AN INVESTIGATION INTO A NEW BINDER

FOR HYDRAULIC BACKFILL

By

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DEDICATION

Thank God.

This thesis is dedicated to:

- My country and culture, which are my strong and deep foundations.
- My mother and father, Mahin Borzoyeh and Mahmoud Fadaei
 Kermani, my mother and father in law, Shamssi Ahmadi and
 Mahmoud Yazdanpanah, which are my inspirations.
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ABSTRACT

Over the last three decades, mine backfilling has progressively integrated into underground mining operations. The high stresses 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 minefill systems are required, but also new techniques are needed to increase the speed of the mining cycle for optimizing the mining operation. Three major purposes of mine backfill are known as 1) providing safe working condition, 2) maximizing ore recovery and 3) improving underground stability. Therefore, mine backfill has contributed greatly to the economics and environmental aspects of mining industry.

In order to improve the mechanical behaviour of fill, cementitious materials are used. These cementitious materials are expensive. As a result the consumption of these cementitious materials has to be optimized and minimized in a way that the required strength is met. The objective of this research is to investigate a new type of backfill, which is known as gelfill. Gelfill binders usually consist of alkali activators such as sodium silicate and the other cementitious materials. Sodium silicate has been used in waste

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treatment and activation of artificial pozzolans such as blast furnace slag and fly ash.

The work presented in this thesis is to evaluate the use of sodium silicate in gelfill. Consequently, the influence of mixing time, mixing sequence and curing time are studied on gelfill and silica sand hydraulic backfill. Various tests including unconfined and confined compressive strength were conducted in order to investigate the mechanical behaviour of samples. By conducting mercury intrusion porosimetery (MIP) and scanning electron microscopy (SEM), microstructure and mineralogical properties of specimens were studied.

The result of this thesis demonstrates that gelfill compared with silica sand hydraulic backfill has better mechanical properties. In addition, other variables, including: mixing time and sequence, have a significant effect on gelfill.

ABRÉGÉ

Durant les trois dernières décennies, l'utilisation de remblais est une pratique qui s'est progressivement intégrée au fonctionnement d'opérations minières souterraines. Les hauts niveaux de contraintes de stress qu'on retrouve en profondeur dans le bouclier canadien exigent une approche innovatrice en ce qui a trait au remblayage; ainsi le remblai doit être assez résistant pour supporter la charge pendant et après l'exploitation de la mine. Pour cette raison, l'amélioration et l'implantation de nouveaux systèmes de remblayage ainsi que l'introduction de nouvelles techniques sont désormais nécessaires pour accélérer le cycle minier et optimiser les opérations. Le remblayage est principalement utilisé pour trois raisons : offrir des conditions de travail sécuritaires, maximiser la quantité de minerais récupérés et améliorer la stabilité souterraine. Toutes ces raisons font en sorte que le remblayage est une pratique qui contribue à améliorer les aspects économiques et environnementaux de l'industrie minière.

Des matériaux à base de ciment peuvent être ajoutés au remblai pour améliorer ses propriétés mécaniques. Ces matériaux sont cependant dispendieux et, par conséquent, leur ajout dans le remblai doit être optimisé de manière à utiliser un minimum de matériaux à base de ciment pour obtenir la résistance recherchée. L'objectif de cette recherche est d'examiner les propriétés d'un nouveau type de remblai connu sous le nom de «gelfill ». Ce type de remblai utilise comme agglomérant des activateurs alcalins tels que du silice de sodium ainsi que d'autres matériaux à base de ciment. La silice de sodium est présentement utilisée dans le traitement des eaux usées et sert aussi d'activateur pour des pouzzolanes artificielles tel que les cendres volantes et les scories produites par les hauts fourneaux.

Le travail présenté dans cette thèse consiste à évaluer l'utilisation de la silice de sodium dans la préparation de « gelfill ». Le temps de mixage, la séquence de mixage ainsi que le temps de cure sont tous des facteurs qui ont été étudiés dans la préparation de divers spécimens de «gelfill » et de remblai hydraulique à base de sable. Par la suite, plusieurs tests ont été faits sur ces spécimens pour examiner leurs propriétés mécaniques telles le test de compression uniaxiale avec et sans confinement. Les spécimens ont aussi été étudiés sous microscope à électrons et soumis à un test d'intrusion porosimétrique au mercure (MIP) pour déterminer leurs microstructures ainsi que leurs propriétés minéralogiques.

Les résultats obtenus démontrent que le «gelfill » possède des propriétés mécaniques supérieures à celles du remblai hydraulique à base de sable de silice. Il a aussi été démontré que d'autres variables tels que le temps et la séquence de mixage ont des effets importants sur les propriétés du «gelfill ».

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CHAPTER 1: INTRODUCTION

1.1 General Background

In the mining industry, backfill is one of the most important aspects. It is defined as a "material or materials, used to fill cavities created by miming activities in order to obtain safe working conditions economically and environmentally" (Wilson, 1979). Mine backfilling has been successfully used all around the world and has progressively practiced with the development of new mining methods such as bulk mining. Backfill generally consists of mill tailings, water and different cementitious materials. Tailings or a mixture of mill tailings and cement are generally delivered to underground stopes by pipelines. These materials are subsequently delivered, consolidated or cured to support the rock walls so that adjacent stopes can be recovered. In fact, the three major purposes of mine backfilling are to i) make working conditions safe, ii) maximize ore recovery and iii) improve underground and surface stability (Hassani et al, 1989).

Normal Portland cement (NPC) used as a cementitious martial in mine backfill in the 1950's for the first time. It was generally used to increase stability of backfill. Since then, NPC and its derivatives have been widely

1 | Page

used all round the world for the same purpose. In fact, by increasing the consumption of binders, the mechanical strength of backfill can be increased (Hassani et al., 1998; Espley et al., 1970). The increasing cost of NPC, especially after the energy crisis of the 1970's, made the use of cement uneconomical; since then, various cementitious alternatives have been investigated including blast furnace slag (Uusitalo et al., 1993), fly ash (Udd et al., 1993), calcined gypsum (Amaratunga, 1995) and sodium silicate (Razavi et al., 2007). These investigations have been done in order to find an alternative which can partially or totally replace NPC. This alternative has to meet the required design strength.

The use of sodium silicate in cemented backfill produced new materials called gelfill. In this thesis, the effect of various parameters on the strength including mixing time, mixing sequence for slurry backfill and gelfill are studied.

1.2 Outline

This thesis is concerned with the fundamental investigation of hydraulic backfill and gelfill. The content of each chapter is described by the following outline:

 Chapter 1 gives a general overview of backfill and the objective of this research.

- Chapter 2 is a review of the literature regarding backfill including backfill materials and methods. It describes the properties of various binders that are used in backfill.
- Chapter 3 describes the properties and characterization of primary materials used in this research.
- Chapter 4 opens with the procedures used for preparing the samples, then explains the standards followed for this research and ends with the experimental procedures and setup.
- Chapter 5 presents the results of the unconfined and confined compressive strength of specimens.
- Chapter 6 presents the results of both scanning electron microscopy (SEM) and X-ray diffraction analysis (XRD) as well as the results of mercury intrusion porosimetery (MIP) on hydraulic back fill and gelfill samples.
- Chapter 7 finally summarizes this thesis and it ends with a recommendation for further research.

CHAPTER 2: LITERATURE REVIEW

2.1 Introduction

Mine backfilling (BF) is defined as a mining process in which the cavities created by mining activities are filled with different materials. It is not exactly clear when this method was invented however, according to F. Hassani and J. Archibald (1998) "The first recorded use of hydraulic mine backfill in North America was in 1864 at Shenandoah, Pennsylvania by the Philadelphia & Reading Coal & Iron Co. to prevent a church from being destroyed due to surface subsidence". Although BF was successfully used before World War II in a few mines, it was not until the 1960's that it became an integral aspect of underground mining methods (Hassani et al., 1998). Between 1960 and 1970, backfill technology evolved dramatically, with the use of binders, especially normal Portland cement (NPC) and its derivatives. Over the last three decades and especially after the energy crisis of the 1970's, as the cost of binders has increased dramatically, researchers have focused on finding new binders and on optimizing the consumption of binders in fill industry.

Throughout the history and development of backfill technology, three elements have had a significant effect; application of binders to BF; conservation of natural resources; and protection of the natural environment. Moreover, it seems these factors will play important roles in the future of this technology.

2.2 Backfill Purposes

As discussed by Hassani (1992), BF has been used for many purposes, including as a wall supporter, a void filler, and a disposal method for tailings, but the most important uses of BF are certainly as methods to i) provide a safe working floor for workers and equipment in mines, ii) increase ore recovery in mines, iii) dispose of waste materials and tailings, and iv) increase underground and surface stability. In cut and fill mining methods, for instance, BF has been used to provide a platform on which mining equipment operates; mining may also be carried out below, beside or through the backfill (F. Hassani et al ,1998; M. Aubertin et al., 2003).

