Electromagnetic Characterization of Cemented Paste Backfill in the Field and Laboratory

by

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ABSTRACT

Cemented Paste Backfill (CPB) is a relatively new backfilling technology for which a better understanding of binder hydration is required. This research uses electromagnetic (EM) wave-based techniques to non-destructively study a CPB consisting of tailings, sand, process water and binder (90% blast-furnace slag; 10% Portland cement). EM experiments were performed using a broadband network analyzer (20 MHz to 1.3 GHz) in the lab and capacitance probes (70 MHz) in the lab and field. Results showed that the EM properties are sensitive to curing time, operating frequency and specimen composition including binder content. The volumetric water content interpreted from dielectric permittivity varied little with curing. Temporal variations in electrical conductivity reflected the different stages of hydration. Laboratory results aided interpretation of field data and showed that a reduction in binder content from 4.5% to 2.2% delays setting of CPB from 0.5 days to over 2 days, which has important implications for mine design.
To my parents, Girilal and Sujatha
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Cemented Paste Backfill (CPB) technology is a backfilling method to fill openings created by underground mining. CPB has been successfully implemented at several Canadian and Australian mines since the late 1990s and is gaining popularity as the backfilling method of choice at mine sites around the world. The technology is more cost effective and versatile than other backfill alternatives such as hydraulic sand and rock fill (le Roux et al., 2005). It can rapidly be poured into mined stopes significantly reducing the mining cycle times. The fines retain water in the backfill reducing the need for dewatering systems. The cement in the paste backfill makes the material self-supporting providing stability as adjacent stopes are mined out and thus reducing dilution. It also supports heavy moving equipment when being used as a trafficked floor. Hardened CPB in filled stopes confine the rock mass surrounding the stope maintaining local and regional stability within mining areas (Rankine and Sivakugan, 2007; le Roux et al., 2005). These benefits improve ore recovery which translates to significant additional revenues for operating mines. In addition, reduction in the volume of surface management of mine tailings and water minimises the environmental footprint and associated costs of remediation and rehabilitation.

CPB is composed of inert materials such as tailings from mineral processing and sand mixed with binding agents such as Portland cement, lime, pulverized fly ash and blast furnace slag (BFS). CPB is prepared as a high-density mixture containing 70% to 80% of solids by total mass and 3% to 8% binder by mass of solids. To inhibit particle settlement and segregation during pipeline transport and placement, at least 15% of the fines are recommended to be smaller than 20 microns or 0.02 mm (Landriault, 1995). The strength of the CPB is attributed to the cohesive strength developed by binding agents and the fine grained nature of CPB.
1.1. Problem Statement

Though CPB is gaining popularity at several mine sites around the world, the geomechanical design of the CPB systems have been adopted from previous backfilling methods. These include the design of fill-fences meant to contain the CPB in the stope and pouring strategies. Most mines pour the paste in two stages to reduce the pressure build up on the fill-fence by initially curing a higher binder containing plug to provide a barrier between the fill-fence and main volume of the stope pour. CPB systems are designed based on strength properties of paste prepared in laboratory environments. The performance of these systems are evaluated in laboratories and through the monitoring of surrounding rock mass using instruments such as extensometers, inclinometers, accelerometers and other transducers. le Roux et al. (2005) investigated the in-situ properties and concluded they were more variable compared to the laboratory prepared samples and that the designs based on laboratory specimens were conservative. Therefore, mines can realize significant cost savings by improving upon the existing CPB systems. For example, a continuous pour of paste with a lower binder into the stope will reduce the mining cycle and reduce binder consumption, the cost of which is a significant portion of the CPB operating costs. However, in order to optimize CPB design, it is important to understand the field performance of CPB.

Most of the field data for CPB performance are either specific to certain test mines or are unpublished (le Roux et al., 2005). To address the lack of in-situ data, a series of instrumentation programs were conducted at three different mining operations. Field work began in 2007 within a 150 m high Alimak stope at Barrick’s Williams Mine in Hemlo, Ontario (Grabinsky et. al., 2007, 2008). In the summer of 2008, instruments were placed in two 30 m high long-hole stopes at Xstrata Copper’s Kidd Mine in Timmins, Ontario and in the fall of 2009, at Inmet’s Cayeli Mine in Turkey (Thompson et. al., 2009a). The aim of the university’s cemented paste backfill research group is to use the field data to understand the different phenomena taking place in CPB-filled stopes by validating with laboratory studies and finally develop a model to rationally engineer CPB systems (Grabinsky et al., 2009).
The challenge in developing the model is to specify parameters for the constitutive behaviour of the CPB which continually evolves with time, especially at early age. To interpret the field data it is important to understand the hydration processes and its effect on the fill’s constitutive behaviour (Grabinsky et al., 2009). Klein and Simon (2006) showed that electromagnetic (EM) properties of laboratory prepared CPB specimens were sensitive to the physicochemical changes that occur during hydration. For this purpose, commercially available capacitance probes that measure the electrical properties of the materials were included in the instrumentation program. The aim of this research study was to characterize the changes in electromagnetic properties of CPB and its individual components to better interpret the field data.

1.2. Research objectives
The non-destructive monitoring of the hydration reactions will provide insight into the microscale interactions in the pastes which affect the macroscale behaviour of CPB. Understanding interactions between paste constituents is critical to optimizing the design of CPB (Klein and Simon, 2006). In this study, field and laboratory EM experiments were performed on CPB and its component materials from the Kidd Mine located in Timmins, Ontario. The objectives of the research were to:

- Characterize the electromagnetic behaviour of CPB mixtures and validate the field data with laboratory experimental data
- Identify the stages of binder hydration by correlating the dielectric properties of CPB
- Study the influence of binder content on hydration of CPB
- Study the influence of sand aggregates on hydration of CPB
- Study the influence of temperature on the EM properties and hydration of CPB

1.3. Thesis organization
Chapter 2 begins with a background on the physicochemical processes that occur in the hydration of hydraulic materials such as Portland cement and cements blended with blast furnace slag. This is followed by a review of the electromagnetic parameters and the
polarization phenomena that occur in the presence of an electric field. The chapter concludes with a review of previous EM studies in soils and cement containing pastes.

Chapter 3 describes the properties of the component materials of CPB used in the mine and in this study follow by a description of the broadband network analyzer and single-frequency capacitance probe measurement techniques used to determine the EM properties of the materials.

Chapters 4 reviews the instrumentation program at Kidd Mine and summarizes the results obtained from the commercially available capacitance probes.

Chapter 5 begins with the description of the laboratory experimental procedure and summarizes the spectral and temporal response of EM results for the different specimens using the network analyzer and capacitance probes.

Chapter 6 examines and discusses the results obtained from the network analyzer and capacitance probes. The chapter begins with the validation of the field data with lab data. The polarization mechanisms that occur are identified through spectral analyses followed by temporal analyses of the EM data that correlates the EM changes to the different stages of hydration in specimens containing 100% binder and 4.5% binder CPB. Influences of binder content, material composition and temperature on the hydration of CPB are also discussed.

Chapter 7 finally summarizes the main results of the field and laboratory experiments and its significance.
CHAPTER 2
LITERATURE REVIEW

This research characterizes cemented paste backfill (CPB) using electromagnetic waves (EM). Portland cement (PC) and Blast Furnace Slag (BFS) are the binder elements added to tailings and mine process water to form CPB. Of the materials mentioned above PC and BFS are hydraulic in nature which causes the change in micro-structural, morphological, thermal, chemical and electrical properties in CPB (Simon, 2004). Therefore, to understand the hydration reactions taking place in CPB, the hydration processes that occur in cements and slag are described. The electromagnetic properties and polarization mechanisms that occur in an electric field are also presented. The chapter concludes by providing a review of the previous EM studies performed in geotechnical applications, cement pastes and finally, CPB.

2.1. Portland cement

2.1.1. Properties of Portland cement

Portland cement is manufactured by mixing pre-blended, finely ground raw materials containing clay, shale and lime into an inclined rotary kiln heated at over 1450°C (Neville, 1995). The materials undergo physical and chemical reactions and fall out the kiln as clinkers. The cooled clinkers are then milled and ground to fine powder. Portland cement primarily consists of the following major chemical elements in the form of oxides: lime (CaO), silica (SiO₂), alumina (Al₂O₃), and iron oxide (Fe₂O₃). Minor chemical elements include: magnesia, alkali oxides (Na₂O and K₂O), titania (TiO₂), phosphoros pentoxide (P₂O₅) and gypsum (Soroka, 1980). The four major constituents of Portland cement clinker are: tricalcium silicate (C₃S), dicalcium silicate (C₂S), tricalcium aluminate (C₃A) and tetracalcium aluminoferrite (C₄AF). As per the cement chemist
notations: C, A, F, S and H represent one mole of CaO, Al₂O₃, Fe₂O₃, SiO₂ and H₂O, respectively.

2.1.2. **Hydration of Portland cement**

The chemical reaction of cement with water is referred to as the hydration of cement (Mehta and Monteiro, 1993). Two different mechanisms of hydration of cement are said to occur: through-solution and topochemical (solid state) reactions. In the former, the anhydrous reactants completely dissolve in water to produce ions in solution. The ions combine with each other and the resulting products precipitate out of the supersaturated solution. In the latter, the reactions take place directly at the surface of the anhydrous cement compounds. Electron microscope studies demonstrate the through-solution mechanism is predominant in the early stages of hydration. At later stages, due to the reduced ionic mobility, the hydration of residual cement may occur by solid-state reactions (Soroka, 1980; Mehta and Monteiro, 1993).

Cement consists of several compounds and its hydration involves a number of chemical reactions which take place simultaneously. The products produced by the hydration of individual compounds (calcium silicates and aluminates) are similar to those resulting from their hydration as part of cement. The typical hydration products of Portland cement are: calcium hydroxide (CH), calcium silicate hydrate (C-S-H) and calcium sulphoaluminate (ettringite).

2.1.3. **Setting and hardening of cement**

The hydration of cement undergoes four distinct stages of hydration: initial, dormant, acceleration or setting, deceleration or hardening. The initial stage lasts for less than half an hour and is usually coupled with the dormant stage. The stages of hydration are illustrated in Figure 2-1 and detailed in the text.

Initial period (0 – 30 minutes): On contact with water, the first set of reactions to occur are the dissolution of the C₃S, C₃A and gypsum during which alkali and hydroxyl ions
are released into the pore solution. Protonation of the cement grains take place, where \( \text{H}^+ \) ions from water and \( \text{SiO}_4^{4-} \) ions from the cement surface react to form soluble \( \text{H}_2\text{SiO}_4^{2-} \) and \( \text{H}_3\text{SiO}_4^{4-} \) ions. Protonation facilitates the formation of the calcium silicate hydrate (C-S-H) gel (Damidot and Nonat, 1992).

Dormant or induction period (30 minutes to 2 hours): Calcium silicates undergo hydrolysis producing C-S-H and a supersaturated solution of calcium hydroxide ions. Gypsum reacts with the C_3A to form needle-like crystals rich in sulphate called ettringite. The C-S-H gel and ettringite surround the C_3S and C_3A grains, respectively, by forming a coating around them. This gel acts as a membrane and retards further hydration of the C_3S and C_3A grains resulting in a dormant period. In this period of relative inactivity the paste is still workable and plastic. The cement grains are separate and the hydrated products are mainly \( \text{Ca(OH)}_2 \) and ettringite. However, the intra-membrane layer allows water to penetrate and further dissolve the cement grains but prevents the migration of the ions into the outer solution. The concentration of the calcium and silica ions within the membrane increases compared to the outer solution giving rise to an osmotic pressure difference. This pressure build-up eventually causes the membrane to rupture resulting in the continuation of the hydration process. This denotes the end of the dormant period and the on-set of the acceleration stage.

Acceleration or setting period (3 hours after mixing): After the rupture of C-S-H gel and ettringite membrane, the silica rich solution mixes with the external solution and the C-S-H gel begins to form particles in the shape of long fibres. Tabular crystals of CH precipitate out of the supersaturated pore solution and ettringite continues to form provided sufficient sulphate ions are present. At this stage, the hydrated products (C-S-H, CH and ettringite) begin to grow on the surface of the grains and bridge the gaps between the particles. This reduces the porosity and increases the stiffness of the paste resulting in the “setting” of the paste.

Deceleration or hardening period (17 to 24 hours after mixing): The C-S-H and CH continues to form at a slower rate forming short fibres and massive deposits, respectively.
By this time, the sulphate ions are also depleted and the less stable ettringite convert into the more stable monosulphate. The hydrated products continue to fill the pores reducing the paste porosity and the rate of hydration gradually decreases.

![Figure 2-1 Schematic description of hydration of cement paste (from Soroka 1980)](image)

**Figure 2-1 Schematic description of hydration of cement paste (from Soroka 1980)**

Cement – shaded areas; CH – hexagonal crystals; ettringite – heavy short lines; C-S-H – thin short lines

### 2.1.4. Water in hydrated cements

The different states of water found in pastes are: free water, adsorbed water and chemically bound water. Free water is held in the large capillaries of the hydrated cement. The water molecules are free from the influence of the attractive forces exerted by the solid surface (Mehta and Monteiro, 1993). Adsorbed water is physically adsorbed to the surface of the solids in the cement due to the influence of the attractive (or surface) forces. Chemically combined water is an integral part of the structure of various hydration products and is not lost on drying. However, it is very difficult to determine the
state of water in pastes because electron microscope examination of hydrated cement pastes require the voids to be empty and dried in high vacuum (Mehta and Monteiro, 1993). As a result, water in pastes can be put into two broad categories: evaporable and non-evaporable. Evaporable water consists of free and part of the adsorbed part that can be removed by drying at 105°C. Non-evaporable water contains the chemically combined water and the part of the adsorbed water that cannot be removed by drying at 105°C (Neville, 1995).

