GYPSUM POWDER RECYCLED FROM WASTE DRYWALLS AS A PARTIAL CEMENT REPLACEMENT IN CONCRETE

by

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DEDICATION

I would like to dedicate this thesis to my mother, Kathy, who always encourages me to be my best and shows me immeasurable love and support.

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ABSTRACT

The ecological impact of the concrete industry can be reduced by re-using waste materials in the cementitious content, thereby decreasing the demand for virgin materials and reducing carbon emissions. In this study, recycled gypsum powder was used as a partial cement replacement, aiming to introduce a more sustainable and environmentally friendly alternative to traditional concrete while maintaining adequate strength and durability. The experimental research was divided into three main phases. Phase I included the fabrication and compressive strength testing of eight different batches of 50 mm cement mortar cubes. Phase II included a total of fifteen different concrete mixes cast into 200 mm x 100 mm cylindrical molds, prepared using 0-20% gypsum and 0, 25 and 50% fly ash as partial cement replacement, and tested for compressive strength at 7, 28 and 90 days. Partially replacing cement with increasing gypsum content decreased strength, however, incorporating fly ash with gypsum as partial cement replacement greatly improved the compressive strength at later ages. Increasing the gypsum content above 0% was beneficial to the 90-day strength of all concrete specimens with 50% fly ash. Phase III was designed to test the durability of a selected concrete mix with 15% gypsum and 50% fly ash in the cementitious material. The compressive strength of specimens’ subject to various dry and wet conditions (seawater and fresh water) was compared after exposure durations of 1000, 3000, and 5000 hours. Strength development throughout all durations indicated that concrete was durable to the tested conditions, with the largest strength increase observed in specimens exposed to wet/dry cycles in seawater. Presented research suggests that incorporating recycled gypsum in concrete is achievable from a structural perspective, and including fly ash is essential.

LIST OF ABBREVIATIONS AND SYMBOLS USED

Abbreviations

ASTM	American Society for Testing and Materials
°C	Degrees Celsius
C	Cement
CFFT	Concrete-filled fibre-reinforced polymer (FRP) tubes
CLSM	Controlled low strength materials
FA	Fly ash
FG	Fine gypsum
GU	General use
HVFA	High volume fly ash
lbs	Pounds
LOI	Loss of ignition
LP	Linear potentiometer
MPa	Megapascal
OPC	Ordinary Portland cement
SCM	Supplementary cementing material
W/C	Water to cement ratio
m	Meter
mm	Millimetre ($1\text{x}10^{-3}$ m)
µm	Micrometer ($1\text{x}10^{-6}$ m) (ie: micron)
nm	Nanometer ($1\text{x}10^{-9}$ m)

Chemical compounds

CO_2	Carbon dioxide
Al_2O_3	Aluminum oxide (Alumina)
BaO	Barium oxide
CaO	Calcium oxide
Cr_2O_3	Chromium (III) oxide
Fe_2O_3	Iron (III) oxide (Ferric oxide)

K_2O	Potassium oxide
MgO	Magnesium oxide
MnO	Manganese (II) oxide
Na_2O	Sodium oxide
P_2O_5	Phosphorus pentoxide
SiO_2	Silicon dioxide
SO_3	Sulfur trioxide (sulfite)
SO_4	Sulfate
SrO	Strontium oxide
TiO_2	Titanium dioxide
V_2O_5	Vanadium oxide
ZrO_2	Zirconium dioxide
$CaSO_4 \bullet 2H_2O$	Calcium sulfate di-hydrate (gypsum)
$Ca_6Al_2(SO_4)_3(OH)_{12} \bullet 26H_2O$	Calcium sulfoaluminate (ettringite)
$Ca_4Al_2O_6(SO_4) \bullet 14H_2O$	Monosulfoaluminate hydrate (monosulfate)
$CaOH_2$	Calcium hydroxide
$3CaO \bullet Al_2O_3$ (ie. C_3A)	Tricalcium aluminate
C_3S	Alite
C_2S	Belite
C_4AF	Calcium aluminoferrite (ferrite)

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CHAPTER 1 INTRODUCTION

1.1 GENERAL

Environmental impacts of cement production and waste accumulation have inspired the research of using industrial solid wastes as partial cement replacements in concrete. Fly ash is commonly used as a supplementary cementing material for its ecological and economic benefits, also demonstrating increased performance of concrete when used alongside gypsum (Puvvadi and Moghal 2011). Incorporating powdered recycled gypsum in concrete mixes reduces the carbon dioxide emissions by reducing the amount of cement required, providing a more sustainable solution while simultaneously helping to keep the material out of landfills, where it is known to cause very harmful reactions with its organic environment. Although gypsum is commonly used in cement in small percentages (3-5%), it is not currently considered as an acceptable supplementary cementing material in larger quantities according to the ASTM 'Standard Specification for Portland Cement' due to its high SO_3 content (Naik et al. 2010) (ASTM 2015). This study aims to challenge the standard by showing that acceptable compressive strength and durability can be obtained in concrete with additional gypsum. The focus of this study was on the structural aspects of incorporating recycled gypsum in concrete, including compressive strength results with varying cementitious contents and after exposure to different durability conditions.

1.2 RESEARCH OBJECTIVES

The objectives of this research are:

- To incorporate recycled gypsum as a partial cement replacement at higher proportions than typically used in effort to produce a more sustainable and ecofriendly alternative to traditional concrete.
- To investigate the compressive behavior of a multitude of concrete mixes containing varying combinations of gypsum, fly ash and cement as the cementitious material, aiming to determine a mix design that maintains adequate compressive strength.
- To examine the durability parameters when concrete is subject to certain environmental conditions.

1.3 THESIS STRUCTURE

This is a paper-based thesis that contains the following six chapters:

- Chapter 1 gives a general introduction to the research.
- Chapter 2 provides a literature review investigating the use of gypsum in general and its application in cement, including with fly ash as a supplementary cementing material and the chemical interactions involved. Environmental and waste management concerns are also discussed along with other relevant research.
- Chapter 3 presents Phase I of the research, including the application of gypsum in cement mortar cubes, which was accepted as a CSCE conference paper.
- Chapter 4 presents Phase II of the research, which is focused on testing cylindrical concrete specimens with numerous different mix designs for compressive strength.
- Chapter 5 presents Phase III of the research, comprising of testing one concrete mix design for durability under various environmental conditions.
- Chapter 6 concludes and summarizes the research, providing recommendations for future research.

1.4 REFERENCES

ASTM. 2015. "C150/150M Standard Specification for Portland Cement." *ASTM International.*

Naik, Turan, Rakesh Kumar, Yoon-moon Chun, and Rudolph Kraus. 2010. *Utilization of Powdered Gypsum-Wallboard in Concrete.* Milwaukee: University of Wisconsin.

Puvvadi, Sivapullaiah, and Arif Ali Baig Moghal. 2011. "Role of gypsum in the strength development of fly ashes with lime." *Journal of Materials in Civil Engineering* (ASCE) 23 (2): 197-206.

CHAPTER 2 LITERATURE REVIEW

Concrete is arguably the most important building material in the world, although it is known to have an enormous environmental impact (Meyer 2009). Since popularity of the concrete is unlikely to decrease, there is an evident demand to adapt it into a more sustainable and environmentally friendly material. Reducing the cement requirement of concrete would undoubtably reduce this environmental footprint, which is attainable with appropriate use of supplementary cementing materials. The use of fly ash is well established as a sustainable alternative in cement applications, and gypsum is known to be used in cement in small percentages. This literature review focuses on gypsum, including its general use, gypsum waste management, and most importantly its valuable role in the concrete industry with the potential to reduce harmful carbon emissions. Consideration was given to chemical interactions, and various relevant studies were reviewed in connection with the use of gypsum as a supplementary cementing material.

2.1 CONCRETE

Concrete is the most widely used engineering material in the world due to its many attractive characteristics, such as its general availability, affordability, mouldability, and excellent mechanical properties (Mehta and Monteiro 2014). However, typical concrete is not considered as an environmentally friendly material, leaving an enormous impact on the planet as billions of tons are produced worldwide every year (Meyer 2009). Concrete is most commonly made by mixing Portland cement with water, coarse aggregate (crushed rock/gravel) and fine aggregate (sand). High amounts of virgin materials need to be extracted and processed for concrete production, using substantial amounts of energy

(Helepciuc et al. 2017). Aggregates are typically sourced from local quarries, which demand energy for extracting, grinding and transportation. Though, in comparison to other materials required, Portland cement in inherently the most unsustainable and environmentally unfriendly as its production emits large amounts of carbon dioxide (CO_2).

Based on concrete's raw materials, there are three obvious opportunities to improve the environmental impact its production. First, reducing the large amount of water currently required, which especially burdensome in locations not fortunate enough to be in close proximity to abundant fresh water sources. Second, reducing the need to quarry virgin aggregate by substituting for various recycled materials. Finally, reducing the cement required by substituting with more sustainable cementitious materials. This is the prime option, as it is well known that the production of cement is very energy intensive and a major factor responsible for carbon emissions. Producing one ton of cement releases nearly one ton of carbon dioxide into our atmosphere (Meyer 2009). Nguyen et al. (2018) studied the life cycle assessment of ordinary Portland cement (OPC) along with five cement alternatives with comparable performance. These alternatives included blending geopolymers (fly ash, slag and metakaolin-based) with OPC, and results indicated that all OPC alternatives reduced the greenhouse gas emissions (Nguyen et al. 2018). Recent developments in the environmental sector have demanded more sustainable and carbon conscious construction practices. Since concrete is the highest consumed building material in the world, any small step in reducing its environmental footprint would have a considerable global impact.

The water to cement ratio (W/C) has the most significant influence on the permeability and therefore on the durability of concrete. Lower W/C decreases the porosity of the cement paste, making the concrete more impermeable, generally producing stronger and more durable concrete (Islam and Islam 2004). The American Concrete Institute (ACI) method for concrete mix design selects a W/C based on the desired compressive strength (ACI 2000).

2.2 GYPSUM

Natural gypsum is mined in every continent of the world, making it one of the most widely used minerals (Olson 2001). Also known as calcium sulfate dihydrate ($CaSO_4 \bullet 2H_2O$), it is mainly formed as a chemically precipitated sedimentary rock and mined in its raw form. A sample of gypsum in its natural (raw) form is shown in Figure 2-1, with a translucent crystalline structure and light grey/white color (University of Waterloo 2020). One of its earliest known uses was on the interiors of the great pyramids of Egypt, dating back to about 3000 B.C. (Sharpe and Cork 2006). Gypsum can also be generated synthetically as a byproduct of industrial processes, typically from coal-fired powerplants with flue-gas desulphurization systems (Olson 2001). In 2003, synthetic gypsum made up 26% of the total gypsum manufactured for wallboard in the United States (Sharpe and Cork 2006).

Figure 2-1 Natural gypsum (by the University of Waterloo 2020)

Different types of gypsum minerals can contain varying amounts of dihydrate ($CaSO_4 \bullet 2H_2O$), hemihydrate ($CaSO_4 \bullet \frac{1}{2} H_2O$), and anhydrite ($CaSO_4$), depending on varying conditions of heat, pressure and water presence in its environment (Chandara et al. 2009). These minerals can convert one form of calcium sulfate to another which can occur simultaneously with other forms in some gypsum deposits. Anhydrite ($CaSO_4$) is the anhydrous form of gypsum with no attached water molecules, which may exist as the primary depositional mineral or the product of deeply buried gypsum that has been dehydrated. Anhydrite is denser than gypsum and contains higher levels of soluble salts that cause impurities and fractures in gypsum deposits, formed in massive rocks or as a mixture of gypsum and anhydrite when partially hydrated. Gypsum, anhydrite or a gypsum-anhydrite blend may be used in the manufacturing process of Portland cement (Sharpe and Cork 2006).

2.2.1 Gypsum Mining in Canada

Canada is the third largest producer of gypsum worldwide, while the United States remains the largest producer and consumer of gypsum in the world. The majority of Canadian produced gypsum is exported to the United States, who receive approximately 97% of the total gypsum exported from Canada (Government of Canada 2017). Much of this raw gypsum is sent by ship to states along the eastern seaboard, such a Massachusetts, New Jersey, and New York. These three states combined receive more than 64% of the total gypsum exported into the United States from Canada (Government of Canada 2019).

Nova Scotia is Canada's leading gypsum producer, operating the world's largest gypsum quarry at Milford Station, about 50 kilometres outside of Halifax. The Milford quarry has been in operation since 1954 and is expected to stay functional for at least another 20 years (Mindat 2019). The open mine pit covers approximately 500 acres and is conveniently located near Canadian National's rail line, where trains are loaded with about 7000 tons of crushed material every day. The trains are sent to a port in Dartmouth, where ships are loaded and delivered to drywall processing plants in eastern United States (Spurlock 2004). The quarry rests on gypsum bedrock, covered in sediments dating back to over 200,000 years ago (Province of Nova Scotia 2014). Several glaciers passed over the quarry, covering the 76-metre thick gypsum rock supply with soil layers about 18 metres thick. To mine the gypsum, this overburden soil is removed, then miners drill holes in the gypsum rock and generate a series of smalls explosions. Controlled blasts loosen rock to later be loaded onto trucks and taken for further crushing and sorting (Spurlock 2004). The total energy required to extract, process and deliver the raw gypsum material is mostly accredited to the transportation to gypsum manufacturing sites (Recycling Council of Ontario 2006).

2.2.2 Other Uses for Gypsum

The predominant use for gypsum is drywall, commonly known as wallboard or gyprock walls. In order for gypsum ($CaSO_4 \bullet 2H_2O$) to be poured between two paper layers to create wallboard, it must first be calcined to become hemihydrate gypsum ($CaSO_4 \bullet \frac{1}{2} H_2O$). The hemihydrate gypsum is then ground into a powder, which is subsequently mixed with water to produce a slurry (Olson 2001). This powdered hemihydrate is commonly referred to as plaster of Paris and is formed after heating to 150°C (Chandara et al. 2009). Gypsum is a very versatile mineral with several other known markets. Firstly, it is widely used for its agricultural benefits, including soil fertility and beneficial changes in soil structure resulting in improved drainage and enhanced plant growth (Construction and Demolition Recycling Association 2019) (Batte and Forster 2015). Additionally, gypsum is used for ancient and modern architectural/artistic uses, animal bedding, medical casts, drugs, cosmetics, toothpaste and even as a food additive (Gratton and Beaudoin 2010) (Mentzer 2018) (Gypsum Association 2019) (Sharpe and Cork 2006).

2.2.3 Gypsum Use in Portland Cement

In order for concrete to maintain an adequate level of workability during mixing and placing, small amounts of gypsum are regularly used to control the initial hydration reaction. The manufacture of Portland cement incorporates about 3-5% calcium sulfates compounds, such as gypsum, as a set retarder (Naik et al. 2010) (Chandara et al. 2009) (Sharpe and Cork 2006). Portland cement is hydraulic and therefor stable in water, setting and hardening very quickly when hydrated. Gypsum on the other hand is non-hydraulic, as it does not harden in water and can be washed away (Chun et al. 2008).

2.2.3.1 Chemical Hydration of Concrete with Gypsum

The chemical name for gypsum is calcium sulfate di-hydrate, and its chemical formula is $CaSO_4 \bullet 2H_2O$. Without using gypsum or another calcium sulfate source as a set regulator in concrete, the Portland cement will set or harden too rapidly upon the addition of water and become unworkable (Barbosa et al. 2018). This is due to the reaction between the tricalcium aluminate ($3CaO \bullet Al_2O_3$ or C_3A) in cement and water, which immediately forms crystalline hydrates and must be slowed down to be used in construction applications. The calcium sulfate provided by gypsum has the capability to retard this reaction (Quennoz and Scrivener 2012). Cement and gypsum powder are considered anhydrous (without water), and the reaction with water is termed "hydration". This reaction is predominantly exothermic, generating a large amount of heat upon mixing. Multiple reactions occur between water and different compounds in the cement clinker at various reaction rates, so there are multiple phases to the hydration process, related to the existing minerals. Five main types of minerals are normally present in cement in the anhydrous state: tricalcium aluminate (C_3A), alite (C_3S), belite (C_2S), and calcium aluminoferrite phase (C_4AF), as well as gypsum (Mehta and Monteiro 2014). The first phase is the C_3A phase is the most reactive of the main cement minerals, and it occurs soon after mixing. The sulfate compounds of gypsum react with the calcium aluminate from cement to form short prismatic crystals of ettringite, or calcium sulfoaluminate ($Ca_6Al_2(SO_4)_3(OH)_{12} \bullet 26H_2O$) in the early stages after mixing. Ettringite formation is necessary to concrete hydration and is the mechanism that controls stiffening as it is dispersed within the cement paste at a microscopic level (Portland Cement Association 2001). The second stage is the dormant period, where concrete transitions from workable to stiff. Following the dormant period, typically lasting 2-3 hours, reactions begin to occur with the alite (C_3S), and belite (C_2S) in cement. Large crystals of calcium hydroxide ($CaOH_2$) and small calcium silicate hydrates start to fill the empty voids previously occupied by dissolving cement particles and water (Mehta and Monteiro 2014). This is the main hydration stage, where much of the concrete strength is developed and the gradual reaction of C_3A is continued. High early strength is largely dependent on the alite, whereas belite hydrates slower and contributes to strength development past one week. The ferrite (C_4AF) reaction phase also begins soon after water is added but slows down during hydration and does not significantly contribute to strength (Portland Cement Association 2001). Calcium silicate hydrate (C-S-H) is the main reaction product in concrete, formed continually when water reacts with calcium silicates in both alite and belite phases.

Depending on the alumina-to-sulfate ratio, the ettringite may become unstable and convert to monosulfoaluminate hydrate, or monosulfate ($Ca_4Al_2O_6(SO_4) \bullet 14H_2O$). This typically occurs when all the sulfates (gypsum) are consumed and the remaining C_3S continues to react with ettringite (Quennoz and Scrivener 2012). The noticeable difference between the chemical formulas of ettringite and monosulfate is the additional sulfate (SO_4) contained in ettringite. Ettringite crystallizes as short prismatic needles, and monosulfate crystallizes as hexagonal plates, taking up less space and typically occurring in later stages of hydration (Mehta and Monteiro 2014). Ettringite tends to hydrate first due to the initially high sulfate to alumina ratio, which changes with the depletion of sulfates from the gypsum and increasing concentration of aluminate ions from renewed hydration of C_3A and C_4AF.

Monosulfate takes about 8-11 days to form at 25°C (Christensen et al. 2004) and is a stable compound unless it is exposed to sulfates, commonly from soils and water. Sulfate exposure reforms monosulfate into ettringite, which is a very fast, expansive and damaging reaction (Christensen et al. 2004) (Wu and Naik 2002). Ettringite can be stabilized by the use of sufficient amounts of gypsum, and replacement of some cement by fly ash aids in reducing the amounts of free aluminate and calcium hydroxide (Naik et al. 2010) (Wu and Naik 2002). However, it is reported that if excessive amounts of calcium sulfate (such as gypsum) are present in cement, inordinate expansions can occur during hydration. The cause for this expansion is attributed to excessive sulfoaluminate formation after hardening that continues until the gypsum is depleted (Portland Cement Association 2001). For this reason, research considering cement with high amounts of gypsum is infrequently studied.

2.2.3.2 Previous Research Using Gypsum in Portland Cement

Naik et al. (2010) presented research on concrete including fly ash and elevated gypsum content closely relating the research presented in this thesis, so it was thoroughly reviewed. In the study, eight concrete mixes were prepared with cement replacements made for fly ash at 20-60% and recycled gypsum powder at 7, 10 and 20% replacement, by mass. Control mixes were also prepared using only Portland cement in the cementitious material. Sodium sulfate was used in select mixes to improve the early strength of concrete with blended fly ash. The mixture proportions used by Naik et al. (2010) are shown in Table 2-1.

The performance of concrete mixes was assessed for compressive strength development, and durability parameters including sulfate resistance and length change. Compressive strength tests showed that the blend containing 20% fly ash and 10% gypsum had the most promising results of all mixes. It developed similar strength to control groups at later ages (28 and 90 days), performing considerably better than the mix with only 10% gypsum replacement and no fly ash. This highlights the positive relationship established between gypsum and fly ash in concrete strength development. The researchers proved that concrete with 40 MPa at 28 days could be successfully made by blending powder gypsum (7-10%) and Class C fly ash (33-55%) in the cementitious material. Premature cracking was observed before 7 days on specimens made with 20% cement, 60% fly ash, 20% gypsum and 2% sodium sulfate due to excessive expansion. Length change of specimens was measured during immersion in saturated limewater, and the results are shown in Figure 2-2 (a) without sodium sulfate and Figure 2-2 (b) with sodium sulfate.

In reference to figures, it is apparent that mixtures with gypsum wallboard showed higher expansion. In Figure 2-2 (a), an excessively large expansion of 0.12% was observed in specimens with 20% fly ash and 20% gypsum. The mix containing 10% gypsum showed a net shrinkage of 0.035%, which is approximately equal to that observed in the control mix (0.036%). In Figure 2-2 (b), the concrete mix with 50% fly ash, 10% gypsum and 1% sodium sulfate showed a rather large expansion (0.043%) during immersion in limewater, although it shrunk almost the same amount during drying, resulting in a net expansion of only 0.006%. Other concrete mixtures with fly ash, gypsum and sodium sulfate showed similar length change to the control mix. Naik et al. (2010) identified this concrete mixture to be used to minimize drying shrinkage in concrete, thereby increasing durability by reducing drying shrinkage cracking. Significantly improved sulfate attack resistance was reported for the blend of 40% cement, 50% fly ash and 10% gypsum, compared to the control mix. Slump reported was between 25 and 55 mm for all specimens, with slightly reduced slump for mixes with gypsum (Naik et al. 2010).

Table 2-1 Proportions of powdered materials used for concrete mixtures (by Naik et al. 2010)

Mixture Designation	C-2	CN-2	CFN-2	CFN-3	C-4	CFNS-3	CFNS-4	CFNS-5
Laboratory mixture designation	Ref-3	New-7	New-8	New-9	Ref-5	New-14	New-15	New-16
Cement (mass % of Cm)	100	90	70	60	100	60	40	20
Fly Ash (mass % of Cm)	0	0	20	20	0	33	50	60
New Gypsum-Wallboard (mass % of Cm)	0	10	10	20	0	7	10	20
Sodium Sulfate (mass % of Cm)	0	0	0	0	0	1	1	2

Cm: Cementitious materials (Cement + Fly Ash + Gypsum-Wallboard).

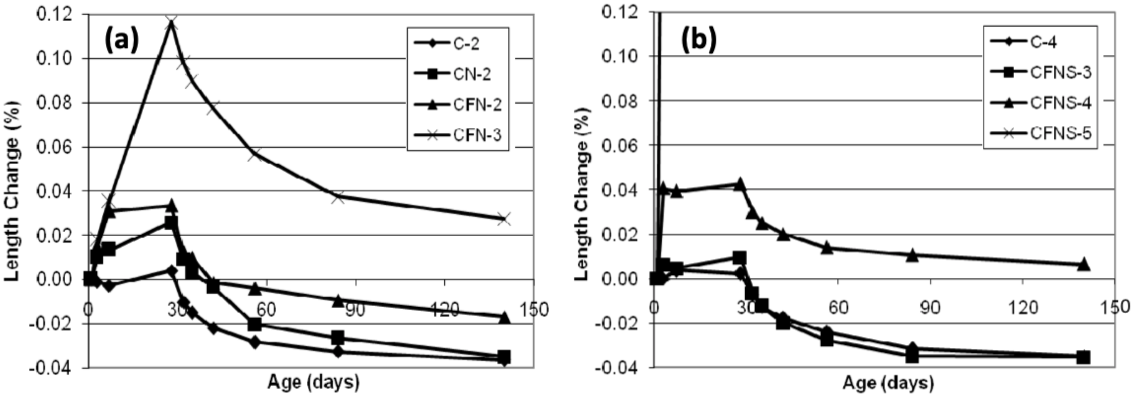

Figure 2-2 Length change concrete mixtures (a) without sodium sulfate; and (b) with sodium sulfate (by Naik et al. 2010)

Mohammed and Safiullah (2018) affirmed that optimum gypsum content is not fixed and varies from one cement to another. In this study, the effect of gypsum content between 0-9% on the strength properties of Algerian Portland cement (CEM I) was investigated. The results are displayed in Figure 2-3 for test ages of 2, 7 and 28 days. The optimum gypsum content was determined to be 5.5%, achieving increased compressive strength compared to all other mixes. This content also produced low values of swelling and drying shrinkage, and minimal heat of hydration. It was recognized that in order to achieve a normal mixing consistency, water demand is increased with the use of gypsum.

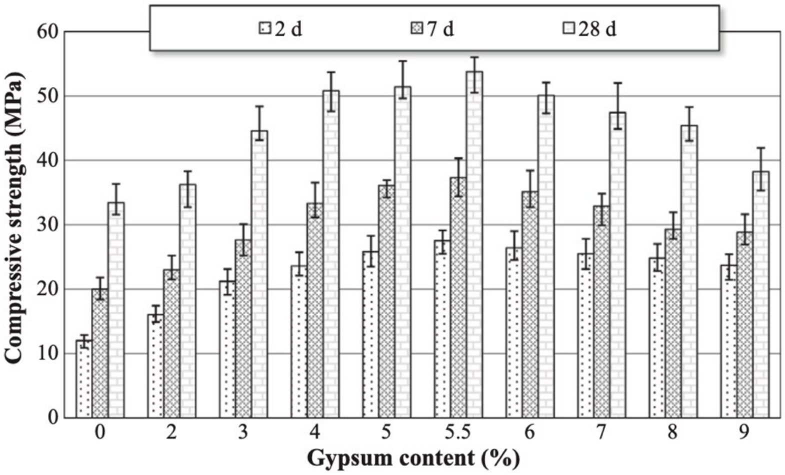

Figure 2-3 Effect of gypsum content on compressive strength (by Mohammed and Safiullah 2018)

Controlled low strength materials (CLSMs) using discarded gypsum wallboard and fly ash as supplementary cementing materials was investigated by Raghavendra and Udayashankar (2015). Quarry dust was used as the fine aggregate, making up half of the dry material by weight. The other half of the dry material was made up of the cementitious materials including cement (13-26%), powdered gypsum (52-61%), and fly ash (22-26%). These mortar mixtures were cast into 80 mm x 40 mm cylindrical molds to be tested in compression after air curing for 3, 7, 28 and 56 days. These were considerably low strength specimens, exhibiting compressive strength values ranging from 0.36 to 3.49 MPa. Materials used in CLSMs increased the typical water demand, and many specimens developed surface cracks at later ages. The highest compressive strengths were attained after 28 days, with reduced strength observed after 56 days. This type of CLSM may be suitable in applications where concrete placement is not permanent and re-excavation is necessary (Raghavendra and Udayashankar 2015).

Antunes et al. (2019) studied that feasibility of reusing gypsum waste in mortar, and concluded that up to 30% of ground gypsum waste can be included in mortar as an aggregate substitute. The mix design for this research had a 1:3 (cement: sand), and gypsum powder was substituted for sand in proportions of 0, 10, 20, and 30% by volume. The researchers considered the water absorption for each of the mix designs in terms of consistency index, and determined that the smooth gypsum particles increased the workability of mortar when compared to sand particles. Delayed curing was observed in specimens containing 10-30% gypsum, which is explained by the increased free water content around gypsum particles. Compressive and flexural strengths were tested at 14 and 28 days and the results are shown in Figure 2-4, with gypsum content along the horizontal axis. Figure 2-4 (a) shows that the cement continued to hydrate between test days, with larger compressive strength development observed in specimens with waste gypsum. Figure 2-4 (b) confirms that delayed curing occurs in samples with waste gypsum, as tensile strength develops significantly between test days with increasing gypsum content.

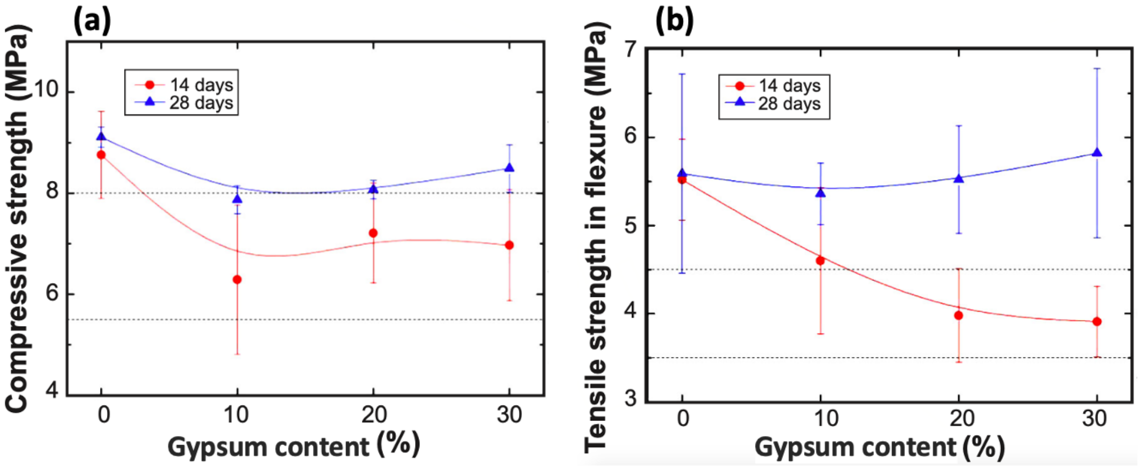

Figure 2-4 Effect of residue ratio on (a) compressive strength; and (b) tensile strength (by Antunes et al. 2019 – with modifications)

2.3 FLY ASH

Fly ash is a by-product of coal combustion in thermal power plants. Depending on the origin of the power plant, the physical and chemical properties of fly ash can vary substantially. The availability of fly ash has increased significantly since environmental regulations were put in place requiring industrial power plants to filter fine particles that were previously liberated into the atmosphere, causing negative environmental impacts (Meyer 2009). These widespread clean air regulations have allowed the concrete industry to profit from this industrial by-product for its advantages as a supplementary cementing material. Unfortunately, large amounts of fly ash still remain unused and are often disposed of directly into landfills, causing environmental pollution and substantial land occupation (Aprianti 2017) (Vargas and Halog 2015).

2.3.1 Fly Ash as a Supplementary Cementing Material

The use supplementary cementing materials (SCMs) is continually increasing due to the benefits from an economic, environmental, and sustainability viewpoint. Of these SCMs, fly ash is the most well-known and widely used as a partial replacement for Portland cement. It is generally less expensive than Portland cement, and has shown the ability to enhance some mechanical properties of concrete when effectively utilized. However, it is widely acknowledged that using fly ash can reduce the early strength in concrete (Vargas and Halog 2015) (Islam and Islam 2013) (Naik et al. 2010) (Wu and Naik 2002). Adding fly ash to a cement mix is likely to increase the workability and has been found to have a water reducing effect on mixtures, as well as slightly decreasing the unit weight while keeping the air content near constant (Puvvadi and Moghal 2011) (Marlay 2011).

Hardening of concrete with fly ash is mostly due to its pozzolanic reaction with calcium hydroxide provided by the cement, although some fly ashes can exhibit cementitious properties. This reaction tends to occur more slowly than typical concrete hydration, so more time for strength development is expected for mixes containing fly ash. Typical Portland cement has adequate oxides and aluminates to react when hydrated, however fly ash necessitates additional chemical activators (such as gypsum) in order to initiate hydration (Marlay 2011). Alumina is a principle component in fly ash, and its activation is based on its ability to react with the sulfate ions from gypsum (Aimin and Sarkar 1991). Hence, hydration of fly ash is improved in the presence of gypsum.

2.3.1.1 Previous Research Using Fly Ash as a Supplementary Cementing Material

Despite notoriously having low early strength, concrete with fly ash as a supplementary cementing material can be designed to exceed durability performance of conventional concrete, while meeting 28-day strength requirements (Bentz and Ferraris 2010). Prusinski and Carrasquillo (1995) reported that in concretes blended with fly ash and additional gypsum (4-5%), the cementitious material generally shows compressive strength that is comparable or improved compared to control mixes through 365 days. Marlay (2011) studied high volume fly ash (HVFA) mortar cubes specimens, using gypsum replacement at 4% due to the higher fly ash content. This proved to be an effective amount, integral in promoting necessary chemical reactions and augmenting early strength.

The effect of lime and gypsum contents, curing period, and several cycles of wetting and drying on the compressive strength of fly ashes was studied by Puvvadi and Moghal (2011). Fly ash was mixed with varying lime contents (1-10% by weight) and gypsum contents (1-2.5% by weight), noting that incorporating gypsum increases the strength of fly ashes at any lime content. This increased strength was not susceptible to repeated alternating cycles of wetting and drying, demonstrated by a minimal loss in strength in the presence of gypsum.

A study by Wu and Naik (2002) blended cements with a combination of Class C fly ash and clean coal ash, using sodium sulfate anhydrite as a chemical activator. Concretes blended with 40-60% coal combustion products showed equivalent or higher compressive strength at all ages. These blended cements also showed much higher resistance to sulfate attacks and alkali silica reactions (Wu and Naik 2002).

Islam and Islam (2013) studied the durability and strength characteristics of concrete made with a blended cement including Class F fly ash. Compressive strength was monitored for 365 days, comparing mixes using fly ash at 0, 10, 20, 30, 40, 50, 60 and 70% cement replacement. Researchers observed lower early strength in all mixes with fly ash, but after 56 days, the compressive strength in all specimens with up to 50% fly ash was higher than the control mix with 0% fly ash. They also reported that the permeability of concrete decreases with the increase of fly ash content up to an optimum value (determined to be 30%), and then starts to increase. Low permeability is desired as it controls the infiltration of water and other chemicals that may affect the durability of concrete. The rate of corrosion of reinforcing steel is limited with low permeability concretes, as the rate of oxygen diffusion is restricted (Islam and Islam 2013).

Cements blended with fly ash and bottom ash, known as geopolymer concretes, were experimentally studied by Xie and Ozbakkaloglu (2015) to determine the fresh concrete properties and mechanical properties. A multi-compound alkaline activator was used containing water, sodium hydroxide solution, and sodium silicate solution. It was reported that mixes with higher fly ash-to-bottom ash ratios exhibited higher strength and durability characteristics, as well as increased workability of fresh concrete. The bottom ash showed large, irregular shaped particles that remain unreacted, resulting higher drying shrinkage. The geopolymer concretes were cured at ambient temperature, and no evidence of exothermic reactions was observed, as is typical with ordinary Portland cement concrete (Xie and Ozbakkaloglu 2015).

As discussed, concrete mixes with HVFA tend to show delayed strength development, which can be a persistent problem in the field if early strength is necessary. However, many concrete structures are not loaded to their design values until several months after placement, such as in dams and heavy foundations, where slower setting times are acceptable (Meyer 2009). Mitigation of excessive hydration retardation caused by fly ash is possible, and was reported to be successful by Bentz and Ferraris (2010). Using calcium hydroxide powder and a rapid set cement (including gypsum), the setting time was significantly reduced. They employed gypsum in all mixtures containing Class C fly ash, as it was indicated that adding at least 2% gypsum was necessary to achieve normal hydration (Bentz and Ferraris 2010).

2.4 WASTE MANAGEMENT

Countless structures are demolished daily and the need to dispose of this rubble has continually been an inevitable burden. Much of construction waste generated can be reduced or recycled, producing benefits for both the environment and the construction industry (Teo and Loosemore 2001). Gypsum is extensively used in construction projects, making the disposal of gypsum wallboard waste a notable issue, reported to make up for 27% of all construction and demolition waste (Recycling Council of Ontario 2006).

Gypsum is successfully recycled around the world, with the ability to be re-used in almost all applications that use natural gypsum. Although unfortunately, large portions of gypsum waste are usually dumped at construction sites and ultimately transported to nearby landfills (Raghavendra and Udayashankar 2015). This is likely attributed to the absence of necessary incentives and resources to support gypsum recycling. For decades, landfills have provided a relatively convenient solution for waste disposal, however the need for more sustainable solutions is apparent with increasing environmental concerns. The application of recycled gypsum waste in concrete is therefore essential, as ready-mixed concrete plants are all over the map and have the ability to use gypsum waste in place of natural gypsum (Naik et al. 2010) (Chandara et al. 2009). This application would not only conserve natural gypsum deposits, but also help preserve valuable landfill space. It is likely that construction companies would be more inclined to recycle their gypsum waste if they had a direct re-use for it in concrete applications, especially at volumes larger than typically used in Portland cement.