2.3 Backfill Properties

Compressive strength and permeability are the two major mechanical properties of fill; they must be considered in order for engineers to accurately design a BF. The compressive strength depends on fill applications and mining methods. For example, the required 28-day compressive strength of BF is about 1 MPa (F. Hassani and J. Archibald, 1998; Benzaazoua, 1999), but it could reach 5 to 7 MPa in delayed backfill, with pillar recovery, depending on the span size of mines (F. Hassani and J. Archibald, 1998).

As dewatering capability is very important in hydraulic backfill, permeability plays a critical role in the design of BF. The generally accepted amount of permeability in mining industries is 100 mm/h or slightly more (F. Hassani and J. Archibald, 1998). Permeability depends on many factors including materials, the size, and distribution of fill, the temperature and density of water (Benzaazoua, 2006). Engineers have to consider the permeability of fill as an important factor in their design.

2.4 Backfill Materials

Backfill materials typically consist of three main components: i) inert materials, ii) cementitious materials (binders), and iii) water. Inert materials are the main components of BF; they are classified into four major groups, depending upon their particle size.

i) Rock

Rocks, measuring up to 300 mm, may be delivered by heavy machinery such as mining trucks and load-hauldumps (LHD), or by conveyors.

ii) Gravel

Gravel, between 1mm and 25 mm, can be transported by a pipeline.

iii) Sand

Sand, 6 µm to 6mm, can be easily transported by pipelines. iv) Mill tails

> The size of tailings depends on mineral processing methods and desliming technologies. Tails may be transported by pipelines.

2.5 Backfill Binders and Additives

Using cementitious materials, especially NPC, to improve the mechanical property of fill started in the late 1950's. Since the 1970's, using different pozzolans, NPC and its derivatives have become an industry standard. In 1971, for instance, 233,000 tonnes of NPC was consumed by Canadian mines (F. Hassani and J. Archibald, 1998). The application of binders has helped industries to improve their mining methods; for instance, the development of bulk mining methods would be nearly impossible without using cemented backfill (CBF). Moreover, the safety and ore recovery of underground mining have been dramatically increased and consequently mining costs and mining sequences have been decreased respectively (Udd, 1993).

Binders are crystalline materials (NPC) or amorphous materials (natural pozzolans); they create cementitious components after their hydration. Binders consist mainly of the three oxide components silica (SiO₂), lime (CaO), and aluminate (Al₂O₃). Table 2-1 represents the typical chemical composition of various binders. The ternary diagram, shown in Figure 2-1, shows the relative placement of major binders with respect to each other. Based on Figure 2-1and Table 2-1, slag is more similar to NPC than fly ash chemically. This is one of the main reasons NPC has been largely replaced by slag rather than by fly ash.

| | Portland Cement | Slag Cement | Fly Ash C | Fly Ash F |
|--------------------------------|--------------------|----------------|--------------|--------------|
| CaO | 65 | 45 | 25 | 3 |
| SiO ₂ | 20 | 33 | 37 | 58 |
| Al ₂ O ₃ | 4 | 10 | 16 | 20 |
| Fe ₂ O ₃ | 3 | 1 | 7 | 10 |
| MgO | 3 | 6 | 7 | · 1 |

Table 2-1 Typical chemical composition of various binders (A. M. Neville, 1998)



Figure 2-1: Ternary diagram of cementitious materials (Douglas and Malhotra,

1989)

According to American Society for Testing and Materials (ASTM ,1996), a pozzolan is defined as "a siliceous or siliceous and aluminous material which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide [lime] at ordinary temperature to form compounds possessing cementitious properties". It consists of natural pozzolans and artificial pozzolans. Burnt clay, volcanic ash, pumicite, opaline shales, cherts and many others, which are found in nature, are natural pozzolans. Artificial pozzolans such as fly ash and slag do not occur naturally; however, they are not only more readily available than natural ones, but also cheaper than NPC

therefore more suitable to replace NPC in mine backfilling. According to Neville (1996), the lists of chemical reactions of the hydration of pozzolans are (CaO=C; SiO₂=S; Al₂O₃=A; Fe₂O₃=F; H₂O=H; SO₃=Ś):

$$C + H \rightarrow CH$$
 (2-1)

$$2S + 3CH \rightarrow C_3 S_2 H_3 \tag{2-2}$$

$$A + F + 8CH \rightarrow C_8 AFH_{26} \tag{2-3}$$

$$A + C\overline{S}H_2 + 3CH + 7H \rightarrow C_4 A\overline{S}H_{12}$$
(2-4)

$$A + 4CH + 9H \rightarrow C_4 AH_{13} \tag{2-5}$$

2.5.1 Normal Portland Cement

The use of cement or cementitious materials is very old. The ancient Egyptians used calcined impure gypsum, and the Greeks and the Romans ground lime and clay tiles to produce pozzolanic cement (A. M. Neville, 1996). The Coliseum and the Pantheon in Rome are some of the structures built with this pozzolanic cement which have survived to this day. However it was not until 1824 that Joseph Monier applied for a patent for Portland cement made by heating fine clay and ground hard limestone until the CO₂ exits the process (Mehta, 1987).

After quarrying, the production of NPC consists of i) grinding raw materials including calcareous materials (i.e. limestone or chalk) and argillaceous materials (i.e. clay or shale), ii) proportioning and finally iii) burning raw materials in a rotating cylindrical kiln. The temperature in a rotary kiln reaches up to about 1450 Centigrade; at this temperature the raw materials partially fuse to produce clinker (Figure 2-3). After adding some gypsum (up to 7% of the total mass), the clinker is cooled and ground to make a very fine powder, known as Portland cement or NPC. The general schematic of the production of NPC is shown in Figure 2-2.



Figure 2-2: The production of Portland cement (www.cement.org)



Figure 2-3: Clinker (www.cement.org)

NPC is a crystalline material consisting of CaO, SiO₂, Al₂O₃, and Fe₂O₃. Four major components, alite (50-70%), belite (15-30%), aluminate, and ferrite (5-15%), constitute cement. The chemical composition and abbreviation of these major components is shown in Table 2-2. The following shortened notations have been used to describe each oxide: CaO=C;

SiO₂=S; Al₂O₃=A; Fe₂O₃=F; H₂O=H; SO₃=Ś.

In the first 30 days of hydration of cement, the strength of Portland cement is derived by alite, and the long term strength (one year or so) is developed by belite (Mehta, 1987).

The lists of chemical reactions of NPC are (Mehta, 1987):

$$\begin{aligned} 2C_{3}S + 6H \to C_{3}S_{2}H_{3} + 3CH & (2-6) \\ 2C_{2}S + 4H \to C_{3}S_{2}H_{3} + CH & (2-7) \\ C_{4}AF + 4\dot{C}H + 22H \to C_{8}AFH_{26} & (2-8) \\ C_{3}A + C\overline{S}H_{2} + 10H \to C_{4}A\overline{S}H_{12} & (2-9) \\ C_{3}A + CH + 12H \to C_{4}AH_{13} & (2-10) \end{aligned}$$

As shown in Equations 2-6 and 2-7, the hydration of Portland cement (alite and bellite) is a lime-producing reaction; however, as can be seen in Equations 2-3 to 2-5, the hydration of pozzolan is a lime-consuming reaction. In addition, both pozzolan and NPC reactions generate calcium silicate hydrates, also known as CSH gel($C_3S_2H_3$, C_8AFH_{26} , $C_4A \ SH_{12}$, and C_4AH_{13}). This is the major cementitious compound produced by the chemical reaction of both type of binders.

Table 2-2 Main compounds of Portland cement (after A. M. Neville, 1996; Mehta,

| Oxide Composition | Name | Abbreviation | Quantity |
|-------------------------------------|-----------------------------|-------------------|-----------------------------|
| | | | (% of total mass of cement) |
| 3CaO.SiO ₂ | Tricalcium silicate (alite) | | 50-70 |
| 2CaO. SiO ₂ | Dicalcium silicate (belite) | C ₂ S | 15-30 |
| 3CaO.Al2O3 | Tricalcium aluminate | C ₃ A | 5-15 <u>p</u> |
| 3CaO.Al ₂ O ₃ | Tetracalcium aliminoferrite | C ₃ AF | 5-15 |

In addition to the major compounds shown in Table 2-2, Portland cement contains many minor compounds, such as MgO, TiO₂, Mn₂O₃, K₂O and Na₂O. The amounts of these minor compounds are usually less than 3% of the total mass of cement; however, they have significant effect on the strength of cement and concrete. For instance, Na₂O and K₂O, known as the alkalis, react with some aggregates causing some products that disintegrate the concrete (A. M. Neville, 1996; Mehta, 1987). Therefore, they are very significant even though they are considered minor compounds by quantity.