2.2. Addition of Supplementary Cementing Materials (SCM)

Portland cement is sometimes blended with additional materials such as fly ash (FA), blast furnace slag (BFS), silica fume and natural pozzolans to enhance the properties and durability of concrete. These materials are also known as mineral admixtures and classified as Supplementary Cementing Materials. Engineering benefits of SCM include: improved resistance to thermal cracking because of lower heat of hydration, enhancement of ultimate strength and impermeability due to the pore refinement and better durability to chemical attacks as a result of reduced alkalinity (Mehta and Monteiro, 1993). The SCM are typically waste products of industrial processes and the blending with Portland cement facilitates their disposal thereby reducing costs and saving energy. The binder material used in this research is a mixture of Portland cement and blast furnace slag and further discussions on SCM will be limited to the blast furnace slag.

BFS is obtained as a waste product in the production of iron from iron ore. The 1600°C molten slag that floats above the molten iron in the furnaces contains about 30-40% SiO₂ and 40% CaO, which is similar to the composition of Portland cement. The rapid quenching of the molten slag produces glass silicates rich in alumina, calcium and magnesia and is then dried and ground. Backscattered electron images of BFS show the slag grains to be angular (Hill and Sharp, 2002).

BFS, unlike fly ash which is a pozzolan, has a latent hydraulic reactivity. The hydration of BFS in pure water is very slow due to the formation of acidic aluminosilicate coating
around the slag grains (Juenger et al., 2006) and also due to the high glass content of BFS which will only react at high alkalinity. However, it can be catalyzed by basic reactors such as CH from Portland cement, calcium sulphates or other alkali compounds. The minimum pH required to activate the reactions is 12 (Zeghici et al., 2005). The release of alkali (Ca$^{2+}$) and hydroxyl (OH$^-$) ions during the hydration of PC increases the alkalinity of the pore solutions triggering the hydration reactions for BFS. The silica glasses become soluble in the solution and react to form secondary growths of C-S-H which fills the capillary pore space left by prior hydration of PC thus increasing strength and porosity. The hydration products of blended cement are comprised of hydration products from both PC and slag hydration. The primary hydration product is C-S-H, however, C-S-H formed by slag has lower Ca/Si ratio compared to PC. Ettringite is also a major product in the hydration of both materials (Juenger et al., 2006; Chen and Brouwers, 2007).

2.3. Electromagnetic Wave Properties

This section begins with the description of the constitutive parameters of electromagnetism that will be used to relate material properties. A broadband network analyzer and single-frequency capacitance probe measurement techniques are used in this study to determine the EM properties of different materials. Therefore, the polarization mechanisms that occur in materials at different frequencies in an electric field are reviewed at the end.

2.3.1. Constitutive Parameters

The three constitutive parameters required to describe the electromagnetic properties of a medium are: dielectric permittivity $\varepsilon$, electrical conductivity $\sigma$ and magnetic permeability $\mu$. The response of a material to an applied electric field depends on the material’s compositional and structural properties, and the frequency of the electrical field.

In dielectrics or insulators, ions or charges are not free to move and have permanent electric dipoles built of positive and negative charges separated by a distance. In an
applied electric field, the dipoles get displaced from a position of equilibrium thereby storing energy or become polarized. A measure of polarizability of a material in an electric field is the dielectric permittivity. Similarly, magnetic materials also possess permanent magnetic dipoles which orient in response to an applied magnetic field and the measure of magnetizability is the magnetic permeability. In an applied electric field, the charges will readily flow in the direction of the electric field. The measure of charge mobility in an electric field is the electrical conductivity, and is described by Ohm’s law as:

\[ J = \sigma E \]  

(2.1)

where \( J \) is the current density and \( E \) is the electric field.

Polarization of bound charges and magnetization of the medium are dynamic effects produced by the forces and torques imposed by electric and magnetic fields. Since these phenomena are opposed by inertial and viscous forces they do not occur instantaneously and are frequency dependent. Also, the response of the materials in an electric field is partially out of phase with excitation and as a result, both the phase and amplitude of the response are captured by defining complex material parameters. Therefore, the constitutive parameters \( \varepsilon \) and \( \mu \) are complex quantities which vary with frequency and are denoted by \( \varepsilon^* \) and \( \mu^* \), respectively. (Santamarina et al., 2001; Griffiths, 1989)

The complex dielectric permittivity \( \varepsilon^* \) consists of a real part \( \varepsilon' \) and an imaginary part \( \varepsilon'' \):

\[ \varepsilon^* = \varepsilon' - j\varepsilon'' \]  

(2.2)

where \( j^2 = -1 \) denotes an imaginary number. The permittivity of the material is normalized with respect to the permittivity of vacuum (\( \varepsilon_0 = 8.85 \times 10^{-12} \) F/m) to give relative permittivity:
\( \kappa' = \frac{\varepsilon^*}{\varepsilon_0} = \kappa' - j\kappa'' \)  
\hspace{1cm} (2.3)

where \( \kappa' \) is the real relative permittivity which represents a material’s ability to polarize in an applied electric field and \( \kappa'' \) is the imaginary relative permittivity which measures the polarization losses.

The electrical conductivity \( \sigma \) is a real quantity without an out-of-phase component. It is also in phase with the imaginary relative permittivity and hence, the losses due to polarization and conduction are measured together. The effective imaginary permittivity and effective electrical conductivity are:

\[ \kappa'_{\text{eff}} = \kappa'' + \frac{\sigma_{\text{dc}}}{\varepsilon_0 \omega} \]  \hspace{1cm} (2.4)

\[ \sigma_{\text{eff}} = \sigma_{\text{dc}} + \kappa'' \varepsilon_0 \omega \]  \hspace{1cm} (2.5)

where \( \omega \) is the angular frequency of the applied field. From the above equations, it is clear that at high frequencies polarization losses dominate and at low frequencies, the effect of conduction losses are higher.

The complex magnetic permeability \( \mu^* \) is similar to permittivity and consists of real and imaginary parts:

\[ \mu^* = \mu' - j\mu'' \]  \hspace{1cm} (2.6)

\[ \mu'_{\text{r}} = \frac{\mu'}{\mu_0} = \mu'_{\text{r}} - j\mu''_{\text{r}} \]  \hspace{1cm} (2.7)

The relative magnetic permeability \( \mu'_{\text{r}} \) is the ratio of the complex magnetic permeability to the permeability of vacuum (\( \mu_0 = 1.26 \times 10^{-6} \text{ H/m} \)). According to Santamarina et al. (2001), for non-ferromagnetic materials \( \mu' \approx \mu_0 \) and thus, \( \mu'_{\text{r}} = 1 \). Most geomaterials
contain insufficient magnetic material to affect complex permittivity. Ideally, this assumption has to be verified as neglecting the presence of ferromagnetic materials can result in a higher permittivity than the true mixture permittivity (Klein and Santamarina, 2000).

2.3.2. **Polarization Mechanisms**

Polarization is the spatial separation of charges in an applied electric field. The response of the material depends on the ability of the charges to polarize at a certain frequency, against opposing viscous and inertial forces. There are two kinds of polarization phenomena: resonance and relaxation. A material that returns to its original state after polarization displays a resonance spectrum (Figure 2-2a) whereas a material that exhibits gradual permanent reduction in polarization displays a relaxation spectrum (Figure 2-2b).

![Figure 2-2 Spectral Response of a material: a) Resonance; b) Relaxation](from Santamarina et al. 2001)

The polarization mechanisms in a single phase, homogeneous materials are: electronic, ionic and orientational polarizations that usually occur at high frequencies. Electronic polarization (Figure 2-3a) is the displacement of electrons with respect to the positive core within the atom and typically occurs at UV frequencies ($10^{16}$ Hz). Ionic polarization (Figure 2-3b) is the relative displacement of atoms in a molecule and occurs at infrared frequencies ($10^{13}$ Hz). Oriental polarization (Figure 2-3c) is the rotation of the dipole molecules, such as water, towards an applied electric field and usually occurs at microwave frequencies ($10^{11}$ Hz). Electronic and ionic polarization display resonance...
phenomenon and are not sensitive to temperature. Oriental polarization is evident as relaxation spectra and is affected by temperature.

![Polarization mechanism in single phase, homogeneous materials](image)

**Figure 2-3** Polarization mechanism in single phase, homogeneous materials:  
a) Electronic; b) Ionic; c) Orientation polarization (from Santamarina et al. 2001)

Single component homogenous materials experience polarization mechanisms at frequencies greater than radio frequencies, however, the presence of a second component causes additional mechanisms to occur at lower frequencies. The additional polarization mechanisms include interfacial-spatial, bound water, double layer and Stern layer polarization (Santamarina et al. 2001).

The main polarization that occurs in heterogeneous mixtures is spatial polarization which is also known as interfacial or Maxwell-Wagner polarization. It is caused by differences in polarizability and conductivity producing charge accumulation at surface interfaces. It typically exhibits a relaxation response at low radio frequencies. Figure 2-4 illustrates the different spatial polarization mechanisms. Stern layer polarization (Figure 2-5a) arises due to the restrained movement of bound ions adjacent to the particle surface. The polarization occurs at high frequencies and is similar to ionic resonance but is affected by temperature. Bound water polarization (Figure 2-5b) caused by the reduced polarizability of water molecules adhered on to surface of a particle. Water can become bound to particles due to a combination of the following forces: adhesive (binding between solid
phase and water), cohesive (binding between water molecules) and osmotic forces (binding due to gradients in chemical potential in electric double layer). Double layer polarization is due to the relative displacement of double layer counter-ion cloud with respect to the charged particle in response to an electric field. The double layer polarizations in different mediums are illustrated in Figure 2-5 c, d, e and f. Additional polarization mechanisms are described in Santamarina et al. (2001). Figure 2-6 summarizes the different polarization that take place in heterogeneous mixtures containing water at different frequencies.

Figure 2-4 Polarization mechanisms in mixtures; Note: ○ cations ● anions
(from Santamarina et al. 2001)
Figure 2-5 Surface-related polarizations (from Santamarina et al. 2001)

Figure 2-6 Polarization mechanisms in heterogenous mixture containing water (modified from de Loor, 1968; taken from Simon, 2004)
2.3.3. Relating EM properties to material properties

Since microscale interparticle electrical forces can be directly related to the macroscale behaviour of particulate mineral materials, the physical interpretation of electromagnetic waves can give insight into the properties about the material (Klein and Wang, 2005).

The dielectric permittivity of polar molecules can become polarized and will orient to an applied electric field. The most common polar liquid is water and has a high relative permittivity of approximately 80 when compared to air ($\kappa' = 1$), mineral soils ($\kappa' = 3$ to 5) or other solids ($\kappa' < 10$). The amount of water in a porous material strongly influences the permittivity of the mixture and thus, can be a good indicator of moisture content in solid-liquid mixtures (Robinson et al., 1999; Simon, 2004). The $\kappa'$ of high specific soils is less than low specific surface soils as water decreases with increasing specific surface area (Klein and Wang, 2005). The $\kappa'$ of water also decreases with increasing ionic concentration as a result of the hindered free water polarization of water molecules which hydrate the ions in the fluid (Hasted, 1974). These factors encourage the use of complex permittivity measurement to monitor physicochemical processes including the curing process of cement hydration (Simon, 2004).

Electrical conductivity is a measure of the availability and mobility of ions in response to an electric field. Since most conduction takes place in the pore space of soils, pore fluid conductivity is an important parameter. Conductivity increases with increasing ionic concentrations but at very high ionic concentrations, the ion-ion interactions reduces the ionic mobility. Ionic mobility is sensitive to temperature, increasing with increasing temperature (Klein and Wang, 2005). Archie (1942) developed a relationship between conductivity, porosity and connectivity of the pore spaces in low specific surface soils and rock. As tortuosity of the pore spaces increases, the conductivity decreases as the ions have to follow a more complex path in response to the electric field. In solid-fluid mixtures, conductivity measurements in different directions provide information on the fabric anisotropy of the mixture which in turn can give useful engineering properties like hydraulic conductivity (Klein and Wang, 2005).
Mitchell and Liu (2006) provided a brief review on relating the electrical properties of soils to determine other geotechnical properties such as: void ratio, soil specific area, compressibility and swelling potential. Void ratio can be related to EM properties by a formation factor. The formation factor is the ratio of pore fluid electrical conductivity and soil electrical conductivity (Archie 1942). Other properties such as soil specific area, compressibility and swelling potential can be related to the decrease in the dielectric behaviour within the frequency range of 1 MHz to 100 MHz.

2.4. Influence of temperature

The dielectric properties of a material are dependent on temperature. The temperature sensitivity of soil dielectric itself is not well understood due to its complex interaction. The real dielectric permittivity ($\varepsilon'$) has a negative correlation with temperature while the imaginary dielectric permittivity ($\varepsilon''$) has a strong positive correlation. In materials with a dominating $\varepsilon'$, an increase in temperature causes a decrease in VWC, while in $\varepsilon''$ dominating soils, an increase in temperature causes an increase in VWC. In soils where the two components balance each other no temperature sensitivity is observed (Cobos and Campbell, 2007). Wraith and Or (1999) also found that the temperature effect on dielectric permittivity depends on specific surface of soil and water content. Low surface area soils with varying water content have a negative relationship. High surface area soils with low water content have a positive relation but high surface area soils with high water content gave differing results.

The influence of temperature on electrical conductivity is better understood and is given by (Kizito et al., 2008):

$$\sigma_b^T = \sigma_b^{ref} \left[ 1 + \alpha(T - T^{ref}) \right]$$  \hspace{1cm} (2.8)

where, $\sigma_b^T$ is the measured bulk EC at given temperature $T$, $\sigma_b^{ref}$ is the measured bulk EC at a reference temperature $T^{ref}$ and $\alpha$ us the temperature coefficient of the bulk EC (~1/°C).
2.5. Previous EM studies

The electromagnetic response of materials depends on their texture, composition and the frequency of the applied EM wave. Unfortunately, very little information regarding the electromagnetic properties of CPB was found in the literature but considerable EM investigations were performed on other materials such as electrolytes, clay, sand soils and hydraulic materials. This section will summarize some of the previous studies performed on materials for geotechnical applications, on cement containing materials and finally, CPB.