2.4.1 Comparing Waste Gypsum and Natural Gypsum for Use in Concrete

A study by Chandara et al. (2009) compared the influence of waste gypsum to natural gypsum in ordinary Portland cement at contents of 3, 4 and 5% by weight of cementitious material. The waste gypsum was taken from a ceramic factory to represent other gypsum product waste such as drywall and plasterboard. The mechanical properties of mortar specimens were investigated in terms of compressive strength, flexural strength and setting time. The water required for each type of gypsum showed no significant difference, although cement with natural gypsum showed higher initial and final setting time when compared to cement with waste gypsum. This is attributed to the various ratios of calcium sulfate forms occurring in the gypsum sample; that is dihydrate ($CaSO_4 \bullet 2H_2O$), hemihydrate ($CaSO_4 \bullet \frac{1}{2} H_2O$) and anhydrite ($CaSO_4$). Dihydrate is the principal component in both natural and waste gypsum, though the hemihydrate component was found to be higher in waste gypsum (Chandara et al. 2009). This research affirms previous reports establishing that the presence of hemihydrate decreases the setting time of cement (Papageorgious et al. 2005). However, Chandara et al. (2009) showed that the setting rate had insignificant effect on compressive and flexural strength. Mortar specimens were tested at 2, 7 and 28 days, and showed negligible strength differences at all ages. Accordingly, waste gypsum can be used in place of natural gypsum in Portland cement clinker without sacrificing mechanical performance.

Suarez et al. (2015) evaluates the environmental impacts of production processes for natural (primary) gypsum compared to recycled (secondary) gypsum for use in the manufacture of Portland cement. Through life cycle assessment methodology, it was determined that recycled gypsum had numerous environmental benefits. It outperformed natural gypsum in all categories evaluated, including greenhouse gas emissions, land occupation, ozone layer depletion, mineral extraction, and carcinogenic effects. The study concluded that the gypsum recycling process emits less than 65% of the greenhouse gases that are produced when obtaining natural gypsum, and also consumes less than 65% of the energy needed to retrieve natural gypsum (Suarez et al. 2015).

2.4.2 Gypsum in Landfills

After many construction and demolition projects, gypsum drywall is often disposed of in landfills along with other waste. Depending on the civic regulations and available waste disposal services, drywall waste may be separated and disposed of more sustainably, however this is not very common and poorly monitored (Construction and Demolition Recycling Association 2019) (Rivero 2016) (Gratton and Beaudoin 2010). Gypsum waste is harmless when isolated, however it can become dangerous when mixed with organic waste in wet anaerobic conditions (Chandara et al. 2009). Certain moisture and temperature conditions can cause the sulfate portion of gypsum to react with other compounds and form high levels of hydrogen sulfide, which is harmful to humans and surrounding eco-systems. Hydrogen sulfide gas is flammable and has the smell of rotten eggs, known to cause serious health effects with human exposure. Sulfide can also be dissolved into the ground as leachates, consequently contaminating nearby water supplies (Raghavendra and Udayashankar 2015) (Naik et al. 2008) (Gratton and Beaudoin 2010). Leachate analysis done by Zhang et al. (2016) indicated that increasing the percentage of gypsum waste in landfills positively correlates to increasing sulfide levels. As the sulfate from gypsum drywall degrades in the landfill, it is also able to form complexes with other unstable metals or elements that are present, the most disturbing being arsenic due to its extreme toxicity (Zhang et al. 2016). Developing a safe and sustainable alternative for gypsum waste is therefore critical to help keep this material out of landfills.

2.5 OTHER RELEVANT RESEARCH

2.5.1 The Effect of SO_3 Content on Concrete

According to the ASTM Standard Specification for Portland Cement C150/C150M (ASTM 2015), a maximum of 3% or 3.5% SO_3 (sulfite) content is applied for general use concrete, with the higher value accepted when C_3A is more than 8%. The reason for this limit is referenced to be the excessive expansions related to elevated SO_3 content. It is permissible to exceed this limit if Test Method C1038/C1038M (ASTM 2019) is used to demonstrate that minimal expansion occurs. Many researchers have explored the influence of SO_3 content on concrete, which is relevant to the present research due to the high amounts of SO_3 contained in gypsum. Sulfite (SO_3) that undergoes oxidation transforms to sulfate (SO_4), and both forms are often present in Portland cements. Hanhan (2004) reported that cement clinker contains additional sulfates from the raw materials and products of fuel combustions. Concrete may also be exposed to sulfates through external sources such as ground water, soils, and seawater. These external sulfates may cause a sulfate attack, discussed in the following section.

The influence of SO_3 content on strength of 50 mm mortar cubes prepared using cement and fly ash was investigated by Chen et al. (2008). It was concluded that increasing the SO_3 content from 1.8% to 8.8% consistently decreased the compressive strength in cement mortar cubes. On the other hand, when the cementitious material of mortar cubes contained 71% fly ash and 29% cement, increasing SO_3 content steadily increased the 28-day compressive strength (Chen et al. 2008). This indicates that elevated SO_3 content is actually beneficial for HVFA concretes.

Hanhan (2004) studied the influence of SO_3 content of cement on the strength and durability when concrete is exposed to a sodium sulfate environment. Some cements were used as received from the manufacturer, and some had additional gypsum to increase the SO_3 content to 3.0% and 3.6%. The durability was determined by measuring the length change of mortar bars exposed to a sodium sulfate environment, and the compressive strengths was tested using mortar cubes. It was determined that optimum SO_3 content was not the same for both durability and strength parameters, and also differed from one cement to another, as well as from one age to another for the same cement. Typically, strength was optimized when SO_3 content was 3.0%, and expansion was increased for SO_3 content beyond 3.0% when C_3A content is low (3%). However, increasing SO_3 content to 3.6% did not increase expansion in cement with higher C_3A (above 7%) (Hanhan 2004). As mentioned, the C_3A phase is the most reactive during concrete hydration, and occurs soon after mixing. It is reported that cements with low percentages of C_3A are especially resistant to external sulfate attack (Portland Cement Association 2001).

Variable C_3A and SO_3 contents of heat-cured cement pastes was researched by Odler and Chen (1995) for the effect on expansion. It was concluded that the extent of expansion increased when outside water was supplied due to delayed ettringite formation. Additionally, increased expansion was reported for increased contents of both C_3A and SO_3, however the SO_3/C_3A ratio showed no effect on expansion (Odler and Chen 1995).

Horkoss et al. (2015) determined that the curing temperature had a significant effect on the expansion of mortars. Specimens with SO_3 sources from gypsum, basanite and high sulfate clinker all showed higher expansion when cured 80°C compared to 20°C, and developed microcracking at higher temperatures. Curing at 20°C, the sample with 3.37%

SO_3 content from gypsum showed expansion of less than 0.005% and 0.015% after 150 days and 660 days, respectively. Delayed ettringite was assumed to form in an expansive reaction due to water exposure, increasing temperature, and increasing SO_3 content.

Although experimental research has given credible explanations behind the microstructure of concrete hydration considering SO_3 content, its complete impacts are still not conclusively accepted, and contradicting test results have been reported (Chen et al. 2008) (Hanhan 2004). It is also noted that the above research was performed on mortars and does not consider any reactions between the paste and coarse aggregates.

2.5.1.1 Sulfate Attack

Sulfate attack was not investigated in this thesis, although some research suggests that having high fly ash and gypsum contents in concrete is beneficial for resistance to sulfate attack (Puvvadi and Moghal 2011) (Marlay 2011) (Prusinski and Carrasquillo 1995). Sulfate attacks are complex and can have numerous forms, all of which are still not fully comprehended. Expansive reactions are the primary response to sulfate attack, characterized by spalling and cracking of concrete. Sulfate exposure can be damaging to structures, with salt crystallization in pores often characterized by surface scaling accompanied by white efflorescence (Nehdi and Hayek 2004). During a chemical sulfate attack, sulfate (SO_4^{2-}) ions react with the hydration products of cement leading to the re-formation of ettringite and gypsum molecules. This additional sulfate is known to cause the formation of secondary ettringite, which is a harmful reaction leading to cracking and decreased structural performance (Vasavan 2017). High SO_4^{2-} concentration may cause ettringite to decompose to form gypsum, however, the disruption caused by gypsum is not well understood (Nehdi and Hayek 2004).

Research by Prusinski and Carrasquillo (1995) reported that using additional gypsum above the typical amount used in Portland cement significantly improved the sulfate resistance when combined with Class C fly ash. This is attributed to the abundant sulfate contained in gypsum that is able to react with all the C_3A and aluminates in concrete at early ages, so these reactions are not available to react with these aluminates at later ages (Prusinski and Carrasquillo 1995).

Two broad classes of sulfate attacks are categorized: external sulfate attack and internal sulfate attack. External sulfate attack often originates from surrounding sources like soils, seawater, wastewater, and ground water. Internal sulfate attack is caused by sulfate sources in the cement clinker, aggregates, water or admixtures. Alkaline-earth metals (calcium and magnesium) and alkali metals (sodium and potassium) are the sulfate compounds responsible for sulfate attack (Vasavan 2017). Research has often focused on the effect of SO_4^{2-} without much attention given to the associated cations. There is ongoing controversy as to the effect of each of these products on the deterioration of concrete, and the traditional view of sulfate attack has been challenged (Nehdi and Hayek 2004).

2.5.2 Particle Fineness

When size distribution and the influence of surface area were analyzed for gypsum particle sizes ranging from approximately 0.2-150 micrometers, it was found that the amount of water lost was essentially the same for all particle sizes (Barbosa et al. 2018). The hydration kinetics of the cementitious paste was altered by the fineness of gypsum, as fine particles reduce the dissolution time of gypsum. Particles with higher fineness were found to be consumed faster, displaying an accelerated rate of ettringite formation as well as exhibiting higher amounts of heat released.

Mehta and Monteiro (2014) report that the reaction between water and cement is also affected by the fineness of cement, with the rate of reactivity increasing with finer particle sizes. Particle sizes larger than 45 μm are slow to hydrate, and complete hydration may not occur in particle sizes larger than 75 μm. Finer particles have a higher surface area relative to their volume that would be in contact with water upon mixing, leading to a higher rate of hydration. The design of high-early strength Portland cement was developed corresponding to this knowledge, showing a shifted particle size distribution curve compared to normal strength Portland cement. Figure 2-5 presents particle size distribution data from ASTM Type I (normal) and Type III (high early strength) Portland cement (Mehta and Monteiro 2014).

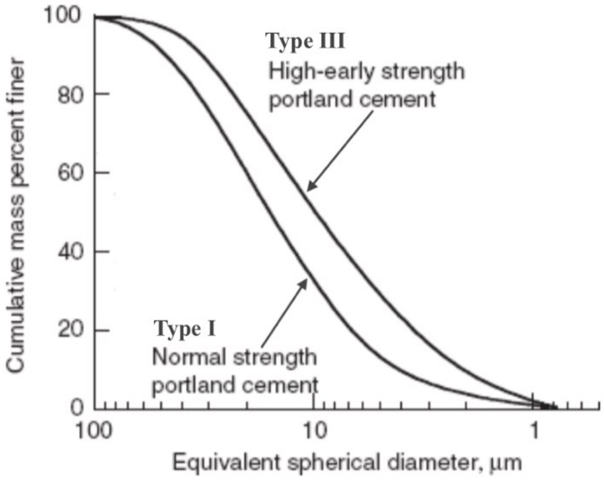

Figure 2-5 Typical particle size distributions data from ASTM Types I and III Portland cement samples (Kumar and Monteiro 2014 – with modifications)

2.5.3 Recycled Concrete as an Aggregate Replacement in Concrete

Using recycled materials in concrete is continually increasing as efforts are made to reduce the environmental impacts of the industry and reduce the accumulation of waste. This prompted the use of recycled concrete as an aggregate replacement in concrete, especially developing relevance after the Second World War. Nixon (1978) studied recycled concrete as an aggregate for concrete and concluded that implicating the recycled material leads to lower compressive and flexural strength. It is also acknowledged that using crushed concrete fines in new concrete increases absorption and therefore sharply increases the water requirement. Contamination by gypsum plaster was discussed as the source of sulfate impurity, identified to cause undesirable expansion in concrete. Less expansion was observed when the specimens were allowed to dry, or when larger particle sizes were used (Nixon 1978). The performance of concrete manufactured with 0, 50 and 100% recycled aggregate was investigated by Olorunsogo and Padayachee (2002) using durability indexes as indicators. They concluded that replacement with recycled aggregate reduced the durability quality for all indexes, including chloride conductivity, oxygen permeability and water sorptivity (Olorunsogo and Padayachee (2001).

Behera et al. (2014) reviewed more recent studies on the status of using recycled aggregate in concrete, concluding that inferior mechanical and durability performance was observed compared to conventional concrete, including reduced compressive and flexural strength. Drying shrinkage was identified as a very prominent feature, with significantly higher drying shrinkage occurring with increased recycled aggregate, likely due to the reduced modulus of elasticity. It is interesting to note that expansion was not reported as a problem in the comprehensive review. Techniques for improving the properties of recycled aggregate concrete were acknowledged, including incorporating mineral admixtures and modifications to the mixing process. Due to the inferior qualities of concrete with recycled aggregate, its application of is currently limited. Although due to its environmental benefits, research on this concrete type continues to advance in many countries (Behera et al. 2014)

2.5.4 Cement-Free Binders Using Fluorogypsum

Garg and Pundir (2016) were able to develop cement-free binders using fluorogypsum (96-99% by weight) ground with different chemical activators (1-4% by weight). Fluorogypsum has essentially the same chemical makeup of gypsum (calcium sulfate and water), but with 1-3% fluoride (F) present (Federal Highway Administration Research and Technology 2016). Strength enhancement was due to the formation of intermediate unstable salts, converting fluorogypsum into gypsum. Concrete specimens developed strength similar to cement, increasing continually in the hydration period with maximum strength attained at 28 days, the latest age tested. At 28 days, all specimens developed compressive strength of at least 15 MPa, with the highest strength attained (38 MPa) in the mix with anhydrous calcium chloride and sodium sulphate activators (Garg and Pundir 2016). This research conveys the possibility that using appropriate chemical activators with recycled gypsum in concrete could be a viable solution for cement replacement.

REFERENCES

Aimin, Xu, and Shondeep L. Sarkar. 1991. "Microstructural study of gypsum activated fly ash hydration in cement paste." Cement and Concrete Research (Pergamon Press plc.) 21: 1137-1147.

American Concrete Institute (ACI). 2000. *ACI Manual of Concrete Practice, Part 1: Materials and General Properties of Concrete.* Manual, Farmington Hills, MI: American Concrete Institute.

Aprianti, E. S. 2017. "A huge number of artificial waste material can be supplementary cementitious material (SCM) for concrete production - a review part II." Journal of Cleaner Production (142): 4178-4194.

Antunes, M. L., A. Botignon de Sa, P. S. Oliveira, and E. C. Rangel. 2019. "Utilization of gypsum from construction and demolition waste in Portland cement mortar." *Ceramica* 65 (1): 1-6.

ASTM. 2019. "C1038/C1038M - 19 Standard Test Method for Expansion of Hydraulic Cement Mortar Bars Stored in Water." *ASTM International.*

ASTM. 2015. "C150/150M - 15 Standard Specification for Portland Cement." *ASTM International.*

Barbosa, W., R. D'Paula Ramalho, and K.F. Portella. 2018. "Influence of gypsum fineness in the first hours of cement paste: hydration kinetics and rheological behaviour." Construction and Building Materials (Elsevier) (184): 304-310.

Batte, Marvin, and D. Lynn Forster. 2015. "Old is new again: the economics of agricultural gypsum use." Journal of ASFMRA 56-65.

Behera, Monalisa, S. K. Bhattacharyya, A. K. Minocha, R. Deoliya, and S. Maiti. 2014. "Recycled aggregate from C&D waste and its use in concrete - a breakthrough towards sustainability in construction sector: A review." *Construction and Building Materials* (Elsevier) 68: 501-516.

Bentz, Dale P., and Chiara F. Ferraris. 2010. "Rheology and setting of high volume fly ash mixtures." Cement and Concrete Composites 32: 265-270.

Chandara, Chea, Khairun Azizli, Zainal Ahmad, and Etsuo Sakai. 2009. "Use of waste gypsum to replace natural gypsum as set retarders in portland cement." Waste Management (Elsevier) (29): 1675-1679.

Chen, X., M.K. Zhou, P.L. Cong, and X. Li. 2008. "Influence of SO3 content on the strength of cement-fly ash stabilized crushed-stones." *4th Asian Regional Conference on Geosynthetics.* Shanghai, China. 398-402.

Christensen, A. Norlund, Torben R. Jensen, and Jonathan C. Hanson. 2004. "Formation of ettringite, Ca6Al2(SO4)3(OH)12-26H2O, AFt, and monosulfate, Ca4Al2O6(SO4)-14H2O, AFm-14, in hydrothermal hydration of Portland cement and of calcium aluminum oxide—calcium sulfate dihydrate mixtures studied by in situ synchrotron X-ray." Journal of Solid State Chemistry 177: 1944-1951.

Chun, Yoon-moon, Tarun Naik, and Rudolph Kraus. 2008. Recycling Opportunity for Gypsum Wallboard in Concrete. Solid waste reserach program, Milwaukee: University of Wisconsin.

Construction and Demolition Recycling Association (CDRA). 2019. Gypsum Drywall. CDRA. April 4. https://cdrecycling.org/materials/gypsum-drywall/.

Federal Highway Administration Research and Technology. 2016. User Guidelines for Waste and Byproduct Materials in Pavement Construction. U.S. Department of Transportation. 08 03. https://www.fhwa.dot.gov/publications/research/infrastructure/structures/97148/sw1.cfm.

Garg, Mridul, and Aakanksha Pundir. 2016. "Energy efficient cement free binder developed from industry waste- A sustainable approach." European Journal of Environmental and Civil Engineering 21 (5): 612-628.

Government of Canada. 2019. Statistics Canada Report - Trade Data Online. https://ic.gc.ca/app/scr/tdst/tdo/crtr.html;jsessionid=0001PxpZoj58tepyrBSIowTV7KM:2G83UKVV8F?lang=eng&hSelectedCodes=32742&searchType=BL&productType=NAICS&currency=CDN&runReport=true&grouped=GROUPED&toFromCountry=CDN&naArea=9999&countryList=SALL&reportTy.

Government of Canada. 2017. Total Canadian Exports - Trade Data Online. https://ic.gc.ca/app/scr/tdst/tdo/crtr.html;jsessionid=0001PxpZoj58tepyrBSIowTV7KM:2G83UKVV8F?lang=eng&hSelectedCodes=32742&searchType=BL&productType=NAICS&currency=CDN&runReport=true&grouped=GROUPED&toFromCountry=CDN&naArea=9999&timePeriod=CustomYears&re.

Gratton, Renee, and Guy Beaudoin. 2010. Gypsum Recycling in the National Capital Region. Ottawa: The Construction Recycling Initiative.

Gypsum Association. 2019. Other Uses of Gypsum. Gypsum Association. https://www.gypsum.org/about/gypsum-101/uses-gypsum/.

Hanhan, Amin A. 2004. *Influence of the SO3 content of cement on the durability and strength of concrete exposed to sodium sulfate environment.* Graduate Thesis and Dissertation, Department of Civil and Environmental Engineering, University of South Florida, Tampa, Florida: Scholar Commons.

Helepciuc, Catalina , Marinela Barbuta, and Dan Babor. 2017. "Investigations on a green concrete obtaining through a partial cement replacement by fly ash." AES Bioflux 9 (3): 201-214.

Horkoss, Sayed, Gilles Escadeillas, Toufic Rizk, and Roger Lteif. 2015. "The effect of the course of cement SO3 on the expansion of mortars." *Case studies in Construction Materials* (Elsevier) 4: 62-72.

Islam, M. M., and M.S. Islam. 2013. "Strength and durability chracteristics of concrete made with fly-ash blended cement." Australian Journal of Structural Engineering (Institution of Engineers Australia) 14 (3): 2204-2261.

Marlay, Kyle Marie. 2011. Hardened Concrete Properties and Durability Assessment of High Volume Fly Ash Concrete. Thesis, St. Louis: Missouri University of Science and Technology.

Mehta, P. Kumar, and Paulo J.M. Monteiro. 2014. "Concrete: microstructure, properties, and materials (Fourth edition)." (McGraw-Hill Education).

Mentzer, A.P. 2018. Uses for Gypsum Powder. Sciencing. November 19. https://sciencing.com/uses-gypsum-powder-6395376.html.

Meyer, C. 2009. "The greening of the concrete industry." Cement & Concrete Composites 31: 601-605.

Mindat. 2019. Milford quarry - National gypsum quarry - East Milford, Nova Scotia, Canada. Hudson Institute of Mineralogy. March 5. https://www.mindat.org/loc-267263.html.

Mohammed, Siline, and Omary Safiullah. 2018. "Optimization of the SO3 content of an Algerian Portland cement: Study on the effect of various amounts of gypsum on cement properties." Construction and Building Materials 164: 363-370.

Naik, Tarun, Rakesh Kumar, Yoon-moon Chun, and Rudolph Kraus. 2010. Utilization of Powdered Gypsum-Wallboard in Concrete. Milwaukee: UWM Center for By-Products Utilization-University of Wisconsin.

Nehdi, M., and M. Hayek. 2005. "Behavior of blended cement mortars exposed to sulfate solutions cycling in relative humidity." Cement and Concrete Research 35: 731-742.

Nguyen, Long, Alexander J. Moseson, Yaghoob Farnam, and Sabrina Spatari. 2018. "Effects of composition and transportation logistics on environmental energy and cost metrics for the production of alternative cementitious binders." *Journal of Cleaner Production* (Elsevier) 185: 628-645.

Nixon, P. J. 1978. Recycled Concrete as an Aggregate for Concrete - A Review. Great Britain: 37-DRC Committee (Demolition and Re-Use of Concrete).

Odler, I., and Y. Chen. 1995. "Effect of cement composition on the expansion of heat-cured cement pastes." *Cement and Concrete Research* (Elsevier) 25 (4): 853-862.

Olorunsogo, F. T., and N. Padayachee. 2002. "Performance of recycled aggregate concrete monitored by durability indexes." *Cement and Concrete Research* (Pergamon) (32): 179-185.

Olson, Donald. 2001. Gypsum. Washington: U.S. Geological Survey Minerals Yearbook.

Papageorgiou, A., G. Tzouvalas, and S. Tsimas. 2005. "Use or inorganic setting retarders in cement industry." Cement and Concrete composiites 27: 183-189.

Portland Cement Association. 2001. *Concrete Information - Ettringite formation and the performance of concrete.* Skokie, Illinois: Portland Cement Association (PCA).

Province of Nova Scotia. 2014. The East Milford Quarry Type Section in the Hants Lowlands. September 29. https://novascotia.ca/natr/meb/fieldtrip/stop3.asp.

Prusinski, J. R., and R. L. Carrasquillo. 1995. "Using medium to high-volume fly ash blended cements to improve the sulfate resistance of high-lime fly ash concrete." Fifth CANMET/ACI International Conference on Fly Ash, Silica Fume, Slag and Natural Pozzolans in Concrete. ACI SP-153. 43-66.

Puvvadi, Sivapullaiah, and Arif Ali Baig Moghal. 2011. "Role of gypsum in the strength development of fly ashes with lime." Journal of Materials in Civil Engineering (ASCE) 23 (2): 197-206.

Quennoz, Alexandra, and Karen Scrivener. 2012. "Hydration of C3A-gypsum system." Cement and Concrete Research (Elsevier) (42): 1032-1041.

Raghavendra, T., and B.C. Udayashankar. 2015. "Engineering properties of controlled low strength materials using flyash and waste gypsum wall boards." Construction and Building Materials 101: 548-557.

Recycling Council of Ontario . 2006. Let's Climb Another Mole Hill - An examination of construction, demolition and renovation (CRD) waste diversion in Canada and associated greenhouse gas emission impacts. Toronto, Ontario: Recycling Council of Ontario .

Rivero, Ana Jimenez. 2016. Gypsum Waste Management in the European Union: Towards a Circular Economy for the Construction Sector. Thesis, Madrid:

Polytechnic University of Madrid- Doctorate Program in Building Technology Innovation.

Sharpe, Roger, and Greg Cork. 2006. "Gypsum and Anhydrite." Industrial Minerals and Rocks (Society for Mining, Metallurgy, and Exploration, Inc.) (7th edition): 519-540.

Spurlock, Nancy. 2004. National Gypsum - Operations - Halifax treasure box supplies plants. https://ngc-heritage.com/op-halifax-quarry.htm.

Suarez, Sindy, Xavier Roca, and Santiago Gasso. 2016. "Product-specific life cycle assessment of recyced gypsum as a replacement for natural gypsum in ordinary Portland cement: application to the Spanish context." Journal of Cleaner Production (Elsevier) 117: 150-159.

Teo, M. M., and M. Loosemore. 2001. "A theory of waste behaviour in the construction industry." Construction Management and Economics (Routledge) 19 (7): 741-751.

University of Waterloo. 2020. *Eart Sciences Museum - Gypsum.* https://uwaterloo.ca/earth-sciences-museum/resources/detailed-rocks-and-minerals-articles/gypsum.

Vargas, J., and A. Halog. 2015. "Effective carbon emission reductions from using upgraded fly ash in the cement industry." Journal of Cleaner Production (Elsevier) (103): 948-959.

Vasavan, Manilal. 2017. "Sulfate attack and sulfate content in concrete: a literature review." *Geology to Concrete Technology.*

Wu, Zichao, and Tarun Naik. 2002. "Properties of concrete produced from multicomponent blended cements." Cement and Concrete Research (Elsevier) 32: 1937-1942.

Xie, Tianyu, and Togay Ozbakkaloglu. 2015. "Behavior of low-calcium fly and bottom ash-based geopolymer concrete cured at ambient temperature." Ceramics International 41: 5945-5958.

Zhang, Jianye, Hwidong Kim, Brajesh Dubey, and Timothy Townsend. 2016. "Arsenic leaching and speciation in C&D debris landfills and the relationship with gypsum drywall content." Waste Management 59 (1): 324-329.

CHAPTER 3 APPLICATION OF RECYCLED GYPSUM WALLBOARDS IN CEMENT MORTAR[1]

ABSTRACT

Gypsum is a naturally formed mineral that is already known to be added to cement at small percentages in order to reduce the speed of reaction with water, however it seems that substantial technical research has not been done concerning larger proportions of gypsum. The primary objective of this study is to use recycled wallboard/drywall powder (hereafter called gypsum) as a partial replacement for cement in cement mortar mixtures to introduce a more sustainable and environmentally friendly solution that lowers carbon dioxide (CO_2) emissions by using recycled materials, while maintaining adequate strength and durability. Used gypsum wallboard is often sent to landfills instead of being recycled, which can cause leachates with harmful environmental and health effects. Eight mixtures containing different combinations of cementitious material including cement, gypsum and fly ash were mixed with water and fine aggregates and placed in 50 mm mortar cube molds. After curing in a moist room, the mortar cubes were tested for compressive strength at the age of 3, 7, 28, and 56 days. Superplasticizers were used to regulate mixture consistency, as adding gypsum was found to dehydrate the mixture. Fly ash was also used, though requiring a longer initial setting time than cement. This study showed that mixtures containing only recycled gypsum and cement showed lower compressive strength at all ages, becoming increasingly weak with increased proportions of gypsum. However,

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combining gypsum and fly ash as partial replacement for cementitious material showed increased compressive strength, especially at later ages.

3.1 INTRODUCTION

It is well known that the production of cement releases large amounts of CO_2 into the atmosphere, causing negative impacts on our environment and contributing to global warming. Using a recycled material decreases the demand on producing virgin materials, ultimately reducing the CO_2 emissions, saving precious landfill space and keeping landfill sites unharmed. The construction industry produces roughly 33% of all solid waste in North America, with gypsum wallboard product comprising about 15% of this waste (Gratton and Beaudoin 2010). Currently, the majority of wallboard waste is disposed of in landfills, which is unsustainable and harmful to the surrounding environment. When exposed to rain and organic waste, landfill sites containing large volumes of gypsum have been shown to produce significant levels of hydrogen sulfide. This sulfide can be absorbed into the leachates or released into the air as a dangerous, flammable gas (Gratton and Beaudoin 2010). When absorbed into the leachates, the groundwater and consequently nearby ecosystems are vulnerable to its harmful effects. As the sulfate contained in gypsum drywall degrades in the landfill, it is also able to form complexes with other unstable metals or elements that are present in landfills, the most concerning being arsenic due to its high toxicity (Zhang et al. 2016). If the ability to use increased volumes of gypsum in cement is discovered to be successful, construction companies would likely be more inclined to recycle their gypsum waste to then be used in concrete applications, instead of producing or purchasing more. Having a sustainable use for recycled gypsum in concrete provides us with a safe and sustainable alternative by keeping the material out of landfills and decreasing CO_2 emissions during production, consequently lowering our environmental footprint.

Suarez et al. (2016) used life cycle assessment methodology to evaluate the environmental impacts of using natural and recycled gypsum in Portland cement production, but consideration was not given to the mechanical properties of concrete. A study by Naik et al. (2010) reported that replacing cement with up to 10% powdered gypsum wallboard did not adversely affect the properties of concrete, and better performance was observed when blending Class C fly ash and powdered gypsum with cement. Mineral additives such as fly ash have been reported to greatly enhance the durability of concrete and resistance to environmental impacts, as well as providing economic and ecological benefits. However, it is widely acknowledged that these by-products can reduce early strength of concrete (Wu and Naik 2002). Marlay (2011) considered that additional gypsum may be required to promote more desirable chemical reactions during concrete hydration when fly ash is used for cement replacement in high volumes. With few research activities concerning the effect of recycled gypsum material from drywalls in concrete, its effect is generally not well-known. This paper reports the results of a preliminary study on the effects of recycled gypsum for use as partial replacement for cement in mortar cubes under compression.

3.2 EXPERIMENTAL PROGRAM

3.2.1 Test Matrix

The main test matrix consisted of five batches of mortar with varying proportions of gypsum for partial replacement by weight for cement, including a control mix containing no recycled gypsum. Additionally, three mixes containing fly ash were prepared and tested under compressive loading. The quantity of sand (2914.8 g) and water (514 mL) was kept constant in each batch. To keep the consistency of the mix to a relatively similar level of workability, variable amounts of superplasticizer were added during mixing. The specimen's batch proportions of are shown in Table 3-1.

Table 3-1 Test matrix and material quantities

Specimen Group ID	Gypsum Content (%)	Fly Ash Content (%)	Cement Content (%)	Gypsum (g)	Fly Ash (g)	Cement (g)	Super-plasticizer (mL)
0	0	0	100	0	0	1060.6	0
1G	10	0	90	106.2	0	954.3	1
2G	20	0	80	211.8	0	848.3	1.5
3G	30	0	70	317.9	0	741.8	2.5
4G	40	0	60	424.4	0	635.9	3.5
5F	0	50	50	0	530.1	530.2	3
25GF	25	25	50	265.1	265.4	265.4	1
1G4F	10	40	50	106.3	424.1	424.1	3

3.2.2 Material Properties

In this research, the gypsum was used as is from a drywall waste recycling company (USA Gypsum, Denver, PA, USA) that processes the material into an ultra-fine consistency with particle sizes ranging from 1/8 in. (3.175 mm) to dust. The cement used in all mixes was Type GU Portland Cement (CRH Canada Group, ON, Canada). Fly ash was available in the lab (Dalhousie University, NS, Canada). The sand was locally sourced (Casey Metro, Halifax, NS, Canada) and was used in air dried condition, so a level of moisture content was accepted. The gypsum and sand were both put through a sieve analysis to determine the particle size distribution curves, shown in Figure 3-1.

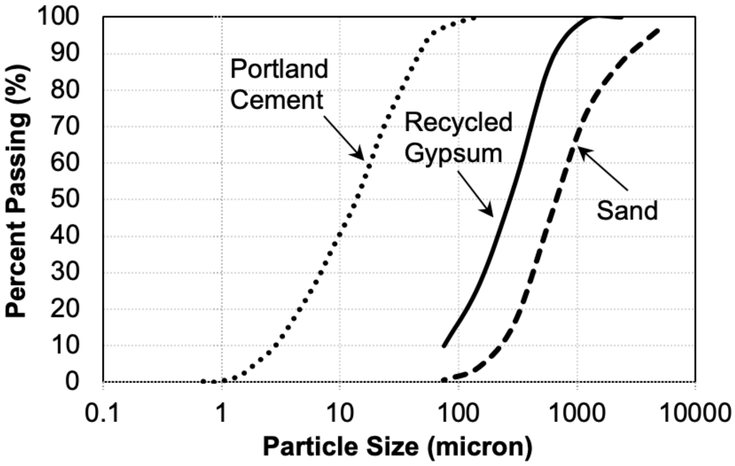

Figure 3-1 Particle size distribution of gypsum, sand and Portland cement. Note: Portland cement data retrieved from Sata et al. (2010)

During the sieve analysis, it was discovered that light-weight fibre-like particles attached together to create small bunches or clusters of material. To produce drywall, two outer sheets of paper contain the gypsum plaster, so it is assumed that these particles are made of paper that remained during the recycling process, however the actual chemical composition is unknown. These bunches tend to be larger and more loosely attached on the smaller number sieves (with larger openings), and more frequent and more tightly packed as the sieve size raises. Passed the No. 100 sieve, these clusters were no longer noticeable. Photos of various sieves retaining the recycled gypsum material are shown in Figure 3-2, including these particle bunches. These particles were only discovered after multiple mixes were cast and were therefore continued to be used in all mortar mixes, potentially causing decreased strength.

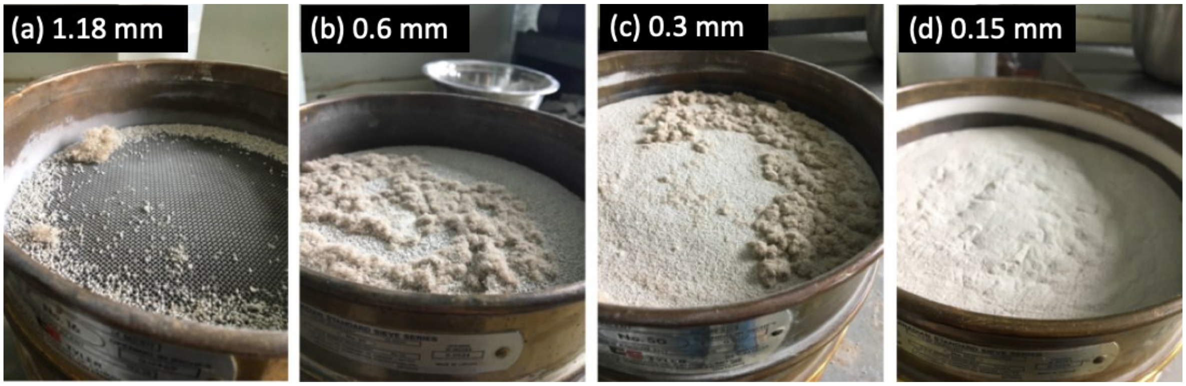

Figure 3-2 Gypsum retained of sieves (a) No. 16 (b) No. 30 (c) No. 50 (d) No. 100

3.2.3 Specimen Preparation

To prepare the specimens, ASTM C109 (2016) was followed for each of the mix designs. All material was slowly added to a tabletop electric mixer and then allowed to mix for 2-3 minutes until a uniform texture was accomplished. Adding gypsum to mortar, even at only 10% of cementitious material, was found to dehydrate the mix and reduce workability. For this reason, the researcher visually and physically assessed each mix and decided whether or not to add superplasticizer based on the workability of the mortar in comparison to the control mix. If needed, superplasticizer was added to the mixer in increments of 0.5 mL using a syringe, evenly distributed and allowed to mix for another minute or so. The consistency of the mortar mix was then assessed again, and the process described above was repeated as necessary. After mixing, the 50 mm cube specimen molds, shown in Figure 3-3, were filled and hand tamped as required by the standard. The molds were removed after 24 hours and the specimens were then labelled and moved to a moist closet for curing.

Figure 3-3 Cube specimen molds

3.2.4 Test Setup and Instrumentation

Specimens were tested promptly after removal from the moist closet and tested at specified ages. The procedure for the determination of compressive strength was in accordance with ASTM C109 (2016). The compression testing machine is shown in Figure 3-4, where the maximum load is measured in pounds (lbs).

Figure 3-4 Compression testing machine

3.3 RESULTS AND DISCUSSION

3.3.1 Compressive Behaviour

Specimens were tested under compressive loading after curing for 3, 7, 28 and 56 days. The average compressive strength was calculated by first converting the measured load from pounds (lbs) to newtons (N), and then to megapascal (MPa). Table 3-2 provides the average results for compressive strength of specimens tested, including the coefficient of variation (CV) taken from 3 mortar cubes per test.

Table 3-2 Summary of compressive strength results

ID	Day 3		Day 7		Day 28		Day 56	
	Average Strength (MPa)	CV (%)	Average Strength (MPa)	CV (%)	Average Strength (MPa)	CV (%)	Average Strength (MPa)	CV (%)
0	26.99	3.82	37.96	5.40	41.22	17.81	52.49	16.17
1	25.5	2.00	28.17	4.83	27.73	3.35	30.1	2.26
2	14.23	6.25	17.2	11.92	24.76	2.75	24.91	6.42
3	13.34	6.67	16.61	8.19	21.5	3.16	21.2	3.21
4	13.05	21.92	12.45	46.83	11.12	25.00	14.68	50.07
5F	20.31	10.78	26.84	0.97	55.75	16.61	51.15	24.50
25GF	8.16	8.33	18.09	9.29	43.59	2.71	49.52	12.74
1G4F	13.2	16.59	26.69	8.80	41.81	5.62	41.96	6.82

Typically, specimens subject to compressive loading failed by breaking into an hourglass shape with larger pieces falling around the centre of faces not in contact with a surface. However, the specimens containing 40% gypsum broke more inconsistently and into smaller pieces that crumbled apart much easier than other specimens. This may be caused by the unknown fibre-like particles that were discovered in the gypsum material during the sieve analysis. The failure modes of various specimens after compression testing are shown in Figure 3-5.

