

**DURABILITY STUDY AND CHARACTERIZATION OF
SELF-COMPACTING CONCRETE DEVELOPED
UTILIZING SILICO-MANGANESE FUME**

BY

MAZEN ANWAR SAEED ABDULLAH

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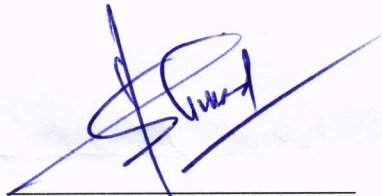
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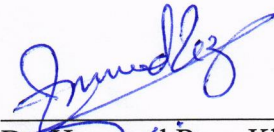
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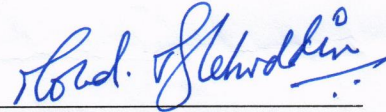
This thesis, written by **MAZEN ANWAR SAEED ABDULLAH** under the direction of his thesis advisor and approved by his thesis committee, has been presented and accepted by the Dean of Graduate Studies, in partial fulfillment of the requirements for the degree of **MASTER OF SCIENCE IN CIVIL ENGINEERING**.



Dr. Shamsad Ahmad
Department Chairman



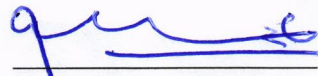
Dr. Hammad Raza Khalid
(Advisor)



Dr. Mohammed Maslehuddin
(Member)



Dr. Suliman Al-Homidan
Dean of Graduate Studies



Dr. Mohammed Al-Osta
(Member)

Date



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This work is dedicated to my beloved parents, brothers, and sisters.

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LIST OF ABBREVIATIONS

SCC	:	Self-compacting Concrete
NVC	:	Normally vibrated Concrete
OPC	:	Ordinary Portland Cement
SMF	:	Silico-manganese Fume
SMS	:	Silico-manganese Slag
LSP	:	Limestone Powder
AABs	:	Alkali-activated Binders
GGBFS	:	Ground Granulated blast furnace slag
SCMs	:	Supplementary Cementitious Materials
SP	:	Superplasticizer
VMA	:	Viscosity Modifying Agent
ASTM	:	American Society for Testing and Materials
RCPT	:	Rapid Chloride Permeability Test
W/C	:	Water/cement ratio
SEM	:	Scanning Electron Microscope
XRD	:	X-ray Diffraction

ABSTRACT

Full Name : MAZEN ANWAR SAEED ABDULLAH

Thesis Title : Durability Study and Characterization of Self-Compacting Concrete
Developed Utilizing Silico-Manganese Fume

Major Field : CIVIL ENGINEERING (STRUCTURES)

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Self-compacting Concrete (SCC) has recently received considerable attention from several structural engineers and researchers due to its exceptional fresh and hardened properties. To overcome the technical, environmental, and economic issues regarding the use of high cement content in SCC, modern approaches are endeavored to replace the cement partially with industrial waste materials such as red mud, slag, palm oil fuel ash, and glass powder. Locally, there is a growing demand to produce steel alloys and thereby generating an enormous amount of waste by-products like slag and silico-manganese fume (SMF). In this thesis, an experimental investigation has been conducted to evaluate the fresh and durability performance of SCC mixes developed by incorporating SMF in weight proportions of 10-70%. All concrete mixes have met the flowability criteria required by EFNARC with good passing ability, filling ability, and segregation resistance. The addition of SMF was reported to increase the setting times as well as retard the hydration reaction of paste samples. Concrete mixes containing up to 20% SMF were superior to the control mix at all curing ages in their compressive strengths. Moreover, SEM of these samples subsidizes the densified microstructure in these samples due to the limited pozzolanic reaction of SMF and the micro filling of its finer particles. The inclusion of SMF up to 30%

in SCC has resulted in lower chloride permeability, and higher electrical resistivity compared to the control sample. The initiation of reinforcement corrosion was delayed in concrete specimens blended with 10% to 40% SMF compared to other mixes. All concrete mixes exhibited deterioration and disintegration when immersed in 2% sulfuric acid due to the formation of gypsum. Moreover, concrete containing 20% and 30% SMF showed lower strength reductions at 3 and 6 months of acid exposure. The results of thermal conductivity of mixes with high SMF content showed lower values compared to the control mix due to the porous microstructure and reduction in density. For the chloride diffusion results, higher values of apparent diffusion coefficient but with lower surface chloride content were obtained due to the increase in SMF content.

ملخص الرسالة

الاسم الكامل: مازن أنور سعيد عبدالله

عنوان الرسالة: دراسة الاديمومة والتحليل الجزيئي لخرسانة ذاتية الدمك طورت باستخدام بودرة السيليكو منغنيز

التخصص: الهندسة المدنية (الإتشاءات)

تاريخ الدرجة العلمية: مايو 2022 م

حظيت الخرسانة ذاتيك الدمك مؤخرا باهتمام كبير من قبل العديد من الباحثين والمهندسين الإنشائيين نظراً لمزاياها وخصائصها الاستثنائية الطازجة والمتصلبة. للتغلب على المشكلات الفنية والبيئية والاقتصادية المتعلقة باستخدام نسبة عالية من الأسمنت في الخرسانة ذاتية الدمك، يتم السعي وراء الأساليب الحديثة لاستبدال الأسمنت جزئياً بمواد النفايات الصناعية مثل الطين الأحمر، والخبث، ورماد وقود زيت النخيل، ومسحوق الزجاج، إلخ. محلياً، هناك طلب متزايد على إنتاج السبائك الصلبة وبالتالي تولد كميات هائلة من المنتجات الثانوية مثل الخبث وبودرة السيليكو منغنيز. في هذا البحث، تم إجراء تحقيق تجريبي لتقييم الأداء الطازج والديمومة لخلائط الخرسانة ذاتية الدمك المطورة من خلال دمج بودرة السيليكو منغنيز بنسب وزن 10-70%. لقد استوفت جميع الخلائط الخرسانية معايير الانسيابية الأوروبية المطلوبة EFNARC مع قدرة تمرير جيدة وملء مقاومة الفصل. إضافة بودرة السيليكو منغنيز ساهم في زيادة وقت الإعداد وكذلك حرارة الإماهة للخلائط الطازجة. كانت الخلائط الخرسانية المحتوية على ما يصل إلى 20% من بودرة السيليكو منغنيز متفوقة على مزيج التحكم في جميع أعمار المعالجة في مقاومتهم للانضغاط. علاوة على ذلك، تؤكد تقنية SEM لهذه العينات البنية المجهرية المكثفة بسبب التفاعل البوزولاني المحدود لبودرة السيليكو منغنيز والتعبئة الدقيقة لجزيئاتها الدقيقة. أدى تضمين بودرة السيليكو منغنيز حتى 30% في الخرسانة ذاتية الدمك إلى انخفاض نفاذية الكلوريد وارتفاع المقاومة الكهربائية مقارنة بعينة التحكم. تأخر بدء تآكل التسليح في عينات الخرسانة الممزوجة بنسبة 10% إلى 40% من بودرة السيليكو منغنيز مقارنة مع الخلطات الأخرى. أظهرت جميع الخلائط الخرسانية تدهوراً وتفككاً عند غمرها في حامض الكبريتيك بنسبة 2% نتيجة تكون الجبس. علاوة على ذلك، أظهرت الخرسانة المحتوية على 20% و 30% من بودرة السيليكو منغنيز انخفاضاً أقل في القوة عند 3 و 6 أشهر من التعرض للحامض. أظهرت نتائج التوصيل الحراري للخلطات ذات المحتوى العالي من بودرة السيليكو منغنيز قيماً أقل مقارنةً من مزيج التحكم بسبب

البنية المجهرية المسامية وانخفاض الكثافة. بالنسبة لنتائج انتشار الكلوريد، تم الحصول على قيم أعلى لمعامل الانتشار الظاهري، ولكن بمحتوى كلوريد سطح أقل بسبب الزيادة في محتوى بودة السيلكو منغنيز.

CHAPTER 1

INTRODUCTION

1.1 General Background about Concrete and SCC

Concrete is the most utilized construction material for various civil engineering applications. In this regard, the global annual consumption is estimated to be about 25 billion tons [1,2]. Moreover, an increase in concrete consumption by 12-23% is predicted by 2050 compared to 2014 [2]. Concrete possesses special characteristics that make it the most versatile material in construction compared to other materials, such as wood and steel. It can be molded into different shapes and sizes and has better performance in terms of fire resistance compressive strength, corrosion resistance, and durability [3]. Nevertheless, the casting of such concrete, which is known as normally vibrated concrete (NVC) involves subsequently vibrating it to ensure good compaction to all layers before placing it [4]. In Japan, the durability performance of NVC has been criticized in the 1980s [5]. This was attributed to the improper consolidation executed by unskilled labor which adversely affected the mechanical and durability properties of concrete [6,7]. Placing the concrete following improper or inadequate compaction may result in low strength, porous, and high permeability concrete which permits the access of aggressive agents to penetrate the concrete and deteriorate the reinforcement. Moreover, this mechanical process generates a lot of noise during construction which negatively affects the labor's health [8].

Furthermore, modern concrete structures represented by the complexity of the construction, congested reinforcement details, and advances in architectural concepts are among the key challenges to the use of NVC. Consequently, there was a vital need to develop a new type of concrete that overcome the difficulties and challenges associated with NVC.

Hence, the solution was lying in developing concrete that can consolidate and flow under its own weight to fill every corner around the reinforcement, thus eliminating the necessity to vibrate it. Therefore, in the period between 1986 and 1989, Self-compacting concrete (SCC) was proposed as a new type of concrete and a prototype was made [5]. SCC can flow in congested reinforcement regions or in places where it is very difficult to perform mechanical vibration such as cast-in-situ foundations, underwater concreting, and walls or columns having dense reinforcement. Nowadays, SCC is considered a breakthrough in concrete technology, and more studies are being conducted to assess and evaluate its properties.

The materials needed to produce SCC are analogous to that of NVC but there is a substantial difference in the relative quantities of each ingredient [9,10]. SCC has to achieve high flowability along with some intrinsic properties such as segregation resistance, passing ability, and filling ability [11]. This demands the use of limited coarse aggregate content and low w/c ratio with the inclusion of certain amounts of chemical admixtures like superplasticizers and higher amounts of cement content and finer materials [9,12]. These quantities are prepared with a well-designed mix and adjusted during the trial mixes to satisfy the flow criteria. However, the usage of higher cement content and

chemical admixtures in SCC can cause a significant upsurge in its cost [13]. Therefore, the current trend is oriented towards reducing the cement content by incorporating SCMs like industrial by-products to produce an eco-friendly SCC.

1.2 Development of Sustainable SCC using Waste Materials

The global growth in the production of cement as the main binder of concrete has contributed significantly to the drastic increase in CO₂ emissions [1,2]. Around 7% of CO₂ produced globally is mainly due to cement production. Accordingly, the production of clinker as the active binding component of the cement is playing a key role in increasing anthropogenic CO₂ emissions [14]. Moreover, the burning of fossil fuels for heating the raw ingredients is further contributed to the CO₂ emissions [14].

Therefore, the current research trend is to utilize industrial by-products in certain proportions to produce alternative concrete binders. Accordingly, large volumes of waste materials are generated annually by industrial sectors. For example, in the UK, nearly three million tons of waste glass are generated, and a small amount of this waste is recycled. Moreover, most of these industrial wastes are non-biodegradable and dumped in landfills which are not the best remedy for their enormous volume [15]. The disposal of these materials in landfills can create critical health problems and real threats to the surrounding environment. For instance, heavy metals can be leached out of fly ash disposed in landfills which eventually leads to possible contamination of groundwater [6]. In addition, most of these hazardous wastes like electric arc furnace dust are very fine and have high heavy metal content which poses a great threat in handling them [16]. Furthermore, significant

expenditure is expected for treatment, processing, transport, and land needed for the disposal endeavors [17]. Therefore, the sustainability of SCC has become an essential solution for most of these waste materials. Numerous recent studies were conducted to incorporate industrial and agricultural waste materials in developing SCC with superior properties, including rice husk ash [18], palm oil fuel ash [19], silica fume [20], plastic waste [21], fly ash [22], glass powder [23], natural pozzolan [24], and red mud [25].

1.3 Steel Alloys By-products and their Current Applications

Silico-manganese alloys are one of the essential additives in the production of high-quality steel [26]. According to International Manganese Institute statistics, the production of silico-manganese alloys reached 16 million tons in 2018 with more than 16% increment compared to 2017 [27]. During the production of these alloys, raw materials including minerals, ores, slags, and reductants (coke and coal) are fed at the top of electric arc furnaces. Then, oxidizing raw materials are smelted in the furnaces under 1500 °C in a carbothermic reduction process to form a raw metal that is tapped, refined, casted, and crushed based on customers' specifications [28–30]. Two by-products are generated in silico-manganese plants, namely Silico-manganese slag (SMS) and Silico-manganese fume (SMF). It has been recently reported that nearly 1.2-1.4 tons and 100 kg of SMS and SMF, respectively, are generated for each ton of alloys produced [29–31]. As industrial by-products, fumes generated in these plants can be disclosed as airborne particulate materials that are formed by the evaporation of liquid metal during the production of the alloys [28]. While the furnaces are the major source of these fumes, modern furnaces are combined with powerful filter and ventilation systems to remove top gases and fumes evaporating

during alloy production [28]. SMF is chemically dominated by manganese and potassium oxides and contains ultrafine agglomerated spherical particles with an average size ranging from micro to nanoscale. Due to its ultrafine particle size and higher manganese oxide content that reaches 35%, the production of SMF has detrimental effects on the health of human beings and the air quality of cities and suburbs [32]. It has been reported that overexposure to higher concentrations of manganese can cause serious health complications in both children and the elderly like Parkinson's and pediatric diseases [33–35]. Pavilonis et al. [35] elucidated that many trace metals including manganese are released as settled dust or suspended aerosols due to the production of steel alloys which adversely affect the population living closer to these plants. The average airborne manganese concentration of $0.15 \mu\text{g}/\text{m}^3$ per year is specified by the World health organization, however, higher manganese levels of $0.5 \mu\text{g}/\text{m}^3$ were measured in the nearby silico-manganese plants [34]. Although there have been many studies that centered on the recycling of dust and fumes back to arc furnaces or smelters, the presence of several impurities in their composition has hindered their reuse [36]. Therefore, most of the waste materials generated in alloy plants are disposed in landfills regularly [33,37,38]. Nevertheless, the disposal of finer materials in landfills may result in several consequences such as land scarcity, leaching of heavy metals, and groundwater contamination [6].

Recently, numerous studies were conducted to incorporate SMF in various applications to lessen environmental and health concerns. Najmuddin et al. [30] developed SMF-based alkali-activated mortars (AAMs) that cured under low temperatures by using different ratios of alkaline activators (NaOH (NH) and Na_2SiO_3 (NS)). Although SMF contains

smaller contents of calcareous and aluminous constituents, the results deduced that SMF is a good precursor in the synthesis of AAMs due to the higher reactivity of MnO_2 with NS and the formation of many gel products such as nchwanningite, glaucochroite, and bustamite. The study also revealed that the spherical nature of SMF particles enhances the flow results. The maximum strength obtained in the developed AAMs was 36 MPa at NS/NH of 2.5 and 12 M of NH. The work on developing AABs using SMF has been advanced by blending it with ground granulated blast furnace slag (GGBFS) at different curing regimes to study the fresh, mechanical, and durability characteristics [38–42]. The developed AABs exhibited acceptable fresh and strength properties with the notable formation of phases such as K-A-S-H, Mn-S-H, and C-Mn-S-H due to the excellent synergy of both precursors. In particular, the best synergistic effect on strength and workability was attained when 70% SMF was blended with 30% BFS [39,40]. Apart from using SMF as a structural material, Razzaq et al. [43] recently studied the bridging capability, flow, and rheology of the marble and SMF water-based fluids (WBF). Compared with marble-based WBFs, a remarkable enhancement of filtration loss and rheological properties including yield point and plastic viscosity has been revealed in SMF-based WBF. It is well established from the previous studies, that the inclusion of SMF has a major influence on workability, rheology, consistency, setting times, and hardened properties. This is mainly ascribed to its intrinsic physicochemical properties that contribute to its growing applications.