As shown in Figure 2-4, the annual global production of cement has dramatically increased since the 1920's, from less than 100 million tonnes to about 1.6 billion tons. In Canada, the total annual production in 1996 was about 10 million tons. As can be seen in Figure 2-5, Eastern Canada (Ontario and Quebec) approximately produced 70% and Western Canada 28%. Archibald et al.,1996, states approximately 5% to 6% of the total annual

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production of cement in Canada are consumed by the Ontario mining industries; the cost of this consumption is about \$70 million dollars per year (assumed NPC price \$150 per ton).





(http://minerals.usgs.gov)



Figure 2-5: Percentage of the production of NPC in Canada

(Total annual production about 10 million tonnes, http://minerals.usgs.gov)
2.5.2 Blast Furnace Slag

Blast furnace slag (BFS) is described as a non-metallic by-product of manufacturing iron and steel in a blast furnace where the residual siliceous and aluminous materials remain after the separation of the metal from ore, which reacts with limestone and coke ash. BFS approximately comprises up to 20% (by mass) of the metal production. Figure 2-6 presents a general schematic of a blast furnace and the production of iron and slag. As shown, the molten slag is lighter and floats on the top of the molten iron.

Molten slag is cooled and ground then, depending on the cooling method, four different types of blast furnace slag are produced: air-cooled, foamed, granulated, and pelletized. In the production of granulated slag, for example, molten slag is cooled by immersing in water or using water jets.

The major setback to using slag as a binder in cement and mining industries is the low rate of hydration and strength acquisition. However, because of the low price and availability of slag, it has been widely used all around the world. For instance, the annual production of BFS is approximately 350,000 tons in Sault Ste. Marie and Hamilton, Ontario. This slag is used as a binder agent for mining backfill in Northern Ontario and used as aggregate in Southern Ontario (F. Hassani; J. Archibald, 1998). One

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of the most important companies that has partially replaced cement with slag is INCO. In this company, a composed binder consisting of 90% slag and 10 % NPC at an overall binder/tailings ratio approximating 1/15 - 1/30 has been employed (Archibald, 1998).

The feasibility and viability of the partial replacement of cement by slag has been demonstrated by many individual investigators (S. M. Razavi et al., 2007; Yu and Counter, 1983; Douglas and Malhotra, 1989). This replacement can cause a dramatic decrease in mine backfill costs, and consequently an increase in mine revenues (Thomas 1978).



Figure 2-6: Blast furnace operation and blast furnace slag production

(www.slagnavigator.net)

2.5.3 Fly Ash

Fly ash (FA), also known as coal ash, is one of the fine residues of the combustion of pulverized coal in thermal power generating plants. Figure 2-7 shows the general schematic of a coal power plant. Coarse fly ash which collects at the bottom of the boiler is called bottom ash. FA is generally captured by mechanical collectors or electrostatic precipitators from the smoke stack. FA comprises up to 10% (by mass) of combusted coal. It used to be treated as waste and disposed of in landfills; however, since the 1930's, it has been used in soil stabilization, waste treatment as well as construction projects.



Figure 2-7: General Schematic of the Production of Fly Ash in a Thermal Power Plant (www.slagnavigator.net).

Fly ash is composed of particles that are generally finer than cement, with a diameter size from 1 μ m to150 μ m. As can be seen in Figure 2-8, these particles are mainly glassy and spherical.



Figure 2-8: The SEM image of fly ash particles (www.concretedecor.net).

According to ASTM C618 (1996), Fly ash is classified as Class-C and Class-F. Produced when sub-bituminous or lignite coals are burned, Class-C is selfcementitious, but Class-F, which is produced by burning bituminous coal, cannot be activated without the addition of lime (F. Hassani et al., 1998). Archibald et al. (1995) states that NPC can be replaced by up to 60% Class-C fly ash in cemented backfilling. In 1994, the Energy Board of Canada estimated the annual national production of fly ash is 3.63 million tonnes of both classes however Class-F is produced more than Class-C. Figure 2-9 shows the production of fly ash in Canadian provinces. As can be seen, Quebec does not produce any fly ash since in this province; there is no coal power generation plant.



Figure 2-9: The annual production of fly ash in Canada (modified after F. Hassani,

1998).

2.5.4 Sodium Silicate

Known as water glass, sodium silicate manufactured from varied proportions of Na_2CO_3 and SiO_2 by smelting silica with sodium carbonate at 1100-1200 Centigrade (Figure 2-10). The major applications of sodium silicate are in pulp and paper industries, detergent industries, and waste treatments and are used as a binder in construction.



Figure 2-10: The schematic of the production of soluble sodium silicate (www.nzic.org.nz).

In civil engineering and mining industries, especially where concrete is used, sodium silicate has been used for different purposes including an alkali activator of slag and fly ash, a penetrating sealant and a hydration accelerator. Once it is added to a concrete admixture, it can react with portlandite, which is an undesirable compound in cement hydration, and then produce a C-S-H gel which is desirable for binding aggregates in the concrete. The elimination of portlandite:

- Reduces the micro porosity of concrete, hence the total porosity.
- Increases the compressive strength of concrete.

- Decreases the permeability of concrete, hence increases its durability.
- Increases the acid attack resistance of concrete.

Sodium silicate has been used as an accelerator for Portland cement, fly ash and slag. This acceleration depends on the type of sodium silicate; for instance, insoluble silicates have a lower acceleration rate because they should be dissolved (Razavi, 2007). This property has been used in many applications like shotcreting to have set immediately. Sodium silicate has been widely used for the solidification and stabilization of waste and hazardous materials. It can be used by its own or in conjunction with the other binders.

2.6 Backfill Method

2.6.1 Hydraulic Backfill

Widely used in mining operations, hydraulic backfill (HBF) or slurry backfill (HBF) is described as "a classified, highly permeable, low solids density mixture of aggregate and water (averaging between 60% to 69% solids, by weight)" (F. Hassani; J. Archibald, 1998). It is classified by two different methods: cemented and uncommented hydraulic backfill. It is usually transported by pipelines.

Use of hydraulic backfill contributes to many technical advantages and disadvantages.

Advantages:

- The infrastructure needed for and the operations using HBF are relatively simple.
- The consistency of backfill materials can be easily maintained.
- The concentrations of solids may be decreased consequently, optimization of cement consumptions can be optimized.
- The control of backfill materials is easy and secured.
- The preparation of slurry can be done both on the surface and underground.
- The possible elimination of pumping systems by optimizing pipeline lay-outs.

Disadvantages:

- The need to recover excess water from stopes.
- The possibility of materials and binders separating.
- The construction of bulkheads and its dewatering facilities are time consuming.
- The possibility of binder washout.

2.6.2 Paste Backfill

Paste backfill is a relatively new technology; it was first used in Germany in the early 1980's. It is defined as a high solid density, low permeable mixture of very fine materials with approximately 15% of materials (by mass) smaller than 45 μm (Hassani and Archibald, 1998; Benzaazoua, 2002). This uniform mixture usually consists of fine mineral processing tails, water and binders. The most important factor in paste backfill design is the distribution of the particle size of tails (Hassani and Archibald, 1998; Benzaazoua, 2003).

There are major advantages and disadvantages associated with paste backfill;

Advantages

- The possibility of using total fine tailings.
- The reduction of mining cycles due to early strength development.
- The reduction of binder consumption.
- The reduction of surface tailing storage.
- The elimination of excess water recovery due to the high solids density of mixtures.

Disadvantages

- The possibility of high pressure in pipelines.
- The need for superior dewatering technologies.
- The higher capital cost due to requirements of dewatering technologies.
- The complexity of the infrastructure, operations and technical supervisions of paste backfill.

2.6.3 Rock Backfill

The use of coarse rock for backfilling underground excavations is known as rock backfill (RBF). It typically consists of sized or unsized coarse waste rock aggregates. In consolidated rockfill (CRF) in addition to rock aggregates, different amounts of various binders are used to increase the mechanical properties of fill (P. Farsangi, 1996). In making CRF generally sized rockfill aggregates have been used. As can be seen in Figure 2-11, these aggregates can be mixed with cement slurry before the placement or can be post consolidated by adding slurry mixture after the placement (Annor, 1999; Hassani, 1999; Razavi, 2007). According to Farrsangi (1996), unconsolidated rockfilling is the best method for filling the void for providing passive support; this type of fill has a negligible free standing height capacity and is used when the stope will not be exposed in future pillar recoveries.



Figure 2-11: The schematic of pre-consolidated rockfill with cement slurry (Farsangi et al., 1996).

Technical advantages and disadvantages associated with paste rock

backfill are

Advantages

- The reduction of surface tailing storage.
- The infrastructure and the operations of RF are relatively simple.
- The elimination of stope dewatering systems.
- The possibility of attaining a relatively high compressive strength.