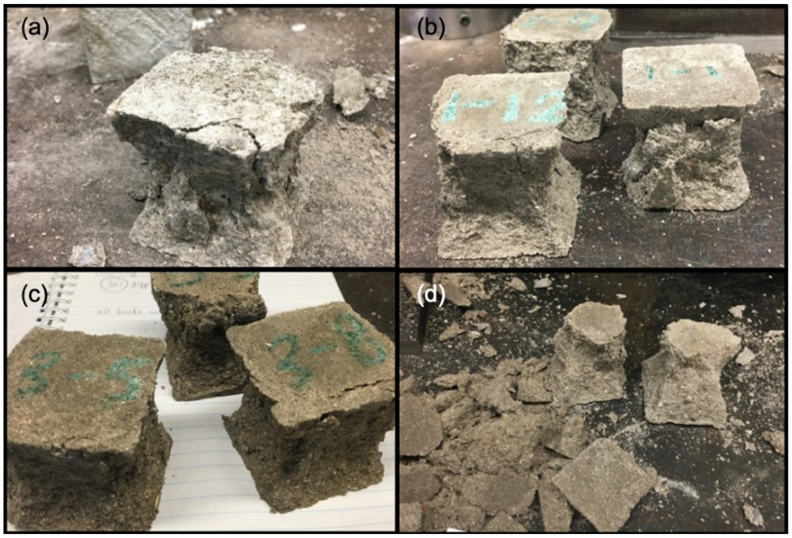

Figure 3-5 Compression testing on specimens (a) 0; (b) G1; (c) G3; (d) G4;

3.3.2 Effect of Gypsum Content

It was detected that gypsum has a lower density in mortar mixes when compared to cement, as the specimens' weight continually decreased with increasing gypsum content. The results shown in Table 3-3 are averaged from 3 cubes weighed on day 56, with the bottom row indicating the average difference (decrease) in weight compared to the control batch which contained no additional gypsum.

Table 3-3 Cube weight varying with gypsum content

Gypsum content (%)	**0**	**10**	**20**	**30**	**40**
Average weight (g)	300.30	289.47	287.93	286.83	278.30
Decrease from control (g)	0	10.83	12.37	13.47	22.00
Decrease from control (%)	0	3.74	4.30	4.70	7.91

Replacing varying percentages of cement with recycled gypsum powder as the cementitious material of the mix was generally found to decrease the compressive strength of specimens. Figure 3-6 shows a comparison of the compressive strength at each test day in relation to the gypsum content, including error bars representing the standard deviation between test specimens. As the gypsum content increased, a continuous decrease in the average compressive strength was observed at all ages in comparison to the control batch. The largest decrease in strength was observed between 0-10%, and noticeably smaller variations were seen for batches containing 20% and 30% gypsum. When the cementitious material contained 40% gypsum, test results were inconsistent with large error bars, and no significant strength gain was developed passed day 3.

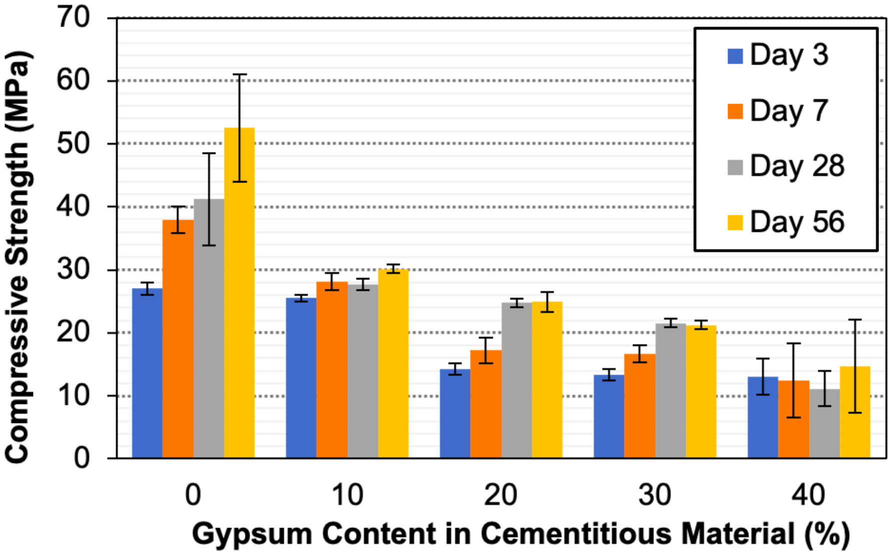

Figure 3-6 Compressive strength with varying gypsum content

Figure 3-6 was split up into separate graphs for each day in order to determine suitable trendlines for the compressive strength with increasing gypsum content. After assessing each of the individual graphs, the trends are generally best described by a negative logarithmic distribution. Table 3-4 highlights the results of the trendline analysis, including the trendline equation and coefficient of variation (R^2) found for each test day. It is accepted that an R^2 value equal to 1 indicates the best fit of the data to the model, so the model is least suitable at day 3. It was also determined that specimens allowed to cure for longer time periods showed a larger decrease in strength in comparison to the control batch. This can be seen as the negative slope of the logarithmic trendline continually decreasing from day 3 through 56. The x-value designates the gypsum content in cementitious material (%), and the y-value designates the compressive strength (MPa).

Table 3-4 Trendline analysis at each test day

Trendline	**Day 3**	**Day 7**	**Day 28**	**Day 56**
Equation	$y = -10.12\ln(x) +28.31$	$y = -16.17\ln(x) +37.96$	$y = -16.61\ln(x) +41.17$	$y = -22.31\ln(x) +50.04$
R^2 value	0.846	0.972	0.938	0.963

3.3.3 Effect of Fly Ash

Three batches of mortar were mixed using fly ash as partial replacement in the cementitious material, one including only cement (5F), and two also including recycled gypsum powder (25GF and 1G4F). The average results from the compression tests for these mixes are presented in Figure 3-7. Significant strength development was observed between 7 and 28 days, however the strength development between 28 and 56 days was variable and considered insignificant. This differs from mixes containing only gypsum and cement shown in Figure 3-6, where the strength gain between test days was less significant and less variable.

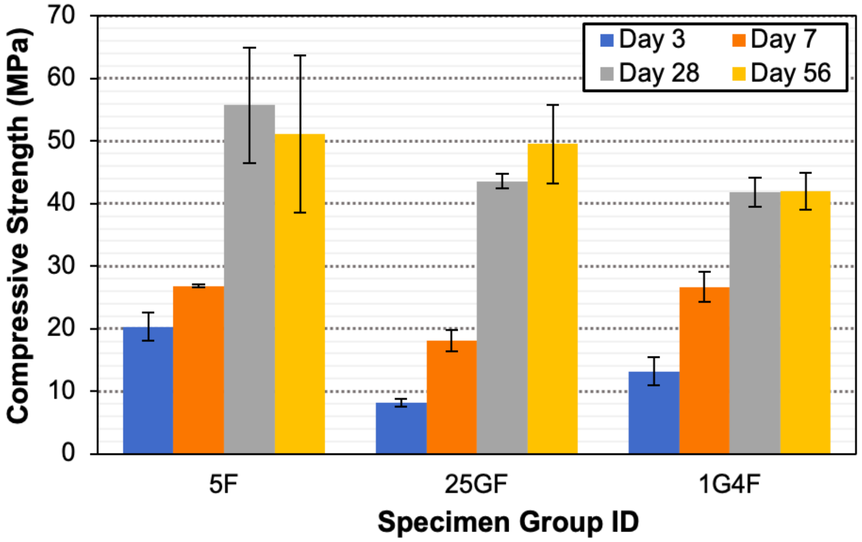

Figure 3-7 Compressive strength with varying gypsum and fly ash content

In order to compare the strength development of mixes containing fly ash to those without fly ash, Figure 3-8 was developed. Figure 3-8 (a) compares test results of mix “1G” containing 10% gypsum and 90% cement to mix “1G4F” containing 10% gypsum, 40% fly ash and 50% cement. Figure 3-8 (b) used the average test results of mixes “2G” and “3G” to create “25G”, hypothetically containing 25% gypsum and 75% cement, to compare with mix “25GF” which contains 25% gypsum, 25% fly ash and 50% cement. These graphs show that mixing gypsum with fly ash and cement yields smaller compressive strengths at day 3, comparable results at day 7, and noticeably higher strengths after longer curation periods of 28 and 56 days. It is therefore concluded that mixes containing fly ash require longer time periods to develop compressive strength.

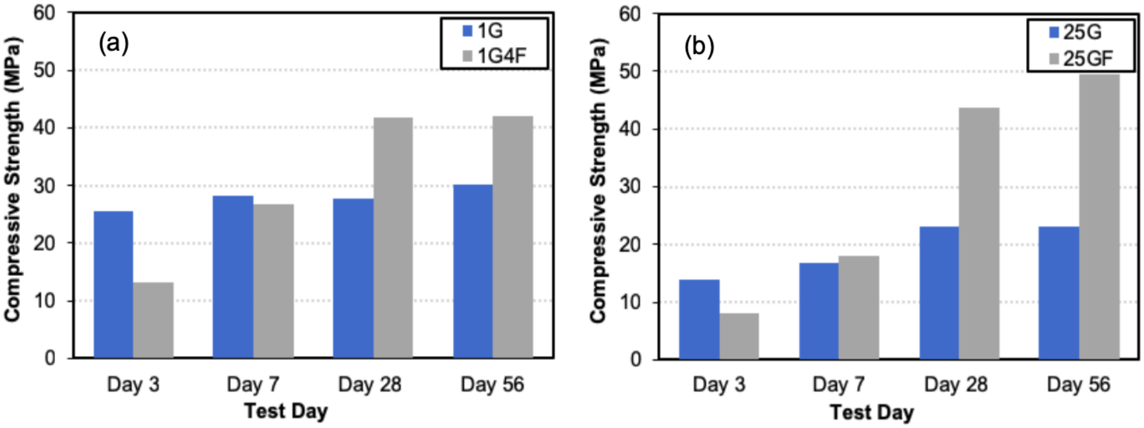

Figure 3-8 Compressive strength comparison with fly ash

3.4 CONCLUSIONS

In this study, eight batches of cement mortar were cast in 50 mm cubes to be tested under compressive loading after 3, 7, 28 and 56 days of curing. The cementitious content of these batches included one control batch with only cement, four batches with recycled gypsum drywall powder replacing cement at 10, 20, 30 and 40%, one batch containing only cement and fly ash, and two batches containing cement, gypsum and fly ash. The results show that partially replacing cement with gypsum yields lighter weight specimens and consistently decreases the compressive strength at all ages in a negative logarithmic trend from 0-40% gypsum replacement. The deviation from the control mix was seen to continually increase with age, with the largest deviation at 56 days. Incorporating fly ash into the mixes with gypsum improved the compressive strength, especially at later ages. This study will be continued by further investigating the effects of recycled gypsum in concrete cylinders to find an optimal design mix. Heavier consideration will be given to mixes containing fly ash due to its positive reaction with gypsum causing increased compressive strength, and less consideration will be given to mixes containing only cement with higher percentages of gypsum as partial replacement.

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REFERENCES

ASTM. 2016. *C109/C109M – 16 Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in or [50-mm] Cube Specimens)*, ASTM International, West Conshohocken, PA, USA.

Gratton, R. and Beaudoin, G. 2010. *Gypsum Recycling in the National Capital Region,* The Construction Recycling Initiative (CRI) – National Capital Region, Ottawa, Ontario, Canada.

Marlay, K. 2011. *Thesis: Hardened concrete properties and durability assessment of high volume fly ash concrete.* Missouri University of Science and Technology, St. Louis, MO, USA.

Naik, T., Kumar, R., Chun, Y., Kraus, R. 2010. *Utilization of Powdered Gypsum-Wallboard in Concrete.* University of Wisconsin, Milwaukee, WI, USA.

Sata, V., Jaturapitakkul, C., Rattanashotinunt, C. 2010. Compressive Strength and Heat Evolution of Concretes Containing Palm Oil Fuel Ash. *Journal of Materials in Civil Engineering.* **22**(10): 1033-1038.

Suarez, S., Roca, X., Gasson, S. 2016. Product-specific life cycle assessment of recycled gypsum as a replacement for natural gypsum in ordinary Portland cement: application to the Spanish context. *Journal of Cleaner Production.* **117**: 150-159.

Wu, Z. and Naik, T. 2002. Properties of concrete produced from multicomponent blended cements. *Cement and Concrete Research.* **32**: 1937-1942.

Zhang, J., Kim, H., Dubey, B., Townsend, T. 2017. Arsenic leaching and speciation in C&D debris landfills and the relationship with gypsum drywall content. *Waste Management*. **59:** 324-329.

CHAPTER 4 RECYCLED GYPSUM FOR PARTIAL CEMENT REPLACEMENT IN CONCRETE[2]

ABSTRACT

The concrete industry is known to leave a massive environmental footprint on our planet, which can be reduced by re-using waste materials in the cementitious content, thereby decreasing cement content. Gypsum wallboards or drywalls used in the building industry are a major source of construction and demolition waste. Gypsum drywall waste contains a valuable mineral capable of being effectively recycled, however it is often disposed of in landfills, where it can cause adverse reactions in its environment. In this experimental study, recycled gypsum powder (hereafter called gypsum) obtained from waste drywalls is used to partially replace cement in concrete mixes. A total of 15 different concrete mixes were prepared containing 0, 5, 10, 15, and 20% gypsum and 0, 25 and 50% fly ash (FA) as partial replacement for cement. Superplasticizer was used to regulate the mixture consistency, as adding gypsum was found to dehydrate the mix. Nine identical specimens per mix were cast into 200 mm x 100 mm cylindrical molds, and 3 of each were tested for compressive strength after curing in a moist room for 7, 28 and 90 days. The mixes were separated into 3 groups based on the FA content, and strength results were compared to those of the respective control mix containing no gypsum, namely the '0% FA control mix', '25% FA control mix' and '50% FA control mix'. The study revealed that using only gypsum as a partial cement replacement was disadvantageous to strength properties, however combining fly ash and gypsum was beneficial at later ages. After 90 days, specimens containing 5% gypsum and 25% FA showed a 15% strength increase from the

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[2] This chapter has been submitted for review as a manuscript to the Journal of Cleaner Production

25% FA control mix. All mixes containing 50% FA maintained equivalent strength to the 50% FA control mix after curing for 90 days, showing that the addition up gypsum up to 20% of cementitious content had no negative effect on the compressive strength at later ages. Incorporating recycled gypsum drywall in concrete not only keeps harmful material out of landfills, but also can provide positive effects on concrete strength when adequately combined with fly ash.

4.1 INTRODUCTION

Gypsum is one of the oldest and most commonly used building materials globally due to its many positive attributes. Firstly, it is abundant, being mined in its natural form, as well as being generated synthetically as a by-product of select industrial processes. It is also economical, fire resistant, versatile and can reduce sound (Olson 2001). Gypsum ($CaSO_4 \bullet 2H_2O$) deposits are formed naturally in sedimentary basins where calcium sulfate ($CaSO_4$) resources were hydrated. Gypsum is predominantly used for wallboard, also known as drywall or gyprock walls, and has been extensively used in the building industry for the construction of interior walls and ceilings. The main component of the wallboards is gypsum, which is extruded between two layers of paper. There are many other known markets for the product, including its widespread use in the farming industry for its agricultural benefits and soil improvement capabilities, as well as for animal bedding (CDRA 2019). The essential role of gypsum is widely acknowledged in cement production, although only used in small percentages. It is incorporated into the cement as a set regulator to control the setting of cement in order to reduce the speed of reaction with water (Naik et al. 2010). Recycled gypsum can be used interchangeably with natural gypsum for almost all applications. A study by Suarez et al. (2016) uses life cycle assessment methodology to evaluate the environmental impacts of using natural and recycled gypsum in Portland cement production. This research showed that using recycled gypsum instead of naturally mined gypsum provides many environmental benefits, including consuming less than 65% of the energy, and emitting less than 65% of greenhouse gases.

Despite being a valuable recyclable material, gypsum is mainly disposed of in landfills, thereby increasing the demand on virgin materials and taking up unnecessary landfill space (Recycling Council of Ontario 2006). It is also important to recognize that the decomposition of gypsum waste in landfills can cause a series of biological and chemical reactions with potential for harmful environmental impacts, mainly due to its sulfate (SO_4) content. Having a sustainable use for recycled gypsum in concrete would provide contractors doing demolition projects with a direct way to re-use the material, expectantly making gypsum recycling more common.

The basic components of concrete are water, coarse and fine aggregates, and cement. Of these components, is well known that the production of cement is the most environmentally impactful. The production of one ton of cement releases nearly one ton of carbon dioxide (CO_2) into our atmosphere, causing negative impacts on our ecosystems and contributing to global warming (Meyer 2009). Since the popularity of concrete is unlikely to decrease and environmental effects are increasingly of concern, there is an apparent need to transition concrete to a more environmentally friendly material. Considering cement is the most harmful, an obvious solution is to use less cement by partially replacing it with other cementitious materials. Common supplementary cementitious materials include by-products of industrial processes, such as fly ash. The availability of fly ash has increased significantly since environmental regulations required power plants to install mechanisms to trap fine particles (Meyer 2009). Incorporating fly ash in concrete mixes provides economic and ecological benefits by utilizing a product that is potentially harmful and costly to dispose of. A study done by Wu and Naik (2002) found that blended cements containing 40% and 60% coal-combustion by-products such as fly ash develop higher compressive strength and a higher resistance to freezing and thawing cycles. However, it is widely acknowledged that fly ash can reduce the early strength of concrete (Wu and Naik 2002). Typical Portland cement has adequate oxides and aluminates to react when hydrated, however fly ash necessitates additional activators (such as gypsum) in order to initiate hydration (Marlay 2011).

As previously mentioned, gypsum, or calcium sulfate di-hydrate ($CaSO_4 \bullet 2H_2O$), is essential to the production of Portland cement and is already incorporated in cement clinkers at approximately 5% (Naik et al. 2010). Without calcium sulfate sources, the tricalcium aluminate ($3CaO \bullet Al_2O_3$ abbreviated to C_3A) in cement will have a rapid reaction with water (H_2O) causing it to harden too quickly and become unworkable (Marlay 2011). The sulfate (SO_4) contained in gypsum reacts at room temperature in early ages with tricalcium aluminate and water to form needle-like crystals of calcium sulfoaluminate, or ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12} \bullet 26H_2O$). Ettringite is a necessary and beneficial component in concrete, likely contributing to its strength (Portland Cement Association 2001). Having an elevated concentration of tricalcium aluminate in comparison to calcium hydroxide ($Ca(OH)_2$) at a later time may cause the ettringite to become unstable and convert to monosulfate ($Ca_4Al_2O_6(SO_4) \bullet 14H_2O$) (Christensen et al. 2004). If sulfate ions get into concrete later, the monosulfate will react with present tricalcium aluminate ions to convert back to ettringite in an expansive reaction (Naik et al. 2010). This expansive reaction is referred to as delayed ettringite formation and is unfavourable as it can crack or damage concrete that has already hardened. It is established that gypsum can stabilize ettringite and replacing some cement with fly ash reduces the amounts of free tricalcium aluminate and calcium hydroxide from cement (Wu and Naik 2002). The activation by gypsum is mainly based on the capability of sulfate ions to react with the alumina provided by fly ash (Aimin and Sarkar 1991). An optimum use of gypsum and fly ash can therefore aid in reducing the quantities of susceptible components (monosulfate and calcium hydroxide) present. However, there is a concern in the literature about undesirable chemical reactions that may occur within the microstructure of concrete during hydration with excess gypsum. It has been reported that excess amounts of activators (gypsum) may cause a "false set" (stiffness) of fresh concrete, meaning the mix did not develop the required densification of the microstructure, affecting the concrete's durability (Naik et al. 2010) (Marlay 2011). It has also been reported that adding high amounts of sulfate sources can cause excessive expansion after hardening (Naik et al. 2010) (Portland Cement Association 2001).

Research done by Naik et al. (2010) considered concrete mixes replacing cement with 0, 7, 10 or 20% powdered gypsum wallboard, combined with 0, 20, 33, 50 or 60% fly ash. The study revealed that mixtures containing powdered gypsum showed lower compressive strength, particularly at early ages. However, the replacement of cement with a combination of fly ash and gypsum performed better than replacing cement with only gypsum. Combining fly ash (20%) and powdered gypsum (10%) with cement (70%) yielded results comparable to plain cement after aging 91 days, demonstrating that up to 10% powdered gypsum can be used in concrete without showing adverse effects on its mechanical properties. During the same experiment, higher expansion (0.043%) was observed in concrete mixtures made with gypsum, implying lower resistance to sulfate attack. Although, a mortar mixture replacing cement with 10% powdered gypsum and 50% fly ash showed much higher resistance to sulfate attack than the control mixture containing only cement. Research by Raghavendra and Udayashankar (2015) was done on the fresh and hardened properties of mortar mixes containing discarded gypsum wallboard and fly ash as secondary cementitious materials in controlled low strength materials (CLSMs). This investigation used quarry dust as the fine aggregate, and the mix proportions of powdered gypsum wallboard as cement replacement are considerably high (51.9-60.9%). They reported reduced compressive strength and increased water demand of CLSM mixes when gypsum and quarry dust were incorporated, with the maximum compressive strength occurring at 28 days. The aforementioned research by Naik et al. (2010) only considers five concrete mixes with gypsum replacing 10% or more of the cementitious material, and the report by Raghavendra and Udayashankar (2015) only considers mortar mixes. Thus, it seems that substantial technical research has not been done concerning large proportions of gypsum as partial cement replacement in concrete.

This study was designed to continue exploring the prospect of reducing the amount of cement needed in concrete by creating multiple mix combinations replacing up to 75% of cementitious material with recycled gypsum powder and fly ash. The goal is to produce a more sustainable and environmentally friendly concrete mix that maintains adequate compressive strength.

4.2 EXPERIMENTAL PROGRAM

4.2.1 Test Matrix

The test matrix consisted of 15 batches of concrete with varying compositions of cementitious material, including control mixes with only cement (Case #1, 6, 11). Gypsum was used as partial replacement by weight for cement at proportions of 0, 5, 10, 15 and 20%. Fly ash was used at 0, 25 and 50% partial replacement by weight for cement. The water to cement ratio (W/C) was kept to 0.475 in all mixes. To keep the consistency of the mix to a relatively similar level of workability, variable amounts of superplasticizer were added during mixing. The specimens' batch proportions are displayed in Table 4-1.

Table 4-1 Test matrix and cementitious content percentage by weight

Case #	**Specimen ID**	**Percentage by weight (%)**			**Number of Specimens**
		Gypsum	**Fly Ash**	**Cement**	
1	FG0-FA0-C100	0	0	100	9
2	FG5-FA0-C95	5	0	95	9
3	FG10-FA0-C90	10	0	90	9
4	FG15-FA0-C85	15	0	85	9
5	FG20-FA0-C80	20	0	80	9
6	FG0-FA25-C75	0	25	75	9
7	FG5-FA25-C70	5	25	70	9
8	FG10-FA25-C65	10	25	65	9
9	FG15-FA25-C60	15	25	60	9
10	FG20-FA25-C55	20	25	55	9
11	FG0-FA50-C50	0	50	50	9
12	FG5-FA50-C45	5	50	45	9
13	FG10-FA50-C40	10	50	40	9
14	FG15-FA50-C35	15	50	35	9
15	FG20-FA50-C30	20	50	30	9
Total	-	-	-	-	135

Note: 3 identical specimens of each group were tested at 7, 28, and 90 days

4.2.2 Material Properties

The cement used in all mixes was Type GU Portland Cement (CRH Canada Group, ON, Canada). The fly ash is ‘Class F’ bituminous coal fly ash donated from a local business (Ocean Contractors, Halifax, NS, Canada). The gypsum used in this study was from a drywall waste recycling company (USA Gypsum, Denver, PA, USA) that processes the material into an ultra-fine consistency with particle sizes ranging from 3.175 mm to dust. The fine aggregate (sand) and coarse aggregate (gravel) were locally sourced (Casey Metro, Halifax, NS, Canada) following ASTM C33/C33M (2018). Moisture content tests revealed that the gravel had a very small average moisture content of 0.12%, so it was used in as-is condition. The gypsum powder and sand showed higher moisture contents (18.29% and 3.39%, respectively), so they were oven-dried overnight, allowed to cool and then stored in airtight containers before use.

A sieve analysis was conducted according to ASTM C136/C136M (2015) for fine aggregate (sand) and the original recycled gypsum material. During the sieve analysis of gypsum, it was discovered that light-weight fibre-like particles attached together to create small bunches or clusters of material. The researchers assume that these particles are made of paper or paint that remained from the outer sheets during the recycling process, however the exact composition is unknown. These bunches tend to be larger and more loosely attached on sieves with larger openings, and more frequent and densely packed as the sieve opening size decreases. These clusters were no longer noticeable passed the No. 100 sieve (0.149 mm opening) and No. 200 sieve (0.074 mm opening). Preliminary testing was done using the original gypsum material (with particle bunches) as well as with only the fine gypsum particles (retained on the No. 100, No. 200 and tray). These trial tests indicated that using only the fine gypsum material showed increased compressive strength results up to 7 days. Accordingly, only the fine gypsum particles were used in the concrete mixes, and the coarse portion was discarded. Photos of various sieves retaining the recycled gypsum material and particle bunches are shown previously in Chapter 3, Figure 3-2.

Figure 4-1 shows magnified polarized light photos taken under a microscope of fine and coarse gypsum, cement, and fly ash. The particles were placed into a puck shape mold, coated with epoxy, cured at air temperature, and then polished before examination with the microscope.

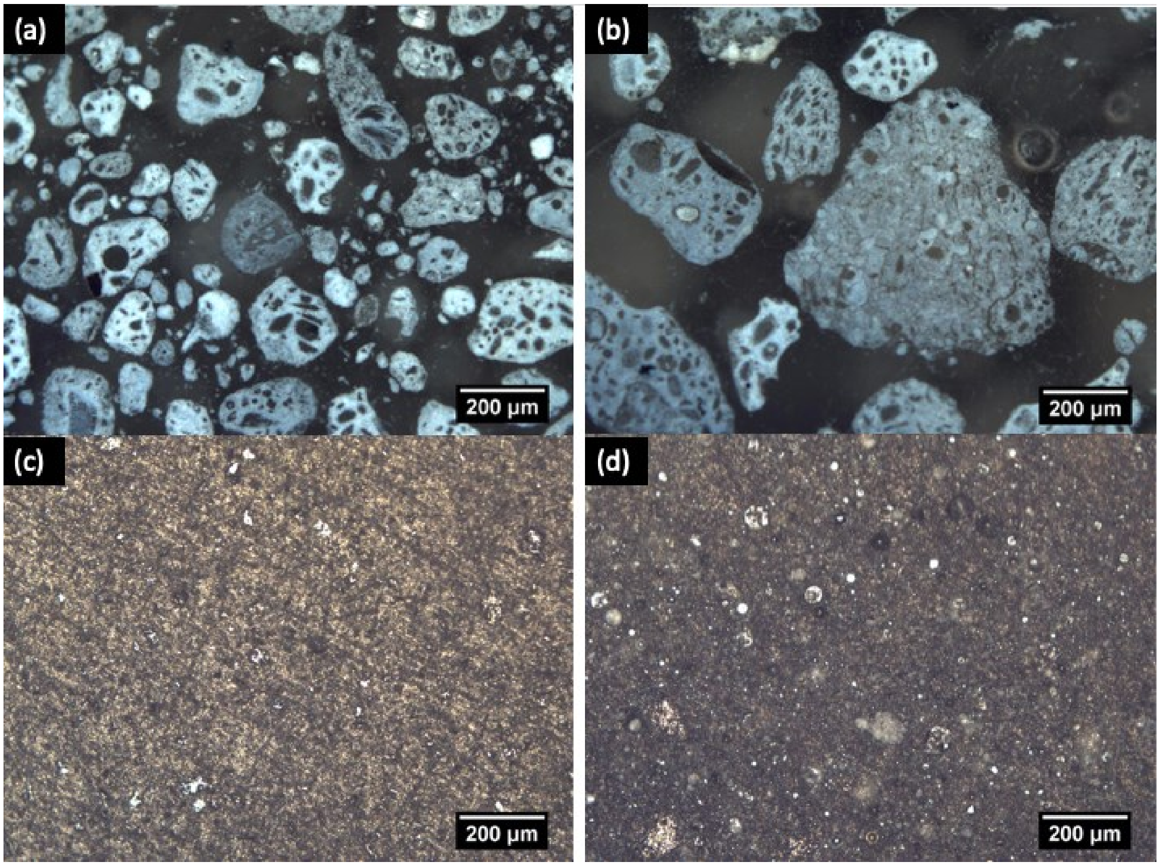

Figure 4-1 Magnified view of (a) fine gypsum; (b) coarse gypsum; (c) cement; (d) fly ash

Additionally, a particle size distribution analysis for the fine particles (fine gypsum, fly ash and Portland cement) was done using laser diffraction by Dalhousie University's

Materials Engineering Center. The particle size distribution curves for these fine materials, along with the sand and original (coarse) gypsum, are shown in Figure 4-2.

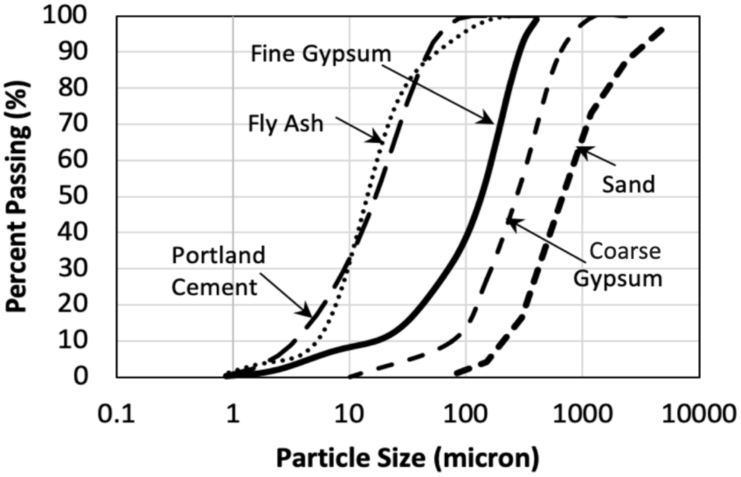

Figure 4-2 Particle size distribution of various materials

4.2.3 Specimen Preparation

To prepare the test specimens, ASTM C192/C192M (2018) was followed for each of the mix designs. All material was added to the mixer and allowed to mix until a uniform texture was accomplished. The mixer was stopped periodically and manually scraped to ensure that minimal material was stuck to the sides and in the centre. Adding gypsum to concrete, even at only 5% of cementitious material, was found to dehydrate the mix and reduce workability. Each mix was therefore visually and physically accessed by the researcher and based on the workability in comparison to the control mix, it was decided whether or not to add superplasticizer. If needed, superplasticizer was added to the mixer in increments of 10 mL using a syringe and evenly distributing the liquid throughout by continuing mixing until uniform. In accordance with previous research, it was realized that mixes containing higher amounts of gypsum would require more superplasticizer. To adhere to the recommendation of ASTM C192/C192M- Section 8.1.2 (2018), the quantity of superplasticizer from the previous (lower gypsum content) mix was added to the water during mixing, instead of directly to the concrete.

After mixing, cylindrical molds with a diameter of 100 mm and a depth of 200 mm were filled and hand tamped as required by the standard. Due to the longer set time required for fly ash noticed by the researcher during trial tests, all molds were removed after 5 days and cured in a moist closet. This differs from the ASTM specification, which indicates removal from molds after 24 hours. Figure 4-3 (a) shows a sample of the dry materials used in concrete mixes, Figure 4-3 (b) shows the researcher hand tamping concrete in the cylindrical molds, and Figure 4-3 (c) shows a sample of specimens with and without fly ash after removal from the molds. It can be seen that specimens containing fly ash (labelled FG0-FA50-C50-X) appear to have a darker colour than those without (labelled FG5-FA0-C95-X); this was more noticeable at early ages. All specimens were cured in a moist room held at approximately 93-96% humidity during their curing periods of 7, 28 and 90 days.

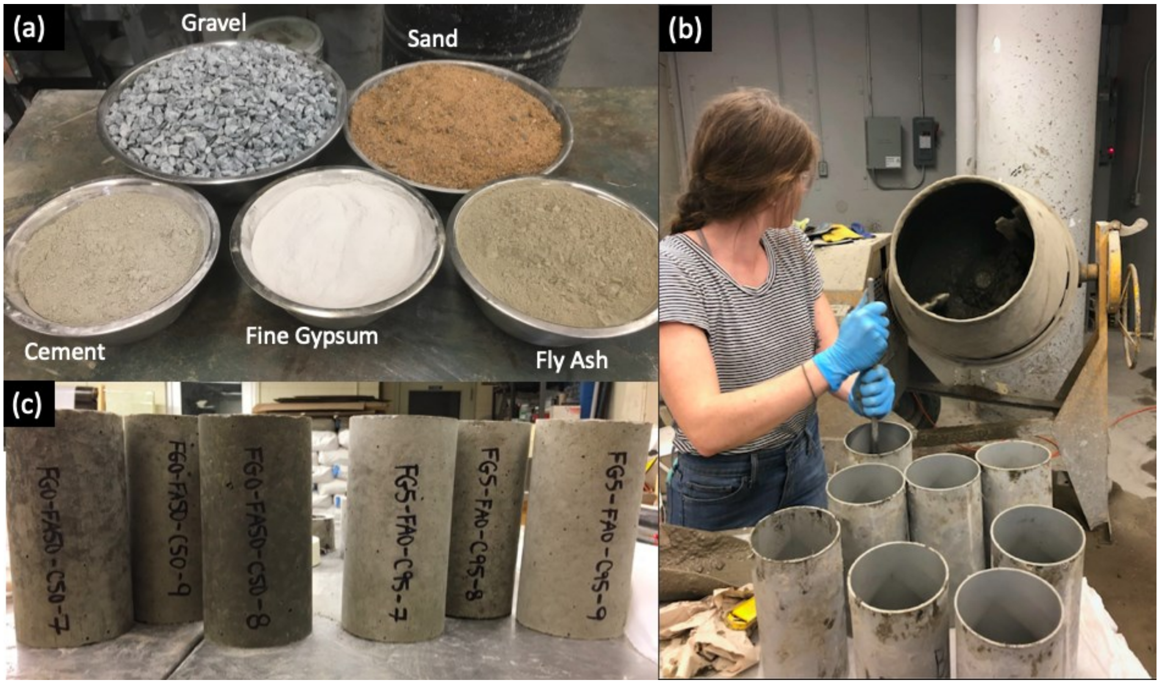

Figure 4-3 Specimen preparation: (a) dry materials; (b) hand tamping; (c) comparison of specimens with and without fly ash

4.2.4 Test Setup and Instrumentation

The procedure for the determination of compressive strength was in accordance with ASTM C39/C39M (2016). To ensure even loading, each end was capped using a sulfur capping compound and allowed to set for about 3 hours prior to testing. The specimens tested on day 7 and 28 were tested using a machine that measured only the maximum compressive load. A spherical platen was used on the upper surface of the compressive machine to minimize any accidental eccentricities. On day 90, compression tests were conducted on a universal testing machine with a constant loading rate of 0.5 mm/min. In addition, the specimens tested on day 90 were also equipped with four linear potentiometers (LPs) to measure axial and lateral strain. Two lateral LPs (LP #1 and LP #2) were placed perpendicular to load and to the cylinders side, at approximately 180 degrees from one another. The axial LPs (LP #3 and LP #4) were fixed parallel to the cylinders side and opposite of one another, using a metal bracing system connected to the cylinder by six bolts. A schematic of the test setup and instrumentation is shown in Figure 4-4 (a), and a photo of the actual setup is shown in Figure 4-4 (b). All specimens were subject to compressive loading until failure, determined to be just after the peak load was attained. The effect on physical properties were also inspected, including specimens' weight and diameter change.

