

## ABSTRACT

PAZMINO, JOSUE ALBERTO. Fat Oil and Grease Formation and Adhesion to Concrete Treated with Soy Methyl Ester. (Under the direction of Dr. Mohammad Pour-Ghaz and Dr. Joel Ducoste).

Concrete sewer collection systems are fundamental parts of the infrastructure in a community since they carry wastewater to a safe disposal location. Sewer systems are designed for a defined flow capacity and if blockages disrupt the flow, they will generate Sanitary Sewer Overflows (SSOs) and release raw sewage to the surrounding environment. Many factors can contribute to blockage in the sewer lines; one of them is Fat, Oil, and Grease (FOG) deposition inside the sewer pipe surface. Research have shown that the FOG formation mechanism is complex and formed by a saponification reaction between long-chain free fatty acids (LCFFAs) and calcium ions present in wastewater or leaching from concrete corrosion that leads to the release of calcium hydroxide. One method to reduce the leaching of calcium hydroxide from concrete is using sealants to eliminate or reduce corrosion of concrete by reducing the interaction of concrete with the wastewater. In this research, Soy Methyl Ester (SME) was used as both an admixture (mixed with concrete) and coating and its impact on FOG deposit formation on concrete was studied. The hypothesis of this research is that SME can reduce leaching of calcium and therefore may reduce the adhesion and formation of FOG deposits in concrete sewer systems.

In this research, SME-treated samples were tested using three different tests evaluating different mechanisms involved during the FOG deposit formation: (i) calcium leaching test from concrete, (ii) acid attack with sulfuric acid, and (iii) FOG deposit formation test.

The concrete used had a water-to-cement ratio (w/c) of 0.42 which is the most common w/c for concrete pipes; two types of aggregates were used: a calcium based aggregate, limestone,

and a silica based aggregate, granite. Concrete samples were treated with topical plain SME and another type of concrete was treated with SME as an admixture during the mixing process.

First, leaching test was conducted to quantify the amount of calcium hydroxide leaching from untreated and SME treated concrete (SME applied topically or intermixed). The test involved exposing concrete to water with controlled pH = 7.0 and measuring calcium leaching in the solution water using the Inductively Coupled Plasma-Atomic Emission Spectrometry test. Results show that SME coating reduces the calcium concentration in the solution, and admixture SME may reduce the calcium hydroxide leaching depending on the concentration used during the mixing process.

The second test are the acid attack on concrete samples following ASTM C 267 test method for chemical resistance against sulfuric acid at pH = 1. Results indicate that SME coating reduced resistance to acid attack when compared with control samples in long-term exposures; however, samples with admixture SME showed better resistance in the first 30 days of testing as compared to control samples.

The third test was the FOG deposit formation test to simulate the conditions of wastewater; the simulated wastewater was made with oleic acid and canola oil in a solution of DI water. Results showed that regardless of the treatment of SME, a considerable amount of FOG deposit forms around the concrete surface, and a high variation was found in each test. Overall, SME treatment did not reduce FOG deposit adhesion to the concrete surface.

Overall, while SME treatment can reduce calcium leaching from concrete, tests results do not reveal why it does not reduce the FOG deposits despite formation. Further research is needed to fully understand the mechanisms involved.

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Fat Oil and Grease Formation and Adhesion to Concrete Treated with Soy Methyl Ester

by  
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## **DEDICATION**

To my family, friends, and all the people that support me, and taught me during this journey.

## **BIOGRAPHY**

Josue was born in Panama City, Panama, on November 24, 1990. He completed his high school studies at Instituto Episcopal San Cristobal in 2008 in Panama City. Thereafter, he attended the Technological University of Panama to obtain a BS in Civil Engineering in February 2015 and then an M.E in Structural Engineering in November 2019 at the same university. Since 2015, Josue starts working as a Civil Engineering in the Laboratory of Testing Materials at the Technological University of Panama involved in control quality projects of construction materials like concrete, metallic materials, wood, and ceramics; also involved in projects of structural pathology, and lecturer of construction material laboratory. In 2019, Josue received a Fulbright-Laspau grant to start an M.S. in Civil Engineering focusing on Mechanics and Materials area at North Carolina State University (NCSU) supervised by Dr. Mohammad Pour-Ghaz and Dr. Joel Ducoste studying the mechanisms of FOG deposit formation in concrete sewer systems, calcium hydroxide leaching, and acid attack in the concrete surface.

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# Chapter 1.

## Introduction

### 1.1 General

The formation of Fat, oil and grease (FOG) deposits is a significant problem affecting the sewer collection systems because FOG deposition inside pipes or other components of the sewer system may reduce their flow capacity and generate a phenomenon known as sanitary sewer overflow (SSO). SSOs lead to the release of raw sewage to the surrounding environment and impact public health. Previous research show one necessary component for the formation and solidification of FOG deposits is the availability of calcium in the wastewater [1]. Concrete, a material used in sewer collection systems, can be a source of calcium release in wastewater because of the calcium hydroxide, a product of the cement hydration which is known to be a water-soluble compound.

Different strategies have been proposed to reduce calcium hydroxide content in concrete. One strategy is the addition of supplementary cementitious materials (SCMs) that react with calcium hydroxide and produce calcium-silicate-hydrate, a more stable compound in concrete [2]. While effective, this strategy can only be applied to new sewer system construction or when the pipes are replaced. Other solutions are needed for the existing sewer systems. In these cases, the protection of the concrete surface with a coating can create a new method to protect concrete against external attacks from the wastewater, reduce calcium leaching, and potentially reduce FOG deposit formation. In wastewater media, coatings may create a barrier that prevents the

leaching of calcium hydroxide from the cement paste reducing the calcium concentration in the water. In this study, Soy Methyl Ester (SME), a derivative of soybean oil is used as a concrete sealant. SME has been used as a sealant for concrete because of the water repellent properties and the capability to fill the pores in cement paste [3-4]. The research questions of the present study include: (i) can SME used as coating and admixture reduce the leaching of calcium hydroxide from concrete? (ii) Can SME increase the durability of concrete against acid attack? and (iii) can SME reduce the formation of FOG deposits in sewer systems as well as reduce the attachment of these deposits to the concrete surface?

To assess these questions, control concrete samples, concrete with SME as admixture, and concrete with SME coating treatment were used to evaluate their capacity to reduce the calcium hydroxide leaching, resist acid attack and FOG deposit formation. In addition to the samples, three different tests were conducted to better understand the complex reactions involved in the formation of FOG deposits on the concrete surface. The first test consists of evaluating the calcium hydroxide leaching of concrete samples with SME as admixture or coating by submerging the samples in water and measuring the calcium content in water up to 50 days by using an Inductively Coupled Plasma-Atomic Emission Spectrometry test. Previous research has shown that the formation of FOG deposits is the product of the reaction of calcium ions in the water with free fatty acids. This reaction generates the release of a hydrogen ion that reduces the pH in the wastewater creating an acid attack around the concrete surface [1-2], and [5]. For this reason, the second test assessed the durability of concrete during acid attack using ASTM C267 standard to concrete samples where concrete control samples and SME treated samples were submerged in a sulfuric acid media with pH of 1. The third test was the FOG deposit formation

test which simulates the conditions in sewer lines and is used to quantify the formation and adhesion of FOG deposits on concrete surface.

## **1.2 Objectives**

The following are the objectives assessed in this work:

1. Investigate the impact of SME used as an admixture and sealant on reducing calcium leaching from concrete
2. Investigate the impact of SME on the resistance of concrete against acid attack by measuring the compressive strength and weight loss during exposure to acid attack
3. Investigate the impact of SME on FOG deposit formation and adhesion to the concrete surface.

## **1.3 Organization**

This thesis is organized in five chapters, starting with this introduction as Chapter 1, Chapter 2 is the literature review where a detailed description of the problem is provided. Chapter 3 is the description of all the methodology used for the investigation of the objectives. Chapter 4 presents the results for the test described in previous chapters. And Chapter 5 presents the final conclusions.

# Chapter 2.

## Literature Review

### 2.1 Sanitary Sewer Overflows

The sewer collection systems are integral parts of the infrastructure in a community protecting the environment by conveying wastewater to a treatment plant. Sewer systems are designed for a defined capacity of flow, and they should operate correctly under these expected conditions, which mean that the system's hydraulic capacity can direct the flow of wastewater. However, some extreme conditions like storm events, or flow restrictions due to the reduction in the inner diameter of the pipe, called blockages, may cause spills from sewer overflow. Blockages are produced from different factors like debris buildup, objects that obstruct the sewer, accumulation of fat, oil, and grease (FOG) deposits, or a combination of these factors. For instance, the Water Environment Research Foundation found a sewer blockage with a combination of roots and FOG deposits [6]. It was reported that 21% and 47% of all sewer line blockages were caused by FOG deposits in Australia and the United States, respectively [6-7].

If blockages interrupt the sewer system's inner flow, this will have risk of flooding and generates Sanitary Sewer Overflows (SSOs). In the United States, 23,000 to 75,000 SSOs occur annually, releasing approximately 3 to 10 billion gallons of untreated wastewater [8], and in the UK, 25,000 flooding events per year are due to sewer blockages [9]. SSOs are mainly wastewater, which harms health and the environment as they contain harmful chemical and biological contaminants that affect the health of communities. In addition, SSOs may release

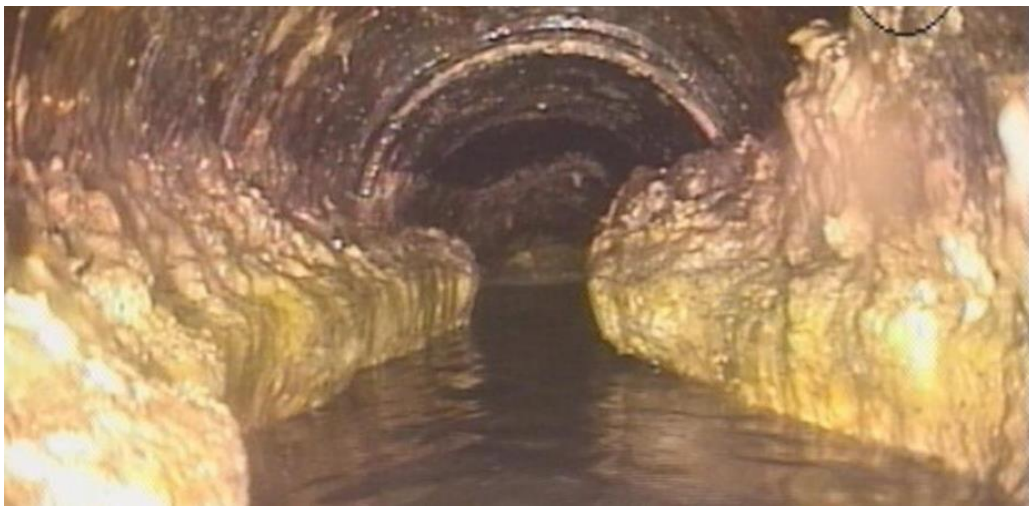
highly toxic contaminants, such as personal care products and endocrine disruptors, among others, that can alter the ecosystem with very low concentrations [7].

The United States Environmental Protection Agency (EPA) estimates that in the United States, the total cost of replacing all the aging sewer systems could be more than \$1 trillion with the replacement of a single large municipality's collection system could be billions of dollars and that of a smaller city on the order of millions of dollars. Sewer collection system rehabilitation to reduce or eliminate SSOs may be too expensive leading to the need to develop an alternative solution. In addition to replacement, sewer maintenance and rehabilitation generate additional value by extending the service life of sewer collection systems. The cost of rehabilitation and other measures to reduce SSOs can vary widely by community size and sewer system type [8].

## **2.2 Fat, oil and grease (FOG) and the formation of solid deposits**

Keener et al. [10] assessed the properties that influence the physical and chemical properties of FOG deposits that are found in sewer collection systems to understand their characteristics and formation. In their study, samples from 23 different locations, mainly in the South East USA were collected and analyzed. Their results showed that FOG deposit chemical and physical properties varied with location, but no correlation was found between water hardness and FOG deposit properties. In 84% of FOG samples, calcium was, however, the most common metal. Based on their results, Keener et al. proposed that the saponification reaction was likely responsible for the FOG deposit formation and possibly encouraged from the use of alkali products such as those in detergents, degreasers, and sanitizers. A later study confirmed this saponification processes in FOG deposit formation using Fourier Transform Infrared (FTIR) spectroscopy and compared similar FTIR profiles between lab based FOG deposits and FOG deposits recovered from sewer collection systems. They clearly showed that FOG deposits are

soaps of fatty acids made of calcium [1]. The free fatty acids found in wastewater are generally caused from chemical and hydrolysis reactions of the oil from food frying or biological processes in the wastewater stream [11] and [5]. While there was a study that did show no correlation between water hardness and FOG deposit formation [10], other researchers have shown that the calcium in the water from calcium hydroxide leaching from concrete can also contribute to the formation of these insoluble deposits [1, 12-13].



**Figure 2.1** FOG formation in sewer system [14]

In addition, Keener [10] showed evidence that a large number of FOG deposits form inside the cross-section of the pipe form above the low-flow water line; this suggests the preference for oil and free fatty acids to accumulate at the water surface. The hydrophobic nature of these compounds may also work as a driving force to accumulate them at the interface between water, air, and concrete surface. FOG formations tests performed by He et al. [12] observed white solid particles of FOG adhered to the concrete surface above the water line; this formation around the concrete may be due to the surface roughness and the free fatty acids from oil able to accumulate all over the concrete helped by the circulation of water throughout the pipe. In addition, Iasmin et al. [5] noticed that the background calcium present in wastewater

forms smaller and more granulated soap in comparison with calcium released from high pH surface conditions such as concrete corrosion, which produces significantly larger and more viscous solids that potentially may adhere more easily to pipe surfaces.

Keener et al. [10] and Williams et al. [13] found a high concentration of iron, aluminum, and silicon; which are readily found in combination with cement (i.e., calcium aluminoferrites, calcium aluminates, and calcium silicates, as mentioned in Kosmatka [15]). Concrete sewer pipes may be exposed to severe corrosion conditions. The concrete pipes not only may corrode by substances in the groundwater (e.g., sulfates) but are also prone to corrosion due to exposure to the sewage and microbial activity [16]. In cement production, the cement reacts with water in order to hold together aggregates and provide mechanical and durability performance. The mixture of water and cement is commonly referred to as cement paste, where the major components of the cement are  $C_3S$  tri-calcium silicates,  $C_2S$  di-calcium silicates,  $C_3A$  tri-calcium aluminates, and  $C_4AF$  tetra-calcium aluminoferrite. Hydration of  $C_2S$  and  $C_3S$  produces CSH, also called calcium-silicate-hydrates, and calcium hydroxide (CH). CSH is a component that is stable and gives strength and durability properties to the cement paste; however, CH is a component that is water soluble, especially in corrosive media or solutions where the pH is lower than concrete pH.

Hence, in FOG deposit formation, calcium released from the corrosion of cementitious materials may have a more adverse impact than background calcium found in wastewater as a high pH saponification reaction occurs at the cementitious materials' surface. Therefore, in addition to the current strategies of limiting FOG discharge from the kitchen sink, reducing calcium ions released into wastewater from concrete corrosion can be an alternative strategy to reduce the FOG deposit formation. A common practice to reduce the amount of CH and increase

the CSH in the components of cement paste the use of Supplementary Cementitious Materials (SCMs) such as fly ash, slag cement, silica fume, or a combination [2]. However, there are existing concrete sewer or pipe systems that were constructed without the addition of SCM; for these cases, other approaches must be evaluated in order to mitigate calcium hydroxide leaching in water.

### **2.3 Acid attack in concrete sewer systems**

For concrete used in sewer and pipe systems, microbial-induced concrete corrosion (MICC) is a problem that may affect the pipe in different stages. MICC has three different stages: First, reduction of moisture pH in the concrete surface, reducing from approximately 13 to 9 through chemical reactions with CO<sub>2</sub> and other acidic gasses in the headspace of sewer systems; Second is the colonization of sulfur-oxidizing microorganisms (SOM) on the moist surfaces of sewer lines that reduces the pH below three; and third is the growth of acidophilic microorganisms that converts sulfur and other reduced sulfur compounds to sulfuric acid, reducing the pH to values closer to 1 [17,18,19]. This final stage of MICC that produces sulfuric acid in combination with the leaching of calcium during the FOG formation is one of the main reasons that create concrete corrosion process inside sewer lines.

Differences between microbial-induced concrete corrosion and chemical acid attacks were reported in previous research studies [20-21]. Additionally, other researchers found that the chemical acid attack in general is similar to the final stage of MICC [18] and [22]. MICC as some of the durability problems, is a long-term process that it is not easily implemented using laboratory experiments [17] because of the microbial factors. For this reason accelerated acid exposure tests on concrete is performed to determine the deterioration of MICC on concrete [18],

and [22-23]. In this research, the durability of concrete treated with SME was evaluated using a chemical acid attack test with a test media at pH 1 using sulfuric acid.

## **2.4 Concrete Topical Sealants**

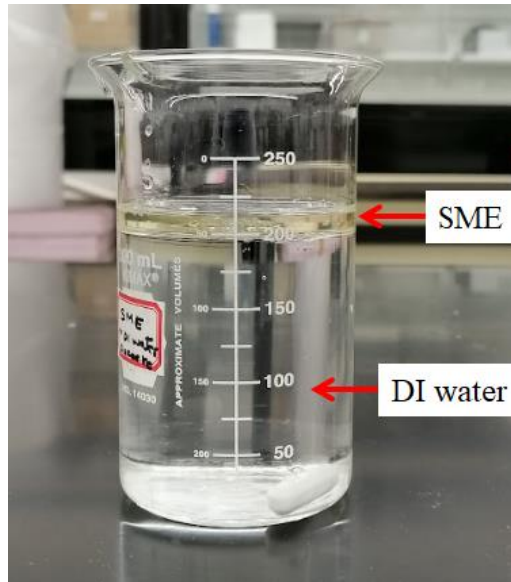
Application of topical concrete sealants and surface treatment is a method to reduce corrosion and calcium leaching from concrete. As Nielsen [24] mentioned, surface treatment in concrete is often used to mitigate the ingress of aqueous components and chemicals from entering into the pores of cement paste. After the hardening of a concrete product of hydration, water can enter the concrete and produce adverse effects on the integrity of the cementitious materials. Many durability problems are associated with the transport of fluids because aqueous fluids are able to dissolve and enter the pores media by diffusion or capillary action. The chemicals that dissolve and transport may be components or ions such as oxygen, sulfates, chlorides, and carbon dioxide that have deleterious effects in cement paste. Sealing pores media of concrete could effectively slow down the mechanisms that are dependent on the exposure to water and other chemicals that can be harmful to concrete and steel reinforcement [24]. Implementing optimized concrete proportioning and modern mixture design and materials may help to reduce water transport and increase the service life of the concrete. However, insufficiency in construction practice and placement of concrete may cause a reduction in the durability properties of the materials; in addition, a phenomena like early age cracking is often a problem that is noted in newly placed concrete elements. Concrete topical sealers can increase the service life of old concrete elements as long as they are applied to surfaces not worse than a low to moderate state of distress, as defined by FHWA-RD-03-031 [25].

## 2.5 Background on Soy Methyl Esters (SME)

One solution used to enhance the durability and, in consequence, the service life of concrete is the use of topically applied surface treatments. In this study, soy methyl ester (SME) will be used as a topical treatment. Generally, oil crops, like soybean, are used to feed livestock [26]. The oil extracted from these products has different uses in the fabrication of soaps, crayons, lubricants, and candles, among others, including the production of SME through alkaline-catalyzed transesterification with methanol, where methyl esters are produced from plant triacylglycerides (oil). SMEs consist of plant oils composed of long-chain fatty acids (14 to 20 carbons), which are esterified to a methyl group. Each molecule of triacylglyceride reacted requires three molecules of methanol, producing three molecules of methyl esters and one molecule of glycerin [3].

Soybean derivatives as SME have proven to be examples of biodegradable, nontoxic, non-corrosive, renewable, and economical resources that could be useful for the construction industry [3]. In the case of concrete, SME has the possibility to repel water (Figure 2.2) or at least reduce the fluid transport inside the pores media as mentioned in previous research where the water absorption is reduced [3-4]. Also, it has been reported that application of SME as sealant to concrete increases concrete's resistance to deteriorations caused by to freezing and thawing, chloride ingress, and ASR [27].

In addition, their biodegradable and non-toxic form may modify the concrete properties to "green" concrete. Studies have shown that SME has the potential to be used in the construction industry as a remover in the case of asphalt or as a concrete sealant to reduce water absorption [27]. The SME has the alternative to being used as a coating or either as an admixture.



**Figure 2.2** SME separates from the DI water

For coating purposes, researchers recommend a rate of application between  $0.02 \text{ g/cm}^2$  and  $0.05 \text{ g/cm}^2$  [28] and [3]. Another factor that has to be considered when SME is used as a coating is the penetration depth which is dependent on concrete mixtures, the viscosity of the SME, and time. As the concrete moisture level increases, the amount of SME absorbed will decrease. When first exposed to the concrete, SME will quickly penetrate. Over time this rate slows [29]. Experimental results show that the greater the penetration depth of SME, the more effective it is at increasing concrete durability [27]. Golias et al. also recommends that the rate of SME applied to concrete elements should be maximized in order to attain a higher resistance to deleterious mechanisms. This can be accomplished by ensuring that the concrete surface is free from moisture before applying the coating and allowing ample time for the sealant to penetrate the concrete. Therefore, it is also important that application not occur when temperatures are below the dew point, which is the point where liquid water will condense on solid surfaces and may reduce the penetration of coatings inside pores. These considerations ensure the pores of the concrete are open and available to absorb the applied SME.