2.5.1. Geotechnical Applications:

Topp et al. (1980) is credited to be the pioneer in developing an empirical relationship between relative permittivity at gigahertz frequency to volumetric water content using time domain reflectometry (TDR) measurements for different types of soils. The classical third order polynomial Topp-model for mineral soils is:

\[
\kappa' = 3.03 + 9.3\theta + 146\theta^2 - 76.7\theta^3
\]  

(2.9)

where, \(\theta\) is the volumetric water content. Over the past three decades, several advances to the model have been made and employed within the agricultural industry to better predict the dielectric properties of the soils. A comprehensive list of different methods have been grouped into phenomenological, volumetric and (semi) empirical methods by van Dam et al. (2005). To understand the relaxation phenomena taking place in soils, studies have been done at different frequencies for soils (Wensink, 1993; Klein and Santamarina, 1997; Hilhorst et al., 2001) and clays (Ishida and Makino, 1999). The main relaxation mechanisms that occur in wet soils are illustrated in Figure 2-7. Typically, the orientational polarization begins to manifest at GHz frequencies, while bound water polarization occurs at the low MHz frequencies.
The Time Domain Reflectometry (TDR) technique is considered to be a reliable and reproducible method to characterize the electrical properties of most soils. Using the TDR technique to measure permittivity and electrical conductivity, Siddiqui et al. (2000) and Yu and Drnevich (2004) developed procedures to measure gravimetric water content and dry density for geotechnical applications. These procedures are now incorporated into an ASTM standard (ASTM D6780). Robinson et al. (1999) and Czarnomski et al. (2005) found that the permittivity measured from capacitance probes agreed well with the permittivity measured from the TDR method. The advantage of using the capacitance probes over TDR instruments is its low cost, data logging capability and ease of use in the field.

2.5.2. Hydration of cement containing materials

The existing methods to measure the changing engineering properties of cement containing mixtures such as penetration tests, unconfined compression tests (UCS) and moisture density are destructive in nature and do not monitor the chemical reactions taking place with time (Drnevich, 2005). The common methods of monitoring the
hydration of cement in the laboratory are: X-ray diffraction, electron microscopy, thermal analyses and the conduction calorimeter. Of these, only conduction calorimetry can continuously study the cement hydration but it can only relate those processes in which changes in heat take place (Pavlík et al., 2003; Zhang et al., 1995).

Changes in water due to hydration process of cement containing materials can be continuously monitored using real relative permittivity. The amount of water required for the formation of hydrates in Portland cement is relatively high and is reflected in the electrical properties (Pavlík et al., 2003). McCarter (1996), Berg et al. (1992) Miura et al. (1998) and Hager and Domszy (2004) evaluated the dielectric properties as a function of free water content in cement containing materials at various frequencies ranging from $10^{-3}$ Hz to 8 GHz. The researchers related the dielectric relaxation processes occurring at the GHz range to monitor the free water content during hydration. The relaxation processes due to bound water and chemically bound water were observed at approximately 100 MHz and 1 MHz, respectively (Miura et al., 1998; Hager and Domszy, 2004). These relaxation phenomena are also observed in soils at similar frequencies (Figure 2-7). Pavlík et al. (2003) monitored the moisture contents of cement pastes using the dielectric properties and tested the samples for their mechanical properties. They observed that that fastest decrease in moisture content coincided at early stage of hardening processes, i.e. between the end of setting and the moment the first measurable values of mechanical parameters were obtained (i.e. beginning of the hardness stage).

Tamas (1992) measured the electrical conductivity during hydration of early age (48 hours) Portland cement pastes at 3 kHz. He observed two maximums in conductivity occurring at 1 and 8 hours. He related the first maxim as a consequence of hydrolysis on introduction to water releasing Ca$^{2+}$, OH$^{-}$ and SO$_4^{2-}$ ions. The consumption of ions during the formation of C-S-H hydrates and ettringite reduced the electrical conductivity. The second maxim was attributed to the transformation of ettringite to mono-sulphate. Therefore, electrical conductivity properties can also be used to study the evolution of micro-structural properties of cement containing materials.
Zhang et al. (1995 and 1996) used microwaves to investigate early age hydration of Portland cement and blended cements containing different amounts of slag at frequencies ranging from 8.2 GHz to 12.4 GHz. They observed that with the addition of slag in cement, the permittivity slightly increased and the induction stage of the hydration process was reduced. The electrical conductivity of slag containing samples was also different from pure cement: the presence of slag delayed the hydration process at early stages of hydration. A second peak was also observed indicating the availability of additional ions to form new hydrated products.

2.5.3. Hydration of CPB

Several studies regarding the static mechanical properties of CPB are available in the published literature; however there is very little information on the hydration processes involved in CPB. Klein and Simon (2006) studied the physicochemical reactions of CPB using electromagnetic waves showing that EM is sensitive to changes in water content, material composition, binder content and frequency of an electric field. They also correlated changes in EM to the different stages of binder hydration. The EM properties reflect the amount of free and bound water in the pastes and the effective electrical conductivity is a good indicator to identify the stages of hydration as shown in Figure 2-8.
Figure 2-8 Relating changes in EM for PC and CPB containing 5% PC specimens to the different stages of hydration (from Klein and Simon, 2006)

Simon and Grabinsky (2007) and Simon et al. (2009) expanded the laboratory work to determine the in-situ dielectric properties in a backfilled mine stope using commercially available capacitance probes. Unfortunately, the dielectric sensors of the capacitance probes were influenced by the high electrical conductivity of the soils and did not reflect the actual VWC. However, the EC measurements from the capacitance probes were similar to that obtained from the network analyzer.

Simon and Grabinsky (2009) studied the retarding effect the supplementary pozzolanic material fly ash has on binder hydration using EM measurements obtained from the broadband network analyzer and capacitance probes. Addition of fly ash shifted the primary EC peak further to later times indicating that EM in CPB is also sensitive to binder type (Figure 2-9). The EC from the capacitance probes were consistent with the network analyzer readings.
Figure 2-9 Temporal response of EM showing the retarding effect of fly ash: a) $\kappa'$ at 1 GHz reflecting free water from network analyser; b) EC from the network analyzer; c) EC from the capacitance probe (from Simon et al., 2009)

In this study, both network analyzer and capacitance probes are used to study the effect of binder content, the addition of aggregates and influence of temperature on EM measurements.
CHAPTER 3
DESCRIPTION OF MATERIALS AND MEASUREMENT TECHNIQUES

Electromagnetic (EM) wave-based experiments were performed on Cemented Paste Backfill (CPB) at Xstrata Copper Canada’s Kidd Mine, Timmins, Ontario and in the laboratory. The following chapter provides a brief description of the raw constituents tested and the instruments used for EM measurements.

3.1. Description of Materials

The material components of CPB were collected and transported from the mine site in sealed five-gallon pails. The components consisted of silica tailings, alluvial sand, binder and mine process water.

3.1.1. Tailings

The tailings used by the mine are obtained from the local gold mines. The chemical composition of the tailings was determined using X-ray Fluorescence (XRF) technique. The composition summary presented in Table 3-1 indicates that the material is rich in silica. Minor amounts of alumina, iron, magnesium and equivalents of calcium and sulphur were also found in their oxide forms. The lower sulphur content in the tailings will not affect the hardening process of CPB as increasing soluble sulphates retards the hydration process of cement hydration (Benzaazoua et al., 2004). The specific gravity of the tailings was 2.80 determined in accordance to ASTM D854 (2006).

Figure 3-1 shows the particle size distribution of the tailings. The tailings were saturated with the sodium hexametaphosphate deflocculant for the hydrometer tests as specified in
ASTM D422 (2007). The average grain size ($d_{50}$) is approximately 35 $\mu$m. Approximately 40% of the tailings is finer than 20 $\mu$m (0.02mm). Landriault (1995) recommended that the at least 15% of the fines in tailings should be smaller than 0.02 mm to prevent particle settlement and segregation during transport.

Since the mine uses tailings that were previously stored on surface, the tailings appear olive-green in colour on the surface as a result of the oxidation from prolonged exposure to the environment.

3.1.2. Sand

The alluvial sand is obtained from local eskers. Alluvial sand is generally rich in silica and was assumed to be chemically inert. Thus, no chemical composition tests were performed. The specific gravity of sand was 2.70 and obtained in accordance with ASTM D854 (2006) and ASTM C127 (2007). The particle size distribution of sand is presented in Figure 3-1. The average grain size ($d_{50}$) is approximately 0.4 mm.

![Figure 3-1 Particle Size Distribution of Tailings and Sand](image-url)
3.1.3. Binder

The binder used at Kidd Mine is premixed at 90% blast furnace slag and 10% Type 10 Portland Cement and is purchased from Lafarge. Typically, cements containing up to 65% BFS are established in the construction industry. Higher levels of replacement are less documented but have advantages in low heat evolution and cost without compromising on mechanical strength (Hill and Sharp, 2002). The chemical composition of the binder as determined by XRF is summarized in Table 3-1. The binder primarily contains calcium and silica with minor amounts of magnesium and alumina.

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Tailings (%)</th>
<th>Binder (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>47.1</td>
<td>30.5</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>13.1</td>
<td>7.3</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>6.7</td>
<td>0.7</td>
</tr>
<tr>
<td>MgO</td>
<td>4.8</td>
<td>11.1</td>
</tr>
<tr>
<td>Ca eq</td>
<td>6.4</td>
<td>47.4</td>
</tr>
<tr>
<td>Na eq</td>
<td>1.7</td>
<td>0.4</td>
</tr>
<tr>
<td>Ba</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td>S eq</td>
<td>4.4</td>
<td>1.1</td>
</tr>
<tr>
<td>K eq</td>
<td>2.7</td>
<td>0.5</td>
</tr>
<tr>
<td>Others</td>
<td>1.0</td>
<td>1.1</td>
</tr>
</tbody>
</table>

3.2. Measurement Techniques

Dielectric properties of CPB were determined in the field using capacitance probes. To aid in the interpretation of the field data, EM tests were performed in the laboratory using the network analyzer and capacitance probes. The interpreted volumetric water content (VWC) data obtained was compared with the oven-drying water content measurements. Digital temperature probes were used to record the evolution of heat specimens. A data logger measuring ambient room temperature was also used at the same time.
3.2.1. **EM Measurements**

To measure the dielectric properties of CPB, two electromagnetic wave-based measurement techniques were employed, namely, a broadband network analyzer (20 MHz to 1.3 GHz) and single frequency capacitance probes (70 MHz). VWC and electrical conductivity (EC) were determined from the dielectric properties of the material.

3.2.1.1. High frequency broadband measurements

Broadband electromagnetic measurements of the CPB in the lab were performed using a HP 85070A dielectric coaxial open-ended termination probe with probe software, in conjunction, with a HP 8752A network analyzer. The face of the probe is composed of stainless steel, a glass to metal hermetic seal and Inconel, a super alloy. The diameter of the face of the probe is 19 mm. The impedance of the coaxial termination probe and coaxial cable is 50 Ω. The system measures permittivity in increasing frequency sweep from 20 MHz to 1.3 GHz (radio frequencies). A more complete description is found in Klein (2004).

![Figure 3-2 Experimental set-up](image)

As shown in the schematic (Figure 3-2), the specimen must be placed in direct contact with the termination probe. An incident wave propagates from the network analyzer
through the coaxial transmission cable to the probe. At the end of the probe, a part of the wave is transmitted through the material in contact and a part of it is reflected back forming a standing wave in the transmission cable. The reflected wave or reflected coefficient depends on the electrical properties of the specimen. The network analyzer measures the amplitude and phase of the reflected coefficient and converts it to complex dielectric permittivity ($\kappa'$ and $\kappa''$) using the probe’s aperture model software. However, the measured reflection coefficient differs from the actual reflection coefficient due to systematic errors such as directivity ($e_d$), frequency responses ($e_r$) and source match errors ($e_s$). The actual reflection coefficient ($\Gamma_a^*$) is:

$$\Gamma_a^* = \frac{\Gamma_m^* - e_d}{e_r + e_s(\Gamma_m^* - e_d)}$$  \hspace{1cm} (3.1)

where, $\Gamma_m^*$ is the measured reflection coefficient. To account for the systematic errors, calibration of the system must be performed prior to each test of the unknown specimen. The systematic errors are determined for each frequency by measuring the reflection coefficient of three known materials: air (open-circuit), metallic shorting block (short-circuit) and deionized water (Blackham and Pollard, 1997). All measurements by the network analyzer described in this thesis use this method of correction.

Other errors that can result in the erroneous interpretation of real permittivity data include electrode polarization and skin depth. Electrode polarization is the migration of electrons from the metal’s probe into the specimen’s pore fluid. It is caused by the difference between the electronegativity of the ions in solution and the metal of the probe. This phenomenon creates a layer of ions at the interface between the probe and specimen. The layer acts as a capacitor in series with the specimen and can manifest as high real relative permittivity at low frequencies thus masking the actual permittivity data. The effects of electrode polarizations increase as electrical conductivity of specimens increases. Typically, this phenomenon occurs in the electrodes of capacitance measurement system at low frequencies (Klein and Santamarina, 1996) but Klein (2004) also showed it to
occur in an open-ended coaxial probe measurement system. Electrode polarization phenomenon was not observed in this study.

The penetration distance of an electromagnetic wave into a specimen that causes a 1/e (e = 2.718) decay in amplitude is called skin depth. For the network analyzer, the maximum skin depth [mm], \( S_d \) is given by (Hewlett-Packard, 1993):

\[
S_d < \frac{20}{\sqrt{|k^*|}}
\]  

(3.2)

3.2.1.2. Capacitance Probe (70 MHz)

The electromagnetic properties of CPB were determined using capacitance probes in the field as the network analyzer is not sufficiently robust for field applications. In addition, these probes are relatively cheap and so it is economically viable to install multiple probes at different locations within a test site. Laboratory experiments were also performed with the capacitance probes to compare with the field data. The set-ups for field and laboratory measurements will be discussed in subsequent chapters.