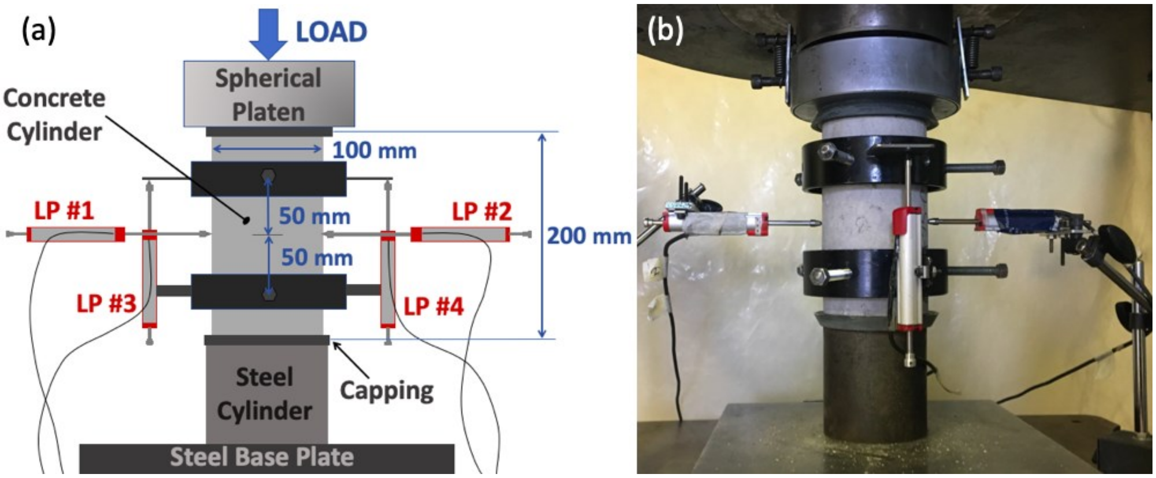

Figure 4-4 Test setup and instrumentation (a) schematic; (b) actual test setup

4.3 RESULTS AND DISCUSSION

4.3.1 Compressive Behaviour

Specimens were tested for compressive strength (f'_c) under axial loading until failure after curing for 7, 28 and 90 days. Table 4-2 presents the summary of compression test results based on the average of three identical specimens for each specimen group, including the coefficient of variation (CV). At failure, all specimens showed observable micro-cracking on the surface, and often audible fracturing of concrete was distinguished as the peak load was approached. It was observed that all specimens failed in compression in relatively the same manner. A combination of longitudinal (vertical) and transvers (horizontal) cracking occurred, often causing larger diagonal cracks. Spalling of the concrete surface was also observed in areas near cracks. The fracture types identified include a combination of conic, diagonal and shear cracking. The severity of cracking depended on how long the specimen was subject to loading past its peak load. More visible and severe cracks occurred on specimens that were left under loading for longer time periods after failure, occasionally leading to complete fracture. Figure 4-5 depicts various specimens after failure, showing cracking patterns.

Table 4-2 Summary of test results for compressive strength

Specimen Group ID	**Day 7**		**Day 28**		**Day 90**	
	Average Strength (MPa)	CV (%)	Average Strength (MPa)	CV (%)	Average Strength (MPa)	CV (%)
FG0-FA0-C100	31.78	5.57	43.16	2.64	50.05	7.37
FG5-FA0-C95	22.05	6.26	33.29	17.24	48.69	4.99
FG10-FA0-C90	23.99	8.55	28.47	4.46	33.71	9.31
FG15-FA0-C85	17.85	2.86	28.38	3.00	32.95	6.59
FG20-FA0-C80	19.78	6.02	24.98	4.16	29.1	4.67
FG0-FA25-C75	24.03	3.04	34.61	4.19	39.46	5.96
FG5-FA25-C70	14.31	8.81	30.69	10.75	45.23	3.25
FG10-FA25-C65	12.61	8.80	21.53	3.02	30.41	17.07
FG15-FA25-C60	14.73	7.88	23.94	2.97	35.71	5.24
FG20-FA25-C55	9.92	5.14	18.98	2.21	27.64	9.95
FG0-FA50-C50	16.71	3.71	29.46	5.47	34.88	10.61
FG5-FA50-C45	6.7	4.33	22.99	4.52	35.83	6.17
FG10-FA50-C40	8.55	3.39	17.38	6.90	37.18	10.09
FG15-FA50-C35	7.18	3.06	17.09	5.97	34.96	4.06
FG20-FA50-C30	5.48	8.39	17.33	2.08	38.25	6.64

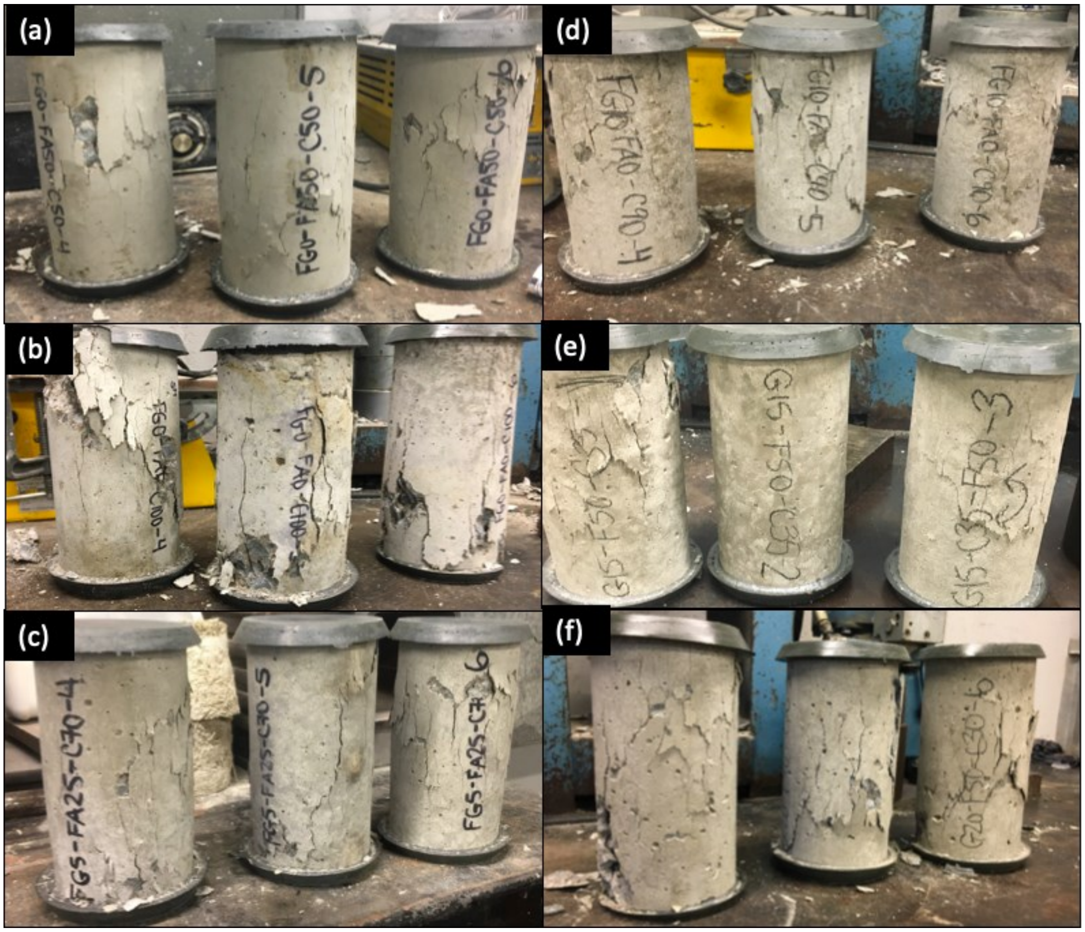

Figure 4-5 Specimens after failure: (a) FG0-FA50-C50; (b) FG0-FA0-C100; (c) FG5-FA25-C70; (d) FG10-FA0-C90; (e) FG15-FA50-C35; (f) FG20-FA50-C30

4.3.2 Effect of Curing Time

Figure 4-6 shows the average compressive strength of specimens with varying amounts of fly ash (FA) as a function of the gypsum content in the cementitious material. Error bars represent the standard deviation above and below the average of the three identical test specimens. Figure 4-6 (a) considers mixes with 0% FA and shows a trend of decreasing compressive strength with increasing gypsum content at all ages. It also shows that compressive strength gradually improved as the curing time increased. Figure 4-6 (b) considers mixes with 25% FA and shows a similar trend as Figure 4-6 (a) of decreasing strength with increasing gypsum content at all ages, although not as distinguished, especially at day 90. There is a notably elevated 90-day strength for mix ID: FG5-F25-C70, where the compressive strength was observed to be 15% higher than the 25% FA control mix (FG0-F25-C75). In Figure 4-6 (b), the strength increase between test days is higher than in Figure 4-6 (a), meaning the effect of curing time becomes more significant with the addition of fly ash. Figure 4-6 (c) considers mixes with 50% FA and shows notably higher strength variability between test days, especially when comparing the 7-day strength to the 90-day strength. In this case, curing time had a very large impact on compressive strength results. The increasingly large strength differences in between test days with increasing FA content indicates that incorporating fly ash retards the development of compressive strength in concrete mixes. This is also evident by comparing the 7-day strength in Figure 4-6 (a) to Figure 4-6 (b) and (c) where mixes containing 25% FA and 50% FA show consistently lower early strength, more noticeable in Figure 4-6 (c) with higher fly ash content. In other words, the effect of curing time has a less significant effect on specimens without fly ash, and the significance of curing time increases by increasing the fly ash content.

A distinctly different trend is observed in Figure 4-6 (c) between the 90-day strengths. The 7-day and 28-day strengths typically follow the previously identified trend of decreasing strength with increasing gypsum content, however after curing for 90 days, the compressive strength is similar for all 50% FA specimens. That is, increasing the gypsum content from 0% to 5, 10, 15 and 20% shows minimal effect on the 90-day strength of concrete specimens with 50% fly ash content in the cementitious material. When comparing with the 50% FA control specimens containing no gypsum (FG0-FA50-C50), mixes with added gypsum actually developed consistently higher 90-day strength with up to 20% gypsum content. Remarkably, the highest average strength of all mixes containing 50% FA was the mix with 20% gypsum and only 30% cement as the cementitious material (FG20-F50-C30). This mix showed a 10% strength increase from the 50% FA control mix (FG0-FA50-C50), rising from 34.88 MPa to 38.25 MPa.

Figure 4-7 was developed to compare mixes containing gypsum to each of the three control mixes that do not contain gypsum; that is, the 0% FA control mix (FG0-FA0-C100), the 25% FA control mix (FG0-FA25-C75) and 50% FA control mix (FG0-FA50-C50). The average strength of the specimens (f'_c) was divided by the applicable average strength of the control specimens ($f'_{c\text{-}control}$) and shown as a function of the gypsum content. Figure 4-7 (a) shows that incorporating only gypsum as partial cement replacement is seen as a disadvantage to the compressive strength at all ages. Figure 4-7 (b) shows that increasing the gypsum content in mixes with 25% FA is also seen as a disadvantage, with the exception of the previously identified 90-day strength of mix FG5-FA25-C70, which is recognized as the graphs highest peak. Figure 4-7 (c) highlights the positive reaction between gypsum and FA, when FA is used at 50% replacement for cement in concrete mixes and allowed to cure for 90 days. In this case, all mixes containing gypsum outperformed the 50% FA control mix in terms of compressive strength, depicted in Figure 4-7 (c) as the top line with $f'_c / f'_{c\text{-}control}$ values above 1.

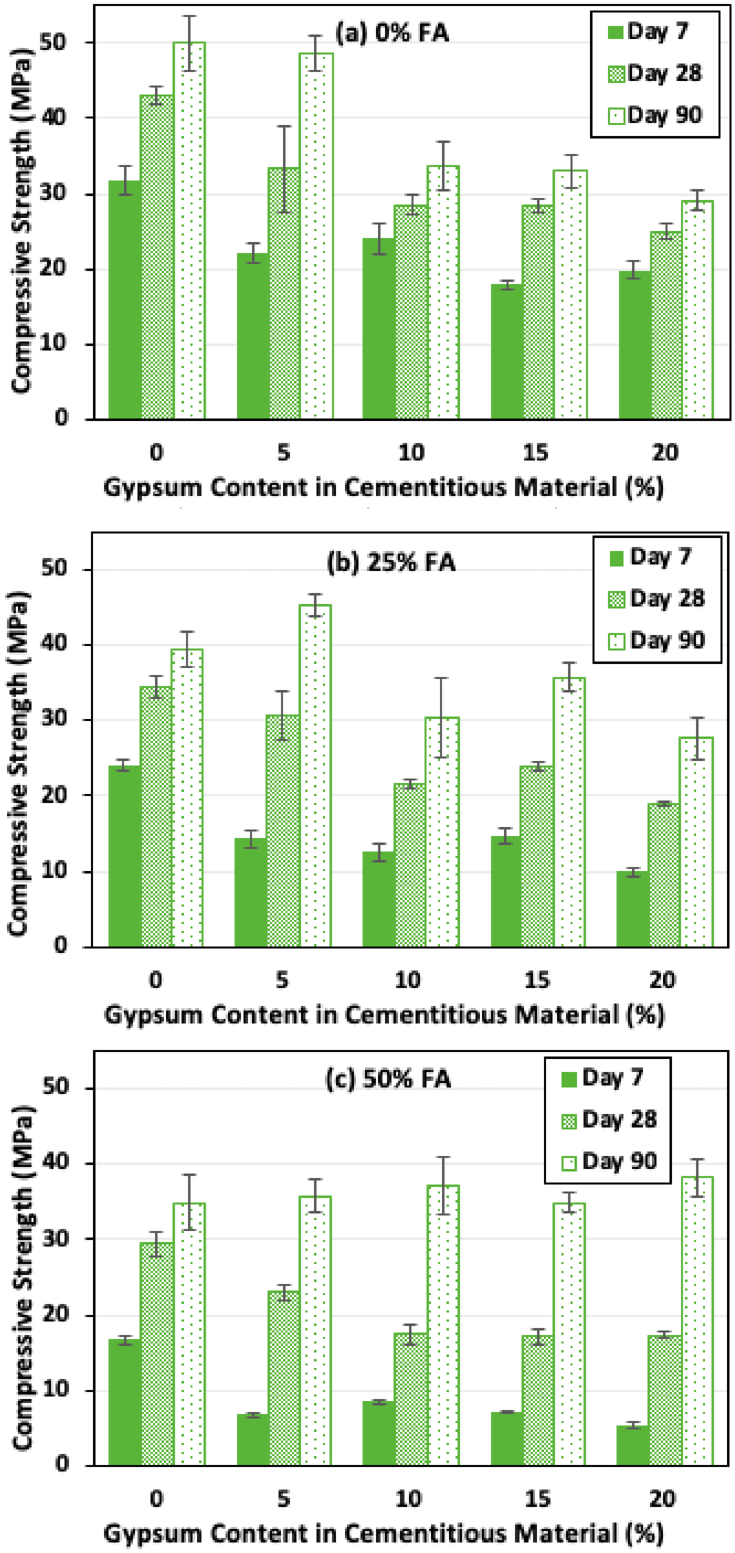

Figure 4-6 Compressive strength with varying gypsum content for (a) 0% FA; (b) 25% FA; (c) 50% FA

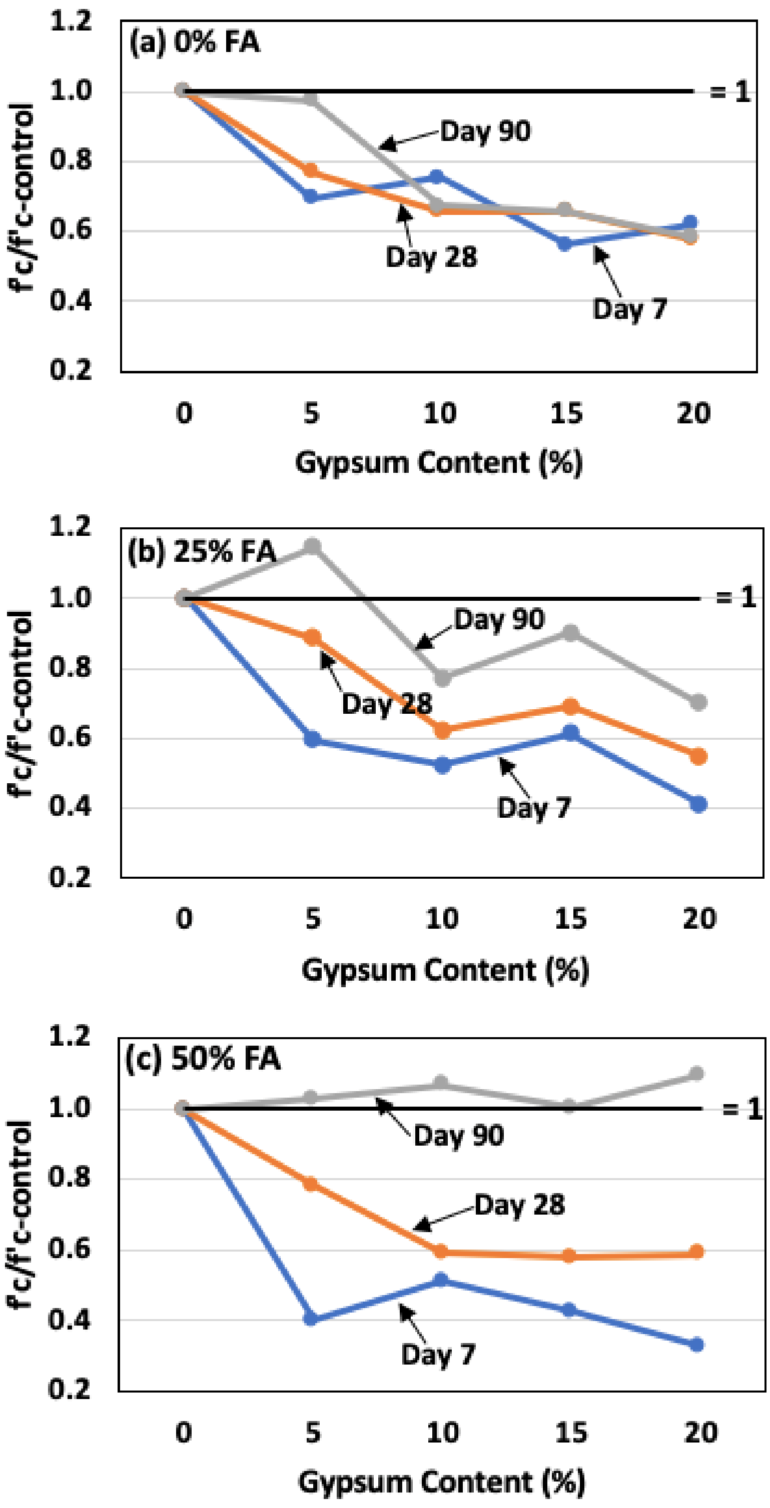

Figure 4-7 Compressive strength comparison to control mixes with varying gypsum content for (a) 0% FA; (b) 25% FA; (c) 50% FA

4.3.3 Effect of Gypsum and Fly Ash on Physical Properties

In terms of workability, the gypsum content was found to have a significant effect during the mixing stage. As previously mentioned, increasing amounts of superplasticizer were needed as the gypsum content increased in order to keep the consistency and workability of fresh concrete similar to the control batch. Mixes containing 5% and 10% gypsum maintained a relatively constant consistency throughout casting, however there was evidence of a chemical reaction occurring at 15% and 20% gypsum content. The reaction caused the concrete in the mixer to "false set" suddenly, leaving the concrete very stiff with severely decreased workability. The surface of the concrete became very hard to the touch and large portions of concrete stuck to the sides of the mixer. This phenomenon occurred during a short period of time, typically after the mixer had been stopped for about a minute and the first or second specimen was being cast (out of 9 specimens per batch). Considerable effort was required to then loosen the hardened concrete and remove it from the sides of the mixer. It is interesting to note that once the concrete was loosened after the false set, the workability improved, allowing the researcher to cast and tamp the remaining specimens. This false set reaction was noticeable for all mixes containing at least 15% gypsum; however, it was more severe and harder to regain workability in mixes with 20% gypsum content. For this reason, no specimens containing more than 20% gypsum were fabricated.

All specimens were weighed on day 5 after being removed from the molds, and the results shown in Figure 4-8 are averaged from all nine specimens of each concrete mix. It was detected that fly ash has a lower density in concrete mixes when compared to cement, as the specimens with 25% FA and 50% FA show a decreased weight in comparison to the average weight of the 0% FA control specimens, as seen in Figure 4-8. For mixes with 25% FA and 50% FA, a generally downward trend is observed with increasing gypsum content from the control mix weight. This indicates that gypsum also has a lower density in concrete mixes when compared to cement. Three specimens per concrete mix were also weighed after curing for 28 and 90 days, confirming that specimens with increased fly ash and gypsum content typically show decreased weight in comparison to the control specimens, especially at later ages. As expected, the largest weight decrease from the control specimens was mix FG20-FA50-C30 at day 90, decreasing 3.4%.

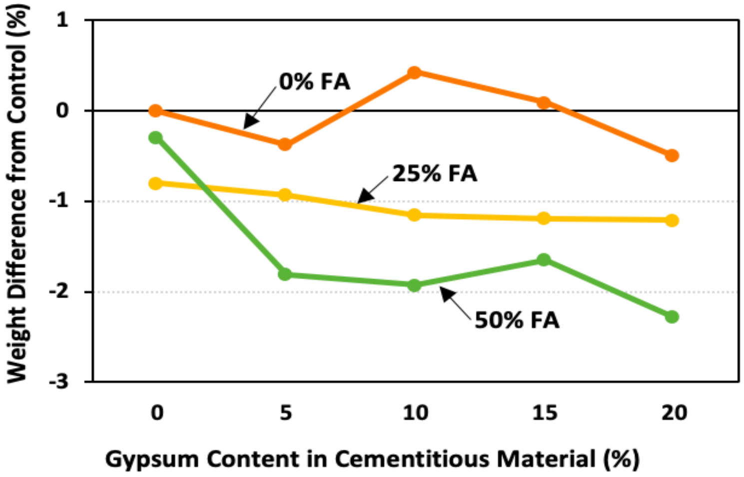

Figure 4-8 Difference of specimens' weight in comparison to control mix on day 5

In the literature, research has shown that adding even small amounts of gypsum to concrete can cause expansion (Naik et al. 2010). Digital calipers were used to measure the diameter of cylinders by marking three different diameters on the specimens and re-measuring the same lines to detect any changes. Three specimens from each concrete mix were measured on day 5 and on day 90, however it was chosen to measure only four mixes periodically: the control mix (FG0-FA0-C100), and all specimens with 20% gypsum content (FG20-FA0-C80, FG20-FA25-C55, FG20-FA50-C30). Based on the measurements, no expansion was observed when comparing to the original diameters measured. If any diameter change did occur in the specimens, it was beyond the accuracy of the calipers. It is recommended that the expansion of cylinders be measured using a more precise measuring tool that is able to accurately evaluate both length and diameter change.

4.3.4 Stress-Strain Behaviour

Axial and lateral strain data was collected for all specimens tested on day-90. The axial stress-strain behaviour is presented in Figure 4-9, separated into each of the three groups: a) 0% FA; b) 25% FA; and c) 50% FA. The vertical axis shows the compressive strength in MPa, and the horizontal axis shows the strain in increments of 0.003 mm/mm. Expectantly, all curves depict typical stress-strain behaviour for concrete with slight variations between specimens. The peak strain behaviour between specimens is analyzed further in the following section. It is believed that substantial error occurred when lateral strain data was collected, rendering data unreliable and unusable for multiple specimens. Due to the high sensitivity of LPs, any small misalignment would cause the lateral LPs (placed perpendicular to the specimens rounded side) to slide in sharp undesirable movements during testing. The misalignment may be caused by spalling of the concrete surface during testing, or by human error during setup if LP was not placed perfectly perpendicular to the rounded surface. The faulty lateral data collected often showed major inconsistencies between identical specimens, including large jumps in strain with minimal stress increase. Lateral stain data is therefore not included in this chapter, however representative graphs including stress and strain data for both lateral and axial directions are found in Appendix A, taken from tests where error/inconsistency was not observed.

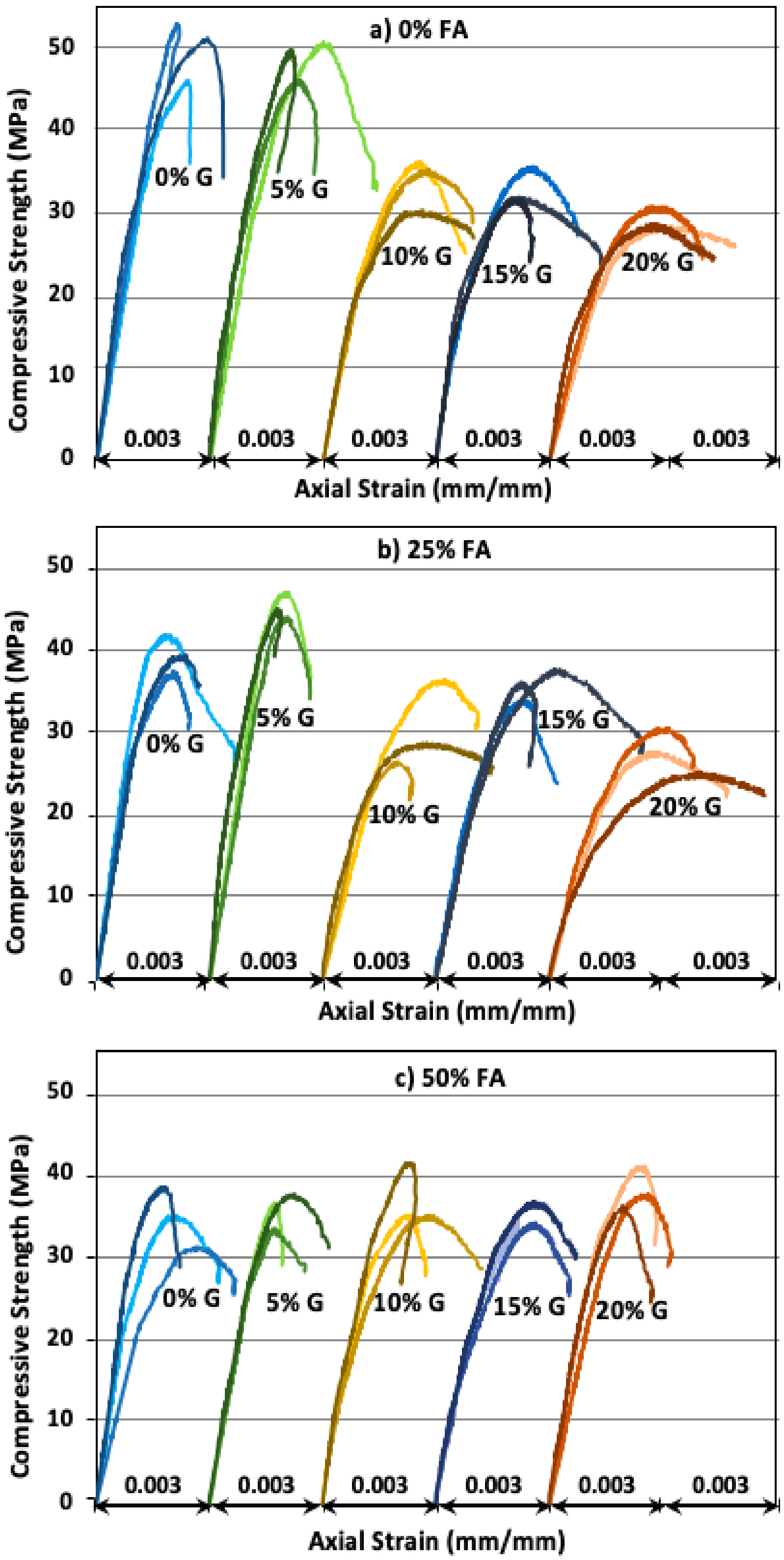

Figure 4-9 Axial strain of specimens with varying gypsum content for a) 0% FA; b) 25% FA; c) 50% FA

Table 4-3 presents a summary of the day-90 peak compressive strength (f'_c), axial strain at peak (ε'_c), and the elastic modulus (E_c), determined based on methods from both CSA (Canadian Standards Association (CSA) 2004) and ASTM (ASTM 2019) standards. The CSA standard uses the equation $E_c = 4500\sqrt{f'_c}$ to calculate the modulus of elasticity for normal density concrete. The ASTM standard uses a customary working stress range from 0-40% of ultimate concrete strength to calculate the modulus of elasticity based on the stress to strain ratio value. The difference between the methods is shown in the last column by dividing E_{c-CSA}/E_{c-ASTM} for each specimen group, including the average difference and standard deviation (SD) between CSA and ASTM methods. The coefficient of variation was calculated to be 12.21%.

Table 4-3 Summary of axial stress-strain and elastic modulus

Specimen Group ID	f'_c (MPa)	ε'_c (mm/mm)	$E_{c\text{-}CSA}$ (MPa)	$E_{c\text{-}ASTM}$ (MPa)	$\frac{E_{c\text{-}ASTM}}{E_{c\text{-}CSA}}$
FG0-FA0-C100	50.05	0.00251	31,822	31,222	0.98
FG5-FA0-C95	48.69	0.00250	31,395	32,903	1.05
FG10-FA0-C90	33.71	0.00256	26,108	24,354	0.93
FG15-FA0-C85	32.95	0.00225	25,821	32,727	1.27
FG20-FA0-C80	29.10	0.00294	24,273	21,973	0.91
FG0-FA25-C75	39.46	0.00206	28,260	30,629	1.08
FG5-FA25-C70	45.23	0.00198	30,260	33,811	1.12
FG10-FA25-C65	30.41	0.00259	24,758	21,864	0.88
FG15-FA25-C60	35.71	0.00259	26,887	23,410	0.87
FG20-FA25-C55	27.64	0.00281	23,637	18,177	0.77
FG0-FA50-C50	34.88	0.00222	26,552	29,106	1.10
FG5-FA50-C45	35.83	0.00185	26,927	25,892	0.96
FG10-FA50-C40	37.18	0.00248	27,417	27,206	0.99
FG15-FA50-C35	34.96	0.00245	26,605	26,412	0.99
FG20-FA50-C30	38.25	0.00230	27,820	26,300	0.95
				Average:	0.99
				SD:	0.12

Figure 4-10 shows the axial strain at peak load of specimens as a function of the gypsum content in the cementitious material, including error bars representing the standard deviation of the three identical specimens. There is noticeable overlap of standard deviation between most specimens which indicates comparable results, with no obvious trend observed. The variability causing high standard deviations can be described by the unavoidable inconsistencies between identical concrete specimens during mixing, as well as possible discrepancies of the LPs during testing.

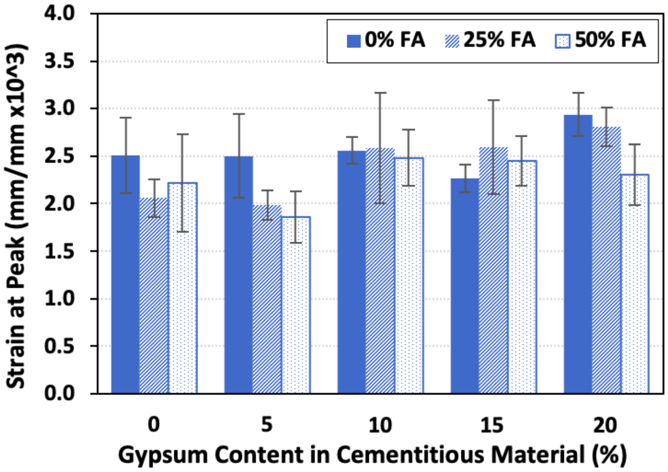

Figure 4-10 Axial strain at peak with varying gypsum content

4.3.5 Statistical Evaluations

An analysis of variance (ANOVA) is commonly performed to analyze factors that may affect a data set. ANOVA compares the variance caused by the between-groups variability (mean square effect or MS_{effect}) with the within-group variability (mean square error or MS_{error}) by means of the F-test. The F-value is calculated from the analysis results as follows: $F = MS_{effect}/MS_{error}$. This F-value is compared to the critical F-value (F_{crit}) that is extracted from statistical tables based on the number of degrees of freedom. The null hypothesis states that the means are equal, and the alternate hypotheses states that they are not. If the F-value exceeds F_{crit}, the null hypothesis is rejected and indicates a statistically significant result deemed unlikely to have occurred by chance. If F_{crit} exceeds the F-value, the null hypothesis is assumed to be true or accepted, indicating a statistically non-significant result. For all statistical evaluations in this study, a confidence level of 95% was used (significance level of 5% or 0.05).

In terms of the compressive strength of specimens after curing for 90 days, ANOVA single factor analysis was used in Microsoft Excel with data from three identical specimens to compare two parameters separately, namely the gypsum content and the fly ash (FA) content. The results are summarized in Table 4-4. When considering specimens with 0% and 25% FA, the gypsum content in the cementitious material showed a significant effect on the compressive strength ($F > F_{crit}$), rejecting the null hypothesis. Alternatively, the analysis showed that gypsum content had a non-significant effect on strength of specimens with 50% FA ($F < F_{crit}$). When considering specimens with 0, 5 and 20% gypsum, the variation of FA content showed a significant effect on the compressive strength. However, the results for specimens with 10% and 15% gypsum indicated that the FA content did not have a significant effect on the 90-day strength.

In terms of the axial strain at peak load, it is concluded that the effect gypsum content is non-significant ($F < F_{crit}$) on the axial strain at peak for all three FA groups at a 95% confidence level, and the null hypothesis is accepted. When the source of variation is FA content, all specimens with 0-15% gypsum showed that the axial strain at peak load was not significantly affected. Specimens with 20% gypsum also showed significant effects of FA content on the strain at peak ($F > F_{crit}$), however the F-value and F_{crit} are similar, indicating a less reliable result. Table 4-4 also shows the summary of results from ANOVA single factor analysis used to compare the elastic modulus obtained by methods from CSA and ASTM standards. The significance is indicated in the last column using ‘S’ and ‘NS’ for a significant and non-significant result, respectively. The results indicate that the method type used has a non-significant effect on the outcome of the elastic modulus.

Table 4-4 Results of ANOVA F-test evaluations

Evaluated Parameter	**Range of data for specimens**	**Source of variation**	**F-value**	**F_{crit}**	**Significance**
Peak compressive strength	With 0% FA	Gypsum content	39.51	3.47	S
	With 25% FA	Gypsum content	16.27	3.47	S
	With 50% FA	Gypsum content	0.78	3.47	NS
	With 0% gypsum	FA content	16.62	5.14	S
	With 5% gypsum	FA content	30.72	5.14	S
	With 10% gypsum	FA content	2.03	5.14	NS
	With 15% gypsum	FA content	1.8	5.14	NS
	With 20% gypsum	FA content	18.75	5.14	S
Axial strain at peak	With 0% FA	Gypsum content	1.98	3.47	NS
	With 25% FA	Gypsum content	2.77	3.47	NS
	With 50% FA	Gypsum content	1.61	3.47	NS
	With 0% gypsum	FA content	0.92	5.14	NS
	With 5% gypsum	FA content	3.76	5.14	NS
	With 10% gypsum	FA content	0.06	5.14	NS
	With 15% gypsum	FA content	0.81	5.14	NS
	With 20% gypsum	FA content	5.25	5.14	S
Elastic Modulus	All specimens	CSA vs ASTM method	0.02	4.2	NS

4.4 APPLICATION AND FUTURE RESEARCH

It should be mentioned that this study does not adhere to the standard composition requirements for sulfur trioxide (SO_3), according to the ASTM Standard Specification for Portland Cement C150/C150M (ASTM 2015). The standard references a maximum of 3.5% SO_3 for general use concrete. An analysis of the major elements by Li-borate fusion was done on the recycled fine gypsum material, showing an SO_3 content of 44.65%. Considering the cement already contains sufficient calcium sulfate activators (such as gypsum), any additional gypsum will exceed this limit. This study shows that having elevated gypsum content in concrete is feasible, however it is still in the initial stages of research and the complete mechanism and impacts are not fully understood. Therefore, appropriate caution should be exercised and/or additional testing done before application. Using this type of concrete mix has several positive aspects, although appropriate concerns are still present. Some general advantages and disadvantages for using recycled gypsum in concrete are summarized here:

4.4.1 Advantages

The main advantage of using recycled gypsum in concrete is the positive environmental impact. Carbon emissions are reduced due to the reduced demand for cement production by using a recycled material. In addition, partially replacing cement with fly ash, an industrial by-product, reduces the demand on virgin materials and lowers the overall carbon footprint. Fly ash is less expensive than Portland cement and already used in many concrete applications, showing a positive relationship with gypsum powder in concrete. It is presumed that contractors would be much more motivated to recycle gypsum from demolition projects if they had a direct use for the material in concrete mixes, potentially giving reason to develop more gypsum recycling facilities. These facilities could also export to other industries that use recycled gypsum, including the previously mentioned agriculture/farming, as well as for architectural/artistic applications, medical casts, drugs, toothpaste, cosmetics, and even as a food additive (Gratton and Beaudoin 2010) (Mentzer 2018) (Gypsum Association 2019).

Increasing gypsum recycling would also help keep the material out of landfills, where it can be dangerous. The sulfate (SO_4) portion of gypsum is particularly harmful when it gets wet and is dissolved into the leachates, conceivably contaminating nearby water supplies (Gratton and Beaudoin 2010) (Government of Canada 2009). Leachate analysis done by Zhang et al. (2017) proved that increasing the percentage of gypsum wallboard in landfills positively correlates to increasing sulfide levels. Sulfide is capable of forming harmful complexes with other metals or debris in landfills, and human exposure to hydrogen sulfide gas has been known to cause serious health effects (Gratton and Beaudoin 2010) (Construction and Demolition Recycling Association 2019). The variation of gypsum content showed a non-significant effect on the axial strain at peak load for all specimens, according to ANOVA single factor analysis. Finally, using recycled gypsum in concrete is suitable for applications where high early strength of concrete is not a requirement.