In addition, Limestone powder (LSP) was also employed in this research to replace 20% of the fine aggregate in all mixes. LSP is considered a non-pozzolanic, calcareous material

which mainly used as a raw material in clinker production [44], but incorporating it in concrete brings about several benefits, including increased early strength, reduced bleeding, improved workability, and superior microstructure densification [45–47]. Moreover, the deformability and viscosity of SCC are improved, and porosity is reduced due to the incorporation of LSP [45]. This is mainly ascribed to the action mechanisms of limestone in cement-based systems like filler, dilution, and chemical effects [48]. Li et al. [49] have carried out an investigation of concrete porosity and durability characteristics using varied limestone content from 30% to 50% by weight. The results showed that incorporating up to 30% of limestone has slightly reduced the porosity and mean pore diameter owing to the filler and the hydration accelerating effects. Moreover, increasing the surface area of limestone led to a decrement in chloride diffusion coefficient due to its role in filling the gaps in the concrete skeleton and improving the packing density. However, it is not recommended to increase the content of limestone beyond 30% to avoid detrimental effects on porosity and durability. Sua-Iam and Makul [47] developed SCC mixes using a combination of limestone powder (LSP) and rice husk ash (RHA) in replacement of fine aggregate. A substantial decrease in water demand in the combined LSP and RHA mixes is due to the reduced flocculation and effective dispersion of particles. The concrete mixes exhibited satisfactory workability properties such as improved slump flow, viscosity, and sufficient segregation resistance. Although strength values were decreasing with increasing RHA, LSP, and LSP/RHA contents, there was a higher strength development at early ages, particularly in LSP-based mixes. This is accredited to the filling effect and the limited pozzolanic reactions.

The research on developing concrete binders using SMF has been limited to alkali-activated binders. Accordingly, the current applications may not meet the increasing SMF volume owing to the growing demands for steel alloys. Thus, there is a pivotal need to expand its potential utility in structural applications, particularly by investigating the possibility of developing SMF-based SCC. SMF was chosen in this study as a good candidate to replace cement in SCC due to its unique physicochemical properties and the need to produce eco-friendly concrete using local waste materials. The proposed research intends to study the possibility of developing SCC in which cement is being replaced with SMF content varying from 0 to 100% by weight on an increment of 10%. Several trial experiments have been done based on acceptance flow criteria to select the optimum mixes. Consequently, specimens will be prepared for durability and thermal conductivity investigation. Finally, characterization techniques, including SEM and XRD are utilized to study morphology and different phases in prepared paste samples. The findings of this study may construct practical avenues for future applications and research works.

1.4 Need for the Research

The last 10 years have witnessed increasingly rapid advances in developing SCC with better properties than conventionally vibrated concrete [50]. Compared with conventional concrete, SCC can offer several advantages such as less-skilled labor needed, no vibration required, reduction in construction time, better surface finishing, freedom in architectural designs, higher durability, higher bond strength, etc. Nevertheless, there is still a need to carry out a comprehensive investigation and assessment of its fresh and durability

properties to encourage the local construction industry to utilize this concrete on future construction projects.

Numerous studies about SCC reported the inclusion of various SCMs or mineral admixtures like rice husk ash, red mud, palm oil fuel ash, marble powder, and slag. These materials are of paramount importance to amend the workability and hardened properties of SCC due to their intrinsic physicochemical properties. However, some of these materials may not be available locally for utilizing them in producing SCC which could increase its cost. Therefore, the modern methodology is to seek alternative local waste materials which are generated in many industries such as the ferroalloy industry and get disposed in landfills. Accordingly, the incorporation of SMF as industrial waste material in producing SCC could be a solution to its disposal costs and potential environmental problems. Moreover, this is a promising topic in concrete technology in which cement is being substituted at certain levels and eventually this will reduce the cost of concrete and the environmental issues associated with its growing production. Recently, SMF has been utilized to develop and assess alkali-activated binders based on SMF alone [30] as well as blended with blast furnace slag. The results showed that SMF is a good precursor for developing AAMs, particularly when blended with slag [38–40]. It has enhanced the workability due to the physical role of its spherical particles and improved the strength and durability owing to the chemical effect and the generation of many gel products.

To date, SMF has never been used in developing SCC. Hence, this study intends to explore the possibility of exploiting SMF in developing an eco-friendly SCC. Moreover, the

durability properties and thermal conductivity will be deeply investigated in this study. XRD and SEM/EDS techniques will be utilized for the characterization of the mixes.

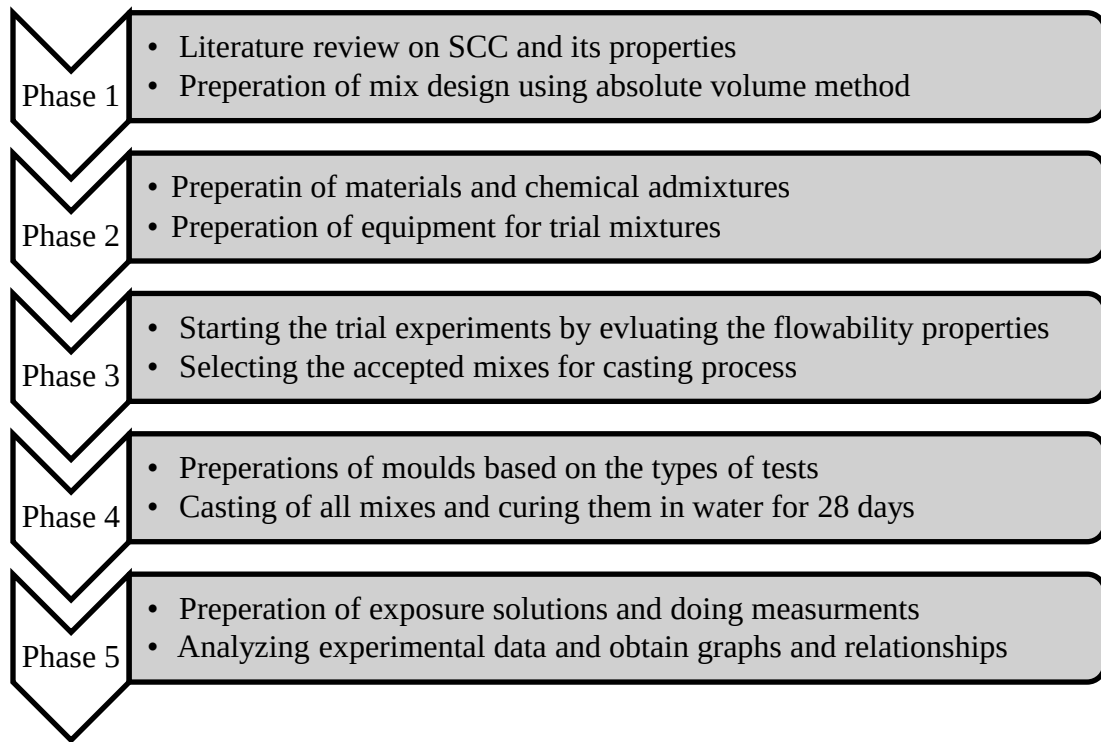
1.5 Objectives

The main objective of this study is to develop SCC utilizing an industrial waste product, namely silico-manganese fume (SMF). The specific objectives of the proposed research are the following:

1. Select the optimum mixes that pass the flow criteria of SCC.
2. Investigate the durability characteristics of developed SCC.
3. Evaluate the thermal conductivity of SCC.
4. Study the heat of hydration of paste samples blended with SMF.
5. Characterize the mineralogy and structure of the developed SCC.
6. Develop correlations between some of the hardened properties of the developed SCC.
7. Provide recommendations on the avenues use of the developed SCC.

1.6 Research Plan

The research work was conducted in different phases as shown in the diagram below:



1.7 Thesis Organization

The frame of this thesis is organized into 6 chapters as summarized below:

Chapter 1 gives an overview about SCC, Steel alloys, SMF, the Significance of this research, and its Objectives.

Chapter 2 reviews the previous studies related to the fresh and durability properties of SCC and the heat of hydration of cementitious materials.

Chapter 3 is devoted to the material's composition and properties, trial mixes, and durability tests and equipment.

Chapter 4 presents the results and discussions as planned in the experimental program.

Chapter 5 focuses on the characterization of some paste samples.

Chapter 6 presents conclusions, recommendations, and suggestions for future studies based on the obtained results.

CHAPTER 2

LITERATURE REVIEW

Since its development in the late 20th century, SCC has gained more attention from many researchers due to its unique flowing properties, improved hardened properties, and wider applications. Therefore, SCC is considered a milestone achievement in concrete technology which needs more research and local adaptation in various structural applications in the Gulf region, especially in Saudi Arabia. In the last twenty years, researchers have focused on developing SCC utilizing different mineral admixtures generated as industrial by-products to replace the cement or aggregates. These studies declare that the inclusion of these materials results in better flowing, rheological and hardened properties and a significant reduction in cost and CO₂ emissions. In this literature review, five main parts will be discussed. The first part presents the historical development of SCC and some of its advantages and drawbacks. The second part discusses the basic ingredients of SCC. In the third part, a review of some studies on SCC utilizing various waste materials and their fresh and durability performance will be introduced. The fourth part briefly highlights the heat of hydration of cementitious materials. The last part presents the summary of the literature review and research gap.

2.1 Self-compacting Concrete History, Applications, and Advantages

Concrete is a very viscous, dense material that demands the use of vibrational techniques to remove any air bubbles or honeycomb-like holes entrapped at the surface during pouring [51]. However, the vibrational process is tedious and sometimes performed improperly or inadequately [51]. In addition, complicated formworks and dense reinforcement regions make the compaction even more difficult [51]. It has been reported that insufficient and uneven vibrations in congested reinforcement regions have adverse effects on the durability and permeability of concrete [52]. Safe working conditions are also declining as more noise is generated during vibration. These reasons were sufficient to upgrade the ordinary vibrated concrete to a newly developed concrete that eliminates the vibration and accelerates the construction process.

Self-compacting concrete was mainly developed in Japan in the 1980s after the emergence of durability concerns over vibrated concrete [7]. This period witnessed a significant reduction in the number of trained workers and the quality of concrete. In the mid and late 1990s, Europe has begun to adapt SCC in many structural applications and set many standards and specifications [6]. For example, transportation networks were implemented in Sweden using SCC [53]. Moreover, SCC has been utilized successfully in Europe in many applications such as walls, tunnels, and bridges. In the United States, standards were also established, and SCC was involved in the precast construction industry [6].

Self-compacting concrete is high-performance concrete that is highly flowable and does not need any external vibrational assistance in compacting its layers [4,54–56]. This

concrete can flow under its own weight to fill the gaps and corners in the formwork and places of congested reinforcement that cannot be reached or is difficult to be compacted by conventional vibration methods [57]. Some of these places include bridges, foundations, and beam-column joints. Moreover, this concrete can be handled without segregation or bleeding as it has enough viscosity and cohesiveness [55].

SCC offers several advantages related to construction and performance. A substantial reduction in construction time and labor cost besides better surface finishing are the main benefits of SCC's unique properties [54,58]. It can also be advantageous in the casting of structural elements of dense reinforcement, complicated shapes, or difficult to reach. Moreover, better working conditions such as reduction of noise and vibration-related injuries are guaranteed when vibration is eliminated in most structural applications [54,59].

Despite the various advantages of using SCC, there exist some drawbacks that consider challenges for researchers and manufacturers of this concrete:

1. To produce SCC, high fluidity and workability should be employed. This can be achieved by increasing cement content and the inclusion of chemical admixtures such as superplasticizers. However, this will raise the cost of concrete and can cause some durability problems like higher shrinkage, cracks, and environmental issues by increasing CO₂ emissions [56,60,61].
2. Another drawback is the segregation and bleeding which occur due to failure in mix proportioning such as using excessive dosages of superplasticizer or not adding viscosity modifying agent [4,62]. During pumping and transporting of this

concrete, especially in restricted areas, the risk of segregation of paste and aggregates is increased which leads to some serious issues such as excessive porosity, honeycombs, reinforcement, and pipe blocking [62,63]. This has an adverse impact on workability and durability properties [62].

3. Another disadvantage that limits the use of SCC is its lower robustness and higher sensitivity. Small changes in the mix proportions, mixing methods, and material properties of SCC can cause considerable changes in the measured properties [64].

Numerous structural applications have been accomplished using SCC in the early stages of its discovery. For instance, two anchorages of a suspension bridge of 1991 m span were constructed with this concrete [65]. In 1998, SCC was utilized in constructing a wall of a natural gas tank where construction time took only 18 months instead of 22 months [65]. The potential uses of SCC have gradually increased worldwide for many structural applications such as precast structures [64]. SCC was used in the pumping of very tall buildings due to its unique flowability properties. A recent application of SCC was seen in the production of hazardous waste storage containers as reported by Esquinas et al. [66]. Mechanical and durability behavior of self-compacting concretes for application in the manufacture. SCC was recently utilized as a retrofitting material in the strengthening and rehabilitation of reinforced concrete due to its excellent fresh properties, less or no vibrational demands, and ability to flow in congested reinforcements [67–69]. Moreover, the appearance of some durability problems such as honeycombing and voids or a possible discontinuity between SCC and old concrete layer can be avoided when SCC is used [69].

Three functional requirements are satisfied in the mix design of any SCC :passing ability, filling ability, and segregation resistance. These requirements are achieved by using high amounts of fines, superplasticizers, and sometimes viscosity modifying agents to control the bleeding and segregation [7].

2.2 Self-compacting Concrete Materials

Although SCC and NVC share similar ingredients in their mix design, they differ in the relative quantities of some ingredients based on essential requirements [9]. For instance, a high amount of SP, powder content, and fine aggregate content with a low w/c ratio are incorporated in SCC to increase the flowability and lubrication and reduce the propensity to segregation [9,70].

2.2.1 Powder

Powder is a general term that includes different cement types, various supplementary cementitious materials, and fillers. In SCC, it is used to refer to a combination of cement and some additives/fillers having particle sizes less than 0.125 mm [71,72]. Although cement is the main binder in various types of concrete, it is significantly considered an extremely energy-intensive and non-environmentally friendly product [71]. Moreover, cement has an adverse influence on concrete properties when exceeded certain limits. The high cement content required by SCC mix design is usually met by replacing it with many additions/fillers in SCC known as supplementary cementitious materials (SCMs) to achieve sustainability. Some common SCMs that are used in SCC are blast furnace slag, limestone powder, fly ash, red mud, glass powder, silica fume, etc. When SCMs are

incorporated in concrete at optimum levels, they bring about several benefits, including a reduction in harmful emissions and costs and enhancement of fresh and durability characteristics [73,74].

SCMs in concrete may contribute to an increase or a decrease in cement hydration due to their physical properties and reactivity [74]. There are many factors affecting their reactivity which include water-to-binder ratio, replacement level, SCM fineness, and chemical composition of cement and SCMs [74]. According to Suraneni et al. [74], these mineral admixtures can be categorized into three types based on their pozzolanic reactivity. The first group includes materials of pozzolanic reactivity that react with water and calcium hydroxide. The next group is known as latent hydraulic materials that react with water if they got activated by temperature, high pH, or other means. The last type consists of chemically inert materials or weak pozzolanic reactivity.