Disadvantages

- The reduction of the ability of tight filling in stopes.
- The possibility of binder washout.
- The possibility of materials and binders separating.

As shown in Table 2-3, a comparison between different backfilling methods was done by Hassani and Archibald (1998). This comparison presents the advantages, disadvantages and properties of slurry fill, paste fill and rockfill.

Table 2-3: Comparison of the principal properties of slurry fill, paste fill and rockfill

| Properties | Slurry Fill | Paste Fill | Rockfill |
|---------------------------------|---|--|---|
| Placement State | 60% to 75% solids (by weight) | 75% to 85% solids (by weight) | Dry |
| Underground Transport System | Borehole/pipeline via gravity | Borehole/pipeline via gravity, can be pumped | Raise, mobile equipment, separate cement system |
| Binder Application | Cemented or uncemented | Cemented only | Cemented or uncemented |
| Water to Cement Ratio (w/c) | High w/c ratio, low binder strength | Low to high w/c ratio. Low to high binder strength | Low w/c ratio, high binder strength |
| Placement Rate | 100 to 200 Tonne/hr | 50 to 200 Tonne/hr | 100 to 400 Tonne/hr |
| Segregation | Slurry settlement and segregation, low strength development | No segregation | Stockpile and placement segregation, reduced strength and stiffness |
| Stiffness | Low stiffness | Low or high stiffness | High stiffness if placed correctly |
| Tight Filling | Cannot tight fill | Easy to tight fill | Difficult to tight fill |
| Binder Quantity | Requires large quantity of binder | Usually lower quantity of binder required | Moderate binder quantities |
| Barricades | Expensive | Inexpensive | Not necessary |
| Water Runoff | Excessive water runoff | Negligible water runoff | No water runoff |
| Capital Costs | Low capital costs | High than for slurry fill | Moderate capital costs |
| Operating Costs | Low distribution costs; lowest cost for an uncemented fill | Lowest cost for a cemented fill | High operating costs |

(Hassani and Archibald, 1998)

2.7 Summary and Conclusion

Mine back operation has become an integral part of the underground mining operation, and any type of optimization or improving on any part of such operation has substantial effect on the economics of the mining operation. One of the serious issues is the reduction in mining cycle. Improving the cycle of minefill operation could contribute to the overall optimization and economics of the mining cycle.

Based on the literature review and mining industry needs, one of the most important methods for the optimization of backfill materials is to increase the fill strength while simultaneously reducing the mining cycle; moreover, this optimization has to consider the operation cost. However, it must be noted that the optimization of fill materials has to meet the safety required by the mining industry.

To optimize backfill materials, numbers of investigations have been conducted. Some of these investigations are as follows;

- The backfill porosity reduction by optimizing size gradation (Hassani et al, 1998; Swan et al., 1993; Razavi et al., 2007).
- The moisture control of backfill materials (Thomas, 1978; Ouellet et al., 2003)

The use of new binders (Hassani et al.,1999; Benzaazoua et al.,1999,2002; Kesimal et al.,2004; Razavi et al., 2007; Doucet et al., 2007).

Since 2004, a new fill material called gelfill has been developed. Gelfill is a fill material which has an alkali activator (sodium silicate or calcium silicate) as part of its constituent. This is usually added to fill materials (sand or tailings), binders (Portland cement and, or other artificial pozzolans). The practical application of this material has not been fully realized. However further research is necessary to detail its influence on fill behaviour.

Between 2005 and 2007, limited laboratory tests were conducted with sodium silicate at the McGill Geomechanics Laboratory and at CANMET Mining and Mineral Sciences Laboratories (MMSL) to evaluate trends in the strength development and water retention of this novel backfill material. These preliminary tests indicated that gelfill behave favourably compared with hydraulic cemented backfills. However, the results of these tests did not answer some of the key questions about many parameters which do have effects on gelfill materials. For instance, it was discovered that the mixing methods dose effect the mechanical properties of gelfill (Razavi et al., 2007, Doucet and Tarr, 2007); however, it was not explained in depth; moreover, the microstructural properties of gelfill have not been investigated in detail. Therefore, although gelfill has economic and mechanical advantage over hydraulic fill, it still has not been practiced for underground mining in Canada.

This research is a fundamental study to contribute to the understanding of the effect of some parameters namely mixing time and sequence, as well as the curing time for gelfill and hydraulic backfill. The research, therefore, mainly focused on the mechanical and physical properties of gelfill as well as the microstructural characteristics of gelfill.

CHAPTER 3: THE PROPERTIES AND CHARACTERIZATION OF PRIMARY MATERIALS

3.1 Introduction

Mine backfill materials consist of three major components namely inert materials, binders and water. The properties of these individual components are the most important factors that define the mechanical and chemical properties of backfill (Fall, 2005; Archibald, 1998; Benzaazoua et al., 2004; Kesimal et al., 2004).

In this chapter, classifications of the primary materials used in this thesis are provided. To conduct such classifications, soil and rock mechanics techniques and standards were applied. Additionally, concrete technology standard methods were used in some cases.

3.2 Silica Sand

Mineral processing tails and alluvial sands are mostly used in mine backfilling (Hassani and Archibald, 1998; Fall, 2005; Razavi, 2007). The mechanical and chemical properties of backfill are significantly influenced by the chemical composition of these inert materials; in order to eliminate the effects of their chemical composition, pure silica sand was used instead to produce backfill specimens in this bench top study. This silica sand was obtained from Opta Minerals Inc. Figure 3-1 shows the texture and shape of the sand, and Table 3-1 shows its chemical composition. The sand's physical properties are shown in Table 3-2.



Figure 3 – 1: SE image of Silica Sand

| Table 3-1: The chemical | composition of sand (| (After the MSDS of Opta | ļ |
|-------------------------|-----------------------|-------------------------|---|
| | Minerals Inc.). | | |

| Material | SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | TiO ₂ | CaO |
|----------|------------------|--------------------------------|--------------------------------|------------------|-------|
| | (%wt) | (%wt) | (%wt) | (%wt) | (%wt) |
| Sand | 99.50 | 0.05 | 0.03 | 0.03 | 0.01 |

| Material | Colour | Mineral | Specific Gravity | Grain | Bulk Density | Hardness |
|----------|--------|---------|------------------|---------|---------------------|----------|
| | | | (gr/cm^3) | Shape | Lbs/ft ³ | (Mohs) |
| Sand | White | Quartz | 2.65 | Rounded | 99 | 7 |

 Table 3-2: The physical properties of sand (Modified from the MSDS of Opta Minerals Inc.).

Since the particle size distribution significantly influences the mechanical strength, permeability and porosity of backfill (Razavi, 2007; Benzaazoua et al., 2006; Kesimal et al., 2004), the size distribution of sand must be standardized. Therefore, in these tests, the silica sand was crushed in order to reach a typical particle size distribution of declaimed tailings from the Creighton mine in Sudbury. For this purpose, a laboratory scale Denver grinding rod mill was used. The grinding media consisted of 17 kg rods, which were made up of four different diameters. Both the mill and the grinding media were made of mild steel.

The particle size distributions of crushed sand and tailings were determined by using the combination of sieving and laser diffraction methods (ASTM C136-96; 1996). The sieve analysis was used for materials retained by the number 100 sieve ($150\mu m$), and for fine materials (finer than $150\mu m$) the Laser Diffraction analysis was deployed; results are shown in Figure 3-2. In order to classify the sand, the Massachusetts Institute of Technology (M.I.T) Classification was conducted and the results are shown in Table 3-3.



Figure 3-2: Particle size distributions of the silica sand

Table 3-3: The particle sizes of the silica sand based on the M.I.T. Classification

System

| Material | Gravel (%) | Sand (%) | Silt and Clay (%) | Category |
|-------------|------------|----------|-------------------|-----------|
| Silica Sand | 0 | 24.6 | 75.4 | Silt-Sand |

In order to classify the sand, Unified Soil Classification System (Bowles, 1988) was employed; the coefficient of uniformity and the coefficient of gradation were calculated. The coefficient of uniformity (Cu) is defined as:

$$C_u = \frac{D_{60}}{D_{10}}$$
(3-1)

Where:

D60 = the diameter of the particle of which 60% of the particles are smaller

D10 = the diameter of the particle of which 10% of the particles are smaller

The C_u shows the general slope of the particle size distribution curve. The range of particle sizes is larger when the value of the coefficient of uniformity is higher.

The coefficient of curvature (C_c), also called the coefficient of gradation (C_g), describes the shape of the particle size distribution curve between D10 and D60 and is defined as:

$$C_g = \frac{(D_{30})^2}{D_{10}D_{60}} \tag{3-2}$$

Where:

D30 = the diameter of the particle of which 30% of the particles are smaller

According to the Unified Soil Classification System (Bowles, 1988), well-graded sand has a C_u value greater than 6 and a C_g value between 1 and 3. Since the coefficient of curvature and the coefficient of uniformity of

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the silica sand used is 0.84 and 2.48 respectively, the sand was classified as poorly graded sand.