4.4.2 Disadvantages

Replacing cement with as little as 5% gypsum dehydrates concrete mixes and decreases the workability, which can be mitigated with the use of superplasticizer. A sudden and severe reduction in workability, referred to as a “false set”, occurred in mixes with 15% and 20% gypsum content. In addition, when only gypsum partially replaces cement in concrete mixes, reduced strength is observed at all ages in comparison to the control mix. Therefore, fly ash should also be incorporated when recycled gypsum is used in concrete. As mentioned, using fly ash in concrete shows low early strength and requires more time for strength development. Expansion of concrete containing gypsum has been of concern to previous researchers, reporting noticeable changes in diameter and length measurements

(Naik et al. 2010). However, this research has found no significant diameter changes to the accuracy of the measurement tools available (digital calipers and later a micrometer).

4.4.3 Future Research

The results of this study will be used to continue research evaluating the durability of specimens by exposing them to various environmental conditions after curing. The conditions considered include dry, submerged in fresh water, submerged in salt (ocean) water, and dry/wet cycles in both fresh and saltwater.

4.5 CONCLUSIONS

In this study, the effect of gypsum and fly ash as partial replacement for cement in concrete was experimentally studied. Fifteen different batches of concrete were prepared by replacing Portland cement with 0, 5, 10, 15, and 20% gypsum, and 0, 25, and 50% fly ash. Three identical cylindrical specimens from each batch were tested for compressive strength after moist curing for 7, 28 and 90 days. Based on the outcomes of the study, the following conclusions can be drawn:

- The positive relationship of fly ash and gypsum powder in concrete was highlighted by showing that either material combined with cement alone had inferior compressive strength, however when mixed together, a strength increase was observed at later ages.
- The effect of curing time is highly significant; results show that mixes containing fly ash and gypsum are slower to develop compressive strength than the 0% FA control mix containing only cement. After 90 days, mix FG5-FA25-C70 showed 15% higher strength than the 25% FA control mix.

- The most noteworthy conclusion considers mixes containing 50% FA. In this group, all mixes containing gypsum actually showed higher compressive strength than the 50% FA control mix after curing for 90 days. The highest strength was surprisingly the mix containing 20% gypsum, showing a 10% strength increase from the 50% FA control mix. This is appealing for projects aiming to be environmentally friendly and where high early strength is not a principle requirement.

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REFERENCES

Aimin, Xu, and Shondeep L. Sarkar. 1991. "Microstructural study of gypsum activated fly ash hydration in cement paste." *Cement and Concrete Research* (Pergamon Press plc.) 21: 1137-1147.

ASTM. 2015. "C136/C136M-14 Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates." *ASTM International.*

ASTM. 2015. "C150/150M- Standard Specification for Portland Cement." *ASTM International.*

ASTM. 2018. "C192/C192M-18 Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory." *ASTM International.*

ASTM. 2018. "C33/C33M-18: Standard Specification for Concrete Aggregates." *ASTM International.*

ASTM. 2016. "C39/C39M-16 Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens." *ASTM International.*

ASTM. 2019. "C469/C469M-14 Standard Test Method for Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression." *ASTM International.*

Batte, Marvin, and D. Lynn Forster. 2015. "Old is new again: the economics of agricultural gypsum use." *Journal of ASFMRA* 56-65.

Canadian Standards Association (CSA). 2004. "CAN/CSA-A23.3-04 Design of concrete structures." *Standards Council of Canada* (Canadian Standards Association).

Christensen, A. Norlund, Torben R. Jensen, and Jonathan C. Hanson. 2004. "Formation of ettringite, Ca6Al2(SO4)3(OH)12-26H2O, AFt, and monosulfate, Ca4Al2O6(SO4)-14H2O, AFm-14, in hydrothermal hydration of Portland cement and of calcium aluminum oxide—calcium sulfate dihydrate mixtures studied by in situ synchrotron X-ray." *Journal of Solid State Chemistry* 177: 1944-1951.

Construction and Demolition Recycling Association (CDRA). 2019. *Gypsum Drywall.* CDRA. April 4. https://cdrecycling.org/materials/gypsum-drywall/.

Government of Canada. 2009. *Guidelines for Canadian Drinking Water Quality: Guideline Technical Document- Sulphate.* 04 24. https://www.canada.ca/en/health-canada/services/publications/healthy-living/guidelines-canadian-drinking-water-quality-guideline-technical-document-sulphate.html.

Gratton, R., and G. Beaudoin. 2010. *Gypsum Recycling in the National Capital Region.* Ottawa: Construction Recycling Initiative (CRI)- National Capital Region.

Gypsum Association. 2019. *Other Uses of Gypsum.* Gypsum Association. https://www.gypsum.org/about/gypsum-101/uses-gypsum/.

Kozicki, Chris, and Carrie Carlson. 2019. *Gypsum Recycling.* Feeco International. https://feeco.com/gypsum-recycling/.

Marlay, Kyle Marie. 2011. *Thesis: Hardened Concrete Properties and Durability Assessment of High Volume Fly Ash Concrete.* St. Louis: Missouri University of Science and Technology.

Mentzer, A.P. 2018. *Uses for Gypsum Powder.* Sciencing. November 19. https://sciencing.com/uses-gypsum-powder-6395376.html.

Meyer, C. 2009. "The greening of the concrete industry." *Cement & Concrete Composites* 31: 601-605.

Naik, Tarun, Rakesh Kumar, Yoon-moon Chun, and Rudolph Kraus. 2010. *Utilization of Powdered Gypsum-Wallboard in Concrete.* Milwaukee: UWM Center for By-Products Utilization-University of Wisconsin.

Olson, Donald. 2001. "Gypsum." *U.S. Geological Survey Minerals Yearbook* 35.1-35.5.

Portland Cement Association. 2001. *Concrete Information - Ettringite formation and the performance of concrete.* Skokie, Illinois: Portland Cement Association (PCA).

Raghavendra, T., and B.C. Udayashankar. 2015. "Engineering properties of controlled low strength materials using flyash and waste gypsum wall boards." *Construction and Building Materials* 101: 548-557.

Recycling Council of Ontario . 2006. *Let's Climb Another Mole Hill - An examination of construction, demolition and renovation (CRD) waste diversion in Canada and associated greenhouse gas emission impoacts.* Toronto, Ontario: Recycling Council of Ontario .

Sata, Vanchai, Chai Jaturapitakkul, and Chaiyanunt Rattanashotinunt. 2010. "Compressive strength and heat evolution on concrete containing palm oil fuel ash." *Journal of Materials in Civil Engineering* (ASCE) 22 (10): 1033-1038.

Suarez, Sindy, Xavier Roca, and Santiago Gasso. 2016. "Product-specific life cycle assessment of recycled gypsum as a replacement for natural gypsum in ordinary Portland cement: application to the Spanish context." *Journal of Cleaner Production* 117: 150-159.

USA Gypsum. 2019. *10 Beneficial Ways To Use Recycled Gypsum.* Agri Marketing Inc. https://www.usagypsum.com/10-beneficial-ways-use-recycled-gypsum.

—. 2019. *Industrial Gypsum.* Agri Marketing Inc. March 26. https://www.usagypsum.com/gypsum-products/industrial-gypsum.

Wu, Zichao, and Tarun Naik. 2002. "Properties of concrete produced from multicomponent blnded cement." *Cement and Concrete Research* 32: 1937-1942.

CHAPTER 5 DURABILITY OF CONCRETE WITH FLY ASH AND GYPSUM AS SUPPLEMENTARY CEMENTING MATERIALS[3]

ABSTRACT

To address the negative contribution of cement production on global carbon emissions, this study aims to reduce the cement content typically required in concrete using partial replacement by more environmentally conscious and sustainable materials. In this experimental study, cement is partially replaced by weight with recycled gypsum powder (15%) and fly ash (50%). The durability was determined by exposing concrete specimens to various environmental conditions and testing for compressive strength at 1000, 3000 and 5000 hours. Five conditions were considered for specimen exposure, all maintained at room temperature. These conditions include dry (control), submerged in fresh water, submerged in seawater (saltwater), and two groups rotated weekly between dry and submerged in either fresh water or seawater. All concrete specimen groups continued to develop strength throughout the exposure periods, although the dry (control) specimens showed the smallest strength increase (16.9%) between 1000 and 5000 hours. Specimens in both fresh water conditions showed inferior compressive strength compared to the control condition at 1000 hours, however ultimately developed 5000-hour strength higher than control specimens, increased by 23.3% for specimens submerged in fresh water and 10.2% for specimens' subject to fresh water wet/dry cycles. Specimens in both seawater conditions showed continually higher compressive strength than the control specimens at all ages. Specimens submerged in seawater showed strength approximately 20% higher

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[3] This chapter will be submitted as a manuscript to a journal

than control specimens at all test ages. Specimens exposed to wetting and drying cycles in seawater showed the largest effect of exposure duration by continuously increasing strength throughout all duration periods, attaining strengths 40.1% and 53.4% higher than control at 3000 and 5000 hours, respectively. After 5000 hours, the lowest strength was observed in control (dry) specimens (35.03 MPa) and the largest strength was observed in specimens in wet/dry seawater condition (53.73 MPa). Results indicate that the concrete mix is considered durable, as strength was not adversely affected by exposure to the conditions considered.

5.1 INTRODUCTION

The production of Portland cement causes negative environmental impacts by releasing large amounts of carbon dioxide (CO_2) into the atmosphere, and appropriately many efforts are being made to reduce the cement needed in concrete (Naik et al. 2010) (Raghavendra and Udayashankar 2015) (Nguyen et al. 2018). Gypsum drywalls are extensively used in the building industry, and are correspondingly a major source of construction and demolition waste that must be disposed of. Gypsum waste can be effectively recycled, but at the present time it appears that there is a lack of motivation to do so, and it is often improperly disposed of in landfills (Construction and Demolition Recycling Association 2019) (Antunes et al. 2019). Disposing of gypsum waste in landfills with other organics and waste materials is likely to trigger adverse chemical reactions in wet environments, responsible for harmful effects to surrounding air and groundwater quality (Naik et al. 2010) (Raghavendra and Udayashankar 2015) (Gratton and Beaudoin 2010). Using recycled gypsum waste as a supplementary cementing material (SCM) reduces the demand for producing virgin materials (cement), saves precious landfill spaces, and keeps landfill sites unharmed. In previous studies considering gypsum in the cementitious material, it was identified that the incorporation of fly ash significantly improves the performance of concrete (Hansen and Sadeghian 2020) (Naik et al. 2010). It has been established that gypsum is essential in the production of Portland cement, commonly used as a set regulator at approximately 3-5% by weight (Naik et al. 2010) (Sharpe and Cork 2006). It is also reported that recycled gypsum can perform the same function in Portland cement (Chandara et al. 2009). However, using large amounts of gypsum has been reported to cause large expansions, and in some cases surface cracking. High gypsum content can also cause a ‘false set’ in fresh concrete mixtures, where the mix stiffens due to the rapid formation of large crystals of gypsum (Mehta and Monteiro 2014) (Naik et al. 2010) (Chun et al. 2008). When the false set is recognized, it can be disrupted by further mixing. Gypsum content in cement is currently limited by the ASTM Standard Specification for Portland Cement C150/150M (2015), which limits the SO_3 content to 3.5%. Gypsum has a particularly high SO_3 content, so amounts above those typically used in cement will exceed this limit (Mehta and Monteiro 2014). For this reason, research considering cement with high amounts of gypsum is infrequently studied, however this experimental research was designed to challenge the limit by going outside the accepted SO_3 range.

A handful of current studies exist using elevated gypsum in concrete, demonstrating promising results on the short-term behaviour of concrete containing recycled gypsum. However, present literature available does not consider long-term durability in any similar conditions to those selected for this study. Mohammed and Safiullah (2018) state that gypsum over the ordinary dosage may have long term negative consequences affecting the durability of concrete, potentially causing substantial damage. These researchers studied the effect of various amounts of gypsum (up to 9%) on properties of an Algerian Portland cement, and concluded gypsum content was optimized at 5.5%, which is above the current recommended value. Specimens were placed in a water bath to monitor for expansion, and results showed that gypsum contents above 7% yielded excessive swelling in water after 28 days (above 0.12 mm/m). Other properties of concrete including compressive strength, water demand, setting time, heat of hydration, and drying shrinkage were all analyzed up to a maximum of 28 days, however no durability measures were studied (Mohammed and Safiullah 2018). Raghavendra and Udayashankar (2015) studied controlled low strength materials using fly ash and waste gypsum wallboards, but only showed strength development up to 56 days and also did not monitor any durability parameters. Antunes et al. (2019) used gypsum from construction and demolition waste as a partial fine aggregate (sand) replacement in Portland cement mortar. They reported that acceptable compressive and flexural strengths can be accomplished when up to 30% ground gypsum waste is included in the aggregate portion, although once again, durability was not considered. Naik et al. (2010) studied concrete with up to 20% recycled gypsum and up to 60% fly as partial cement replacement. The study revealed promising results, indicating that gypsum could be used as a supplementary cementing material up to 10% weight replacement without adversely affecting the properties of concrete, with fly ash included in the SCM. The durability of specimens was tested based on length change and sulfate resistance up to 140 days, generally indicating inferior durability with increased gypsum content in comparison the control mix (Naik et al. 2010).

Durability of concrete is generally considered as its ability to resist the influences and effects of the environment, while performing its desired function (Hoff 1991).

Concrete is inherently resistant to serious deterioration from water, unlike other fundamental building materials such as wood and steel, making it an ideal building material for structures aiming to transport, store or restrain water. Its historical use by Romans in aqueducts and waterfront retaining walls is well known, and its application has translated it into a common construction material for dams, caissons, canal linings and other waterside structures (Mehta and Monteiro 2014). When excessive concrete shrinkage or expansion occur, the likelihood of infiltrating damaging substances is substantially increased. Accordingly, concrete durability is often considered in terms of the volume (or length) change.

The volume of concrete begins to change immediately upon contact with water, and a reasonable level of drying shrinkage is expected. Small amounts of expansion are also expected in concrete specimens' subject to wet/dry cycles, where expansion occurs during wetting and shrinkage occurs during drying. However, it is generally understood that excessive volume changes can cause cracking and potential loss of strength. Olivia and Nikraz (2011) reported that incorporating fly ash in cement blends produces less expansion and drying shrinkage. Volume changes are often monitored by length change, and ordinarily range from about 0.0001% to 0.1% in concrete, which is equivalent to a maximum of 1 mm per 1 m (Portland Cement Association 2001). Based on literature reviews, average length change values and values causing deleterious expansion can vary considerably between researchers based on many conditions. However, it is generally understood that length changes above approximately 0.04% are significant enough to have damaging effects, often consistent with visible cracking on the concrete surface (Naik et al. 2010) (Chun et al. 2008) (Hanhan 2004) (Shehata and Thomas 2000).

Supplementary cementing materials are commonly used to reduce the amount of cement required in concrete mixes. Among the most common of these materials is fly ash, which has been thoroughly studied and is regularly used in the construction industry. Fly ash is a by-product of coal-fired power plants and when incorporated in cement, it exemplifies industrial ecology because it reduces the environmental impact of two industries by integrating them together (Mehta and Monteiro 2014). Using fly ash in concrete is not only environmentally friendly, but it also reduces costs without sacrificing strength (Islam and Islam 2013). In fact, improved strength and durability characteristics have been reported by many researchers using fly ash, often identifying gypsum as an essential activator to promote strength development with fly ash. Although, it is well known in the concrete industry that cements blended with fly ash are slower to develop strength (Mehta and Monteiro 2014) (Marlay 2011) (Puvvadi and Moghal 2011) (Wu and Naik 2002) (Aimin and Sarkar 1991).

Aimin and Sarkar (1991) investigated the compressive strength of mortar cubes with fly ash replacing cement at 30% and 60%, considered as high-volume fly ash (HVFA), and added gypsum (not recycled). They concluded that the addition of 3% and 6% gypsum was beneficial to 28-day strength, although higher early strength was observed in specimens without gypsum. It was hypothesized that gypsum is not a fully effective activator until it reaches dissolved state. Puvvadi and Moghal (2011) also report that the hydration of fly ash is better in the presence of small amounts of gypsum (1%), determined to increase compressive strength at any lime content. These researchers also investigated the effect of soaking, and repeated wetting and drying cycles after curing. Soaking specimens showed a general reduction in strength, however incorporating gypsum minimized the strength loss from soaking. The strength loss from repeated cycles of wetting and drying was minimal for all specimens, and it is interesting to note that the strength showed a slight increase with the presence of gypsum. This is attributed to the development of cementitious complexes during the wetting and drying cycles (Puvvadi and Moghal 2011).

The durability and strength characteristics of concrete made with a blended cement including Class F fly ash have been studied extensively by Islam and Islam (2013), indicating the compressive strength of all mixes containing up to 50% fly ash were higher than the control mix after 90 days. This research revealed an optimal fly ash content of 30%, showing higher tensile and compressive strength than the control mix. Additionally, the study revealed that incorporating fly ash in concrete up to the optimum value achieves higher resistance against water permeability and can effectively reduce the corrosion of the internal steel reinforcement in comparison to the ordinary Portland cement mix (Islam and Islam 2013).

Early deterioration of concrete structures is common in marine environments exposed to seawater. This is primarily due to the corrosion of reinforcing steel, which occurs at a pH below 11. The pH value of seawater varies between 7.4 and 8.4, therefor impermeability is essential, and the cement must supply alkalinity in severe environments (Gani 1997). The long-term durability is therefore dependent upon the protection of the internal reinforcing steel. A passive layer with high pH from the cement is formed on the surface of steel and breaking down this layer by carbonation results in corrosion (Law et al. 2014). Chloride, magnesium, sodium, calcium and potassium are among the primary chemical constituent of seawater. The main mode of attack on concrete is crystallisation due to the reaction of magnesium sulphates, which is followed by the disintegration of concrete and eventual structural failure. Using proper concrete mix design and construction procedures, engineers are able to produce dense and impermeable concrete with sufficient cover to resist the attack of seawater on internal reinforcements (Wegian 2010). Incorporating fly ash in cements has been beneficial in wet environments. Concrete mixtures containing blended cements with fly ash and blast-furnace slag (geopolymer concretes) are well known for long term durability to sulfate and seawater attack in Europe and North America (Mehta and Monteiro 2014). Carbonation testing by Law et al. (2014) indicated that geopolymer concrete has an initially lower pH, but becomes higher than ordinary Portland cement after carbonation, and can accordingly provide necessary protection of reinforcing steel.

A study by Wegian (2010) considered three types of concrete mixing and curing conditions: mixed and cured in fresh water (control), mixed with fresh water and cured in seawater, and mixed and cured in seawater. The first two conditions are most relevant, as they also considered in the present study. Specimens mixed and cured in freshwater developed compressive strength up to 28 days, however strength was decreased after 90 days. Specimens mixed with fresh water and cured in seawater showed increased compressive strength development up to 14 days as compared to the control. In this group, the rate of strength gain decreased at ages over 28 days, and some specimens experienced a strength loss between 28 and 90 days (Wegian 2010). Water sources are important for proper concrete hydration, however almost all deteriorative mechanisms responsible for concrete degradation result from external water exposure. Water is required to transport aggressive substances known to cause deleterious reactions in concrete and corrosion of embedded steel, and abrasion is more severe in the presence of water (Hover 2011). Zhang et al. (2012) reported that one of the most aggressive environmental conditions suffered by concrete is drying and wetting cycles.

Using recycled gypsum and fly ash in concrete provides a safe and sustainable alternative use for these waste materials, ultimately lowering the environmental footprint of concrete production. Durability to repeated wetting and drying cycles is important for the construction of road embankments, geotechnical applications below water line, and in marine environments. It would be greatly beneficial to develop a durable concrete incorporating waste materials which maintains strength during prolonged exposure to salt and fresh water. The presented research aims to study the durability of concrete by monitoring compressive strength after exposure to certain environmental conditions that are common to concrete. This provides an alternative method of concrete durability testing that has not previously been considered by researchers using gypsum waste and fly ash as SCMs.

5.2 EXPERIMENTAL PROGRAM

The experimental program was carried out to test the durability of a selected concrete mix when exposed to various conditions by comparing compressive strength. After curing in a moist room for 28 days, specimens endured exposure periods of either 1000, 3000 or 5000 hours. The five conditions considered include dry, submerged in fresh water, submerged in seawater (saltwater) and weekly wet/dry cycles in both fresh and seawater. These exposure conditions were chosen to simulate some of the situations that concrete is exposed to in various environments.

The study consisted of five batches of the same concrete mix, containing 15% fine gypsum, 50% fly ash and 35% Portland cement, by weight. This ratio of cementitious materials was selected based on previous research (Hansen and Sadeghian 2020) as a representative mix due to its considerably high gypsum and fly ash content. The water to cement ratio (W/C) was kept to 0.48 in all mixes, including the liquid proportion from the superplasticizer (SP), which was used in each mix to maintain an adequate level of workability of fresh concrete. Each batch provided enough concrete to prepare nine cylindrical specimens, so a total of 45 concrete specimens were prepared. The mix proportions used for concrete are displayed in Table 5-1.

Table 5-1 Mix proportions for specimens tested for durability

Material	**Quantity (for 1 m^3)**
Water (L)	188
Fine Aggregate (kg)	575
Coarse Aggregate (kg)	1184
Gypsum (kg)	59
Fly Ash (kg)	198
Cement (kg)	138
Superplasticizer (% of water)	1.2

5.2.1 Material Properties

Type GU Portland cement was used in all concrete mixes (CRH Canada Group, ON, Canada). The bituminous coal fly ash was donated from a local contractor (Ocean Contractors, Halifax, NS, Canada). The superplasticizer used was ‘Plastol 6400’, a high range water reducing admixture donated by Euclid Chemical (Dartmouth, NS, Canada). Another local business (Casey Metro, Halifax, NS, Canada) supplied the coarse and fine aggregate, and both materials follow the specifications identified in ASTM C33/C33M

(2018). The coarse aggregate had a considerably small average moisture content (0.12%) and was therefore used in as-is condition in the concrete mixes. The fine aggregate showed an average moisture content of 3.39%, and was oven-dried and allowed to cool before use. The gypsum powder also showed a significantly high average moisture content of 18.29%, so it was also oven-dried before use. Fine aggregate and gypsum powder were stored in airtight containers during any periods after drying and before use, to keep the materials from absorbing any humidity. The gypsum material was supplied by a drywall recycling company, which processed drywall waste material into pieces with sizes ranging from 1/8 in (3.175 mm) to dust (USA Gypsum, Denver, PA, USA). When a sieve analysis was conducted on the original gypsum material from the recycling company according to ASTM C136/C136M (2015), it was found to have a particle size distribution more similar to sand than to Portland cement (referring back to Figure 3-1). During the same sieve analysis, bunches of fibre like particles were found to cluster together on sieves larger than the No. 100 sieve (0.149 mm opening). These particle clusters were previously discussed in Chapter 3, and photos of various sieves retaining gypsum and these clusters are shown in Figure 3-2. Rather than using this irregular and coarse recycled gypsum material, only the ‘fine gypsum’ powder was used as a partial cement replacement in the concrete mixes, with all particles passing sieve No. 50 (0.3 mm). Figure 5-1 shows a photo comparing the fine gypsum used in study to the coarse gypsum (including particle clusters), which was discarded.

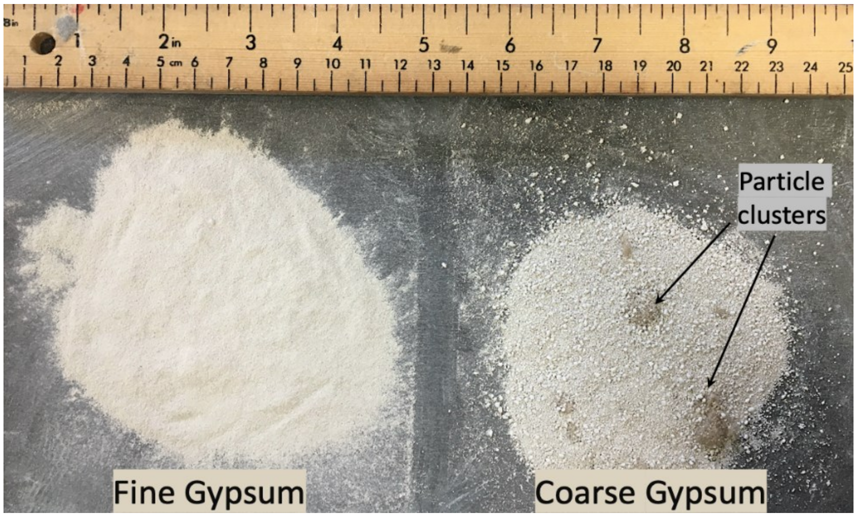

Figure 5-1 Comparison of fine and coarse gypsum material

The particle size analysis for both coarse (original) and fine (sieved) gypsum material, fly ash, Portland cement, and sand was previously reported in Chapter 4 (Figure 4-2). The particle sizes were evaluated using laser diffraction measurement techniques on a Mastersizer 3000 machine in the Department of Mining Engineering at Dalhousie University. Using this measurement system, a laser beam is passed by a group of particles and the angle and intensity of light scatter is analyzed (Panalytical 2020). The angle of light scattering shows an inversely proportional relationship with particle size, meaning small angles relative to the incident light indicate large particles and larger angles indicate smaller particle size (ATA Scientific Instruments 2018).

To compare and identify the chemical elements of each of the materials used in the cementitious portion of mixes, an analysis on the major elements and oxides was conducted using the lithium-tetraborate (Li-borate) fusion technique. This was also conducted in the Department of Mining Engineering at Dalhousie University, and the results are shown in Table 5-2. The nomenclature for each compound name can be found in the Abbreviations section. ASTM C150/150M (2015) presents the Standard Specification for Portland cement, including standard composition requirements. According to the standard, the cement used in this research adheres to all requirements for Type I cement except for the requirement regarding the maximum loss of ignition. The standard states that loss of ignition should be below 3% when limestone is not an ingredient, however the results presented indicate that the loss of ignition of the cement used was 7.21%. ASTM C618 (2017) presents the standard specification for using coal fly ash and raw or calcined natural pozzolans in concrete. According to the chemical composition requirements presented in the specification, the fly ash used conforms to all requirements.

Table 5-2 Major oxides or elements in fly ash, cement and fine gypsum

Oxide or Element	**Units**	**Fly Ash**	**Cement**	**Fine Gypsum**
Al_2O_3	Wt. %	20.87	4.00	0.77
BaO	Wt. %	0.09	0.02	<0.01
CaO	Wt. %	1.44	59.88	32.05
Cr_2O_3	Wt. %	0.02	0.01	<0.01
Fe_2O_3	Wt. %	6.55	2.34	0.35
K_2O	Wt. %	2.30	0.80	0.18
MgO	Wt. %	1.84	2.21	0.71
MnO	Wt. %	0.09	0.05	0.02
Na_2O	Wt. %	1.06	0.17	0.05
P_2O_5	Wt. %	0.15	0.10	0.03
SiO_2	Wt. %	59.39	19.54	3.80
S (as SO_3)	Wt. %	1.24	3.29	44.65
SrO	Wt. %	0.04	0.09	0.07
TiO_2	Wt. %	0.92	0.20	0.06
V_2O_5	Wt. %	0.94	0.01	< 0.01
ZrO_2	Wt. %	0.02	0.02	< 0.01
LOI (1000°C)	Wt. %	2.96	7.21	17.30
Total	Wt. %	99.94	99.93	100.04

The seawater used for the experiment was drawn from the Atlantic Ocean at the Halifax harbour waterfront, and the fresh water was taken from the tap, which is sourced from the Halifax municipal water supply. Water was kept in sealed plastic buckets at room temperature (approximately 22 °C) during exposure times. To obtain 28-day compressive strength of 35 MPa in non-air-entrained concrete, the corresponding water to cement ratio is reported to be 0.48 by weight (ACI 2000), which is accordingly used in this research.

5.2.1.1 Scanning Electron Microscopy

In order to gain a better understanding of the microstructure of the cementing materials being used, a scanning electron microscope (SEM) was used to analyze the shape, texture and particle size of fine (powdered) materials. SEMs use electrons with high energy in a focused beam to interact with atoms while scanning the surface of solid material particles. This generates a variety of signals containing information that reveals the electron-sample interactions, giving the ability to view particles less than 1 nm (Amidon et al. 2017).

Two general sizes of gypsum powder were analyzed with SEM: coarse and fine. The coarse gypsum material was used as supplied directly from a drywall recycler. When it was previously put through a sieve analysis, bunches of fibre-like material were identified, likely containing paper or other undesirable material particles, so this material was not used in the concrete mixes. Gypsum material retained on the No. 100 sieve (0.149 mm opening), No. 200 sieve (0.074 mm opening), and in the tray after sieve analysis was considered as the fine gypsum, and was used in the concrete mixes. The other materials analyzed were Portland cement and fly ash.

The SEM technique first requires small (milligram) quantities of material to be coated in epoxy. Each powder was placed in circular molds (pucks) approximately 20 mm in diameter, and a two-component epoxy was lightly mixed in with the powder. Subsequently, the specimens were placed in a pressure sealed curing container for two days to harden. Figure 5-2 (a) shows the molds after mixing epoxy with each of the four powder samples, and Figure 5-2 (b) shows the specimens curing. After specimens were fully hardened, the bottoms were polished to expose a random cross section. The cured and polished specimens were taken to the Scientific Imaging Suite in the Biology department at Dalhousie University to be gold plated. A gold sputtering of about 20 nm was applied to the polished side of each circular specimen. The specimens were then carefully transported to the lab with the SEM and camera apparatus, ensuring not to scrape or scuff the gold-plated end.

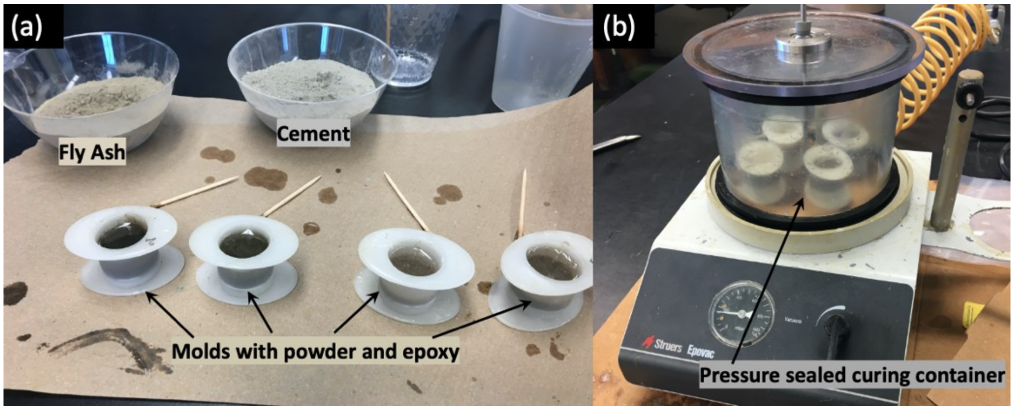

Figure 5-2 Epoxy pucks with powder materials during (a) preparation; (b) curing

Selected SEM photos are depicted at various magnifications in the following figures, showing each individual microstructure. In certain SEM images, rectangular dotted boxes are attached to arrows, representing a section of the image that is enlarged in another image, indicated by the pointing arrow. Figure 5-3 shows the fine gypsum particles (less than 0.3 mm), highlighting a dense pore structure surrounding a recognizable solid circular shape. Figure 5-4 shows the coarse gypsum particles, exhibiting two distinct configurations. Figure 5-4 (a) shows bunches of densely packed pores surrounding a more solid surface, and Figure 5-4 (b) shows a particularly different formation resembling a crystalline structure. The SEM images of Portland cement particles are shown in Figure 5-5. These particles also show densely connected pores surrounding a solid surface, as previously identified in the gypsum particles. The microstructure of fly ash particles is noticeably different, displayed in Figure 5-6, where solid rounded, almost spherical, shapes are noticed. It seems as though these bead-like grains are being surrounded by smaller interconnected particles that are not fully adhering to the grains.