2.2.2 Aggregates

Although there is a lower relative proportion of aggregates in SCC compared to normally vibrated concrete, it is still considered a major constituent of SCC and has a significant influence on its properties. In SCC, The maximum allowable aggregate size is usually in the limited range of 12 to 20 mm [75]. The ratio of coarse to fine aggregate in SCC is reduced to allow free flowability of mortar layer between coarse aggregate grains and to reduce possible interlocking and bridging when concrete flows in narrow gaps between reinforcement [75]. The maximum size and shape of coarse aggregates and their particle size distribution have a direct influence on the flowability and passing ability of SCC [58]. For instance, blocking can be eliminated when using more spherical aggregate particles as

a result of lower internal friction [75]. In recent studies, there is a considerable tendency toward replacing natural aggregates in SCC with waste materials [18,52,59]. Environmental challenges due to the growth in consumption of solid wastes and excessive depletion of natural resources constitute the main reasons behind partially replacing aggregates with waste materials [76].

2.2.3 Chemical Admixtures

The quality of concrete is enhanced using different chemical admixtures in mixing, transporting, placing, and curing [77]. Some examples include accelerators, retarders, shrinkage reducers, corrosion inhibitors, superplasticizers, viscosity modifying agents, etc. High water reducers such as superplasticizers are among the essential admixtures in SCC mixes which ensure the necessary flowability. Previous generations of superplasticizers were based on Naphthalene and Melamine, however, now the developed ones are based on poly-carboxylic ether. Some SCMs such as palm oil fuel ash (POFA) possess a higher surface area which increases the water demand [56]. Therefore, workability is reported to reduce with higher POFA content. The addition of a small superplasticizer dosage (1.3%-1.8% of binder) was a beneficial solution to prevent any addition of extra water that makes the concrete susceptible to deterioration and degradation. At a fixed w/b ratio, the workability of concrete containing fly ash increases with high fly ash content. Therefore, lower superplasticizer dosages were added compared to the control mix and mix having silica fume [78]. On the other hand, silica fume having a higher surface area requires the addition of higher doses of superplasticizers to satisfy required workability values [78].

Viscosity modifying agents are also one of the common chemical admixtures that are employed in SCC mixes. Mixture viscosity is strictly related to segregation and filling ability which is modified using a suitable viscosity admixture [79]. Lower dosages of this admixture are often added to SCC compared with SP to prevent any segregation and control the viscosity [80]. Unlike common liquid VMA that is produced in some companies, waste materials replacing the cement can be described as viscosity modifying agents such as silico carbide waste [4]. Some of the waste materials reported in the literature with their w/p and dosages of admixtures are depicted in Table 2-1. Most of these materials require the inclusion of SP higher than 1.2% and not exceeding 5%. However, VMA was not added in most of these studies due to improved viscosity with the high SCMs content.

Table 2-1: SP and VMA dosages used in SCC in some studies

Waste material	Powder content (kg/m ³)	W/P	SP (%)	VMA (%)	Reference
Sandstone waste	600	0.33 and 0.35	1.35 - 1.65	0.10%	[81]
Glass powder	450	0.4	4 - 4.8	-	[82]
sugarcane bagasse ash and metakaolin	444.13	0.48	0.52 – 3.27	-	[83]
Oil palm shell and fly ash	520	0.31-0.33	1.65	-	[84]
Red mud, marble, and granite	500	0.37	1.4	-	[85]
Alum sludge	500	0.36-0.4	1.18-2.6	-	[86]

2.3 Performance of SCC Developed with Waste Materials

Apart from using conventional filler materials in SCC like slag and fly ash, recent studies exploited the feasibility of using various by-products such as red mud, palm oil fuel ash, waste ceramic powder, sugarcane ash, etc. When SCMs are incorporated in concrete at

optimum levels, several benefits can be attained, including significant reductions in harmful emissions, natural resources depletion, and total concrete costs, in addition to enhancement of fresh and durability characteristics [74]. The next two subsections present a review of the fresh and durability performance of SCC developed using different SCMs.

2.3.1 Studies on Fresh Properties of SCC

The workability of SCC is enhanced through the inclusion of finer mineral admixtures. In addition, the size and surface area of some waste materials replacing the cement is significantly affecting the workability and flowability. A recent study by Al-Oran et al. [87] was conducted in which six mixes were prepared (A0: OPC only, A1: 10% MK, A2-A5: 10% MK, and GGBS ranging from 15% to 30%). The mixes were prepared using a binder content of 500 kg/m³, w/b ratio of 0.38, and SP content between 1% to 1.5% of binder content. All fresh properties of concrete mixes were in the range specified by flowability criteria. The slump flow of A1 has shown a reduction of 1.5% and an increase of 16.7% and 9.1% in T_{500mm} and V-funnel, respectively. However, mixes A2-A4 had a maximum improvement of 4.5 % in slump flow diameter and a reduction in T_{500mm} and V-funnel by 10% and 25%, respectively. It was reported that the higher surface area of GGBS compared to MK and cement accelerates the hydration reaction which enhances the flowability and workability and increases the viscosity of the A2-A4 mixes. Moreover, it was recommended to replace the cement at a maximum of 25% GGBS to get the best workability properties.

In a recent study by Ren et al. [4], silicon carbide waste (SCW) that is generated in the photovoltaic industry was used to replace cement at ratios of 5,10,15, and 20%. It was

reported that there is an upsurge in superplasticizer dosages with the increase in SCW replacement level owing to its high surface area and water absorption. The range of slump flow of all mixes was between 650 and 700 mm. Moreover, the slump flow were having an increasing trend after 30 and 60 minutes due to the increase in SCW content. This was accredited to the limited hydration activity of SCW at an early age. The values of $T_{500\text{mm}}$ were between 2.3 and 6.4 sec, V-funnel was between 7.7 and 19.8 sec, and segregation resistance was between 6.3 and 17.3. The control mixture showed poor passing ability, filling ability, and sieve segregation due to aggregate accumulation. However, SCW generally enhanced these properties, and 10% and 15% replacement ratios were the best improvement.

Alsubari et al. [88] conducted a study on the use of treated palm oil fuel ash as a partial replacement of cement in SCC. Four mixes of 0%, 50 %, 60%, and 70% replacement ratios were developed with a w/b ratio of 0.35, and SP content was kept constant. The slump flow diameters ranged between 700 and 740 mm and were increasing with the increment in POFA content. Higher content of fine particles of POFA results in a reduction in LOI and an increase in paste volume to fill the gaps between the aggregate particles and this has improved the flowability. Shorter V-funnel times were obtained with the increase in POFA content due to high paste volume which enhances the filling ability and reduces the viscosity. A good passing ability was observed by conducting the L-box test in which the range was between 0.94 and 1. The segregation resistance values were increasing with POFA content, but they were below 15%, the limit specified by EFNARC guidelines.

Ranjbar et al. [56] investigated the fresh properties of SCC developed by replacing cement with palm oil fuel ash at 10, 15, and 20% by weight. The superplasticizer content was increased from 1.3% in the control mix to 1.8% in a mix of 20% replacement to achieve a slump flow of 680 ± 20 mm. There was a slight reduction in slump flow and workability with the increase in POFA content due to higher surface area which requires more water to ease the rolling and movement of particles. T_{50} and V-funnel times were in the range of 3-4.57 sec and 5.6-8.75 sec, respectively. The high content of POFA led to an increase in the viscosity of concrete as indicated by the results of slump flow, $T_{500\text{mm}}$, V-funnel, and L-box. The range of segregation resistance values was between 6.67 and 13.9. It was reported that cement can be replaced with 20% with no adverse effect on the fresh properties of SCC.

Safhi et al. [89] developed SCC using treated marine sediments (TMS) as a partial replacement of cement at three levels: 0%, 10%, and 20%. The three mixes were prepared using 0.4 w/c and cement content of 450 kg/m^3 . The results of workability showed good filling ability, passing ability, and stability. The slump flow measured for SCC mixes was 690 ± 5 mm. However, T_{50} readings were approximately 2 sec, and a V-funnel time was between 6 and 8 sec. Moreover, the L-box was in the range of 0.85 and 0.98 and all mixes had sieve segregation lower than 10%.

A recent study conducted by Ofuyatan et al. [55] in which SCC is developed using eggshell powder (ESP) and ground blast furnace slag (GBFS). Besides the control mix, three concrete mixes of each waste material were prepared to replace cement in proportions of 10%, 20%, and 30% by weight. The slump flow and $T_{500\text{mm}}$ were within the acceptable

limits (slump flow: 550-580, $T_{500\text{mm}}$: 5.19-6.05). These results were better in mixes with GGBFS compared with ESP. However, both materials attained the best workability at 10% replacement ratio. The V-funnel flow time had an increasing trend in both materials but higher values with ESP. The passing ability measured by the L-box test was improved in both materials reaching its maximum value at 30% replacement ratio.

Basu et al. [90] developed SCC utilizing sandstone waste (SS) as a partial replacement for cement. In this study, five concrete mixes were prepared (replacement ratios: 0% ,5%, 10% ,15% and 20%) with constant w/p ratio of 0.33. The utilization of SS in SCC increased SP demand to achieve a slump flow diameter of 800 ± 50 mm. The slump flow was reduced with the increase in SS content due to the high fineness of SS compared to cement and the reduction in binder content. This study reported an increase in the viscosity of SCC mixes due to uneven surface and angular form of SS particles. Segregation resistance was improved by increasing the replacement level of SS and this was attributed to the inert nature of SS that performs better compared to pozzolanic fillers.

A recent study by Larissa et al. [83] was conducted to produce SCC using high levels of sugarcane bagasse ash (SCBA) and metakaolin. Five concrete mixes in which cement was replaced with a blend of SCBA and metakaolin (REF, R30M20, R20M20, R30M10, and R30; R refers to SCBA and M refers to metakaolin). The demand for SP was increasing with the increase in mineral additive content. For instance, 3.27% by weight of binder was used as SP dose in R30M20, while only 0.52% was used in the control mix. The increase in demand is attributed to the high surface area of the particles and irregular shape and high porosity of SCBA particles. The diameters of slump flow ranged between 680 and 740 mm,

$T_{500\text{mm}}$ was between 1.17 and 1.63, V-funnel was between 4.86 and 7.66 and L-box was between 0.98 and 1.

Sheen et al. [80] employed stainless steel reducing slag (SSRS), a waste material of the steel industry, in developing SCC. OPC was replaced by SSRS from 0% to 50% with an increment of 10%. Water demand and workability of concrete mixes are affected by properties of materials used in these mixes such as shape, smoothness of surface, and particle size distribution. The slump flow has seen an improvement of up to 20% of SSRS replacement reaching a maximum diameter of 653 mm. The reduction in size and volume of the void was seen as a possible reason for this improvement and the SP effect in dispersing the particles and reduction of friction was also attributed to this enhancement. Beyond 20% SSRS substitution, there was a reduction in workability due to the higher surface area of SSRS and its irregular shape. The results of the $T_{500\text{mm}}$ test showed longer times in SSRS mixes compared to the control mix. The flow times in the V-funnel test have extended with the increase in SSRS content and reached a maximum of 20 sec in the last replacement.

In their major study, Subaşı et al. [72] investigated the usability of waste ceramic powder (WCP) as a filler material in SCC. Cement was substituted with WCP at proportions of 5%, 10%, 15%, and 20% by weight. Although the slump flow of WCP mixes was lower than the control mix, it was reported that flowability was slightly improved with the increase in the content of WCP. The range of slump flow was between 758 and 778 while $T_{500\text{mm}}$ was between 2.63 and 3.02. The passing ability measured by L-box has increased from 0.856 in the control mix to 0.955 in the last replacement level.

The study of higher replacement levels of 40-60% of three fly ashes (A, B, and C) in SCC was conducted by Matos et al.[91]. Superplasticizer content was reduced when cement was replaced by FAs due to its smooth texture and spherical shape. The values of slump flow were in the range of 675 and 715 mm. There was a general improvement in flowability, especially at replacement levels of 50% and 60%. The viscosity of SCCs was reduced in all types of fly ashes compared to the control mix. For instance, V-funnel was 19 sec in the control mix while it was in the range of 8-14 sec in mixes having FAs. This was explained by the texture and smooth shape of fly ash which reduces the friction between particles. Fly ash type C is more ground compared to type B which increases its surface area and resulted in a slight increase in viscosity.

Ghalehnovi et al. [85] conducted a study in which red mud (RM) was replaced the cement at varying levels (2.5%, 5%, 7.5%, and 10%). Three groups of concrete mixes were prepared, and each group has a filler with a constant content (LP: limestone powder, GP: granite, and MP: marble). Generally, a reduction in workability was observed for each group with the increase in RM content. The large surface area of RM and the high porosity of its particles led to an increase in water demand and slump flow reduction. Compared to the control mix, all groups at 2.5% RM showed a reduction in workability except the MP group which enhanced the flowability. The time of the V-funnel test is generally increased with increasing RM content for group LP and GP, but it was decreased for group MP. All concrete mixes exhibited larger values of V-funnel times which did not meet EFNARC guidelines. The finer particles of RM led to an increase in paste volume and higher viscosity.

Zareei et al. [92] published a recent article on SCC developed utilizing sugarcane bagasse ash (SBA) as cement replacement at ratios 5,10,15,20 and 25%. The slump showed a slight increase with the increase in SBA content. The density of SCC incorporated a higher amount of SBA was lowered due to the lower density of SBA compared with cement. The results of $T_{500\text{mm}}$ were in the range of 1.9-4 sec with the lowest value at 25% SBA replacement level. The addition of SCBA beyond 20% level led to degradation of workability and flowability. The filling ability and passing ability were enhanced with the inclusion of SBA.

Gupta and Siddique [93] investigated the fresh properties of SCC developed using copper slag (CS) as a replacement for fine aggregate. Slump flow exhibited an increasing trend and was in the range of 685-750 mm. The glass, smooth texture, and low water absorption of slag grains resulted in higher slump flow. For segregation resistance, SCC mixes were stable with values lower than 15% and increased with an increase in CS content. The values of $T_{500\text{mm}}$ were decreasing with a surge in CS content which indicates a lack of cohesion. It was reported a decline in V-funnel time from 7.55 to 5.01 sec as the CS content increased from 0 to 60%. Moreover, a strong correlation was found between V-funnel and $T_{500\text{mm}}$ with R^2 value of 0.9726. In terms of passing ability, the L-box ratio was in the range of 0.82 to 0.98 which indicates sufficient passing ability, especially in higher replacement levels.

Singh and Siddique [94] conducted a study on developed SCC in which iron slag used as a partial replacement of fine aggregate at levels of 0,10,25 and 40%. All SCC mixes exhibited a slump flow in the range of 680-780 mm. These values were decreasing as the

amount of iron slag increased due to the rough texture that contributed to the inter-particle friction. L-box and U-box values ranged between 0.84-0.91 and 32-35, respectively. A slight increase in V-funnel time with the higher slag content reaching a value of 13 sec compared with only 11 sec in the control mix. Therefore, a decrease in workability was noticed with the utilization of iron slag in SCC.