The ECH2O-TE capacitance probes, manufactured by Decagon Devices Inc., were used to measure the volumetric water content (VWC), electrical conductivity (EC) and temperature in the lab and field. Kizito et al. (2008), Czarnomski et al. (2005) and Bandaranayake et al. (2007) tested the ECH2O sensors for reliability and accuracy in soil moisture measurements for use in agricultural applications. The probe consists of 3 prongs as shown in Figure 3-3. The prongs are 50 mm long, 5.5 mm wide and spaced apart at 4.7 mm.
The probe calculates VWC based on dielectric permittivity of the material, while the dielectric permittivity is measured using the capacitance technique. In this technique, the dielectric permittivity of the medium is obtained by measuring the charge time of a capacitor. The probe’s prongs act as a conductor and the surrounding medium is the dielectric, thus forming a capacitor. The time $t$ to charge the capacitor from a starting voltage $V_i$, to a voltage $V$, with an applied voltage, $V_f$ is given by:

$$
t = -RC \ln \left[ \frac{V - V_f}{V_i - V_f} \right]
$$

where, $R$ is the series resistance and $C$ is the capacitance. The resistance and voltage ratio are held constant. Capacitance is a function of the dielectric permittivity ($\kappa$) of the medium and can be calculated by:

$$
C = \frac{\kappa A}{S}
$$
where, $A$ is the area of the capacitor and $S$ is the separation between the probe’s prongs. Since $A$ and $S$ are fixed values, the charge time on the capacitor is a linear function of the dielectric permittivity of the surrounding medium, shown by (Decagon Application Note 2009):

$$\frac{1}{\kappa} = \frac{t}{1} \left[ \frac{RA}{S} \ln \left( \frac{V - V_f}{V_i - V_f} \right) \right]$$  \hspace{1cm} (3.5)

The probe sends out a 70 MHz oscillating wave and the time taken to charge the surrounding medium is recorded by the dielectric sensors. The raw output data in millivolts (mV) is converted into volumetric water content using the probe’s linear mineral soil calibration model:

$$\theta = 1.087 \times 10^{-3} \times Raw - 0.629$$  \hspace{1cm} (3.6)

where, $\theta$ is the volumetric water content in m$^3$/m$^3$ and $Raw$ is the raw output data in mV (ECH2O-TE, 2007). In actuality, the calibration model is quadratic from 0 to 100% VWC but the linear calibration model shown in Equation 3.6 provides a good fit for VWC ranges from 0% to approximately 35%. Customized calibrations will be required to perform higher water content ranges.

The probe is capable of measuring temperature using a thermistor, located underneath the plastic mould above the probes, as shown in Figure 3-3. It is soldered to the copper ground trace on the board and is in contact with the probes. The temperature output is an average temperature along the prong surface (ECH2O-TE, 2007).

Electrical conductivity was measured by four gold strips located on two of the prongs of the probe as shown in Figure 3-3. The gold strips serves as electrodes to form a 4-probe Wenner array. Electrical conductance is calculated by applying an electric current to the outer electrodes and measuring the voltage between the inner electrodes. This is then
converted to electrical conductivity (EC) by multiplying the voltage and conductance by the cell constant which is the ratio of distance of the electrode to the surface area.

3.2.2. Direct Water Content Measurements

Gravimetric water content (GWC) of the specimens by mass is determined in accordance with ASTM D2216 (2005) and was compared with volumetric water content (VWC) data obtained from EM measurements. The relationships between the different water content measurements are:

\[
VWC = \theta = \frac{\text{Volume of water}}{\text{Total Volume of material}}
\]  
(3.7)

\[
GWC = w = \frac{\text{Mass of water}}{\text{Mass of dry soil}}
\]  
(3.8)

\[
\text{Mining } WC = \frac{\text{Mass of water}}{\text{Total Mass (soil, air and water)}} = \frac{\theta}{\rho}
\]  
(3.9)

where, \( \rho \) is the bulk density of the paste.

3.2.3. Temperature Measurements

Heat is generated during the hydration of binder while curing. To determine the temperature increase during the curing of CPB in EM experiments, temperature data were simultaneously recorded for a few experiments. Temperature data captured by the thermal resistor in the capacitance probes were compared with measurements taken using a BK Precision digital thermometer equipped with a type K probe. The K probe was inserted into paste contained in a small cylinder (diameter = 5 cm; height = 10 cm) and covered with plastic wrap. In addition, the relative humidity and room temperature were recorded using the HOBO® U14-001 LCD data logger by Onset.
This chapter begins with a brief description on the setting of the field work and summarizes the results of the in-situ EM characterization of CPB using capacitance probes in two long-hole stopes at Xstrata Copper’s Kidd Mine located in Timmins, Ontario. A detailed description of the installation of the instruments at Kidd Mine can be found at Thompson et al. (2009b). The field description that is pertinent to the EM study is presented in this section.

4.1. Field Set-up

4.1.1. Objective
The aim of the field work was to gather in-situ data at different locations within a backfilled stope to better understand the field performance of CPB. To capture several aspects of the paste’s evolving properties a multi-transducer approach was initiated. EM measurement devices were included in instrumentation clusters to identify the hydration processes taking place.

4.1.2. Instrumentation
The field program at Kidd Mine involved placing “instrument clusters” at different locations along the vertical height of the stope. Each cluster contained the following instruments:

- Total earth pressure cells (TEPC) – in three orthogonal positions, to measure total stresses
- Piezometer – to measure pore water pressure
- Heat dissipative sensor – to measure negative pore water pressure or suction
- Capacitance probe – to measure the electromagnetic properties
- Biaxial tilt meter – to determine the orientation of cluster
- Tri-axial accelerometers and dynamic pore pressure transducer (in select cages) – for dynamic measurements

The instruments were assembled within a wire cage of dimensions 74 cm x 60 cm x 54 cm, which in turn was mounted into a protective steel frame shown in Figure 4-1. The locations of the instruments relative to each other within the cage are shown in Figure 4-2. A closed-circuit camera visually confirmed that the paste flowed into the wired cage encapsulating the instruments thus ensuring good contact between the material and the instruments.

Figure 4-1 (A) Instruments mounted within cage; (B) Cages ready for installation; (C) Capacitance probe in cage (from Thompson et al., 2009a)
Six cages were positioned in the test stopes at Kidd Mine. Cages 1 and 2 were placed at the brow of the opening while cages 3, 4, 5 and 6 were suspended vertically from the back of the top sill at heights 3m, 8m, 15m, 22m, respectively (Figure 4-3). The installation of the cages in the stope took approximately 8 days. More information on the preparation and installation of the cages in Kidd Mine are reported in Thompson et al. (2009a and 2009b). The data from the capacitance probes were recorded using a Campbell Scientific CR1000 data logger.
4.1.3. **Test Stopes**

Two test stopes were successfully instrumented and backfilled at Kidd Mine. The first test stope, 67SL1 is located in the South Lens of 6700 level. The second test stope, 88 947 is located in the main ore body on the 8800 level. Henceforth, for the sake of convenience, stope 67 SL1 will be referred to as Stope 67 and 88 971 as Stope 88.

Stope 67 is at approximately 2,000 m from the earth’s surface with a floor-to-floor height of 32 m, with an undercut area of 28 m x 12 m and an overcut area of approximately 19 m x 28 m. The geometry of the stope from Cavity Monitoring Surveys (CMS) is presented in Figure 4-4 with the approximate locations of fill fences and instrumentation cages. The stope was filled with approximately 32,680 tonnes of paste backfill. The initial average air temperature recorded at this stope was approximately 22°C.

![Figure 4-4 CMS of Stope 67 showing the hanging cages (from Thompson et al., 2009b)](image)

Stope 88 is at 2,680m from the earth’s surface. The dimensions of the stope are 18 m x 11.5 m in the undercut and 24 m x 11.5 m in the overcut. The height of the stope is 40 m. The geometry of the stope from CMS is shown in Figure 4-5. The stope was filled with approximately 22,770 tonnes of paste backfill. The initial average air temperature
recorded at this level was approximately 33°C, an increase of 10°C from the previous stope. The difference in temperature between the two stopes may be due to a combination of poor ventilation below 6900 level and the thermal gradient of the rock.

Figure 4-5 CMS of Stope 88 (from Thompson et al., 2009b)

4.1.4. Backfilling Detail

The mine employed a two-stage filling system in which the paste is first poured into the stope until the pressure in the bulkheads approaches approximately 100 kPa (15 psi). It is then allowed to cure until the pressure decreases and the paste reaches strength capable of withstanding the internal pressure generated from remainder of the pour in the stope. The paste is typically poured at 82% solids by mass. The density of the paste is 2100 kg/m³. For the first stage of the pour, referred to as “Plug 3” in the mine, 4.5% binder is added. For the second stage of the pour 2.2% binder is added and this mix is referred to as the “Main 10”.
The total volume of CPB delivered to Stope 67 was 15,560 m$^3$. A plug of 6.4 m thickness was poured using the “Plug 3” mix. The remainder of the stope was filled using the “Main 10” mix. The stope was backfilled between May 29 and June 4, 2008. There were four plant shutdowns during this period due to operational problems. The actual pouring time for the stope was 128 hours. Figure 4-6 is a schematic diagram of the stope with stages of pouring indicated with reference to instrument cage position.

In Stope 88, the total volume of CPB delivered was 11,844 m$^3$. A plug of 16.3 m thickness was poured using the “Plug 3” mix with the remainder of the stope filled with “Main 10” mix. The stope had to be backfilled in two periods as an extended shutdown was required due to paste line blockage in the upper levels of the mine. The first period was from July 6 to July 8, 2008 during which, there were two stoppages. Pouring resumed on July 27 and was completed on July 29, 2008. There was one stoppage in this period.
The total pour time was 82 hours. Figure 4-7 shows a schematic of various stages of pour with reference to the instrument cages.

![Figure 4-7 Stages of pouring in Stope 88. The hanging cages are indicated by black squares. (from Thompson et. al., 2009b)](image)

### 4.2. Results and Discussion

This section summarizes the results obtained from the capacitance probes in the cages at different locations in the two stopes. The capacitance probes measure the dielectric permittivity and records it as a voltage output in millivolts (mV). The dielectric permittivity measurements, which are dimensionless, are converted from voltage to VWC using the calibration equation 3.6. The EC is measured in decisiemens-per-metre (dS/m). A thermistor located under the mould of the probes records the temperatures in the material.

Though the paste was mixed at the paste-plant on surface and took approximately 40 minutes to reach underground, the field data and graphs in this report for VWC, EC and
temperature are presented starting from the time the paste came in contact with the prongs of the probes. When the probes are completely encapsulated by the paste a capacitor is formed with the prongs serving as the conductor and the surrounding paste as the dielectric or insulator. The raw data values for VWC in the probes sharply increases from approximately 430± mV in air to 900± mV in pastes and the time at which this occurs was identified as the arrival time of CPB. The raw voltage data for VWC for stopes 67 and 88 showing the arrival times are shown in Figure 4-8 and Figure 4-9, respectively. The arrival times of cages in both stopes are summarized in Table 4-1. The time shown in these graphs is the real time from when the data logger started to record the data.

![Figure 4-8 VWC Raw data voltage for Stope 67](image-url)
Capacitance probes were present in all 12 of the instrument-cluster cages in both stopes combined; however, probes in cages 4 and 5 in Stope 67 malfunctioned and were not included in the analyses. Of the remaining probes, 8 were located in cages placed in the plug (4.5% binder) and the remaining 2 were located in cages in the main pour (2.2% binder), as shown in the schematic summary in Figure 4-10. The high and low binder volumes of CPB in both stopes are henceforth referred to as “plug” and “main pour”. Table 4-1 provides a summary of the location of the probes, the type of CPB they were in and arrival times.

<table>
<thead>
<tr>
<th>Cages</th>
<th>Location</th>
<th>Stope 67</th>
<th>Stope 88</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Arrival Time (min)</td>
<td>Binder Type</td>
</tr>
<tr>
<td>1</td>
<td>Brow</td>
<td>780</td>
<td>Plug</td>
</tr>
<tr>
<td>2</td>
<td>Brow</td>
<td>780</td>
<td>Plug</td>
</tr>
<tr>
<td>3</td>
<td>Stope</td>
<td>560</td>
<td>Plug</td>
</tr>
<tr>
<td>4</td>
<td>Stope</td>
<td>1600</td>
<td>Transition</td>
</tr>
<tr>
<td>5</td>
<td>Stope</td>
<td>-</td>
<td>Main Pour</td>
</tr>
<tr>
<td>6</td>
<td>Stope</td>
<td>5380</td>
<td>Main Pour</td>
</tr>
</tbody>
</table>
Chapter 4: Field Experiments

**Figure 4-10 Summary of field set-up**

<table>
<thead>
<tr>
<th></th>
<th>Stope 67</th>
<th>Stope 88</th>
</tr>
</thead>
<tbody>
<tr>
<td>Depth from Surface (m)</td>
<td>2000</td>
<td>2680</td>
</tr>
<tr>
<td>Volume of CPB (m³)</td>
<td>15,560</td>
<td>11,844</td>
</tr>
<tr>
<td>Initial Air Temperature (°C)</td>
<td>22</td>
<td>33</td>
</tr>
<tr>
<td>Plug Height (m)</td>
<td>6.4</td>
<td>16.3</td>
</tr>
<tr>
<td>Cages in Plug</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>Cages in Main Pour</td>
<td>2</td>
<td>1</td>
</tr>
</tbody>
</table>

Figure 4-11 and Figure 4-12 show comparative overviews of VWC, EC and temperature data for each probe in all the cages in Stopes 67 and 88. Electrical conductivity in dS/m is plotted in the primary y-axis, while temperature and VWC, expressed in degree Celsius and percentage, respectively, are plotted in the secondary y-axis with respect to time in days. Detailed analyses are provided in the following sections.
Figure 4-11 Comprehensive overview of EM results in Stope 67; EC (primary y-axis); VWC and Temperature (secondary y-axis); a) Cage 1, b) Cage 2, c) Cage 3, d) Cage 4 and e) Cage 6
Figure 4-12 Comprehensive Overview of EM results in Stope 88; EC (primary y-axis); VWC and Temperature (secondary y-axis); a) Cage 1, b) Cage 2, c) Cage 3, d) Cage 4, e) Cage 5 and f) Cage 6
Significant increases in temperature were evident in the field data due to the exothermic nature of binder hydration reactions which can affect the dielectric properties of the material. For this reason, the presentation of the field results begins with temperature followed by EC and VWC data.