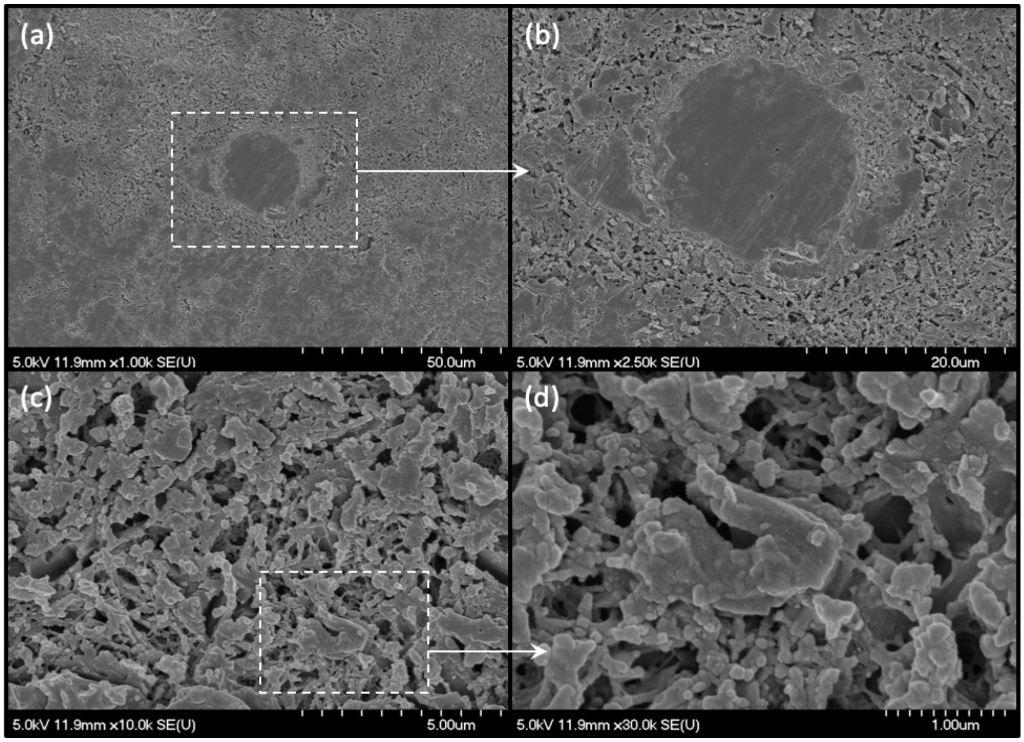

Figure 5-3 Fine gypsum particles under SEM magnified at: (a) 1000x (b) 2500x (c) 10,000x (d) 3000x

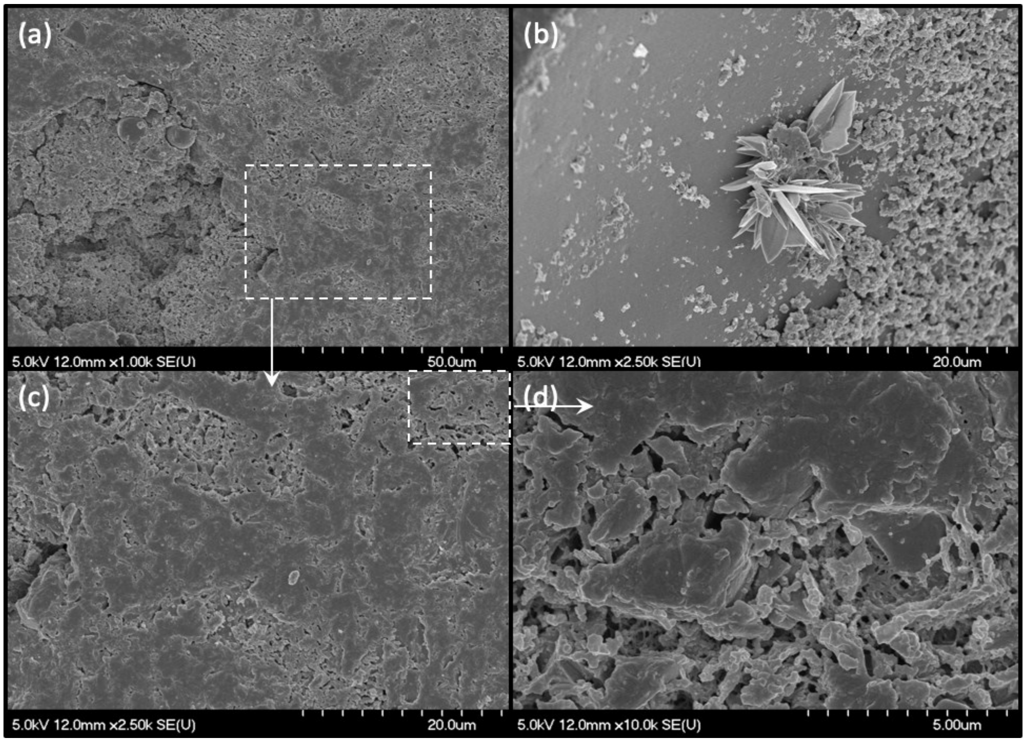

Figure 5-4 Coarse gypsum particles under SEM magnified at: (a) 1000x; (b) 2500x; (c) 2500x; (d) 10,000x

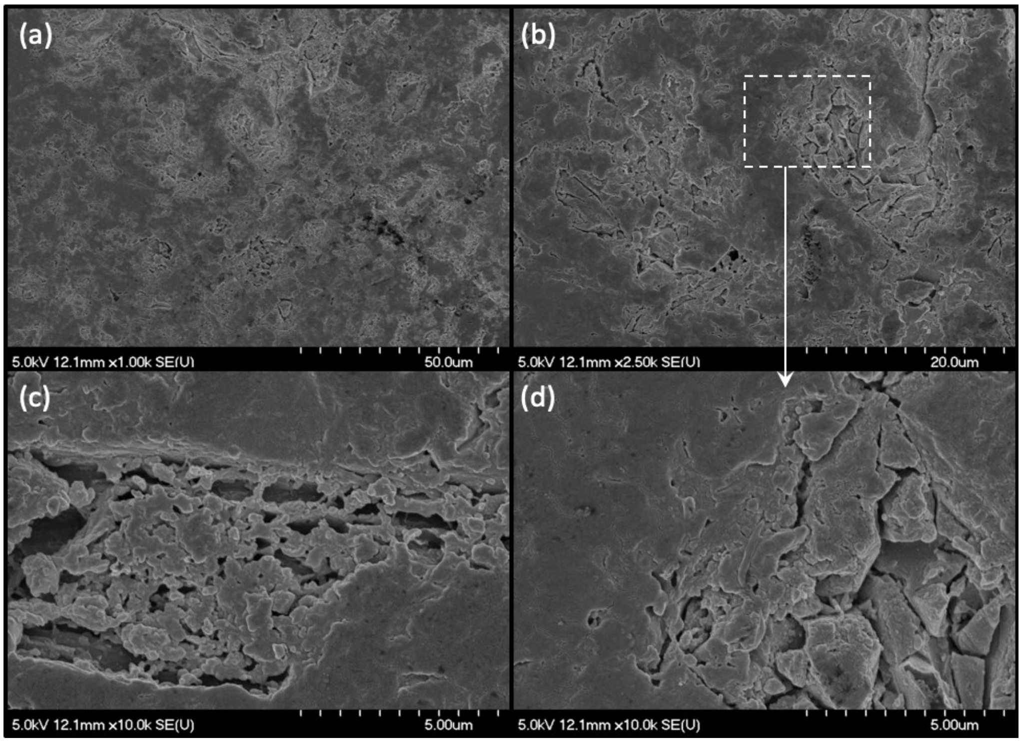

Figure 5-5 Portland cement particles under SEM magnified at: (a) 1000x; (b) 2500x; (c) 10,000x; (d) 10,000x

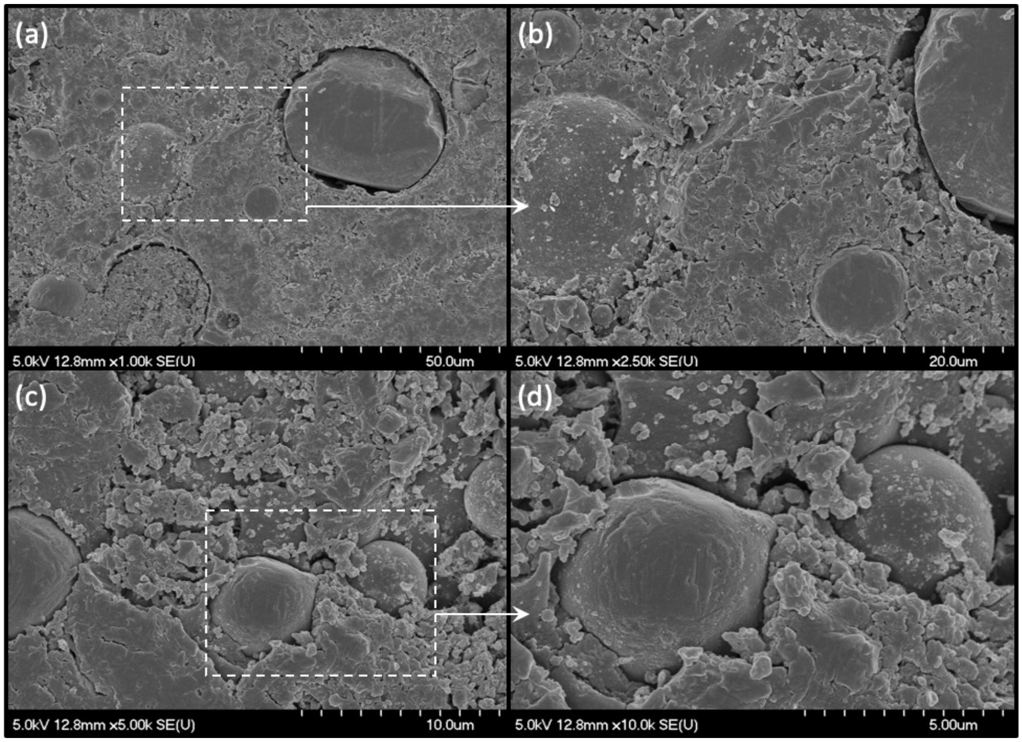

Figure 5-6 Fly ash particles under SEM magnified at: (a) 1000x; (b) 2500x; (c) 5000x; (d) 10,000x

5.2.2 Specimen Preparation

The concrete test specimens were prepared by following ASTM C192/C192M (2018). All the dry material was added to the mixer and allowed to mix with water and superplasticizer until a uniform texture was accomplished. In order to ensure that none of the dry materials were stuck to the sides and centre of the mixer, it was stopped periodically and manually scraped. To adhere to the recommendation of ASTM C192/C192M- Section 8.1.2, the superplasticizer was added to the water before mixing, instead of directly to the concrete. After the concrete was adequately mixed, cylindrical molds with a diameter of 100 mm and a height of 200 mm were filled and compacted by hand tamping, as required by the standard. Based on previous research experience mixing this type of concrete, it was deemed necessary to keep the mixer moving whenever possible to avoid hardening of the concrete. Intense stiffening of the fresh concrete in the mixer was previously observed after several minutes of remaining static, referred to as a 'false set'. The determination of slump was conducted on fresh concrete in according with ASTM C143/C143M (2015). The slump was measured on three of the five batches of identical concrete to obtain a representative average of 95.77 mm. Figure 5-7 shows the dry materials used in the concrete mixes.

Figure 5-7 Dry materials

The researcher noticed that specimens with fly ash had a longer set time than specimens containing only cement, meaning they did not fully solidify in the expected 24 hours. To mitigate this, the molds were removed after 5 days, differing from the ASTM specification which indicates removal from molds after 24 hours. Subsequently, specimens were cured in a moist room for 28 days, which was held at a measured humidity level between 93-96% at all times.

5.2.2.1 Environmental Exposure

After curing, specimens were labelled and placed into one of the five environmental exposure conditions, which were all held at room temperature (approximately 22 °C). Group E1 is the control group, which was left to dry for the entire duration of the exposure condition. Groups E2 and E3 were submerged in sealed buckets of water for the entire exposure period, with group E2 in fresh water (tap water), and group E3 in seawater (saltwater). Group E4 was submerged in sealed in buckets of fresh water for one week, then removed and allowed to dry for one week, and this process was repeated until the test date. This rotating wet/dry process was also applied to group E5, where specimens were placed in sealed buckets of saltwater for one week, and then removed and allowed to dry for the next week. Table 5-3 outlines the specimen groups that were conditioned in each of the exposure environments considered.

Table 5-3 Exposure environments for each specimen

Exposure Environment	Specimen ID	Number of Specimens
Dry (Control)	E1	9
Fresh Submerged	E2	9
Salt Submerged	E3	9
Fresh Wet/Dry	E4	9
Salt Wet/Dry	E5	9
Total		45

Figure 5-8 shows specimens being exposed to select environmental conditions, including (a) specimens left in dry condition, and (b) specimens submerged in buckets of seawater and fresh water. The buckets were sealed for the entire exposure duration, opened only when rotating select specimens between wetting and drying cycles. In Figure 5-8 (b), there are noticeable light-coloured salt deposits on the surface of specimens submerged in seawater. These salt deposits were not observed on specimens submerged in fresh water.

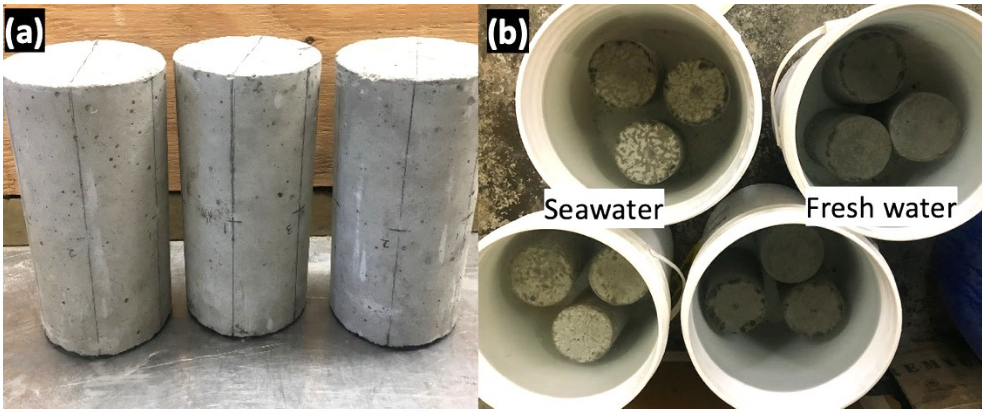

Figure 5-8 Specimens in exposure conditions including (a) dry; and (b) submerged in seawater and fresh water (note: the sealed covers were removed for taking photo).

5.2.3 Compression Tests

The procedure for the determination of compressive strength was in accordance with ASTM C39/C39M (2016). To ensure even loading, each end was capped using a sulfur capping compound and allowed to set for approximately three hours prior to testing. The specimens were tested using a universal testing machine that measured the maximum compressive load, with a spherical platen on the upper surface of the machine to minimize any accidental eccentricities. Figure 5-9 (a) shows specimens from all groups before being tested in compression after curing for 3000 hours, including the capping on cylinder ends. Figure 5-9 (b) shows a specimen in the testing machine prepared to be tested in compression. It is noted that groups E2 and E3 were submerged full time in fresh water and seawater, respectively, until the test date. These specimens were only allowed to dry for a couple hours before capping, so they appear to have a darker surface than other specimens. This is more noticeable in specimens from group E3 (submerged in seawater), where the darker surface colour was more pronounced and retained for longer after removal from the water compared to group E2 (submerged in fresh water). It should be noted that specimens rotated between dry and submerged weekly were tested in dry condition, likely representing a higher strength than if they were tested in moist condition.

Figure 5-9 (a) A group of specimens after environmental conditioning to be tested under compression; (b) specimen prepared for compression test

5.2.3.1 Diameter Change Measurements

To inspect for any expansion or shrinkage of concrete cylinders, researchers measured the diameter of many cylindrical specimens. The micrometer tool used measured to a precision of 0.0001 inches (25.4 μm), but only for lengths between 4 and 5 inches (101.6 mm – 127 mm). The cylindrical molds used to cast cylinders typically had diameters slightly larger than 4 inches, however due to the imperfectly circular cross sections, some diameters were under 4 inches and therefore immeasurable with the micrometer. The circular plane surface on each specimen's end was divided into six marked sections of 60°. Due to the inequality of cylinders' cross-sections, this division is not exact and was measured by hand as accurately as possible. The 60° sections on each end were connected along the cylinders' curved surface, and a '+' mark was made at approximately mid-height (100 mm). Three different diameters were marked per specimens, and the six '+' marks at mid-height were used as the repeated measurement points on each specimen. A total of five measurements was taken at each diameter on each day, and the averages were calculated. Figure 5-10 (a) shows a photo of the technique used by the researcher to measure diameters with the micrometer device, including the marks drawn onto the concrete specimens. Figure 5-10 (b) shows a schematic of a circular plane at the end of a cylinder that was divided into 60° marked sections to be measured at each of the three diameters (D1, D2, D3).

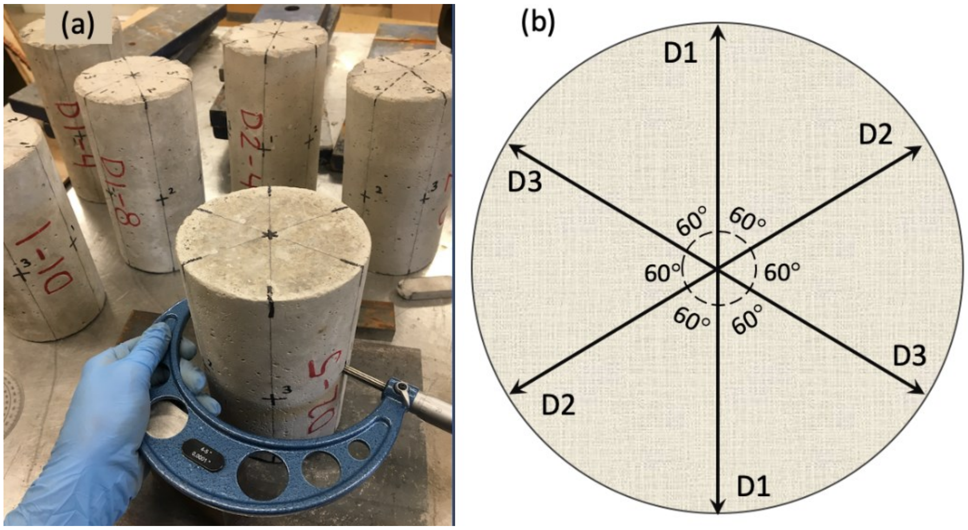

Figure 5-10 Measurement of cylinder diameters showing (a) actual setup; and (b) schematic

Measurements were made regularly on specimen groups exposed to three of the different exposure conditions: dry, and rotating weekly between dry and submerged in either fresh water or seawater. The first group includes six specimens left to dry at room temperature. Since group E1 contains specimens in dry condition, three of these specimens were measured after curing for 28 days, and then again on days 30, 32, 35, and 40. In effort to recognize any diameter changes that occurred in dry concrete directly after being removed from the molds, three specimens were mixed specifically to be measured in dry condition without the 28-day curing period. These specimens were labelled Group M, and their diameters were measured on day 5 (after removal from mold) and then again on days

7, 10, 14, 19, 24, 35, 48, 60, 80, and 100. It is noted that all measurements were made within +/- 1 day from the date stated. The second group measured included three specimens that were rotated weekly between being submerged in fresh water for a week and left to dry the following week (E4). The third group includes three different specimens that were also rotated weekly, but they were submerged in seawater and then left to dry (E5). Measurements were made on specimens immediately after being submerged in water for a week, and then after drying out for one full week. That is, 'wet' measurements were taken bi-weekly and 'dry' measurements were also taken bi-weekly, on opposing weeks. Measurements were taken for nine consecutive weeks. Summary tables and all raw data of diameter measurements recorded for groups M, E1, E4 and E5 are tabulated in Appendix B, including calculations for the average and standard deviation of the five measurements taken at each diameter.

5.3 RESULTS AND DISCUSSION

5.3.1 Compressive Behaviour

After curing for 28 days, specimens were moved to one of five selected exposure conditions. Specimens were tested for unconfined compressive strength under uniaxial compressive loading until failure after exposure periods of 1000, 3000 and 5000 hours. Results are presented in Table 5-4, including the coefficient of variation (CV) between the three specimens tested.

Table 5-4 Compression test results after 1000, 3000, and 5000 hours of exposure

	1000 hrs		**3000 hours**		**5000 hours**	
Condition	**Average Strength (MPa)**	**CV (%)**	**Average Strength (MPa)**	**CV (%)**	**Average Strength (MPa)**	**CV (%)**
Dry (Control)	29.98	8.32	34.00	4.91	35.03	6.70
Sub. Fresh	26.77	5.52	40.94	3.81	43.20	4.41
Sub. Salt	35.88	4.78	41.55	6.21	42.49	2.85
Wet/Dry Fresh	28.66	9.46	31.78	3.63	38.62	5.34
Wet/Dry Salt	35.74	2.70	47.64	5.75	53.73	4.64

All specimens failed in compression in comparable manners, with micro-cracking observed on the surface of all specimens after failure. Both longitudinal (vertical) and transvers (horizontal) cracking was revealed to develop into larger diagonal cracking. Spalling of concrete was visible on all specimens to some extent, more apparent in areas near cracking. After the peak load was attained, the testing machine was promptly stopped, and the load removed. If not stopped, the specimen was subject to considerable loading past its peak, and expectantly cracking became much more severe and complete fracture and crumbling of concrete would occur. Often, the fracturing of concrete was loud and sudden as the peak load was attained, especially noticeable at higher strengths. Photos of a specimen from each group after failing in compression during 5000-hour tests are shown in Figure 5-11, all having the load removed promptly after reaching the peak strength. Some large pieces of concrete ruptured from the sides of specimens in an explosive reaction, especially noticeable at higher strengths, observed in Figure 5-11 (b), (c) and (e).

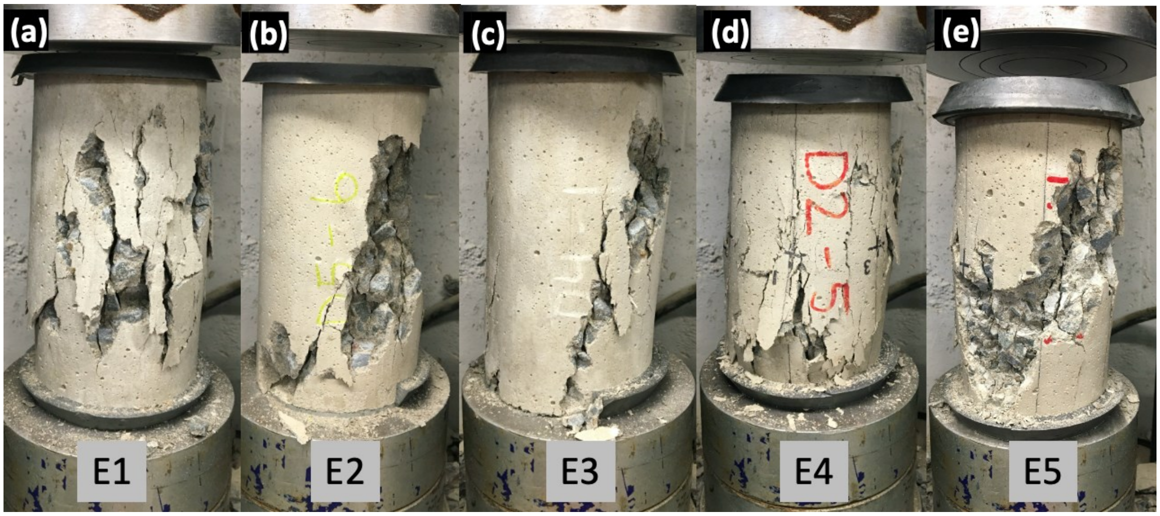

Figure 5-11 Specimens after compression tests following 5000-hour exposure to conditions: (a) dry; (b) submerged in fresh water; (c) submerged in saltwater; (d) wet/dry fresh water; (e) wet/dry saltwater

5.3.2 Effect of Exposure Duration

Figure 5-12 shows the compressive strength of specimens after varying exposure durations as a function of the environmental condition endured. Error bars represent the positive and negative standard deviation of three identical specimens tested at each time. All specimens indicated a positive effect with increased exposure duration, as strength continued to improve between all periods tested.

Of all exposure conditions considered, the concrete proved to be least durable in dry (control) condition. The dry specimens showed the lowest effect of exposure duration, indicated by the smallest strength increase between tests, rising only 16.9% from 1000 to 5000 hours. This group also showed the lowest average 5000-hour strength (35.03 MPa). The duration of exposure showed the most significant effect on specimens subject to wet/dry saltwater condtions. This group showed a steady compressive strength increase throughout conditioning, increasing 33.3% between 1000 and 3000 hours, and another 12.8% between 3000 and 5000 hours.

After being subject to the conditions for 1000 hours (42 days), the highest compressive strength was observed in specimens subject to saltwater conditions. Groups submerged in saltwater and subject the wet/dry saltwater cycles both showed 1000-hour strength of about 36 MPa. After conditioning for 3000 hours (125 days), specimens from groups in saltwater conditions continue to show higher compressive strength than other specimens. There was a notable strength gain between 1000 and 3000 hours for specimens submerged in fresh water, increasing 52.9%, from 26.77 MPa to 40.94 MPa. The strength gain observed in all specimens between 1000 and 3000 hours is likely due to the delayed pozzolanic reaction occuring with fly ash, which is known to incur strength at later ages. Minimal strength increase (less than 6%) was observed in specimen groups that were not touched between 3000 and 5000 hours of exposure, that is the dry and both submerged conditions. However, specimens subject to weekly wet/dry cycles continued to increase compressive strength between 3000 and 5000 hours for both fresh water and saltwater conditions. This may indicate that the wetting and drying process is advantageous to strength for this type of concrete. After 5000 hours (208 days) of exposure, all specimens developed compressive strength of at least 35 MPa. The highest overall strength observed was 53.73 MPa for specimens subject to wet/dry saltwater conditions, over 10 MPa higher than all other groups.

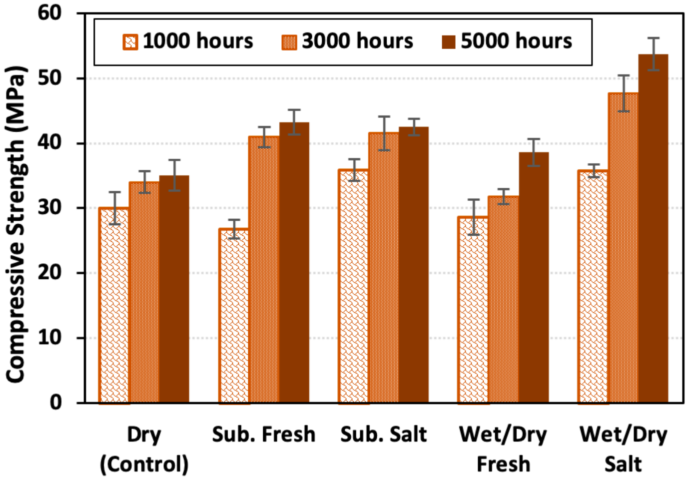

Figure 5-12 Compressive strength after exposure for 1000, 3000, and 5000 hours

Figure 5-13 was produced to more clearly compare the effect of each of the wet exposure conditions to the dry (control) condition. The average strength of the specimens from each group (f'_c) was divided over the average strength the control specimens ($f'_{c\text{-}control}$) for each duration, including error bars representing the standard deviation between specimens. The horizontal line along ($f'_c / f'_{c\text{-}control}$) = 1.0 maintains the performance of the control group, so results above and below 1.0 indicate increased and decreased performance, respectively.

After being subject to the conditions for 1000 hours, specimen groups in fresh water conditions showed inferior compressive strength compared to the control. Specimens submerged in freshwater and rotated in fresh water wet/dry cycles showed strength decreases of 10.7% and 4.4%, respectively, in comparison to control specimens. Despite showing low early strength, specimens submerged in fresh water full-time developed higher strength than control specimens at later ages, increased by 20.4% and 23.3% at 3000 and 5000 hours, respectively. Between 1000 and 3000 hours, specimens in fresh water wet/dry cycles showed a slight decrease in strength compared to control specimens, however it was deemed insignificant considering the overlapping standard deviations. There was a clear strength development for this group between 3000 and 5000 hours, rising 10.2% above the strength of the control group after 5000 hours of wet/dry exposure in fresh water.

Specimen groups in saltwater conditions showed continuously higher strength than control specimens at all ages. Specimens submerged in saltwater full-time showed $f'_c / f'_{c\text{-}control}$ approximately constant around 1.2 at all ages (20% higher than control), indicating that this exposure condition is beneficial to strength development at early ages, but does not have significant effect on strength after 1000 hours of exposure. Specimens subject to wet/dry saltwater conditions showed the largest strength developed between exposure durations, showing $f'_c / f'_{c\text{-}control}$ values notably higher than all other groups after 3000 and 5000 hours of exposure. In comparison to control, this group showed strength improving by 19.2%, 40.1% and 53.4% at 1000, 3000 and 5000 hours, respectively. After 5000 hours, all specimens outperformed the control group. It can therefore be concluded that exposure to wet conditions is beneficial to compressive strength at later ages for the studied concrete type. Specimens in saltwater conditions consistently outperformed the control group, with more obvious strength increase in specimens subject to wetting and drying. Additional strength gain in saltwater conditions may be the result of beneficial chemical complexes being formed with saltwater and concrete.

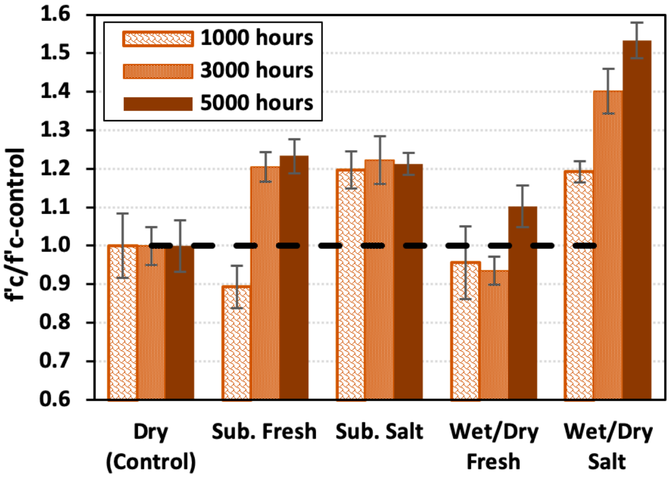

Figure 5-13 Compressive strength comparison of each exposure to dry (control) condition

5.3.3 Diameter Change

Undesirable and excessive expansion in concrete containing large amounts of gypsum has been reported as a concern by many researchers (Nixon 1978) (Naik et al. 2010) (Marlay 2011) (Mehta and Monteiro 2014). With this in mind, the researchers attempted to monitor any diameter changes in concrete specimens with the use of a micrometer. This tool was used after measurements with digital calipers proved to be unreliable in previous research. This method of measurement is not consistent with any specific standard and is considered an approximate method of measurement with considerable possibility of human error. The maximum total shrinkage measured in this research was 0.026% between day 5 and day 100. This calculation can be found in Appendix B. None of the concrete specimens showed surface cracking prior to testing, which is typical of specimens that have experienced excessive shrinkage or expansion. Although the measured shrinkage was acceptably small, this data should not be relied on for definitive expansion and shrinkage measurements.

To compare specimen's diameter changes after time to the original average diameter (*Do*), all diameter measurements taken after various times of exposure duration (*Dt*) were divided by the initial average diameter measured; that is (*Dt/Do*). This ratio is shown as the vertical axis on the following figures, with days along the horizontal axis. Results with (*Dt/Do)* above 1 indicate that the average specimen diameter expanded, and (*Dt/Do)* results below 1 indicate that the specimen diameter shrunk. Figure 5-14 shows the diameter changes observed in dry specimens, including group M (made specifically for measuring), and group E1 (control specimens). Both groups show a continuous decline in diameter length observed over time in comparison to the initial average diameter (*Do*), indicating shrinkage of the concrete cylinder. However, this may not truly be the case, as testing error is assumed to have occurred during continual use of the micrometer fixture. In order to make an accurate measurement with the micrometer, two metal bars have to be clicked to tighten fixture into place at the exact location of the marked (+) locations. Since metal is harder than concrete, slight grinding of the concrete surface was occasionally observed when the metal micrometer bars were clicked into place. The contact made between the concrete and the metal was sufficient to detach fine concrete particles (dust) from the cylinders surface, thereby reducing the diameter at the location where specimens were continually measured, resulting in an inaccuracy of reported measurements. The results are not compatible with published principles indicating that increasing the gypsum content, and therefore the SO_3 content, is likely to cause expansion (ASTM 2019) (ASTM 2015) (Naik et al. 2010).

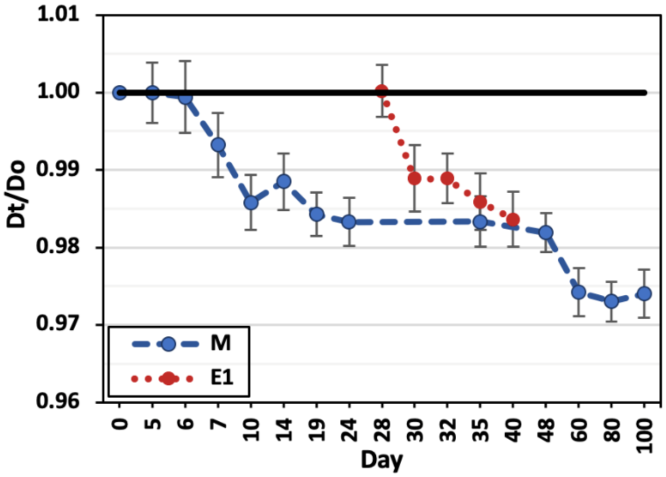

Figure 5-14 Diameter measurements of dry specimens from groups M and E1

Figure 5-15 shows the diameter changes observed in specimens that were rotated weekly in wet (submerged) and dry cycles. Figure 5-15 (a) presents data measured from specimens submerged in fresh water bi-weekly and Figure 5-15 (b) shows data from specimens submerged in saltwater bi-weekly. In both graphs, it can be seen that the diameter of specimens measured at the end of the wet cycle were continually larger than specimens measured at the end of the dry cycle. This was anticipated based on the established knowledge that concrete expands in wet conditions and shrinks while drying. At day 21, expansion was observed in specimens at the end of both wet cycles (fresh and salt), as well as in saltwater specimens at the end of the dry cycle. At all times after day 21, a steady decline in diameter length is observed in all specimens. This is likely attributed to the previously stated assumption that testing error has occurred while measuring, unintentionally reducing the diameters.

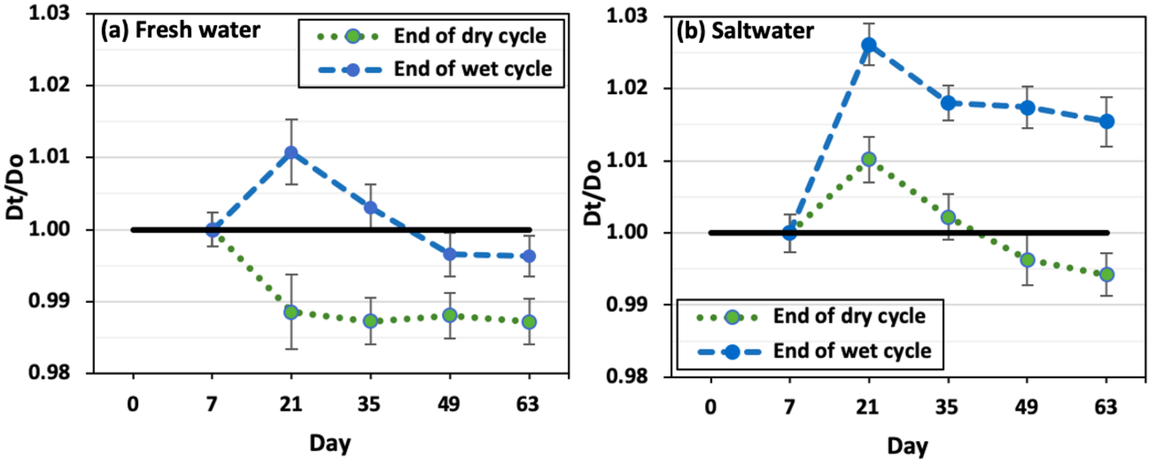

Figure 5-15 Diameter measurements of specimens at the end of wet/dry cycles in (a) fresh water; (b) saltwater

5.4 DISCORDANCE WITH STANDARD FOR SO_3 CONTENT

As mentioned, this study does not adhere to the standard composition requirements for sulfur trioxide (SO_3), according to ASTM C150/C150M (2015). The standard references a maximum of 3% or 3.5% SO_3 content for general use concrete, with the higher value accepted when C_3A is more than 8%. The percentage of C_3A is calculated based on chemical analysis involving a simple calculation using the percentages of aluminum oxide (Al_2O_3) and ferric oxide (Fe_2O_3). According to this chemical analysis calculation, the C_3A content was determined to be 24.65%. Details of the calculation can be found in Appendix C, which were made based on the process laid out in ASTM C150/C150M (2015). The standard also states that this SO_3 limit may be surpassed if it can be demonstrated that expansion exceeding 0.020% will not develop, tested according to ASTM C1038/C1038M (2019). This test method determines the expansion amount of a mortar bar when it is stored in water, and states that excess sulfate may be a cause excessive expansion. This test was not performed during this research as the necessary equipment was not available. The cementitious materials used in this research were analyzed by Li-borate fusion to determine their major elements and oxides. Through this analysis, SO_3 content by weight was reported as 1.24%, 3.29% and 44.65%, for fly ash, cement and gypsum, respectively. The cementitious content of the mix design used in this study contains 50% fly ash, 35% cement, and 15% gypsum. This combines to a total SO_3 content of 8.47%, over double the specified limit. The effect of SO_3 content on concrete was previously discussed in the literature review.

This experimental research focuses on compressive strength parameters, showing promising results in this regard. Although expansion of specimens was not able to be precisely measured, it is recognized that if any expansion did occur, it was not sufficient to produce visible microcracking in concrete prior to testing. This suggests that gypsum can be implicated as part of the cementitious material with inconsequential effects to compressive strength.

5.5 RECOMMENDATIONS FOR FUTURE RESEARCH

Concrete is utilized in many harsh environments, so it is recommended that durability to other environmental conditions be considered, such as cycles of heating and cooling or freezing and thawing. It would also beneficial to monitor the resistance to common chemical attacks, including attacks from sulfates, chlorides or alkalis. Future testing should similarly include saltwater and fresh water wet/dry conditions, as improved strength was observed in this research for specimens exposed to wet conditions. Durability testing beyond 5000 hours is also of interest.

Only one mix design was considered in this study, however it is recommended that future research compares mix designs with different fly ash and gypsum contents in the cementitious material, including a control mix with only Portland cement. As inordinate expansions have been reported in the literature at high gypsum contents (Naik et. al 2010) (Marlay 2011) (Mehta and Monteiro 2014), it could be useful to study the confined strength of this concrete type, such as in concrete-filled fibre-reinforced polymer tubes (CFFTs). Larger-scale tests are also of interest, including beams, columns and slabs. Flexural strength testing should also be considered.

It is recommended that future researchers use a more precise measuring device that is able to monitor expansion and shrinkage more accurately. The standard test method existing for measuring expansion of hydraulic cement mortar bars stored in water (ASTM C1038) requires a length comparator that conforms to apparatus specification requirements outlined in ASTM C490, which was not available at the time of research. It is recommended that length changes are not only monitored in specimens stored in water, but also in dry specimens, as previous research indicates that concrete with gypsum can show increased expansion in water.

5.6 CONCLUSIONS

In this research, concrete containing gypsum powder recycled from waste drywalls (15% by weight) combined with fly ash (50% by weight) as partial cement replacements was investigated for durability to different wet and dry conditions. Specimens were exposed to one of five environmental conditions, namely: continuously dry (control), continuously submerged in seawater and fresh water, and rotating weekly between dry and submerged seawater and fresh water. Durability of concrete to these conditions was evaluated using the average compressive strength of three specimens tested after exposure for 1000, 3000, and 5000 hours. The following conclusions were drawn from the presented research:

- Compressive strength was not adversely affected by exposure to any of the environmental conditions considered. Strength increases were observed in all specimens after prolonged exposure, indicating that concrete strength and durability was maintained up to at least 5000 hours.
- Specimens left to dry (control) showed the smallest effect of exposure duration, developing minimal strength during conditioning periods. Between 1000 and 5000 hours, this group showed the smallest strength increase (16.9%), attaining the lowest overall compressive strength after the full exposure duration (35.03 MPa). This indicates a beneficial relationship between water exposure and compressive strength of concrete.
- In comparison to the control, specimens in fresh water conditions showed inferior compressive strength after 1000 hours of exposure, but greater strength after exposure for the maximum duration (5000 hours). After 5000 hours, specimens submerged in fresh water and specimens in fresh water wet/dry cycles showed strength increases of 23.3% and 10.2%, respectively, when compared to the control.
- Specimens in saltwater conditions showed higher strength at all test ages in comparison to the control. Specimens submerged in saltwater maintained compressive strength approximately 20% higher than the control group throughout the entire exposure duration. Specimens rotated weekly between dry and submerged in saltwater showed the most significant effect of exposure duration, continually improving strength throughout exposure. After 3000 and 5000 hours, specimens in wet/dry saltwater condition attained compressive strengths 40.1% and 53.4% higher than the

control, respectively. It was speculated that cementitious complexes were formed during wetting and drying cycles in concrete exposed to seawater.