A recent study of the fresh properties of self-compacting concrete prepared with metakaolin (MK) and rice husk ash (RHA) was conducted by Gill and Siddique [18]. In their investigation, cement was replaced by MK in three levels (5,10, and 15%) while RHA was substituted for fine aggregate in only 10%. Both MK and RHA have no significant effect on the workability of SCC mixes. There was a slight increase in water demand with the increase in MK content and consequently, the SP dose increased up to 2% compared with 1.5% in the control mix. The small additions of MK with high surface area and round shape and fine size of RHA particles led to higher water demand. The slump flows, U-box, L-box, and V-funnel of these mixes were between 680-755, 8-12, 0.8-0.9, and 8-10, respectively. The results showed a slight increment in U-box and V-funnel with the increase in MK content.

2.3.2 Studies on Durability Properties of SCC

Basu et al. [81] investigated the durability properties of six SCC mixes developed with sandstone slurry (SS) as a partial replacement of cement (0%,5%,10%,15%,20%,25% and 30%). The results showed an increase in sorptivity with the increase in SS content due to higher fineness and specific surface area of SS which caused a decrement in binder content. The porosity of SCC mixtures was increasing with SS content. The highest depth of

carbonation was measured in a sample of 30% SS. The results of chloride ion penetration showed an increase with the increment of SS content. For instance, replacing cement with 30% SS resulted in 12% higher chloride penetration compared with the control sample and a maximum depth of penetration of 17.5 mm. This was attributed to the increment in porosity because of using porous SS that permits the penetration of chloride ions into the concrete sample. The losses in compressive strength due to freeze-thaw cycles were 0%, 2%, 4.5%, 6.8%, 7.3%, 8.6% and 9.5% for the six samples.

In a recent study, Silva and Delvasto [95] studied the performance of SCC prepared with the residue of masonry (RM) in two sulfate solutions (5% Na_2SO_4 and 5% MgSO_4). Minor deterioration of concrete mixes incorporated RM was observed compared with the control mix due to the smaller content of calcium hydroxide and the pozzolanic reaction of RM. A mass gain for all mixes was noticed at 28.90 and 180 days except the control mix submerged in Na_2SO_4 solution. However, after 360 days, there was a reduction in mass due to the formation of expansive products such as gypsum and ettringite. The compressive strength has seen an increase up to 180 days of immersion in both solutions due to the formation of gypsum and secondary ettringite.

Laidani et al. [96] evaluated the durability performance of two different types of calcined bentonite (CgB and CsB) as a partial replacement of OPC in SCC. It was reported that CgB and CsB can be incorporated up to 25% and 15% respectively for porosity to be lower than the control mix. This was attributed to the finer particles of CB that fill the porous system and the production of additional C-S-H gel. However, the porosity of mixes of higher CB content was reduced due to deficiency in hydration which absorbs more water and produces

a high viscous concrete. The concrete mixes with replacement levels of CB below 20% showed lower electrical charges passed at 90 days which indicates a higher resistance to chloride penetration, and they were classified as low chloride permeability. The reduction of porosity due to pozzolanic reaction is the main reason for the improvement in chloride-ion penetration. The results of gas permeability showed an improvement in permeability up to 20% and 10 and 15% mixes were the best permeable mixes. The filling of voids during the hydration and the filling ability of CB led to this enhancement in gas permeability.

Ghalehnovi et al. [85] investigated the electrical resistivity, sulfuric acid resistance, and water absorption of SCC mixes prepared with red mud (RM) as a replacement of cement with Limestone powder (LP), granite (GP), and marble (MP) were added in a constant content for each group. The water absorption for all groups showed an increase in water absorption with an increase in RM content due to the presence of pits and folds on the surface of RM. However, water absorption was slightly higher in MP mixes reaching the maximum value. All concrete mixes showed a higher electrical resistivity compared to the control mix. This is related to the finer sizes of RM particles. The control sample exhibited the highest durability against sulfuric resistance with only a 9% reduction in compressive strength. However, a concrete mix of 10% RM with GP showed the lowest resistance with 22% reduction in compressive strength. The higher voids in RM led to a general reduction in compressive strength in all groups.

Aarthi and Arunachalam [97] in a recent article conducted a study on using Granite Sawing Waste (GSW) in fiber-reinforced self-compacting concrete. It was reported that water

absorption is initially slightly reduced due to the inclusion of finer GSW then it increased. Moreover, at 5% and 10%, water absorption was lowered than the control mix in all fiber additions due to the continuous process of hydration and reduction in porosity compared to these replacement levels. The rapid chloride permeability values in mixes up to 20% of GSW were lower than the control mix due to the dense condition of concrete with GSW. The minimum chloride penetration was observed at 10% of GSW without any fibers. All mixes showed a very low value of RCPT. The weight loss due to acid attack was reduced in mixes incorporated with GSW and fibers compared to the control mix. The inclusion of GSW showed an improvement in sulfate resistance compared to control concrete. For instance, a reduction of 36.34% in compressive strength was noticed for 20% GSW while 48.83% happened in the control mix. The reactivity of GSW which improves the bonding between cement and aggregates could be the reason behind this improvement.

Ranjbar et al. [56] conducted a study on SCC performance by replacing the cement with palm oil fuel ash (POFA) at 10, 15, and 20% by weight. The inclusion of POFA resulted in a reduction of initial surface water absorption compared to the control mix and the minimum flow rate occurred at 20% POFA at all ages. This was attributed to the small particle sizes of POFA and lower pore networks in the concrete matrix. The lowest reduction in compressive strength due to 5% MgSO_4 was found at 20% replacement level with 7.2% due to the pozzolanic reaction of POFA. Acid attack due to 3% HCl also showed a similar trend with the highest loss in compressive strength occurring at 20% POFA. The pozzolanic reaction of POFA and conversion of Ca(OH)_2 to additional C-S-H led to densification of the matrix and enhancement of durability properties.

Sheen et al. [80] incorporated stainless steel reducing slag (SSRS) in SCC as a partial replacement of cement from 0% to 50% with an increment of 10%. The durability performance of this concrete was evaluated using water absorption, electrical resistivity, and sulfate resistance. The water absorption was in the acceptance range of (3-5%) with the lowest value at 30% SSRS replacement level. The volume of voids was lowered with the increase in SSRS content and SSRS has finer particles compared to OPC which supports the improvement in water absorption. The highest and lowest electrical resistivity (ER) of 12.33 k Ω .cm and 7.08 k Ω .cm was attained at 10% SSRD at 91 days. With the increase in SSRS content, it was reported a general decline in ER compared to the control mix. Increasing the SSRS substitution resulted in higher weight loss and damage due to sodium sulfate attack. However, only 10% of SSRS showed better resistance compared to the control mix.

Gupta and Siddique [52] studied the water absorption, rapid chloride permeability (RCPT) and sorptivity of seven SCC mixes in which sand is being substituted with copper slag (CS) in ratios 0-60%. All concrete mixes exhibited water absorption of less than 6.5% at all curing ages. The lowest water absorption was found at 20% CS due to improvement in packing of aggregates and reduction in pores. Further addition of CS has slightly increased the water absorption due to the rise in the amount of free water that created new voids. All concrete mixes were classified under the category of very low to low in RCPT results at 28 days. RCPT exhibited a substantial reduction in the charges passed up to 30% CS at all ages. This was attributed to the lower porosity of up to 30% CS. The effect of copper slag up to 30% was also apparent in the lower values of sorptivity. Further reduction of the

values .of sorptivity was observed with the progress of time due to the formation of denser concrete.

Gill and Siddique[18] published a recent study of the durability performance of SCC developed by incorporating metakaolin (MK) as a partial replacement of cement while fine aggregate was replaced by 10% of rice husk ash (RHA). A reduction in water absorption and porosity of mixes incorporating MK and RHA as compared with the control mix. This was attributed to the pozzolanic reaction that promotes the additional production of C-S-H gel. However, there was an increment in porosity and water absorption with higher replacement levels of MK. This was associated with a higher surface area of MK and RHA and higher water demand in which there was a gradual decrease in workability. All concrete mixes exhibited better resistance to sulfate solutions compared with the control mix due to lower alumina content in the blended MK and RHA. Furthermore, the compressive strength loss due to sulfate attack showed the highest loss in the control mix and the minimum was in the mix of 10% MK. All concrete mixes were in the category of low and very low as per ASTM C1202. Regarding the rapid chloride permeability results, a significant drop in charges passed at the age of 28 days for mixes with MK. For instance, the concrete mix with 15% MK had a 22% drop in chloride penetration as compared with the control mix.

A recent study by Ren et al. [4] was conducted to study the durability performance of silicon carbide waste (SCW) as a partial substitution of cement in SCC. Incorporating SCW led to a reduction in initial water absorption due to lower interconnected capillary pores and the filling effect of SCW. However, final water absorption was found to be higher compared to the control mix in replacement levels below 10%. The results of chloride

permeability showed a higher reduction in the total charges of mixes containing SCW compared with the control mix. The microstructure densification explained such improvements in durability properties of SCC made with SCW.

Based on the literature of some durability studies, some conclusions can be inferred:

1. Most of the studies showed an improvement in durability properties up to a certain replacement level, then there was a decrement in some of these properties.
2. Two common features of some SCMs blended in SCC; they have smaller particle sizes compared with cement and they exhibit pozzolanic reaction that accounts for augmentation in some hardened properties.
3. Most of the improvement in durability properties is attributed to the pozzolanic reactivity of the substituted materials besides their smaller sizes and higher surface areas.
4. Higher replacement levels of cement by SCMs are not recommended in some of these studies due to clear degradation of durability properties, particularly chemical resistance.

2.4 Heat of hydration of Cementitious Materials

During the hydration of cement, around 120 Cal/g of energy is evolved in an exothermic process [98] which is known as the heat of hydration. In concrete elements, the generated heat creates a temperature difference between the core and the surface of the concrete element which causes thermal stresses and cracks even without external restraint [99]. Therefore, the service life of structures is reduced due to penetration of various harmful

substances leading to corrosion and deterioration of reinforcement. The heat of hydration has a major influence on early-age properties such as workability, setting time, hardening behavior of cement, and also on durability properties of concrete. The heat evolution process relies on many factors such as material fineness, w/b ratio, ambient temperature, the chemical compositions of cement, mixture proportion, chemical and mineral admixture, and curing conditions of concrete [19,100]. The heat of hydration can be controlled by several methods which include cooling the aggregate, cooling the mixing water, using special types of cement, reducing cement content, and using supplementary cementitious materials [101]. It is reported that early-age heat of hydration is smaller when SCMs are blended with cement compared with Portland cement alone [101]. The incorporation of mineral admixtures such as in SCC does not offer only economic benefits but also contributes to the reduction of the heat of hydration [13,102].

A recent study by Moghaddam et al. [103] has examined how fly ash fineness affects the flow and hydration of cement pasts blended with a different fineness of fly ashes. In this study, cement is being replaced partially by fly ashes of different fineness at 20% and 40% by weight of cement. These fly ashes were having an average particle size of 17.4, 11.3, and 5.7 μm , respectively. The results of the isometric calorimetry test showed a decrease in cumulative heat evolution due to the increase in fly ash content. However, the heat generated by the coarse grade fly ash of 17.4 μm was higher than the finer one of 5.7 μm . The SEM, XRD analysis, and compressive strength results confirm the denser microstructure in the finer grade fly ash. The rapid pozzolanic reactivity of the finer grade fly ash is contributed to the denser microstructure.

The increased quantity of C_3S and C_3A in the composition of cement is playing a vital role in increasing the heat of hydration [98]. However, lower heat is generated for supplementary cementitious materials due to a slower pozzolanic reaction compared with cement hydration [98]. The heat of hydration of another pozzolanic material, palm oil fuel ash, has been investigated by Awal and Shehu [104]. The peak temperature reached by the OPC mix was 57.3 °C, whereas temperatures of 46.8 and 44.2 °C were recorded for 50% and 60% of POFA mixes after 24 hours of casting. The performance of POFA concrete mixes in reducing the heat of hydration was better than the control mixture (100% OPC).

A. Gheni et al. [105] studied the heat of hydration of mortar mixes prepared with different proportions of rubber-fiber powder (RFP) and sand during a period of 65 h after the addition of water to the mixture. The results of the heat flow curve show that the normalized heat flow per gram of cement has not significantly changed due to the increase in sand content. However, temporal variation was noticed with the addition of RFP. This industrial by-product requires additional heat to reach a certain temperature because of its higher specific heat compared with cement. Peak heat flow of mixes having 15, 20 and 35% RFP was lowered by 20.7, 25.5, and 29.4%, respectively. Moreover, the induction period was increased by 24.3, 31.5, and 78.5% for the three mixes.

Kamali and Ghahremaninezhad [106] investigated the hydration of three cement pastes partially replaced with two types of glass powders (20% GP1, 20% GP2) and class F fly ash (20% FA) using a semi-adiabatic calorimeter. A fixed w/b ratio of 0.4 was used to prepare the paste samples and the temperature was recorded every one minute for 24 h. Figure 2-1 shows that peak temperature and rate of hydration were lowered in modified

cement pastes compared to the control mix. However, 20% GP1 had higher peak hydration compared to 20% GP2 and FA. This contributed to the high surface area of GP1 that provided more nucleation sites for hydration to occur.

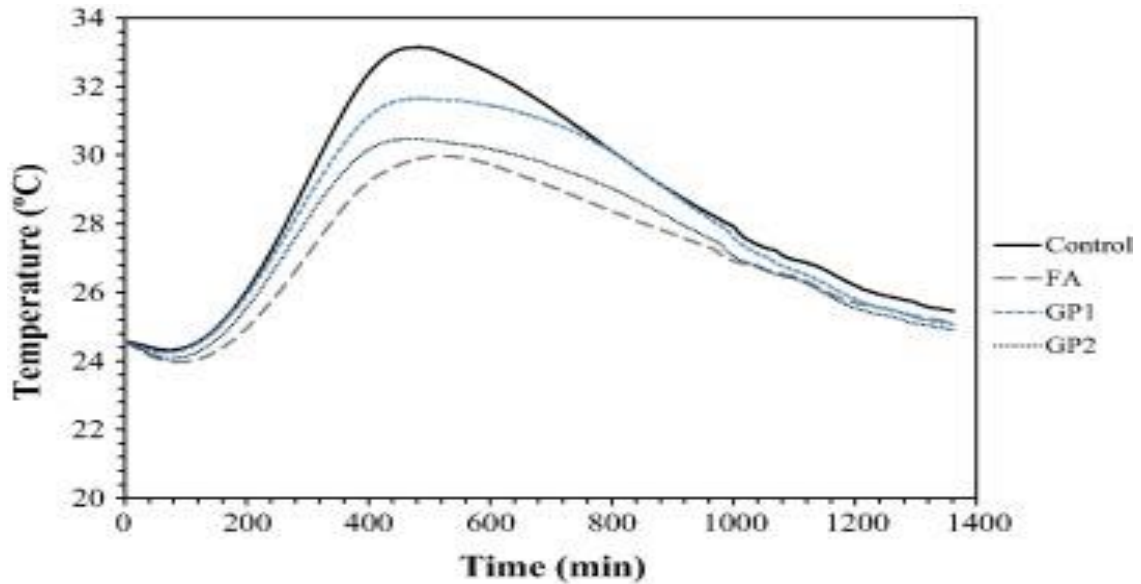


Figure 2-1: Temperature evolution curves for control and modified samples [106]

Wu et al. [107] used an isothermal calorimeter to study the influence of silica fume on heat of hydration measured for 60 h. Five concrete mixes of constant w/b of 0.18 were prepared in which cement is replaced with silica fume in a proportion of 0-25%. The heat evolution results showed a reduction in the dormant period from 12 to 9 h with the increase in silica fume content. Moreover, a mixture of 25% SF showed an accelerated hydration peak. It was also reported that the heat generated evolved quickly in mixes incorporated SF compared to control and this is attributed to additional C-S-H formed.