Because of the hydrophobic nature of SME, using this component as an admixture presents practical problems in mixing. Bisnett et al. [28] observed that when SME is in contact with dry constituent materials like unhydrated cement and aggregates before they come into contact with mixed water; the SME will coat the particles of materials and will tend to agglomerate during the mixing process, and only high shear rates of mixing could solve the agglomeration of particles. One recommendation to avoid the condition of agglomeration is the addition of SME into cementitious mixtures after all other constituent materials have been added to the mixture in accordance with ASTM C305, resulting in more homogeneous mixtures. However, the workability of the concrete mixture tends to reduce because of the oily behavior of SME; therefore, new alternatives should be tested to improve this property in fresh concrete.

Since SME reduces the water absorption in concrete it can possibly reduce calcium hydroxide leaching because calcium hydroxide leaching requires water transport. Reduced calcium hydroxide leaching can also reduce the formation of FOG inside concrete sewer systems.

SME can dissolve polystyrene (PS) to create a SME-PS blend. Adding polystyrene to the SME alters the viscosity of the SME. These modifications also influence the sealant capacity of SME to adhere to the surface of the concrete and in consequence may help to reduce the fluid transport into concrete [29].

# Chapter 3.

## Methodology

### 3.1 Test Overview

This chapter presents descriptions of the tests performed in the laboratory. As mentioned in Chapter 2, the soluble calcium hydroxide will leach from the concrete sewer pipes; this available calcium in the solution reacts with the free fatty acids in the wastewater, producing a saponification reaction in the solution and also at the concrete surface-air-wastewater interface and produce FOG deposits; in addition, the saponification reaction or also microbial attack will produce concrete corrosion through an acid attack. In this research, concrete treated with SME as coating or admixtures was tested. Unless otherwise noted, for this research, plain SME without any addition of polystyrene was used for coating and admixture treatment.

To simulate and quantify the calcium leaching from concrete, a laboratory-made leaching test was performed by placing concrete cubes of 1-inch in water inside a beaker over a magnetic plate. Samples from water were taken periodically to measure the concentration of calcium ions in the solution. Acid attack tests was performed by following the procedure of ASTM C267 with concrete 2-inch cubes submerged inside a solution of sulfuric acid and evaluated after several days of deterioration changes in dimensions, weight, physical appearance, and compression strength. In addition, a FOG formation test was performed by partially submerging concrete samples in laboratory-made synthetic wastewater. The FOG generated from the saponification reaction of available calcium and free fatty acids was weighted to measure the total FOG

generated. Additionally, 4x8 concrete cylinders with 5% SME by mass of cement were tested in compression to determine if the addition of 5% SME reduces the concrete strength. Experimental tests are summarized in Table 3.1 and in the following sections of this chapter.

**Table 3.1** Testing Program Overview

Test Description	Test Method	Mixture*	SME Treatment
Compression Test	ASTM C39	LO	Control
		LSI5.0	Admixture
	Concrete Cubes	LO	Control, Coating (1 dose and 4 doses)
		GO	
		LSI2.5	Admixture
LSI5.0			
Leaching Test	Calcium leaching from concrete samples	LO	Control, Coating (1 dose and 4 doses)
		GO	
		LSI2.5	Admixture
		LSI5.0	
		GSI2.5	
Durability Test	ASTM C267, in acid media	LO	Control, Coating (1 dose and 4 doses)
		GO	
		LSI2.5	Admixture
		LSI5.0	
FOG Formation Test	Fat, oil and grease formation under synthetic wastewater	LO	Control, Coating (1 dose and 4 doses)
		GO	
		LSI2.5	Admixture
		LSI5.0	
		GSI2.5	

\* Mixture proportions defined in Table 3.3

To avoid misinterpretation with the description and nomenclature of samples in further sections of this document, each sample tested is identified with the type of concrete used, description, and nomenclature shown in Table 3.2.

**Table 3.2** Samples Identification for this project

Concrete Samples	Description	Nomenclature
Limestone Only	Concrete with limestone aggregate with no SME treatment	LO
Limestone SME Intermix 2.5%	Concrete with limestone aggregate with 2.5% of SME by mass of cement as admixture	LSI2.5
Limestone SME Intermix 5.0%	Concrete with limestone aggregate with 5.0% of SME by mass of cement as admixture	LSI5.0
Limestone SME Topic 1 Layer	Concrete with limestone aggregate with 1 dose of SME as coating	LST1
Limestone SME Topic 4 Layer	Concrete with limestone aggregate with 4 dose of SME as coating	LST4
Granite Only	Concrete with granite aggregate with no SME treatment	GO
Granite SME Intermix 2.5%	Concrete with granite aggregate with 2.5% of SME by mass of cement as admixture	GSI2.5
Granite SME Topic 1 Layer	Concrete with granite aggregate with 1 dose of SME as coating	GST1
Granite SME Topic 4 Layer	Concrete with granite aggregate with 4 dose of SME as coating	GST4
Granite SME Topic 1 Layer - PS 2%	Concrete with granite aggregate with 1 dose of SME and 2% of polystyrene as coating	GST1-2%PS

### 3.2 Materials and mixture proportioning

In this study, two types of coarse aggregates were used to prepare concrete mixtures: (i) Limestone, a calcium-based aggregate with a specific gravity of 2.36 and absorption of 3.9%, and (ii) Granite, a silica-based aggregate with a specific gravity of 2.61 and absorption of 0.60%. The fine aggregate was the same for all mixtures and has a specific gravity of 2.60 and absorption of 0.82%. ASTM C150 Type I ordinary Portland cement (OPC) was used, and all the concrete mixtures were prepared with a water-to-cement ratio (w/c) of 0.42 where the mix water was regular tap water and conditioned to 23°C± 2°C. Five types of concrete mixtures were

prepared: 1) only limestone (LO), 2) limestone with 2.5% by mass of cement of SME intermix (LSI2.5), 3) limestone with 5.0% by mass of cement of SME intermix (LSI5.0) 4) only granite (GO), and 5) granite with 2.5% by mass of cement of SME intermix (GSI2.5). Where mixtures identified as LO and GO are control samples, and LSI2.5, LSI5.0, and GSI2.5 are samples treated with admixture SME. The amounts of materials used for this project are shown in Table 3.3.

**Table 3.3** Mixture proportioning

Materials	LO	LSI2.5	LSI5.0	GO	GSI2.5
Cement (Type I) (kg/m <sup>3</sup> )	407	407	407	407	407
Water (kg/m <sup>3</sup> )	171	171	171	171	171
Fine Aggregate (kg/m <sup>3</sup> )	780	780	780	780	780
Coarse Aggregate (kg/m <sup>3</sup> )	944	944	944	1044	1044
SME Intermix (kg/m <sup>3</sup> )	-	10.17	20.35	-	10.16
Super-P Plasticizer (g/100g cem.)	-	-	0.45	-	-



**Figure 3.1** Homogenization of SME and water with an ultrasonic probe

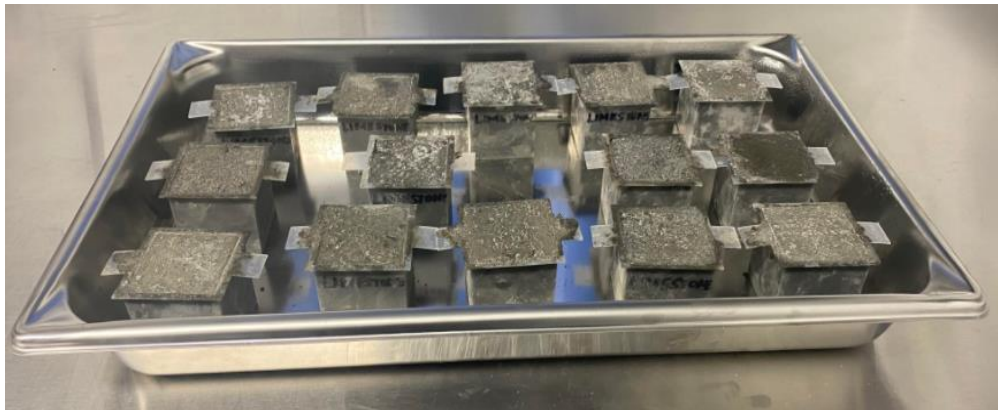
### 3.3 Casting and Curing

Cast concrete mixtures with SME intermix is challenging, as described in Section 2.5, for this reason, two procedures were used for the 2.5% SME intermix (LSI2.5) and 5.0% SME intermix (LSI5.0). In the case of LSI2.5, SME was added after cement, aggregates, and water were mixed. However, for LSI5.0, the increase of SME also increases mixture's viscosity and reduces workability. Therefore at first, the SME was mixed with a handheld ultrasonic homogenizer 8 mm probe in a beaker with approximately 200 mL of the mix water, as shown in Figure 3.1, and then immediately added to the rest of the mixture materials in the mixer. When all the materials (including the homogenized SME) were mixed, 0.45% by mass of cement plasticizer (based on polycarboxylate polymer technology) was added to increase the workability of the mixture.

A 2x2 inch cross-section square PVC tube of 12-inch height was used to cast samples for the leaching and FOG deposit formation test (Figure 3.2). Molds were filled in three layers and vibrated for compaction after each layer. A flexible PVC cap was used on both ends of the PVC tube to seal the mold. For the chemical durability test, 2-inch cube samples were prepared using a cube mold and sealed cured at room temperature at  $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$  until tested (Figure 3.3). All the samples were cured for 90 days prior to tests. In addition, half of the control samples (LO and GO) was coated with topical treatment of SME (as described in Section 3.4) to evaluate their resistance against calcium leaching, FOG deposit formation and acid attack.



**Figure 3.2** PVC square tubes of 2-inch of width by 12-inch of length



**Figure 3.3** Two inches cubes for durability test

### **3.4 Topical treatment of SME in concrete surface**

After the curing period of 90 days, the specimens were cut from the control samples to the desired size using a wet saw cut; then cut specimens were left to dry in a conditioning room at a relative humidity of 50% for 15 days; this step is important because it will define the absorption capacity of SME inside concrete samples. Then samples were treated with SME as a coating applying a dose of  $0.02 \text{ g/cm}^2$  or  $0.150 \text{ mL/in}^2$  (assuming a density of  $0.8565 \text{ g/mL}$ , [28]) using a brush evenly across the surface. In the case of samples treated with 4 layers of SME, each layer was applied one week after the previous. After coating, the samples with the SME treatment were left curing in the conditioning room for another 30 days until the test started.

### 3.5 Compressive strength test

Compressive strength measurements were performed on cylindrical specimens (4-inch diameter and 8-inch height) LO and LSI5.0 at 14 and 28 days according to ASTM C39 [30] to assess the influence of the SME admixture. Before testing, the surface of the sample was monitored for flatness; any undulation on the surface was ground to meet the ASTM testing standard tolerance. Samples were capped with bearing plates on both ends, and the compression test was performed at a loading rate of 35 psi/s (Figure 3.4). Each sample contained three replicates, and the average of the three-compressive strength was reported.



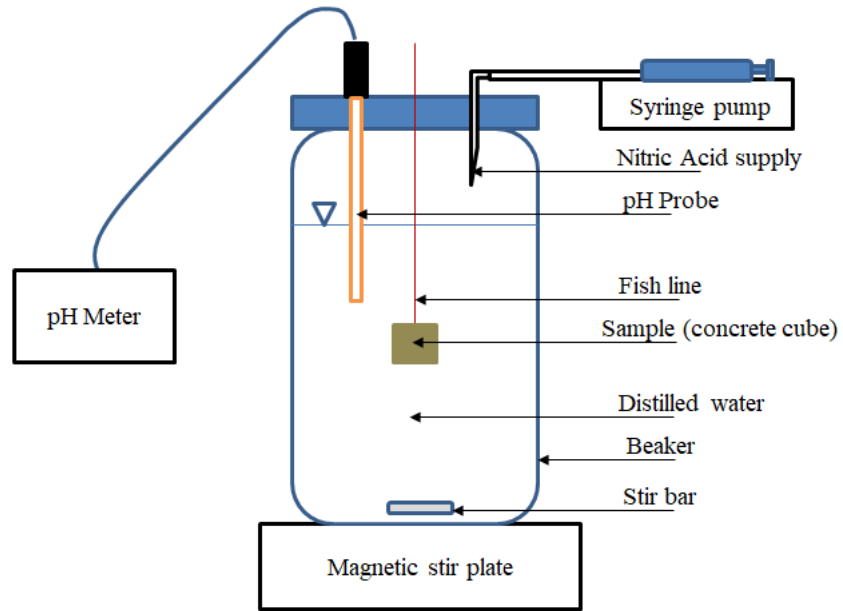
**Figure 3.4** Compression test on cylindrical samples

As mentioned by Bisnett [28], the compressive strength of 4x8 concrete cylinders with a dose of 1.25 % by mass of cement of SME-PS (Polystyrene addition) was studied, and the samples just showed slightly lower average strength in comparison with control samples, where the maximum reduction was 18% for the sample with SME-5%PS at 28 days.

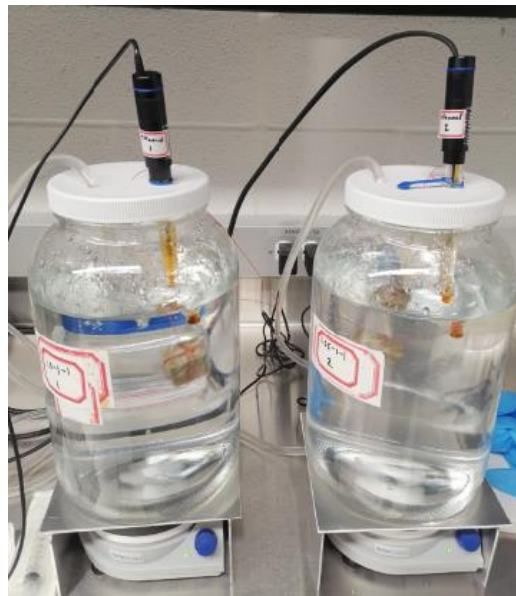
However, for the assessment of this project, a dose of 5% by mass of cement of SME was tested to determine the reduction in strength since the amount of SME used is greater than 1.25% by mass of cement. In addition, this test is also to determine whether, even with the reduction because the SME, the concrete meets with the minimum required compressive strength of 4 ksi according to ASTM C76 [31] and ASTM C655 [32].

### **3.6 Calcium Leaching Test**

The leaching test aims to determine whether the concrete treated with SME reduces the dissolution of calcium hydroxide (CH) in an aqueous solution. After 90 days of sealed curing, a 1-inch cube concrete sample was cut from the 2-inch PVC tube using a diamond blade saw. Then dimensions and weight of all samples were measured and then washed with DI water to remove dust from the cutting process. Three specimens of each type of mixture or treatment were tested, and the average is reported in the final result. The sample was fully submerged into a 3L leachate solution (distilled water) using a fish line. A magnetic stir plate was used to stir the water inside the beaker. Figure 3.5 and Figure 3.6 show the schematic diagram and experimental setup for the leaching test. The leaching test was performed for 50 days at  $\text{pH} = 7$  to simulate potential sewer wastewater conditions. The DI water solution pH tends to increase because of the leaching of CH from the cement paste; in consequence, the pH was monitored every 30 minutes by using a pH meter and maintained at the desired pH level by supplying nitric acid or sodium hydroxide solution through a syringe pump as needed.



**Figure 3.5** Schematic diagram for the leaching test



**Figure 3.6** Experimental setup for the leaching test

For calcium ion concentration measurement, leachate solution was taken from each test container three times a week in 15 ml centrifuge tubes. Leachate solution was then tested for calcium concentration by using Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES).

### 3.7 Concrete Durability Test against Sulfuric Acid Attack

To determine the suitability of SME treatment in sewer line construction materials, a chemical durability test on control and SME-treated samples was performed following ASTM C267 [33]. 2-inch cube concrete samples were cast using cube molds and cured under sealed conditions for 90 days. After the curing period, samples were taken out of the mold, weighed, and sample dimensions were measured using calipers. O'Connell [34] states that although the normal pH of sewage is slightly acidic and in the range of pH 5~6; previous research has shown that the surface pH of the sewer line can go as low as a range of 1 and 2 due to microbial acid attack [21] [35]. Hence, a test media of pH 1 was prepared using sulfuric acid to simulate the potential microbial acid attack inside sewer lines. Test media was replaced whenever the pH of the test media exceeded 1.5 for the first month of the total test duration. After 1 month of testing, the test media was replaced once a week. The duration of this test was 60 days. At the age of 0, 14, 30 and 60 days, three replicates from each container were observed to identify changes in physical appearance, weight, thickness, and compressive strength. Concrete cubes were submerged inside a 5 gallons container where the liquid-to-solid ratio was kept constant in all three containers at 4.00 of the test media (Figure 3.7).



**Figure 3.7** Chemical durability test set-up

The percentage loss or gain in weight of the specimens during exposure to acid for each examination period was calculated by the following equation:

$$\text{Weight change, \%} = [(W-C)/C] \times 100 \quad \text{Equation 3.1}$$

where, C = initial weight of specimen before immersion, and W = weight of specimen after immersion.

The thickness, weight, and compressive strength after immersion were measured by brushing the surface of the exposed concrete cubes, rinsing using running tap water, and then drying by blotting with a paper towel. After the final blotting, each specimen was allowed to dry for half hour before weighing. Following the period of half hour, the samples were weighted to the nearest 0.001 g. As mentioned in the literature, the weight change and thickness significantly differ between samples that are brushed and those not brushed samples [21].

Similarly, change in compressive strength was also measured by the following equation:

$$\text{Change in compressive strength, \%} = [(S2-S1)/S1] \times 100 \quad \text{Equation 3.2}$$

where, S1 = average compressive strength of a set of specimens following the conditioning period and S2 = average compressive strength of a set of specimens following the test period.

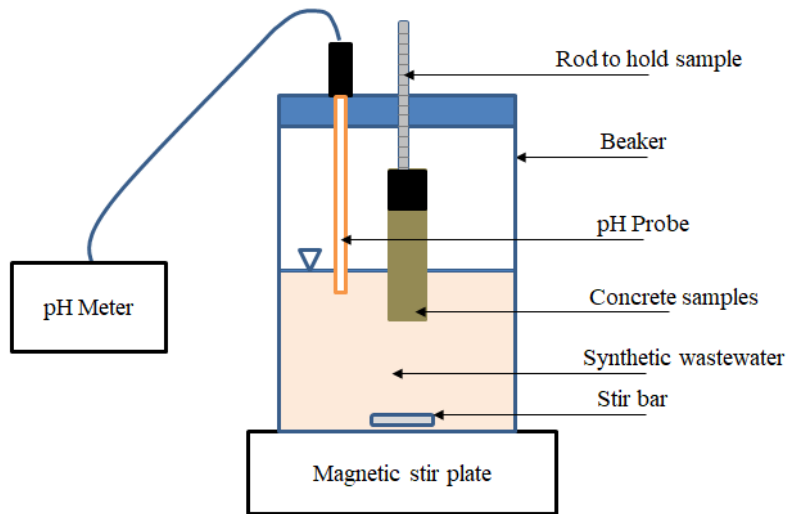
### **3.8 FOG deposit Formation Test**

For this task, concrete with limestone or granite aggregate using the mixture proportion of Table 3.3 was cast in a 2x2x12 inch PVC tube. From these tubes, concrete segment samples of 1-inch x 1-inch x 3-inch size were cut using a wet saw. After cutting, dimensions and weight of samples were measured and then washed with DI water to remove any dust remaining from the cutting process. Concrete specimens were exposed to synthetic wastewater with two different scenarios tested separately: the first one using deionized water (DI water) with external calcium

addition to the background bulk solution and the second scenario with DI water without any external addition of calcium. In the first scenario, the synthetic wastewater was prepared by mixing 700 ml of deionized water with the addition of 75 mg/l of calcium adding calcium chloride as a background calcium source to analyze the effect of residual calcium content in wastewater on FOG deposit formation inside sewer lines; in the second alternative, 700 ml of DI water without calcium background condition to specifically assess the calcium leaching component from the concrete to FOG deposit formation. Then, 30 g of oleic acid and 20 g of canola oil were added in all cases to simulate the occurrence of free fatty acids from oil hydrolysis and degradation in wastewaters. At first, canola oil, oleic acid, and DI water were mixed properly by mixing the resulting solution overnight using a magnetic stir plate and a stir bar. After that time, one-third of the 3-inch long concrete coupon was submerged in the synthetic wastewater; the rest of the coupon remained outside the solution and attached to the cap of the beaker with a rod and tape (Figure 3.8).