- Cylinder diameters were measured periodically in dry (control) condition, as well as at the end of dry and wet cycles in both fresh and saltwater, to monitor for any changes. Specimens in dry condition generally showed a continuous reduction in diameter, potentially attributed to error incurred with the measuring device used. As expected, specimens measured at the end of wet cycles had larger diameters than specimens measured at the end of dry cycles.
- Before the possibility of using this type of concrete can become a practical reality, more in-depth investigations should be conducted. Recommendations for future research include varying the cementitious content, exposure to other environments, application of other test methods, and using a more precise measuring device to monitor volume changes.

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REFERENCES

Aimin, Xu, and S. L. Sarkar. 1991. "Microstructural study of gypsum activated fly ash hydration in cement paste." *Cement and Concrete Research* (Pergamon Press plc.) 21: 1137-1147.

American Concrete Institute (ACI). 2000. *ACI Manual of Concrete Practice, Part 1: Materials and General Properties of Concrete.* Manual, Farmington Hills, MI: American Concrete Institute.

Amidon, G. E., P. J. Meyer, and D. M. Mudie. 2017. "Chapter 10 - Particle, Powder, and Compact Characterization." *Developing Solid Oral Dosage Forms - Pharmaceutical Theory and Practice* (Academic Press) Second Edition: 271-293.

Antunes, M. L., A. Botignon de Sa, P. S. Oliveira, and E. C. Rangel. 2019. "Utilization of gypsum from construction and demolition waste in Portland cement mortar." *Ceramica* 65 (1): 1-6.

ASTM. 2019. "C1038/C1038M - 19 Standard Test Method for Expansion of Hydraulic Cement Mortar Bars Stored in Water." *ASTM International.*

ASTM. 2015. "C136/C136M-14 Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates." *ASTM International.*

ASTM. 2015. "C143/C143M - 15a Standard Test Method for Slump of Hydraulic-Cement Concrete." *ASTM International.*

ASTM. 2015. "C150/150M - 15 Standard Specification for Portland Cement." *ASTM International.*

ASTM. 2018. "C192/C192M - 18 Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory." *ASTM International.*

ASTM. 2018. "C33/C33M - 18 Standard Specification for Concrete Aggregates." *ASTM International.*

ASTM. 2016. "C39/C39M - 16 Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens." *ASTM International.*

ASTM. 2017. "C490/C490M - 17 Standard Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete." *ASTM International.*

ASTM. 2017. "C618 - 17a Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete." *ASTM International.*

Chandara, Chea, Khairun Azizli, Zainal Ahmad, and Etsuo Sakai. 2009. "Use of waste gypsum to replace natural gypsum as set retarders in portland cement." *Waste Management* (Elsevier) (29): 1675-1679.

Chun, Yoon-moon, Tarun Naik, and Rudolph Kraus. 2008. *Recycling Opportunity for Gypsum Wallboard in Concrete.* Solid waste reserach program, Milwaukee: University of Wisconsin.

Construction and Demolition Recycling Association (CDRA). 2019. *Gypsum Drywall.* CDRA. April 4. https://cdrecycling.org/materials/gypsum-drywall/.

Gani, M.S.J. 1997. "Cement and concrete." In *Cement and concrete: 1st edition*, 49-169. England: Chapman and Hills.

Gratton, Renee, and Guy Beaudoin. 2010. *Gypsum Recycling in the National Capital Region.* Ottawa: The Construction Recycling Initiative.

Hanhan, Amin A. 2004. *Influence of the SO3 content of cement on the durability and strength of concrete exposed to sodium sulfate environment.* Graduate Thesis and

Dissertation, Department of Civil and Environmental Engineering, University of South Florida, Tampa, Florida: Scholar Commons.

Hansen, Sarah, and Pedram Sadeghian. 2020. "Recycled gypsum for partial cement replacement in concrete." *Journal of Cleaner Production (In review).*

Hoff, G. 1991. "Durability of offshore and marine concrete structures." *2nd International Conference (ACI SP-127).* Montreal, Canada: American Concrete Institute. 33-64.

Hover, Kenneth. 2011. "The influence of water on the performance of concrete." *Construction and Building Materials* (Elsevier) 25: 3003-3013.

Islam, M. M., and M.S. Islam. 2013. "Strength and durability chracteristics of concrete made with fly-ash blended cement." *Australian Journal of Structural Engineering* (Institution of Engineers Australia) 14 (3): 2204-2261.

Law, David, Andi Arham Adam, Thomas K. Molyneaux, Indubhushan Patnaikuni, and Arie Wardhono. 2014. "Long term durability properties of class F fly ash geopolymer concrete." *Materials and Structures* 48: 721-731.

Marlay, Kyle Marie. 2011. *Thesis: Hardened concrete properties and durability assessment of high volume fly ash concrete.* St. Louis: Missouri University of Science and Technology.

Mehta, P. Kumar, and Paulo J.M. Monteiro. 2014. "Concrete: microstructure, properties, and materials (fourth edition)." (McGraw-Hill Education).

Mohammed, Siline, and Omary Safiullah. 2018. "Optimization of the SO3 content of an Algerian Portland cement: Study on the effect of various amounts of gypsum on cement properties." *Construction and Building Materials* 164: 363-370.

Naik, Turan, Rakesh Kumar, Yoon-moon Chun, and Rudolph Kraus. 2010. *Utilization of Powdered Gypsum-Wallboard in Concrete.* Milwaukee: University of Wisconsin.

Nguyen, Long, Alexander J. Moseson, Yaghoob Farnam, and Sabrina Spatari. 2018. "Effects of composition and transportation logistics on environmental energy and cost metrics for the production of alternative cementitious binders." *Journal of Cleaner Production* (Elsevier) 185: 628-645.

Nixon, P. J. 1978. *Recycled Concrete as an Aggregate for Concrete - A Review.* Great Britain: 37-DRC Committee (Demolition and Re-Use of Concrete.

Olivia, Monita, and Hamid Nikraz. 2011. "Properties of fly ash geopolymer concrete designed by Taguchi method." *Materials and Design* (Elsevier) 36: 191-198.

Panalytical, Malvern. 2020. *Mastersizer 3000.* Worcestershire, United Kingdom: Spectris Company.

Puvvadi, Sivapullaiah, and Arif Ali Baig Moghal. 2011. "Role of gypsum in the strength development of fly ashes with lime." *Journal of Materials in Civil Engineering* (ASCE) 23 (2): 197-206.

Raghavendra, T., and B.C. Udayashankar. 2015. "Engineering properties of controlled low strength materials using flyash and waste gypsum wall boards." *Construction and Building Materials* 101: 548-557.

Sharpe, Roger, and Greg Cork. 2006. "Gypsum and Anhydrite." *Industrial Minerals and Rocks* (Society for Mining, Metallurgy, and Exploration, Inc.) (7th edition): 519-540.

Shehata, Medhat, and Michael Thomas. 2000. "The effect of fly ash composition on the expansion of concrete due to alkali-silica reaction." *Cement and Concrete Research* (Pergamon) 30: 1063-1072.

Wegian, Falah M. 2010. "Effect of seawater for mixing and curing on structural concrete." *The IES Journal Part A: Civil & Structural Engineering* 3 (4): 235-243.

Wu, Zichao, and Tarun R. Naik. 2002. "Properties of concrete produced from multicomponent blended cements." *Cement and Concrete Research* 32: 1937-1942

Zhang, Jun, Yuan Gao, Yudong Han, and Wei Sun. 2012. "Shrinkage and interior humidity of concrete under dry-wet cycles." *Drying Technology International Journal* (Taylor & Francis) 30 (6): 583-596.

CHAPTER 6 CONCLUSIONS AND RECOMMENDATIONS

6.1 CONCLUSIONS

In this thesis, three phases of research were presented investigating the potential of using recycled gypsum material as a partial cement replacement. The primary objective was to design a more sustainable and environmentally friendly solution to traditional concrete by using recycled gypsum, and to provide sufficient experimental evidence that adequate strength and durability is maintained. In the first phase of research, eight mortar mixtures containing different combinations of cementitious material (including cement, gypsum and fly ash) were tested for compressive strength after curing for 3, 7, 28, and 56 days. In the second phase of research, fifteen different concrete mixtures were prepared containing 0, 5, 10, 15, and 20% gypsum and 0, 25 and 50% fly ash as partial replacement for cement. Specimens were tested for compressive strength at the age of 7, 28, and 90 days. The third and final phase of research selected one mix designs from Phase II including 15% recycled gypsum, 50% fly ash and 35% cement to be tested for durability, which was evaluated by testing the compressive strength of specimens after exposure to various wet and dry conditions for 1000, 3000 or 5000 hours. The following conclusions were drawn from the experimental studies:

- Mortar specimen tests indicated that gypsum is disadvantageous to compressive strength at all ages when partially replacing cement; however, improved strength was observed at later ages in specimens that also used fly ash as a partial cement replacement.
- A positive relationship between fly ash and gypsum in concrete repeatedly confirmed, highlighting that either material combined with cement alone had inferior

compressive strength, however when mixed together, a strength increase was observed at later ages. This confirmed the work of previous researchers stating that the hydration of fly ash is improved with gypsum.

- Gypsum content between 5-20% was found to have a non-significant effect on compressive strength of concrete containing 50% fly ash at later ages. All mixes with additional gypsum slightly outperformed the 50% fly ash control specimens (no gypsum) at 90 days, with the highest compressive strength observed in specimens with 20% gypsum content.
- All specimens tested for durability continued to develop compressive strength throughout exposure to all conditions, including dry, submerged in saltwater (seawater) and fresh water, and rotated between wetting and drying cycles in saltwater and fresh water. Specimens in dry (control) conditions showed the smallest strength increase throughout exposure, and specimens in saltwater conditions showed the highest strengths. Specimens subject to saltwater wetting and drying cycles revealed the highest strength increase between 1000 and 5000 hours (50.3%), and maximum compressive strength (53.73 MPa).
- Gypsum was found to dehydrate mixes, so the use of a superplasticizer was necessary. A 'false set' in the concrete mix can occur with large amounts of gypsum that stiffens due to crystal formation. This can be avoided by keeping the mix moving, or if a false set is recognized, it can be disrupted by further mixing.

6.2 RECOMMENDATIONS

Based on the presented study, it is believed that further research on gypsum as a supplementary cementing material in combination with fly ash is worthwhile and beneficial. The following recommendations are made for future research:

- Investigation of other exposure conditions such as cycles of freezing and thawing, and/or heating and cooling. These exposures should also be considered in wet and dry conditions, as improved strength was observed for this concrete mix when exposed to wet conditions.
- Additionally, it would be useful to compare the durability of concrete exposed to conditions similar to those presented in this research, but varying gypsum and fly ash contents.
- Durability to prolonged exposure (beyond 5000 hours) is recommended.
- Proper equipment to precisely measure any expansion or shrinkage of wet and dry concrete specimens is highly suggested.
- Resistance to various chemical attacks is also pertinent, including attacks from sulfates, chlorides or alkalis. Sulfate (SO_4) attack may be of increased importance due to the additional sulfates provided by gypsum.
- Testing of various larger scale specimens is also of interest, including beams, columns, slabs, and concrete-filled fibre-reinforced polymer tubes (CFFTs).
- Analyzing SEM photos of dry cementitious materials to determine differences in particle shapes its effect on the hydration process of concrete.
- Taking SEM photos of concrete after failure to analyze cracking patterns, including microcrack patterns and microcrack density.

- It would be interesting to explore the possibility of using seawater as the mix water, considering the results of this research indicate that submerging in seawater was beneficial to strength for this concrete type. With rising sea levels, many coastal areas around the world are experiencing shortages of fresh water and this would be an ideal sustainable alternative to traditional concrete construction.

REFERENCES

Aimin, Xu, and Shondeep L. Sarkar. 1991. "Microstructural study of gypsum activated fly ash hydration in cement paste." *Cement and Concrete Research* (Pergamon Press) 21: 1137-1147.

American Concrete Institute (ACI). 2000. *ACI Manual of Concrete Practice, Part 1: Materials and General Properties of Concrete.* Manual, Farmington Hills, MI: American Concrete Institute.

Amidon, G. E., P. J. Meyer, and D. M. Mudie. 2017. "Chapter 10 - Particle, Powder, and Compact Characterization." *Developing Solid Oral Dosage Forms - Pharmaceutical Theory and Practice* (Academic Press) Second Edition: 271-293.

Aprianti, E. S. 2017. "A huge number of artificial waste material can be supplementary cementitious material (SCM) for concrete production - a review part II." *Journal of Cleaner Production* (142): 4178-4194.

Antunes, M. L., A. Botignon de Sa, P. S. Oliveira, and E. C. Rangel. 2019. "Utilization of gypsum from construction and demolition waste in Portland cement mortar." *Ceramica* 65 (1): 1-6.

ASTM. 2019. "C1038/C1038M - 19 Standard Test Method for Expansion of Hydraulic Cement Mortar Bars Stored in Water." *ASTM International.*

ASTM. 2016. "C109/C109M – 16 Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in or [50-mm] Cube Specimens)", *ASTM International.*

ASTM. 2015. "C136/C136M-14 Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates." *ASTM International.*

ASTM. 2015. "C143/C143M - 15a Standard Test Method for Slump of Hydraulic-Cement Concrete." *ASTM International.*

ASTM. 2015. "C150/150M- Standard Specification for Portland Cement." *ASTM International.*

ASTM. 2018. "C192/C192M-18 Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory." *ASTM International.*

ASTM. 2018. "C33/C33M-18: Standard Specification for Concrete Aggregates." *ASTM International.*

ASTM. 2016. "C39/C39M-16 Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens." *ASTM International.*

ASTM. 2019. "C469/C469M-14 Standard Test Method for Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression." *ASTM International.*

ASTM. 2017. "C490/C490M - 17 Standard Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete." *ASTM International.*

ASTM. 2017. "C618 - 17a Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete." *ASTM International.*

Barbosa, W., R. D'Paula Ramalho, and K.F. Portella. 2018. "Influence of gypsum fineness in the first hours of cement paste: hydration kinetics and rheological behaviour." *Construction and Building Materials* (Elsevier) (184): 304-310.

Batte, Marvin, and D. Lynn Forster. 2015. "Old is new again: the economics of agricultural gypsum use." *Journal of ASFMRA* 56-65.

Behera, Monalisa, S. K. Bhattacharyya, A. K. Minocha, R. Deoliya, and S. Maiti. 2014. "Recycled aggregate from C&D waste and its use in concrete - a breakthrough towards sustainability in construction sector: A review." *Construction and Building Materials* (Elsevier) 68: 501-516.

Bentz, Dale P., and Chiara F. Ferraris. 2010. "Rheology and setting of high volume fly ash mixtures." *Cement and Concrete Composites* 32: 265-270.

Canadian Standards Association (CSA). 2004. "CAN/CSA-A23.3-04 Design of concrete structures." *Standards Council of Canada* (Canadian Standards Association).

Chandara, Chea, Khairun Azizli, Zainal Ahmad, and Etsuo Sakai. 2009. "Use of waste gypsum to replace natural gypsum as set retarders in portland cement." *Waste Management* (Elsevier) (29): 1675-1679.

Chen, X., M.K. Zhou, P.L. Cong, and X. Li. 2008. "Influence of SO3 content on the strength of cement-fly ash stabilized crushed-stones." *4th Asian Regional Conference on Geosynthetics.* Shanghai, China. 398-402.

Christensen, A. Norlund, Torben R. Jensen, and Jonathan C. Hanson. 2004. "Formation of ettringite, Ca6Al2(SO4)3(OH)12-26H2O, AFt, and monosulfate, Ca4Al2O6(SO4)-14H2O, AFm-14, in hydrothermal hydration of Portland cement and of calcium aluminum oxide—calcium sulfate dihydrate mixtures studied by in situ synchrotron X-ray." *Journal of Solid State Chemistry* 177: 1944-1951.

Chun, Yoon-moon, Tarun Naik, and Rudolph Kraus. 2008. “Recycling Opportunity for Gypsum Wallboard in Concrete”. Solid waste research program, Milwaukee: University of Wisconsin.

Construction and Demolition Recycling Association (CDRA). 2019. Gypsum Drywall. CDRA. April 4. https://cdrecycling.org/materials/gypsum-drywall/.

Federal Highway Administration Research and Technology. 2016. User Guidelines for Waste and Byproduct Materials in Pavement Construction. U.S. Department of Transportation. 08 03. https://www.fhwa.dot.gov/publications/research/infrastructure/structures/97148/sw1.cfm.

Gani, M.S.J. 1997. "Cement and concrete." *Cement and concrete: 1st edition*, 49-169. England: Chapman and Hills.

Garg, Mridul, and Aakanksha Pundir. 2016. "Energy efficient cement free binder developed from industry waste- A sustainable approach." *European Journal of Environmental and Civil Engineering* 21 (5): 612-628.

Government of Canada. 2009. *Guidelines for Canadian Drinking Water Quality: Guideline Technical Document- Sulphate.* 04 24. https://www.canada.ca/en/health-canada/services/publications/healthy-living/guidelines-canadian-drinking-water-quality-guideline-technical-document-sulphate.html.

Government of Canada. 2017. Total Canadian Exports - Trade Data Online. https://ic.gc.ca/app/scr/tdst/tdo/crtr.html;jsessionid=0001PxpZoj58tepyrBSIowTV7KM:2G83UKVV8F?lang=eng&hSelectedCodes=32742&searchType=BL&productType=NAICS&currency=CDN&runReport=true&grouped=GROUPED&toFromCountry=CDN&naArea=9999&timePeriod=CustomYears&re.

Government of Canada. 2019. Statistics Canada Report - Trade Data Online. https://ic.gc.ca/app/scr/tdst/tdo/crtr.html;jsessionid=0001PxpZoj58tepyrBSIowTV7KM:2G83UKVV8F?lang=eng&hSelectedCodes=32742&searchType=BL&productType=NAICS&currency=CDN&runReport=true&grouped=GROUPED&toFromCountry=CDN&naArea=9999&countryList=SALL&reportTy.

Gratton, R. and Beaudoin, G. 2010. *Gypsum Recycling in the National Capital Region,* The Construction Recycling Initiative (CRI) – National Capital Region, Ottawa, Ontario, Canada.

Gypsum Association. 2019. “Other Uses of Gypsum.” Gypsum Association. https://www.gypsum.org/about/gypsum-101/uses-gypsum/.

Hanhan, Amin A. 2004. *Influence of the SO3 content of cement on the durability and strength of concrete exposed to sodium sulfate environment.* Graduate Thesis and Dissertation, Department of Civil and Environmental Engineering, University of South Florida, Tampa, Florida: Scholar Commons.

Hansen, Sarah and Pedram Sadeghian. 2019. "Application of recycled gypsum wallboards in cement mortar." *CSCE Annual Conference.* Laval, Quebec, Canada: Canadian Society for Civil Engineering. MA5-1:8.

Hansen, Sarah, and Pedram Sadeghian. 2020. "Recycled gypsum for partial cement replacement in concrete." *Journal of Cleaner Production (In review).*

Helepciuc, Catalina, Marinela Barbuta, and Dan Babor. 2017. "Investigations on a green concrete obtaining through a partial cement replacement by fly ash." AES Bioflux 9 (3): 201-214.

Hoff, G. 1991. "Durability of offshore and marine concrete structures." *2nd International Conference (ACI SP-127).* Montreal, Canada: American Concrete Institute. 33-64.

Horkoss, Sayed, Gilles Escadeillas, Toufic Rizk, and Roger Lteif. 2015. "The effect of the course of cement SO3 on the expansion of mortars." *Case studies in Construction Materials* (Elsevier) 4: 62-72.

Hover, Kenneth. 2011. "The influence of water on the performance of concrete." *Construction and Building Materials* (Elsevier) 25: 3003-3013.

Islam, M. M., and M.S. Islam. 2013. "Strength and durability chracteristics of concrete made with fly-ash blended cement." *Australian Journal of Structural Engineering* (Institution of Engineers Australia) 14 (3): 2204-2261.

Kozicki, Chris, and Carrie Carlson. 2019. *Gypsum Recycling.* Feeco International. https://feeco.com/gypsum-recycling/.

Law, David, Andi Arham Adam, Thomas K. Molyneaux, Indubhushan Patnaikuni, and Arie Wardhono. 2014. "Long term durability properties of class F fly ash geopolymer concrete." *Materials and Structures* 48: 721-731.

Marlay, Kyle Marie. 2011. Hardened Concrete Properties and Durability Assessment of High-Volume Fly Ash Concrete. Thesis, St. Louis: Missouri University of Science and Technology.

Mehta, P. Kumar, and Paulo J.M. Monteiro. 2014. "Concrete: microstructure, properties, and materials (Fourth edition)." (McGraw-Hill Education).

Mentzer, A.P. 2018. Uses for Gypsum Powder. Sciencing. November 19. https://sciencing.com/uses-gypsum-powder-6395376.html.

Meyer, C. 2009. "The greening of the concrete industry." *Cement & Concrete Composites* 31: 601-605.

Mindat. 2019. Milford quarry - National gypsum quarry - East Milford, Nova Scotia, Canada. Hudson Institute of Mineralogy. March 5. https://www.mindat.org/loc-267263.html.

Mohammed, Siline, and Omary Safiullah. 2018. "Optimization of the SO3 content of an Algerian Portland cement: Study on the effect of various amounts of gypsum on cement properties." *Construction and Building Materials* 164: 363-370.

Naik, Tarun, Rakesh Kumar, Yoon-moon Chun, and Rudolph Kraus. 2010. Utilization of Powdered Gypsum-Wallboard in Concrete. Milwaukee: UWM Center for By-Products Utilization-University of Wisconsin, Milwaukee, WI, USA.

Nehdi, M., and M. Hayek. 2005. "Behavior of blended cement mortars exposed to sulfate solutions cycling in relative humidity." *Cement and Concrete Research* 35: 731-742.

Nguyen, Long, Alexander J. Moseson, Yaghoob Farnam, and Sabrina Spatari. 2018. "Effects of composition and transportation logistics on environmental energy and cost metrics for the production of alternative cementitious binders." *Journal of Cleaner Production* (Elsevier) 185: 628-645.

Nixon, P. J. 1978. “Recycled Concrete as an Aggregate for Concrete” - A Review. Great Britain: 37-DRC Committee (Demolition and Re-Use of Concrete).

Odler, I., and Y. Chen. 1995. "Effect of cement composition on the expansion of heat-cured cement pastes." *Cement and Concrete Research* (Elsevier) 25 (4): 853-862.

Olivia, Monita, and Hamid Nikraz. 2011. "Properties of fly ash geopolymer concrete designed by Taguchi method." *Materials and Design* (Elsevier) 36: 191-198.

Olorunsogo, F. T., and N. Padayachee. 2002. "Performance of recycled aggregate concrete monitored by durability indexes." *Cement and Concrete Research* (Pergamon) (32): 179-185.

Olson, Donald. 2001. "Gypsum." *U.S. Geological Survey Minerals Yearbook* 35.1-35.5.

Panalytical, Malvern. 2020. *Mastersizer 3000.* Worcestershire, United Kingdom: Spectris Company.

Papageorgiou, A., G. Tzouvalas, and S. Tsimas. 2005. "Use or inorganic setting retarders in cement industry." *Cement and Concrete composites 27*: 183-189.

Portland Cement Association. 2001. *Concrete Information - Ettringite formation and the performance of concrete.* Skokie, Illinois: Portland Cement Association (PCA).

Province of Nova Scotia. 2014. The East Milford Quarry Type Section in the Hants Lowlands. September 29. https://novascotia.ca/natr/meb/fieldtrip/stop3.asp.

Prusinski, J. R., and R. L. Carrasquillo. 1995. "Using medium to high-volume fly ash blended cements to improve the sulfate resistance of high-lime fly ash concrete." *Fifth CANMET/ACI International Conference on Fly Ash, Silica Fume, Slag and Natural Pozzolans in Concrete.* ACI SP-153. 43-66.

Puvvadi, Sivapullaiah, and Arif Ali Baig Moghal. 2011. "Role of gypsum in the strength development of fly ashes with lime." *Journal of Materials in Civil Engineering* (ASCE) 23 (2): 197-206.

Quennoz, Alexandra, and Karen Scrivener. 2012. "Hydration of C3A-gypsum system." *Cement and Concrete Research* (Elsevier) (42): 1032-1041.

Raghavendra, T., and B.C. Udayashankar. 2015. "Engineering properties of controlled low strength materials using flyash and waste gypsum wall boards." *Construction and Building Materials* 101: 548-557.

Recycling Council of Ontario. 2006. “Let's Climb Another Mole Hill - An examination of construction, demolition and renovation (CRD) waste diversion in Canada and associated greenhouse gas emission impacts.” *Recycling Council of Ontario,* Toronto, Ontario.

Rivero, Ana Jimenez. 2016. Gypsum Waste Management in the European Union: Towards a Circular Economy for the Construction Sector. Thesis, Madrid: Polytechnic University of Madrid- Doctorate Program in Building Technology Innovation.

Sata, Vanchai, Chai Jaturapitakkul, and Chaiyanunt Rattanashotinunt. 2010. "Compressive strength and heat evolution on concrete containing palm oil fuel ash." *Journal of Materials in Civil Engineering* (ASCE) 22 (10): 1033-1038.

Sharpe, Roger, and Greg Cork. 2006. "Gypsum and Anhydrite." Industrial Minerals and Rocks (Society for Mining, Metallurgy, and Exploration, Inc.) (7th edition): 519-540.

Shehata, Medhat, and Michael Thomas. 2000. "The effect of fly ash composition on the expansion of concrete due to alkali-silica reaction." *Cement and Concrete Research* (Pergamon) 30: 1063-1072.

Spurlock, Nancy. 2004. National Gypsum - Operations - Halifax treasure box supplies plants. https://ngc-heritage.com/op-halifax-quarry.htm.

Suarez, S., Roca, X., Gasson, S. 2016. Product-specific life cycle assessment of recycled gypsum as a replacement for natural gypsum in ordinary Portland cement: application to the Spanish context. *Journal of Cleaner Production.* **117**: 150-159.

Teo, M. M., and M. Loosemore. 2001. "A theory of waste behaviour in the construction industry." Construction Management and Economics (Routledge) 19 (7): 741-751.

University of Waterloo. 2020. *Eart Sciences Museum - Gypsum.* https://uwaterloo.ca/earth-sciences-museum/resources/detailed-rocks-and-minerals-articles/gypsum.

USA Gypsum. 2019. *10 Beneficial Ways To Use Recycled Gypsum.* Agri Marketing Inc. https://www.usagypsum.com/10-beneficial-ways-use-recycled-gypsum.

USA Gypsum. 2019. *Industrial Gypsum.* Agri Marketing Inc. March 26. https://www.usagypsum.com/gypsum-products/industrial-gypsum.

Vargas, J., and A. Halog. 2015. "Effective carbon emission reductions from using upgraded fly ash in the cement industry." *Journal of Cleaner Production* (Elsevier) (103): 948-959.

Vasavan, Manilal. 2017. "Sulfate attack and sulfate content in concrete: a literature review." *Geology to Concrete Technology.*

Wegian, Falah M. 2010. "Effect of seawater for mixing and curing on structural concrete." *The IES Journal Part A: Civil & Structural Engineering* 3 (4): 235-243.

Wu, Zichao, and Tarun Naik. 2002. "Properties of concrete produced from multicomponent blended cements." *Cement and Concrete Research* (Elsevier) 32: 1937-1942.

Xie, Tianyu, and Togay Ozbakkaloglu. 2015. "Behavior of low-calcium fly and bottom ash-based geopolymer concrete cured at ambient temperature." *Ceramics International* 41: 5945-5958.

Zhang, Jianye, Hwidong Kim, Brajesh Dubey, and Timothy Townsend. 2016. "Arsenic leaching and speciation in C&D debris landfills and the relationship with gypsum drywall content." *Waste Management* 59 (1): 324-329.

APPENDIX A LATERAL AND AXIAL STRESS/STRAIN DIAGRAMS

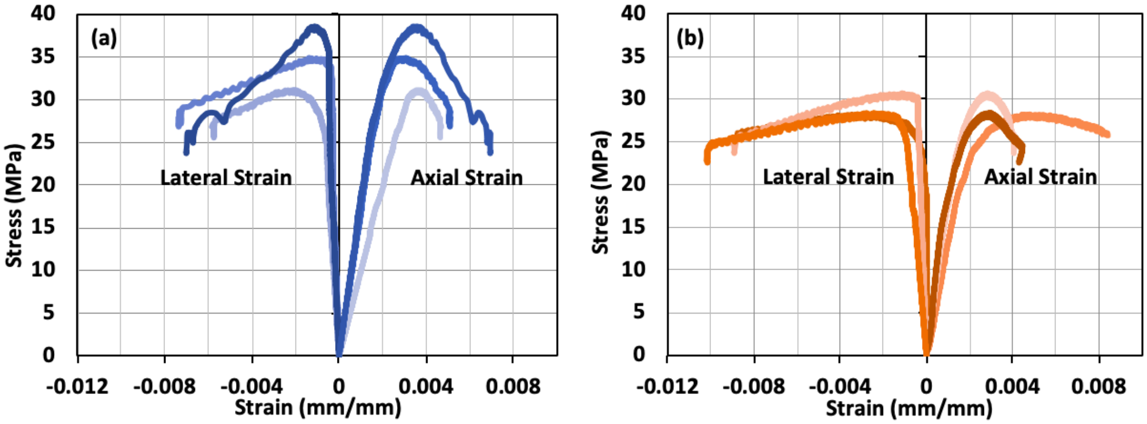


Figure A - 1 Lateral and axial stress/strain diagrams for specimens (a) FG0-FA50-C50 and (b) FG20-FA0-C80.

APPENDIX B DIAMETER MEASUREMENT DATA FOR SELECT DURABILITY SPECIMENS

This appendix includes data recorded while monitoring the diameter change of concrete specimens that are dry (M and E1), rotated wet/dry in fresh water (E4), and rotated wet/dry in seawater (E5). The numbers recorded are measured in inches to the accuracy of 0.0001. The micrometer used for measurements was designed to measure lengths between 4 and 5 inches, so each of the numbers recorded is however much above 4 inches the diameter length was measured to be. In the following tables, ‘Avg’ signifies the average, and ‘SD’ signifies standard deviation.

Table B - 1 Diameter change of group M between day 5 and day 100

		Day 5	Day 100	Day 5-Day 100
M1-1	1	0.0701	0.0686	0.0014
	2	0.0078	0.0076	0.0002
M1-2	1	0.0189	0.0183	0.0005
	2	0.0729	0.0705	0.0024
M1-3	1	0.0489	0.0472	0.0017
	2	0.0264	0.0257	0.0007
	3	0.0080	0.0079	0.0001

Avg = 0.0010 inches

To calculate the % change (shrinkage):

Average change = (0.0010 inches) x (25.4 mm/inch) = 0.0255 mm

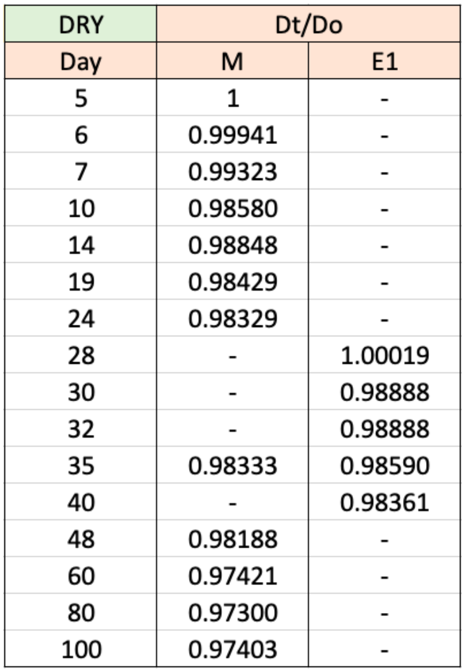
Diameter = 100 mm, therefore:

Diameter change = (0.0255 mm) / (100 mm) = 0.000255 mm/mm

Change to percent:

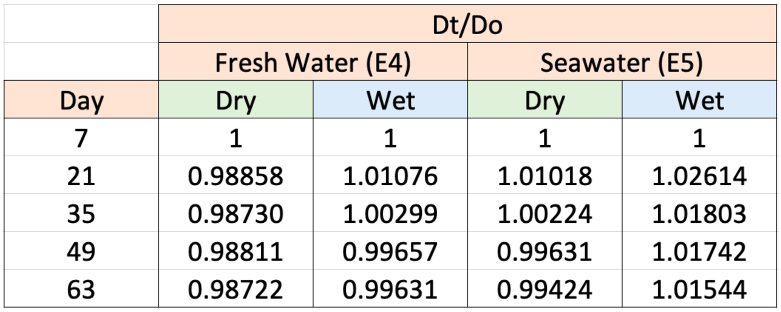
(0.000255 mm/mm) x (100%) = 0.0255% = 0.026%

Table B - 2	Summary table for D/Davg results for dry specimens (M and E1)



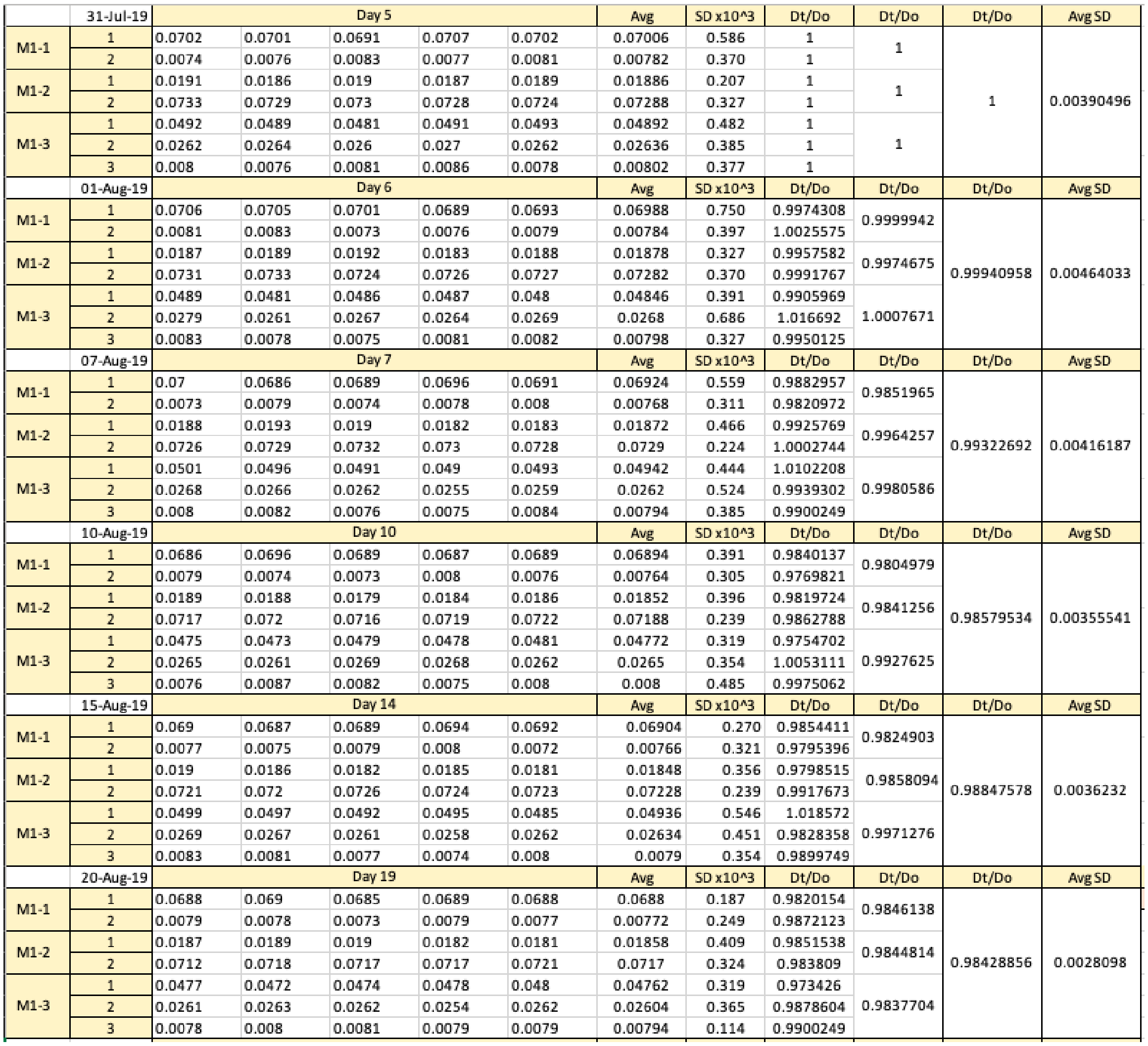
DRY	Dt/Do	
Day	M	E1
5	1	-
6	0.99941	-
7	0.99323	-
10	0.98580	-
14	0.98848	-
19	0.98429	-
24	0.98329	-
28	-	1.00019
30	-	0.98888
32	-	0.98888
35	0.98333	0.98590
40	-	0.98361
48	0.98188	-
60	0.97421	-
80	0.97300	-
100	0.97403	-

