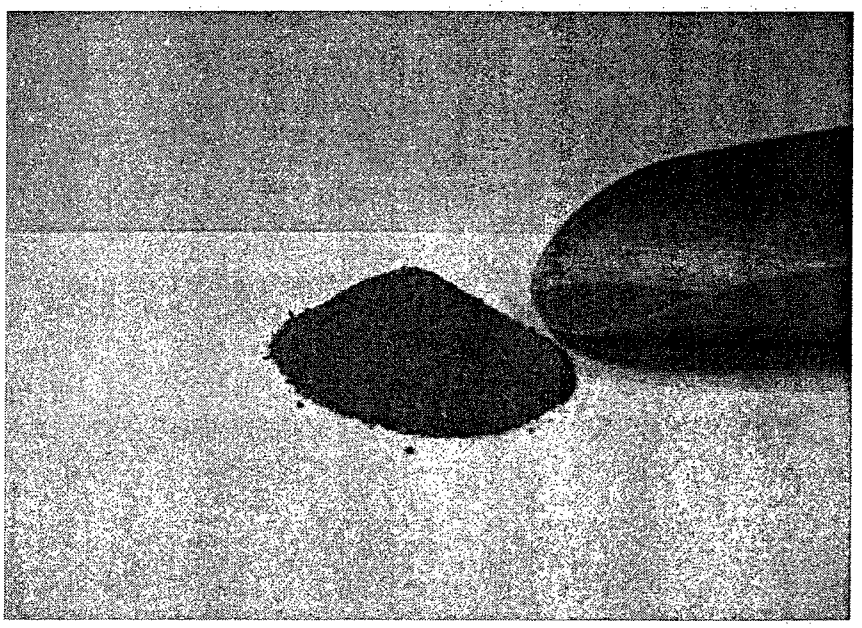

Figure 3. 2: Tailings from Creighton mine near Sudbury, Ontario.

Figure 3. 3: SEM image of tailings, magnification 200X, length of bar 50μm (Razavi, 2007)

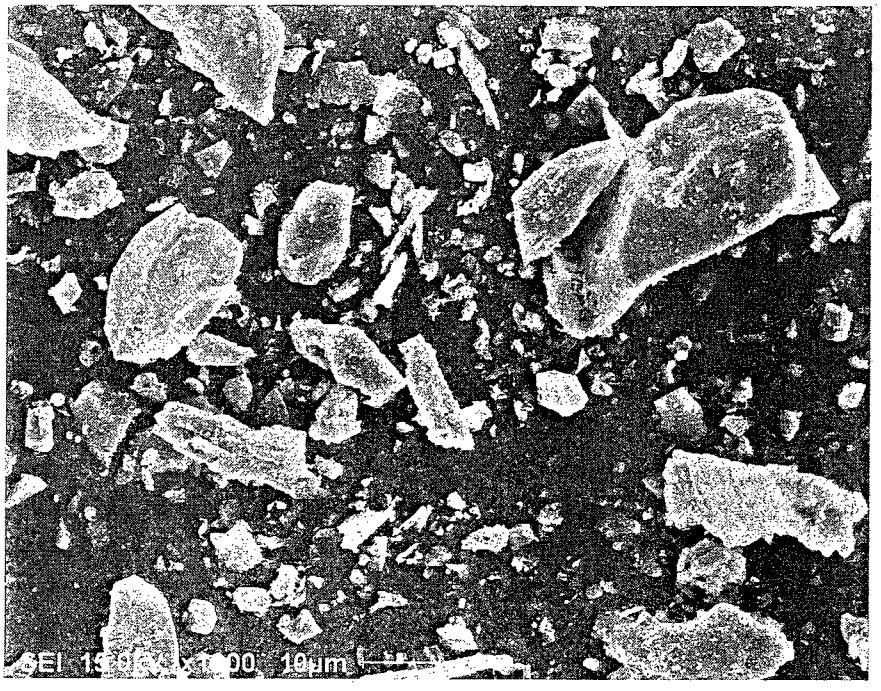

Figure 3. 4 SEM image of tailings, magnification 1000X, length of bar 10μm (Razavi, 2007)

The figures show that the particle shapes were angular and the surface textures were rough. In geological terms, "rough" describes jagged fractures of fine or medium grained rock without any crystalline constituent.

3.2.4. MINERALOGY

The backfill material used in this research was de-slimed tailings from the Creighton Mine in Sudbury, and the x-ray diffraction technique was used to find its mineralogical content. First, a portion of the tailings was dried in an oven at 50°C for 24 hours and ground into a powder. It was then placed in an aluminum cup, which was positioned in an x-ray generator. The spectrums were then compared to those from known minerals with a special computer program that was also used to determine the mineralogy. The X-ray diffraction results are shown in the Figure 3.5.

The tailings were made up mainly of quartz, albite, and anorthite with smaller amounts of calcite, muscovite, actinolite, chalcopyrite, biotite, pyrrhotite, epidote, and chlorite.

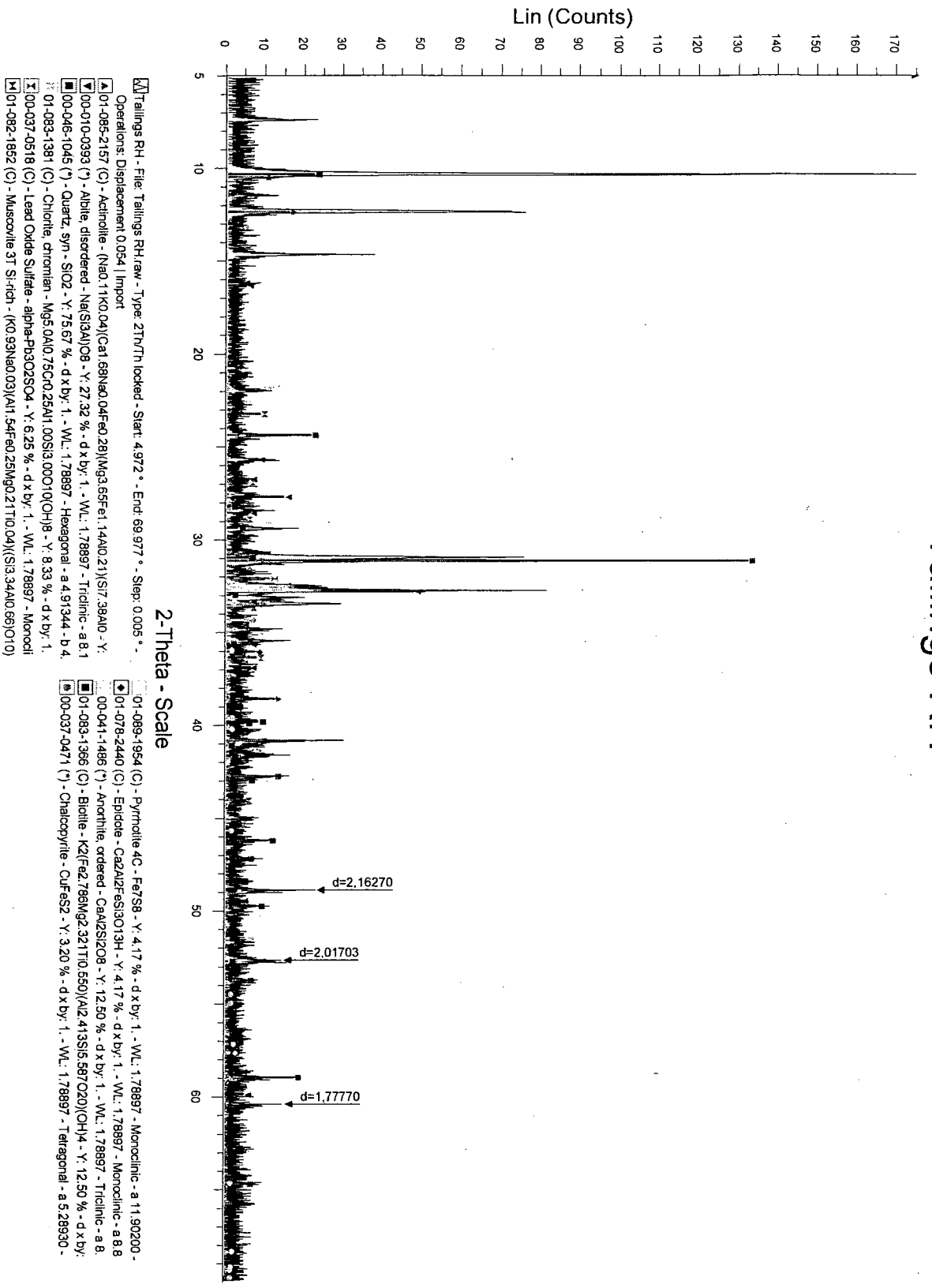

Figure 3. 5: The X-ray diffraction results (Razavi, 2007)

3.3. BINDERS

In this research project, a binder mixture proportion was used as currently used in the mining industry for backfill. A mix of 10% Portland cement type 10 (Type GU) and 90% blast furnace slag was used as the major binders, and both these materials were obtained from Lafarge Toronto, Ontario.

3.3.1. CEMENT

For the material used in the research project, the particle size distribution of cement was determined by the laser light diffraction technique, and the results are presented in Figure 3.6. From the cement particle size graph, D10=1.21μm and D50=3.45μm.

Comparing the D10 and D50 values and particle size distribution graph with the M.I.T. classification system, this cement is in the "silt-clay" category. To have an overview about the shape size and texture of cement, an SEM image was taken and presented in Figure 3.8. A chemical analysis was performed as well using the XRF test and its results are shown in Table 3-1. Figure 3.7 shows cement that was used for this research as one of the combination of binders.

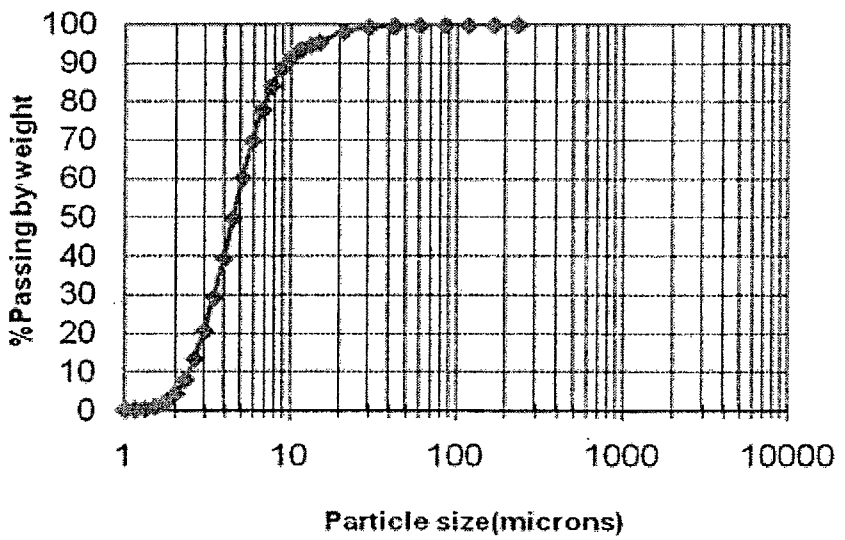

Figure 3. 6: Particle size distribution of Lafarge type 10 (Type GU) Portland cement.

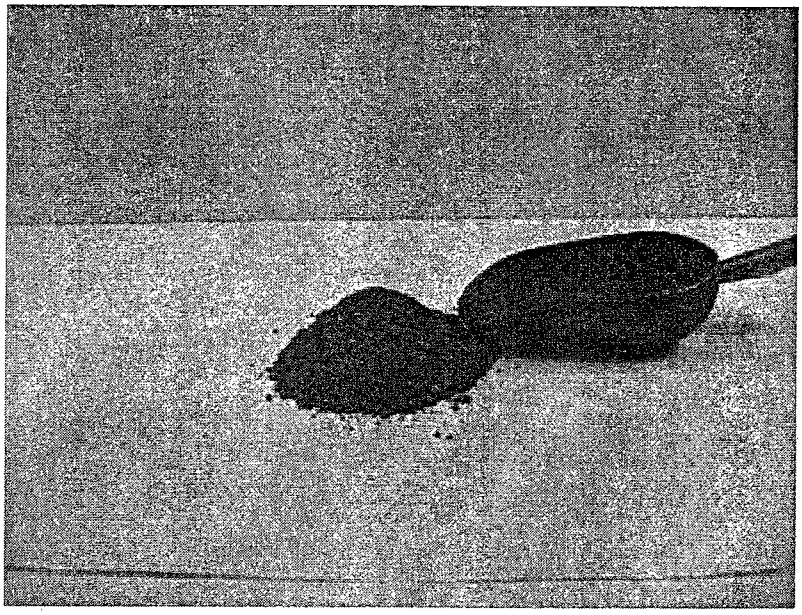

Figure 3. 7: Type 10 (Type GU) Portland cement from Lafarge.