This FOG deposit formation test setup previously designed [2] and is adopted in this study to quantify the amount of FOG deposit formation and to analyze the effect of concrete corrosion on FOG formation inside sewer lines (Figure 3.8 and Figure 3.9). In the experimental setup, specimens were be exposed to synthetic wastewater for 20 days, with the stir bar rotating continuously to simulate the running water conditions in the sewer system. After completing the tests, two kinds of FOG deposit formation were analyzed; the first is the FOG deposit that forms and is attached to the surface of the concrete, and the second is the FOG deposit that forms and remains in the surrounding water at the end of the test, as shown on Figure 3.10. FOG deposits formed on the surface of the concrete coupons was removed with a spatula, then weighed in the moist condition on a scale, and then dried in an oven to determine the FOG deposit weight

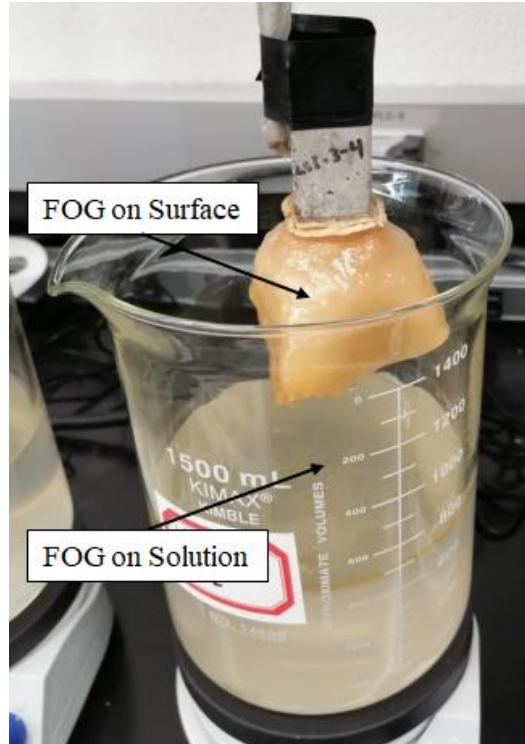
without moisture. FOG deposits formed in synthetic wastewater solution combines small particles of FOG deposits inside the water and oily content floating on the water. The oily content was first picked up with a spoon, and the small particles were filtered through a qualitative filter paper (sizes  $> 45 \mu\text{m}$ ); both contents were placed on an aluminum dish, the moist weight was measured, and then dried to measure the FOG deposit that formed inside the solution.



**Figure 3.8** Schematic diagram for the FOG deposit formation test



**Figure 3.9** Experimental setup for the FOG deposit formation test



**Figure 3.10** Difference between FOG deposit form on the surface and FOG on the solution

In addition to the availability of calcium in the wastewater, two pH conditions were tested. First, samples were tested in synthetic wastewater with adjustment of pH = 7 to simulate the field conditions where wastewater generally is in neutral state. Second, tests were performed without any pH adjustment to assess only the contribution of leaching of calcium hydroxide from concrete and it influences the FOG deposit formation. A summary of the FOG deposit formation tests is shown in Table 3.4.

**Table 3.4** Test Matrix for FOG deposit formation test in the laboratory

<b>Concrete Samples</b>	<b>With Ca<sup>2+</sup>, pH Adjustment</b>	<b>With Ca<sup>2+</sup>, No pH Adjustment</b>	<b>Without Ca<sup>2+</sup>, pH Adjustment</b>	<b>Without Ca<sup>2+</sup>, No pH Adjustment</b>
Limestone Only	✓	✓	✓	NT
Limestone SME Intermix 2.5%	✓	✓	✓	NT
Limestone SME Intermix 5.0%	NT	✓	NT	NT
Limestone SME Topic 1L	✓	✓	✓	NT
Limestone SME Topic 4L	NT	✓	NT	NT
Granite Only	✓	✓	NT	✓
Granite SME Intermix 2.5%	✓	NT	NT	NT
Granite SME Topic 1L	✓	✓	NT	✓
Granite SME Topic 4L	NT	✓	NT	NT
Granite SME Topic 1L - PS 2%	✓	NT	NT	NT

NT: Not tested

### 3.9 FTIR Analysis of FOG deposits

Fourier Transform Infrared (FTIR) analysis was performed on all the FOG deposit samples as well as on pure canola oil, calcium chloride solution, and oleic acid. The surface FOG deposits from the FOG deposition tests with and without background calcium were analyzed using FTIR to observe the changes in bond absorbance due to the saponification process, and their properties were evaluated using the criteria used by Poulenat [36]. The soap content of the FOG deposit was determined using Equation 3.3. For this analysis, two areas on the surface FOG deposit were studied. The first area identified as Surface layer (SL) is the FOG deposit that was directly adjacent to the surface of the concrete, and the second area is identified as Outer layer (OL), which is far from the concrete surface and directly adjacent to the water layer, as shown in Figure 3.11. Infrared absorption spectra of these samples were determined with a Digilab FTS-6000 Fourier Transform Infrared (FTIR) spectrometer using a mounted crystalline Zinc Selenide attenuated total internal reflection (ATR) sampling attachment (Pike Technologies Inc., MIRacle™ Single Reflection ATR). The absorbance spectra were computed from 400 cm<sup>-1</sup> to 4000 cm<sup>-1</sup> wavelength, and baseline correction was performed. The absorbance and wavelength data were then processed using data processing software (Microcal Origin, v7.0, Microcal Software Inc., Northampton, MA).

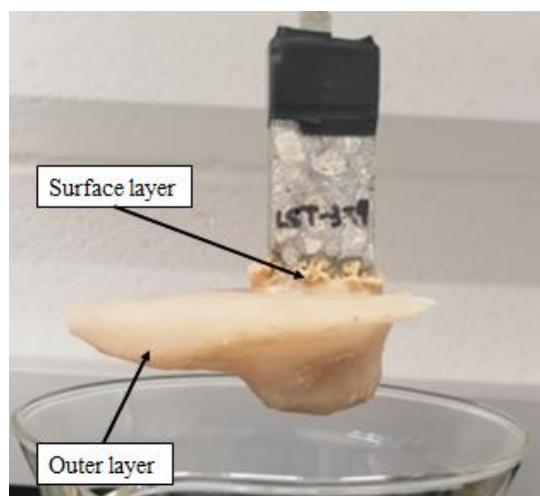
Soap(%)

$$= \frac{\text{Soap absorbance}(1577 \text{ cm}^{-1} + 1541 \text{ cm}^{-1}) * 100}{\text{Soap absorbance}(1577 \text{ cm}^{-1} + 1541 \text{ cm}^{-1}) + \text{Oil absorbance } 1740 \text{ cm}^{-1} + \text{LCFFA absorbance } 1707 \text{ cm}^{-1}}$$

Equation 3.3

According to Poulenat [36], the formation of calcium soaps can be attributed to four regions of wavelengths; region-1: 4000-3000 cm<sup>-1</sup>, region-2: 1800-1350 cm<sup>-1</sup>, region-3: 1350-1180 cm<sup>-1</sup> with additional side-band near 720 cm<sup>-1</sup>, and finally, region-4: near 670 cm<sup>-1</sup>. In

addition Poulenat also stated that calcium soap in hydrated form shows two peaks characteristic vibration at  $1577\text{ cm}^{-1}$  and  $1541\text{ cm}^{-1}$ .



**Figure 3.11** Difference between Surface layer and Outer layer in FTIR analysis

# Chapter 4.

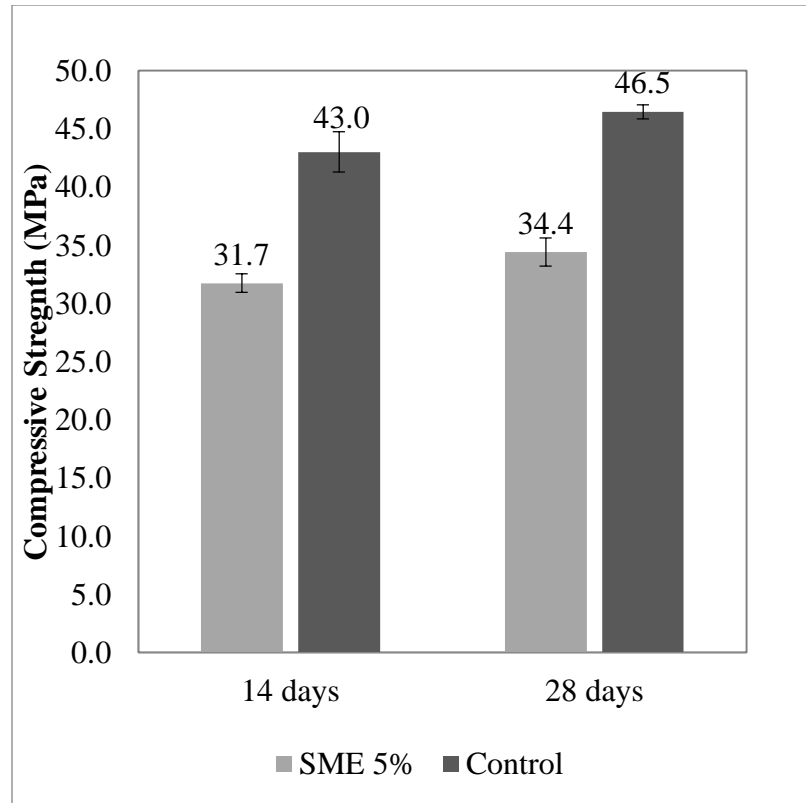
## Results and Discussion

### 4.1 Compression Test of samples treated with SME

#### 4.1.1 The effect of 5.0% of SME intermix on concrete cylinders

Compressive strength tests at 14 and 28 days of sealed curing were carried out to measure the strength of concrete containing 5% SME by mass of cement and compare against the control mixture that did not contain any SME. Figure 4.1 represents the average compressive strength of three cylinders at each age. In Figure 4.1, at 14 and 28 days, the strength of the LSI5.0 sample is 26.0% lower as compared to the control concrete. This suggest that SME as intermix may reduce the bonding between aggregates and reduce the hydration of cement paste in early ages, however, more studies are needed to confirm this hypothesis.

According to ASTM C76 and ASTM C655 for class 3/ class 4 standard reinforced concrete pipe segments, compressive strength of 4 ksi (28 MPa) is required for sewer line construction. For both control concrete and concrete with 5.0% of SME as an admixture, the strength was 34.4 MPa, exceeding the requirement of the ASTM standard.



**Figure 4.1** Compressive strength of LO and LSI5.0 over time

#### **4.1.2 Concrete cubes treated with SME**

Section 3.7 mentions the compressive strength of cubes testes in durability test against acid attack. Besides, concrete cubes were tested in compression at zero days of acid attack after a curing time of 90 days. These results can be used to quantify the effect of SME intermixing on compressive strength of concrete.

Table 4.1 and Table 4.2 show the compressive strength of concrete cubes with limestone and granite aggregates, respectively. Similar to 4x8 inches cylinder, concrete samples with admixed SME, LSI2.5 and LSI5.0 had reductions of 10% and 46%, respectively. These reductions may be caused by SME during the mixing process where SME may have coated the particles of cement preventing them to hydrate during the curing period. It is also hypothesized that the SME covers the aggregates reducing the bonding between aggregates and cement paste,

reducing the compressive strength. This also may affect the shear strength of concrete. Other factors that may affect the results of the tests are the size and shape of the specimens, or the ratio of maximum size of aggregates and the width of the specimens.

**Table 4.1** Compressive strength test in concrete with limestone aggregate cubes

Sample	Compressive Strength (psi)	Compressive Strength (MPa)	Reduction (%)
LO	5440	38.1	-
LSI2.5	4893	34.3	-10.0
LSI5.0	2912	20.4	-46.5
LST4	4672	32.7	-14.1

**Table 4.2** Compression test in concrete with granite aggregate cubes

Sample	Compressive Strength (psi)	Compressive Strength (MPa)	Reduction (%)
GO	6444	45.1	-
GST4	5186	36.3	-19.5

Samples treated with four layers of SME used as sealant (LST4 and GST4) also present a reduction in their compressive strength. LST4 had a reduction of 14%, and GST4 had a reduction of almost 20% when compared with control samples. It should be mentioned that control samples and samples treated with 4 layers of SME are from the same concrete batch. This means that concrete treated with excessive amount of SME may have reduction in the mechanical properties; however, further studies are needed to confirm this data.

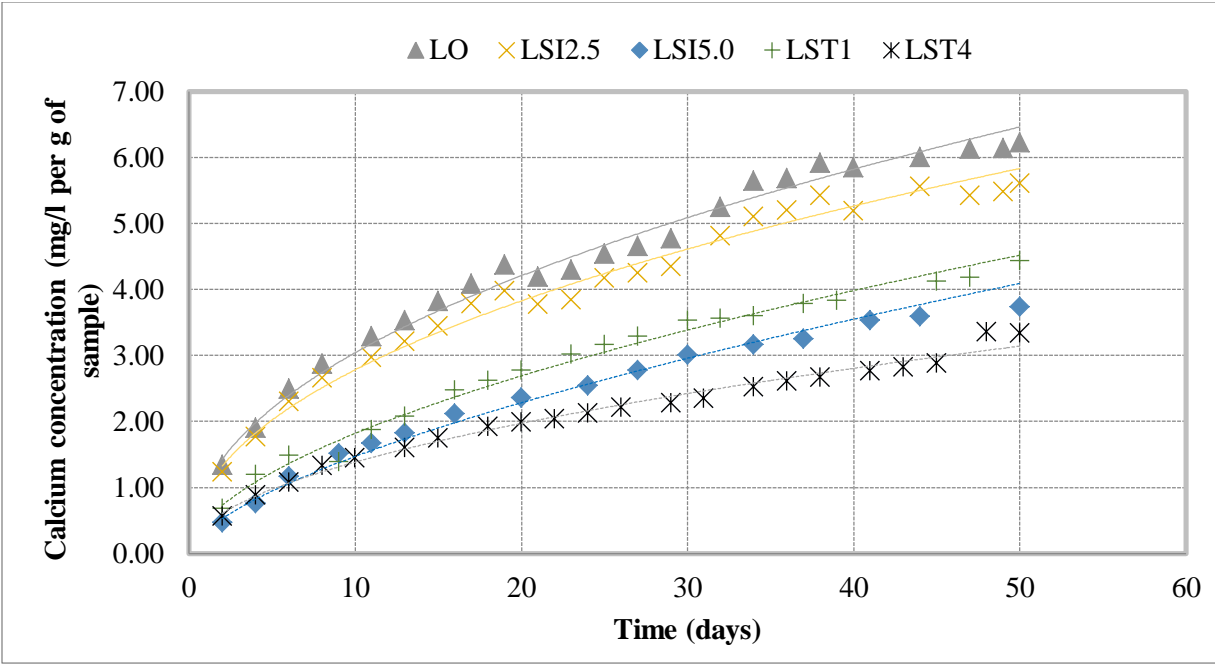
## 4.2 Influence of SME on calcium leaching from concrete

Calcium leaching tests were performed for 50 days. Measurements were performed on three specimens per mixture and the results are reported in Figure 4.2 (concrete with limestone)

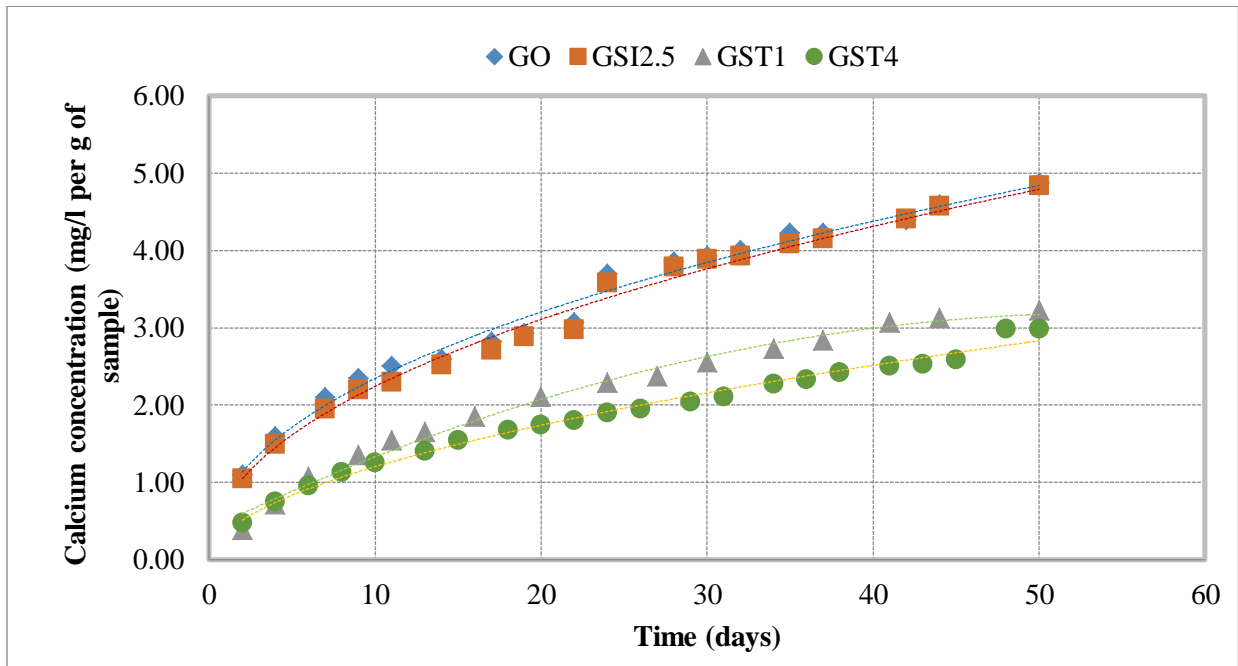
and Figure 4.3 (concrete with granite). The measured calcium concentration is normalized to the sample mass to remove the error that may come from non-identical concrete sample mass between the replicates. A trend line of the leaching behavior is drawn to better visualize the leaching profile of calcium ions when a concrete coupon is submerged into DI water at pH 7.

The maximum calcium leaching was observed in the control sample made with limestone aggregates (identified by LO) which was 6.25 mg/L per g of sample after 50 days, followed by concrete treated with 2.5% of SME intermix, which was 5.61 mg/L per g of sample after 50 days of testing (Figure 4.2). Control concrete with granite aggregates (GO) and concrete with granite containing SME 2.5% intermix (GSI2.5) showed lower calcium leaching than concrete limestone aggregates. GO and GSI2.5 after 50 days of testing (Figure 4.3) showed a calcium concentration of 4.87 mg/L per g of sample and 4.84 mg/L per g of sample, respectively. The high concentration of LO and LSI2.5 may be due to the additional calcium leached from the limestone aggregates.

Comparing LO and LSI2.5 samples, the results show that they follow similar profiles and calcium concentrations after 50 days. As mentioned previously, LO and LSI2.5 had a calcium concentration of 6.23 and 5.61 mg/L per g of sample, respectively, which means a reduction of 9.95% for the LSI2.5 sample. For 5% of admixture SME (LSI5.0), the calcium concentration was 3.74 mg/L per g of sample, which signifies a reduction of 40% when compared with LO. In the case of GO and GSI2.5 at 50 days, GO presents a calcium concentration of 4.87 mg/L per g of sample; meanwhile, GSI2.5 showed 4.84 mg/L per g of sample; resulting in a reduction of almost 1%, which suggests no significant contribution from the addition of 2.5% of SME intermix by mass of cement.



**Figure 4.2** Leaching test results in concrete with limestone aggregates samples



**Figure 4.3** Leaching test results in concrete with granite aggregates samples

Comparing concrete with only limestone (control, LO) and concrete with one layer (LST1) or 4 layers of SME topical treatment (LST4), results show that after 50 days of testing,

the calcium concentration was 4.43 mg/L per g of sample for LST1 and 3.34 mg/L per g of sample for LST4, which means a reduction of 29% for LST1 and 46% for LST4 when compared with LO. In the case of granite control samples (GO), the calcium leaching after 50 days was 4.87 mg/L per g of sample. For the coating treatment with one layer of SME (GST1), the calcium leaching was 3.22 mg/L per g of sample and for 4 layers of SME (GST4) was 2.99 mg/L per g of sample, resulting in a reduction of 34% and 39% respectively. These results suggest that topical treatment reduce the leaching of calcium from concrete materials and produce similar results to the addition of 5% SME intermix. This observation was made because a 5% SME intermix was added to the concrete mix using an emulsification process that helps the SME to distribute uniformly in the concrete, similar to the uniform application of topical SME with the brush.

In general, it was observed that the treatment of SME reduces the calcium hydroxide leaching at a pH = 7. This is due to the ability of SMEs to reduce the water ingress to the pores media of concrete and with less water in contact with the cement paste, less calcium hydroxide will leach into water. Samples with 2.5% of SME by mass of cement have a calcium leaching profile similar to the control samples, and this may be because the SME was added during the concrete mix without any emulsification or plasticizer; emulsification makes the mixture more uniform and therefore may increase the efficiency of SME.

### **4.3 Influence of SME against acid attack**

This section presents the results of the specimens exposed to a solution of sulfuric acid at a pH of 1; the method is described in section 3.7. Table 4.3 and Figure 4.4 show the percent reduction in the compressive strength of LO, LSI2.5, LSI5.0, and LST4 (concrete with limestone

aggregate and different SME treatments). The samples with SME used as an admixture (LSI2.5 and LSI5.0) in the concrete initially improve the performance when compared with the control samples (LO). After 14 days, the control samples had a reduction of 9.5%, while LSI2.5 and LSI5.0 reduced by 0.3% and 0.9%, respectively. After 30 days of testing, LO reduced by 25.4%, whereas LSI2.5 had 2.3% and LSI5.0 had 17.2%. On the other hand, samples with 4 layers of SME had the most significant reduction of all samples; LST4 displayed a decrease of 16.5% at 14 days and 39.7% at 30 days, which is greater than the reduction of the control sample. Despite the improved performance of LSI2.5 and LSI5.0 at 14 and 30 days, the reduction in compressive strength after 60 days was similar to control samples LO; meanwhile LST4 maintain a greater reduction than LO.