4.2.1. Temperature

Figure 4-13 presents the temperature data for Stope 67. The probe in cage 1 increased from 25°C and peaked to 38°C at approximately day 9 and then decreased towards the initial temperature. Similar trends were observed for cages 2 and 3: the temperature increased from 24°C and 23°C, respectively, and reached the maximum value of approximately 37°C and 38.6°C, respectively by around day 10 and day 20, respectively. Temperature data for cages 4 and 5 for Stope 67 were acquired using the thermistor in the piezometers as the TE probes in those cages malfunctioned. The temperature in cage 4 increased from 23°C to 36°C by day 24. The temperature remained steady for another 25 days and then began to decrease. Cages 5 and 6 showed similar trends as temperature increased from approximately 22°C and 23°C, temperature rise in Cage 4, located in the transition zone between plug and main pour, exhibits trends of both high and low binder containing CPB.
Figure 4-13 Temperatures in Stope 67

Figure 4-14 presents the temperature data for Stope 88. Similar trends as that observed in the Stope 67 were evident in Stope 88. The temperature in cages 1 and 2 in the plug increased from 34°C and 32°C, respectively to approximately 47°C by day 10 and then decreased steadily towards the initial temperature. The temperature in cages 3, 4 and 5 also located in the plug increased from 32°C, 32°C and 33°C, respectively and peaked at 48°C, 49°C and 50°C, respectively by day 20. The temperature of cage 6 in main pour plateaued from 31°C to 42°C by day 15.
The 10°C initial difference in temperature between the two stopes 67 and 88 may be due to a combination of the geothermal gradient of rock and poor ventilation below level 6900 in the mine. Distinct trends were, however, discernible for the different binder contents at different locations in the both stopes irrespective of the initial conditions. The normalized rise in temperature for the cages in stopes 67 and 88 are compared for different binder content within the stope: plug (Figure 4-15b) and main pour (Figure 4-15c). Comparisons were also made at different boundary conditions in the plug: under the brow (Figure 4-15a) and main stope (Figure 4-15b). These are summarized in Table 4-2. The slope of the rise in temperature in all the cages from stopes 67 and 88 are identical. The temperature in the cages under the brow in both stopes increased and decreased at similar rates peaking at ~13°C in ~10 days. Similarly, for the cages located in stope plug in Stopes 67 and 88, the temperature increased at identical rates peaking at 15°C by day 20 and decreasing. The temperatures in cages in the main pour of Stopes 67 and 88 increased and plateaued between 10°C and 12°C at approximately day 20. The implication of these graphs is that the rise in temperature in not influenced by the initial temperature conditions at the mine or the volume of high binder content but just by the
binder content in CPB. Therefore, a temperature curing function can be easily incorporated into the modeling tools to assess the physical impacts of temperature such as expansion and shrinkage in the design of CPB systems.

Kizito et al. (2008) and Cobos and Campbell (2007) confirmed that temperature has little effect on the sensor electronics of ECH2O-TE capacitance probes using air tests. Therefore, the changes in temperature observed in the field should not affect the recorded EM data. However, the EM properties of a material are influenced by temperature. According to the manufacturers, the ECH2O-TE probe in soils for VWC has a maximum temperature sensitivity of ~0.003 m^3 m^-3 per °C (Decagon FAQ, 2006). The ECHO2O-TE probes use an internal calibration to correct EC for temperature fluctuations and Kizito et al. (2008) verified the calibration is adequate for soils. The influence of temperature on the dielectric properties of CPB will be addressed in subsequent sections and chapters.

Table 4-2 CPB Temperature trends observed in field

<table>
<thead>
<tr>
<th>Location</th>
<th>Binder in CPB (%)</th>
<th>Peak ΔT (°C)</th>
<th>Time to peak (days)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brow</td>
<td>4.5</td>
<td>13</td>
<td>10</td>
</tr>
<tr>
<td>Stope</td>
<td>2.2</td>
<td>15</td>
<td>20</td>
</tr>
<tr>
<td>Stope</td>
<td>2.2</td>
<td>10 - 12</td>
<td>20</td>
</tr>
</tbody>
</table>
Figure 4-15 Comparison of rise in temperature in both stopes; 
  a) Brow – Plug, b) Stope – Plug, c) Stope – Main Pour
4.2.2. **Electrical Conductivity (EC)**

Electrical conductivity reflects the availability of free ions in a material and was discussed in Chapter 2 as a useful method to monitor the stages of hydration in cement-containing samples. When cement containing materials come in contact with water, ions will be released into the pore solution which will be consumed to form hydration products. The ionic activity is measured by EC by the electrodes in the capacitance probes. The EC for materials with higher binder content will decline prior to materials with lower binder content since materials with higher binder content will harden more quickly.

Figure 4-11 shows the EC data for individual cages in Stope 67. The data in cages 1, 2 and 3 were scattered until day 1. The probe in cage 4 recorded data for the first 12 days since the start of the test and then malfunctioned. The probe in cage 5 also malfunctioned and no good data was obtained from it. The data in cage 6 showed lower EC values than the other cages and a high degree of scatter was observed in the data until day 3. Cages 1, 2, 3 were located in the plug containing higher binder; cage 4 was located in the transition zone between the plug and lower binder containing main pour while cages 5 and 6 were located in the main pour. The scatter in the EC data for the initial days ranges from 0.67 to 4.5 dS/m for the cages in the plug and 0.5 to 1.9 dS/m for the cage in the main pour. Figure 4-16 compares the EC of the probes in all the cages. Due to the scatter observed in the initial days, it is difficult to discern any peaks but two trends can be observed: the EC declines rapidly near day 1 and 3 for cages in the plug and main pour, respectively.
Figure 4-12 shows the EC data for the individual cages in Stope 88. Cages 1, 2, 3, 4 and 5 are located in the plug while cage 6 is located in the main pour. The data in cages 1, 2, 3, 4 and 5 are not as scattered as the data in the previous stope but only becomes legible after approximately half a day. The data in cage 6 has a higher degree of scatter for the first two days. Similar trends as that observed in the cages in Stope 67 are observed in Stope 88: the EC declines rapidly prior to day 1 and 3 for cages in plug and main pour, respectively. The pre-peak EC data ranges from 0.4 to 1.96 dS/m for cages in the plug and 0.4 to 1.5 dS/m for main pour as shown in Figure 4-17. The spike in EC observed in cages 3, 4 and 5 on approximately day 22 reflects the resumption of pour in Stope 88 following break in the paste pipeline.
The scatter observed in the data may be due to the contamination of the gold electrodes during installation of the probes into the cages. EC measurements are very sensitive to the presence of non-conducting contamination, such as skin-oils from finger prints, especially at high EC. In a high conductive material the resistance of the material is low but the resistance from the contamination can dominate the total resistance leading to erroneous electrical conductivity data (ECH2O-TE, 2007). When binder reacts with water in the initial stages of hydration, a lot of ions are released into the pore fluid increasing the electrical conductivity of the material. The scatter in the data at the initial days may be due to the dominating resistance of the contaminant. As free ions in the pore are consumed to form hydration products, the total resistance will be dominated by the CPB which may be the reason the data is more legible at later days of monitoring.

Increasing temperature increases the rate of binder hydration reactions in CPB (Fall and Samb, 2006). Since higher temperatures were observed in Stope 88, the hydration products in Stope 88 should form faster than in Stope 67. This is reflected in the EC data and is more prominent for data in the plug (Figure 4-18) than the main pour (Figure
The EC data for the plug in Stope 88, represented by warmer colours, decline prior to EC data for plug in Stope 67 which is represented by the cooler colours.

Figure 4-18 Influence of temperature on 4.5% binder (plug) in in-situ EC

Figure 4-19 Influence of temperature on 2.2% binder (main pour) in in-situ EC
The EC data for cages in plug and main pour in both stopes follow similar trends. The peak in EC can be hypothesized to occur prior to days 1 and 4 for plug and main pour, respectively. This hypothesis will be verified by comparing the field results with laboratory EM experiments in subsequent chapters.

4.2.3. Volumetric Water Content (VWC)

Figure 4-20 shows the VWC data for the cages in Stope 67. The cages under the brow (cages 1 and 2) show an initial VWC of approximately 35% and 40%, respectively. With the exception of a small perturbation of ~2%, the VWC in cages 1 and 2 remained steady for 20 days after which the VWC started to decrease. The initial VWC recorded by cages 3, 4 and 6 were approximately 48%, 40% and 40%, respectively. The VWC data in cage 3 showed the largest change showing a steady increase peaking at 55% by day 5 and then decreasing. The VWC data in cage 6 remained relatively constant at approximately 40% during the monitored time period. The probe in cage 4 malfunctioned after 12 days and the probe in cage 5 did not work.
Figure 4-21 presents the VWC data from Stope 88. Cages 1 and 2 measured initial water contents at approximately 40%. Cages 3 and 4 recorded initial VWC of 35% and cage 5 recorded 40%. These cages were located in the stope with high binder CPB. The data in these cages increased steadily and decreased after 20 days. The data in cage 6, located in the main pour of the stope, remained relatively constant hovering at 40%. Perturbations were observed in cages 1, 2 and 3 at day 1. The spikes in VWC observed in cages 3, 4 and 5 on day 22, corresponded to the resumption of CPB pour into the stope after the operational delay. A sudden increase in VWC in cages 1, 3 and 5 were also observed around day 100.

The initial VWC data, measured from the ECH2O-TE probes, in cages from both stopes when converted to gravimetric water content (GWC) match the designed GWC of ~22% at which paste is pumped underground. There is an increase in VWC for 20 days after which it decreases, similar to the temperature trends. The VWC also appears to be sensitive to binder content, with data in both stopes showing more variability in higher
binder content cages. Two distinct trends are observable in the individual cages in both stopes (Figure 4-11 and Figure 4-12). Firstly, a drop in VWC occurs as EC decreases. This would indicate that the water content is decreasing as a result of the hydration of CPB. Secondly, there is a positive influence of temperature on VWC data. The VWC increases with increasing temperature, peaking at the same time the temperature peaks and decreases with decreasing temperature. Water content should not increase since additional water was not pumped into the stope. In previous field studies, the water content of CPB, as determined by ASTM D2216 (2005), had remained relatively constant even after approximately 9 months of curing (Grabinsky et al., 2008).

The manufacturers tested the ECH2O sensors in different media to study the temperature dependence and observed little response in soils with low specific surface area. Moderate temperature sensitivity of 0.003 m³ m⁻³ per °C was observed for fine textured soils (Decagon FAQ, 2006; Campbell, 2001). Since tailings is a high specific surface medium, the above factor was used to correct the temperature sensitivity in VWC for the probes in the cages in Stopes 67 and 88 and is presented in Figure 4-22 and Figure 4-23, respectively. Corrected data from the probe in cage 4 in Stope 67 is not presented as the probe malfunctioned after 12 days. Data from cage 5 is also not presented as the probe did not work at all. Though the temperature sensitivity in VWC was minimized, the corrected VWC still greatly fluctuated with time. Since the dielectric sensors in ECH2O-TE probes were developed and tested for soils and not for cemented materials with evolving properties, the field VWC results will require to be compared with laboratory results to verify whether the probes are reflecting the actual VWC of CPB.
Figure 4-22 Temperature sensitivity corrected VWC for probes in Stope 67

a) Cage 1, b) Cage 2, c) Cage 3 and d) Cage 6
Figure 4-23 Temperature sensitivity corrected VWC for probes in Stope 88

a) Cage 1, b) Cage 2, c) Cage 3, d) Cage 4, e) Cage 5 and f) Cage 6
4.3. Summary

To summarize, the capacitance probe used to determine the EM properties of CPB is a commercially available agricultural product. A thermistor located in the probe provides information on temperature. The temperature recorded in Stope 88 was 10°C higher than that recorded in Stope 67. However, the rise in temperature for the cages in the two stopes was identical. The temperature increase in the cages under the brow peaked at ~13°C in ~10 days. Similarly, the temperature increase in cages in the stopes within the higher binder peaked at 15°C and the cages in the main pour plateaued between 10°C and 12°C by day 20. Therefore, the rise in temperature is dependent on binder content and boundary conditions and can be used to develop a temperature function into modelling tools.

The gold electrodes in the probes reflected the ionic activity in the material as EC during the curing of CPB in the stope. A lot of scatter was observed in the data from cages in both stopes, especially in the initial days. Despite the scatter and noise in the data, distinct trends were identified for the different binder contents in CPB. The peak in EC was hypothesized to occur prior to days 1 and 4 for plug and main pour, respectively. The scatter in the data in the initial days may have been due to contamination of the electrodes during installation. In the initial period, the resistance due to contaminants will dominate the total resistance resulting in erroneous EC. As CPB hardens with time, the resistance in CPB dominates the total resistance and the EC data becomes more accurate. The hydration reactions of the binder in CPB were affected by temperature and were reflected in the EC data as the trends shifted to earlier times.

Though the dielectric sensors of the probe reflected the designed water content at the initial day, the field data fluctuated with time and therefore, need to be verified with the lab data to better interpret the field data.

To conclude, laboratory tests are required to verify the accuracy of the field data, especially the VWC data and will be discussed in subsequent chapters.
Electromagnetic (EM) experiments of cemented paste backfill (CPB) were performed in laboratory using the same model capacitance probes as in the mine to validate the field results with a more accurate and reliable TDR method using a dielectric coaxial termination probe in conjunction with a broadband high frequency network analyzer. Laboratory EM experiments were also used to interpret the binder hydration processes that take place. Experiments were performed on mixtures and raw constituents of Kidd mine CPB. Experiments were also performed at elevated temperatures to investigate the influence of temperature on hydration rates and instrument sensitivity. The chapter begins with the description of the experimental set-up and concludes with the presentation and preliminary discussion of the results.