D10 and D50, defined as the diameter of the particle of which 10% and 50% of the particles are smaller, are the particles mostly used in mining industries and soil mechanics. Their measurements are shown in Table 3-4.

Table 3-4: the Silica sand D50 and D10 values

| Material | D10 (microns) | D50 (microns) |
|-------------|---------------|---------------|
| Silica Sand | 21.80 | 45.40 |

3.3 Binders

A combination of 10% of type 10 Portland cement (PC_x) and 90% of blast furnace slag (BFS) was used as the main binder in this research. Both of PC_x and BFS were obtained from Lafarge.

3.3.1 Blast Furnace Slag

The particle size distribution of binder is one of the most important factors in the hydration of binders (Metha, 1987; Neville 1996). As the

particles of BFS and PC_x were too small to do the sieve analysis, the laser light diffraction technique was employed to obtain the particle size distribution of BFS; the results are shown in Figure 3-3. *D10* and *D50* values of this slag are 1.53 μ m and 4.41 μ m respectively. Using the M.I.T. classification system, the Lafarge BFS can be classified in the "Silt-Clay" category. Figure 3-4 is the SEM image of this slag, which shows the shape, size and texture of slag. To determine the chemical compositions of the BFS, the XRD analysis was conducted, and the results are shown in Table 3-5.



Figure 3-3: The particle size distribution of Lafarge BFS

| | SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | CaO | MgO | K ₂ O | Na ₂ O | SO3 |
|----------|------------------|--------------------------------|--------------------------------|-------|-------|------------------|-------------------|-------|
| Material | (%wt) | (%wt) | (%wt) | (%wt) | (%wt) | (%wt) | (%wt) | (%wt) |
| Slag | 36.42 | 10.21 | 0.58 | 38.12 | 9.42 | 0.44 | 0.52 | 3.01 |

Table 3-5: The chemical composition of BFS



Figure 3-4: SEM image of slag (Razavi, 2007)

3.3.2 Type 10 Portland Cement

As with the slag, the laser light diffraction technique was used in order to obtain the particle size distribution of Portland cement; results are shown in Figure 3-5. *D10* and *D50* values of this cement are 1.21 µm and 3.45 µm respectively. Considering the particle size distribution, this cement is classified in the "Silt-Clay" category according to the M.I.T. classification system. To determine the chemical compositions of this Portland cement, the XRD analysis was deployed, and the results are shown in Table 3-6. Figure 3-6 is the SEM image of typical Portland cement, which shows the shape, size and texture of slag.



Figure 3-5: The particle size distribution of Lafarge type 10 Portland cement



Figure 3-6: SEM image of Lafarge type 10 Portland cement (Razavi, 2007)

| | SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | CaO | MgO | K ₂ O | Na ₂ O | SO3 |
|----------|------------------|--------------------------------|--------------------------------|-------|-------|------------------|-------------------|-------|
| Material | (%wt) _ | (%wt) | (%wt) | (%wt) | (%wt) | (%wt) | (%wt) | (%wt) |
| Cement | 19.39 | 4.61 | 2.27 | 61.13 | 2.01 | 0.71 | 2.01 | 3.3 |

 Table 3-6: The chemical composition of Portland cement

3.3.3 Sodium silicate

Type N[®] sodium silicate was used in this investigation. This sodium silicate was provided by the PQ National Silicate Company. Table 3-7 shows the properties of the sodium silicate.

| Sodium silicate properties | Standard | Maximum | Minimum |
|-------------------------------------|----------|---------|---------|
| Na ₂ O, % | 8.90 | 9.10 | 8.70 |
| SiO ₂ , % | 28.66 | 29.00 | 28.20 |
| Weight Ratio, % | | | |
| SiO ₂ /Na ₂ O | 3.22 | 3.27 | 3.15 |
| Density, @ 20° C | 41.00 | 41.50 | 40.5 |
| Specific Gravity, @20° C | 1.394 | 1.401 | 1.388 |
| Viscosity @ 20°C Centipoises | 177 | 213 | 141 |
| Solids, % | 37.56 | 38.10 | 36.90 |

Table 3-7: The properties of sodium silicate (PQ National Silicate)

3.4 Water

The effect of the soluble chemical components of water on the strength of cemented backfill and mortar is known and has been investigated by many researchers including Neville, 1996; Hassani and Archibald, 1998; Kesimal, 2005; Razavi, 2007 and many others. Due to use of mine process water, this effect is significant in mining industries. However, in this research, in order to standardize and eliminate the effect of water chemical compositions, distilled water was used; therefore, the influence of soluble chemical components in water is almost negligible as compared to mine process water.

CHAPTER 4: EXPERIMENTAL PROCEDURES AND SETUP

4.1 Objective of the Experiments

A literature review of minefill processes indicates that many parameters do affect the physical and chemical properties of sand, hydraulic backfill and gelfill. However, in this research the effects of the following factors were investigated:

- The effect of binder types.
- The effect of quantity of sodium silicate.
- The effect of mixing time.
- •) The effect of the sequential addition of ingredients.
- The effect of curing time.

In order to conduct this investigation, many hydraulic backfill specimens were prepared and consequently many different tests were conducted. In this chapter, the procedure for preparing the specimens is described. In addition, the different test procedures employed in this project are defined. Finally, it is important to note that the standard tests used were done primarily according to a standard developed at the McGill University Geomechanics Laboratory, as well as other standard tests, including the American Standard and the British Standard.

4.2 Preparation of Backfill Mixture

After measuring the desired amount of silica sand for a mixture (3300 gr), the sand was poured into a 5-quart, stainless steel bowl. For mixing the ingredients, a kitchen appliance (Kitchenaid, Commercial 5 Series, Model: KM25G0X) was used. In this mixer, a stainless steel wire whip blade was used for blending and preparing homogenous samples. At first, for a short period of time, the dry sand was mixed alone. At this stage, the rate of the agitator was set at the lowest possible speed of the 150-RPM mixer to make uniformly mixed sand. Then, according to a specific sequence of ingredient addition, the remaining materials (water, binders and sodium silicate) were added to the mixing bowl in the following order. First, $\frac{3}{4}$ of the water was added to the sand in the bowl, then the binders, including Portland cement and slag, were poured into the mixture (5% of total dry weight). Consequently, sodium silicate was inserted (0.4% of total dry weight) for the gelfill specimens. Finally the remaining water $\binom{1}{4}$ was poured into the mixture.

All mixtures were mixed for 5 minutes, except those made in order to investigate the effect of mixing time on the strength of the samples. In these cases, mixing time was changed from 5 to 30 minutes.

After the required mixing time, the mixtures were poured into cylindrical, plastic polyvinyl chloride (PVC) moulds. The specifics of this PVC mould are shown in Figure 4-1. As can be seen, the mould meets the recommended ratio of 2:1 for height to diameter, recommended by American Society for Testing and Materials (ASTM ,1996),for unconfined compressive strength (UCS) tests. In order to simulate drainage in the laboratory, as happens in hydraulic backfill practice in a mine stope, the bottom of the moulds were perforated by 30 equally distributed holes (Figure4-1b).However, in order to prevent fine particles from escaping, a filter made of a geo-textile was used (Figure4-1b). This geo-textile material supplied by Texel Company and was cut to conform to the bottom of the moulds.





Figure4-1: A cylindrical, plastic, polyvinyl chloride (PVC) mould (a), and a geo-textile filter (b).

In order to cure the samples, all the moulds were transferred to a humidity chamber (Figure 4-2). In this humidity chamber, the temperature and humidity were adjusted and controlled to $90 \pm 5\%$ and the temperature to 25 ± 2 °C. In this way, underground mine conditions were simulated in the laboratory (Razavi, 2007; Benzaazoua et al., 2002). As humidity and temperature have a significant effect on the hydration rate and so the strength of samples, by controlling humidity and temperature, their effects can be mitigated, and thus other effects can be investigated accurately. All the samples were kept in the moulds during their curing time (3, 7 or 28 days).



Figure 4-2: Geomechanics Laboratory humidity chamber.

4.3 Uniaxial (Unconfined) Compressive Strength Test

Well-known and commonly used in mining and civil engineering, the uniaxial (or unconfined) compressive strength (UCS) test is used to measure the strength of backfill, intact rock, soil and many other mediums. Some of the reasons this test has been widely used and accepted as the initial factor in engineering are:

- The most important properties of backfill are directly related to compressive strength.
- Backfill has little tensile strength and is used primarily in compression.
- Structural design is based on compressive strength, modulus of elasticity and Poisson's ratio as well as shear strength.
- The test can quickly obtain the mechanical parameters of cohesive mediums.
- The test is relatively simple and inexpensive to perform.