2.5 Summary of Literature Review and Research Gap

The previous research works demonstrate the importance of incorporating SCMs in concrete to achieve sustainability and improve the fresh and hardened properties of SCC. The fresh properties are influenced by the physical properties of SCMs like specific gravity, specific surface area, size and shape of particles, and particle size distribution. The increase in water demand of some SCMs necessitates the inclusion of chemical admixtures such as superplasticizers in suitable proportions to enhance the rheology and workability. The strength and durability performance of SCC blended with SCMs are dominated by some factors like the physical and chemical properties of SCMs, the porosity of mixes, and pore interconnectivity. Finer SCMs exhibit some degree of pozzolanic reactivity which contributes to the formation of additional gel products like secondary C-S-H gel and the improvement of hardened properties. However, the increase of SCMs content may degrade the properties due to the dilution effect and the increase of pores and voids in the mixes. The heat of hydration of concrete was reported to decrease with the inclusion of SCMs which makes the concrete less susceptible to thermal cracking and deterioration against aggressive agents.

The increase in the production and demand of steel alloys will inevitably generate a considerable amount of by-products like slag and SMF. These materials are collected and disposed in the landfills as previously mentioned. Accordingly, the utilization of these materials in various applications presents a feasible way of safe disposal of these wastes that would otherwise need huge areas for their enormous volumes. Hence, recent studies were published on the synthesis of alkali-activated binders using SMF and the study of

some properties like strength, flow, setting time, and durability. There are no available durability studies of concrete where SMF is replacing part of the cement. Therefore, this research focuses on the thermal and durability characteristics of SCC mixes prepared by substituting cement with SMF (10-70% by weight).

CHAPTER 3

METHODOLOGY OF RESEARCH

3.1 Introduction

In this chapter, the materials and chemicals utilized in this research are listed and demonstrated along with their main characteristics and sources. Moreover, the mix design approach, design parameters, and weight proportions of trial mixes are presented. The selection of the accepted 8 concrete mixes was based on EFNARC flowability criteria [108]. Furthermore, the test methods to evaluate the durability performance of concrete mixes, such as electrical resistivity, rapid chloride permeability, acid attack, and sulfate resistance are described in terms of test procedures, equipment, exposure solutions, and duration of the tests.

3.2 Materials Utilized in SCC Mixes

The materials and chemicals utilized in the experimental program are cementitious materials (Portland cement, and silico-manganese fume), coarse and fine aggregates, water, superplasticizer, and viscosity modifying agent.

3.2.1 Cementitious Materials & Characterization

1. Ordinary Portland Cement

ASTM C150 [109] Type I Portland cement of specific gravity of 3.15 was utilized as the main binder. This cement was sourced from a local supplier and kept in a dry condition.

2. Silico-manganese Fume

This industrial waste material was obtained from a local Ferro Alloy company, SABAYEK, located in Jubail, Saudi Arabia. This plant is well-known as the only ferroalloy producer in the GCC region, and its products include high carbon ferromanganese alloys and Ferro silico-manganese. In this local plant, SMF is collected through installed baghouses and stored in dedicated storage rooms before final disposal in landfills [33,38].

Table 3-1 shows the chemical composition and the physical properties of cement, LSP, and SMF. The collected SMF raw material from the same silico-manganese plant may vary greatly in its chemical and physical characteristics from one batch to another, so characterization and analysis of its properties are crucial. Compared with cement, SMF is very rich in MnO and K₂O (sum = 61.35%) but has a lower content of CaO and comparable content of SiO₂. SMF is considered acidic when evaluating its basicity coefficient and hydration modulus (<1) according to [33,40]. Bruker Benchtop D2 phaser XRD (Cu-K α radiation, 20 mA and 30 kV) was used to obtain the mineralogy of the raw materials as shown in Figure 3-1. XRD spectrum of SMF showed prominent crystalline phases such as quartz, sylvite, wollastonite, bixbyite, eckermannite, and manganese ferrite while the main

peaks presented in Portland cement are alite, belite, gypsum, tricalcium aluminate (C_3A) and tetra calcium aluminoferrite (C_4AF).

The morphologies and surface textures of cement and SMF were examined using JEOL JSM-6610LV SEM as shown in Figure 3-2. It was observed that Cement particles are rough, angular, and irregular in shape while SMF particles consist of smooth agglomerated spheres which contribute to the enhancement of workability of SCC mixes due to the ball-bearing effect. A closer look at the dispersion of SMF particles revealed that the bigger particles are well-distributed within the smaller particles. SMF has a higher specific surface area ($10.10 \text{ m}^2/\text{g}$) than cement ($0.33 \text{ m}^2/\text{g}$) according to the BET method.

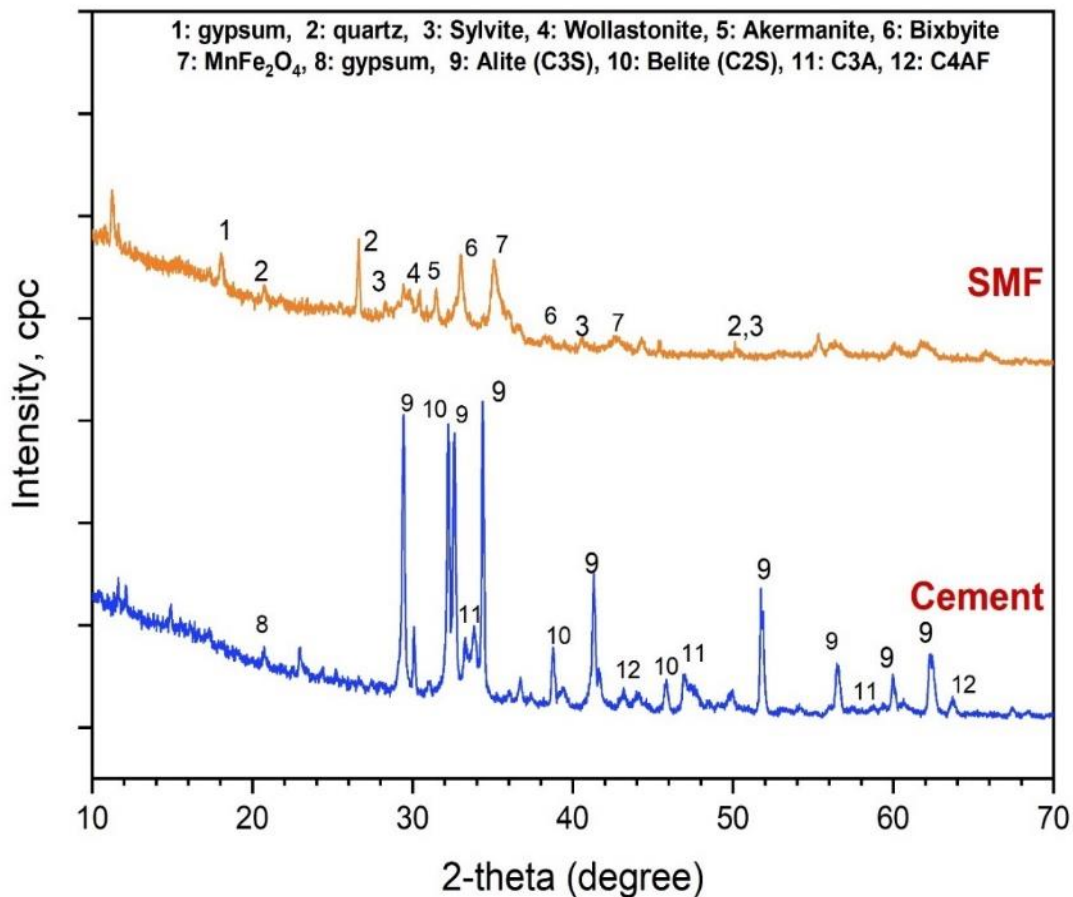


Figure 3-1: XRD spectra of SMF and Cement

Table 3-1: Chemical and Physical Properties of raw materials

Material	Cement	SMF
SiO ₂	22.00	19.50
Al ₂ O ₃	5.64	1.46
Fe ₂ O ₃	3.80	3.35
CaO	64.35	7.24
MgO	2.11	5.71
SO ₃	2.10	6.24
K ₂ O	0.36	16.62
Na ₂ O	0.19	1.73
MnO	-	35.42
Appearance	Grey	Brownish
Loss on ignition	0.70	5.82
Specific gravity	3.15	2.88
Specific surface area BET(fineness), m ² /g	0.33	10.10

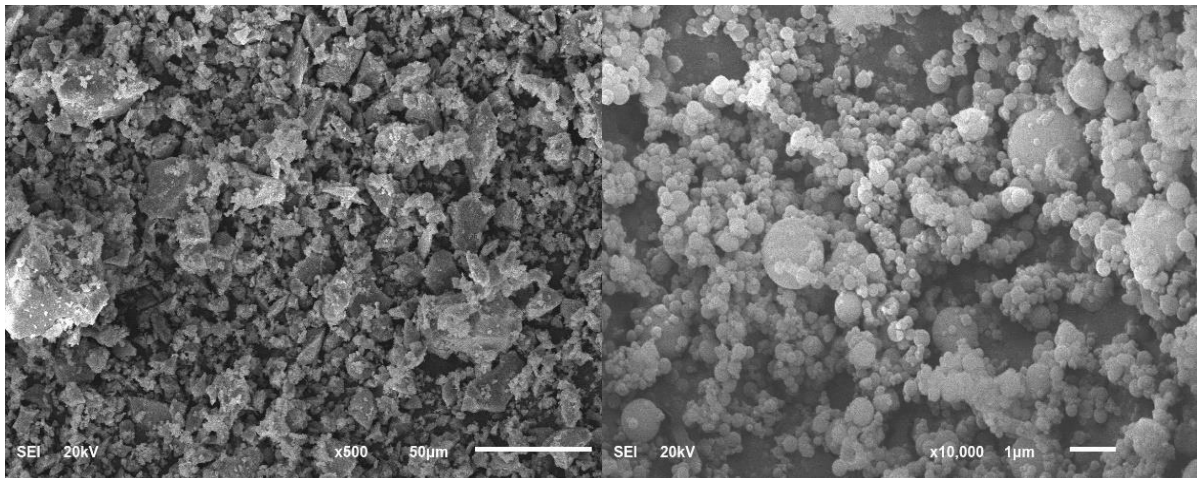


Figure 3-2: SEM of Cement (left) and SMF (right)

3.2.2 Fine Aggregates

Locally available sand was utilized as a fine aggregate with SSD water absorption and specific gravity of 0.4% and 2.55, respectively [110]. Table 3-2 shows the grading of fine aggregates used in this study. Part of the sand in this study was replaced by locally available limestone of specific gravity of 2.67. This limestone was obtained from a quarry in the Eastern province of KSA and sieved before using it in the experimental program.

Table 3-2: Fine aggregates grading [110]

Sieve #	Size (mm)	% Passing
4	4.75	100
8	2.36	100
16	1.18	100
30	0.6	76
50	0.3	10
100	0.15	4

3.2.3 Coarse Aggregates

The crushed limestone in a local quarry was used to supply coarse aggregates. The maximum size of these aggregates is 19 mm. Moreover, these aggregates have a density of 2.64 and water absorption of 1.4% [110]. The grading of coarse aggregates is shown in Table 3-3.

Table 3-3: Coarse aggregates grading [110]

Sieve size (mm)	% Passing
19	100
12.5	65
9.5	30
4.75	10
2.36	0

3.2.4 Superplasticizer

Superplasticizers which are known as high-range water reducers are used to maintain the workability during the production of concrete mixes with a lower water/binder ratio. Therefore, workability increases without the addition of SP to the mixes.

In this research work, Master-Ease 3741, a product of BASF company, was used as a superplasticizer to enhance the flowability and rheology of SCC. This product is based on Poly Aryl Ether (PAE) polymers that are mainly made for ready-mix concrete. The technical data of the used SP is depicted in Table 3-4. The dosage of SP is added carefully to SCC mixes based on the workability needs to minimize disturbing the homogeneity of the mixes or degrading the hardened properties.

3.2.5 Viscosity Modifying Agent

The incorporation of Viscosity modifying agents in SCC mixes helps to improve the viscosity and reduce the segregation. Master Matrix 110, a new chemical admixture produced by the BASF company, was utilized as a stabilizer to reduce the bleeding and segregation that might occur due to higher fines content. VMA was only used in the control

mix to control the apparent bleeding using a dosage of 0.15 % by weight of the binder content.

The technical properties of this product as provided by the manufacturer are shown in Table 3-4.

Table 3-4: Technical data of SP and VMA

Property	SP	VMA
Appearance	light brown	Light to dark brown
Specific gravity	1.08 to 1.16	1.009
PH-value	5 to 7	9.5
Chloride content	“Chloride free”	< 0.1 %

3.2.6 Mixing Water

Potable tap water that has no chemicals was used in the casting and curing of SCC mixes and specimens. In addition, distilled water was used in the preparation of exposure solutions with durability properties such as rapid chloride permeability.

3.3 Design of SCC Trial Mixes

The trial investigation was conducted on 11 mixes starting with the control mix of cement content only. Then, cement was replaced by SMF at proportions of 10% to 100%.

3.3.1 Mix design Approach

The weight proportions of the ingredients were obtained using the absolute volume method. Using a fixed cement content, coarse to fine aggregate ratio and w/c ratio, the mass of total

aggregates was determined using the absolute volume method. The derivation of the aggregate masses is shown below:

Consider the equation of the absolute volume:

$$V_{TA} + \sum_i V_i = 1 \quad (1)$$

where V_{TA} represents the total volume of aggregates and V_i is the volume of each constituent in the concrete except the aggregates.

Then, the volume of total aggregate is expressed as mass over density in equation (2) in which the volume of each material other than aggregate is known.

$$V_{TA} + \sum_i \frac{m_i}{\rho_i} = 1 \quad (2)$$

Then the volume of each aggregate (coarse aggregate + fine aggregate) is summed to obtain the volume of the total aggregate as in equation (3) :

$$V_{TA} = \frac{m_{CA}}{\rho_{CA}} + \frac{m_{sand}}{\rho_{sand}} + \frac{m_{limestone}}{\rho_{limestone}} \quad (3)$$

To obtain the mass of total aggregate, equation (3) is substituted into equation (2) and each aggregate mass is expressed in terms of total aggregate mass. Finally, the amount of water is corrected using the absorption percentage of each aggregate.

3.3.2 Key Parameters of the Trial Mixes

The key parameters considered in the design of trial mixes are shown in Table 3-5. A constant binder content, coarse/total aggregate ratio, and water to binder ratio of 500 kg/m^3 , 0.5 and 0.4, respectively. The dosages of the chemical admixtures were varied in the trial experiments to produce a self-compacting concrete conforming to EFNARC [108] specifications and flowability criteria.

Table 3-5: Mix design parameters

Cement Content	(0-500) kg/m^3 (Variable)
Total mineral SMF	(0-500) kg/m^3 (Variable)
Total Binder	500 kg/m^3
Water/Cement	0.4
CA/TA	0.5
Sand/TA	0.5
LSP/FA	0.2
SP Dosage	(0.8-1.1) %
VMA Dosage	(0-0.15) %

3.3.3 Weight Proportions of SCC Trial Mixes

Table 3-6 presents the weights of the different materials in SCC trial mixes. SP dosages were started with 0.8% in the control mix and then gradually increased to enhance the flowability of the concrete. However, VMA was only used as a stabilizer in the control mix which had clear bleeding. The next mixes show little or no segregation in which VMA was not employed.