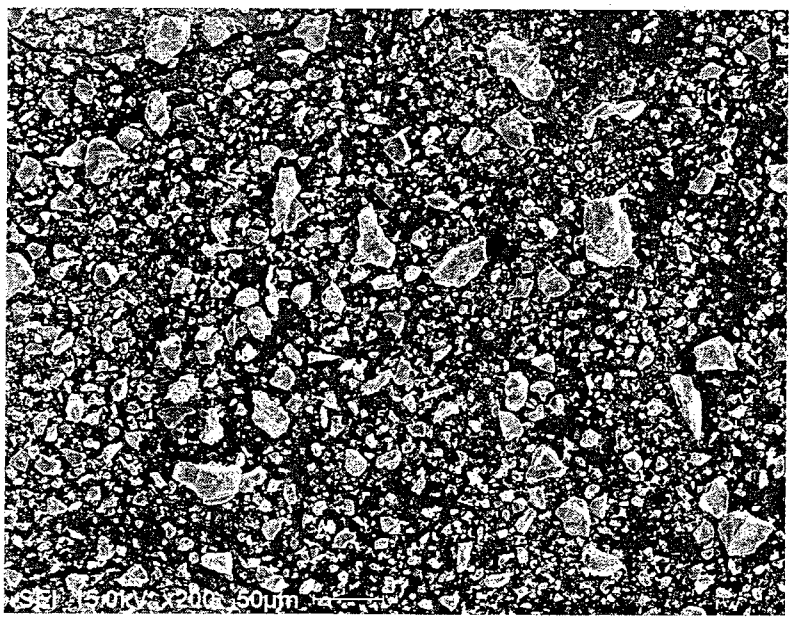

Figure 3. 8: SEM image of Lafarge type 10 Portland cement (Razavi, 2007)

Table 3. 1: Chemical Composition of Lafarge Portland cement type 10 (Kermani, 2008)

Material	SiO_2 (wt%)	Al_2O_3 (wt%)	Fe_2O_3 (wt%)	CaO (wt%)	MgO (wt%)	K_2O (wt%)	Na_2O (wt%)	SO_3 (wt%)
Cement	19.4	4.6	2.3	61.1	2.0	0.7	2.0	3.3

3.3.2. BLAST FURNACE SLAG (BFS)

To identify slag properties and the fine grain size of slag particles, the laser light diffraction technique was applied and the particle size distribution graph is presented in Figure 3.9. From the graph, D10 and D50 values are read to be 1.52 μm and 4.41 μm, respectively. Classifying the Lafarge BFS with the M.I.T. method designated it in the silt-clay category.

Shape, size, and texture of slag are shown in an SEM image given in Figure 3.11. and Figure 3.10 shows natural scale of slag. XRF analysis was applied to investigate the chemical composition and the results are given in Table 3-2.

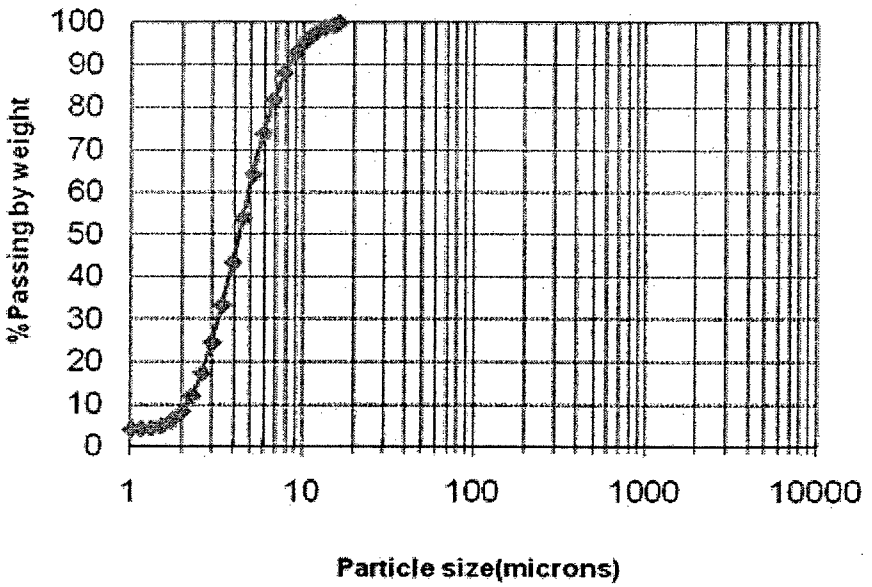

Figure 3. 9: Particle size distribution of Lafarge BFS (Razavi, 2007)

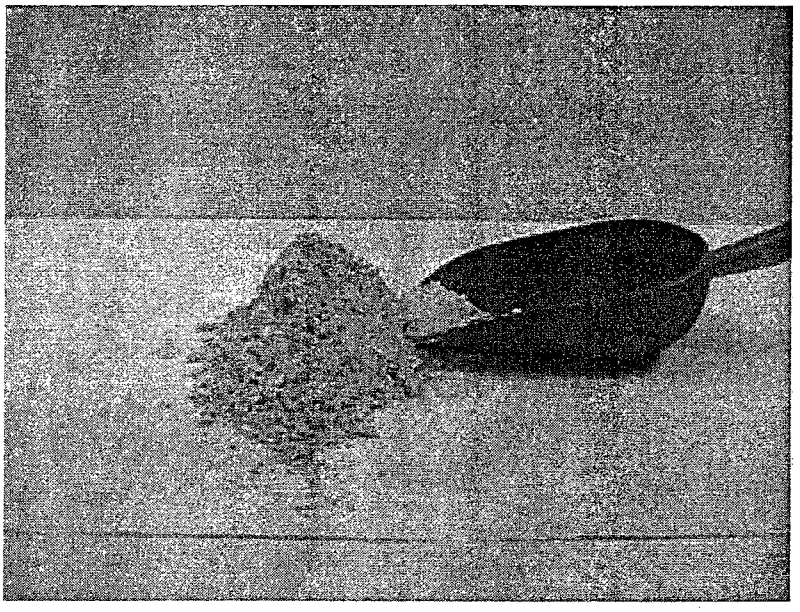

Figure 3. 10: Blast furnace slag from Lafarge

Figure 3. 11: SEM image of Lafarge BFS (Razavi, 2007)

Table 3. 2: Chemical Composition of Lafarge BFS (Kermani, 2008)

Material	SiO_2 (wt%)	Al_2O_3 (wt%)	Fe_2O_3 (wt%)	CaO (wt%)	MgO (wt%)	K_2O (wt%)	Na_2O (wt%)	SO_3 (wt%)
Cement	36.4	10.2	0.6	38.1	9.4	0.4	0.5	3.0

3.3.3. SODIUM SILICATE

For this experiment, type N® sodium silicate was used, which was provided by the PQ National Silicate Company in the form of liquid gel. The properties of sodium silicate are presented in Table 3.3.

Table 3. 3: Properties of sodium silicate (PQ National Silicate)

Sodium Silicate Properties	Standard	Maximum	Minimum
Na_2O,%	8.90	9.10	8.70
SiO_2,%	28.66	29.00	28.20
Weight Ratio, % SiO_2/Na_2O	3.22	3.27	3.15
Density (g/cm^3), @20°C	1.38	1.40	1.36
Specific Gravity, @20°C	1.394	1.401	1.388
Viscosity @20°C (Pascal/Second)	0.177	0.213	0.141
Solids, %	37.56	38.10	36.90

3.4. WATER

Water plays two important roles in the mixtures; it hydrates the binders and it assists with the transportation of backfill material. Chemical components, soluble in water, have an effect on the chemical reactions of binders, and several researchers (Hassani and Archibald, 1998; Kesimal et al., 2005; Razavi, 2007) take this factor into account. Considering that the mining industry uses process water for their backfill, its properties have a significant effect on the product of the backfill. Nevertheless, in this research experiment, the effect of soluble chemicals was not studied and distilled water was used throughout.

CHAPTER : 4 TEST PROCEDURES

The physical and chemical properties of tailings influence the final strength of backfill. The current chapter explains the preparations for and the tests conducted on the tailings in this research project. Experimental procedures were carried out at Concordia University and the McGill University geotechnical laboratory. All the tests and their setup were conducted according to American Society for Testing Materials (ASTM) and other references.

In the project, various tests were done to study the physical, mechanical, and microstructural properties of the materials. For instance, the direct shear test was applied to study the shear strength behaviour of the tailings without any binder or moisture added to it. The Vicat apparatus was used to check the reaction of binders in different mixes and at various curing temperatures. The uniaxial compressive strength (UCS) test was run to observe the strength behaviour of samples for two types of binders, five different curing conditions, and three different curing periods. A triaxial test was done on both types of binder at one curing period and temperature. The mercury intrusion porosimetry (MIP) test was used to identify the microstructural network in samples for both types of binders, three critical curing temperature conditions, and one curing time.

4.1. PREPARING SAMPLES

This section explains the procedure for the preparing and setup of the raw materials as well as the samples made from them.

4.1.1. DRYING TAILINGS

The principal material used for making slurry backfill is tailings. Tailings have been brought to laboratory in barrels from the INCO Creighton mining site located in Sudbury, Ontario. Tailings were brought in the wet condition so they were air dried for one or two weeks to control moisture of samples.

4.1.2. PREPARING MOULDS

The moulds used for making the samples were cylindrical PVC ones as seen in Figure 4.1, with the dimensions being 50mm (two inches) in diameter and 100mm (four inches) in height to satisfy the ratio requirement of 2 to 1. The moulds were extended and taped as shown in Figure 4.1.b, and the reason for this extension is that the samples settled after pouring; the extra water filtered out at the end. The extension provided a method of pouring in extra material so that after the settlement, the sample satisfied the ratio requirements.

To simulate the drainage condition in the underground mine, holes were drilled in the bottom of moulds, and mattress filters were placed at bottom of moulds to stop particles from running out with the extra water. For a better performance during sample extraction, the inner walls of the moulds were sprayed with oil.

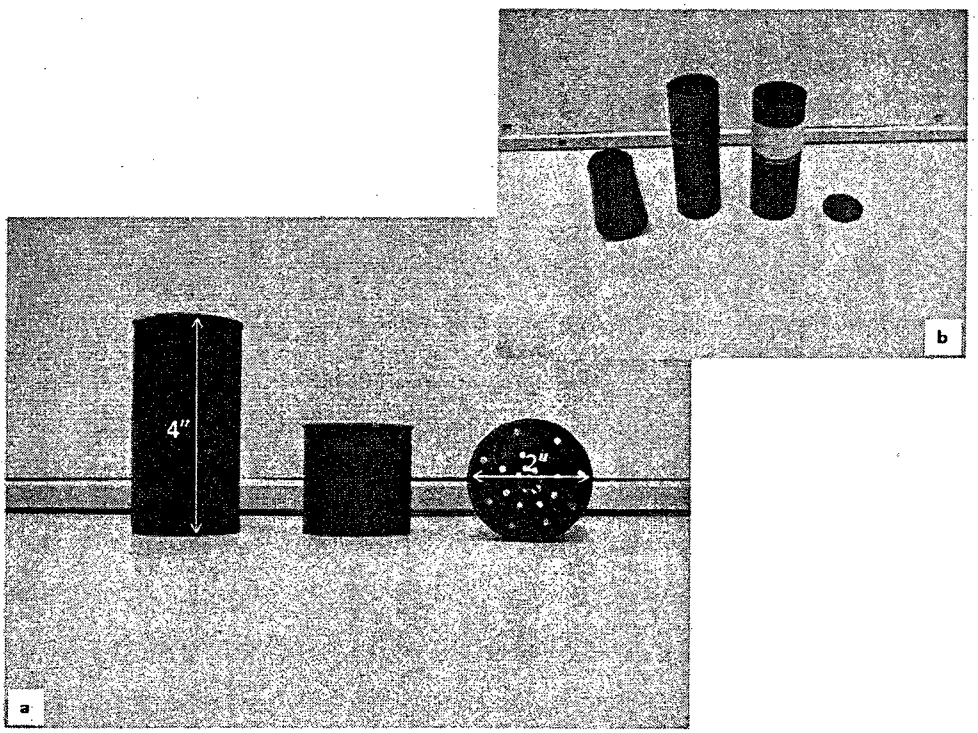

Figure 4. 1: Cylindrical shaped moulds showing a) Dimensions of the mould, b) Extension part of mould and filter for drainage purposes

4.1.3. MAKING SAMPLES

To mix different components, a kitchen stand mixer was used (Kitchenaid, Commercial 5 Series, Model: KM25G0X). The speed of mixer was set such that the materials inside the bowl did not come out and the mixture material did not settle down. A PVC pipe with a valve was attached to the lower part of the bowl to increase the efficiency of pouring the mixes into the moulds. To prevent the material from blocking the pipe, a small piece of rubber was placed in the entrance during mixing time; Figure 4.2 illustrates the details of the mixing procedure. Tailings were weighed in the mixing bowl. Binders such as cement and slag were weighed in a separate jar. Water was measured separately at a room temperature of 25°C, and some of the mixtures contained sodium silicate. All of the measurements have been done by the weight of materials. In order to homogenize the mixtures, the mixer was started with the tailings after which about ¾ of the water volume was added, and finally the binders were poured into the mixing bowl. If the addition of sodium silicate was necessary, it was put in before all the binders were poured into the bowl, and then the rest of the water was added. Afterwards, the materials were mixed for five minutes, and were then poured into the prepared moulds.