Table 4.4 and Figure 4.5 display the compressive strength reduction of concrete samples with granite aggregates; control samples (GO) are compared with concrete samples with 4 layers of topical SME. At 14 days, GO samples had a reduction of 12.2%, and the GST4 sample had a more significant decrease of 21.6%. This trend was seen until 60 days, when the reduction was 63% for GST4 samples, whereas GO had a reduction of 49.4%.

Figure 4.6 and Table 4.3 summarize the percentage of weight reduction for concrete with limestone samples. At 14 days, the lowest reduction in weight was for the control sample LO and the 5.0% SME intermix sample (LSI5.0), with both results at 0.4%, followed by LST4 and LSI2.5 with 2.1% and 2.6% weight reduction, respectively. After 30 days of exposure to acid, the reduction in weight of LO samples increased to 1.8%, LSI5.0 was 1.2%, and LST4 was 7.0%. LSI2.5 maintained a weight loss with a reduction of 2.4%. LST4 samples showed the largest reduction in weight, consistent with the highest reduction in compression strength shown

in Figure 4.4. At 60 days, LO presents a reduction of 8.6%; LSI2.5 reduces to 7.4% and LSI5.0 to 5.2% showing fewer reductions than control, and LST4 reduces to 11.7%.

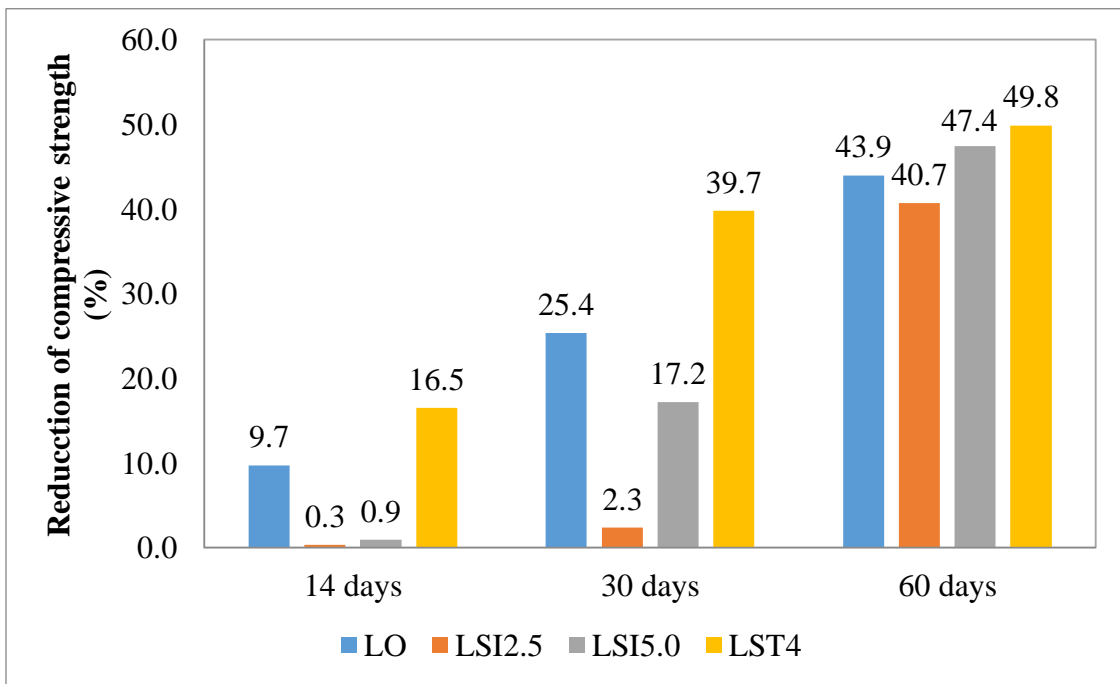
In addition, Figure 4.7 and Table 4.4 present the weight reduction of concrete with granite aggregates. In Figure 4.7, the results show a similar behavior as concrete with limestone aggregates where the control sample GO displays a reduction of 0.8% at 14 days, 3.1% at 30 days, and 10.9% at 60 days. In addition, GST4 samples had a greater reduction of 3.0% at 14 days and 9.5% at 30 days, and 16.7% at 60 days.

**Table 4.3** Durability test in acid media with concrete with limestone aggregates

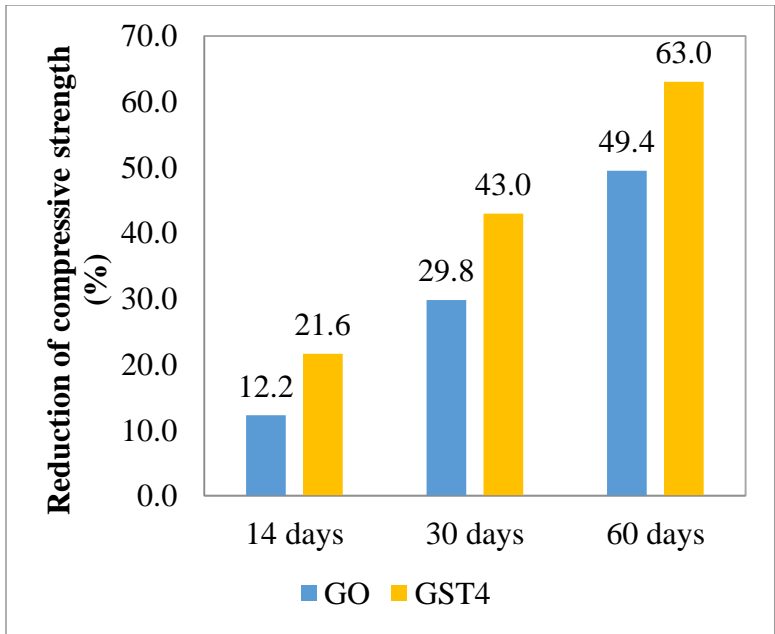
Sample	Age (days)	Change in thickness (%)	Reduction in Weight (%)	Reduction in Compressive strength (%)
LO	14	-1.4	0.4	9.7
	30	-1.5	1.8	25.4
	60	-1.0	8.6	43.9
LSI2.5	14	-0.7	2.6	0.3
	30	-0.9	2.4	2.3
	60	-0.4	7.4	40.7
LSI5.0	14	0.1	0.4	0.9
	30	-0.2	1.2	17.2
	60	-0.4	5.2	47.4
LST4	14	-0.7	2.1	16.5
	30	0.8	7.0	39.7
	60	-1.4	11.7	49.8

**Table 4.4** Durability test in acid media with concrete with granite aggregates

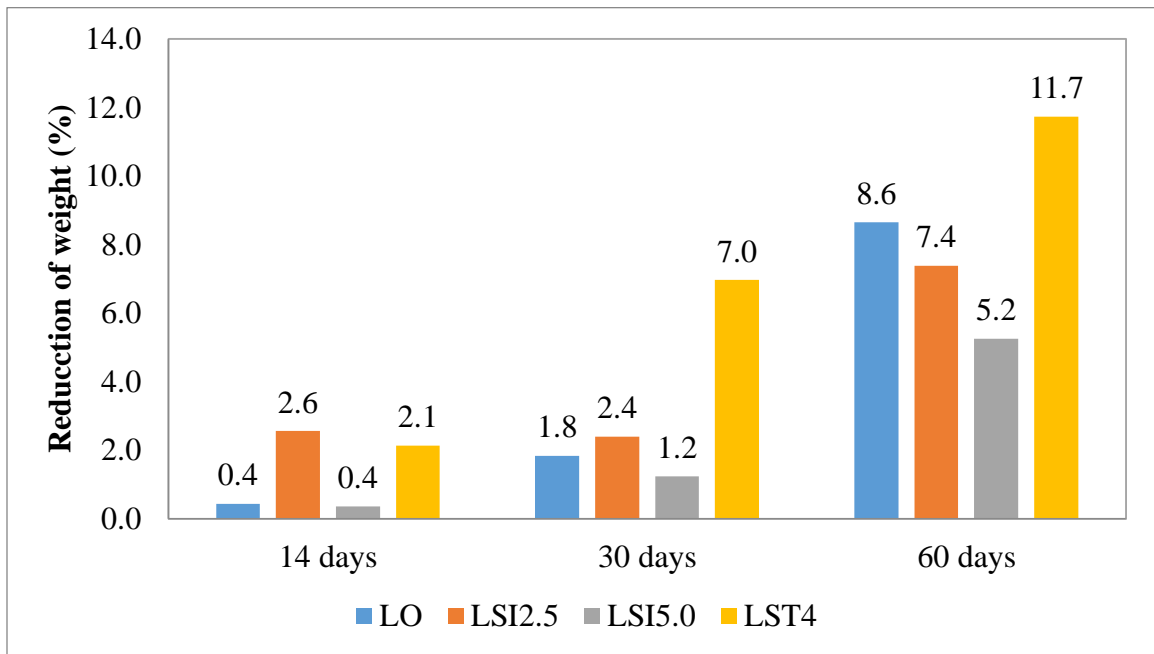
Sample	Age (days)	Change in thickness (%)	Reduction in Weight (%)	Reduction in Compressive strength (%)
GO	14	1.0	0.8	12.2
	30	1.0	3.1	29.8
	60	-1.2	10.9	49.4
GST4	14	-0.1	3.0	21.6
	30	2.3	9.5	43.0
	60	-1.2	16.7	63.0



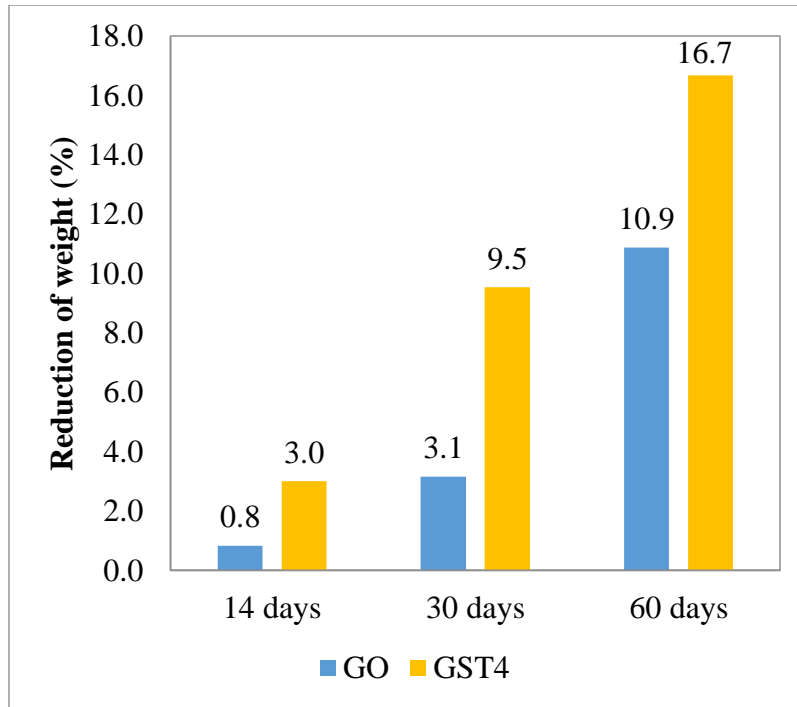
**Figure 4.4** Reduction of compressive strength in percentage of concrete samples with limestone aggregates



**Figure 4.5** Reduction of compressive strength in percentage of concrete samples with granite aggregates



**Figure 4.6** Reduction of weight in percentage of concrete samples with limestone aggregates



**Figure 4.7** Reduction of weight in percentage of concrete samples with granite aggregates

Before performing compressive strength test or weight loss measurements, the dimensions of all samples were measured; however, the result did not show a consistent trend. Consequently, it was concluded that sample dimensions is not a precise parameter to quantify the behavior in acid attack. It is worth mentioning that the acid attacks mainly the cement paste and to a lesser degree the aggregates; hence the majority of the weight loss from the specimens is from loss of the cement paste from the exposed surface of the concrete. As shown in Table 4.3 and Table 4.4, there is a combination of increase and reduction in the percentage of the specimen thickness that could be due to the irregularities in the surface caused by the exposed aggregates. Figure 4.8 and Figure 4.11 display the visual inspection performed after different intervals. In the case of LST4 and GST4 samples, more exposed aggregates are seen on the surface which is a product of the greater mass loss compared to the other samples tested.



**Figure 4.8** Visual inspection of LO (left), LSI2.5 (center), and GO (right) after 14 days of testing



**Figure 4.9** Visual inspection of LST4 (left), LSI5.0 (center), and GST4 (right) after 14 days of testing



**Figure 4.10** Visual inspection of LO (left), LSI2.5 (center), and GO (right) after 30 days of testing



**Figure 4.11** Visual inspection of LST4 (left), LSI5.0 (center), and GST4 (right) after 30 days of testing

Comparing the two types of aggregates used in this research, concrete with limestone aggregates (LO) presents less weight reduction than concrete with granite aggregates (GO). For instance, at 60 days, the reduction of the LO sample was 8.6% and for GO was 10.9%, and this same tendency was presented at 14 and 30 days. This observation was also observed in other research [37], which suggests that limestone aggregates may be used as a sacrificial medium to reduce the acid concentration near the concrete surface and decrease the rate of deterioration of concrete subjected to acid attack when compared concrete with siliceous aggregate and limestone concrete.

#### **4.4 Influence of SME in formation of FOG Deposits**

As mentioned in the Methodology Section 3.8, the FOG deposit formation test was conducted under two different test conditions to identify the effect of calcium availability on FOG deposit formation and adhesion mechanisms for 20 days. When background calcium was used, 75 mg/l of calcium using  $\text{CaCl}_2$ , was added to the synthetic wastewater and FOG deposit formation on concrete samples was quantified.

As an example, Figure 4.12 to Figure 4.16 display the FOG deposit formation and changes in the synthetic wastewater over time for control concrete with limestone aggregate. In

Figure 4.12, the coupons are just added inside the synthetic wastewater solution with all the components mixed as mentioned in Section 3.8. In Figure 4.13 and Figure 4.14, at 3 and 6 days respectively, test results display FOG deposit formation around the concrete coupons at the air water interface along with a change in the color of the water from ongoing saponification. Figure 4.15 and Figure 4.16, at 13 and 20 days respectively, the same amount of FOG deposit has accumulated around the concrete coupon, however the color of the FOG deposit changes from white to light brown. Similar behavior was observed in all the samples, regardless of the SME treatment; and also was reported by other authors in the literature [2] and [12]. The change in colors during the time may be because of the fatty acid oxidative mechanisms that happen in the presence of iron ions or other metals from the cement paste [2]. In the following sections of this chapter, a more detailed description of each tested sample is shown with the quantities related to FOG deposit formed on the surface of the concrete.



**Figure 4.12** FOG deposit formation test of control concrete with limestone aggregates at 0 days



**Figure 4.13** FOG deposit formation test of control concrete with limestone aggregates at 3 days



**Figure 4.14** FOG deposit formation test of control concrete with limestone aggregates at 6 days



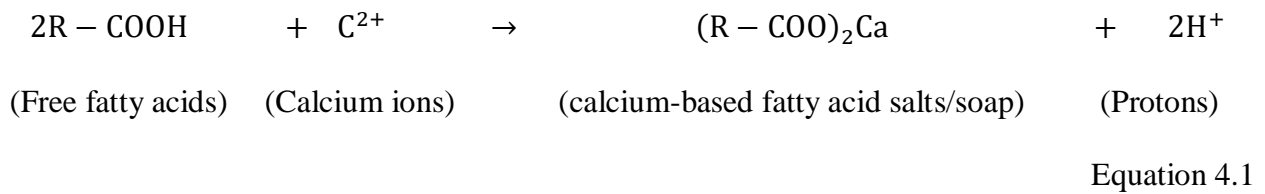
**Figure 4.15** FOG deposit formation test of control concrete with limestone aggregates at 13 days



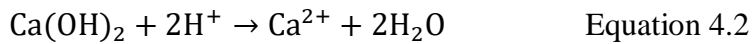
**Figure 4.16** FOG deposit formation test of control concrete with limestone aggregates at 20 days

Another characteristic found during the FOG deposit formation test is the deterioration of the portion of the concrete samples in the wastewater. At the beginning of the test, just after demolding and cutting the samples, the concrete has a regular surface with two faces of mainly

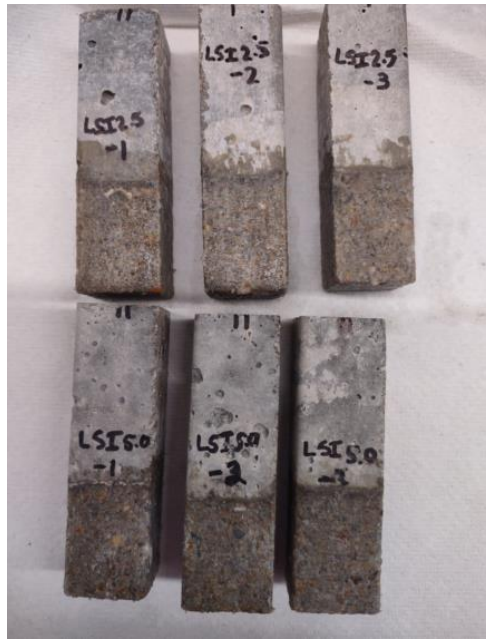
cement paste that was cast against the mold as shown in Figure 4.17; however, after 20 days of testing, a general deterioration was observed around the underwater surface of all the concrete coupons and the air-water interface. As an example, Figure 4.18 displays the deterioration of LSI2.5 and LSI5.0 samples. This corrosion of the cement paste may be due to the acid attack that happens in the concrete and water interface during the saponification reaction. This reaction was described by other research [5], [2] as shown in Equation 4.1.



As mentioned in [38], highly acidic conditions may exist in food and animal-processing industries where free fatty acids may be available in the wastewaters. The hydrogen ion produced during the saponification reaction accelerates the leaching of calcium hydroxide due to a reduction in pH:



**Figure 4.17** Concrete samples before the start of the FOG deposit formation test



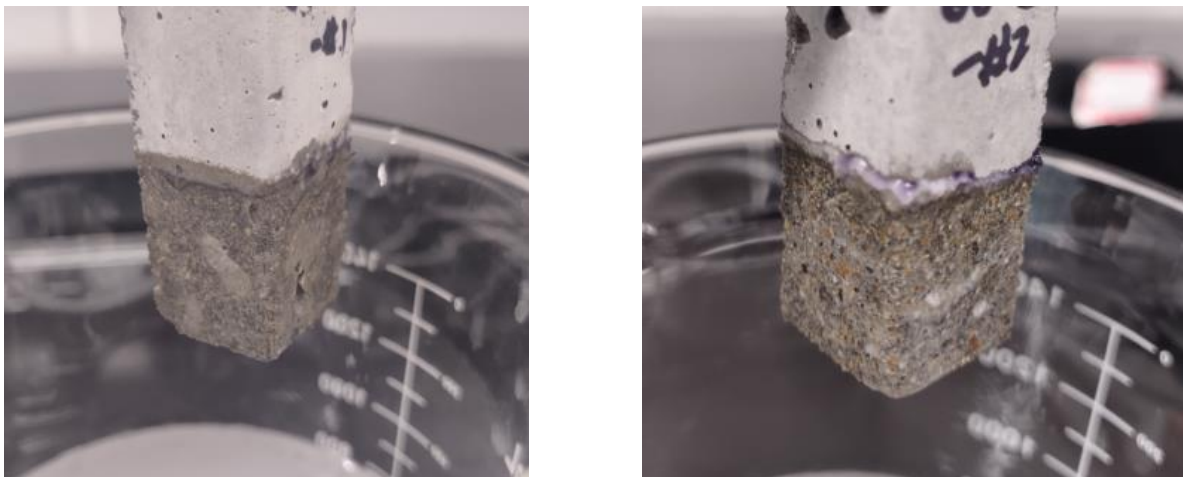
**Figure 4.18** Concrete samples after 20 days of the FOG deposit formation test

This observation is important because the loss of the first layer of cement paste in all the concrete coupons evaluated in this study during the FOG deposit formation may be due to the additional acid attack during the saponification reaction that releases more calcium hydroxide to the wastewater than in comparison with the mechanisms involved with the only leaching of calcium hydroxide at a pH of 7. Figure 4.19 shows a visual comparison between concrete samples after different tests performed in this study. At the right is a specimen after 50 days of leaching test, and is seen that after this period, the cement paste remains on the sample. However, in the case of the acid attack after 14 days of testing and the FOG deposit formation test after 20 days, test results reveal a considerable reduction of the first layer of the cement paste and exposed aggregates.

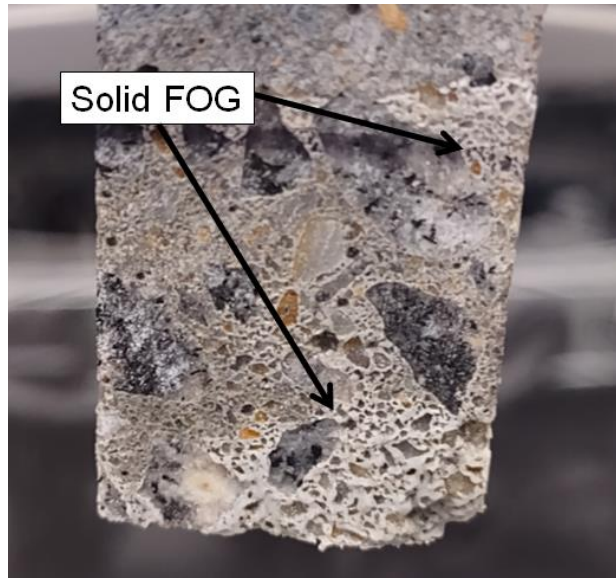


**Figure 4.19** Concrete samples; after 50 days of leaching test (left), after 14 days of acid attack (center), after 20 days of FOG deposit formation test (right)

Additionally, two control GO specimens were tested at 7 hours and 24 hours to check the deterioration generated by the synthetic wastewater during the FOG deposit test. Figure 4.20 shows the GO specimens after 7 hours of FOG deposit testing on the left and after 24 hours of testing on right. After 7 hours of testing, the cement paste is still present in the specimen but a thin layer of FOG deposit starts to form around the cement paste of the concrete specimen as shown in Figure 4.21. However, after 24 hours of testing, the first layer of cement paste starts to deteriorate and particles of aggregate are visible, similar to samples tested until 20 days. Further studies are needed to find the mechanism of deterioration over time of concrete samples during the FOG deposit formation.