According to ASTM D 2166-96 *Standard test method for unconfined compressive strength of cohesive soil* (1996), 221 triplicates (663 total numbers) of slurry sand backfill samples were tested to determine the UCS values of samples. To conduct the tests, all selected samples were removed from the humidity chamber just before performing the tests. Their moulds

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were removed, and their ends were flattened properly in order to reach a ratio of 2:1 height to diameter.

Figure 4-3 shows the Wykeham Farrance 100 KN stepless press machine used to perform the tests. This press had a 50 KN load cell and a 25 mm LVDT sensor. A normal loading rate of 1 mm/min was applied by the press control panel. A computer setup and digital acquisition data boards were used to store the data in a computer, and special software called (Intertech XY 6) was used to show the results graphically.



Figure 4-3: Unconfined compressive setup

4.4 Triaxial Confined Compressive Strength Test

This test determines the mechanical strength of backfill specimens namely: shear strengths at various lateral pressures, angle of internal friction, cohesion and Young's modulus. This test is based on the fact that, the compressive strength of backfill increases by the increase of the confining pressure. This phenomenon of backfill samples has been described by Coulomb's Law. This law is generally recognized as a failure criterion to describe the behavior of cohesive mediums under different degrees of confinement; according to this law:

$$\tau_f = c + \sigma_n \tan\theta \tag{4-1}$$

Where:

 τ_{f} = The shear stress on the failure surface at failure

c = The cohesion

 σ_n = The normal stress on the failure surface

 θ = The angle of shear resistance

To determine the confined compressive strength, cohesion and angle of shear resistance, the Mohr circles are mostly used (Razavi, 2007). As is shown in Figure 4-4, a set of Mohr circles are usually drawn for every identical specimen at various confining pressures and the line drawn tangential to these circles represents Coulomb's Law.



Figure 4-4: Mohr Circles of Stress

According to the ASTM standard D 4767-88 Standard test method for consolidated-undrained triaxial compression test on cohesive soils (1988), all tests were performed by a Wykeham Farrance 100 KN stepless press machine, using a 50 KN load cell, a 25 mm LVDT sensor and a triaxial cell. A normal loading rate of 1 mm/min was applied by the press control panel. This press is shown in Figure 4-5.

Same to the UCS tests, selected samples were removed from the humidity chamber just before performing the tests. Their moulds were removed, and their ends were flattened properly in order to reach a ratio of 2:1 height to diameter. These samples were positioned into a rubber membrane sealed. These sealed samples, then, were positioned in a

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 σ_n

compression triaxial cell which was filled with de-aired water at various confining pressures range between 200 to 600 KPa.

A computer controlled data acquisition system was used to store the data in a computer. Special software was used to analyze and calculate the results. This computer controlled setup is same as the setup was used for the unconfined compression tests.



Figure 4-5: Confined compression setup, the compression triaxial cell (a) and the de-aired water system (b).

4.5 Mercury Intrusion Porosimetery (MIP)

Mercury Intrusion Porosimetery (MIP) is a technique widely used to investigate the microstructure of porous materials such as backfill, concrete
and rocks. This technique can accurately determine pore size distribution and pore structure data, including bulk and apparent density, total pore volume, as well as median and average pore diameters ranging from 360 to 0.005 μ m. Therefore, MIP is an important technique in the investigation of pore size distribution.

Based on the theoretical foundation of E. Washburn (1921), the principal of mercury intrusion porosimetery is that a non-wetting liquid has to be forced in order to penetrate into the pores of materials, and this penetration is directly depend on the amount of the applied pressure. This relationship between the applied pressure and pore size is shown Equation 4-2 (the Washburn's Equation). Finally, Since Mercury is a non-wetting liquid for almost all substances, it has been used for this technique.

 $D = \frac{4\gamma \cos\varphi}{P}$

(4-2)

Where:

D = pore opening diameter.

P = applied pressure.

 γ = surface tension of a non-wetting liquid (approximately 480 dyne/cm for mercury).

 φ = contact angle between a non-wetting liquid and pore wall (approximately 140° for mercury).

In order to study and characterize the total porosity (MP_{tot}), free access porosity (MP_{free}) and trapped porosity (MP_{trap}) of the cemented backfill materials, a Micromeritics 9320-PoreSizer porosimeter was used. A digital data acquisition system was used to store the data in a computer. Special software was used to analyze and show the results graphically. This porosimeter and its computer setup are shown in Figure 4-6 and 4-7.

The samples are subjected to two different cycle of mercury intrusion. The difference between the two cycles depicts the level of trapped mercury intrusion. In this investigation, a total amount of eight samples were subjected to the mercury intrusion porosimetery test.

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Figure 4-6: Micromeritics 9320-PoreSizer porosimeter



Figure 4-7: Mercury Intrusion Porosimetery setup

4.6 X- ray Diffraction Analysis and Scanning Electron Microscopy

The combination of X- ray diffraction analysis (XRD) and scanning electron microscopy (SEM) has been widely and successfully used to characterize mineral and geochemical of microstructure of cement-based materials (Razavi, 2007; Hassani et al., 1998; Benzaazoua, 2002, 2006; Fall et al., 2005).

Samples were studied in a SEM vacuumed chamber using a Hitachi 3500-N scanning electron microscope at the Electron Microscopy Facility of McGill University. A typical scanning electron microscope is shown in Figure 4-8. Different techniques including backscattered electron imaging mode (BSE), energy dispersive spectrometry (EDS) and X- ray diffraction analysis were used to study cement phase textures and morphology as well as the effect of sodium silicate in the cement phase.



Figure 4-8: A typical scanning electron microscope and its setup

(www.geosci.unc.edu).

CHAPTER 5: MECHANICAL TEST RESULTS and DISCUSSION

5.1 Introduction

In this chapter, the effect of the mixing order, and mixing time on the short term mechanical behaviour and microstructures of slurry sand backfill and gelfill specimens were investigated and compared. In order to achieve these objectives, a total number of 351 samples from 39 mixtures with various recipes were made and cured for 7, 14 or 28 days for this purpose. The binders used for preparing the slurry fill samples were a combination of 90:10 blast furnace slag and Portland cement (S/C) with a 5% of total dry weight dosage. The binders for the gelfill samples were a combination of blast furnace slag, Portland cement (90:10 slag: cement) and sodium silicate (0.4% of total dry weight). The amount of binder added was 5% of the total dry weight for preparing the gelfill samples. The following notations are used in this chapter:

- S= blast furnace slag
- C= Portland cement
- SS= sodium silicate

5.2 The Optimization of Sodium Silicate

In the first step, it was necessary to optimize the amount of sodium silicate in the gelfill. Therefore, seven different recipes were made to evaluate the effect of sodium silicate on the UCS value of backfill samples during 28 days with a 5-minute mixing time period.

These seven recipes are:

- 1) S/C: consisting of a 90:10 slag: cement binder,
- S/C/SS 0.1wt%: consisting of a 90:10 slag: cement binder and the amount of sodium silicate added was 0.1% of the total dry weight.
- 3) S/C/SS 0.2wt%: consisting of a 90:10 slag: cement binder and the amount of sodium silicate added was 0.2% of the total dry weight.
- 4) S/C/SS 0.3wt%: consisting of a 90:10 slag: cement binder and the amount of sodium silicate added was 0.3% of the total dry weight.
- 5) S/C/SS 0.4wt%: consisting of a 90:10 slag: cement binder and the amount of sodium silicate added was 0.4% of the total dry weight.
- 6) S/C/SS 0.5wt%: consisting of a 90:10 slag: cement binder and the amount of sodium silicate added was 0.5% of the total dry weight.
- 7) S/C/SS 0.7wt%: consisting of a 90:10 slag: cement binder and the amount of sodium silicate added was 0.7% of the total dry weight.

The UCS tests were conducted on the 63 samples after a curing time of 7, 14 or 28 days and the results are shown in Figures 5-1 and 5-2. The similarity between the results of samples and the trends of UCS values can be a good indication of the homogeneity of samples, and accuracy of data. The results show that in a given curing time, the UCS values are increased by increasing the amount of sodium silicate up to 0.4% of the total dry weight (wt), but are decreased with any further increase of sodium silicate. Moreover, the UCS values significantly decrease when the amount of sodium silicate goes above 0.5% of the total dry weight. This is may be due to the increase in the total porosity of samples and the amount of moisture trapped in the samples. Finally, it can be concluded that the optimized amount of sodium silicate is 0.4% of the total dry weight.







Figure 5-2: Effect of sodium silicate on the 28-day UCS values of gelfill samples with a 5- min mixing time period.