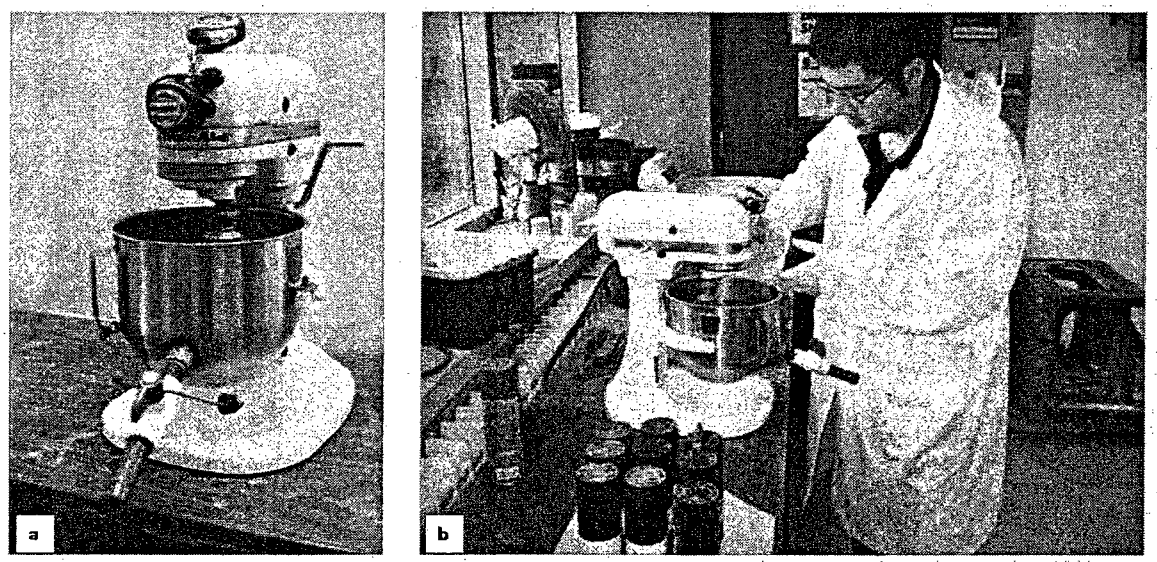

Figure 4. 2: Mixing procedure with a) Mixing bowl details, b) Mixing operation

4.1.4. CURING SAMPLES

Samples remained in the moulds throughout the curing period, and were treated at different curing temperatures, which were 5°C, 15°C, 25°C, 35°C, and 50°C. The relative humidity was controlled with an Intelligent Growing System apparatus for 90%±5% as shown in Figure 4.3. The purpose of applying humidity was to simulate the conditions of an underground mine. To maintain temperatures of 5°C and 15°C, two different commercial refrigerators were used and for the 35°C and 50°C values, two soil laboratory ovens were used. The 25°C temperature was applied in a chamber humidity room. Samples used for the USC and triaxial tests were stored at constant conditions until testing.

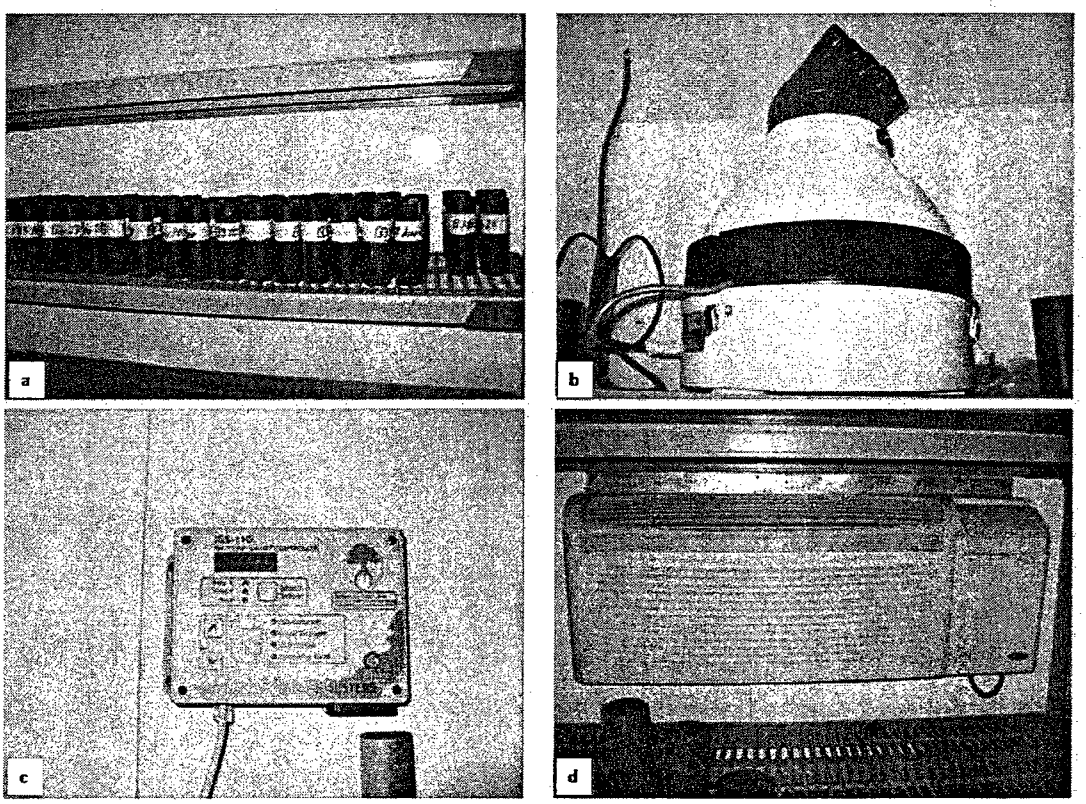

Figure 4. 3: Curing samples for UCS, Triaxial and MIP Test with a) Samples in Moulds, b) Humidifier, c) Humidity room controller, d) Curing room AC

4.2. TEST PROCEDURES

4.2.1. DIRECT SHEAR STRENGTH TEST

The laboratory tests were carried out on mine tailings using the direct shear strength apparatus as specified by ASTM D 3080 (2004). The length and breadth of the box was measured using a meter rule. A filter paper was placed in the shear box and the box filled with tailings up to the brim. The shear box was then placed in the machine and the screws were removed. In the direct shear test, the top half of the specimen was translated relatively to the bottom half, which created a shear plane across the middle of the specimen (Zhang and Thornton, 2007). Horizontal and vertical external forces were applied on the shear box or shear cell and the ratio of horizontal to vertical load were calculated. This provided an estimated average ratio of shear to normal stress acting in the shear band and hence a direct measure of the angle of internal friction could be obtained (Zhang and Thornton, 2007).

The loading conditions of the shear test were applied by two separate loading frames. The normal force was applied by weight and the shear force was applied through an electrical gear engine as appears in Figures 4.4 and 4.5. A spherical ball bolt was placed on the cap of the shear box that contained the specimen. The relative movement was caused by the sliding of the lower part under the upper part, and the displacement of the lower half of the specimen relative to its shear box was measured by displacement sensor. The normal load acting on the specimen was changed each time in separate experiments. Increasing weights were first measured and then added to the weight hanger to increase the vertical or normal stress. Each test was carried out using a rate of shear deformation of about 1 mm/min.

Using a computerized data logging system, the data regarding the shear force, shear, and normal displacement were collected. The readings were taken until the horizontal shear load peaked and then fell or until the horizontal displacement reached 15% of the length of the box.

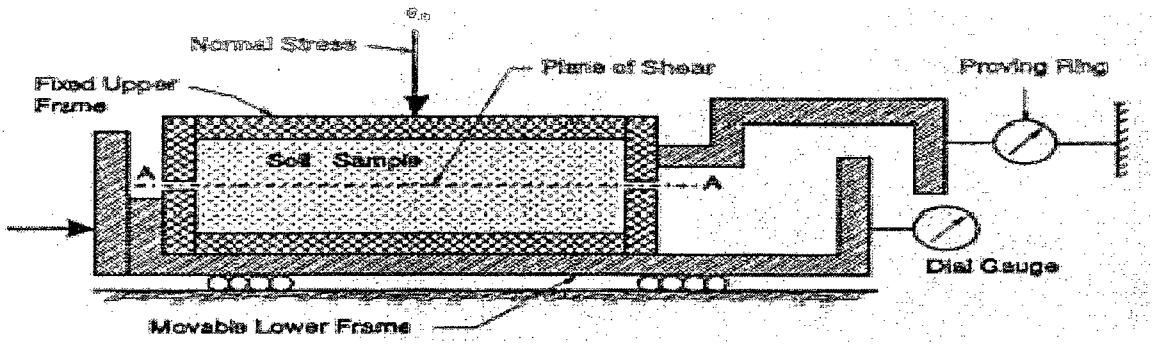

Figure 4. 4: Schematic representation of direct shear apparatus (Yagiz, 2001)

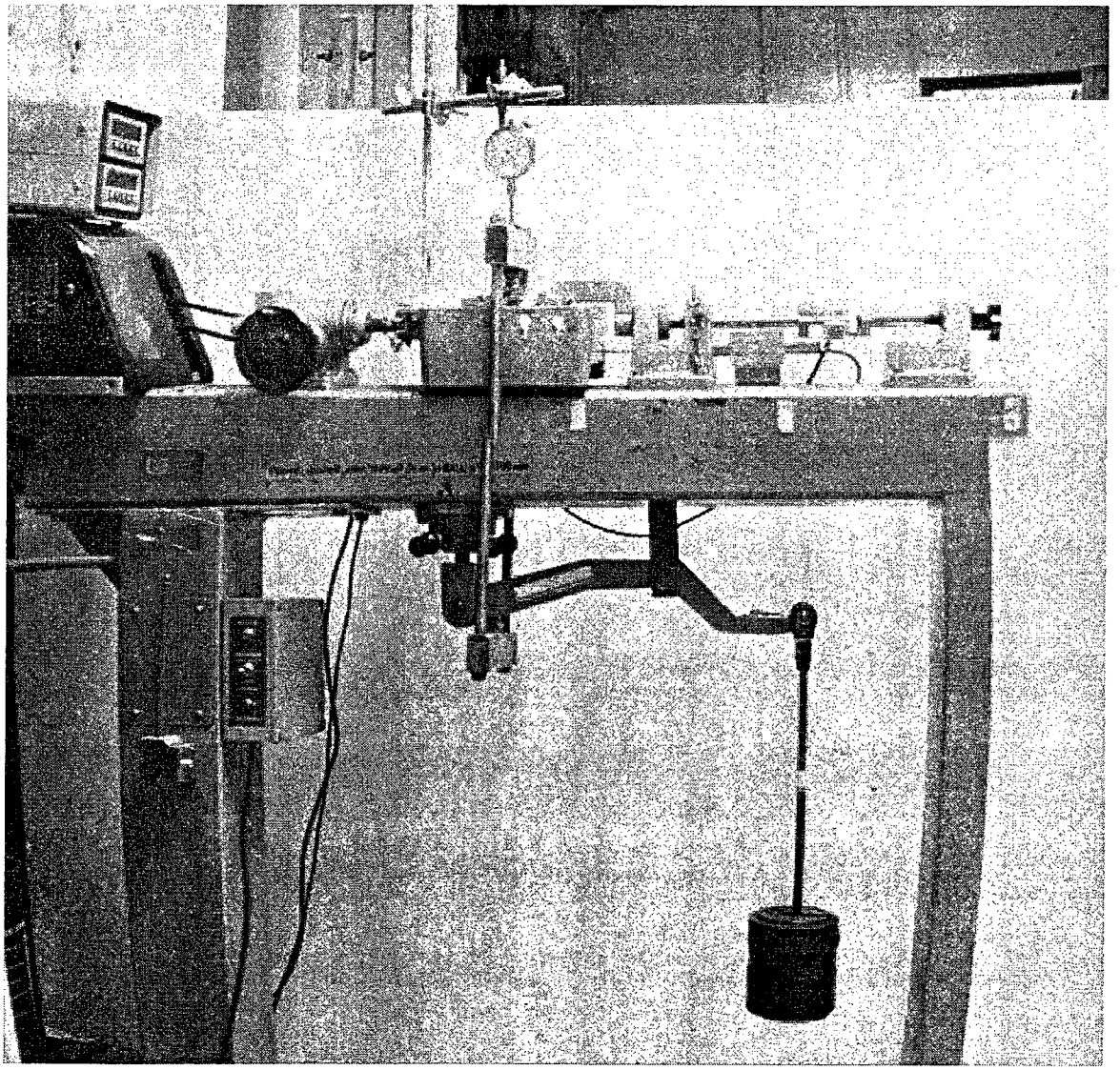

Figure 4. 5: Computerized direct shear apparatus used for test

4.2.2. SETTING TIME TEST

The Vicat needle test is a standard procedure to determine the setting time of hydraulic cement (ASTM C 191, 2004). The setting time is the period required for a hydraulic cement paste mixture to set and harden. Standard setting time methods and procedures were used for this test, and the apparatus is presented in Figure 4.6.