**Figure 4.20** Concrete samples; FOG test after 7 hours (left), FOG test after 24 hours (right)



**Figure 4.21** Thin layer of FOG deposit starts to form around cement paste after 7 hours of testing

#### **4.4.1 Concrete with Limestone Aggregate under Calcium Background condition**

Table 4.5 and Figure 4.22 summarize the results of the FOG deposit formation test in concrete with limestone aggregates in the calcium background condition using a concentration of 75 mg/l of calcium. At first, comparing the control samples (LO) with the adjustment of pH, the average FOG deposit formed around the surface of the concrete is  $1.43 \text{ g/cm}^2$  and, with no adjustment of the pH, the average is  $1.59 \text{ g/cm}^2$  which are slightly greater. Even though the FOG deposit form in the solution with no adjustment is greater, considering the standard deviation as shown in Table 4.5, both samples show similar results. Regarding the pH level in the synthetic wastewater solution, in samples with calcium background and no pH adjustment, pH values were between  $\text{pH} = 5-6$ ; this is slightly less when compare with samples in background calcium and  $\text{pH} = 7$ . This suggests the presence of background calcium and maintaining the pH at 7 with

ammonium hydroxide did not have a significant impact on FOG deposit formation when the results are compared with FOG deposit formation on samples with no adjustment of pH. In addition, this also suggests that the high pH generated by the calcium hydroxide from the cement paste is sufficient to form FOG deposits, and the effect of additional alkaline conditions is minimal.

Comparing the control samples with the SME treated samples when the pH =7, the control sample LO produces a FOG deposit of 1.43g/cm<sup>2</sup>, LSI2.5 produces 1.40 g/cm<sup>2</sup>, and LST1 produces 1.54 g/cm<sup>2</sup>. Taking into account the standard deviation, LO has 0.14 g/cm<sup>2</sup>, LSI2.5 0.20 g/in<sup>2</sup>, and LST1 0.11 g/in<sup>2</sup> which suggests that these three samples with background calcium and pH adjustment to 7 had similar behavior.

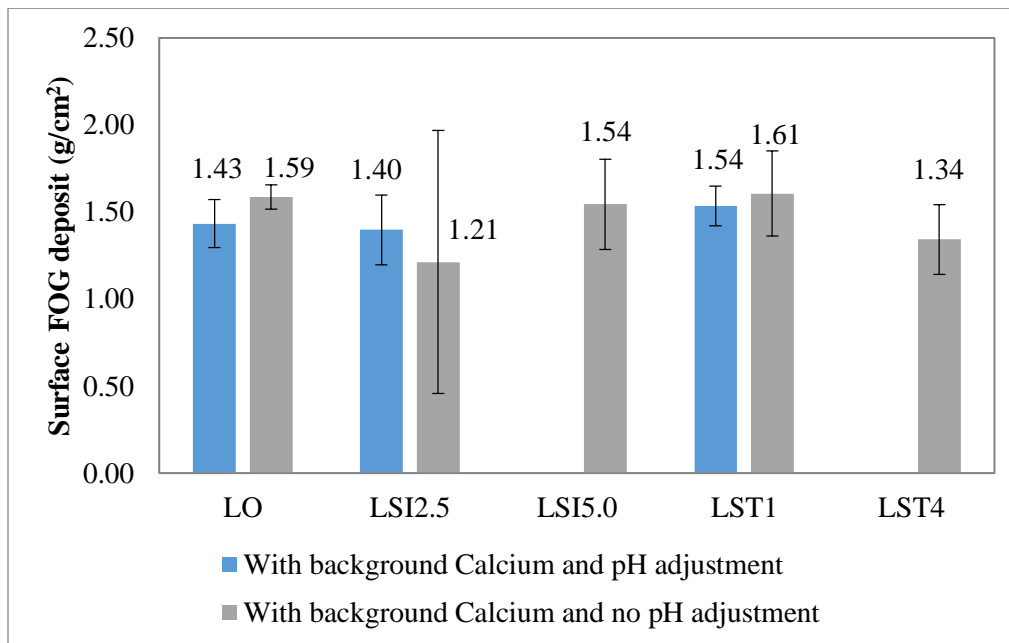
For samples with no adjustment of pH (pH = 5-6), the control sample LO produced an average 1.59 g/cm<sup>2</sup> of FOG deposit, while LST1 and LST4 produce 1.61 g/cm<sup>2</sup> and 1.34 g/cm<sup>2</sup>, of FOG deposit respectively. The samples with SME as admixture (LSI2.5 and LSI5.0) also had similar behavior as control samples.

For samples with calcium background condition and in a range of pH = 5-7, FOG deposit formation test produce similar results, regardless the SME treatment. The results suggest that the treatment with SME did not reduce the formation of FOG deposit around the surface of the samples because of the high standard deviation that makes them in the same range as control samples, as shown in Figure 4.22.

**Table 4.5** Amount of Surface FOG deposit formed in concrete with limestone in ( $\text{g}/\text{cm}^2$ )

Concrete Sample	with $\text{Ca}^{2+}$ , Adjust pH	with $\text{Ca}^{2+}$ , No Adjust pH	Without $\text{Ca}^{2+}$ , Adjust pH
Limestone Only (LO)	$1.43 \pm 0.14$	$1.59 \pm 0.07$	$1.09 \pm 0.12$
Limestone SME Intermix 2.5% (LSI2.5)	$1.4 \pm 0.2$	$1.21 \pm 0.76$	$1.14 \pm 0.12$
Limestone SME Intermix 5.0% (LSI5.0)	NT	$1.54 \pm 0.26$	NT
Limestone SME Topic 1L (LST1)	$1.54 \pm 0.11$	$1.61 \pm 0.24$	$1.43 \pm 0.2$
Limestone SME Topic 4L (LST4)	NT	$1.34 \pm 0.2$	NT

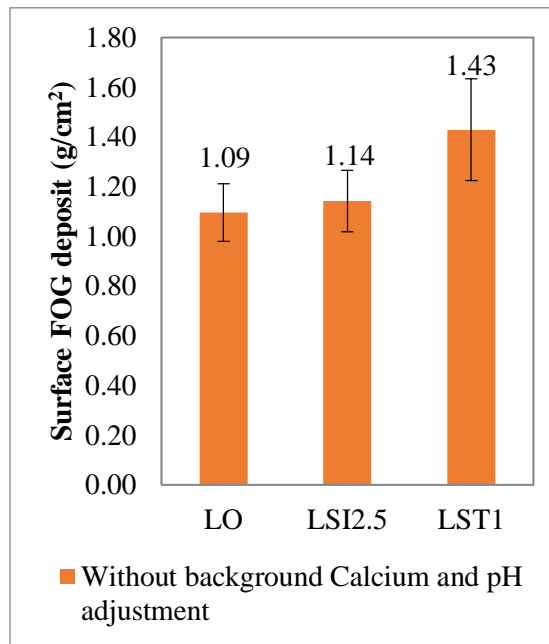
NT: Not tested



**Figure 4.22** Surface FOG deposit form around concrete with limestone aggregate in background calcium media

#### 4.4.2 Concrete with Limestone Aggregate without Calcium Background condition

Figure 4.23 displays the results for samples in a solution without background calcium; a reduction in the amount FOG deposit formation was observed as compared to the samples with background calcium. For instance, the control samples with limestone aggregates (LO) showed a FOG deposit quantity of 1.43 g/cm<sup>2</sup> in the presence of background calcium, while LO without calcium background reduced to 1.09 g/cm<sup>2</sup> in the absence of external calcium. The FOG deposit formation was for the control sample with 1.09 g/cm<sup>2</sup>; LSI2.5 was 1.14 g/cm<sup>2</sup> and LST1 was 1.43 g/cm<sup>2</sup>. These results indicate that, similar to the samples with background calcium solution, SME treatment does not reduce the FOG deposit formation.



**Figure 4.23** Surface FOG deposit form around concrete with limestone aggregate without calcium background media

#### 4.4.3 Concrete with Granite Aggregate under Calcium Background condition

Table 4.6 and Figure 4.24 summarize the results of the FOG deposit formation test on concrete with granite aggregates. At first, the control samples (GO) with calcium background condition and with the adjustment of pH to 7, the average FOG deposit weight formed around the surface of the concrete is  $1.71 \text{ g/cm}^2$  and, with no adjustment of pH (pH = 5-6), the average value is  $1.57 \text{ g/cm}^2$ . This indicates that maintaining the pH 5 to 7 will generate similar mass of FOG deposits around the concrete surface.

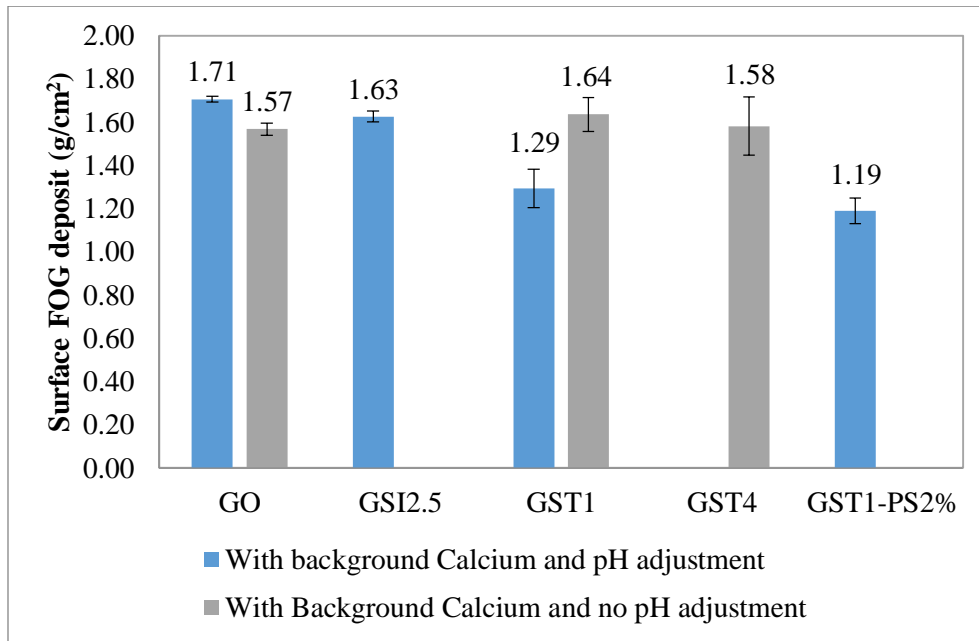
Comparing the control sample (GO) with the SME treated samples, in the presence of calcium background and with pH adjusted to 7, GO produced  $1.71 \text{ g/cm}^2$ , GSI2.5 produced  $1.63 \text{ g/cm}^2$ , and GST1 produced  $1.29 \text{ g/cm}^2$  surface FOG deposit. In this project, one sample was treated with one layer of SME with an addition of 2% by mass of SME of polystyrene (GST1-2%PS) and tested in a solution with background calcium and pH adjusted to 7 generating an average mass of  $1.19 \text{ g/cm}^2$  of FOG deposits, which is a reduction of 30.2% as compared with the control sample. Despite the reduction presented with GST1-2%PS, further studies are needed to understand this treatment in wastewater conditions, leaching test, and acid attack test.

The GO, GST1, and GST4 were tested in the calcium background solution without pH adjustment (pH =5-6). For this case, the control sample GO generated a surface FOG deposit amount of  $1.57 \text{ g/cm}^2$ , GST1 generated  $1.64 \text{ g/cm}^2$ , and GST4 generated  $1.58 \text{ g/cm}^2$  of surface FOG deposit.

**Table 4.6** Amount of Surface FOG deposit formed in concrete with granite in (g/cm<sup>2</sup>)

Concrete Samples	with Ca <sup>2+</sup> , Adjust pH	with Ca <sup>2+</sup> , No Adjust pH	Without Ca <sup>2+</sup> , No Adjust pH
Granite Only (GO)	1.71 ± 0.01	1.57 ± 0.03	1.15 ± 0.29
Granite SME Intermix 2.5% (GSI2.5)	1.63 ± 0.03	NT	NT
Granite SME Topic 1L (GST1)	1.29 ± 0.09	1.64 ± 0.08	1.25 ± 0.17
Granite SME Topic 4L (GST4)	NT	1.58 ± 0.13	NT
Granite SME Topic 1L - PS 2% (GST1- 2%PS)	1.19 ± 0.06	NT	NT

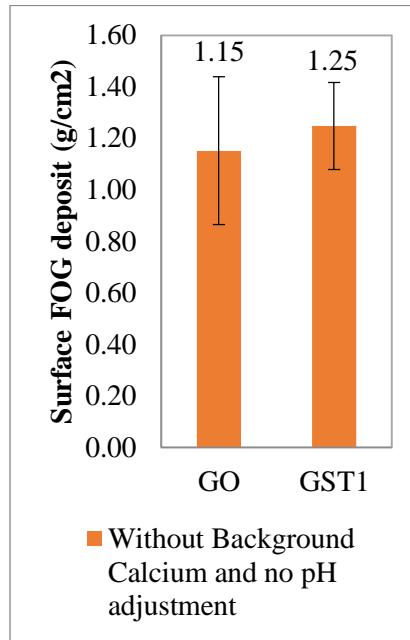
NT: Not tested



**Figure 4.24** Surface FOG deposit form around concrete with granite aggregate in background calcium media

#### 4.4.4 Concrete with Granite Aggregate without Calcium Background condition

Additionally, GO and GST1 samples were tested in solution without calcium background and no pH adjustment. For the control sample, the amount of FOG deposits generated was 1.15 g/cm<sup>2</sup> and for GST1 was 1.25 g/cm<sup>2</sup>, as shown in Figure 4.25.



**Figure 4.25** Surface FOG deposit form around concrete with granite aggregate without calcium background media

#### 4.4.5 FTIR analysis of FOG deposit samples

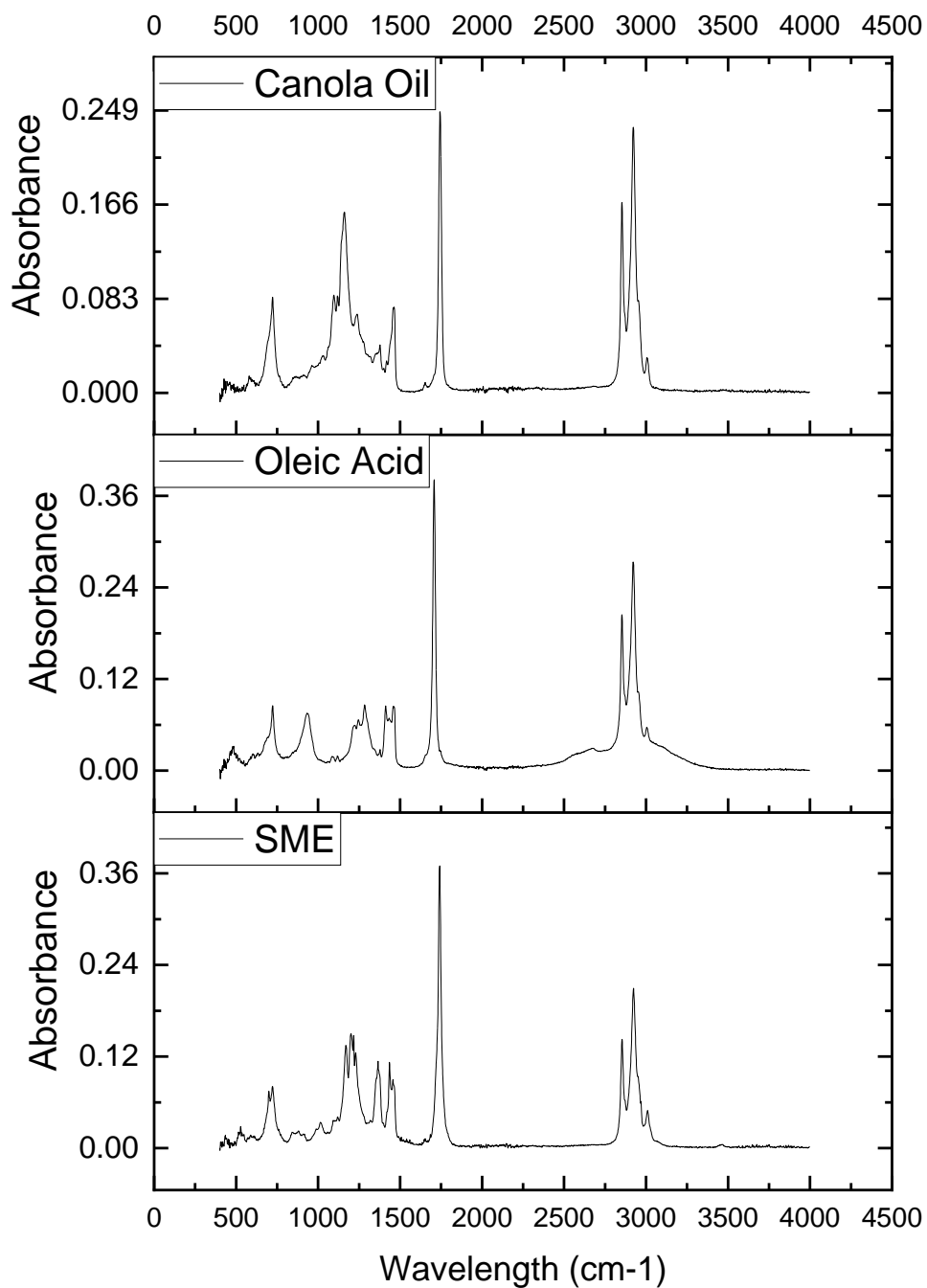
Figure 4.26 shows the typical profile spectrum for canola oil and oleic acid used as free fatty acids in synthetic wastewater. A stretching vibration at 1745 cm<sup>-1</sup> is observed which is attributed to the frequency of ester bond found in oil; SME also exhibits a similar peak at this wavelength. Figure 4.27 and Figure 4.29 shows the FTIR profile of the outer layer FOG deposit using concrete with granite and limestone aggregate, respectively, in a solution with background

calcium. In this case, the farthest part from the concrete surface shows a low absorption for the characteristic band in 1541 and 1577  $\text{cm}^{-1}$  indicating a low saponification reaction. In contrast, the surface layer profile, as shown in Figure 4.28 and Figure 4.30 for granite and limestone aggregate, respectively, shows an increase in the bands that characterizes the calcium saponification reaction in 1541 and 1577  $\text{cm}^{-1}$  of wavelength which comes with a reduction of the band peaks related with oleic acid and canola oil.