5.1. Test Set-up

5.1.1. Specimen Preparation

Electromagnetic experiments in the laboratory were performed on CPB samples made using the same mix designs (i.e. same tailings-aggregate-binder ratios and water contents) as used in the field. The samples consisted of approximately 55% tailings (MT) and 45% sand (S) by mass mixed with binder (B) and mine process water (PW). The paste was prepared at 82% solids by mass. Since the mine employed a two-stage backfilling strategy with binder contents of 4.5% (plug) and 2.2% (main pour), experiments were performed on both samples. The water-to-binder ratio is approximately 12.82 and 6.37 for plug and main pour, respectively.

In addition, to better understand the EM properties of the CPB, experiments were also performed on individual components and different mixtures of the paste. Table 5-1
provides a summary of the specimens tested with their initial mining water contents, water-to-binder ratios and wet densities. Some of the specimens were prepared at different water contents to maintain the same slump consistency (fluidity) as the CPB mixture at the mine. Tests for the different mixtures were performed at least twice for reproducibility of results.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Mining water content (%)</th>
<th>Water-to-binder ratio</th>
<th>Wet Density (g/cm³)</th>
<th># of NA Expts</th>
<th># of TE Expts</th>
</tr>
</thead>
<tbody>
<tr>
<td>Individual Components</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tailings (MT)</td>
<td>28</td>
<td>1.93</td>
<td>2</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td>Sand (S)</td>
<td>16</td>
<td>2.07</td>
<td>2</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>Binder (B)</td>
<td>29</td>
<td>0.41</td>
<td>1.86</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Process water (PW)</td>
<td>100</td>
<td>-</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Deionized water (DW)</td>
<td>100</td>
<td>-</td>
<td>1</td>
<td>1</td>
<td>1</td>
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<tr>
<td>Mixtures</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MT + S</td>
<td>22</td>
<td>-</td>
<td>1.99</td>
<td>-</td>
<td>1</td>
</tr>
<tr>
<td>MT + 2.2B</td>
<td>28</td>
<td>18.57</td>
<td>1.9</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>MT + 4.5B</td>
<td>28</td>
<td>8.64</td>
<td>1.87</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>MT + S + 2.2B</td>
<td>22</td>
<td>12.82</td>
<td>1.99</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>MT + S + 4.5B</td>
<td>22</td>
<td>6.27</td>
<td>1.93</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Temperature Effects @ 40°C</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MT + S + 2.2B</td>
<td>22</td>
<td>12.82</td>
<td>1.99</td>
<td>-</td>
<td>1</td>
</tr>
<tr>
<td>MT + S + 4.5B</td>
<td>22</td>
<td>6.27</td>
<td>1.93</td>
<td>-</td>
<td>2</td>
</tr>
</tbody>
</table>

1 NA: Network Analyzer
2 TE: Capacitance Probe

The tailings were mixed with mine supplied process water to the required water content for one minute using a high torque paint mixer. Other components, such as sand and binder, were added and mixed after one minute intervals for approximately 4 minutes. The sand was oven-dried and made to pass the No. 10 sieve (2 mm) to prevent the larger particles from blocking the EM wave source point of the dielectric termination probe, the diameter of which is approximately 2 mm. This ensures that the reflected coefficient recorded by the network analyzer is that of the paste and not blocked by larger particle sizes. The time from which the binder is in contact with the wet mixture was recorded and served as the start time of the experiment. Initial water content and density measurements were taken prior to pouring the paste into the containers. Wet density was
calculated by weighing the mass of paste in a cylinder of known mass and volume. As the paste was poured into the containers, the paste was tapped with a flat spatula to minimise air voids.

5.1.2. **Procedure**

EM measurements in the laboratory were performed using the network analyzer and capacitance probes described in Chapter 3. The experimental set-up is shown in Figure 5-1. The dielectric coaxial probe from the network analyzer is held within a holder and a hollow cylinder (Ø = 10 cm; height = 15 cm) is placed above it such that the paste will be in contact with the dielectric probe. Three 5 mm vertical slits, spaced 5 mm apart, were cut approximately 3.5 cm to 4 cm from the bottom of the hollow cylinder to insert the ECH$_2$O-TE capacitance probe with flat side vertical. Two other probes were similarly inserted into two identical cylindrical containers. The probes were sealed with silicon caulking to prevent water leaking. The containers were hermetically sealed with plastic wrap and lids to prevent the evaporation of water. Three experiments were performed using only the capacitance probes in a scientific drying oven with the temperature set to approximately 40°C, in an attempt to “simulate” the temperatures observed at the mine site. The three oven experiments were performed on the following CPB samples:

- 2.2% binder added to tailings and sieved sand
- 4.5% binder added to tailings and sieved sand
- 4.5% binder added to tailings and un-sieved sand

Temperature within the CPB paste and the surrounding ambient conditions were monitored simultaneously as the EM experiments on a few CPB specimens. The temperature within the paste were monitored using the prongs of the capacitance probe which is in thermal contact with a thermistor located under the plastic mould and by type K thermometer probe inserted into a separate small container. A HOBO® data logger monitored the ambient temperature relative humidity.
Additional samples prepared at the same time as EM experiments were stored in smaller containers and allowed to cure in the same conditions as the EM experiments to obtain water content measurements at different time intervals during the test. At the end of each test, samples were taken from close proximity of the EM probes to obtain water content measurements. Gravimetric water content measurements were obtained by oven drying to a constant mass at 110°C in accordance with ASTM D2216 (2005) and converted to volumetric water content.

Readings from the broadband network analyzer were taken at logarithmic time intervals and the data was stored in the computer connected to the analyzer. The data from the capacitance probes were continuously monitored using the Em50 Data logger, manufactured by Decagon Devices Inc. The duration of all tests, excluding 100% binder was at least 6 days. To prevent permanent damage to the capacitance probes, the duration of the 100% binder was limited to 10.5 hours.

1. Samples for oven dried water content measurements
2. EM experiment using capacitance probes
3. Em50 — Data logger for capacitance probes
4. Coaxial cable
5. Network Analyzer
6. Prongs of a capacitance probe
7. Dielectric termination probe

Figure 5-1 Laboratory EM set-up; Inset – View from top
5.2. Results

The following section summarizes the results obtained from the different EM experiments using the broadband frequency network analyzer and single frequency capacitance probes. The output parameters that feature in the results were discussed in Chapter 2. Refer to Table 5-1 for the notations used in the graphs.

5.2.1. Spectral response of materials

Figure 5-2 shows the variations in real relative permittivity (Figure 5-2a), imaginary relative permittivity (Figure 5-2b) and effective electrical conductivity (EC) (Figure 5-2c) across the frequency range of the network analyzer (20 MHz to 1.3 GHz) for different materials 30 minutes after the specimens were mixed.

In Figure 5-2a, the real relative permittivity for both process water (PW) and deionized water (DW) is approximately 80 and remains constant through the frequency spectrum. The real relative permittivities of the various CPB mixtures and the raw constituents are much lower and increase with decreasing frequencies. The increase in real relative permittivity with decreasing frequency at MHz range is indicative of bound water polarization. The orientational polarization of free water in the different specimens begin to manifest at 1 GHz (10^9 Hz) and is confirmed by the deviations in effective imaginary relative permittivity and effective electrical conductivity trends (van Dam et al., 2005; Klein and Simon, 2004). Thus, the temporal response of real relative permittivity of bound and free water is presented at 70 MHz and 1 GHz, respectively. The real relative permittivity is presented at 70 MHz to compare the volumetric water content (VWC) determined from the dielectric properties using the capacitance probe which operate at a frequency of 70 MHz.

The linear decrease in effective imaginary permittivity with increasing frequency in the log-log scale (Figure 5-2b) indicates the prevalence of conduction losses. Deviations from this linear trend at higher frequencies (closer to GHz frequencies) indicate polarization losses due to the orientational polarization of free water. This phenomenon
begins to manifest in most materials at approximately 1 GHz and is more evident for process and deionized water. The process water also appears to be conductive as the conduction losses dominate in the lower frequencies. Deionized water is not conductive as the data is scattered in the lower frequencies but are dominated by polarization losses in the GHz frequency range.

Similarly, an increase in effective conductivity with increasing frequency (Figure 5-2c) is due to the free water polarization. The frequency at which polarization losses become noticeable depends on the composition of the specimen (Klein and Simon, 2004). For this reason, the temporal responses for effective conductivity are presented at a lower frequency of 70 MHz. This is also the operating frequency of the capacitance probe.
Figure 5-2 Spectral response of materials after ~30 minutes: 

a) real relative permittivity; 
b) imaginary relative permittivity; 
c) effect conductivity
5.2.2. Temporal response of materials

The following section summarizes the temporal response of materials with respect to volumetric water content (VWC), electrical conductivity (EC) and temperature.

5.2.2.1. VWC and $\kappa'$

Figure 5-3 to Figure 5-7 compares the real relative permittivity ($\kappa'$) obtained from the network analyzer and VWC from the capacitance probes and oven drying with time for various mixtures. The $\kappa'$ at lower MHz range and GHz is a good indicator of bound and free volumetric water content, respectively. In this study, the $\kappa'$ at 70 MHz and 1 GHz were selected to observe the bound and free water changes taking place in the material. The capacitance probe expresses the dielectric properties of the material as raw voltage using an operating frequency of 70 MHz. The raw voltage output is then converted to VWC using a linear calibration equation. The capacitance probe operates at a single frequency and therefore, does not recognize the polarization mechanism that occurs. According to the manual, the VWC readings from the capacitance probes are accurate within ± 3% VWC up to 8dS/m (ECH₂O-TE, 2007).

The volumetric water contents determined by the capacitance probes for the mine tailings (Figure 5-3), sand (Figure 5-4) and the two CPB mixtures with 2.2% (Figure 5-5) and 4.5% (Figure 5-6) binder showed relatively little change with time. These values were in agreement to within ± 3% VWC determined by oven drying, which decreased slightly during the duration of the tests. The oven-dried VWC at 10 minutes is higher than the VWC at later times due to the excess water in the mine tailings-water mixture. With time, the excess water bleeds out of the mixture due to self-weight consolidation.

The VWC from the capacitance probes for the 100% binder (Figure 5-7) steadily increased peaking to approximately 80% VWC at 400 minutes and then decreased. The more realistic VWC determined by oven drying remained constant closer to the actual initial VWC at approximately 50%. On contact with water, the binder releases ions increasing the conductivity of the pore fluid to approximately over 6 dS/m (Figure 5-11).
This is close to the threshold limit of the capacitance probes to accurately measure VWC. Therefore, the dielectric sensors in the capacitance probes are affected by the high conductivity of the medium resulting in the erroneous VWC output (Figure 5-11). Simon et al. (2009) also faced similar issues with the probes in high conductive tailings and concluded though the probes inaccurately measure VWC, its sensitivity to variations in EC allows the interpretation of cement hydration stages.

The real relative permittivity $\kappa'$ at 70 MHz and 1 GHz reflects bound and free volumetric water content, respectively. For cement-containing materials, the free water content decreased while the bound water content increased slightly with hydration time indicating that the state of water is changing either due to the consumption of free water to form hydration products or water becoming bound to the solid particles. These trends are most prominent in the 100% binder (Figure 5-7). For the non-cement containing materials, the trends in $\kappa'$ at 70 MHz and 1 GHz are similar implying the state of water is not changing.

![Figure 5-3 Temporal response of VWC by capacitance probes and oven-dried samples and $\kappa'$ at 70 MHz and 1 GHz by network analyzer for Mine Tailings (MT)](image-url)
Figure 5-4 Temporal response of VWC by capacitance probes and oven-dried samples and $\kappa'$ at 70 MHz and 1 GHz by network analyzer for Sand (S).

Figure 5-5 Temporal response of VWC by capacitance probes and oven-dried samples and $\kappa'$ at 70 MHz and 1 GHz by network analyzer for 2.2B+MT+S.
Figure 5-6 Temporal response of VWC by capacitance probes and oven-dried samples and $\kappa'$ at 70 MHz and 1 GHz by network analyzer for CPB with 4.5% binder.

Figure 5-7 Temporal response of VWC by capacitance probes and oven-dried samples and $\kappa'$ at 70 MHz and 1 GHz by network analyzer for 100 % binder (B).
Figure 5-8 to Figure 5-10 shows the performance of the capacitance probes at elevated temperatures by comparing the VWC from the capacitance probes with oven-dried values for different CPB specimens cured in a scientific oven at approximately 40°C. Since the scientific oven used in the experiments were for applications requiring 100°C, temperature uniformity could not be maintained at 40°C and sudden spikes in temperature were observed during the duration of the tests, especially during the curing of the 2.2% binder specimen (Figure 5-8). Two sharp increases in VWC were observed which corresponded to spikes in temperature. However, no increases in VWC were observed during the sustained temperature increase at the start of the tests. The spikes in temperature did not alter the general trend of the VWC and the VWC data appeared to be in agreement with the oven-dried VWC readings for the different specimens. Unlike the field data, the dielectric sensors in the probe did not seem to be affected by the temperature.

![Figure 5-8 Temporal response of VWC by capacitance probes and oven-dried samples for CPB specimen with 2.2% binder cured at elevated temperatures (40°C)](image-url)
Figure 5-9 Temporal response of VWC by capacitance probes and oven-dried samples for CPB specimen with 4.5% binder cured at elevated temperatures (40°C)

Figure 5-10 Temporal response of VWC by capacitance probes and oven-dried samples for CPB specimen with 4.5% binder cured at elevated temperatures (40°C). The sand used for this CPB mixture was not sieved
5.2.2.2. Electrical Conductivity

The capacitance probes operate at 70 MHz and so, the electrical conductivity (EC) data from the probes are compared with the effective electrical conductivity data from the network analyzer at 70 MHz with time in Figure 5-11. When binder reacts with water, ions are released into the pore solution. EC will reflect the ionic activity in the pore fluid of the specimens. The initial conductivities for all the specimens are identical in both instruments. The mine tailings and sand show no change in electrical conductivity with time and remains fairly constant at approximately 1.5 dS/m and 0.4 dS/m, respectively. The network analyzer and capacitance probes also capture the hydration of the 100% binder specimen containing Portland cement and slag. Two maxima were observed for the 100% binder specimen at approximately 6.2 dS/m and 6 dS/m occurring at approximately 4 and 6 hours, respectively.