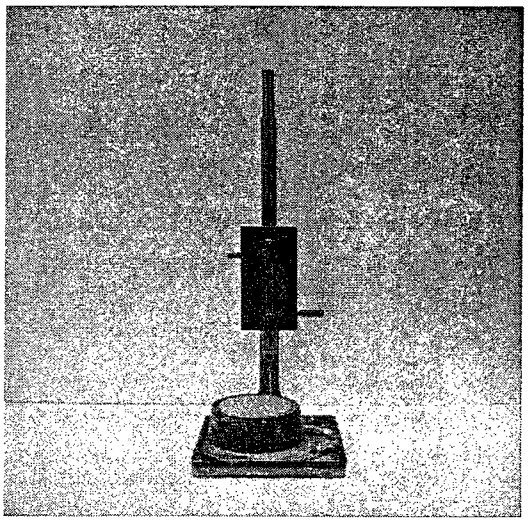

Figure 4. 6: Vicat apparatus used to determine setting time of binders

The Vicat apparatus consists of a frame with a moveable rod, a 50mm scale graduated in millimetres, and a removable steel needle. The rod is reversible and adjustable, and includes an adjustable indicator which moves over the graduated millimetre scale. At the bottom is the non-absorptive plate on which a conical ring with cement paste is placed (ASTM C 191, 2004).

A paste consisting of water, cement, slag and sodium silicate was mixed thoroughly and made homogeneous. As it started to take shape, it was rolled and made into a sphere and tossed six times from one hand to another, maintaining a 6 inch gap in between. The paste was then inserted from the bottom of the Vicat mould and pressed gently and levelled using a spatula. With the use of the Vicat apparatus the penetration of the needle was measured. The moulded paste was kept in a moist cabinet and allowed to start setting.

Periodic penetration tests were performed on the paste by allowing penetration of the Vicat needle (1mm diameter) into the paste. At each time interval, the process was repeated and the penetration was measured. It was not possible to get an exact 25mm penetration from the instrument. Thus, the data was interpolated and this number was set as the initial set time. The final setting time was taken at the point where the needle left prints on the hardened mixture and did not penetrate (ASTM C 191, 2004). The final setting time was also recorded and a graph plotted using the data. The mould paste was then immediately transferred into a humid room in a moist cabinet and allowed to set.

4.2.2.1 Calculation:

The Vicat time of initial setting was determined using the formula below (ASTM C 191, 2004):

$$T_{25} = [\frac{(H - E)}{(C - D)}] \times (C - 25) + E$$

Where:

T_{25}= Time in minutes at 25mm penetration

E= time in minutes of last penetration greater than 25mm

H=time in minutes of first penetration less than 25mm

C=penetration reading at time E

D=penetration reading at time H.

Apart from taking the measurements of the needle penetration, the mould was kept in the humid room, where the temperature was steady at 25°C (± 2°C). The humidity of the room was kept at about 90%. Same procedure followed for other curing temperatures.

4.2.2.2 Precaution:

During the reading procedure, any vibration to the Vicat instrument was avoided. The needle was kept clean and straight at all times to prevent cement from causing friction and decreasing the penetration rate.

4.3. MECHANICAL TESTS

This section includes uniaxial compressive strength and triaxial test procedures, with the former being done on all samples and the latter conducted based on the optimum points of the UCS results.

4.3.1. UNIAXIAL COMPRESSIVE STRENGTH (UCS) TEST

Different specimens were made and their material components are presented in table 4.1. The specimens were mixed and poured in moulds as described in section 4.1.3, and they were made to have similar heights. They were then taken and cured as in 4.1.4. After 7, 14, and 28 days of curing, samples were forced out of the mould with the use of air pressure, and were handled with care so that they did not break. They were cut and levelled such that the height to diameter ratio was around 2, and the height of the sample was measured after resizing. The sample was put in the loading frame and centered at the bottom of the platen, and the loading frame was adjusted carefully so that the upper platen touched the sample. A probe sensor was attached to measure the displacement and maximum force, the displacement was set to zero, and the machine was started. The rate of strain applied on the sample was set at a rate of 1mm/min. The maximum load that was borne by the sample was measured; the data was obtained with LVDT, and saved on the computer.

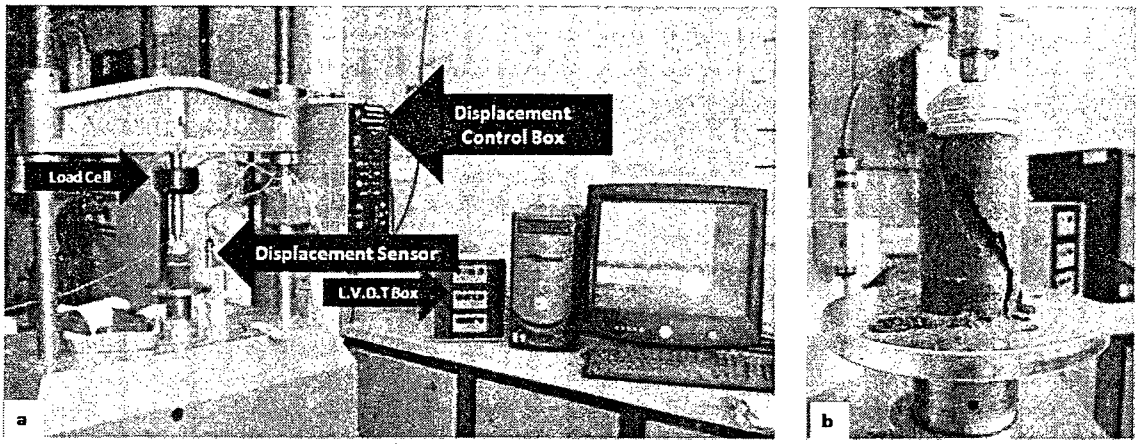

Figure 4. 7: UCS apparatus setting with a) Different parts of the apparatus, b) Sample at the end of a test

Table 4. 1: Mixture Recipe for the UCS Test (Total Weight)

Binder Type	Slag BFS (wt%)	Portland Cement (wt%)	Sodium Silicate (wt%)	Pulp Density
SCSS 0.3	4.5	0.5	0.3	70%
SC	4.5	0.5	0	70%

4.3.2. MERCURY INTRUSION POROSIMETRY (MIP) TEST

Mercury porosimetry was performed on a few mixtures to investigate the effects of sodium silicate addition and curing temperature on microstructure. These samples were chosen as critical point in manner of highest and lowest curing temperature plus higher strength values from UCS test for both types of binders. A small specimen is dried at 50°C for at least twenty four hours to empty the pores of any existing fluid. It is then weighed and put in a chamber which is evacuated and the specimen is surrounded by mercury (Diamond, 2000). Pressure is applied to the mercury and the intrusion is monitored, as presented in Figure 4.9. Since mercury does not wet to cementitious solid, it does not intrude unless pressure is applied. The set of pressure steps and consequent volumes intruded gives the pore size distribution calculations. The Washburn equation is used for the measurement of the pore radii and is dependent on the accuracy of the values

of the contact angle of mercury with the material and surface tension of mercury (Kumar and Rao, 2002).

The Washburn model used to convert mercury intrusion data into pore size distribution invokes two discrete assumptions: i) pores are cylindrical and ii) they are equally and completely accessible to the outer surface of specimen (Diamond, 2000). The choice of cylindrical pore geometry was used for convenience in order to avoid complexities regarding mean radii and contact angles in pores of irregular cross sections (Leon and Carlos, 1998).

The process of MIP involves the introduction of porous samples into an evacuated chamber and the samples are then surrounded by mercury. The pressure applied to the mercury is then gradually increased. As the pressure is increased, the mercury is forced into the pores on the surface of the sample (Cook and Hover, 1999). The pore width at which the highest rate of mercury intrusion per change in pressure is achieved is called the threshold pore width threshold diameter. The results of MIP are expressed as the total mercury volume intruded into a sample or in terms of the incremental volume intruded at different pressure. The total volume of pore space that can be intruded by mercury is useful to compare the porosity of the system. This volume varies with the water to cement ratio and age in the same manner as threshold diameter. The volume that can be intruded includes air voids and intrinsic paste pores, and the air void content may fluctuate depending on mixing procedure (Diamond, 2000).

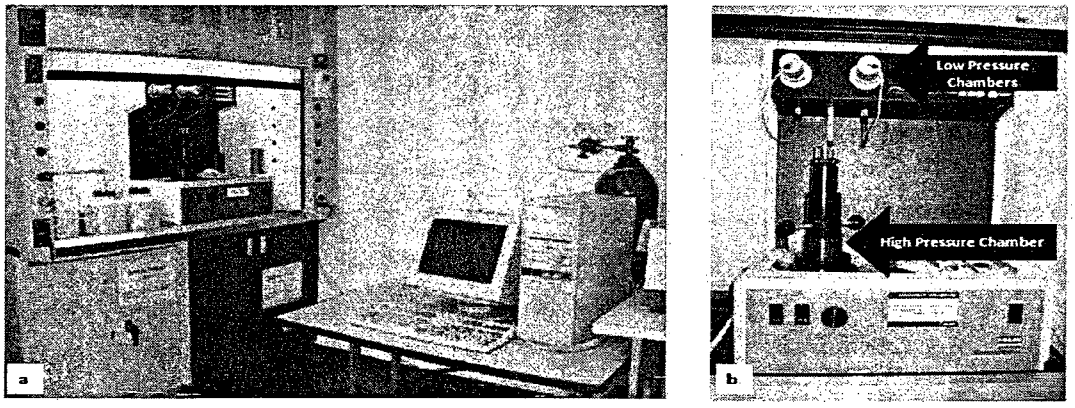

Figure 4. 8: a) Mercury Intrusion Porosimetry apparatus setting, b) High and low pressure chambers

CHAPTER : 5 RESULTS AND DISCUSSION

In this chapter, the results of the series of tests performed to study the effect of temperature on behaviour of sodium silicate in backfill samples are presented. This part has two related sections. The first section of the chapter includes results of tests. For instance, the direct shear test was performed on dry tailing and then the setting time test for different binders was determined and finally mixtures of material were tested for uniaxial compressive strength (UCS) and Mercury Intrusion Porosimetry (MIP). The second section compares and analyzes results of different tests in first section.

5.1. TEST RESULTS

Purpose of this section is to gain the properties and understand the behaviour of basic materials and mixtures of them. To satisfy this goal, the Direct Shear Test was applied on tailings to determine cohesion and angle of friction. Setting time was used to study the effect of temperature on different combination of binders. Uniaxial Compressive Strength (UCS) test was used on samples that were cured at different temperatures for two types of binders. Last but not least, Mercury Intrusion Porosimetry test inspects effect of various curing temperatures and both binders on microstructure of samples.

5.1.1. DIRECT SHEAR TEST

The Direct Shear Test is one of the simplest tests that are able to produce valuable information about the shear behaviour of a material. This test was applied to study the behaviour of the tailing component of the backfill mixture. These results can be compared to the results of the triaxial test and this comparison gives perspective for the effect of binders on backfill mixtures. The tailing, used for this test, was air dried and four different incremental normal stresses were applied to produce the Mohr-Coulomb envelope.

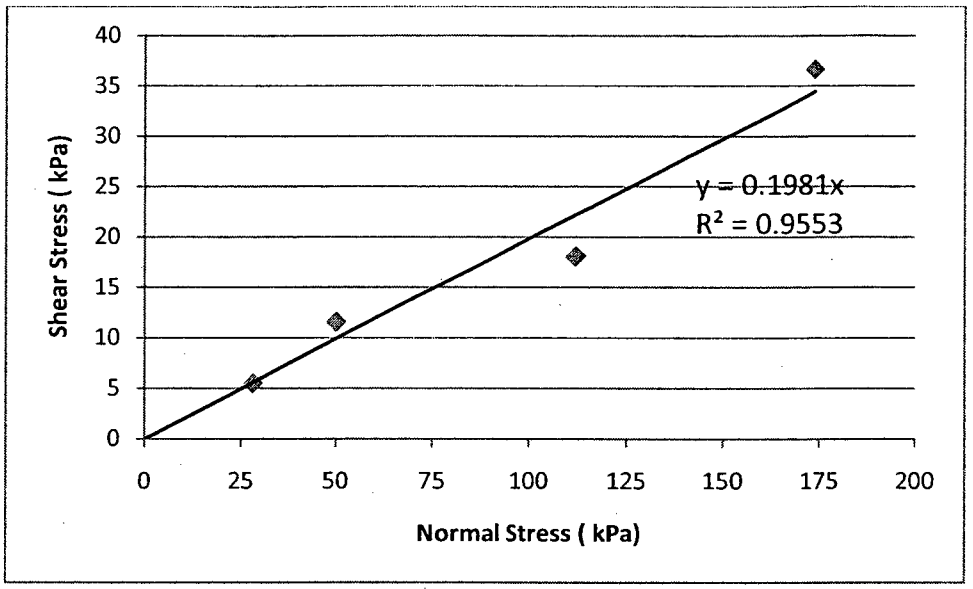

Figure 5. 1: Plot of Four Direct Shear Test on Tailing with C=0(kPa) and Ø=11°.

Figure 5.1 shows the results of the direct shear test experiment. The normal and shear forces were obtained from the four tests and are presented in terms of kPa. These results were then used to plot a best fit straight line. With an increase in applied load to normal stress, it was found that the shear stress increases linearly and is thus directly proportional to the normal one. From the graph, it can be seen that there is no y-intercept; C = 0 (kPa). The angle that the line makes with the horizontal gives the Ø value; Ø=11°, which is the angle of internal friction. An important mechanical property is determined in this manner since the C value – cohesive stress – is zero for the tailings.

5.1.2. SET TIME

The use of a Vicat needle for determining the setting times of cements is a simple procedure which has been standardized and is universally used. Setting time test was performed in this part to monitor the behaviour of binders in their first phase of hydration and for purpose of the research some conditions such as curing temperature and binders' type changed. Six different combinations of binders were under investigation which had different percentages of sodium silicate that were mixed with binders of slag, Portland cement and water; slag and cement were mixed by 9 to 1 ratio. In addition, five of the combinations were tested at two other curing temperature conditions; 5°C and 40°C.