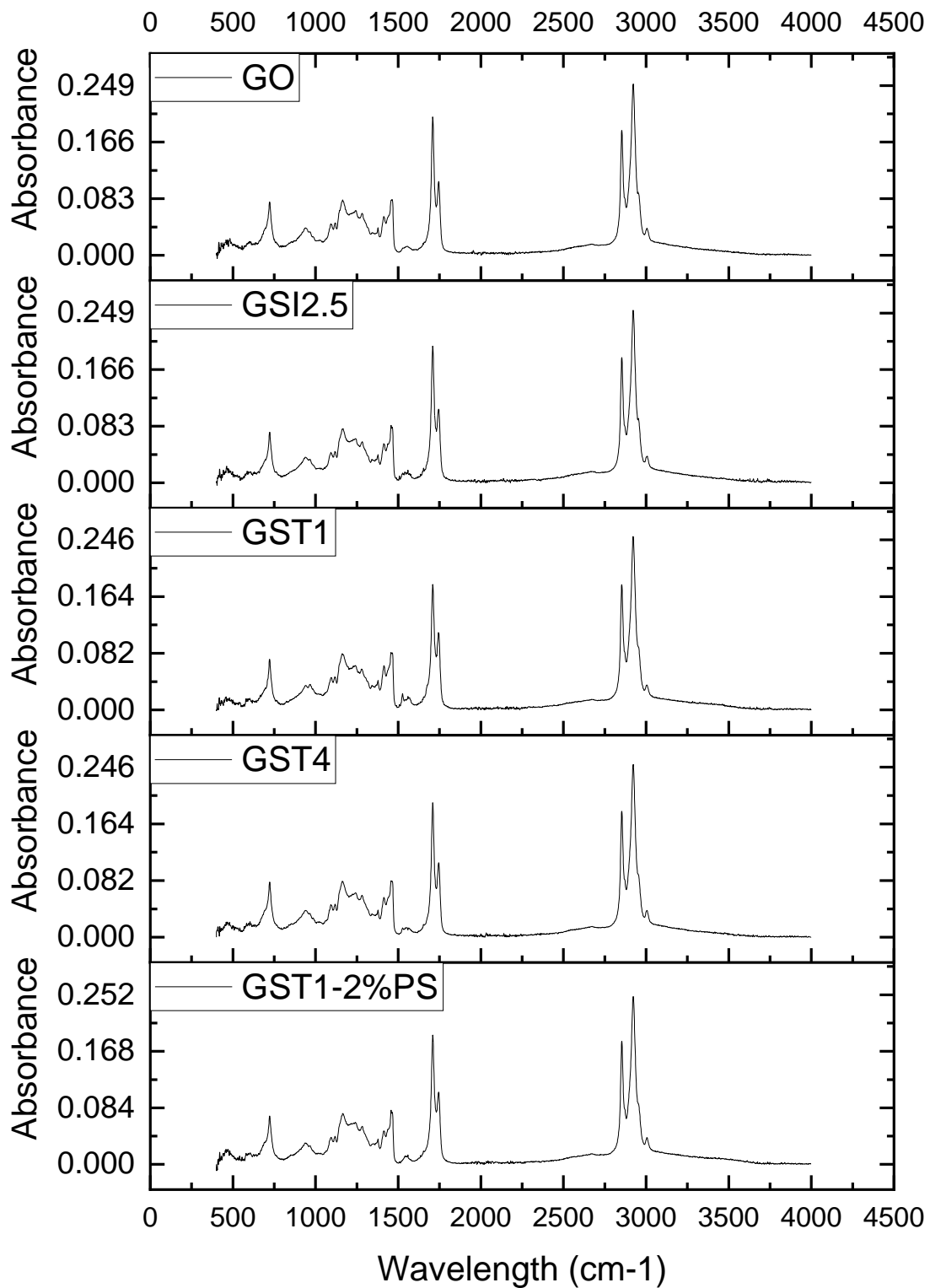
Similar FTIR profiles were observed for FOG deposits collected from sewer systems or produced in labs [39] and [1]. When analyzing Figure 4.27- Figure 4.30, the FTIR profile describes the calcium soap band at 1468  $\text{cm}^{-1}$ , 1541  $\text{cm}^{-1}$ , 1577  $\text{cm}^{-1}$ , wide profile at 3400  $\text{cm}^{-1}$ , and the disappearance of 1740  $\text{cm}^{-1}$  and 1707  $\text{cm}^{-1}$  due to the consumption of FFA and oil during saponification reaction. Besides these characteristics of calcium soap bands, high absorbance values are observed close to 3000–2800  $\text{cm}^{-1}$  containing four peaks, located at 3004  $\text{cm}^{-1}$  (=C–H stretching vibration), 2955  $\text{cm}^{-1}$  (-C-H ( $\text{CH}_3$ ) asymmetric stretching vibration), 2922  $\text{cm}^{-1}$  (-C-H ( $\text{CH}_2$ ) asymmetric stretching vibration), and 2851  $\text{cm}^{-1}$  (-C-H ( $\text{CH}_2$ ) symmetric stretching vibration) as mentioned in [36].

However, the intensity of this absorbance varied based on the availability of background calcium. For the FOG deposit formed on samples without background calcium, Ca-O band absorption was lower than that formed on samples with calcium background solution. The difference between the FOG deposit layers in this study may be due to the difference in pH, calcium gradient between the concrete surface and wastewater, and availability of fatty acids. Iasmin [5] reported that at a very high pH (pH = 14), solid soap was formed; meanwhile, at lower pH (pH = 10), semi-solid soap was formed. Due to the leaching of CH from concrete, a high pH and calcium availability at the concrete surface are expected [40-41]. Along with the high

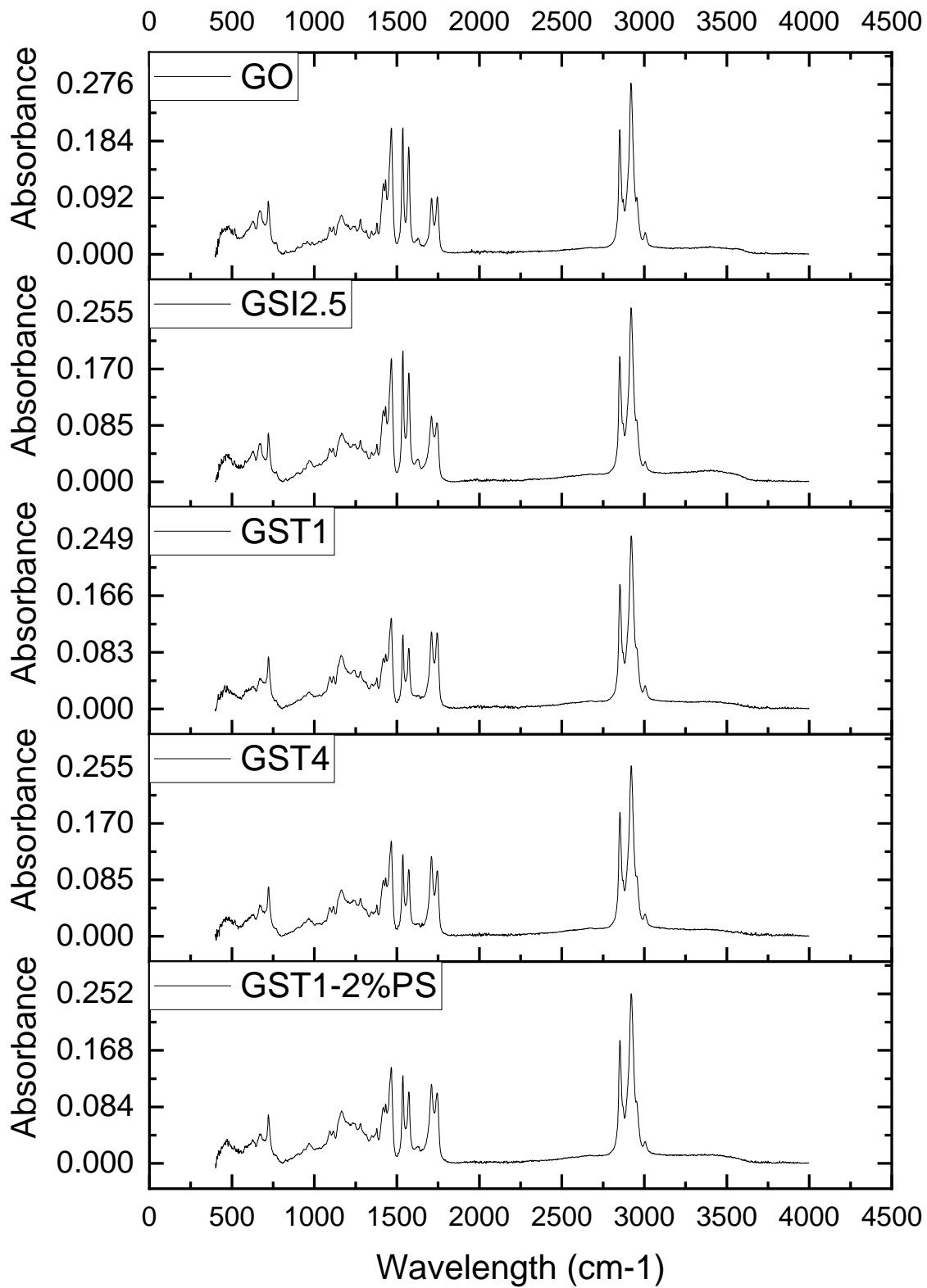
concentration of available fatty acids near the air-wastewater interface, the saponification reaction proceeds at a higher rate at this location and results in the formation of white solid soap particles.



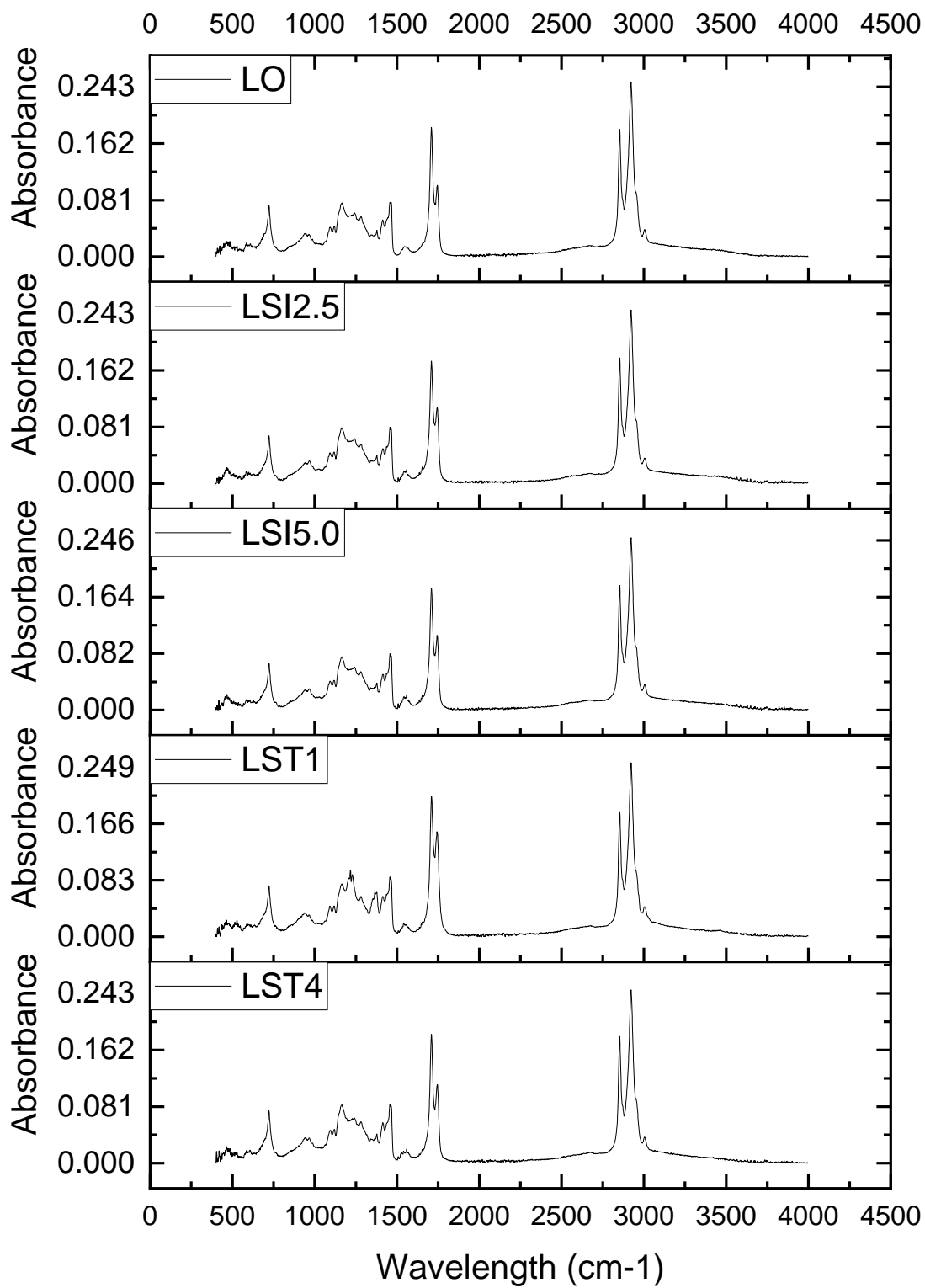
**Figure 4.26** FTIR spectra for Canola oil, oleic acid, and SME



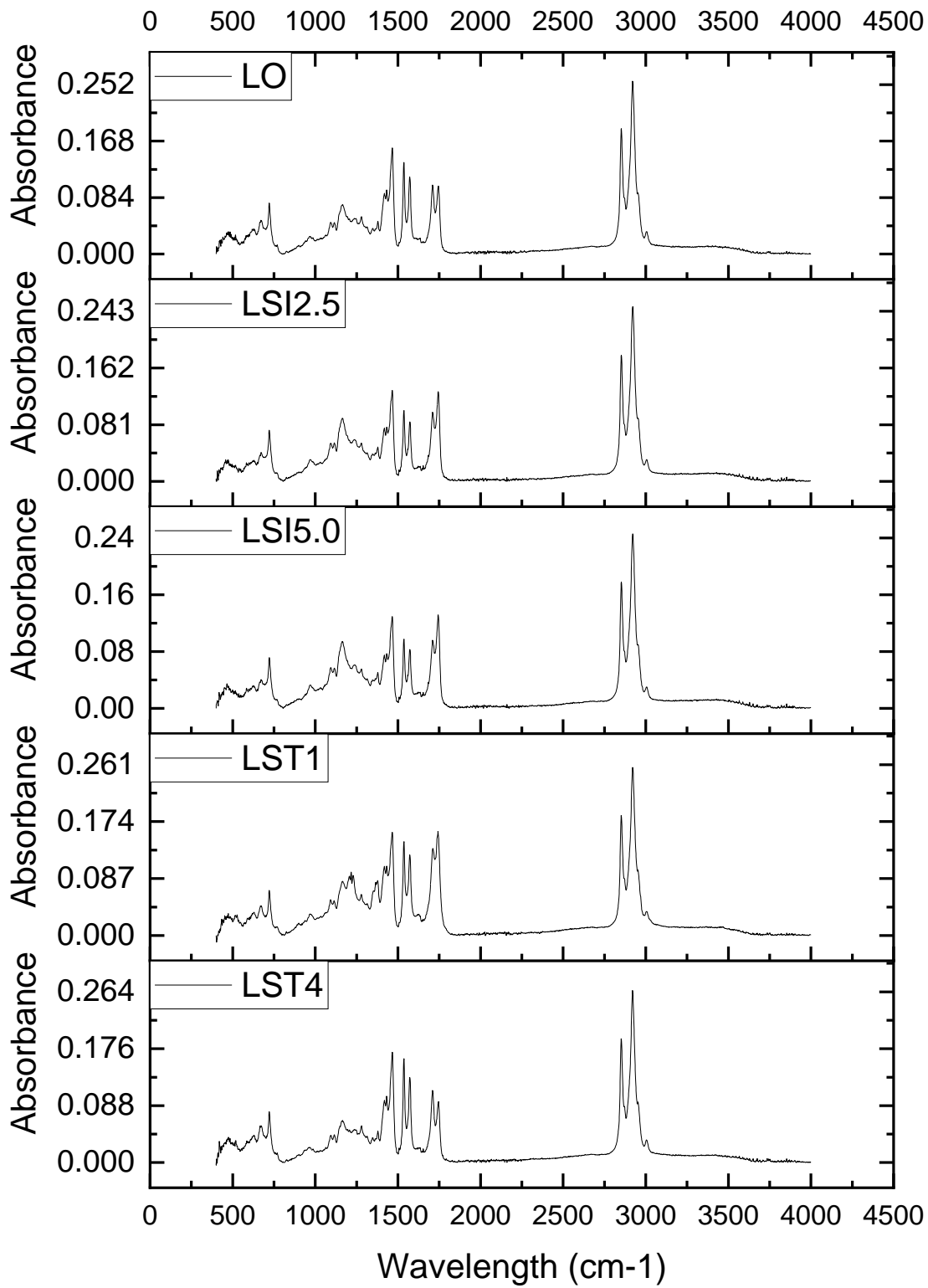
**Figure 4.27** FTIR spectra of outer layer FOG deposits with calcium background for concrete with granite aggregates



**Figure 4.28** FTIR spectra of surface layer FOG deposits with calcium background for concrete with granite aggregates



**Figure 4.29** FTIR spectra of outer layer FOG deposits with calcium background for concrete with limestone aggregates



**Figure 4.30** FTIR spectra of surface layer FOG deposits with calcium background for concrete with limestone aggregates

As mentioned in Section 3.8, the FTIR analysis was made in the surface layer and the outer layer of FOG depositions, as shown in Figure 3.11. Table 4.7, Table 4.8, and Table 4.9 show the soap absorbance for the surface layer, outer layer, and FOG deposit after oven drying, respectively, for concrete samples with limestone aggregates. Table 4.10, Table 4.11, and Table 4.12 show the soap absorbance for concrete samples with granite aggregates. These tables display the soap content (%) determined from the FTIR spectrum using Equation 3.3. Overall, the soap absorbance of the FOG deposit maintains similar percentage in all the samples, regardless of the coarse aggregate used or the SME treatment. And this soap absorbance percentage decreases between the surface layer and the outer layer. The soap content also increased with the presence of background calcium. Additionally, FOG deposits with higher soap absorbance are comparatively rigid and adhere firmly to the concrete surface.

From FTIR spectra, the FOG deposit formed on concrete surfaces is, as expected, calcium soap of long chain free fatty acids. However, the different calcium hydroxide dissolution rates from concrete materials resulted in a high to low pH and calcium ion gradient from the concrete surface toward the wastewater solution. As a result, granular and hard calcium soap formed on the surface layer during the saponification reaction at a higher pH and calcium availability. Meanwhile, the soft soap created on the outer layer shows high absorbance at  $1740\text{ cm}^{-1}$  and  $1707\text{ cm}^{-1}$  indicates the accumulation of partially saponified or unreacted FOG at a lower pH and calcium availability.

**Table 4.7** Soap absorbance in surface layer FOG deposit in concrete with limestone

Concrete Sample ID	Soap in Surface Layer (%)		
	With background calcium and pH adjustment	With background calcium and no pH adjustment	Without background calcium and pH adjustment
LO	39.73 ± 5.47	43.39 ± 14.69	37.49 ± 10.6
LSI2.5	40.09 ± 10.08	40.05 ± 13.43	29.99 ± 8.31
LSI5.0	NT	38.03 ± 11.87	NT
LST1	36.75 ± 6.2	44.28 ± 18.77	22.68 ± 5.7
LST4	NT	41.01 ± 18.3	NT

NT: Not tested

**Table 4.8** Soap absorbance in outer layer FOG deposit in concrete with limestone

Concrete Sample ID	Soap in Outer Layer (%)		
	With background calcium and pH adjustment	With background calcium and no pH adjustment	Without background calcium and pH adjustment
LO	10.36 ± 2.58	7.49 ± 0.96	7.5 ± 1.72
LSI2.5	9.67 ± 1.03	9.65 ± 7.13	7.02 ± 1.76
LSI5.0	NT	6.57 ± 0.70	NT
LST1	6.73 ± 0.87	7.78 ± 1.54	6.75 ± 1
LST4	NT	8.77 ± 0.91	NT

NT: Not tested

**Table 4.9** Soap absorbance in oven dry FOG deposit in concrete with limestone

Concrete Sample ID	Soap in Oven dry (%)		
	With background calcium and pH adjustment	With background calcium and no pH adjustment	Without background calcium and pH adjustment
LO	22.54 ± 4.71	16.36 ± 0.43	17.41 ± 0.82
LSI2.5	21.33 ± 2.26	19.13 ± 7.32	15.41 ± 1.08
LSI5.0	NT	12.93 ± 1.50	NT
LST1	11.9 ± 0.24	12.07 ± 1.42	11.86 ± 0.88
LST4	NT	15.15 ± 0.50	NT

NT: Not tested

**Table 4.10** Soap absorbance in surface layer FOG deposit in concrete with granite

Concrete Sample ID	Soap in Surface Layer (%)		
	With background calcium and pH adjustment	With background calcium and no pH adjustment	Without background calcium and no pH adjustment
GO	38.7 ± 15.21	44.48 ± 14.32	38.05 ± 9.95
GSI2.5	37.84 ± 10.43	NT	NT
GST1	39.05 ± 8.58	45.06 ± 13.40	38.72 ± 10.99
GST4	NT	40.98 ± 10.99	NT
GST1-2%PS	43.28 ± 11.69	NT	NT

NT: Not tested

**Table 4.11** Soap absorbance in outer layer FOG deposit in concrete with granite

Concrete Sample ID	Soap in Outer Layer (%)		
	With background calcium and pH adjustment	With background calcium and no pH adjustment	Without background calcium and no pH adjustment
GO	8.3 ± 1.46	8.85 ± 1.42	4.88 ± 2.03
GSI2.5	9.44 ± 2.46	NT	NT
GST1	8.92 ± 8.58	9.02 ± 1.58	6.51 ± 0.51
GST4	NT	6.67 ± 0.95	NT
GST1-2%PS	6.71 ± 1	NT	NT

NT: Not tested

**Table 4.12** Soap absorbance in oven dry FOG deposit in concrete with granite

Concrete Sample ID	Soap in Oven dry (%)		
	With background calcium and pH adjustment	With background calcium and no pH adjustment	Without background calcium and no pH adjustment
GO	19.38 ± 4.31	13.85 ± 1.00	9.04 ± 3.88
GSI2.5	17.46 ± 3.59	NT	NT
GST1	19.79 ± 2.23	12.76 ± 1.68	15.17 ± 0.89
GST4	NT	11.78 ± 0.57	NT
GST1-2%PS	20.8 ± 1.4	NT	NT

NT: Not tested

## 4.5 Discussion of results

Analyzing the results of compressive strength test of concrete treated with SME, it is observed that the addition of SME as an admixture reduces the compressive strength of concrete. Also, concrete cubes treated with 4 layers of SME, showed a reduction in compressive strength. This means that SME may reduce the bonding between aggregates and cement paste, or in the case of admixture SME, reduce the hydration of cement particles in the early stages. Further studies are needed to determine why the mechanical properties of concrete are affected in the presence of SME.