Table 3-6: Weight proportions of trial mixes in kg/m³

Materials		SMF0	SMF10	SMF20	SMF30	SMF40	SMF50	SMF60	SMF70	SMF80	SMF90	SMF100
Cement		500	450	400	350	300	250	200	150	100	50	0
SMF		0	50	100	150	200	250	300	350	400	450	500
Water		200	200	200	200	200	200	200	200	200	200	200
Coarse Aggregate	1/2	82.5	81.5	80.5	79.4	78.4	77.4	76.4	75.4	74.4	73.4	72.4
	3/8	330.0	325.9	321.8	317.8	313.7	309.7	305.6	301.6	297.5	293.5	289.4
	3/16	288.7	285.2	281.6	278.1	274.5	271.0	267.4	263.9	260.3	256.8	253.2
	3/23	123.7	122.2	120.7	119.2	117.7	116.1	114.6	113.1	111.6	110.0	108.5
Dune Sand		659.9	651.8	643.7	635.6	627.5	619.4	611.2	603.1	595.0	586.9	578.8
LSP		165.0	162.9	160.9	158.9	156.9	154.8	152.8	150.8	148.8	146.7	144.7
SP %		0.8	0.8	0.83	0.87	0.9	0.92	0.96	0.98	1.03	1.06	1.1
VMA %		0.15	0	0	0	0	0	0	0	0	0	0

3.4 Tests of Self-compactibility on Trial Mixes

Five tests were performed on each trial mixture to determine their self-compactibility: slump flow, U-box, L-box, V-funnel, and sieve segregation resistance as shown in Figure 3-3 and Figure 3-4. Bleeding and segregation were observed during measuring slump flow.



Figure 3-3: Equipment used in measuring fresh properties



Figure 3-4: Measurement of fresh properties in lab

3.5 Selection of Accepted SCC Mixes

The selection of accepted concrete mixes was based on EFNARC criteria [108] as illustrated in the previous work done by our research member, Qawah et al. [111]. In this regard, the values of some tests like V-funnel in SMF 80, SMF 90, and SMF 100 were reported to be below the minimum requirement of the European criteria. Therefore, the

previous mixes were discarded and only the remaining eight mixes were further considered for mechanical and durability studies.

3.6 Details of Test Standards and Specimens

Table 3-7 summarizes the strength and durability tests conducted in this research with details of specimen type and size, test age, and exposure solution.

Table 3-7: Summary of test methods

Property	Specimen type	Specimen shape and size	Test age	Exposure solution
Compressive strength	Concrete	Cube: 100-mm	7,14,28 and 90 days	-
Acid Attack	Concrete	Cube: 50-mm	0,3 and 6 months	2% H ₂ SO ₄
Sulfate Resistance	Paste	Cube: 25-mm	0, 3 and 6 months	2.1% MgSO ₄
	Paste	Prism: 25 × 25 × 285 mm	At regular periods	2.1% Na ₂ SO ₄
Chloride Diffusion	Concrete	Cylinder: 75× 150 mm	3 months	5% NaCl
Rapid Chloride Permeability Test	Concrete	Cylinder disks: 75× 50 mm	After 28 days of water curing	Two cells filled with NaCl and NaOH
Electrical Resistivity	Concrete	Cylinder: 75× 150 mm	After 28 days of water curing	Water exposure
Thermal Conductivity	Concrete	Cylinder disks: 50× 25 mm	After 28 days of curing	-
Reinforcement Corrosion	Concrete	Cylinder with 12 mm bar: 75× 150 mm	After 28 days of water curing	5 % NaCl

3.7 Tests on the Selected Mixes

3.7.1 Heat of Hydration

In this test, the temperature evolution of paste samples was measured at early ages. A portable data logger (TDS-303) and a thermocouple of type K were utilized to detect and

record the temperature of paste samples for approximately 24 hours which begins immediately after the addition of water to the powder.

Initially, paraffin wax is used to insulate the internal part of a plastic vial. Then, an external coating is applied to the plastic vial. After that, a paste sample is prepared in which a 0.45 w/c ratio was used. The time of adding water to the powder was recorded with the initial temperature from the data logger. Then, approximately 70 g of paste sample was poured into the coated plastic vial. This weight was maintained in all mixes to get comparable results. This step is followed by inserting the thermocouple in the middle of the plastic vial and closing it tightly with a screw cap. A duct tape was applied to the top of the screw cap to hold the thermocouple and screw cap in place.

Following sample preparation, the vial was inserted into a chamber that is surrounded by a thick polystyrene container as shown in Figure 3-5. This container is protected by a wooden box. Then, small polystyrene foam pieces were used to encapsulate the plastic vial for more heat insulation as shown in Figure 3-6.



Figure 3-5: Preparation of specimen and placing it inside the insulating chamber

The thermocouple embedded in the paste sample was connected to a data logger to measure the temperature every 7 minutes from the first reading. The readings continued for 24 hours which were taken for generating heat of hydration graphs. The experimental setup is shown in Figure 3-7.



Figure 3-6: Insulating chamber with polystyrene foam



Figure 3-7: Experimental setup used in Heat of hydration

3.7.2 Rapid Chloride Permeability Test (RCPT)

RCPT test of SCC specimens was determined in accordance with ASTM C1202 [112]. The test started with cutting out three disks (75 dia. $\times$ 50 mm thickness) from each of 75 $\times$ 150 mm cylindrical specimen. Then, an epoxy coat was prepared and applied carefully to the curved surface of the disks as presented in Figure 3-7. The disk specimens were situated in a vacuum desiccator for 4 hours and this was followed by keeping them saturated in water for about 18 hours as shown in Figure 3-8.



Figure 3-8: Specimens preparation for RCPT

After that, the saturated disks were fitted between two half cells in which a potential of 60 V was maintained during the testing period. Two solutions were used to fill each cell: 3% NaCl and 0.3 M of NaOH. The adjusted coulombs or charges were recorded in the software after the 6 hours of testing. The experimental setup of the RCPT test is depicted in Figure 3-9.



Figure 3-9: RCPT test setup

3.7.3 Electrical Resistivity

In concrete, the estimated probability of reinforcement corrosion is related to its measured electrical resistivity. Moreover, porosity and size of pores play a vital role in changing the electrical resistivity. In this study, the electrical resistivity of 75×150 mm cylindrical specimens was evaluated using a digital ground resistance apparatus having probes connected to spongy terminals according to the two-probe Wenner method. The terminals were kept moist in each measurement to have good electrical contact with the concrete ends. The concrete specimen was maintained in a contact with wet spongy terminals using the mechanical part. The length and diameter of each concrete specimen were initially recorded. Then, specimens were placed in a water bath for 48 hours as shown in Figure 3-10. The specimens were taken out of the water to measure their resistance along with weight. The initial resistance was taken as saturated surface dry to have the first moisture content. After that, they were kept in the lab under atmospheric conditions to lose weight gradually. The specimens were monitored continuously by measuring first their weights.

The resistance was measured when there was a drop in weight to have several readings. Finally, the electrical resistivity and moisture content were calculated, and a graph was drawn showing their relationship. Moreover, the electrical resistivity was determined at 3% moisture content to compare the different concrete mixes. Figure 3-11 shows the experimental setup used in measuring the electrical resistivity of cylindrical specimens.



Figure 3-10: Specimens immersed in water for 48 hours



Figure 3-11: Setup for electrical resistivity

3.7.4 Sulfate Resistance

Two types of paste specimens were prepared for sulfate investigation: 1-inch cubes and $25 \times 25 \times 285$ mm prisms. The specimens were immersed in saturated solutions of Na_2SO_4 and MgSO_4 .

i. Length change

The prism specimens were taken after 28 days of water curing to measure their initial length. Then, lengths were measured at continuous intervals. The apparatus of this test consists of an LVDT attached to a portable data logger (TDS-301) as shown in Figure 3-12. The frame has a smooth base which keeps the specimen held in a vertical position during the test. Before measuring the length of a prism, a standard rod of a known length was used for calibration. The length change or difference between the length of the standard rod and the measured prism data logger was shown on the data logger in each measurement.



Figure 3-12: Setup used for measuring the expansion of prism specimens

ii. Compressive strength

Small cubes of 25 mm were tested for each mix using MATEST compressive testing machine with a load capacity of 500 kN and the applied loading rate was 2 kN/s. The compressive strength was computed based on the measured maximum force at failure. Then, the average of the readings was reported in this test. Figure 3-13 presents the compressive strength machine used to test the cubes.



Figure 3-13: Compressive strength machine

3.7.5 Acid Attack

In this test, 50-mm cubes were prepared and immersed in 2% sulfuric acid solution for a period of 3 and 6 months as shown in Figure 3-14. The compressive strength of cubes was measured and the visual examination of the effect of acid on specimens was observed after the end of the exposure period.



Figure 3-14: Preparation and testing of specimens exposed to acid solution

3.7.6 Reinforcement Corrosion

To assess the corrosion status of the concrete specimens, 3 cylindrical specimens of each mix of size 75×150 mm were prepared with a 12-mm steel bar placed in the middle. Then, cement paste was used to coat the 12-mm bars and the epoxy coating was applied at the concrete-air interface and bottom of the specimen. After that, the samples were cured in water before they were partially placed in a plastic container filled with 5% NaCl solution. Then, the evaluation of reinforcement corrosion was done by measuring the corrosion potentials and the corrosion current density.

i. Corrosion potential

A saturated reference electrode connected to a digital voltmeter was used to measure the corrosion potentials at continuous intervals as shown in Figure 3-15. The positive and negative terminals of a digital multimeter dipped in the chloride solution were connected to the reference electrode and the steel bar, respectively. The reading on the voltmeter represents the potential difference between the two terminals. The corrosion status is

determined based on corrosion potential reading according to ASTM C876 [113]. For example, if the measured potential of the reinforced concrete specimen reaches the limit of -270 mV, then its condition is classified as a high risk of corrosion with a 90% probability of initiation of corrosion.



Figure 3-15: Measurement of corrosion potential

ii. Corrosion current density

This test is used to indicate the rate of progress of reinforcement corrosion. Therefore, it's considered an important test in predicting the remaining service life and determining the extent of the corrosion damage to help in taking decisions on possible repair and rehabilitation works. The linear polarization resistance method was used to obtain the corrosion current density using three electrodes. Figure 3-16 demonstrates the setup used in measuring corrosion current density. The working electrode, counter, and reference electrode were connected to a Potentiostat/Galvanostat. The polarization of steel was done at ± 10 mV of the corrosion potential at a rate of 0.1 mV/s and the resulted current was measured.

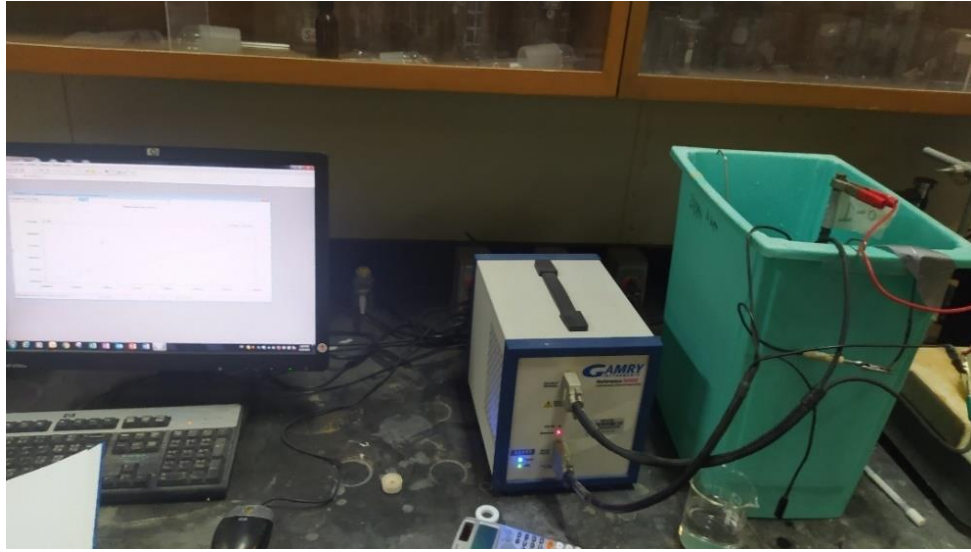


Figure 3-16: Setup of corrosion current density test

3.7.7 Chloride Diffusion

In this test, 75×150 mm concrete cylinders were coated with epoxy sealant from all sides with the cast surface left uncoated in accordance with ASTM C1556 [114]. Then, a saturated calcium hydroxide solution was prepared to neutralize the specimens for at least two cycles (each cycle was 24 hours) until there was no record of weight change. After that, a solution of 5% sodium chloride was prepared and specimens were exposed for 3 months. After the exposure period has finished, 6 concrete disks of 5-mm thickness for each specimen were obtained at different depths as shown in Table 3-8. These disks were pulverized in a machine as shown in Figure 3-17 and then passed through sieve #100. Following the pulverizing process, all samples were analyzed for water-soluble free chloride content in a procedure given below:

1. For each sample, 5 grams of powder was transferred into a beaker.
2. Then, hot distilled water of 50 ml was added to the sample and mixed thoroughly, and left for 24 hours for digestion of chlorides.
3. After that, the solution was filtered into a flask in which distilled water was added to the filtrate to attain 100 ml.
4. Then, 0.2 ml of the solution was taken, and distilled water was added to make it 10 ml.
5. 2 ml of each mercury thiocyanate and ferric ammonium sulfate was added to 10 ml solution.
6. The solution was gently shaken to ensure adequate mixing before it was put in a test tube.
7. The Spectro-photometer set at 460 nm wavelength was used to determine the absorbance value of each sample in the test tube.
8. The chloride calibration curve was used to calculate the free chloride concentration.
9. The chloride profile was used to find the diffusion coefficient according to Fick's law of diffusion

Figure 3-18 shows the experimental arrangement for determining the water-soluble free chloride content.

Table 3-8: Depth of 6 layers obtained for each specimen

Layer no.	Depth, mm	Average depth, mm
1	5-10	7.5
2	15-20	17.5
3	25-30	27.5
4	45-50	47.5
5	70-75	72.5
6	95-100	97.5



Figure 3-17: Pulverizing of specimens' layers after 3-months of chloride exposure



Figure 3-18: Chemical analysis to determine free chloride content

3.7.8 Thermal Conductivity

Three disks of 50-mm diameter and 25-mm thickness were prepared for each mix to determine their thermal conductivity. In this test, the FOX50 heat flow meter apparatus was employed in accordance with ASTM C518-21 [115] as shown in Figure 3-19. The setup includes a cooling unit (air pump and water flow) that works in link with FOX50 and is connected to a computer using the software “WinTherm50”. After the temperature of the cooling unit was fixed at 18 °C, the computer software “WinTherm50” was used to input

the file name as well as upper and lower temperatures. In our measurements, the temperature of the upper plate was set at 45 °C which is 20 °C higher than the temperature of the lower plate (25 °C) to resemble the real conditions of buildings. The upper and lower plates have sensitive heat flow thermocouples and transducers. Initially, the top and bottom surfaces of the specimens were polished because of the higher sensitivity of the plates in the presence of air gaps.