Table 5. 1: Setting Time Results (in minutes)

Binder Type		**Slag**	**Slag + 10% Sodium Silicate**	**Slag + Cement**	**Slag + Cement + 2% Sodium Silicate**	**Slag + Cement + 6% Sodium Silicate**	**Slag + Cement + 10% Sodium Silicate**
Temperature [C]	**(Minute)**						
5	Initial Setting Time		140	590	130	94	75
	Final Setting Time		590	1360	404	329	285
25	Initial Setting Time	816	105	180	93	80	65
	Final Setting Time	3820	340	535	215	190	145
40	Initial Setting Time		100	138	75	69	50
	Final Setting Time		295	348	190	169	140

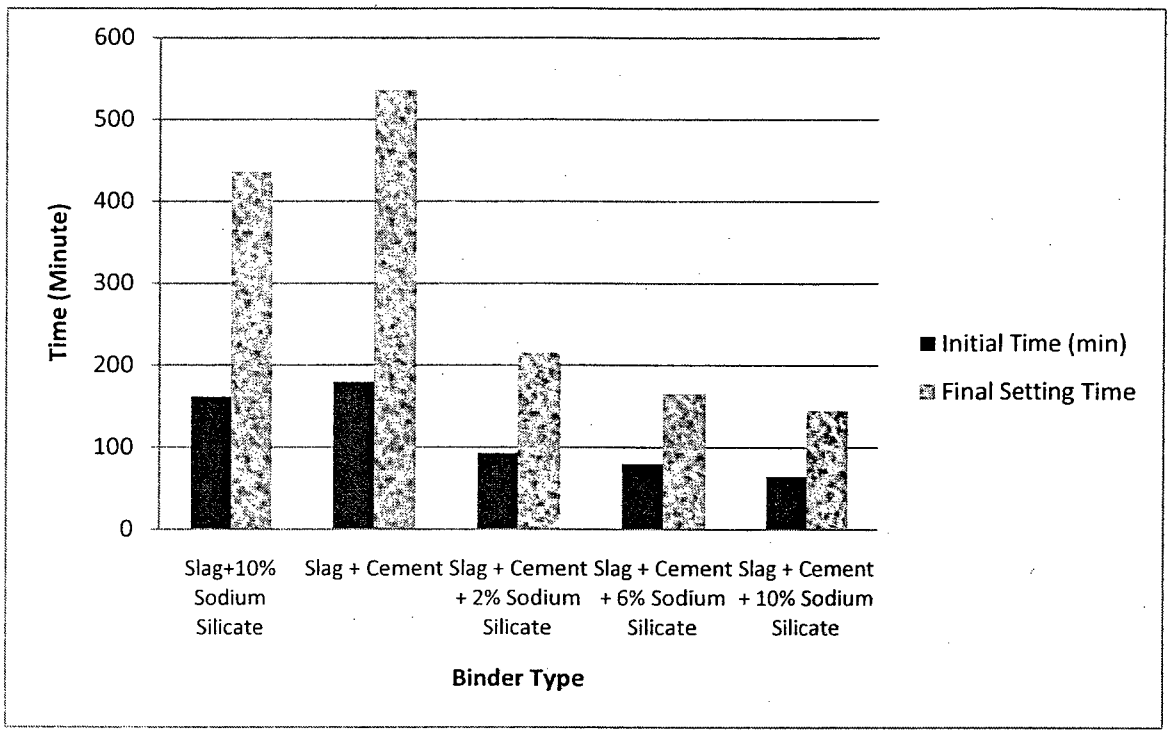

Figure 5. 2: Initial and Setting Time for Different Percentages of Reagents in the Mixture Cured at 25°C

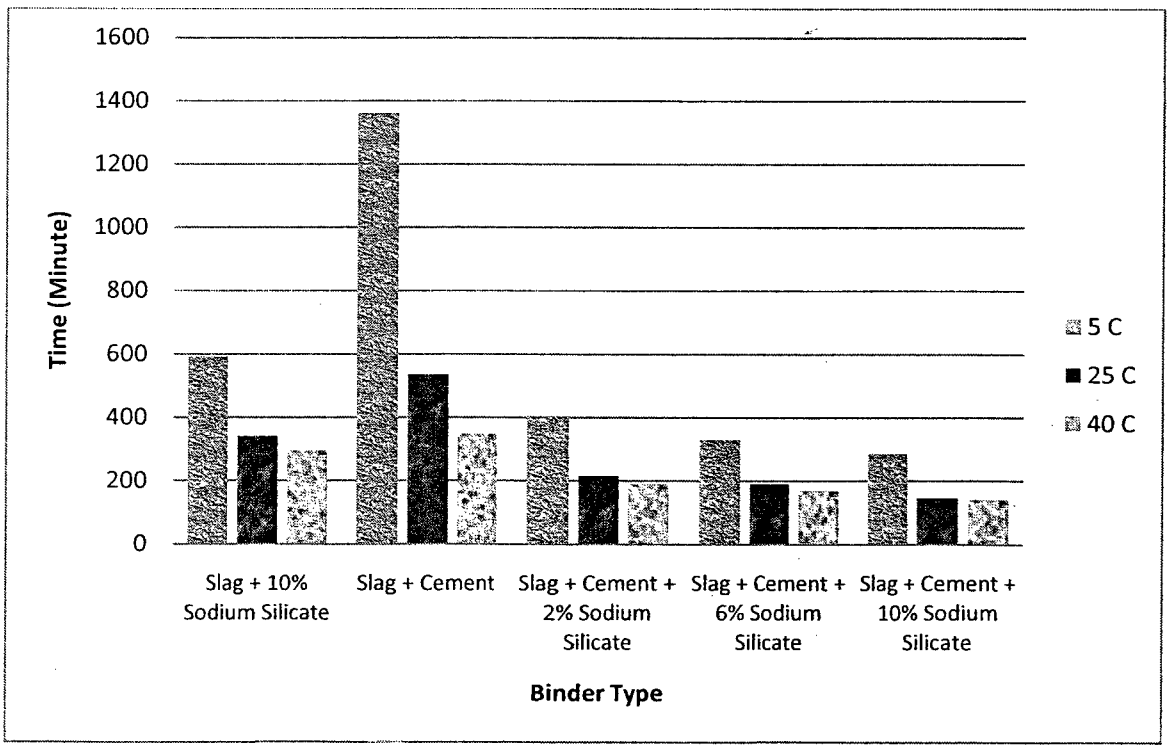

Figure 5. 3: Effect of Temperature and Sodium Silicate on the Final Setting Time.

Table 5.1 presents the changes in setting time due to different proportions of reagents in the mixture. First of all, it is observed from Table 5.1 that the slag only mixture takes a much longer time to set when compared to the others. It can be seen that without the presence of cement or sodium silicate, the setting time differs significantly from the other ones. The initial setting time is greater than the final setting times for the rest of the mixtures. Due to inadequate early strength, this mixture was not tested for set at other temperatures or for hardened properties. It should be also noted that adding Sodium Silicate to Slag or adding Cement to Slag reduces setting time for slag combination, it can be concluded from table 5.1 as slag and other combinations of slag are used i.e. slag cement and slag sodium silicate. Since slag results showed low rate of hydration for this test then slag binder was not practiced for other curing temperatures due to obvious behaviour. When comparing the two sodium silicate mixtures at 10% addition from Figure 5.2 and Table 5.1, it can be observed that there is a marked difference in setting time and the only difference between the two recipes is that the one was prepared with Slag, Cement and 10% Sodium Silicate, and the other one was prepared with Slag and 10% Sodium Silicate. Thus, cement can be seen to play a major role in determining the setting time, as anticipated. As the sodium silicate percentage gradually increases, and the relationship between percentage of sodium silicate and setting time becomes obvious in Figure 5.2. As the percentage of sodium silicate in the mixture increases, the initial and setting times both decrease. Thus, setting time is inversely proportional to the percentage of sodium silicate added to the mixture.

It is evident that the types of binders and dosage of sodium silicate are significant factors influencing the properties of the alkali slag cement. This is a consequence of the fact that the nature and dosage of activators play a decisive role in determining the hydration and structure development of alkali activated cements, and thus the setting time and workability are characteristics which indicate the hardening process in its initial period (Zivica, 2006). It was also found that sodium silicate, along with cement or slag alone, does not form a good cementitious binder. It is a good mixture of all the components which is responsible for a faster setting time of the resultant product.

These experiments were carried out again with the same mixtures except for altering the curing temperatures; as presented in Table 5.1 and Figure 5.3, 5°C and 40°C. The general trends indicate that both the initial and final setting times decrease as the curing temperature increases, which could be due to an increase in the rate of hydration at higher temperatures. It was found that the initial and final setting times of ordinary Portland cement are shortened with an increase in the curing temperature as well as with blended pastes (Heikal et al., 2004). Thus, less time is needed for the same mixture to hydrate at higher temperatures, e.g. 40°C, than at lower ones. When the experiment was repeated at lower temperatures, e.g. 5°C, the initial and final time were found to be longer as shown in Table 5.1. In addition, the workability of the mix during handling, consolidation, and finishing decreases due to quick setting at these temperatures and adding more percentage of sodium silicate.

In the literature, it is mentioned that the initial and final setting times of liquid sodium silicate and sodium hydroxide activated slag occur much faster than Portland cement (Atis et al., 2007). The final setting times of liquid sodium silicate activated slag paste are also reduced with increase in the silicon dioxide/sodium oxide ratio (Atis et al., 2007).

5.1.3. UNIAXIAL COMPRESSIVE STRENGTH

Studying the trend of strength gain for backfill samples leads to the results of the Uniaxial Compressive Strength test. This test was done on cured backfill samples at three curing periods; 7, 14 and 28 days, and five different curing temperatures; as 5, 15, 25, 35 and 50°C in combination of two types of binders. The binders are chosen to represent mining industry usage as it was advised to observe their strength behaviour in various curing conditions. As it was mentioned before, proportion of material recipe are in this order; Slag and Cement (SC) binder ratio is 9 to 1 and Slag, Cement and Sodium Silicate (SCSS) binder has the same ratio at slag and cement plus 6% weight of binder sodium silicate.

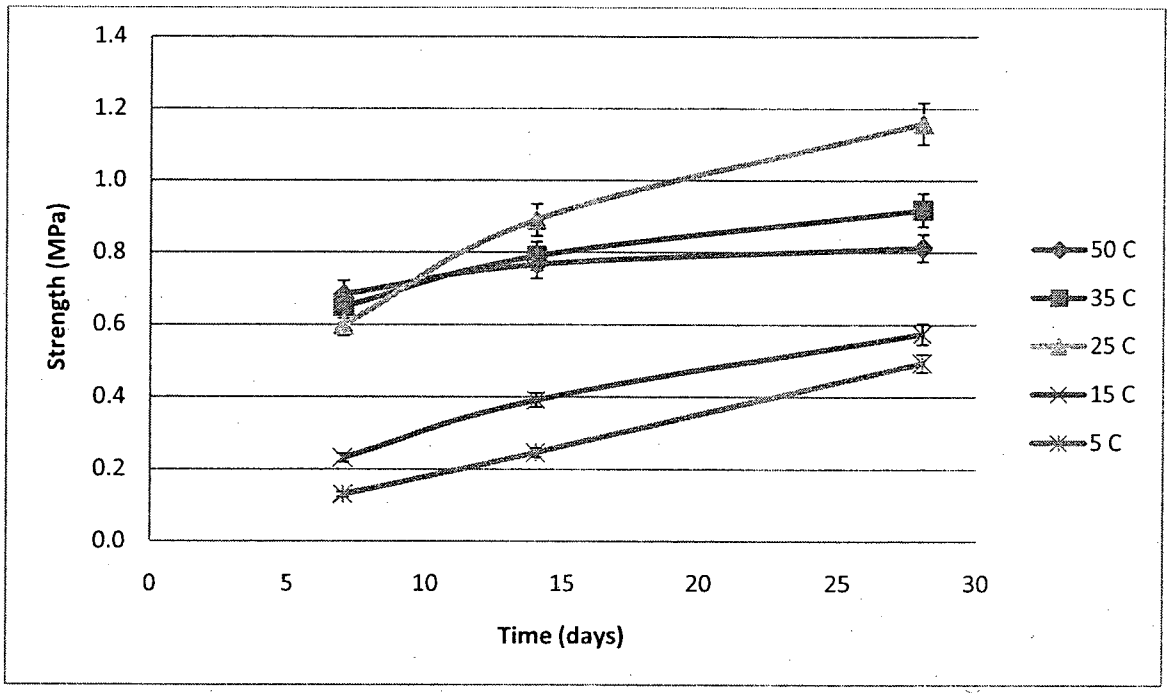

Figure 5. 4: Strength versus Curing Time at Different Temperatures in the Presence of Sodium Silicate (SCSS).