In the case of leaching test, SME treatment reduces the leaching of calcium at a pH = 7 because the SME reduces the ingress of water to the pores media, which also reduce leaching of CH out of the sample. However, samples treated with SME during the acid attack test present deterioration of the superficial cement paste of each specimen, similar to the deterioration of samples during the FOG deposit formation test. This suggests that FOG deposit formation on concrete is a combination of calcium leaching accelerated by the acid attack on the cement paste, and further tests are needed to assess the behavior of SME in acid media whether the compound is used as admixture or coating treatment of concrete. Also, the results suggest that the pH of the bulk solution of wastewater may be lower in zones closer to the concrete surface. This observation creates the need for better understand and quantify the actual pH in the interface between the concrete surface and wastewater where the calcium hydroxide, the free fatty acids, and the saponification reaction are involved. Also further studies are needed to address the calcium hydroxide leaching of concrete treatments with SME at different pH conditions and whether SME reacts inside the pores media of cement paste because of the alkaline nature of this material.

Regarding the FOG deposit formation test, variability and scatter data in samples treated with SME show that SME treatment is not suitable to reduce the FOG deposit formation on the concrete surface. As expected, samples tested in calcium background media formed more FOG deposits around the concrete coupons than samples without calcium background, but similar to previous research [2], it was observed that calcium leaching from concrete itself is enough to form FOG deposits because of the calcium hydroxide present in the concrete. However, more studies are needed with concrete treated with SME with the addition of polystyrene (SME-PS); this addition enhances the attachment properties of SME to the concrete surface and also may increase the resistance of concrete to acid attack. It was observed that the FOG deposit formation test is affected by variables such as the calcium concentration in the bulk solution, duration of the test, and alkaline components used to stabilize the pH; these, variables that need to be studied to better understand the FOG deposit formation.

# Chapter 5.

## Conclusions

### 5.1 Summary

The formation of fat, oil, and grease (FOG) deposits in sewer systems is a major challenge for owners and operators of infrastructure because of the contamination as a consequence of the sanitary sewer overflows. For this reason, new alternative materials or treatments must be developed to enhance the system's resilience against FOG deposit formation. In the case of this study, the plan was to evaluate the possibility of using SME, a concrete sealant product from soybean oil, to reduce the FOG deposit formation in concrete pipes and sewer systems. This thesis also evaluated the effect of SME treatment on leaching of calcium from concrete on the resistance of concrete against acid attack.

### 5.2 Conclusion from the experimental results

1. Results from the leaching tests, shown in Figure 4.2, indicate that the SME reduces the leaching of calcium in water with a pH of 7. The exception was the sample with 2.5% SME (by mass of cement) used as admixture used which showed the same calcium leaching rate as the control samples. On the other hand, samples with four layer of topical treatment of SME (LST4 and GST4) showed a reduction of approximately 40% when compared with control samples. Samples with one layer of SME and 5.0% of admixed SME also show a decrease of calcium leaching compared

with control samples with a reduction of 29% for LST1, 34% for GST1 and 40% for LSI5.0.

2. The results of sulfuric acid attack on concrete samples with SME treatment indicate that samples with SME as admixture (LSI2.5 and LSI5.0) showed a good performance in terms of compressive strength and weight loss up to 30 days of exposure; these samples showed less reduction in strength and weight in comparison with control samples as shown in Table 4.3 and Table 4.4. However, after 60 days of exposure, the compressive strength of LSI2.5 and LSI5.0 decreased to similar percentage as control samples. Weight reduction of LSI2.5 and LSI5.0 samples was less than control samples after 60 days. Samples treated with four layers of topical SME (LST4 and GST4) showed the lower performance, with reductions more significant than the control samples (LO and GO). LST4 and GST4 had reductions in compressive strength and weight loss values at 30 days, similar to the reductions of control samples at 60 days. In addition, concrete with limestone aggregates has less weight reduction than concrete with granite aggregate.
3. SME as an admixture can reduce the compressive strength of concrete and the reduction in strength is more significant when 5% SME by mass of cement is used and when the surface is completely saturate with SME, as shown in Section 4.1. Further studies are needed to determine the mechanisms of SME that reduce the mechanical properties of the concrete.
4. The FOG deposit formation test results showed no apparent differences between the control samples and those treated with SME (topically or admixed) due to the high variability between of test results, as seen in the standard deviation in the results of

Section 4.4. The FOG test also evaluates the difference between the adjustment of pH at 7 and samples without any pH adjustment (pH = 5-6) in a solution of DI water, 30 g of oleic acid, 20 g of canola oil, and 75 mg/l of  $\text{Ca}^{2+}$ , and no difference where found. This suggests a lower pH and complex mechanisms in the interface between the solution of synthetic wastewater and the cement paste; therefore further studies are needed in this area to better understand the mechanisms involved. FOG deposit formation is a combination of acid attack and calcium leaching in the concrete surface. Regarding the FTIR analysis, all tests performed in this study showed a profile of calcium soap from the saponification reaction between the free fatty acids and the calcium from the synthetic wastewater and the cement paste.

### **5.3 Future Work**

Based on the objectives of this thesis and the results found during the experimental work, future work needs to focus on understanding the mechanisms that occur at a small scale between the surface of the concrete and the wastewater, such as pore distribution of the cement paste, pH adjacent to the concrete surface and next to the wastewater. This will help modify the type of test that simulates these phenomena in laboratories conditions. Determining the actual pH between the concrete surface and the wastewater interface will help develop a more accurate prediction of the concrete's deterioration during the exposure and to propose durability and leaching tests that more closely simulate field conditions.

This research studied the treatment of concrete samples with plain SME without adding polystyrene. Because of the properties of SME to work as a solvent, polystyrene concentration by weight of SME may be used to improve the coating and attaching properties to concrete surfaces. Additionally, studies related to understand the mechanisms involved during the

hydration of the cement with the addition of the SME, to can explain the reason for the reduction of the compressive strength and other mechanical properties. Also determine if concrete saturated with SME.

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## APPENDICES

## Appendix A

### Leaching Test Supplementary material

Table A. 1 Sample measurements

Sample average properties					
ID	weight (g)	Surface (mm <sup>2</sup> )	Surface (cm <sup>2</sup> )	Volume (cm <sup>3</sup> )	Density (g/cm <sup>3</sup> )
GO	28.7	3220.4	32.2	12.4	2.3
GSI2.5	28.7	3241.6	32.4	12.5	2.3
LO	26.6	3152.9	31.5	12.0	2.2
LSI2.5	29.2	3342.8	33.4	13.1	2.2
LST4	25.4	3040.9	30.4	11.4	2.2
GST4	27.1	3114.4	31.1	11.8	2.3
LST1	27.8	3279.8	32.8	12.7	2.2
GST1	28.7	3272.3	32.7	12.7	2.3
LSI5.0	27.9	3283.9	32.8	12.8	2.2

Appendix A shows the supplemental data from the leaching test. Table A. 1 shows the average measurements of the coupons (1-inch cubes) used during the leaching test. And between Table A. 2 to Table A. 10 show the data of calcium concentration used for the leaching test. Also show the calcium concentration normalized to the weight of the sample, weight of the cement paste and the surface of the sample.

**Table A. 2 LO leaching results**

Sample ID	Date	Time (Days)	sqrt of time	Sample Ca Conc. (mg/L)					per weight		per paste weight		per surface	
				1	2	3	Average	SD	Ca Conc. (mg/L/g) per weight	SD	Ca Conc. (mg/L/g) per paste weight	SD	Ca Conc. (mg/L/cm2) per surface	SD
LO-1-1,2,3 01/30/2022	1/30/2022	2	1.4	38.8	36.9	31.2	35.6	3.9	1.34	0.15	5.36	0.59	1.13	0.12
LO-1-1,2,3 02/01/2022	2/1/2022	4	2.0	53.6	51.6	46.5	50.6	3.7	1.90	0.14	7.60	0.55	1.60	0.12
LO-1-1,2,3 02/03/2022	2/3/2022	6	2.4	71.1	66.6	61.8	66.5	4.7	2.50	0.18	10.00	0.70	2.11	0.15
LO-1-1,2,3 02/05/2022	2/5/2022	8	2.8	80.6	77.4	71.7	76.6	4.5	2.88	0.17	11.50	0.68	2.43	0.14
LO-1-1,2,3 02/08/2022	2/8/2022	11	3.3	90.7	90.4	81.5	87.5	5.2	3.29	0.20	13.15	0.79	2.78	0.17
LO-1-1,2,3 02/10/2022	2/10/2022	13	3.6	97.3	97.0	87.6	94.0	5.5	3.53	0.21	14.12	0.83	2.98	0.17
LO-1-1,2,3 02/12/2022	2/12/2022	15	3.9	103.6	105.1	96.5	101.7	4.6	3.82	0.17	15.29	0.69	3.23	0.15
LO-1-1,2,3 02/14/2022	2/14/2022	17	4.1	110.7	111.4	103.9	108.7	4.1	4.08	0.16	16.33	0.62	3.45	0.13
LO-1-1,2,3 02/16/2022	2/16/2022	19	4.4	118.8	118.7	111.7	116.4	4.1	4.37	0.15	17.49	0.61	3.69	0.13
LO-1-1,2,3 02/18/2022	2/18/2022	21	4.6	114.87	113.53	106.10	111.5	4.7	4.19	0.18	16.75	0.71	3.54	0.15
LO-1-1,2,3 02/20/2022	2/20/2022	23	4.8	118.20	115.17	109.90	114.4	4.2	4.30	0.16	17.19	0.63	3.63	0.13
LO-1-1,2,3 02/22/2022	2/22/2022	25	5.0	122.03	121.37	119.50	121.0	1.3	4.54	0.05	18.18	0.20	3.84	0.04
LO-1-1,2,3 02/24/2022	2/24/2022	27	5.2	127.27	125.93	118.23	123.8	4.9	4.65	0.18	18.60	0.73	3.93	0.15
LO-1-1,2,3 02/26/2022	2/26/2022	29	5.4	129.60	128.27	123.30	127.1	3.3	4.77	0.12	19.09	0.50	4.03	0.11
LO-1-1,2,3 03/01/2022	3/1/2022	32	5.7	143.37	141.87	134.03	139.8	5.0	5.25	0.19	21.00	0.75	4.43	0.16
LO-1-1,2,3 03/03/2022	3/3/2022	34	5.8	155.07	152.57	143.10	150.2	6.3	5.64	0.24	22.58	0.95	4.77	0.20
LO-1-1,2,3 03/05/2022	3/5/2022	36	6.0	153.63	154.30	145.83	151.3	4.7	5.68	0.18	22.73	0.71	4.80	0.15
LO-1-1,2,3 03/07/2022	3/7/2022	38	6.2	157.87	156.57	158.07	157.5	0.8	5.92	0.03	23.67	0.12	5.00	0.03
LO-1-1,2,3 03/09/2022	3/9/2022	40	6.3	163.73	156.30	146.73	155.6	8.5	5.84	0.32	23.38	1.28	4.93	0.27
LO-1-1,2,3 03/13/2022	3/13/2022	44	6.6	163.87	163.93	151.73	159.8	7.0	6.00	0.26	24.02	1.06	5.07	0.22
LO-1-1,2,3 03/16/2022	3/16/2022	47	6.9	169.30	165.73	154.70	163.2	7.6	6.13	0.29	24.53	1.14	5.18	0.24
LO-1-1,2,3 03/18/2022	3/18/2022	49	7.0	167.03	165.93	157.23	163.4	5.4	6.14	0.20	24.55	0.81	5.18	0.17
LO-1-1,2,3 03/19/2022	3/19/2022	50	7.1	168.53	170.20	158.70	165.8	6.2	6.23	0.23	24.92	0.93	5.26	0.20

**Table A. 3 LSI2.5 leaching results**

Sample ID	Date	Days	sqrt of time	Sample Ca Conc. (mg/L)					per weight		per paste weight		per surface	
				1	2	3	Average	SD	Ca Conc. (mg/L/g) per weight	SD	Ca Conc. (mg/L/g) per paste weight	SD	Ca Conc. (mg/L/cm2) per surface	SD
LSI-1-1,2,3 01/30/2022	1/30/2022	2	1.4	34.5	36.0	37.1	35.9	1.3	1.23	0.04	4.92	0.17	1.07	0.04
LSI-1-1,2,3 02/01/2022	2/1/2022	4	2.0	50.6	52.0	52.0	51.5	0.8	1.77	0.03	7.07	0.11	1.54	0.02
LSI-1-1,2,3 02/03/2022	2/3/2022	6	2.4	66.2	66.7	68.0	67.0	0.9	2.30	0.03	9.19	0.12	2.00	0.03
LSI-1-1,2,3 02/05/2022	2/5/2022	8	2.8	76.5	77.9	78.2	77.5	0.9	2.66	0.03	10.63	0.13	2.32	0.03
LSI-1-1,2,3 02/08/2022	2/8/2022	11	3.3	84.5	86.8	88.6	86.7	2.0	2.97	0.07	11.89	0.28	2.59	0.06
LSI-1-1,2,3 02/10/2022	2/10/2022	13	3.6	92.5	93.9	94.7	93.7	1.1	3.21	0.04	12.85	0.15	2.80	0.03
LSI-1-1,2,3 02/12/2022	2/12/2022	15	3.9	98.6	101.5	101.2	100.4	1.6	3.44	0.06	13.77	0.22	3.00	0.05
LSI-1-1,2,3 02/14/2022	2/14/2022	17	4.1	108.5	110.6	111.8	110.3	1.7	3.78	0.06	15.13	0.23	3.30	0.05
LSI-1-1,2,3 02/16/2022	2/16/2022	19	4.4	114.5	116.5	116.7	115.9	1.2	3.98	0.04	15.90	0.17	3.47	0.04
LSI-1-1,2,3 02/18/2022	2/18/2022	21	4.6	108.33	109.53	111.83	109.9	1.8	3.77	0.06	15.08	0.24	3.29	0.05
LSI-1-1,2,3 02/20/2022	2/20/2022	23	4.8	110.87	111.07	114.43	112.1	2.0	3.85	0.07	15.38	0.27	3.35	0.06
LSI-1-1,2,3 02/22/2022	2/22/2022	25	5.0	118.83	120.30	125.47	121.5	3.5	4.17	0.12	16.67	0.48	3.64	0.10
LSI-1-1,2,3 02/24/2022	2/24/2022	27	5.2	122.00	124.40	125.33	123.9	1.7	4.25	0.06	17.00	0.24	3.71	0.05
LSI-1-1,2,3 02/26/2022	2/26/2022	29	5.4	124.50	126.07	129.23	126.6	2.4	4.34	0.08	17.37	0.33	3.79	0.07
LSI-1-1,2,3 03/01/2022	3/1/2022	32	5.7	137.60	140.40	142.80	140.3	2.6	4.81	0.09	19.24	0.36	4.20	0.08
LSI-1-1,2,3 03/03/2022	3/3/2022	34	5.8	145.30	153.07	148.17	148.8	3.9	5.10	0.13	20.42	0.54	4.45	0.12
LSI-1-1,2,3 03/05/2022	3/5/2022	36	6.0	148.40	151.60	154.70	151.6	3.2	5.20	0.11	20.79	0.43	4.53	0.09
LSI-1-1,2,3 03/07/2022	3/7/2022	38	6.2	155.83	158.73	159.97	158.2	2.1	5.42	0.07	21.70	0.29	4.73	0.06
LSI-1-1,2,3 03/09/2022	3/9/2022	40	6.3	149.60	151.60	152.93	151.4	1.7	5.19	0.06	20.77	0.23	4.53	0.05
LSI-1-1,2,3 03/13/2022	3/13/2022	44	6.6	161.83	162.30	162.30	162.1	0.3	5.56	0.01	22.24	0.04	4.85	0.01
LSI-1-1,2,3 03/16/2022	3/16/2022	47	6.9	156.47	159.53	157.97	158.0	1.5	5.42	0.05	21.67	0.21	4.73	0.05
LSI-1-1,2,3 03/18/2022	3/18/2022	49	7.0	157.70	162.77	158.73	159.7	2.7	5.48	0.09	21.91	0.37	4.78	0.08
LSI-1-1,2,3 03/19/2022	3/19/2022	50	7.1	163.00	165.57	162.03	163.5	1.8	5.61	0.06	22.43	0.25	4.89	0.05

**Table A. 4 LSI5.0 leaching results**

Sample ID	Date	Time (Days)	sqrt of time	Sample Ca Conc. (mg/L)					per weight		per paste weight		per surface	
				1	2	3	Average	SD	Ca Conc. (mg/L/g) per weight	SD	Ca Conc. (mg/L/g) per paste weight	SD	Ca Conc. (mg/L/cm2) per surface	SD
LST5.0-1-1 (4) 08/15/2022	8/15/2022	2	1.4	13.57	12.49	12.97	13.01	0.54	0.47	0.02	1.87	0.08	0.40	0.02
LST5.0-1-1 (4) 08/17/2022	8/17/2022	4	2.0	21.16	21.78	20.08	21.00	0.86	0.75	0.03	3.02	0.12	0.64	0.03
LST5.0-1-1 (4) 08/19/2022	8/19/2022	6	2.4	32.84	31.09	33.23	32.38	1.14	1.16	0.04	4.65	0.16	0.99	0.03
LST5.0-1-1 (4) 08/22/2022	8/22/2022	9	3.0	42.48	42.39	42.06	42.31	0.22	1.52	0.01	6.08	0.03	1.29	0.01
LST5.0-1-1 (4) 08/24/2022	8/24/2022	11	3.3	47.35	47.74	44.73	46.60	1.63	1.67	0.06	6.70	0.23	1.42	0.05
LST5.0-1-1 (4) 08/26/2022	8/26/2022	13	3.6	50.96	52.36	49.45	50.92	1.46	1.83	0.05	7.32	0.21	1.55	0.04
LST5.0-1-1 (4) 08/29/2022	8/29/2022	16	4.0	59.04	59.77	57.92	58.91	0.93	2.12	0.03	8.46	0.13	1.80	0.03
LST5.0-1-1 (4) 09/02/2022	9/2/2022	20	4.5	64.92	67.76	64.31	65.66	1.84	2.36	0.07	9.43	0.26	2.00	0.06
LST5.0-1-1 (4) 09/06/2022	9/6/2022	24	4.9	69.8	72.6	70.0	70.80	1.56	2.54	0.06	10.17	0.22	2.16	0.05
LST5.0-1-1 (4) 09/09/2022	9/9/2022	27	5.2	76.8	80.2	74.7	77.25	2.80	2.77	0.10	11.10	0.40	2.36	0.09
LST5.0-1-1 (4) 09/12/2022	9/12/2022	30	5.5	84.3	85.0	82.0	83.77	1.58	3.01	0.06	12.04	0.23	2.55	0.05
LST5.0-1-1 (4) 09/16/2022	9/16/2022	34	5.8	90.3	87.6	86.2	88.02	2.07	3.16	0.07	12.65	0.30	2.68	0.06
LST5.0-1-1 (4) 09/19/2022	9/19/2022	37	6.1	88.9	93.0	89.8	90.56	2.15	3.25	0.08	13.01	0.31	2.76	0.07
LST5.0-1-1 (4) 09/23/2022	9/23/2022	41	6.4	95.6	102.3	97.4	98.44	3.45	3.54	0.12	14.14	0.50	3.00	0.11
LST5.0-1-1 (4) 09/26/2022	9/26/2022	44	6.6	99.6	103.1	97.5	100.06	2.82	3.59	0.10	14.38	0.41	3.05	0.09
LST5.0-1-1 (4) 10/2/2022	10/2/2022	50	7.1	105.9	103.7	102.6	104.02	1.68	3.74	0.06	14.95	0.24	3.17	0.05

**Table A. 5 LST1 leaching results**

Sample ID	Date	Time (Days)	sqrt of time	Sample Ca Conc. (mg/L)					per weight		per paste weight		per surface	
				1	2	3	Average	SD	Ca Conc. (mg/L/g) per weight	SD	Ca Conc. (mg/L/g) per paste weight	SD	Ca Conc. (mg/L/cm2) per surface	SD
LST1-1-1,2,3 06/20/2022	6/20/2022	2	1.4	23.43	19.56	14.33	19.10	4.57	0.69	0.16	2.74	0.66	0.58	0.14
LST1-1-1,2,3 06/22/2022	6/22/2022	4	2.0	35.77	33.15	31.11	33.34	2.33	1.20	0.08	4.79	0.34	1.02	0.07
LST1-1-1,2,3 06/24/2022	6/24/2022	6	2.4	45.56	40.41	38.12	41.36	3.81	1.49	0.14	5.94	0.55	1.26	0.12
LST1-1-1,2,3 06/27/2022	6/27/2022	9	3.0	36.61	39.80	39.48	38.63	1.76	1.39	0.06	5.55	0.25	1.18	0.05
LST1-1-1,2,3 06/29/2022	6/29/2022	11	3.3	57.40	51.01	47.73	52.05	4.91	1.87	0.18	7.48	0.71	1.59	0.15
LST1-1-1,2,3 07/01/2022	7/1/2022	13	3.6	66.33	55.21	51.81	57.78	7.59	2.08	0.27	8.30	1.09	1.76	0.23
LST1-1-1,2,3 07/04/2022	7/4/2022	16	4.0	77.73	65.89	63.15	68.92	7.75	2.48	0.28	9.90	1.11	2.10	0.24
LST1-1-1,2,3 07/06/2022	7/6/2022	18	4.2	82.93	69.42	66.50	72.95	8.77	2.62	0.31	10.48	1.26	2.22	0.27
LST1-1-1,2,3 07/08/2022	7/8/2022	20	4.5	85.91	74.44	71.46	77.27	7.63	2.78	0.27	11.10	1.10	2.36	0.23
LST1-1-1,2,3 07/11/2022	7/11/2022	23	4.8	97.00	80.06	74.90	83.98	11.56	3.02	0.42	12.07	1.66	2.56	0.35
LST1-1-1,2,3 07/13/2022	7/13/2022	25	5.0	100.70	83.50	79.96	88.05	11.10	3.16	0.40	12.65	1.59	2.68	0.34
LST1-1-1,2,3 07/15/2022	7/15/2022	27	5.2	105.60	87.38	81.39	91.46	12.61	3.29	0.45	13.14	1.81	2.79	0.38
LST1-1-1,2,3 07/18/2022	7/18/2022	30	5.5	113.55	94.14	87.25	98.31	13.64	3.53	0.49	14.13	1.96	3.00	0.42
LST1-1-1,2,3 07/20/2022	7/20/2022	32	5.7	112.75	94.35	90.65	99.25	11.84	3.56	0.43	14.26	1.70	3.03	0.36
LST1-1-1,2,3 07/22/2022	7/22/2022	34	5.8	116.40	93.92	90.44	100.25	14.09	3.60	0.51	14.40	2.03	3.06	0.43
LST1-1-1,2,3 07/25/2022	7/25/2022	37	6.1	121.80	98.66	95.64	105.37	14.31	3.78	0.51	15.14	2.06	3.21	0.44
LST1-1-1,2,3 07/27/2022	7/27/2022	39	6.2	124.20	100.99	95.12	106.77	15.38	3.84	0.55	15.34	2.21	3.26	0.47
LST1-1-1,2,3 08/02/2022	8/2/2022	45	6.7	137.70	105.75	100.70	114.72	20.06	4.12	0.72	16.48	2.88	3.50	0.61
LST1-1-1,2,3 08/04/2022	8/4/2022	47	6.9	138.55	107.85	102.50	116.30	19.45	4.18	0.70	16.71	2.80	3.55	0.59
LST1-1-1,2,3 08/07/2022	8/7/2022	50	7.1	151.00	112.80	106.60	123.47	24.05	4.43	0.86	17.74	3.45	3.76	0.73