However, unlike the capacitance probes, the network analyzer does not capture the drop in EC for CPB specimens with 2.2% and 4.5% binder. One of the reasons for this may be due to the fact that the specimen is not uniformly hydrating but hydrating from the boundaries based on visual inspection of the end-of-test specimen (Figure 5-12). The dielectric coaxial termination probe is still in the wet region while a part of the region near the capacitance probes appears to be dry. The network analyzer gathers data from a 2 mm source point in the termination probe, while the data from the capacitance probe is averaged along the length of the 5 cm long prongs. Thus, EC data from the capacitance probes were considered to be more reliable and will be presented for subsequent analyses.
Figure 5-11 Temporal comparison of EC by capacitance probe (TE) and network analyzer (NA) at 70 MHz for select specimens

Figure 5-12 Photo of the bottom of the specimen holder at the end of the test showing the “dry” and “wet” regions for CPB specimen with 4.5% binder

Figure 5-13 shows the EC data from the capacitance probes for the different specimens with time. Materials with no binder showed little or no change in EC during the duration
of the tests. Process water was previously identified to be conductive (Figure 5-2b) and its conductivity hovered around 0.7 dS/m. The electrical conductivity of mine tailings and sands were 1.45 dS/m and 0.41 dS/m, respectively. The tailings exhibited the highest conductivity after the binder but the tailings and sand mixture reduced the conductivity to approximately 1 dS/m. Binder containing specimens release ions on contact with water and this is reflected by the steady increase in EC with time. The EC for all cement containing materials reach a maximum EC and decline, indicating that the ions are being consumed faster than new ions being released. The EC for higher cement containing materials attain the maxima earlier than lower binder content materials. Another observation is that the EC for CPB mixtures containing sands peak prior to mixtures without sand. This implies that CPB with sands gains strength earlier than CPB without sand.

The temporal response of EC for 2.2% binder CPB cured at 40°C temperature is presented in Figure 5-14. Contrary to the VWC data (Figure 5-8), an inverse relationship
is observed between EC and the spikes in temperature. However, the spikes in temperature do not appear to affect the general EC trend. The EC increases from 0.78 dS/m to a maximum of 0.99 dS/m and declining at approximately 2740 minutes (~ 45 hours).

![Figure 5-14 Temporal response of EC for CPB with 2.2% binder cured at 40°C](image)

The temporal response of EC for 4.5% binder CPB specimens containing sand and sand sieved to less than 2 mm is shown in Figure 5-15. The EC for both sieved and un-sieved specimens increased from 0.97 dS/m to 1.14 dS/m and declining approximately 200 minutes (~ 3.3 hours). The identical EC trends observed in both sieved and un-sieved specimens suggest that sieving of sand to less than 2 mm did not alter the EM property of the material.
Table 5-2 summarizes the EC results for all the CPB mixtures as well as its raw constituents observed in the figures above. As mentioned previously, the maxima in EC is observed in those specimens containing binder. The correlation of EC to the different stages of hydration of binder and CPB as well as the influences and significance of the binder content, addition of sand aggregate and temperature will be discussed in the next chapter.
<table>
<thead>
<tr>
<th>Specimen</th>
<th>Curing Temperature (°C)</th>
<th>Peak EC (dS/m)</th>
<th>Time @ Initial set (min)*</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Individual Components</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tailings (MT)</td>
<td>22 ± 2</td>
<td>1.52</td>
<td>NA</td>
</tr>
<tr>
<td>Sand (S)</td>
<td>22 ± 2</td>
<td>0.42</td>
<td>NA</td>
</tr>
<tr>
<td>Binder (B)</td>
<td>22 ± 2</td>
<td>6.2</td>
<td>250</td>
</tr>
<tr>
<td>Process water (PW)</td>
<td>22 ± 2</td>
<td>0.68</td>
<td>NA</td>
</tr>
<tr>
<td><strong>Mixtures</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MT + S</td>
<td>22 ± 2</td>
<td>0.98</td>
<td>NA</td>
</tr>
<tr>
<td>MT + 2.2B</td>
<td>22 ± 2</td>
<td>1.28</td>
<td>6600</td>
</tr>
<tr>
<td>MT + 4.5B</td>
<td>22 ± 2</td>
<td>1.23</td>
<td>1440</td>
</tr>
<tr>
<td>MT + S + 2.2B</td>
<td>22 ± 2</td>
<td>0.99</td>
<td>3200</td>
</tr>
<tr>
<td>MT + S + 4.5B</td>
<td>22 ± 2</td>
<td>1.12</td>
<td>700</td>
</tr>
<tr>
<td><strong>Temperature Effects @ 40°C</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MT + S + 2.2B</td>
<td>40</td>
<td>0.99</td>
<td>2740</td>
</tr>
<tr>
<td>MT + S + 4.5B</td>
<td>40</td>
<td>1.13</td>
<td>200</td>
</tr>
<tr>
<td>MT + S (un-sieved) + 4.5B</td>
<td>40</td>
<td>1.14</td>
<td>200</td>
</tr>
</tbody>
</table>

* Drop in EC

5.2.2.3. Temperature Monitoring

To determine the heat generated by the laboratory specimens, changes in temperature were recorded using two instruments, the capacitance probes and a digital thermometer. Ambient temperature conditions were also recorded. Figure 5-16 and Figure 5-17 shows that for the CPB volumes used in the EM experiments, the temperature increases by 0.75°C to 1°C in the first two hours. During the remainder of the curing, the change in temperature in the paste is influenced by the ambient laboratory conditions. This contrasts with the temperature measured underground implying that the amount of material is critical and that internal heating effect controls the process rather than the boundary conditions.
Chapter 5: Laboratory Experiments

Figure 5-16 Temporal comparison of change in temperature in paste and ambient for CPB with 2.2% binder

Figure 5-17 Temporal comparison of change in temperature in paste and ambient for CPB with 4.5% binder
5.3. Summary

To summarize the chapter, the changes in $\kappa'$ at 70 MHz and 1 GHz using the broadband network analyzer reflects the state of water at any given time and can be correlated to understand the hydration stages of binder. The capacitance probes which can be used in the field provide VWC information without disturbing the material but its use is limited to material with electrical conductivity less than 8 dS/m. Oven experiments confirmed the dielectric sensors in the probe instrument are not affected by temperature up to 40°C and reliably reflects the dielectric properties of the material as the VWC recorded by the capacitance probes were in agreement with the VWC data determined by oven-dried samples. However, previous studies in CPB have shown that it takes very little change in water content for the material to change from liquid state to solid state. Since the probes can only determine VWC to within ± 3%, the VWC from the probes cannot be used to monitor the setting of the pastes.

The binder exhibited higher EC than other materials. The electrical conductivity of the tailings at Kidd Mine is higher than the materials mixed with binder. The correlation of EC to the different stages of hydration of binder and CPB as well as the influences and significance of the binder content, addition of sand aggregate and temperature will be discussed in the next chapter.
CHAPTER 6
VALIDATION AND DISCUSSION

This chapter compares and verifies the field results with the results obtained from laboratory experiments. The chapter also discusses the relaxation processes identified during the hydration of cemented paste backfill (CPB) across the frequency spectrum as well as the different stages of hydration and concludes with the discussion on the influence of material, binder composition and temperature on the hydration of CPB using electromagnetic (EM) data.

6.1. Validation of laboratory and field data

This section compares laboratory data with the field data for volumetric water content (VWC), electrical conductivity (EC) and temperature obtained from ECH2O-TE capacitance probes.

6.1.1. VWC

The initial field VWC recorded by the capacitance probes in both stopes at Kidd Mine was ~ 40% VWC which corresponded to the designed mining water content of ~22%. With time, however, the VWC data followed the same trends as the temperature in both stopes (Figure 4-11 and Figure 4-12). This phenomenon was not observed in experiments performed in the laboratory where the VWC from the probes were in agreement with oven-dried VWC (See Figure 5-3 to Figure 5-6) and also for experiments set at 40°C (Figure 5-8 to Figure 5-10). The temperatures in the stopes were within the -40°C to 50°C operating temperature limits specified in the manual. The maximum temperature dependence of the soil on VWC is 0.003 m³ m⁻³ per °C. Despite factoring in the correction the VWC still fluctuated with time. In previous field studies, there was no significant change in water content in the CPB even after approximately 9 months of
curing (Grabinsky et al., 2008). As a result, the in-situ VWC from the probes in the cages at Kidd mine were not analyzed for characterizing the CPB.

Also, the measurement precision of the VWC using the commercial capacitance probe is only accurate to ±3% (ECH2O-TE, 2007). This level of accuracy may be sufficient for agricultural studies, for which the probes are manufactured, but not for monitoring the hydration of CPB as it takes very little change in water content for the material to change from liquid to a solid state (Grabinsky et al., 2007b). Therefore, the dielectric sensors in ECH2O-TE’s capacitance probes may not be an ideal instrument to monitor the changes in water content during the hydration of CPB.

6.1.2. EC

A high degree of scatter was observed in the in-situ electrical conductivity data of the CPB, especially in the first day of pouring. Fortunately, with time the scatter in EC decreased and distinct trends were observable for main pour and plug. The scatter in the data may be due to contamination of the gold electrodes during installation of the probes into the cages. EC measurements are very sensitive to the presence of non-conducting contamination, such as skin-oils from finger prints, especially at high EC (ECH2O-TE, 2007). Figure 6-1 compares the temporal response of in-situ EC data from select cages in the plug and laboratory EC data for CPB containing 4.5% binder. The cages in Stope 67 and 88 are represented by pale blue and rose colour, respectively to distinguish the higher curing temperatures observed in Stope 88. The EC for CPB cured at higher temperature decrease prior to CPB cured at lower temperature indicating that the binder hydration reactions are increase at higher temperatures. This is evident in the field data and is also consistent with laboratory data performed at room and 40°C. Figure 6-2 compares the temporal data for the cages in the main pour and laboratory EC data for 2.2% binder CPB. Due to the lower binder content, the influence of temperature on binder hydration reaction is not prominent but the trends in in-situ EC data are identical to the laboratory data.
Figure 6-1 Comparison of temporal response of in-situ and laboratory EC for CPB containing 4.5% binder (plug)

Figure 6-2 Comparison of temporal response of in-situ and laboratory EC for CPB containing 2.2% binder (main pour)
The field and laboratory EC data in Figure 6-1 and Figure 6-2 appear to be consistent. The implication of these figures is that despite the scatter in the field EC data, the interpretation and characterizations of laboratory prepared CPB specimens can also be applied to in-situ EM data monitored at the mine.

6.1.3. Temperature

The temperature in laboratory specimens was observed to go up by ~1°C during the first day after which temperature in the paste was influenced by ambient laboratory conditions (Figure 5-16 and Figure 5-17). The volume of paste used in the laboratory experiments is insufficient to generate comparable temperatures as observed in the underground mines.

6.2. Discussion

This section discusses the results obtained from laboratory experiments in Chapter 5 which also apply to CPB cured in the field. It begins with the spectral analyses which identifies the relaxation processes that occur during hydration followed by the correlation of EM properties to the stages of hydration and concludes with the influence of material and binder composition and temperature on hydration.

6.2.1. Relaxation processes

Heterogeneous solid-water mixtures experience both single and multi-phase polarizations such as Maxwell-Wagner spatial, double layer bound and free polarization. These polarization processes have been identified for different materials at different frequencies. Ishida and Makino (1999) observed bound and free polarization at 1-10 MHz and 20 GHz, respectively in clay minerals. McCarter and Garvin (1989) investigated moisture condition in cement pastes at low frequencies in the range of 20 Hz and 110 MHz and concluded that Maxwell-Wagner polarization is the dominant at early stages of cement hydration but as cement pastes harden, bound water polarization becomes dominant. Miura et al. (1998) and Hager and Domszy (2004) observed three relaxation processes in cement pastes: free water polarization at 20 GHz, bound water at 100 MHz and chemical-
bound water polarization at 1 MHz. Bound water polarization at low MHz frequencies and free water polarization at higher GHz frequencies were the only two relaxation processes evident in electromagnetic studies of CPB at spot frequencies from 20 MHz to 1.3 GHz by Klein and Simon (2006) and Simon (2009). They asserted that Maxwell-Wagner polarization occurs at much lower frequencies than the bound water polarization and the occurrence of double layer polarization was assumed to be unlikely due to the close proximity of neighbouring particles. Though the free water polarization occurs between 10-20 GHz ranges, it begins to manifest at 1 GHz as seen in Figure 5-2c. The bound water polarization occurs at the lower MHz frequencies.

The amount of bound water binding on the surface of a particle depends on the mineralogy and specific surface area of the material. More water molecules will become adsorbed to materials with high specific surface area. Figure 6-3 and Figure 6-4 show the spectral response of real relative permittivities $\kappa'$ for binder containing specimens and non-binder containing specimens, respectively at 10 minutes and 10 hours. These figures essentially show the effect of curing with respect to time. The amount of bound water increases with increasing binder content. The difference in $\kappa'$ at approximately 10 minutes and 10 hours at lower frequencies is more prominent in the 100% binder specimen. Similarly, the real relative permittivity $\kappa'$ at high frequencies has decreased in binder containing specimen (Figure 6-3). This can be attributed to a combination of self-weight consolidation and consumption of free water to form hydrated products. In comparison, the spectral responses for real relative permittivity at 10 minutes and 10 hours show no change in materials that do not contain any binder (Figure 6-4).
Figure 6-3 Spectral response of $\kappa'$ for materials containing binder at 10 minutes and 10 hours

Figure 6-4 Spectral response of $\kappa'$ for materials that do not contain binder at 10 minutes and 10 hours
6.2.2. Hydration of CPB

On mixing with water, cement pastes undergo physicochemical changes to form hydrated products. These changes are sensitive to the electrical properties of the paste and therefore, can be used as a tool to continuously monitor the hydration of cemented pastes. Several studies have shown that the electrical properties of a hydrating material are sensitive to curing time, operating frequency and material composition including binder content and binder type.