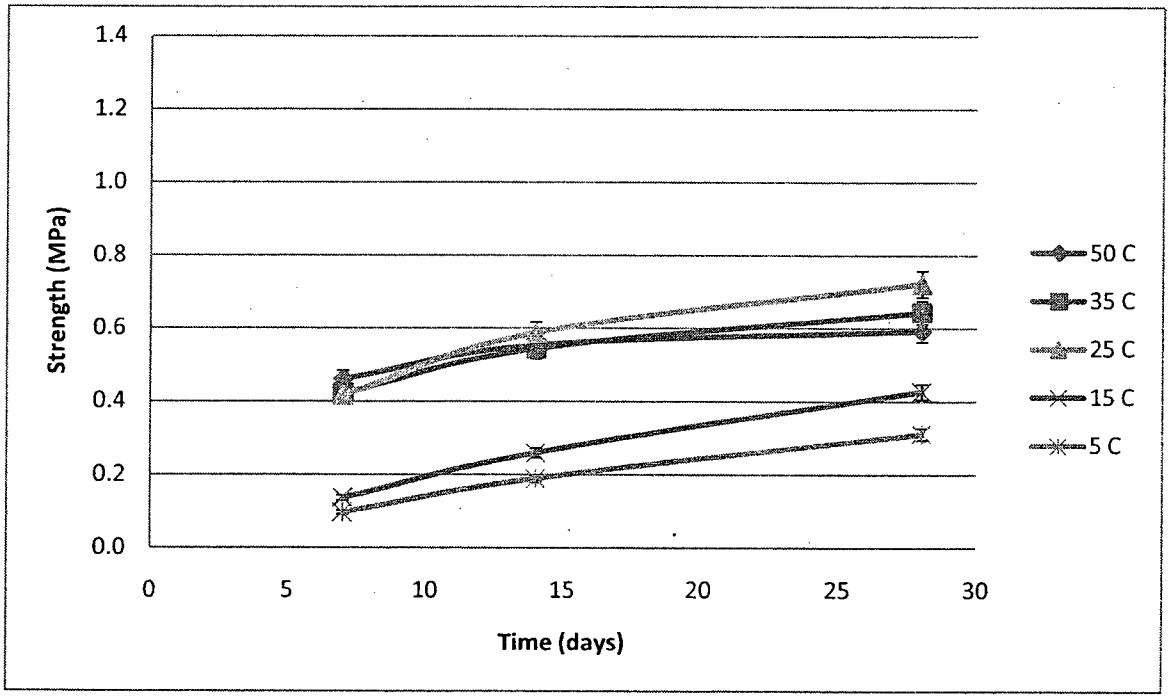

Figure 5. 5: Strength versus Curing Time at Different Temperatures in the Absence of Sodium Silicate (SC).

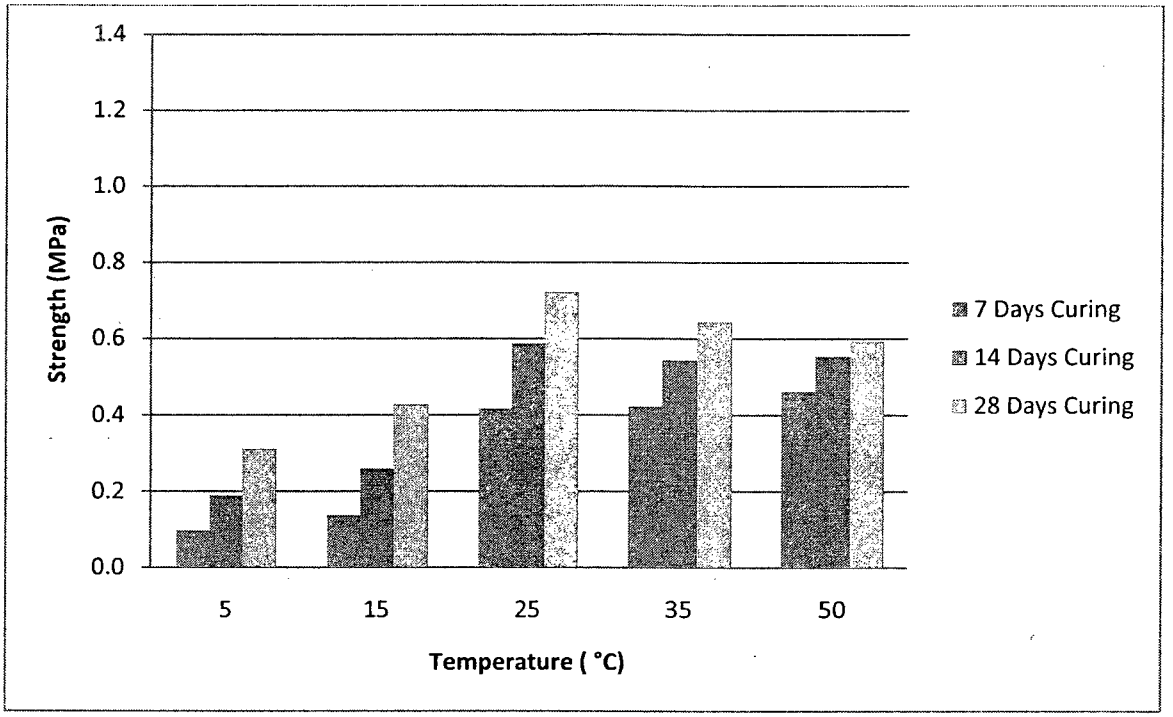

Figure 5. 6: Relationship between Strength and Curing Time at Different Temperatures in the Absence of Sodium Silicate (SC).

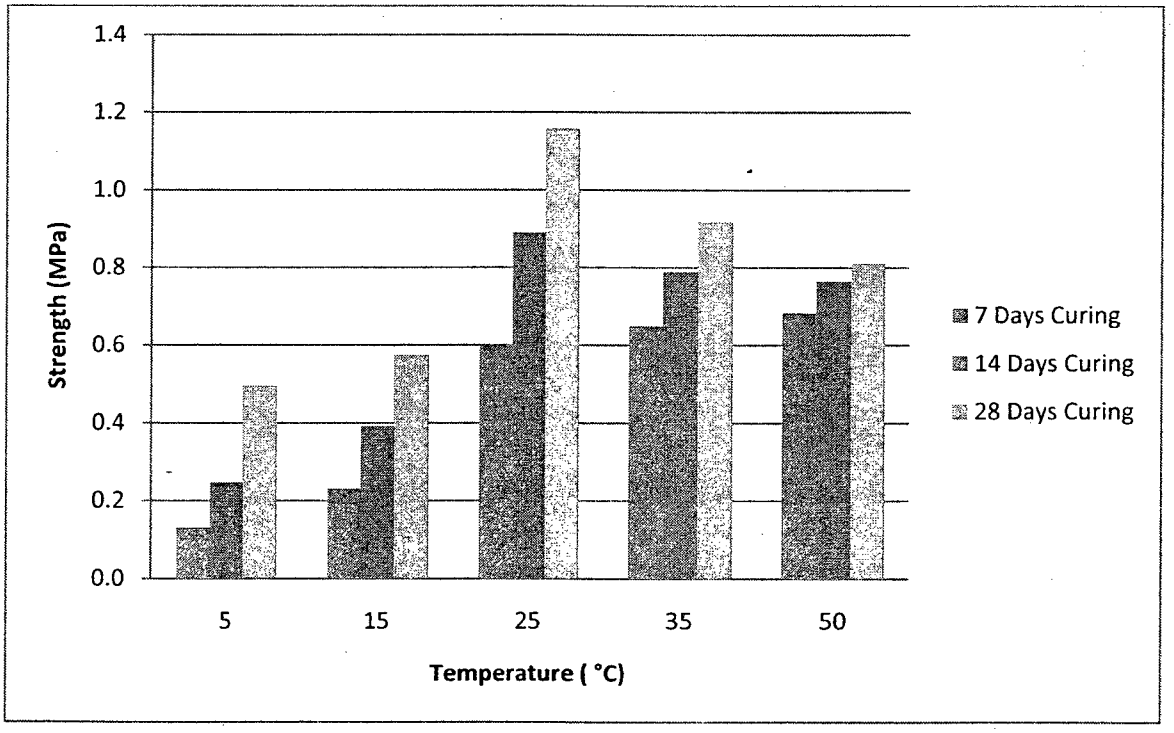

Figure 5. 7: Relationship between Strength and Curing Time at Different Temperatures in the Presence of Sodium Silicate (SCSS).

When dealing with the manufacture of cement backfill mixtures, two factors are emphasized more than others; the economical aspect and the strength of the mixture. From Figures 5.4 and 5.5 presented above, it can be seen that up to 25°C, the strength increases with increasing curing temperature. At 25°C, the strength is found to be at a maximum for samples with and without sodium silicate. At temperatures higher than 25°C, a different trend can be observed. At early ages, the strength is marginally higher at elevated temperatures but as the curing time increases, the strength increase becomes less and the final strengths are lower at temperatures above 25°C. A number of conclusions can be drawn from the above mentioned behaviour. Firstly, it is possible that at temperatures above 25°C, the water evaporates at a much faster rate, which is unfavorable for hydration since less water would be available, resulting in lower strength values. Secondly, it is also possible that initially the rate of hydration is high, resulting in a higher strength values. However, with an increase in curing time, the water is used up by the initial rate of hydration, and not enough water is left, resulting in a slower rate of hydration at elevated temperatures. As the curing time increases, the strength for all the samples increase regardless of the temperature, but the rate of strength gain decreases for temperatures above 25°C. Another observation was that samples at the same temperature that included sodium silicate had higher strengths as can be seen in Figures 5.4 and 5.5. Figure 5.6 and 5.7 illustrate the fact that for all temperatures values (5, 15, 25, 35 and 50°C) the strength is greater with an increase in curing time. However, it can be concluded from the Figure 5.6 and 5.7 that at 25°C, the final strength is greater and the rate of increase are higher than the others for samples with and without sodium silicate.

5.1.4. MERCURY INTRUSION POROSIMETRY (MIP)

The Mercury Intrusion Porosimetry (MIP) test was used to study the effect of temperature on the microstructure of backfill samples. This test was performed after 28 days curing on 5, 25 and 50°C curing temperature samples and for both type of binders i.e. Slag and Cement (SC) and Slag, Cement and Sodium Silicate (SCSS).

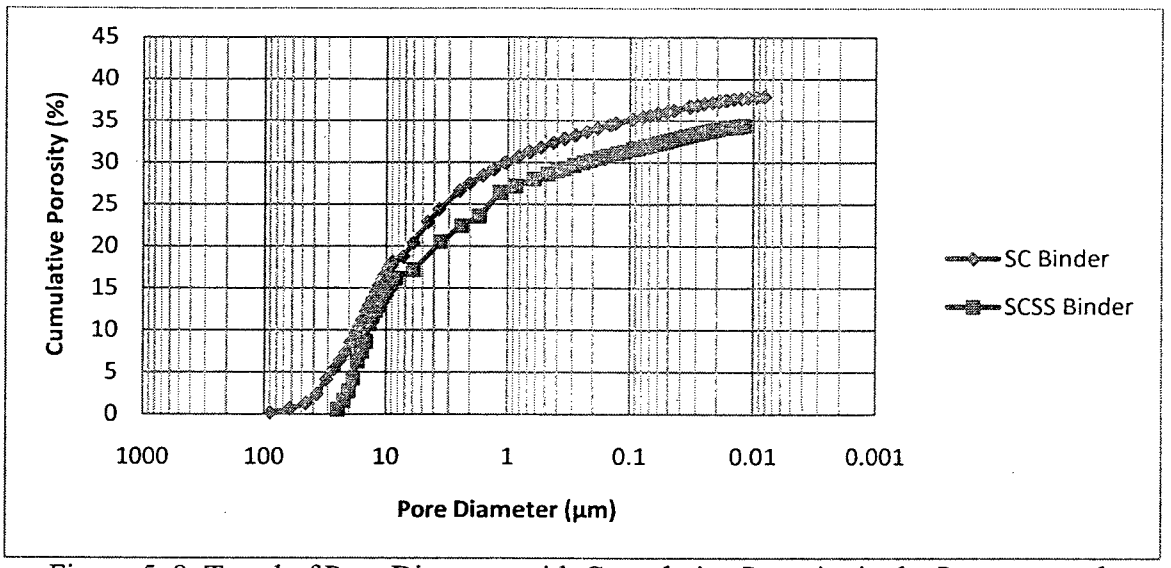

Figure 5. 8: Trend of Pore Diameter with Cumulative Porosity in the Presence and Absence of Sodium Silicate for 25˚C.

From Figure 5.8, the effect of sodium silicate on pore diameter and cumulative porosity can be studied. It is quite obvious that with adding sodium silicate content the cumulative porosity decreases, this can be explained as the change of behaviour of hydration in the presence of sodium silicate that builds less porous and finer structures.