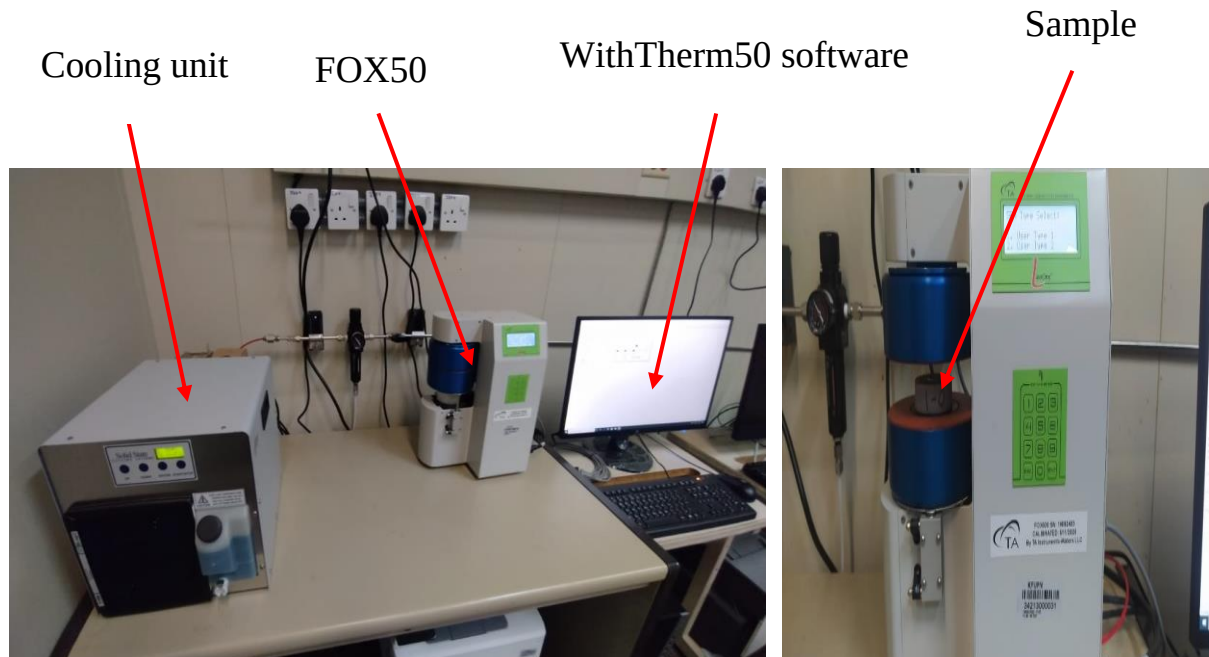


Figure 3-19: FOX50 heat flow setup

3.8 Microstructural Analysis and Characterization

3.8.1 SEM

Scanning electron microscopy (SEM) is a technique that generates images of a sample by scanning it with a beam of electrons. The interaction of electrons with different atoms at

the sample surface produces several signals that reveal information about the surface composition and morphology. Small portions of fractured concrete and paste samples were obtained from cubes exposed and cured in water for 28 days and cubes exposed to acid solutions. Their surfaces were coated with gold to minimize electric charge and enhance the resolution. JEOL SEM JSM-6610LV device used in this study is presented in Figure 3-20.

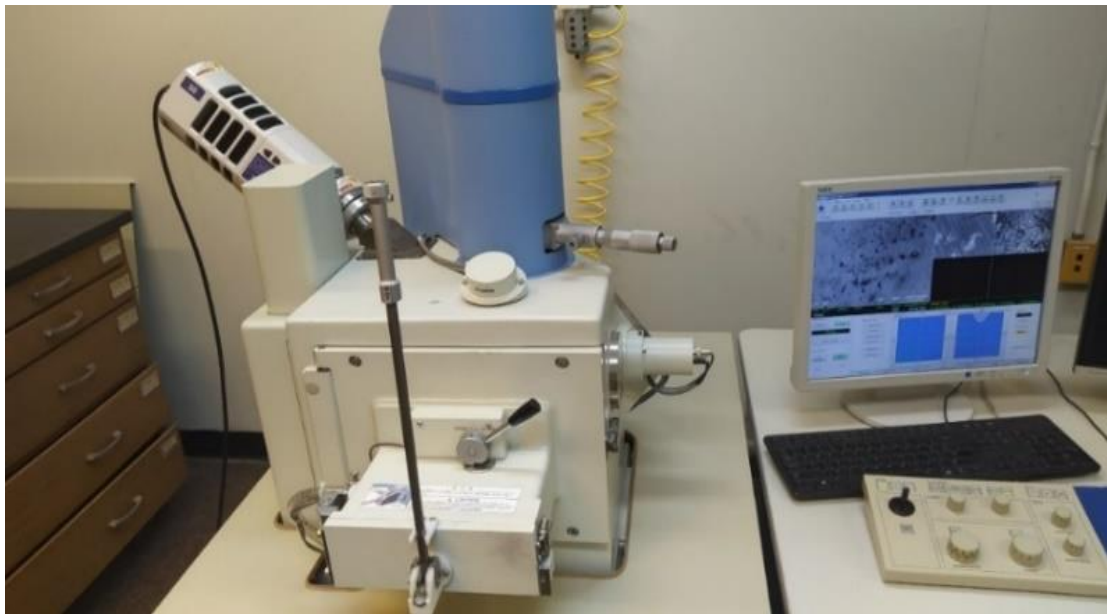


Figure 3-20: SEM JOEL used for morphology

3.8.2 XRD

X-ray diffraction (XRD) technique has been developed to study and characterize the mineralogical composition of the materials. This technique provides characterization information about material constituents based on their diffraction pattern. Powdered paste samples were prepared, sieved, and analyzed using Bruker Benchtop D2 phaser XRD equipment to identify the different phases of the constituents that existed in the samples as

shown in Figure 3-21. The diffraction angle 2θ varied from 5 to 80. Then, the rotation of samples was performed using the specified angles and the intensity of diffracted X-rays was measured in counts per second.



Figure 3-21: Benchtop D2 phaser XRD

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Setting Times

The setting time of fresh concrete is a paramount property since it is related to the timing of placing and finishing the concrete. Figure 4-1 depicts the initial and final setting times of paste samples according to ASTM C191 [116]. The results showed that both initial and final setting times were systematically increasing with the increase in SMF content. Moreover, all blended paste mixes exhibited higher setting times compared to the control paste sample. The range of initial and setting times was 2.33-5.82 hours and 3.75-8 hours, respectively. The increase in setting time is ascribed to the retardation effect of SMF due to the slower reaction of SMF at early ages. The low ratio of CaO/SiO_2 and the high MnO content of SMF are also contributing to the extension of setting times [39]. Moreover, the results of the thermal evolution of paste samples conducted using semi-adiabatic calorimetry subsidize the prolonging of setting times with subsequent replacement of SMF. Incorporating mineral admixtures such as slag, sugarcane bagasse ash, fly ash, and metakaolin in concrete is generally retarding the hydration process due to the lower cement content and lower hydration rate of these materials [93].

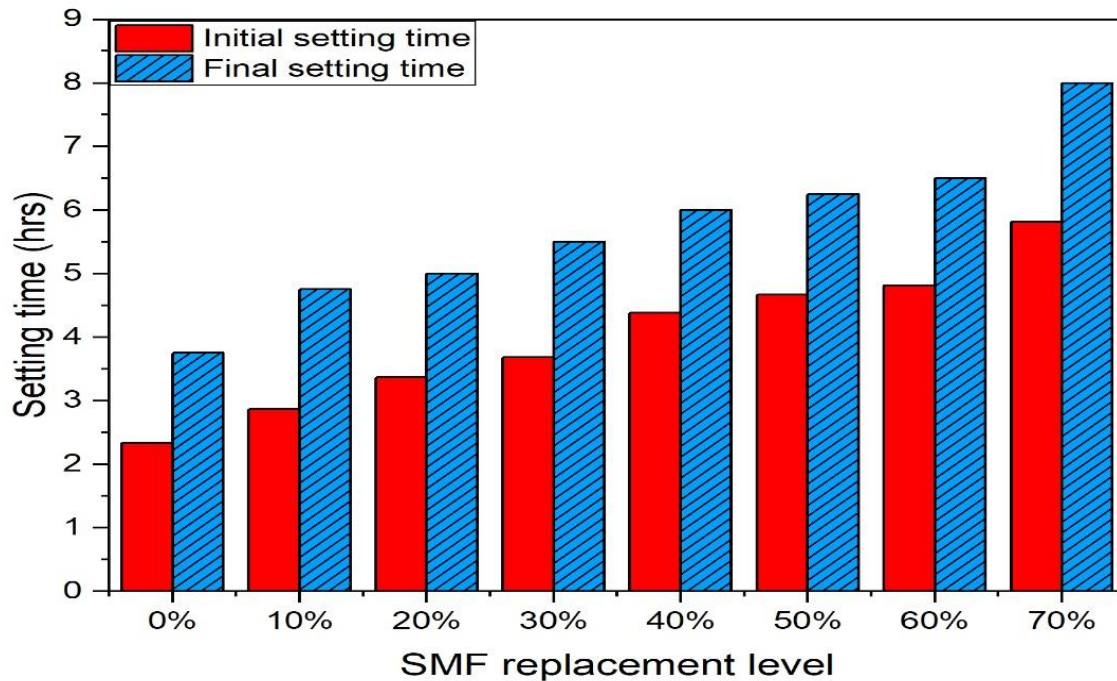


Figure 4-1: Initial and final setting times of SCC mixes (cf Qawah et al. [111])

4.2 Heat of Hydration of Paste Samples

The heat of hydration of cement pastes is an exothermic process that is released during the chemical reaction, and it depends on the major constituent in their chemical compositions such as calcium in cement [104]. Although high cement content is beneficial to obtain higher initial strengths, the higher heat generated can negatively affect the durability which could result in higher shrinkage and more cracks [104]. Therefore, thermal cracking and corrosion of reinforced steel in concrete are avoided by using materials of lower heat of hydration such as fly ash, silica fume, slag, and rice husk ash [104].

In this study, the temperature profiles of cement paste samples prepared with 0-70% SMF were recorded for up to 24 hours as presented in Figure 4-2. The data logger was used in the recording of temperatures of paste samples. Table 4-1 shows the dormant period, initial

and peak temperature, and time to peak temperature. The initial temperature rise reaching the peak temperature is followed by a drop in temperature, upon which the temperature stabilized. All paste mixes underwent a series of exothermic chemical reactions. However, their heat of hydration behavior has seen a significant variance in some parameters such as dormant period and peak temperatures. The inclusion of SMF deterred the rise of temperature compared to Portland cement paste. In the cement paste mix, the increase in temperature was noted after about 2.28 h. However, this period has seen an upsurge with values of 2.87, 4.08, 6.37, and 9.97 in SMF 10, SMF 20, SMF 50, and SMF 70, respectively. The early hydration starting with the initial temperature of the samples was shown to be fairly equal, however, a significant variation of peak temperatures was observed. The heat of hydration was significantly affected by the increase in SMF replacement levels. Therefore, there was a drop in the peak temperatures of the samples with the addition of SMF. For instance, the peak temperature of SMF 70 was the lowest with a value of 26.3 °C compared to the highest value of 32.8 °C in the control sample. The time to peak temperature has initially increased with increasing the quantity of SMF up to 30%. Accordingly, this time was at its lowest value of 14.3 h in the cement paste with 0% SMF while it was 14.30, 15.25, 17.27 and 23.20 h in paste samples with 10% to 30% SMF. The obtained results proved the effectiveness of SMF in reducing the peak temperatures and delaying the time to reach peak temperatures. This is mainly attributed to the acidity and slower reactivity of SMF, in addition to its high MnO content with low CaO/SiO₂. According to Awal and Shehu, the lower heat of hydration obtained for higher replacement of palm oil fuel ash with cement (50%-70%) is attributed to the pozzolanic reaction of the fly ash and the lower cement content in these mixes. Based on the previous discussions,

the utilization of SMF as a retarding admixture in structural applications can be beneficial and practical under hot weather conditions, especially in gulf countries. Moreover, most commercial retarders are expensive and supplied in the liquid form in which their shelf-life is limited [117]. Therefore, the use of solid waste materials as retarding admixtures minimizes the costs of concrete production and the environmental problems due to the disposal of these materials [117]. Some correlations can be developed between the setting times and the parameters of the heat of hydration. In this regard, a strong correlation was observed between the setting times and the peak temperatures of paste samples as shown in Figure 4-3. The values of R^2 were 0.918 and 0.903 for initial and final setting times with peak temperatures, respectively. In addition, Figure 4-4 presents a strong correlation between the dormant period and peak temperatures of paste samples with R^2 of 0.892.

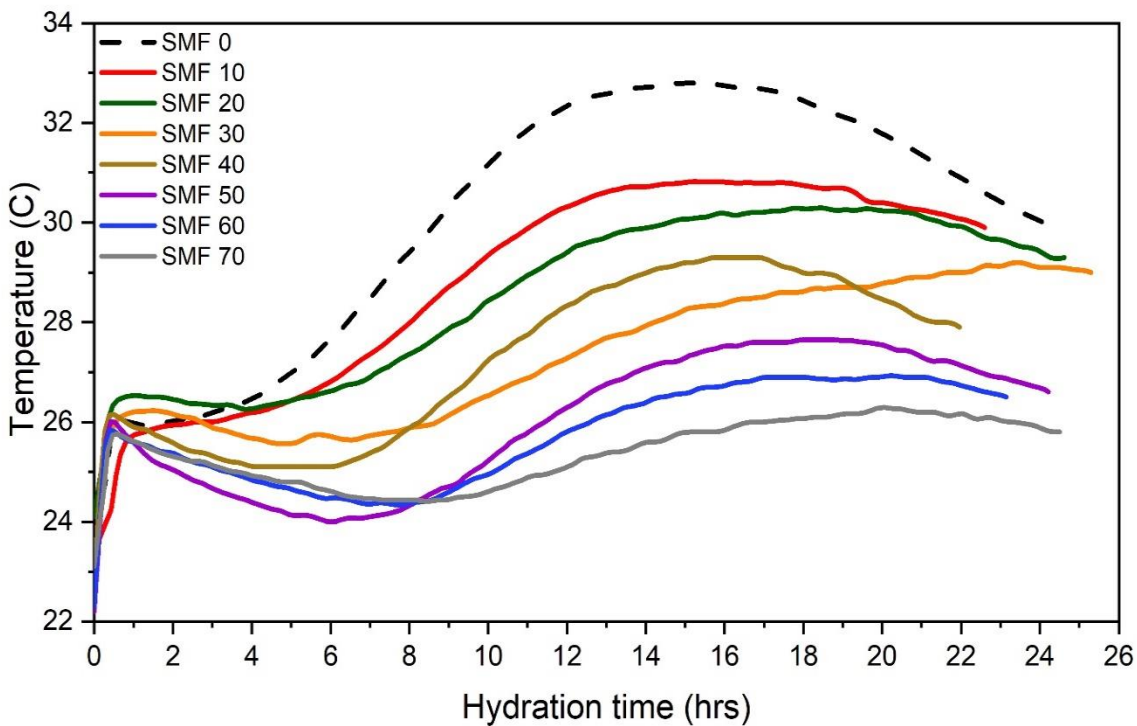


Figure 4-2: Temperature evolution due to hydration in paste samples

Table 4-1: Influence of SMF on Dormant period, Peak Temperature, and Time to reach Temperature

SMF replacement	Dormant period (Hours)	Initial temperature (°C)	Peak Temperature (°C)	Time to Peak Temperature (Hours)
0%	2.28	23.6	32.8	14.3
10%	2.87	23.4	30.9	15.25
20%	4.08	24.1	30.3	17.27
30%	5.12	23.2	29.2	23.20
40%	6.35	22.8	29.3	15.55
50%	6.37	22.2	27.7	17.92
60%	9.38	22.3	26.9	16.85
70%	9.97	23.1	26.3	19.83