During the hydration of cemented pastes, some of the water molecules get consumed to form hydration products while some water molecules get bound to solid particles (Fam and Santamarina, 1996). The polarization mechanisms of free and bound water occur at different frequencies and hence, the real relative permittivity of free water can be used as a tool to monitor cement hydration (Miura et al., 1998; McCarter and Garvin, 1989; Berg et al., 1992; Pavlik, 2003). In addition, electrical conductivity reflects the availability of ions in the material and can be used to correlate the different stages of cement and cement paste hydration (Tamas, 1982; Moukwa et al., 1991; Fam and Santamarina, 1996; Zhang et al., 1995 and 1996; Klein and Simon, 2006; Simon, 2009).

The distinct stages of hydration for 100% binder and CPB containing 4.5% binder with tailings and sand aggregate are identified in Figure 6-5 and Figure 6-6, respectively and discussed in detail below.

Stage 1: Initial period

In the first 30 to 40 minutes after mixing for both specimens, the EC appears to be slowly increasing. This can be attributed to the initial dissolution of binder grains and the release of ions such as Ca$^{2+}$, Na$^+$, K$^+$, OH$^-$ and SO$_4^{2-}$ into the pore solution to form a C-S-H gel. In both specimens, the real relative permittivity ($\kappa'$) at 1 GHz appears to gradually decrease while no change is observed in $\kappa'$ at 70 MHz. Most of the ions and molecules at this stage are unbound in the aqueous phase and can easily polarize. Zhang et al. (1996) and Klein and Simon (2006) observed similar trends for the initial period for cement pastes and CPB with 5% PC binder, respectively.
Stage 2: Induction period
The slope of the EC increases after ~ 40 minutes in both specimens. The rise in EC continues for ~ 4 hours after mixing for the 100% binder specimen, while for the 4.5% CPB specimen it lasts until ~ 8 hours after mix. At this stage, the binder grains are surrounded by a C-S-H gel membrane. Water is allowed to penetrate the semi-permeable gel membrane dissolving the blended-cement grains and releasing ions but the ions are not allowed to migrate into the outer solution resulting in a concentration of ions in the solution.

As the concentration of ions continue to build up, EC will increase reaching a maximum at which point the C-S-H membrane is ruptured due to the osmotic pressure difference. However, two maxima are observed in EC for the 100% binder specimen at ~ 4 and 6 hours. Tamas (1982) observed two maxima in EC during the hydration of Portland cement at ~1 hour and ~ 5 hours after initial mix. He attributed the first peak to dissolution of the cement grains and the second peak to the re-crystallization of ettringite to mono-sulphate. The latter phenomenon theoretically occurs at later stages of hydration approximately 17 hours for pure cement samples (Neville, 1995) and Klein and Simon (2006) only observed one peak during 100% PC hydration. It can be speculated since the binder only contains 10% cement, the cement grains quickly dissolve forming hydrated products such as C-S-H and portlandite (CH) as well as an increase in the alkalinity of the solution (Hill and Sharp, 2002). This corresponds to the drop in EC after the first maxima in the 100% binder specimen. The increased alkalinity of the pore solution and availability of CH activates the hydration of slag grains resulting in concentration of ions and the second peak at 6 hours in EC for 100% binder. In the 4.5 binder CPB specimen, double peaks are not observed but the EC plateaus at ~3.5 hours and rapidly decrease at ~8 hours. Similar reactions as described for 100% binder will occur in the 4.5% binder CPB but is not prominent in the EC graph due to the small amount of binder. The $\kappa'$ at 1 GHz for both specimens continue to decline at the same rate as before.
Stage 3: Acceleration period
Following the peaks in EC observed at ~ 6 hours and ~ 8 hours after initial mix for 100% binder and 4.5% binder CPB specimens, respectively, the EC rapidly decreases. This is indicative of the removal of ions in the pore solution due to consumption of ions to form hydrated products at an accelerated rate. The hydrated products build up on the solid particles forming a crystalline network and increasing the tortuosity of the paste. This further affects the conductivity as the ions will now have to traverse a more complex path to respond to the electric field. The sudden decrease corresponds to the initial setting of the paste. The $\kappa'$ at 1 GHz appears to abruptly decrease at this stage for the 100% binder where as for the 4.5% binder CPB specimen, the $\kappa'$ decreases at a slightly faster rate than previous stages. The $\kappa'$ at 70 MHz for 100% binder gradually increases implying that the state of free water is changing to bound water. This is more prominent for the 100% binder specimen than CPB with 4.5% binder. Similar trends were identified by Klein and Simon (2006) and Zhang et al. (1996).

Klein and Simon (2006) observed the onset of the acceleration stage in EC for PC at approximately 2 hours. For the 100% blended cement with 90% slag this stage occurred at approximately 6 hours. The retarding effect of slag is due to the decrease in cement content, especially C_3S which is responsible for early setting. This was also observed by Zhang et al. (1996). Simon and Grabinsky (2009) showed the retarding effect on hydration of cement pastes by fly ash (another supplementary cementing material) using EM methods.

Stage 4: Deceleration period
The deceleration stage is not observed in the 100% binder specimen due to the short duration (~ 10 hours) of the experiment to prevent permanent damage to the instruments. In the 4.5% binder CPB specimen, the slope of declining EC becomes less inclined at ~ 2.5 days indicating the slow continuous formation of C-S-H and other hydration products of the blended cements. No other peaks were identified at the deceleration stage.
Chapter 6: Validation and Discussion

Figure 6-5 Stages of hydration in specimen containing 100% binder
Stages of hydration: 1) Initial; 2) Induction; 3) Acceleration

Figure 6-6 Stages of hydration in CPB specimen with 4.5% binder
Stages of hydration: 1) Initial; 2) Induction; 3) Acceleration; 4) Deceleration
6.2.3. **Influence of sand aggregates**

Figure 6-7 compares the temporal response of electrical conductivity data for cemented paste backfill comprised of tailings mixtures with and without sand for 2.2% and 4.5% binder. The addition of sand aggregates lowers the peak in electrical conductivity from 1.2 dS/m to 0.9 dS/m and rapidly declines from ~6600 min to ~3200 min in the 2.2% binder CPB specimen. For the 4.5% binder, the EC is also lowered from 1.2 dS/m to 1 dS/m, and declines from ~1440 min to ~700 min. The particle packing density of tailings changes with the addition of sand. The tailings-sand mixture particles will have friction at many points of contact providing good interlocking and contain fewer voids. In addition, the hydration products of the blended cements will take less time to build up on these particles and form a rigid structure increasing the tortuosity of the pore spaces (Zhang et al., 1996). As a result, conductivity will decrease since the ions must now follow a more complex path to respond to the electric field (Klein and Wang, 2005). The implication of Figure 6-7 is that cement paste backfill comprising of tailings and sand aggregates will harden and gain strength prior to cemented tailings mixtures without sand.
6.2.4. **Influence of binder content**

Figure 6-8 shows the temporal response of CPB with 2.2% and 4.5% binder along with the components of CPB. Materials with no binder such as tailings, sand, process water and tailings-sand mixture show little to no change in electrical conductivity. However, with the addition of binder the EC maxima decline at ~3200 min and ~700 min for 2.2% and 4.5% binder CPB specimens, respectively. With higher binder content, more cement grains get dissolved and more hydrated products form around the tailings-sand mixture thereby decreasing the porosity of the medium earlier. As a result, the CPB mixture with higher binder sets prior to CPB containing lower binder.

![Figure 6-8 Temporal response of EC on CPB components and mixture](image)

6.2.5. **Influence of temperature**

The initial temperature observed at Stope 88 was 10°C higher than what was observed in Stope 67. In addition, due to the exothermic nature of the binder hydration reactions, the temperature in Stope 67 increased from 22°C to over 40°C and in Stope 88 from 33°C to over 50°C (Figure 4-13 and Figure 4-14). To investigate the effect of temperature and
simulate mine conditions, EM experiments were conducted on specimens placed in a drying oven set at ~40°C. Figure 6-9 compares the EM experiments conducted using capacitance probes at room and oven temperatures for CPB specimens containing 4.5% binder. The EC maximum for the specimen cured at the elevated temperature occurs prior to the specimen cured at room temperature. This trend was not as clear for the 2.2% binder CPB cured at 40°C, probably due to the very low binder content. With increasing temperature, the rate of hydration reactions in the binder increases (Fall and Samb, 2006). As a result, the hydration products in the CPB form more quickly compared to CPB cured at room temperature. Morsy (1999) and Heikal et al. (2005) noted the rate of hydration in cements increased at high curing temperature resulting in the shifting of the EC maxima to a shorter hydration time. Therefore, to conclude, the ECH₂O-TE probes reflect the changes in hydration of CPB due to increased temperature.

Figure 6-9 Temporal comparison of EC for CPB with 4.5% binder cured at 40°C and room temperature
Figure 6-10 Temporal comparison of EC for CPB with 2.2% binder cured at 40°C and room temperature
CHAPTER 7

CONCLUSION

7.1. Conclusion

This study demonstrated the in-situ and laboratory electromagnetic (EM) measurement techniques that can provide useful information about paste characteristics and about physicochemical changes that occur in the hydration of cemented paste backfill (CPB).

Temporal responses of EM parameters measured using commercially available, single frequency ECH2O-TE capacitance probes were used to study the hydration processes of CPB in the field and in the laboratory. A coaxial dielectric termination probe in conjunction with a broadband network analyzer was also used in the electromagnetic characterization of CPB in the laboratory. The capacitance probes provided information on the volumetric water content (VWC), EC and temperature and were used to obtain in-situ data from two test stopes in an underground mine. The field data was then compared with the laboratory data tested with the same model probes and the network analyzer.

In the field, the dielectric sensors of the capacitance probe appeared to be influenced by the temperature and did not provide useful data. A high degree of scatter was observed in the EC data for the initial days of monitoring, and may be attributed to contamination of the electrodes during installation. Despite the scatter, distinct trends in EC were identified for the different binder contents used in CPB at the mine (Figure 4-16 and Figure 4-17). The maximums in EC were observed to occur at approximately half a day and over 2 days for plug and main pour, respectively. The maximum in EC is identified corresponding to the onset of the acceleration stage in binder hydration reactions. The rise in temperature for the cages in the two stopes was identical despite the initial conditions in the stope (Figure 4-15). Elevated temperatures increase the rate of hydration reactions of the binder in CPB and were reflected in the field EC data (Figure 4-18 and
Figure 4-19). In the laboratory, the VWC data from the capacitance probes were consistent with the oven-dried VWC data and were not influenced by temperature (from Figure 5-8 to Figure 5-10). The trends in EC in laboratory prepared CPB were in good agreement with the trends observed in the field EC data (Figure 6-1 and Figure 6-2). The volumes of the samples used in the laboratory experiments were insufficient to compare the heat evolution arising from binder hydration. The VWC sensor of the probe is only accurate to ±3% and therefore, may not be useful for monitoring the changes in water content during CPB hydration as very little change in VWC can change the state of CPB from liquid to solid, i.e. greater precision is required for this application (Figure 5-5 and Figure 5-6). The electrodes of the probe which measure EC, however, provide useful information regarding the hydration of CPB. Therefore, the capacitance probe is a useful and relatively inexpensive instrument that can be used to monitor the physicochemical changes of CPB in the field or laboratory.

Variation in the spectral response of EM wave measurements provides information on the polarization mechanisms that take place during the hydration of the CPB (Figure 5-2). Two polarization mechanisms were identified within the 20 MHz to 1.3 GHz frequency spectrum: bound and free water polarization occurring at low MHz and over 1 GHz, respectively. For materials containing binder, the real relative permittivity at 1 GHz decreased while the real relative permittivity at 70 MHz increased slightly with hydration time indicating that the state of water is changing either due to the consumption of free water to form hydration products and/or water becoming bound to the solid particles.

Additionally, temporal variations in the effective electrical conductivity indicate changes in ion availability or mobility which can be correlated to identify the different stages of hydration (Figure 6-6). The factors that affect the electrical properties are binder content, material composition, temperature and the frequency of the electric field. As binder content in the paste increases, the water-to-binder ratio decreases resulting in the formation of more hydration products. The interconnectivity between the tailings and cement particles increases, i.e. porosity of the paste decreases. The hydration reactions are reflected in the electrical conductivity data which decline more quickly as the ions
have to travel a more tortuous path in an electric field (Figure 6-8). Similarly, addition of sand aggregates to the paste increases the inter-particle contacts in the paste lowering the porosity even further and increasing the strength (Figure 6-7). The CPB mixture at Kidd mine comprises tailings and sand aggregates mixed with 4.5% binder and 2.2% binder for the plug and main pour, respectively. At room temperature, the maximums in EC for 4.5% binder CPB occurred close to half a day while the maximum EC for CPB with 2.2% binder occurred a little over 2 days. The rate of the binder hydration reactions increases with increasing temperature and is evident in the EC data as the peak EC occurs prior to samples cured at room temperature (Figure 6-9 and Figure 6-10).

7.2. Significance of results

CPB systems are designed based on strength properties of paste prepared in laboratory environments. However, due to the high volume of CPB poured into the stopes and the exothermic reactions of binder, the curing temperature of in-situ CPB is significantly higher than laboratory conditions. The higher temperature increases the rate of hydration reactions in CPB and will achieve strength prior to samples cured at room temperature. Knowledge of in-situ initial and final set for different binder contents can be used to optimize CPB designs and reduce the mining cycle of each stope. Cement and other binding agents are a significant influence on cost performance of CPB systems and therefore backfill costs can be critically examined to identify potential cost savings.

The EM capacitance probes were just one of the instruments placed in the multi-transducer field program initiated by the university’s cemented paste backfill research group. The data from this study will be used to guide interpretation of data from other field instruments. Measurement of EM properties of CPB is just one of the methods to monitor the evolving properties of the paste. EM properties can be used in conjunction with other techniques such as heat evolution and elastic wave velocities to obtain an integrated interpretation of the hydration behaviour of binder used in CPB.
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