5.3 The Effect of Mixing Time on the Strength of Gelfill and Slurry Backfill

In order to determine the effect of mixing time on slurry backfill and gelfill, 72 samples (24 triplicate samples) were prepared with various mixing times (between 5 to 20 minutes). The binders used for preparing the slurry fill samples were a combination of blast furnace slag and Portland cement (S/C), and the binders for the gelfill samples were combination of blast furnace slag (BFS), Portland cement and sodium silicate (S/C/SS). The content of binders and sodium silicate were 5% and 0.4% of total dry weight,

respectively. For all samples, the pulp density of mixtures was kept constant at 76%.

Figures 5-3 and 5-4 show the development of the UCS as a function of mixing time for four different mixing times for slurry backfill and gelfill over a 28-day period. As expected, the UCS values increase with an increase in curing time (due to the hydration of normal Portland cement and blast furnace slag). It is important to note that these increases in the mixtures with sodium silicate are more than in the mixtures without sodium silicate. Furthermore, as shown in Figure 5-4, there is no clear relation between mixing time and the UCS of slurry backfill samples however, the test results shown in Figure 5-3 indicate that for gelfill samples, the increase in mixing time reduces the UCS values.

The fact that the mechanical strength of slurry samples is unaffected by changes in mixing time is due to the slow hydration process of Portland cement and slag. This has been discussed by number of researchers including Neville, 1996; Benzaazoua et al, 2002; Fall et al 2005 and Razavi, 2007. The results of UCS values of gelfill specimens could be due to the fact that the setting time of BFS is dramatically decreased by the addition of alkali activators, especially sodium silicate. In this case by extending the mixing time, the initial binding between aggregates could be broken resulting in a decrease in strength values. The similarity between the results of triplicate samples indicates the homogeneity of samples, and reproducibility of the data.

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Figure 5-3: The evolution of UCS values over curing duration for gel and slurry backfill with various mixing orders with a 5 min mixing time period.



Figure 5-4: Effect of mixing time on compressive strengths of hydraulic sand fill and gelfill after a 28-day curing time period.

5.4 The Effect of Mixing Sequence on the Strength of Gel and Slurry Backfill

The preliminary results regarding the effect of mixing time on gel and slurry backfill indicate that the mixing sequence of materials may have an effect on the mechanical strength of gelfill. To investigate this parameter, 90 samples (30 triplicate samples) were made with two mixing times, 5 and 10 minutes. Binders used consisted of a combination of blast furnace slag, Portland cement and sodium silicate (S/C/SS) for gelfill samples. The content of binders and sodium silicate were 5% and 0.4% of the total dry weight respectively. The pulp density of mixtures was kept constant at 76% for all samples. The UCS test was conducted for all samples.

5.4.1 Five Minutes Mixing Time

The development of the UCS value of backfill mixtures over a 28-day period of time for four different mixing orders is shown in Figure 5-5.

These four mixing orders are:

- 1) S/C: consisting of a 90:10 slag: cement binder
- S/C/SS: consisting of a 90:10 slag: cement binder, and sodium silicate added immediately after the binder
- S/C/SS@2: consisting of a 90:10 slag: cement binder, and sodium silicate added to the mixture after 2 minutes
- S/C/SS@4: consisting of a 90:10 slag: cement binder, and sodium silicate added to the mixture after 4 minutes

Figure 5-5 shows the UCS test results, which indicate that for a given mixing time, a higher mechanical strength is expected when sodium silicate added with a delay. This may be verified by the effect of mixing time on the gelfill strength in which the mechanical strength of gelfill decreases by increasing the mixing time.

As Figure 5-6 shows, the S/C/SS@2 and S/C/SS@4 mixtures have UCS values that are slightly more than of the S/C/SS. Moreover, the mechanical strength of gelfill samples are almost 20% more than the slurry backfills' without sodium silicate.

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Figure 5-5: The evolution of UCS values over curing duration for gel and slurry backfill with various mixing orders with a 5 min mixing time period.





with a 5 min mixing time period after 28 days curing time.

5.4.2 Ten Minutes Mixing Time

Six different mixing orders were applied to 54 samples, to evaluate the effect of mixing sequence on the UCS value of backfill samples over 28 days with a 10-minute mixing time period. These six mixing orders are termed as:

- 1) S/C: consisting of a 90:10 slag:cement binder (5% of total dry weight).
- S/C/SS: consisting of a 90:10 slag:cement binder (5% of total dry weight), and sodium silicate added immediately after the binder.
- S/C/SS@2: consisting of a 90:10 slag:cement binder (5% of total dry weight), and sodium silicate added to the mixture after 2 minutes.
- S/C/SS@4: consisting of a 90:10 slag:cement binder (5% of total dry weight), and sodium silicate added to the mixture after 4 minutes.
- 5) S/C/SS@6: consisting of a 90:10 slag:cement binder (5% of total dry weight), and sodium silicate added to the mixture after 6 minutes.
- S/C/SS@8: consisting of a 90:10 slag:cement binder (5% of total dry weight), and sodium silicate added to the mixture after 8 minutes

The results are shown in Figures 5-7and 5-8. As expected, the UCS values of gelfill samples increase with curing time and these values also increase when sodium silicate is added to the mixtures after a delay. For instance, the average UCS values of S/C/SS@6 and S/C/SS@8 mixtures

are slightly more than the UCS values of S/C/SS@4, S/C/SS@2 and S/C/SS as well as S/C.



Figure 5-7: The evolution of UCS values over curing duration for gel and slurry backfill with various mixing orders with a 10- min mixing time period.





5.5 The Elasticity Modulus of Hydraulic Backfill and Gelfill

In order to determine the effect of sodium silicate on the elasticity modulus of backfill, a total of 18 samples with two different recipes were made and cured for 28 days. These two recipes are:

 S/C: consisting of a 90:10 slag:cement binder and a binder dosage of 5% of the total dry weight. 2) S/C/SS: consisting of a 90:10 slag:cement binder (5% of total dry weight), and the amount of sodium silicate added was 0.4%.

The UCS tests were conducted on the samples and the results shown in Table 5-1. As can be seen, the elasticity modulus of gelfill (S/C/SS) is almost 15% higher than the elasticity modulus of slurry backfill (S/C). This can be attributed to the increase of the formation of CSH gel and the decrease of the porosity.

Table 5-1: The modulus of elasticity values for slurry fill and gelfill after with 28

| | Binder proportion | Elasticity modulus | | |
|--------|-------------------|--------------------|--|--|
| recipe | (wt %) | (MPa) | | |
| SC | 5 | 0.34 | | |
| SCSS | 5 | 0.39 | | |

days curing

5.6 The Cohesion and Internal Fraction Angle of Hydraulic Backfill and Gelfill

A total of 18 samples with two different recipes were made to evaluate the effect of sodium silicate on the internal angle of friction (ϕ) and cohesion (C) of hydraulic backfill. The two recipes are:

- S/C: consisting of a 90:10 slag:cement binder and the binder dosage of 5% of the total dry weight.
- S/C/SS: consist of a 90:10 slag:cement binder (5% total dry weight), and the amount of sodium silicate added was 0.4%.

The samples were cured for 28 days in a humidity room and were subjected to consolidated drained triaxial compression tests. In these tests, the confining pressures were 0, 300 and 600 KPa. The axial deformation rate was kept constant at the rate of 1mm/min.

The results of triaxial tests are shown in Table5-2. The results indicate that by using sodium silicate, the cohesion and the internal angle of friction were decreased 12% and 20% respectively.

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Table 5-2: The result of triaxial tests for sand hydraulic backfill and gelfill (28 days

| | Binder proportion | Cohesion | Internal angle of |
|-------------|-------------------|----------|-------------------|
| Binder type | (wt %) | (MPa) | friction (φ) |
| | | | (degrees) |
| SC | 5 | 0.91 | 20 |
| | | | |
| SCSS | 5 | 1.06 | 25 |
| а. С | | | |

of curing)

CHAPTER 6: MINERALOGY AND MICROSTRUCTRE RESULTS

6.1 Introduction

The effect of sodium silicate on the mineralogy and microstructures of silica sand hydraulic backfill are investigated by using scanning electron microscopy (SEM) and mercury intrusion porosimetry (MIP) techniques. A variable pressure Hitachi S-3000N scanning electron microscope equipped with an energy dispersive spectrometer (SEM/EDS) was used for Scanning Electron Microscopy (SEM), and a Micromeritics 9320-PoreSizer porosimeter was used for Mercury Intrusion Porosimetery (MIP).