Table B - 3	Summary table of Dt/Do results for rotated dry/wet specimens in fresh water and seawater

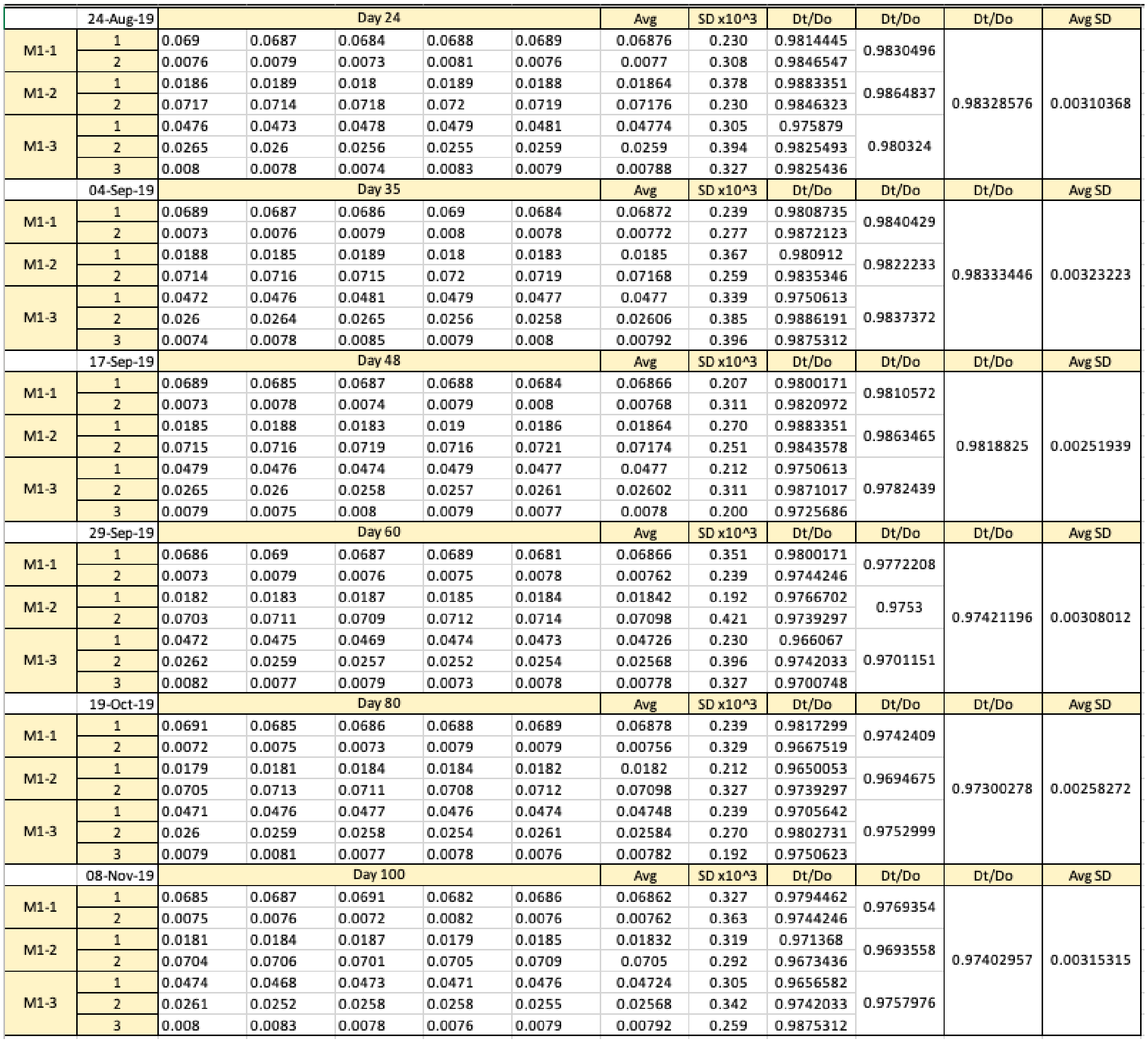


	Dt/Do			
	Fresh Water (E4)		Seawater (E5)	
Day	Dry	Wet	Dry	Wet
7	1	1	1	1
21	0.98858	1.01076	1.01018	1.02614
35	0.98730	1.00299	1.00224	1.01803
49	0.98811	0.99657	0.99631	1.01742
63	0.98722	0.99631	0.99424	1.01544

Table B - 4 Diameter measurements of dry specimen group M

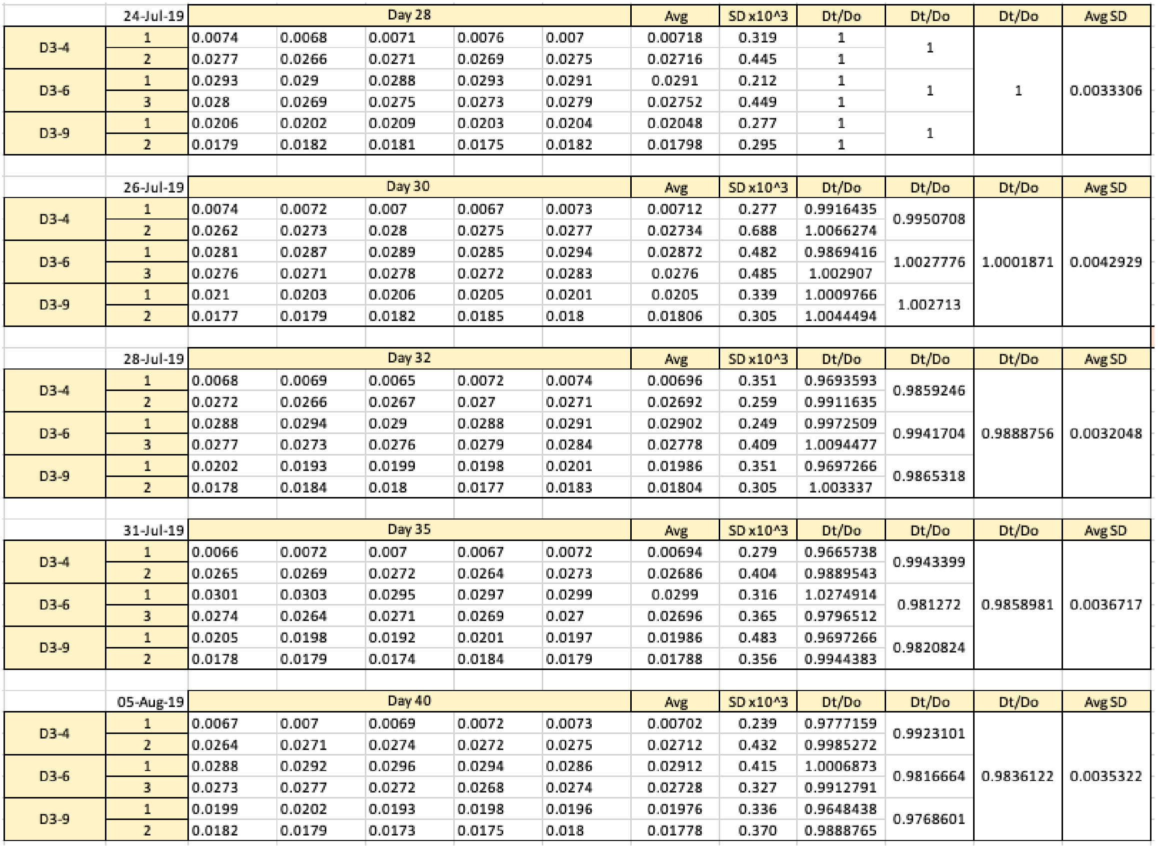


	31-Jul-19	Day 5					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
M1-1	1	0.0702	0.0701	0.0691	0.0707	0.0702	0.07006	0.586	1	1	1	0.00390496
	2	0.0074	0.0076	0.0083	0.0077	0.0081	0.00782	0.370	1			
M1-2	1	0.0191	0.0186	0.019	0.0187	0.0189	0.01886	0.207	1	1		
	2	0.0733	0.0729	0.073	0.0728	0.0724	0.07288	0.327	1			
M1-3	1	0.0492	0.0489	0.0481	0.0491	0.0493	0.04892	0.482	1	1		
	2	0.0262	0.0264	0.026	0.027	0.0262	0.02636	0.385	1			
	3	0.008	0.0076	0.0081	0.0086	0.0078	0.00802	0.377	1			
	01-Aug-19	Day 6					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
M1-1	1	0.0706	0.0705	0.0701	0.0689	0.0693	0.06988	0.750	0.9974308	0.9999942	0.99940958	0.00464033
	2	0.0081	0.0083	0.0073	0.0076	0.0079	0.00784	0.397	1.0025575			
M1-2	1	0.0187	0.0189	0.0192	0.0183	0.0188	0.01878	0.327	0.9957582	0.9974675		
	2	0.0731	0.0733	0.0724	0.0726	0.0727	0.07282	0.370	0.9991767			
M1-3	1	0.0489	0.0481	0.0486	0.0487	0.048	0.04846	0.391	0.9905969	1.0007671		
	2	0.0279	0.0261	0.0267	0.0264	0.0269	0.0268	0.686	1.016692			
	3	0.0083	0.0078	0.0075	0.0081	0.0082	0.00798	0.327	0.9950125			
	07-Aug-19	Day 7					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
M1-1	1	0.07	0.0686	0.0689	0.0696	0.0691	0.06924	0.559	0.9882957	0.9851965	0.99322692	0.00416187
	2	0.0073	0.0079	0.0074	0.0078	0.008	0.00768	0.311	0.9820972			
M1-2	1	0.0188	0.0193	0.019	0.0182	0.0183	0.01872	0.466	0.9925769	0.9964257		
	2	0.0726	0.0729	0.0732	0.073	0.0728	0.0729	0.224	1.0002744			
M1-3	1	0.0501	0.0496	0.0491	0.049	0.0493	0.04942	0.444	1.0102208	0.9980586		
	2	0.0268	0.0266	0.0262	0.0255	0.0259	0.0262	0.524	0.9939302			
	3	0.008	0.0082	0.0076	0.0075	0.0084	0.00794	0.385	0.9900249			
	10-Aug-19	Day 10					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
M1-1	1	0.0686	0.0696	0.0689	0.0687	0.0689	0.06894	0.391	0.9840137	0.9804979	0.98579534	0.00355541
	2	0.0079	0.0074	0.0073	0.008	0.0076	0.00764	0.305	0.9769821			
M1-2	1	0.0189	0.0188	0.0179	0.0184	0.0186	0.01852	0.396	0.9819724	0.9841256		
	2	0.0717	0.072	0.0716	0.0719	0.0722	0.07188	0.239	0.9862788			
M1-3	1	0.0475	0.0473	0.0479	0.0478	0.0481	0.04772	0.319	0.9754702	0.9927625		
	2	0.0265	0.0261	0.0269	0.0268	0.0262	0.0265	0.354	1.0053111			
	3	0.0076	0.0087	0.0082	0.0075	0.008	0.008	0.485	0.9975062			
	15-Aug-19	Day 14					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
M1-1	1	0.069	0.0687	0.0689	0.0694	0.0692	0.06904	0.270	0.9854411	0.9824903	0.98847578	0.0036232
	2	0.0077	0.0075	0.0079	0.008	0.0072	0.00766	0.321	0.9795396			
M1-2	1	0.019	0.0186	0.0182	0.0185	0.0181	0.01848	0.356	0.9798515	0.9858094		
	2	0.0721	0.072	0.0726	0.0724	0.0723	0.07228	0.239	0.9917673			
M1-3	1	0.0499	0.0497	0.0492	0.0495	0.0485	0.04936	0.546	1.018572	0.9971276		
	2	0.0269	0.0267	0.0261	0.0258	0.0262	0.02634	0.451	0.9828358			
	3	0.0083	0.0081	0.0077	0.0074	0.008	0.0079	0.354	0.9899749			
	20-Aug-19	Day 19					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
M1-1	1	0.0688	0.069	0.0685	0.0689	0.0688	0.0688	0.187	0.9820154	0.9846138	0.98428856	0.0028098
	2	0.0079	0.0078	0.0073	0.0079	0.0077	0.00772	0.249	0.9872123			
M1-2	1	0.0187	0.0189	0.019	0.0182	0.0181	0.01858	0.409	0.9851538	0.9844814		
	2	0.0712	0.0718	0.0717	0.0717	0.0721	0.0717	0.324	0.983809			
M1-3	1	0.0477	0.0472	0.0474	0.0478	0.048	0.04762	0.319	0.973426	0.9837704		
	2	0.0261	0.0263	0.0262	0.0254	0.0262	0.02604	0.365	0.9878604			
	3	0.0078	0.008	0.0081	0.0079	0.0079	0.00794	0.114	0.9900249			



	24-Aug-19	Day 24					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
M1-1	1	0.069	0.0687	0.0684	0.0688	0.0689	0.06876	0.230	0.9814445	0.9830496	0.98328576	0.00310368
	2	0.0076	0.0079	0.0073	0.0081	0.0076	0.0077	0.308	0.9846547			
M1-2	1	0.0186	0.0189	0.018	0.0189	0.0188	0.01864	0.378	0.9883351	0.9864837		
	2	0.0717	0.0714	0.0718	0.072	0.0719	0.07176	0.230	0.9846323			
M1-3	1	0.0476	0.0473	0.0478	0.0479	0.0481	0.04774	0.305	0.975879	0.980324		
	2	0.0265	0.026	0.0256	0.0255	0.0259	0.0259	0.394	0.9825493			
	3	0.008	0.0078	0.0074	0.0083	0.0079	0.00788	0.327	0.9825436			
	04-Sep-19	Day 35					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
M1-1	1	0.0689	0.0687	0.0686	0.069	0.0684	0.06872	0.239	0.9808735	0.9840429	0.98333446	0.00323223
	2	0.0073	0.0076	0.0079	0.008	0.0078	0.00772	0.277	0.9872123			
M1-2	1	0.0188	0.0185	0.0189	0.018	0.0183	0.0185	0.367	0.980912	0.9822233		
	2	0.0714	0.0716	0.0715	0.072	0.0719	0.07168	0.259	0.9835346			
M1-3	1	0.0472	0.0476	0.0481	0.0479	0.0477	0.0477	0.339	0.9750613	0.9837372		
	2	0.026	0.0264	0.0265	0.0256	0.0258	0.02606	0.385	0.9886191			
	3	0.0074	0.0078	0.0085	0.0079	0.008	0.00792	0.396	0.9875312			
	17-Sep-19	Day 48					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
M1-1	1	0.0689	0.0685	0.0687	0.0688	0.0684	0.06866	0.207	0.9800171	0.9810572	0.9818825	0.00251939
	2	0.0073	0.0078	0.0074	0.0079	0.008	0.00768	0.311	0.9820972			
M1-2	1	0.0185	0.0188	0.0183	0.019	0.0186	0.01864	0.270	0.9883351	0.9863465		
	2	0.0715	0.0716	0.0719	0.0716	0.0721	0.07174	0.251	0.9843578			
M1-3	1	0.0479	0.0476	0.0474	0.0479	0.0477	0.0477	0.212	0.9750613	0.9782439		
	2	0.0265	0.026	0.0258	0.0257	0.0261	0.02602	0.311	0.9871017			
	3	0.0079	0.0075	0.008	0.0079	0.0077	0.0078	0.200	0.9725686			
	29-Sep-19	Day 60					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
M1-1	1	0.0686	0.069	0.0687	0.0689	0.0681	0.06866	0.351	0.9800171	0.9772208	0.97421196	0.00308012
	2	0.0073	0.0079	0.0076	0.0075	0.0078	0.00762	0.239	0.9744246			
M1-2	1	0.0182	0.0183	0.0187	0.0185	0.0184	0.01842	0.192	0.9766702	0.9753		
	2	0.0703	0.0711	0.0709	0.0712	0.0714	0.07098	0.421	0.9739297			
M1-3	1	0.0472	0.0475	0.0469	0.0474	0.0473	0.04726	0.230	0.966067	0.9701151		
	2	0.0262	0.0259	0.0257	0.0252	0.0254	0.02568	0.396	0.9742033			
	3	0.0082	0.0077	0.0079	0.0073	0.0078	0.00778	0.327	0.9700748			
	19-Oct-19	Day 80					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
M1-1	1	0.0691	0.0685	0.0686	0.0688	0.0689	0.06878	0.239	0.9817299	0.9742409	0.97300278	0.00258272
	2	0.0072	0.0075	0.0073	0.0079	0.0079	0.00756	0.329	0.9667519			
M1-2	1	0.0179	0.0181	0.0184	0.0184	0.0182	0.0182	0.212	0.9650053	0.9694675		
	2	0.0705	0.0713	0.0711	0.0708	0.0712	0.07098	0.327	0.9739297			
M1-3	1	0.0471	0.0476	0.0477	0.0476	0.0474	0.04748	0.239	0.9705642	0.9752999		
	2	0.026	0.0259	0.0258	0.0254	0.0261	0.02584	0.270	0.9802731			
	3	0.0079	0.0081	0.0077	0.0078	0.0076	0.00782	0.192	0.9750623			
	08-Nov-19	Day 100					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
M1-1	1	0.0685	0.0687	0.0691	0.0682	0.0686	0.06862	0.327	0.9794462	0.9769354	0.97402957	0.00315315
	2	0.0075	0.0076	0.0072	0.0082	0.0076	0.00762	0.363	0.9744246			
M1-2	1	0.0181	0.0184	0.0187	0.0179	0.0185	0.01832	0.319	0.971368	0.9693558		
	2	0.0704	0.0706	0.0701	0.0705	0.0709	0.0705	0.292	0.9673436			
M1-3	1	0.0474	0.0468	0.0473	0.0471	0.0476	0.04724	0.305	0.9656582	0.9757976		
	2	0.0261	0.0252	0.0258	0.0258	0.0255	0.02568	0.342	0.9742033			
	3	0.008	0.0083	0.0078	0.0076	0.0079	0.00792	0.259	0.9875312			

Table B - 5 Diameter measurements of specimen group E1



	24-Jul-19	Day 28					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D3-4	1	0.0074	0.0068	0.0071	0.0076	0.007	0.00718	0.319	1	1	1	0.0033306
	2	0.0277	0.0266	0.0271	0.0269	0.0275	0.02716	0.445	1			
D3-6	1	0.0293	0.029	0.0288	0.0293	0.0291	0.0291	0.212	1	1		
	3	0.028	0.0269	0.0275	0.0273	0.0279	0.02752	0.449	1			
D3-9	1	0.0206	0.0202	0.0209	0.0203	0.0204	0.02048	0.277	1	1		
	2	0.0179	0.0182	0.0181	0.0175	0.0182	0.01798	0.295	1			
	26-Jul-19	Day 30					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D3-4	1	0.0074	0.0072	0.007	0.0067	0.0073	0.00712	0.277	0.9916435	0.9950708	1.0001871	0.0042929
	2	0.0262	0.0273	0.028	0.0275	0.0277	0.02734	0.688	1.0066274			
D3-6	1	0.0281	0.0287	0.0289	0.0285	0.0294	0.02872	0.482	0.9869416	1.0027776		
	3	0.0276	0.0271	0.0278	0.0272	0.0283	0.0276	0.485	1.002907			
D3-9	1	0.021	0.0203	0.0206	0.0205	0.0201	0.0205	0.339	1.0009766	1.002713		
	2	0.0177	0.0179	0.0182	0.0185	0.018	0.01806	0.305	1.0044494			
	28-Jul-19	Day 32					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D3-4	1	0.0068	0.0069	0.0065	0.0072	0.0074	0.00696	0.351	0.9693593	0.9859246	0.9888756	0.0032048
	2	0.0272	0.0266	0.0267	0.027	0.0271	0.02692	0.259	0.9911635			
D3-6	1	0.0288	0.0294	0.029	0.0288	0.0291	0.02902	0.249	0.9972509	0.9941704		
	3	0.0277	0.0273	0.0276	0.0279	0.0284	0.02778	0.409	1.0094477			
D3-9	1	0.0202	0.0193	0.0199	0.0198	0.0201	0.01986	0.351	0.9697266	0.9865318		
	2	0.0178	0.0184	0.018	0.0177	0.0183	0.01804	0.305	1.003337			
	31-Jul-19	Day 35					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D3-4	1	0.0066	0.0072	0.007	0.0067	0.0072	0.00694	0.279	0.9665738	0.9943399	0.9858981	0.0036717
	2	0.0265	0.0269	0.0272	0.0264	0.0273	0.02686	0.404	0.9889543			
D3-6	1	0.0301	0.0303	0.0295	0.0297	0.0299	0.0299	0.316	1.0274914	0.981272		
	3	0.0274	0.0264	0.0271	0.0269	0.027	0.02696	0.365	0.9796512			
D3-9	1	0.0205	0.0198	0.0192	0.0201	0.0197	0.01986	0.483	0.9697266	0.9820824		
	2	0.0178	0.0179	0.0174	0.0184	0.0179	0.01788	0.356	0.9944383			
	05-Aug-19	Day 40					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D3-4	1	0.0067	0.007	0.0069	0.0072	0.0073	0.00702	0.239	0.9777159	0.9923101	0.9836122	0.0035322
	2	0.0264	0.0271	0.0274	0.0272	0.0275	0.02712	0.432	0.9985272			
D3-6	1	0.0288	0.0292	0.0296	0.0294	0.0286	0.02912	0.415	1.0006873	0.9816664		
	3	0.0273	0.0277	0.0272	0.0268	0.0274	0.02728	0.327	0.9912791			
D3-9	1	0.0199	0.0202	0.0193	0.0198	0.0196	0.01976	0.336	0.9648438	0.9768601		
	2	0.0182	0.0179	0.0173	0.0175	0.018	0.01778	0.370	0.9888765			

Table B - 6 Diameter measurements of specimens rotated wet/dry in fresh water (E4)

E4- Fresh water												
DRY	31-Jul-19	Day 7					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D2-8	2	0.0876	0.0877	0.088	0.0876	0.0875	0.08768	0.192	1	1	1	0.002317781
	3	0.0263	0.0261	0.026	0.0264	0.0261	0.02618	0.164	1			
D2-4	1	0.0246	0.0242	0.0251	0.0243	0.0246	0.02456	0.351	1	1		
	3	0.0273	0.0273	0.0275	0.0272	0.027	0.02726	0.182	1			
D2-5	1	0.0223	0.0221	0.0224	0.0222	0.0225	0.0223	0.158	1	1		
	2	0.0357	0.0355	0.0361	0.0363	0.0356	0.03584	0.344	1			
WET	07-Aug-19	Day 14					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Average SD
D2-8	2	0.0878	0.0881	0.0876	0.0873	0.0879	0.08774	0.305	1.00068431	1.00645369	1.010763665	0.004500725
	3	0.0266	0.0258	0.0263	0.027	0.0268	0.0265	0.469	1.01222307			
D2-4	1	0.0249	0.0256	0.0251	0.0245	0.0252	0.02506	0.404	1.02035831	1.01348069		
	3	0.0272	0.028	0.0278	0.027	0.0272	0.02744	0.434	1.00660308			
D2-5	1	0.023	0.0226	0.0222	0.0225	0.0224	0.02254	0.297	1.01076233	1.01235661		
	2	0.037	0.0371	0.0366	0.0354	0.0356	0.03634	0.792	1.01395089			
DRY	14-Aug-19	Day 21					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Average SD
D2-8	2	0.0879	0.0881	0.0877	0.0878	0.0874	0.08778	0.259	1.00114051	0.99522266	0.988583677	0.00516935
	3	0.026	0.0264	0.0256	0.0254	0.0261	0.0259	0.400	0.98930481			
D2-4	1	0.0249	0.0242	0.0246	0.0238	0.0241	0.02432	0.432	0.99022801	0.98961144		
	3	0.0272	0.0279	0.0264	0.0263	0.027	0.02696	0.650	0.98899486			
D2-5	1	0.022	0.0224	0.0221	0.0224	0.0199	0.02176	1.055	0.97578475	0.98091693		
	2	0.0354	0.0349	0.0357	0.0352	0.0355	0.03534	0.305	0.98604911			
WET	21-Aug-19	Day 28					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Average SD
D2-8	2	0.0881	0.0876	0.0883	0.0875	0.0876	0.08782	0.356	1.00159672	0.99621471	1.002989506	0.003268068
	3	0.0265	0.0261	0.0259	0.0257	0.0255	0.02594	0.385	0.9908327			
D2-4	1	0.0241	0.0246	0.0247	0.0247	0.0248	0.02458	0.277	1.00081433	1.01214598		
	3	0.028	0.0286	0.0278	0.0275	0.0276	0.0279	0.436	1.02347762			
D2-5	1	0.0223	0.0221	0.0225	0.022	0.0223	0.02224	0.195	0.99730942	1.00060783		
	2	0.0358	0.0365	0.0357	0.036	0.0359	0.03598	0.311	1.00390625			
DRY	28-Aug-19	Day 35					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Average SD
D2-8	2	0.0881	0.0879	0.0873	0.087	0.0875	0.08756	0.445	0.99863139	0.9912943	0.987300919	0.0032641
	3	0.0257	0.0256	0.0254	0.026	0.0261	0.02576	0.288	0.98395722			
D2-4	1	0.0241	0.0246	0.0245	0.0243	0.0239	0.02428	0.286	0.98859935	0.98732975		
	3	0.0274	0.0271	0.0265	0.0264	0.027	0.02688	0.421	0.98606016			
D2-5	1	0.0221	0.022	0.0222	0.0216	0.0218	0.02194	0.241	0.9838565	0.9832787		
	2	0.0355	0.0351	0.0353	0.0348	0.0354	0.03522	0.277	0.98270089			
WET	04-Sep-19	Day 42					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Average SD
D2-8	2	0.088	0.0881	0.0877	0.0876	0.0881	0.0879	0.235	1.00250912	0.99934471	0.996569475	0.003055866
	3	0.0266	0.026	0.0265	0.0255	0.0258	0.02608	0.466	0.99618029			
D2-4	1	0.0244	0.0247	0.0241	0.0245	0.0248	0.0245	0.274	0.997557	0.99767799		
	3	0.0271	0.0274	0.027	0.0272	0.0273	0.0272	0.158	0.99779897			
D2-5	1	0.022	0.0223	0.0226	0.0219	0.0225	0.02226	0.305	0.99820628	0.99268573		
	2	0.0354	0.0351	0.0349	0.0356	0.0359	0.03538	0.396	0.98716518			
DRY	11-Sep-19	Day 49					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Average SD
D2-8	2	0.0882	0.0878	0.0873	0.0874	0.0875	0.08764	0.365	0.9995438	0.9944243	0.988108719	0.0031583
	3	0.0255	0.0257	0.0261	0.0262	0.026	0.0259	0.292	0.98930481			
D2-4	1	0.024	0.0244	0.0247	0.0242	0.0243	0.02432	0.259	0.99022801	0.98741041		
	3	0.0272	0.027	0.0265	0.0266	0.0269	0.02684	0.288	0.98459281			
D2-5	1	0.0218	0.022	0.0223	0.0219	0.0214	0.02188	0.327	0.98116592	0.98249144		
	2	0.0353	0.0352	0.0347	0.0354	0.0357	0.03526	0.365	0.98381696			
WET	18-Sep-19	Day 56					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Average SD
D2-8	2	0.0871	0.0873	0.088	0.0875	0.0876	0.0875	0.339	0.99794708	0.99859157	0.996312247	0.002811371
	3	0.0264	0.0262	0.0263	0.0259	0.026	0.02616	0.207	0.99923606			
D2-4	1	0.024	0.0249	0.0244	0.0246	0.0245	0.02448	0.327	0.99674267	0.99727082		
	3	0.0272	0.0275	0.0274	0.027	0.0269	0.0272	0.255	0.99779897			
D2-5	1	0.0222	0.0224	0.0221	0.0219	0.0226	0.02224	0.270	0.99730942	0.99307435		
	2	0.0355	0.0351	0.0352	0.0356	0.0358	0.03544	0.288	0.98883929			
DRY	25-Sep-19	Day 63					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Average SD
D2-8	2	0.0869	0.0868	0.0872	0.0864	0.0871	0.08688	0.311	0.99087591	0.98932642	0.987216807	0.003148164
	3	0.0256	0.0255	0.0261	0.0263	0.0258	0.02586	0.336	0.98777693			
D2-4	1	0.0242	0.0246	0.0245	0.024	0.0247	0.0244	0.292	0.99348534	0.98977275		
	3	0.0273	0.0265	0.0271	0.0266	0.0269	0.02688	0.335	0.98606016			
D2-5	1	0.0218	0.0219	0.0221	0.0224	0.0214	0.02192	0.370	0.98295964	0.98255125		
	2	0.035	0.0352	0.035	0.0352	0.0356	0.0352	0.245	0.98214286			

Table B - 7 Diameter measurements of specimens rotated wet/dry in saltwater (E5)

E5- Salt water												
DRY	26-Aug-19	Day 7					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D1-4	1	0.0759	0.0751	0.0758	0.0757	0.0752	0.07554	0.365	1	1	1	0.002640148
	3	0.0069	0.0077	0.0071	0.0073	0.007	0.0072	0.316	1			
D1-1	1	0.012	0.0114	0.0119	0.0121	0.0116	0.0118	0.292	1	1		
	3	0.0253	0.0254	0.0251	0.025	0.0251	0.02518	0.164	1			
D1-8	1	0.0019	0.0018	0.0022	0.0017	0.002	0.00192	0.192	1	1		
	2	0.0309	0.0314	0.0308	0.0311	0.0308	0.031	0.255	1			
WET	02-Sep-19	Day 14					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D1-4	1	0.0762	0.0758	0.076	0.0763	0.076	0.07606	0.195	1.00688377	1.039553	1.02613686	0.002875698
	3	0.0081	0.0078	0.008	0.0072	0.0075	0.00772	0.370	1.07222222			
D1-1	1	0.0122	0.0116	0.012	0.0124	0.0121	0.01206	0.297	1.0220339	1.02650543		
	3	0.0261	0.0259	0.0259	0.0254	0.0265	0.02596	0.397	1.03097697			
D1-8	1	0.0021	0.0017	0.002	0.0022	0.0018	0.00196	0.207	1.02083333	1.01235215		
	2	0.031	0.0308	0.0311	0.0315	0.0312	0.03112	0.259	1.00387097			
DRY	09-Sep-19	Day 21					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D1-4	1	0.0753	0.0759	0.0761	0.0764	0.0755	0.07584	0.445	1.00397141	1.00059681	1.010182194	0.003156881
	3	0.0072	0.0076	0.0071	0.0067	0.0073	0.00718	0.327	0.99722222			
D1-1	1	0.0123	0.0119	0.0121	0.0122	0.012	0.0121	0.158	1.02542373	1.01271186		
	3	0.0253	0.0251	0.0257	0.025	0.0248	0.02518	0.342	1			
D1-8	1	0.0022	0.0016	0.0021	0.0022	0.0018	0.00198	0.268	1.03125	1.0172379		
	2	0.0309	0.0307	0.0316	0.031	0.0313	0.0311	0.354	1.00322581			
WET	16-Sep-19	Day 28					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D1-4	1	0.0763	0.0765	0.0761	0.0767	0.0762	0.07636	0.241	1.01085518	1.0040387	1.018033468	0.002408938
	3	0.007	0.0073	0.0071	0.0074	0.0071	0.00718	0.164	0.99722222			
D1-1	1	0.0121	0.0126	0.012	0.0122	0.0125	0.01228	0.259	1.04067797	1.03185606		
	3	0.0256	0.0253	0.0257	0.0262	0.026	0.02576	0.351	1.02303415			
D1-8	1	0.002	0.0018	0.0024	0.0018	0.0019	0.00198	0.249	1.03125	1.01820565		
	2	0.0311	0.0309	0.0312	0.0314	0.0312	0.03116	0.182	1.00516129			
DRY	23-Sep-19	Day 35					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D1-4	1	0.0754	0.0758	0.076	0.0757	0.0755	0.07568	0.239	1.00185332	0.99814888	1.002236143	0.003151153
	3	0.0071	0.0073	0.0076	0.007	0.0068	0.00716	0.305	0.99444444			
D1-1	1	0.0119	0.0122	0.0126	0.012	0.0116	0.01206	0.371	1.0220339	1.00823697		
	3	0.0248	0.0251	0.025	0.0256	0.0247	0.02504	0.351	0.99444003			
D1-8	1	0.0019	0.0017	0.0022	0.002	0.0018	0.00192	0.192	1	1.00032258		
	2	0.0313	0.0308	0.0311	0.0304	0.0315	0.03102	0.432	1.00064516			
WET	30-Sep-19	Day 42					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D1-4	1	0.076	0.0765	0.0758	0.0762	0.0766	0.07622	0.335	1.00900185	1.04616759	1.017418747	0.002870134
	3	0.008	0.0076	0.0074	0.0079	0.0081	0.0078	0.292	1.08333333			
D1-1	1	0.0123	0.0121	0.0118	0.0122	0.012	0.01208	0.192	1.02372881	1.01424725		
	3	0.0254	0.0248	0.0259	0.0251	0.0253	0.0253	0.406	1.00476569			
D1-8	1	0.0021	0.0016	0.002	0.0018	0.0019	0.00188	0.192	0.97916667	0.9918414		
	2	0.031	0.0313	0.0315	0.0307	0.0312	0.03114	0.305	1.00451613			
DRY	07-Oct-19	Day 49					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D1-4	1	0.0757	0.0756	0.0761	0.0753	0.0754	0.07562	0.311	1.00105904	0.99636285	0.9963119	0.00348788
	3	0.0075	0.0071	0.0069	0.0069	0.0073	0.00714	0.261	0.99166667			
D1-1	1	0.0117	0.0121	0.0116	0.0125	0.0115	0.01188	0.415	1.00677966	1.00100699		
	3	0.025	0.0254	0.0255	0.0246	0.0248	0.02506	0.385	0.99523431			
D1-8	1	0.002	0.0016	0.0023	0.0017	0.0019	0.0019	0.274	0.98958333	0.99156586		
	2	0.0307	0.0311	0.0314	0.0305	0.0303	0.0308	0.447	0.99354839			
WET	14-Oct-19	Day 56					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D1-4	1	0.0761	0.0762	0.0755	0.076	0.0754	0.07584	0.365	1.00397141	1.03115237	1.01543912	0.003437839
	3	0.0081	0.0077	0.0076	0.0074	0.0073	0.00762	0.311	1.05833333			
D1-1	1	0.0125	0.012	0.0115	0.0122	0.0121	0.01206	0.365	1.0220339	1.01617978		
	3	0.0257	0.0249	0.0253	0.0254	0.0259	0.02544	0.385	1.01032566			
D1-8	1	0.0019	0.0017	0.0023	0.0015	0.0021	0.0019	0.316	0.98958333	0.99898522		
	2	0.0311	0.0308	0.0316	0.0313	0.0315	0.03126	0.321	1.0083871			
DRY	21-Oct-19	Day 63					Avg	SD x10^3	Dt/Do	Dt/Do	Dt/Do	Avg SD
D1-4	1	0.0755	0.0756	0.0755	0.0751	0.0756	0.07546	0.207	0.99894096	0.98974826	0.994239018	0.002973177
	3	0.0071	0.0069	0.0067	0.0075	0.0071	0.00706	0.297	0.98055556			
D1-1	1	0.012	0.0122	0.0117	0.0114	0.0115	0.01176	0.336	0.99661017	0.99393654		
	3	0.0252	0.0246	0.0255	0.0247	0.0248	0.02496	0.378	0.99126291			
D1-8	1	0.0018	0.0016	0.0023	0.0018	0.0021	0.00192	0.277	1	0.99903226		
	2	0.0311	0.031	0.0313	0.0307	0.0306	0.03094	0.288	0.99806452			

APPENDIX C CHEMICAL ANALYSIS CALCULATIONS FOR TRICALCIUM ALUMINATE

Calculations according to ASTM C150/150M (2015) - Section A1.3

Calculation is based on the percentages of aluminum oxide (Al_2O_3) and ferric oxide (Fe_2O_3)

Table C - 1 Weight % of fly ash, cement and gypsum (as displayed in Table 5-2):

Oxide	Units	Fly Ash	Cement	Gypsum
Al_2O_3	Weight %	20.87	4.00	0.77
Fe_2O_3	Weight %	6.55	2.34	0.35

The cementitious material of the concrete mix was made up of 50% fly ash, 35% cement, and 15% gypsum, so the total weight % of each of the oxides is calculated as follows:

Al_2O_3 : 20.87(0.5) + 4.00(0.35) + 0.77(0.15) = 11.95%

Fe_2O_3 : 6.55(0.5) + 2.34(0.35) + 0.35(0.15) = 4.15%

Percentage of aluminum oxide to ferric oxide (Al_2O_3/Fe_2O_3): 11.95/4.15 = 2.81

Since (Al_2O_3/Fe_2O_3) > 0.64, the tricalcium aluminate is be calculated as follows:

Tricalcium aluminate (C_3A) = (2.650 x % Al_2O_3) – (1.692 x % Fe_2O_3)

C_3A = 2.650 (11.95) – 1.692 (4.15)

C_3A = 24.65 %