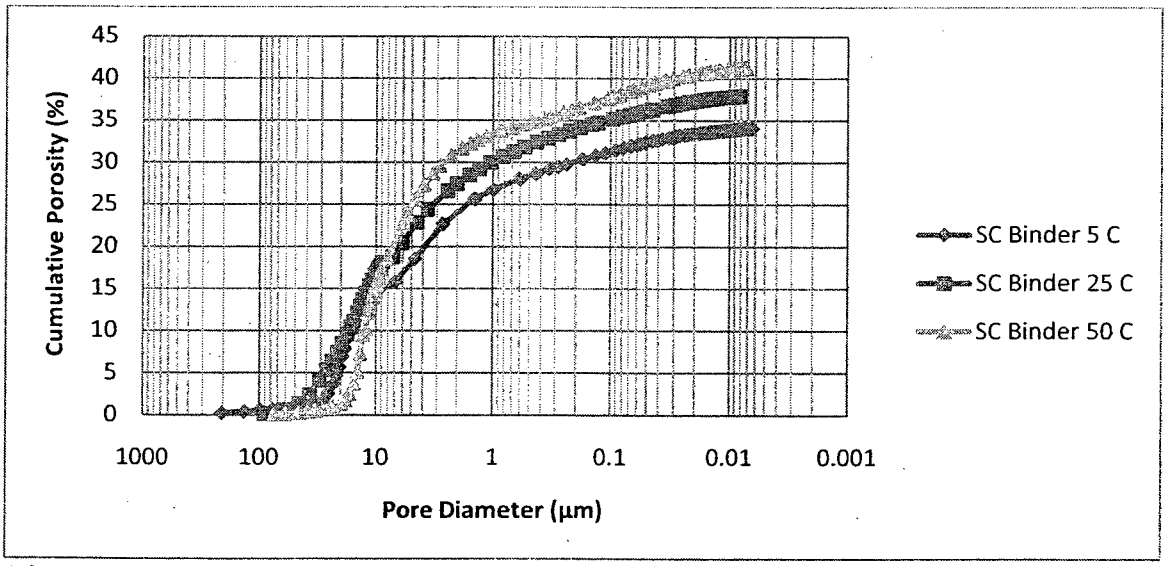

Figure 5. 9: Trend of Pore Diameter with Cumulative Porosity for different Temperature (SC Binder).

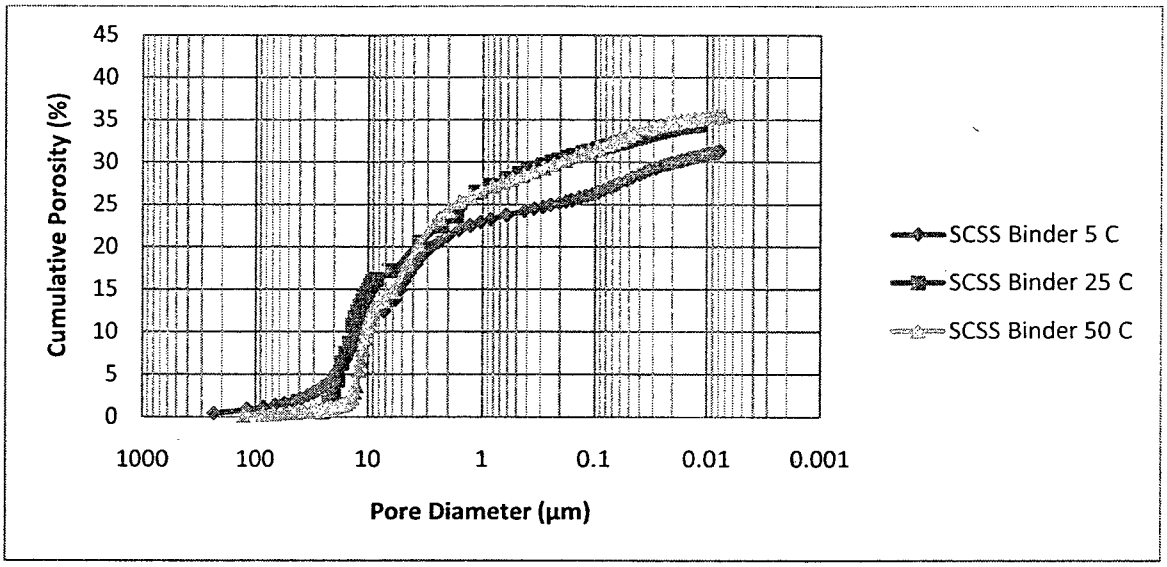

Figure 5. 10: Trend of Pore Diameter with Cumulative Porosity for different Temperature (SCSS Binder).

As illustrated in Figures 5.9 and 5.10 with increasing temperature, the cumulative porosity percentages increases for samples with and without sodium silicate. That could be the result of the fast hydration of binding agents in their early age hydration which does not allow the whole binding agents to hydrate; moreover, in the case of the samples cured at 50 degree Celsius, the water needed for hydration could be consumed and the hydration could not continue without adequate moisture.

5.2. DISCUSSION

In this section of chapter, different results of experimental part which were discussed in previous section are compared with related individual outcome.

5.2.1. RELATIONSHIP BETWEEN SETTING TIME AND UCS

Based on results from part one of this chapter and literature review chapter 2, sodium silicate is one of the best additives to the mixture recipe as it improves setting time behaviour and strength gain. Using different types of minerals as binders in addition to cement depends on mining scenarios like price, accessibility and chemical matters in individual mines. The longer the curing period, the higher strength gains no matter type

of binder or temperature conditions, but in case of set time, the increased usage of sodium silicate causes faster set. At last but not least, higher curing temperature has faster hydration rate that it observed in setting time test and it has caused crossover effect for longer period strength as it observed during UCS test.

5.2.2. MATURITY METHOD

The Maturity Method is a door to estimate strength of mixtures in a certain curing condition in relation to time and temperature. This method is applied to concrete products and has been standardized with ASTM (C 1074, 2004) but since there are similarities with backfill products mixtures using binders in them, this method may be able to assist research. Curing time and temperature are taken to account to create an equation for samples in this practice and compare this model with experimental part of research. Finally, if this model is acceptable, it will be applied to laboratory and in-situ usage.

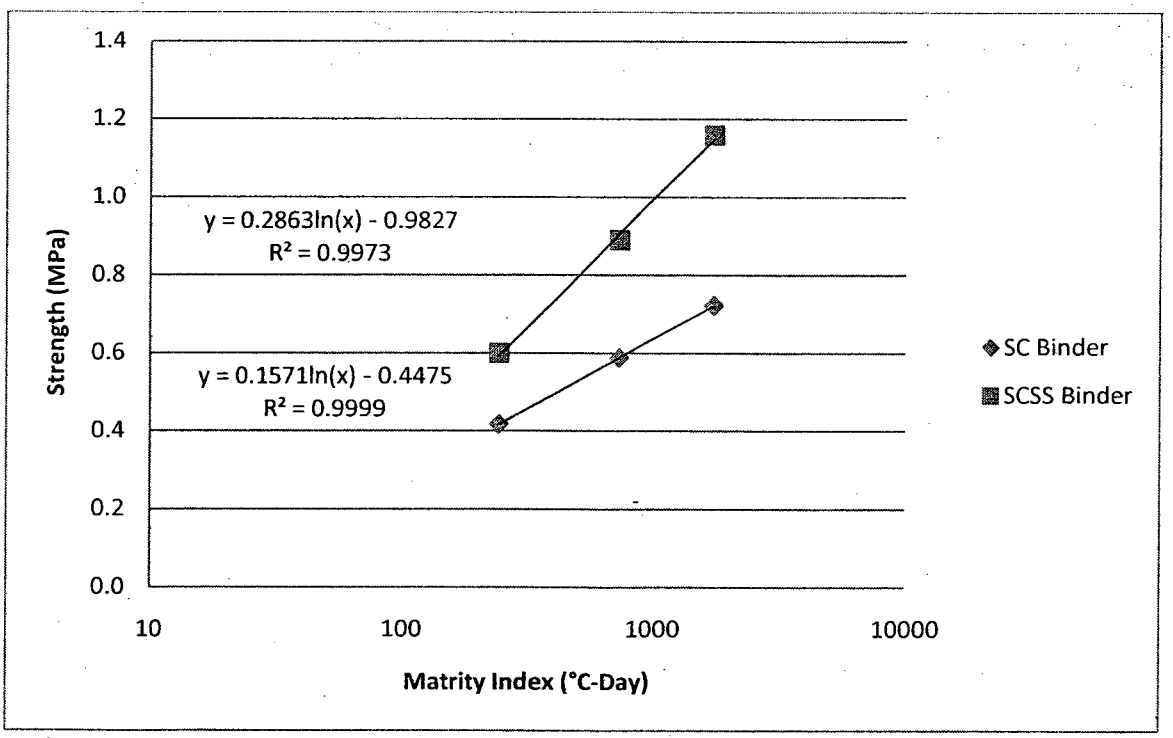

Figure 5. 11: Strength and Maturity Index for 25°C with for Both Type of Binders SC and SCSS

Figure 5.11 illustrates the maturity index relationship for the two combinations of binders in this experiment. The UCS values obtained at 25°C are used to generate the figure. From the logarithmic graphs plotted, the relationship between strength and maturity index can be read. Topcu et al. (2007) derived an equation for ultimate strength determination as follows:

$$fc = a + b.\ln(M)$$

Where fc is compressive strength of sample, a and b are regression coefficients and M is the maturity index and datum temperature considered as -10°C (Topcu et al., 2007).

As shown in Figure 5.11, both trend lines show that a good correlation of these equations should be able to estimate strength at any desired time and temperature. Taking this point to account, each binder type needs its own equation. As they appear and have been expected, SCSS binder type has a higher strength than SC binder type. Considering that changing of in-situ condition will affect the behaviour of mixture and the model cannot cover all the various and model will require modification.

In this part, two related tests i.e. Uniaxial Compressive Strength (UCS) and Maturity Method, results were compared with each other and areas which should be more considered are highlighted. The relationship determined at 25°C will be tested for its ability to predict strengths at other temperatures.

The maturity method enables to estimate the compressive strengths of mixture samples. The compressive strength of samples cured in the presence or absence of sodium silicate at different temperatures was investigated experimentally. Samples were prepared and allowed to set at constant temperatures, and the UCS test was conducted after 7, 14, and 28 days of curing. It was considered to make a comparison between the UCS values and the results obtained using the maturity index test (Figure 5.11).

Using the general equations, the UCS strength for the other temperatures (5, 15, 35 and 50°C) was predicted, shown in Table 5.2. The results were compared with the experimental ones, and it was found that the predicted UCS values were much higher

than the experimental ones as shown in Figures 5.12 and 5.13. Thus, it was concluded that a generalized equation should not be used to estimate the strength at different temperatures using the maturity indexes.

Table 5. 2: the UCS values obtained using maturity index and the normal equations at 25°C for a) Slag, Cement and Sodium Silicate Binder, b) Slag and Cement Binder.

Curing Temperature	Curing Period (Day)		
	7	14	28
5°C	0.3497	0.6643	0.907
15°C	0.496	0.8105	1.053
25°C	0.5923	0.9068	1.155
35°C	0.6643	0.9788	1.221
50°C	0.7466	1.061	1.304
a) y=0.2863lnx-0.9827 SCSS			

Curing Temperature	Curing Period (Day)		
	7	14	28
5°C	0.2836	0.4562	0.5893
15°C	0.3639	0.5365	0.6695
25°C	0.4167	0.5893	0.7256
35°C	0.4562	0.629	0.762
50°C	0.5014	0.674	0.8071
b) y=0.1571lnx-0.4475 SC			

From the above results, it is concluded that to make more clear and realistic predictions; effective curing conditions should be kept in mind for each different sample cured at other circumstances. Tables 5.2.a and 5.2.b are showing varieties of model for other temperatures that were experimented.

Comparison of results of strength in Figures 5.4 and 5.5 illustrate that the Crossover effect occurred (Verbeck and Helmuth, 1968; Carino, 1991; Brooks et al., 2007). The Crossover effect states that samples which were cured at a higher temperature in their initial curing time have higher strength at short term but at a longer period these samples are gaining less strength than the samples were cured at a lower temperature.

In the future, one of the suggestions can be made is for different bracket of temperatures diverse regression coefficients to be carried out or short term and long term equations are taken under consideration.

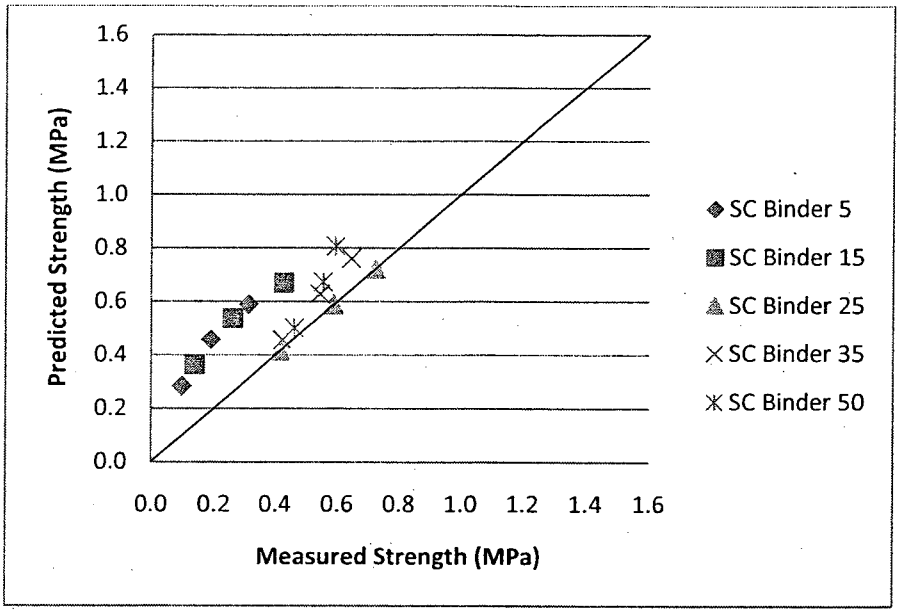

Figure 5. 12: Slag, Cement Binder Results Showing Predicted Strength vs. Measured Values.