6.2 Scanning Electron Microscopy

To evaluate the effect of sodium silicate on the mineralogy of hydraulic and gelfill samples, 12 samples with four different recipes were made and cured for 7, 14 or 28 days. Table 6-1 shows the notation and the composition of these recipes.

| Sample | Water (wt %) | Material | BFS (wt %) | PCX (wt %) | Sodium Silicate (wt %) |
|--------|-----------------|----------|---------------|---------------|---------------------------|
| S331 | 20 | Sand | 27 | 3 | 3 |
| S311 | 20 | Sand | 27 | 3 | 1 |
| S301 | 20 | Sand | 27 | 3 | 0 |
| S001 | 20 | Sand | 0 | • 0 | 0 |

Table6-1: Notation and composition of samples

Sample S001 was a reference sample and no binder was added to the silica sand. Figures 6-1, 6-2 and 6-3 are the SEM images of S001. The images show isolated sand grains without any aggregation which is due to lack of binders and sodium silicate. The chemical composition of the samples in three spots- S1, S2 and S3- are shown in Figures 6-4, 6-5, and 6-6. Since the samples were made of silica sand, the main components are silicium (Si) and oxygen (O).

Sample S301 was made of silica sand and binders (10% wt). Figures 6-7, 6-8, 6-9 and 6-10 are the SEM images of S301. As can be seen, a few grains of sand are coated by the process of hydrating the binders. They could be alite or belite. As can be demonstrated, the amount of these coating materials is not very much. The chemical composition of these coating materials are shown in Figures 6-11and 6-12. It can be seen that sodium (Na) was not included in the chemical composition of this sample , likely due to the fact that sodium silicate was not used to make this sample.



Figure6-1: SEM image of Sample S001



Figure6-2: SEM image of S3, Sample S001



Figure 6-3: SEM image of P3, Sample S001



Figure6-4: Spectrum of S1



Figure6-5: Spectrum of S2



Figure6-6: Spectrum of S3



Figure6-7: SEM image of Sample S301



Figure 6-9: Magnified SEM image of P3 S301



Figure6-8: Magnified SEM image of P2 S301



Figure6-10: Magnified SEM image of P4 S301 c.f., S1



Figure6-11: Spectrum of S1 of P4 S301; in cement above C-tape



Figure 6-12: Spectrum of S1 of P4 S301; Cement on quartz grain

As can be seen in Table 6-1, 1% and 3% of total dry weight sodium silicate were used for manufacturing Samples S311 and S331, respectively, and the amount of binders were the same as in Sample 301(10% wt). Figures 6-13, 6-14 and 6-15 demonstrate the SEM images of S311. Compare with S301, the amount of coating materials is more in S311. This can be attributed to the use of sodium silicate in preparing this sample. The chemical composition of these coating materials are determined in Figures 6-16, 6-17,6-18 and 6-19; the presence of sodium(Na) in the chemical composition confirms the presence of sodium silicate in the production of these coating materials.



Figure6-13: SEM image of Sample S311



Figure 6-14: Magnified SEM image of P2 S311



Figure6-15: Magnified SEM image of P3 S311



Figure6-16: Spectrum of S1, S301



Figure6-17: Spectrum of S2 S311



Figure6-18: Spectrum of S3 S311



Figure6-19: Spectrum of S4, S311

The SEM images of Sample S331 are shown in Figures 6-20, 6-21, 6-22, 6-23 and 6-24. As can be seen, the coating materials in sample S331 is much more abundant than S301 and S311. Therefore, it can be concluded that by increasing sodium silicate, the amount of coating materials also increases. The chemical compositions of the binding materials are given in Figures 6-25, 6-26, 6-27 and 6-28. These compositions, which are the same as Sample S311, show the presence of sodium (Na) in the coating materials, due to use of sodium silicate.



Figure6-20: SEM image of Sample S331



Figure6-21: Magnified SEM image of P2, S331



Figure6-22: Magnified SEM image of P3, S331



Figure6-23: SEM image of Sample S331



Figure6-24: Magnified SEM image of P5, S331 c.f. S2, S3, S4



Figure6-25: Spectrum of S1, S331



Figure6-26: Spectrum of S2, S331



Figure6-27: Spectrum of S3, S331



Figure6-28: Spectrum of S4, S331

6.3 Mercury Intrusion Porosimetery (MIP)

In order to investigate the microstructure of backfill samples and the effect of sodium silicate on stabilized backfill samples, the results of the MIP tests of five samples were studied. Initially, three samples with various binders were studied; these three samples were as follows:

- 1. C: made with Portland cement.
- 2. SC: made with the combination of blast furnace slag and Portland cement.
- 3. SSS: made with the combination of sodium silicate and blast furnace slag.

The results of MIP tests are shown in Figures 6-29, 6-30 and 6-31. As can be seen, both samples C and SC have one pore size family that dominates the pore size distribution, and the size of pores in this family is between 10,000 to 100,000 Å. Moreover, the total porosity of these samples are 27% for Sample SC and about 29% for Sample C.

The sample with sodium silicate and slag (SSS) has an overall porosity which is similar to SC and C samples (30%),but the pore size distribution of SSS are dominated by pores whose size are between 100 to 1000 Å.

It may be concluded that the better mechanical behaviour is obtained from samples made with sodium silicate. This may be attributed to different pore size distributions. In fact, in a given overall porosity of a sample when the pore sizes are decreasing, the distribution of an applied stress is more likely to be homogeneous and uniform. To validate this assumption, the results of MIP tests for two samples were studied. It has been shown that the mechanical strength of backfill specimens improves by increasing binder dosages. This phenomenon has been discussed by many researchers including Hassani and Archibald, 1998; Benzaazoua, 2002, 2006; Razavi, 2007; Fall et al., 2004 and others. The MIP results of two samples with two different binder usages (7% and 9% wt) are shown in Figures 6-32 and 6-33. As can be seen, the overall porosity of these samples are almost the same at about 30% however, the dominant pore size in the sample with 9% binder dosage is significantly less than the sample with 5%. Therefore, it can be concluded that by decreasing pore sizes, the strength of samples increase.



Figure 6-29: MIP result of a backfill sample made with cement (9 % wt)



Figure 6-30: MIP result of a backfill sample made with cement and slag (9 % wt)



Figure 6-31: MIP result of a backfill sample made with slag and sodium silicate (9 % wt)



Figure 6-32: Pore size distribution and overall porosity of a backfill sample with 7% wt binder (SSS).



Figure 6-33: Pore size distribution and overall porosity of a backfill sample with 7% wt binder (SSS).
CHAPTER 7: CONCLUSION AND FURTHER WORK

7.1 Conclusion

In this research, a number of the properties of slurry backfill and gelfill have been investigated and compared namely curing time, mixing time and mixing sequence. In order to determine the influence of each abovementioned variable, a series of laboratory experiments were conducted with various recipes. The experiments focused mainly on the mechanical behaviour of gelfill and slurry backfill samples as well as the microstructural and mineralogical properties of specimens. Based on these results, the following conclusions can be made:

- Gelfill and hydraulic backfill UCS values increase over a 28 day curing time.
- Gelfill materials behave favourably compared with hydraulic backfill.
- Mixing time plays a significant role in strength of gelfill however it does not in hydraulic backfill. By increasing mixing time, the strength of gelfill decreases.

- Mixing order has a crucial effect on the strength of gelfill, but not on the strength of hydraulic backfill..
- The required sodium silicate in gelfill differs at each specific mine materials.

Adding sodium silicate sharply changes pore size distribution of silica sand hydraulic backfill.

 The presence of sodium silicate affects the mineralogy of specimens in a way that the amount of coating materials in samples is increased by adding sodium silicate.

7.2 Further Work

In this thesis, a number of variables affecting the strength and performance of gelfill have been studied; nevertheless, a wide range of variables remain to be studied in further research, including:

> Curing temperature has a crucial role in the strength development of cemented backfill; therefore, the effect of curing temperature should be investigated.

- Since water temperature changes the viscosity of water dramatically, consequently, the effect of water temperature has to be investigated.
- The effect of size grading of inert materials on the strength of gelfill has to be determined.

In order to fully explain the mechanical behaviours of gelfill, an in-depth chemical investigation including scanning electron microscopy (SEM) and X-Ray diffraction is needed.

- The hydration development and chemical and mineralogical changes over time of samples with different composition have to be investigated by transmission electron microscopy (TEM) and energy dispersive X-ray analysis (EDX).
- A detailed study should be conducted to determine the possible effects of sodium silicate used for backfill on the flotation of the mined ore.
- Comprehensive studies on applications of sodium silicate in waste treatment and cement and concrete materials have shown that sodium silicate has the capability of fixing and encapsulating the heavy metals; therefore, the effect of sodium silicate on high sulphide content backfill is required.

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 In order to validate the laboratory results on gelfill specimens, in-situ specimens have to be obtained from stopes and compared with the laboratory work discussed in this thesis.

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APPENDIX 2- A SEM image of a particle of silica sand and effect of the crystals bound



Print of a sand grain formerly pasted by sodium metasilicate coating, as frequently seen in this friable sample.

APPENDIX 3- A SEM image of a particle of silica sand and effect of the crystals bound



Several sand grains bond together by sodium metasilicates



Two sand grains cemented by "amorphous" sodium metasilicate





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APPENDIX 5- EDX spectrum of sodium metasilicate in sample A1033