Table A. 6 LST4 leaching results

Sample ID	Date	Time (Days)	sqrt of time	Sample Ca Conc. (mg/L)					per weight		per paste weight		per surface	
				1	2	3	Average	SD	Ca Conc. (mg/L/g) per weight	SD	Ca Conc. (mg/L/g) per paste weight	SD	Ca Conc. (mg/L/cm2) per surface	SD
LST4-1-1,2,3 03/31/2022	3/31/2022	2	1.4	15.08	14.03	13.93	14.35	0.64	0.57	0.03	2.26	0.10	0.47	0.02
LST4-1-1,2,3 04/02/2022	4/2/2022	4	2.0	24.08	22.45	20.91	22.48	1.58	0.89	0.06	3.55	0.25	0.74	0.05
LST4-1-1,2,3 04/04/2022	4/4/2022	6	2.4	28.61	27.66	26.01	27.42	1.32	1.08	0.05	4.33	0.21	0.90	0.04
LST4-1-1,2,3 04/06/2022	4/6/2022	8	2.8	35.08	33.65	32.14	33.62	1.47	1.33	0.06	5.30	0.23	1.11	0.05
LST4-1-1,2,3 04/08/2022	4/8/2022	10	3.2	38.37	36.60	35.32	36.76	1.53	1.45	0.06	5.80	0.24	1.21	0.05
LST4-1-1,2,3 04/11/2022	4/11/2022	13	3.6	41.41	40.89	39.85	40.72	0.79	1.61	0.03	6.42	0.13	1.34	0.03
LST4-1-1,2,3 04/13/2022	4/13/2022	15	3.9	46.05	44.17	42.71	44.31	1.67	1.75	0.07	6.99	0.26	1.46	0.06
LST4-1-1,2,3 04/16/2022	4/16/2022	18	4.2	50.40	47.92	47.94	48.76	1.43	1.92	0.06	7.69	0.23	1.60	0.05
LST4-1-1,2,3 04/18/2022	4/18/2022	20	4.5	51.34	50.36	49.80	50.50	0.78	1.99	0.03	7.97	0.12	1.66	0.03
LST4-1-1,2,3 04/20/2022	4/20/2022	22	4.7	53.37	51.32	50.47	51.72	1.49	2.04	0.06	8.16	0.24	1.70	0.05
LST4-1-1,2,3 04/22/2022	4/22/2022	24	4.9	55.64	53.17	52.80	53.87	1.54	2.12	0.06	8.50	0.24	1.77	0.05
LST4-1-1,2,3 04/24/2022	4/24/2022	26	5.1	58.30	55.14	54.53	55.99	2.02	2.21	0.08	8.83	0.32	1.84	0.07
LST4-1-1,2,3 04/27/2022	4/27/2022	29	5.4	60.04	57.19	56.52	57.91	1.87	2.28	0.07	9.14	0.29	1.90	0.06
LST4-1-1,2,3 04/29/2022	4/29/2022	31	5.6	62.75	57.83	57.93	59.51	2.81	2.35	0.11	9.39	0.44	1.96	0.09
LST4-1-1,2,3 05/02/2022	5/2/2022	34	5.8	66.77	62.86	62.02	63.89	2.53	2.52	0.10	10.08	0.40	2.10	0.08
LST4-1-1,2,3 05/04/2022	5/4/2022	36	6.0	69.70	65.16	63.63	66.16	3.16	2.61	0.12	10.44	0.50	2.18	0.10
LST4-1-1,2,3 05/06/2022	5/6/2022	38	6.2	71.08	66.22	65.65	67.65	2.99	2.67	0.12	10.67	0.47	2.22	0.10
LST4-1-1,2,3 05/09/2022	5/9/2022	41	6.4	74.11	68.95	67.54	70.20	3.46	2.77	0.14	11.08	0.55	2.31	0.11
LST4-1-1,2,3 05/11/2022	5/11/2022	43	6.6	75.41	70.57	68.93	71.64	3.37	2.83	0.13	11.30	0.53	2.36	0.11
LST4-1-1,2,3 05/13/2022	5/13/2022	45	6.7	77.25	71.82	70.25	73.11	3.68	2.88	0.14	11.53	0.58	2.40	0.12
LST4-1-1,2,3 05/16/2022	5/16/2022	48	6.9	90.35	81.38	83.63	85.12	4.67	3.36	0.18	13.43	0.74	2.80	0.15
LST4-1-1,2,3 05/18/2022	5/18/2022	50	7.1	88.54	83.66	81.96	84.72	3.42	3.34	0.13	13.37	0.54	2.79	0.11

**Table A. 7 GO leaching results**

Sample ID	Date	Time (Days)	sqrt of time	Sample Ca Conc. (mg/L)					per weight		per paste weight		per surface	
				1	2	3	Average	SD	Ca Conc. (mg/L/g) per weight	SD	Ca Conc. (mg/L/g) per paste weight	SD	Ca Conc. (mg/L/cm2) per surface	SD
GO-1-1,2,3 11/17/2021	11/17/2021	2	1.4	30.3	28.7	35.4	31.4	3.5	1.09	0.12	4.38	0.49	0.98	0.11
GO-1-1,2,3 11/19/2021	11/19/2021	4	2.0	42.7	41.4	53.4	45.9	6.6	1.60	0.23	6.39	0.92	1.42	0.20
GO-1-1,2,3 11/22/2021	11/22/2021	7	2.6	58.0	55.6	67.3	60.3	6.2	2.10	0.21	8.40	0.86	1.87	0.19
GO-1-1,2,3 11/24/2021	11/24/2021	9	3.0	64.4	62.1	75.4	67.3	7.1	2.34	0.25	9.37	0.99	2.09	0.22
GO-1-1,2,3 11/26/2021	11/26/2021	11	3.3	70.4	68.1	77.4	72.0	4.8	2.50	0.17	10.02	0.67	2.23	0.15
GO-1-1,2,3 11/29/2021	11/29/2021	14	3.7	75.0	70.2	78.5	74.6	4.1	2.60	0.14	10.38	0.58	2.32	0.13
GO-1-1,2,3 12/2/2021	12/2/2021	17	4.1	79.2	77.3	86.2	80.9	4.7	2.82	0.16	11.26	0.66	2.51	0.15
GO-1-1,2,3 12/04/2021	12/4/2021	19	4.4	84.1	79.2	89.1	84.1	5.0	2.93	0.17	11.72	0.69	2.61	0.15
GO-1-1,2,3 12/07/2021	12/7/2021	22	4.7	86.9	84.6	92.6	88.0	4.1	3.06	0.14	12.26	0.58	2.73	0.13
GO-1-1,2,3 12/09/2021	12/9/2021	24	4.9	105.7	101.9	110.7	106.1	4.4	3.69	0.15	14.77	0.61	3.29	0.14
GO-1-1,2,3 12/13/2021	12/13/2021	28	5.3	108.9	107.8	115.6	110.8	4.2	3.86	0.15	15.42	0.59	3.44	0.13
GO-1-1,2,3 12/15/2021	12/15/2021	30	5.5	109.9	109.5	119.3	112.9	5.5	3.93	0.19	15.72	0.77	3.51	0.17
GO-1-1,2,3 12/17/2021	12/17/2021	32	5.7	112.4	111.4	121.2	115.0	5.4	4.00	0.19	16.01	0.75	3.57	0.17
GO-1-1,2,3 12/20/2021	12/20/2021	35	5.9	120.3	118.1	125.9	121.4	4.0	4.23	0.14	16.91	0.56	3.77	0.12
GO-1-1,2,3 12/22/2021	12/22/2021	37	6.1	118.9	119.2	126.1	121.4	4.1	4.23	0.14	16.90	0.57	3.77	0.13
GO-1-1,2,3 12/27/2021	12/27/2021	42	6.5	124.0	123.6	131.2	126.3	4.3	4.40	0.15	17.58	0.60	3.92	0.13
GO-1-1,2,3 12/29/2021	12/29/2021	44	6.6	131.0	128.6	136.0	131.9	3.8	4.59	0.13	18.36	0.53	4.09	0.12
GO-1-1,2,3 01/04/2022	1/4/2022	50	7.1	135.8	135.5	148.7	140.0	7.5	4.87	0.26	19.49	1.05	4.35	0.23

**Table A. 8 GSI2.5 leaching results**

Sample ID	Date	Days	sqrt of time	Sample Ca Conc. (mg/L)					per weight		per paste weight		per surface	
				1	2	3	Average	SD	Ca Conc. (mg/L/g) per weight	SD	Ca Conc. (mg/L/g) per paste weight	SD	Ca Conc. (mg/L/cm2) per surface	SD
GSI-1-1,2,3 11/17/2021	11/17/2021	2	1.4	31.7	29.5	29.2	30.1	1.4	1.05	0.05	4.19	0.19	0.93	0.04
GSI-1-1,2,3 11/19/2021	11/19/2021	4	2.0	44.0	43.1	41.4	42.9	1.3	1.49	0.05	5.97	0.19	1.32	0.04
GSI-1-1,2,3 11/22/2021	11/22/2021	7	2.6	57.2	57.0	54.1	56.1	1.7	1.95	0.06	7.80	0.24	1.73	0.05
GSI-1-1,2,3 11/24/2021	11/24/2021	9	3.0	65.1	63.6	60.6	63.1	2.3	2.20	0.08	8.78	0.32	1.95	0.07
GSI-1-1,2,3 11/26/2021	11/26/2021	11	3.3	67.1	67.9	63.1	66.0	2.6	2.30	0.09	9.19	0.36	2.04	0.08
GSI-1-1,2,3 11/29/2021	11/29/2021	14	3.7	72.2	74.2	70.7	72.4	1.7	2.52	0.06	10.07	0.24	2.23	0.05
GSI-1-1,2,3 12/02/2021	12/2/2021	17	4.1	78.6	77.9	77.1	77.9	0.7	2.71	0.03	10.84	0.10	2.40	0.02
GSI-1-1,2,3 12/04/2021	12/4/2021	19	4.4	83.0	84.6	81.3	83.0	1.7	2.89	0.06	11.55	0.23	2.56	0.05
GSI-1-1,2,3 12/07/2021	12/7/2021	22	4.7	86.5	86.1	84.2	85.6	1.2	2.98	0.04	11.91	0.17	2.64	0.04
GSI-1-1,2,3 12/09/2021	12/9/2021	24	4.9	103.2	105.9	99.8	103.0	3.0	3.58	0.11	14.33	0.42	3.18	0.09
GSI-1-1,2,3 12/13/2021	12/13/2021	28	5.3	108.9	112.0	105.8	108.9	3.1	3.79	0.11	15.15	0.43	3.36	0.10
GSI-1-1,2,3 12/15/2021	12/15/2021	30	5.5	109.7	115.3	110.2	111.7	3.1	3.89	0.11	15.55	0.43	3.45	0.10
GSI-1-1,2,3 12/17/2021	12/17/2021	32	5.7	111.8	115.1	112.1	113.0	1.8	3.93	0.06	15.72	0.25	3.49	0.06
GSI-1-1,2,3 12/20/2021	12/20/2021	35	5.9	114.8	121.2	116.4	117.5	3.3	4.09	0.12	16.35	0.46	3.62	0.10
GSI-1-1,2,3 12/22/2021	12/22/2021	37	6.1	118.8	123.0	116.3	119.4	3.4	4.15	0.12	16.61	0.47	3.68	0.10
GSI-1-1,2,3 12/27/2021	12/27/2021	42	6.5	125.1	131.6	123.4	126.7	4.3	4.41	0.15	17.63	0.60	3.91	0.13
GSI-1-1,2,3 12/29/2021	12/29/2021	44	6.6	129.3	136.6	128.7	131.5	4.4	4.58	0.15	18.30	0.61	4.06	0.14
GSI-1-1,2,3 01/04/2022	1/4/2022	50	7.1	136.4	142.5	138.2	139.0	3.1	4.84	0.11	19.35	0.44	4.29	0.10

**Table A. 9 GST1 leaching results**

Sample ID	Date	Days	sqrt of time	Sample Ca Conc. (mg/L)					per weight		per paste weight		per surface	
				1	2	3	Average	SD	Ca Conc. (mg/L/g) per weight	SD	Ca Conc. (mg/L/g) per paste weight	SD	Ca Conc. (mg/L/cm2) per surface	SD
GST1-1-1,2,3 08/15/2022	8/15/2022	2	1.4	13.61	11.67	8.04	11.10	2.83	0.39	0.10	1.55	0.39	0.34	0.09
GST1-1-1,2,3 08/17/2022	8/17/2022	4	2.0	24.84	21.19	15.68	20.57	4.61	0.72	0.16	2.87	0.64	0.63	0.14
GST1-1-1,2,3 08/19/2022	8/19/2022	6	2.4	33.63	32.19	26.37	30.73	3.84	1.07	0.13	4.29	0.54	0.94	0.12
GST1-1-1,2,3 08/22/2022	8/22/2022	9	3.0	43.77	38.84	33.93	38.84	4.92	1.35	0.17	5.42	0.69	1.19	0.15
GST1-1-1,2,3 08/24/2022	8/24/2022	11	3.3	48.56	45.11	39.36	44.34	4.65	1.55	0.16	6.19	0.65	1.35	0.14
GST1-1-1,2,3 08/26/2022	8/26/2022	13	3.6	51.60	48.73	41.61	47.31	5.14	1.65	0.18	6.60	0.72	1.45	0.16
GST1-1-1,2,3 08/29/2022	8/29/2022	16	4.0	57.34	53.30	48.34	52.99	4.51	1.85	0.16	7.39	0.63	1.62	0.14
GST1-1-1,2,3 09/02/2022	9/2/2022	20	4.5	66.52	60.56	54.00	60.36	6.26	2.11	0.22	8.42	0.87	1.84	0.19
GST1-1-1,2,3 09/06/2022	9/6/2022	24	4.9	71.5	65.1	60.3	65.63	5.57	2.29	0.19	9.16	0.78	2.01	0.17
GST1-1-1,2,3 09/09/2022	9/9/2022	27	5.2	73.4	67.0	63.3	67.89	5.12	2.37	0.18	9.47	0.71	2.07	0.16
GST1-1-1,2,3 09/12/2022	9/12/2022	30	5.5	79.8	72.1	67.6	73.17	6.17	2.55	0.22	10.21	0.86	2.24	0.19
GST1-1-1,2,3 09/16/2022	9/16/2022	34	5.8	84.4	78.0	72.2	78.21	6.08	2.73	0.21	10.91	0.85	2.39	0.19
GST1-1-1,2,3 09/19/2022	9/19/2022	37	6.1	89.9	80.2	74.0	81.38	8.00	2.84	0.28	11.35	1.12	2.49	0.24
GST1-1-1,2,3 09/23/2022	9/23/2022	41	6.4	96.5	85.6	81.8	87.98	7.63	3.07	0.27	12.27	1.06	2.69	0.23
GST1-1-1,2,3 09/26/2022	9/26/2022	44	6.6	97.8	88.2	82.8	89.59	7.60	3.12	0.27	12.50	1.06	2.74	0.23
GST1-1-1,2,3 10/2/2022	10/2/2022	50	7.1	100.7	91.3	85.2	92.36	7.81	3.22	0.27	12.89	1.09	2.82	0.24

**Table A. 10 GST4 leaching results**

Sample ID	Date	Days	sqrt of time	Sample Ca Conc. (mg/L)					per weight		per paste weight		per surface	
				1	2	3	Average	SD	Ca Conc. (mg/L/g) per weight	SD	Ca Conc. (mg/L/g) per paste weight	SD	Ca Conc. (mg/L/cm <sup>2</sup> ) per surface	SD
GST4-1-1,2,3 03/31/2022	3/31/2022	2	1.4	12.72	14.48	11.18	12.79	1.65	0.47	0.06	1.89	0.24	0.41	0.05
GST4-1-1,2,3 04/02/2022	4/2/2022	4	2.0	20.10	22.69	18.46	20.42	2.13	0.75	0.08	3.01	0.31	0.66	0.07
GST4-1-1,2,3 04/04/2022	4/4/2022	6	2.4	25.03	28.05	24.51	25.86	1.91	0.95	0.07	3.81	0.28	0.83	0.06
GST4-1-1,2,3 04/06/2022	4/6/2022	8	2.8	30.41	33.08	28.33	30.61	2.38	1.13	0.09	4.51	0.35	0.98	0.08
GST4-1-1,2,3 04/08/2022	4/8/2022	10	3.2	33.83	36.67	31.47	33.99	2.61	1.25	0.10	5.01	0.38	1.09	0.08
GST4-1-1,2,3 04/11/2022	4/11/2022	13	3.6	38.20	39.96	36.17	38.11	1.90	1.40	0.07	5.62	0.28	1.22	0.06
GST4-1-1,2,3 04/13/2022	4/13/2022	15	3.9	42.03	44.06	39.53	41.87	2.27	1.54	0.08	6.17	0.33	1.34	0.07
GST4-1-1,2,3 04/16/2022	4/16/2022	18	4.2	45.73	47.53	42.84	45.37	2.37	1.67	0.09	6.69	0.35	1.46	0.08
GST4-1-1,2,3 04/18/2022	4/18/2022	20	4.5	47.84	49.13	44.78	47.25	2.24	1.74	0.08	6.97	0.33	1.52	0.07
GST4-1-1,2,3 04/20/2022	4/20/2022	22	4.7	49.21	51.09	46.39	48.90	2.37	1.80	0.09	7.21	0.35	1.57	0.08
GST4-1-1,2,3 04/22/2022	4/22/2022	24	4.9	52.15	53.28	48.80	51.41	2.33	1.90	0.09	7.58	0.34	1.65	0.07
GST4-1-1,2,3 04/24/2022	4/24/2022	26	5.1	53.33	55.15	50.22	52.90	2.49	1.95	0.09	7.80	0.37	1.70	0.08
GST4-1-1,2,3 04/27/2022	4/27/2022	29	5.4	56.23	57.67	52.22	55.38	2.82	2.04	0.10	8.17	0.42	1.78	0.09
GST4-1-1,2,3 04/29/2022	4/29/2022	31	5.6	57.99	59.45	54.23	57.22	2.69	2.11	0.10	8.44	0.40	1.84	0.09
GST4-1-1,2,3 05/02/2022	5/2/2022	34	5.8	62.50	64.31	58.17	61.66	3.15	2.27	0.12	9.09	0.46	1.98	0.10
GST4-1-1,2,3 05/04/2022	5/4/2022	36	6.0	63.93	65.87	59.92	63.24	3.03	2.33	0.11	9.33	0.45	2.03	0.10
GST4-1-1,2,3 05/06/2022	5/6/2022	38	6.2	66.37	68.67	61.78	65.61	3.51	2.42	0.13	9.67	0.52	2.11	0.11
GST4-1-1,2,3 05/09/2022	5/9/2022	41	6.4	69.45	70.87	63.62	67.98	3.84	2.51	0.14	10.02	0.57	2.18	0.12
GST4-1-1,2,3 05/11/2022	5/11/2022	43	6.6	69.24	72.11	64.10	68.48	4.06	2.52	0.15	10.10	0.60	2.20	0.13
GST4-1-1,2,3 05/13/2022	5/13/2022	45	6.7	72.07	73.18	65.22	70.16	4.31	2.59	0.16	10.35	0.64	2.25	0.14
GST4-1-1,2,3 05/16/2022	5/16/2022	48	6.9	83.18	83.97	75.63	80.93	4.60	2.98	0.17	11.93	0.68	2.60	0.15
GST4-1-1,2,305/18/2022	5/18/2022	50	7.1	83.65	83.74	75.74	81.04	4.60	2.99	0.17	11.95	0.68	2.60	0.15