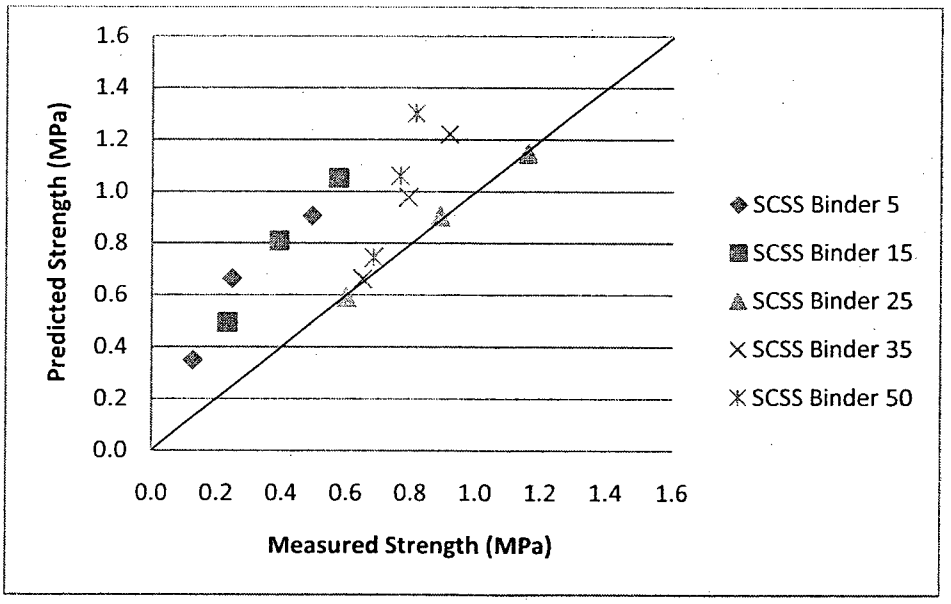

Figure 5. 13: Slag, Cement and Sodium Silicate Binder Results Showing Predicted Strength vs. Measured Values.

5.2.3. MIP AND UCS

The results are as noted already in first section of this chapter; in this portion, the results of MIP and UCS tests are discussed and relation between them to support each other is argued. The MIP test was performed on the critical points of UCS test and these points are highest and lowest curing conditions plus the highest strength point after 28 days of curing which is 25°C. These critical points were chosen for both type of binders i.e. Slag and Cement (SC) and Slag, Cement and Sodium Silicate (SCSS).

It has already seen in Figures 5.4 and 5.5; samples made with SCSS binder have higher strength than SC binder. When these observations are put side by side, in Figure 5.8, it is notable that SC binders contain more Cumulative Porosity than SCSS binders and can explain their lower strength outcome. Strength gaining process is engaged in coating surface of aggregates and other components of mixture with binders' particles, the more area needed to be covered, the more binder amount is needed. One of the reasons for higher strength SCSS samples are that sodium silicate covers more surfaces than SC binder. Cement and slag mixed binder has advantages over pure cement binder for filling fine voids in samples and SC binder builds finer and denser matrix structure, also Blast Furnace Slag is more active than other additive (Li et al., 2006).

It should be emphasized that curing temperature also has direct effect on strength and porosity of samples, higher temperature condition causes higher cumulative porosity and lower compressive strength. Generally, increasing porosity reduces strength and many researchers investigated the relationship between porosity and strength, some studied pore size in selective brackets and some other made general equation but it is not clear yet since other factors influence outcome (Ouellet et al. 2007). Strength of void materials depends on form, quality and distribution of pores (Neville, 1995; Collins and Sanjayan, 2001; Katz and Kolver, 2004; Bahar et al., 2004).

Again by comparing the result of UCS and MIP tests this phenomenon is notable and it illustrates highest curing temperature has highest porosity regardless of binder type. As it explained in previous parts, higher temperature does not necessarily produces higher compressive strength and other conditions are playing role here. A reason that can be

noted is expansion of materials and causing cracks and increasing porosity. The best performance in hydration of cemented material is the situation that there is less difference between ambient temperature and concrete temperature (Ortiz et al., 2005). Longer curing time reduces porosity and most of mineral additives react in longer period therefore hydration continues and strength will be higher (Li et al., 2006). As Cook and Hover mentioned, reducing water to binder ratio is more effective than increasing curing time on porosity (Cook and Hover, 1999).

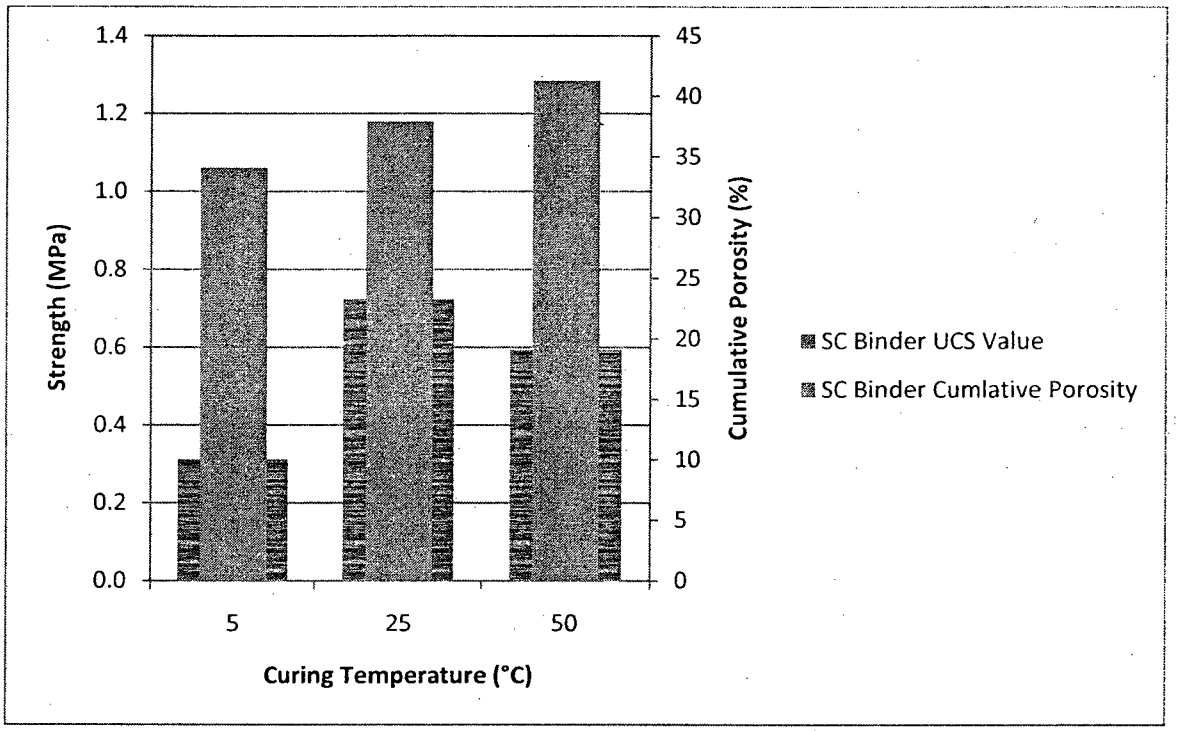

Figure 5. 14: Comparison of Strength and MIP Results vs. Curing Temperature for SC Binder

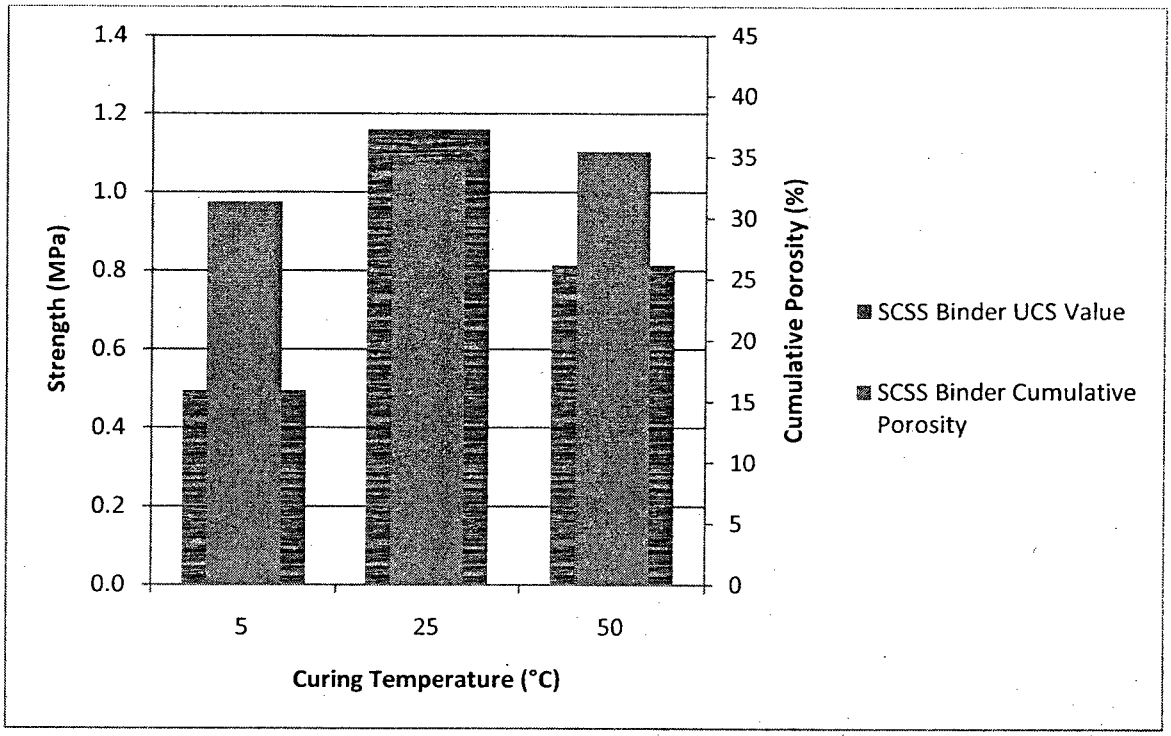

Figure 5. 15: Comparison of Strength and MIP Results vs. Curing Temperature for SCSS Binder

As Figures 5.14 and 5.15 illustrate, by increasing the temperature porosity increases but strength value as it already mentioned has alter behaviour and behaviour of porous are not clear by comparing strength of samples. This trend is similar for both type of binders but SCSS has a lower porosity and higher strength. Other important factor is that samples for MIP test should be taken from similar area of UCS test.

CHAPTER : 6 CONCLUSION

Various properties of backfill materials were investigated and different tests were carried out to find the effect of binder, sodium silicate combination, and curing temperature. Compressive strength was determined by carrying UCS. Direct shear test was a simple method employed to find the shear and normal stress properties of soil particles. To find the setting time of slag, cement and sodium silicate mixtures, Vicat test was used. With the help of mercury intrusion porosimetry test, particle size properties and other microstructural properties were drawn. Samples were cured at different temperatures using various recipes and the compressive strengths were determined. From the various test results, the key conclusions determined are:

• **The curing temperature had significant impact on strength.** In the presence of sodium silicate, the compressive strength is greater for all temperatures. The rate of increase of strength with curing time increases with temperature until 25°C. Early age strength for higher temperatures is more but the final strength is less than 25°C. There can be few conclusions that can be derived from the above behaviour. Firstly, it is possible that at higher temperatures above 25°C, the water evaporated at a much faster rate which is unfavourable for hydration. Since less water is available, thus less hydration occurs and hence less strength at higher temperatures. Secondly, it is also possible that initially, the rate of hydration is so high that its strength is also high and due to fast shaping of gelation structures there is not enough room for further reaction taking place.

• **With the increase in sodium silicate percentage, the setting time usually reduced at any temperature.** Sodium silicate accelerates hydration rate and it benefits hydraulics backfill for shortening mining cycle but it should be considered in case of distance between batching plan and empty stopes due to prevent blocking in transportation path.

• **MIP results show that in presence of sodium silicate the average cumulative porosity decreases.** Maximum incremental porosity occurs in the range between 1 to 10 micrometer pore diameter for all temperatures in presence and absence of sodium silicate.

• **The maturity method was found not to be able to predict strength of samples cured at various temperatures.** The maturity index was established and then it was used to show the relationship with compressive strength at different temperatures. A logarithmic graph was plotted and the equation of the graph was also obtained. The maturity index calculated for one temperature should not be used to accurately predict other properties of the same concrete.

•This research were investigated specific type of tailings from identical mine and same conclusions for other type of tailings may not be comparable.

CHAPTER : 7 FUTURE WORK

Although in this research was concentrated on a specific type of material and conditions which were a requirement of a mine in Sudbury, further work is needed to cover all the angles of this concept, some of them are mentioned below:

- Different ranges of particle size and other types of tailing materials
- Considering different binders due to economical issues
- Modification and improvement of mathematical model for Maturity-strength equation.
- Higher ranges of curing temperatures
- Investigation the behaviour of samples with help of triaxial test.
- Studying microscopic and chemical behaviour of binder in their early age
- Investigation of chemical effect on hardening process of binders on different tailing
- Use of industrial water to obtain effects on set and strength
- Following laboratory results for in-situ conditions

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