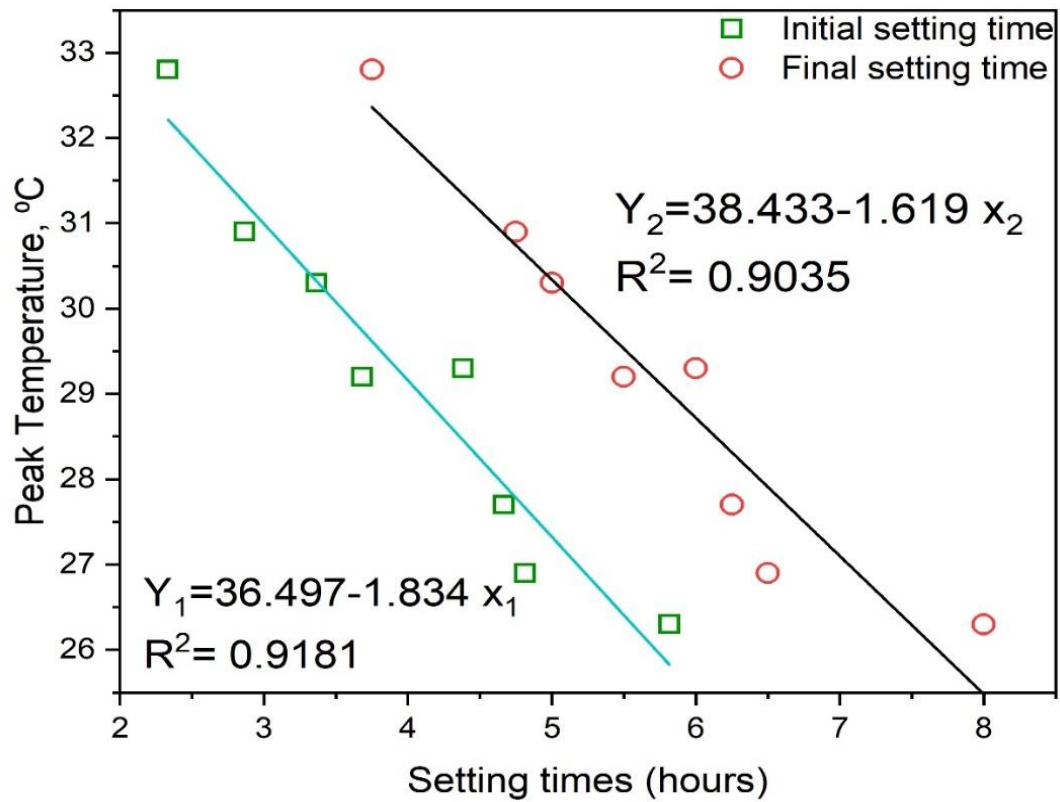


Figure 4-3: Correlation between Peak temperatures and Setting times

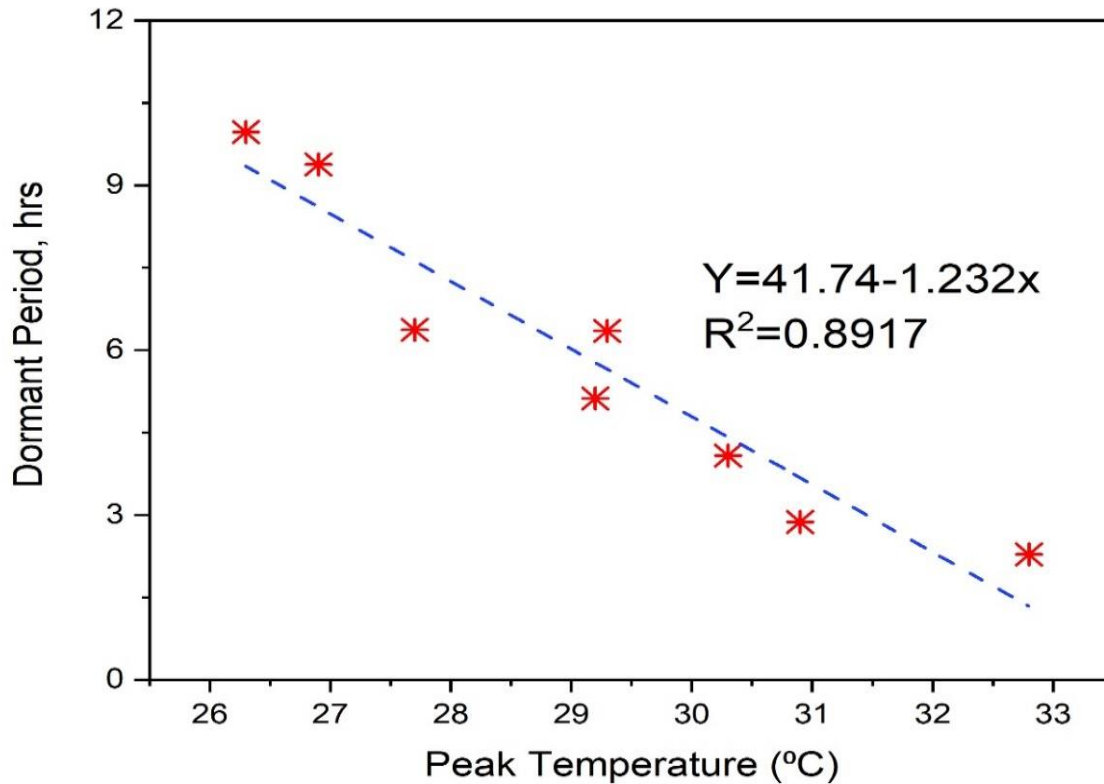


Figure 4-4: Correlation between Dormant Periods and Peak temperatures

4.3 Compressive Strength

The compressive strength development of SCC mixes incorporating SMF at 7,14,28 and 90 days of water curing is presented in Figure 4-5. As shown in the figure, the strength values of SCC concrete blended with 10%-20% SMF were greater than those for control mixes, measuring at all curing ages. Accordingly, the control mix attained a compressive strength of 36.41 MPa at 7 days which improves to 43.05 MPa and 38.03 MPa for mixes made with 10% and 20% SMF, respectively. The highest compressive strength at 28-days was recorded for substitution level of 10% with a value of 55 MPa; whilst the lowest was obtained for SMF 70 with a value of 27.6 MPa. The range of compressive strength at 7, 14, 28, and 90 days was 14.76-43.05, 23.00-51.79, 27.63-54.9, and 35.6-57.9, respectively.

The addition of SMF up to 30% was not adversely affecting the compressive strength results in comparison with the control mix, especially at higher curing ages. For instance, the 28- and 90-day compressive strengths of SMF 30 were 42.9 MPa and 47.8 MPa compared with 45 MPa and 47.3 MPa in the reference mix. However, the compressive strength of mixes of high SMF content beyond 40% has significantly dropped especially in early curing periods of 7 and 14 days. For instance, strength reduction at 70% SMF at 7, 14 and 28 days was 59.46 %, 45.39 %, and 30.17%, respectively, while it was only 24.79% at 90 days. This indicates that a high volume of SMF gains compressive strength at later ages due to the slower reaction of SMF. The increment in compressive strength values at 10-20% SMF may be attributed to two key roles of SMF: Physical and Chemical effects. The physical role of SMF finer particles (higher surface area compared to cement) to fill the gap between aggregates and cement contributes to the densification of the microstructure. Moreover, the chemical effect of SMF promotes the pozzolanic reaction of SMF with valuable lime to produce secondary C-S-H. On the other hand, mixes, in which cement was replaced by more than 40%, exhibited lower compressive strength values in comparison with the control mix. This may be accredited to the dilution effect due to the limited portlandite (Ca(OH)_2) content produced in these mixes which reduces the formation of hydration products. In addition, the slower reactivity of SMF due to higher MnO content (35%) and lower SiO_2 content may contribute to the decline in strength in replacement levels exceeding 40%. Some materials such as Granite waste (finer than cement) showed a similar effect at higher contents when replaced by cement [54]. The presence of voids and cracks in concrete mixes prepared with high content of SMF was confirmed by

studying the morphologies using SEM in the next section which further elucidates and explains the decrease in strength.

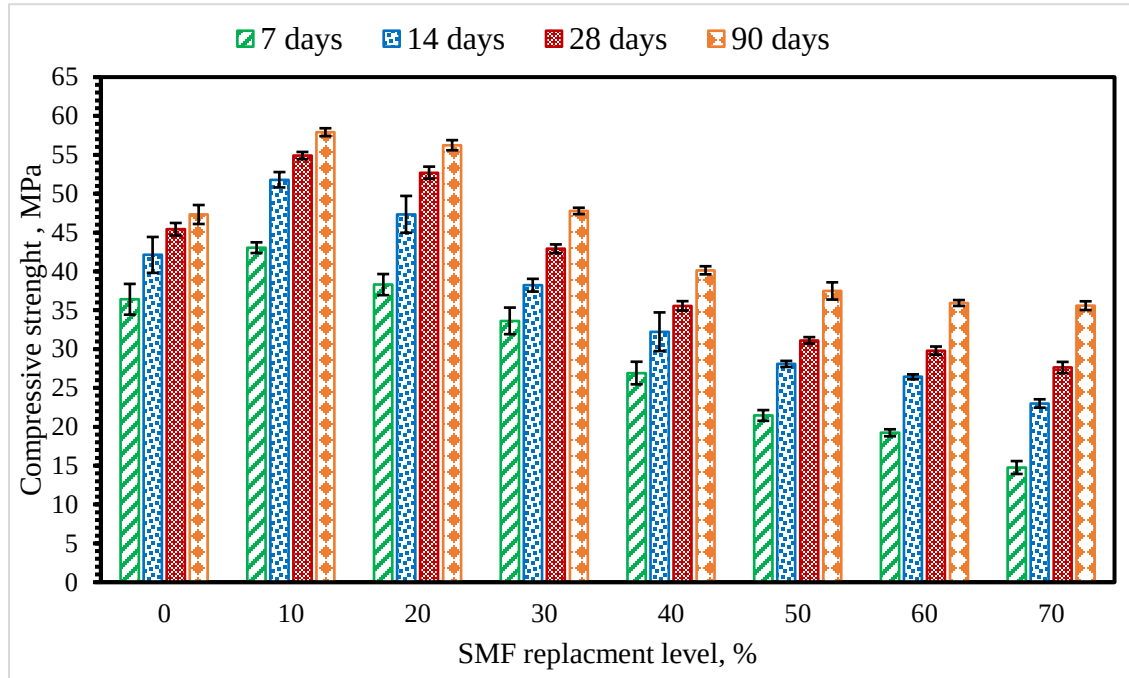


Figure 4-5: Compressive strength evolution for SCC mixes (cf Qawah et al. [111])

4.4 Morphology of Concrete Samples at 28 Days

The morphology and the microstructure of the fractured concrete samples cured in water for 28 days were examined using SEM as shown in Figure 4-6 and Figure 4-7. SEM pictures showed a denser microstructure in the control sample and samples with 10% to 30% SMF with some cracks and voids in SMF 30. However, the inclusion of high content of SMF beyond 40% showed a less densified microstructure with more cracks and pores which are filled by needle-like shapes (ettringite). These pictures may explain the effect of the addition of SMF on the strength and durability properties in which the improvement was limited to lower SMF replacement levels up to 30%.

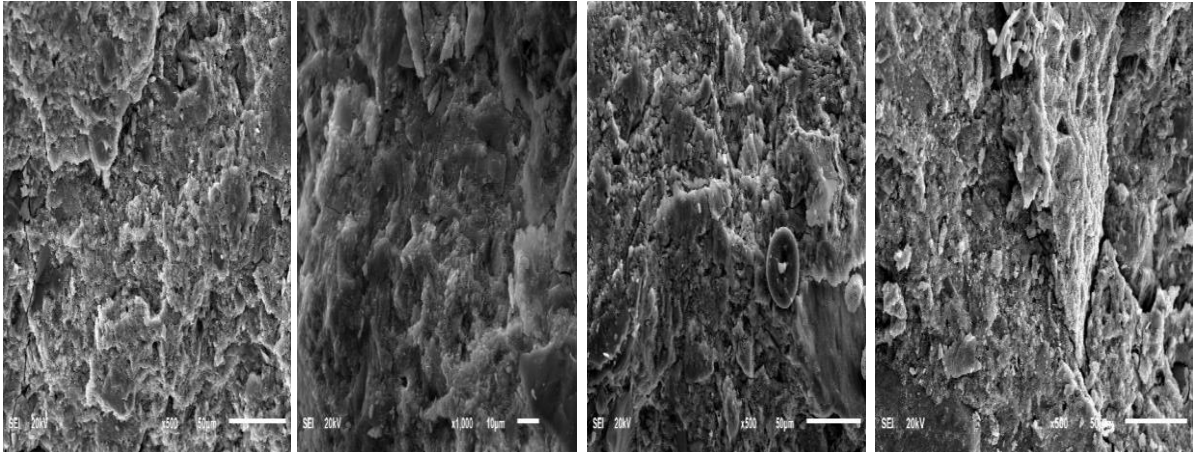


Figure 4-6: SEM images of concrete samples from left (control) to right (SMF 30)

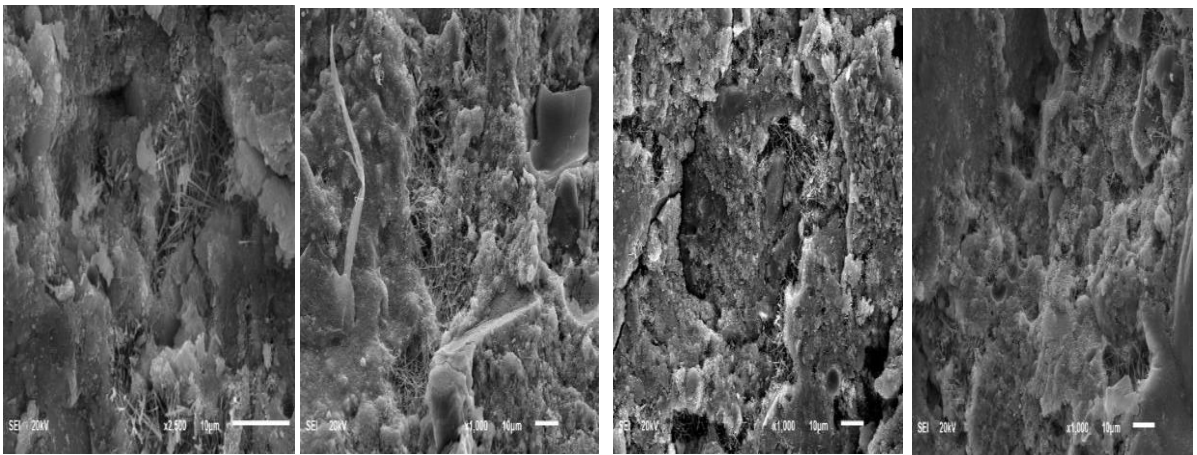


Figure 4-7: SEM images of concrete samples from left (SMF 40) to right (SMF 70)

4.5 Rapid Chloride Permeability Test (RCPT)

There are two mechanisms behind the transport of chloride ions in concrete, namely, the binding of chlorides to the cement matrix and the penetration of chloride ions through the interconnected pores which react with C_3A [118]. Corrosion of reinforced bars is initiated with the presence of free chloride ions [118]. The total charges passed in this test give an indication of the chloride ion permeability of the concrete specimen.

Depending on the SMF content, the total charges passed vary from 1287 to 3112 coulombs. Moreover, SCC mixes with a replacement level up to 30% showed the lowest charges; this indicates a higher resistance to chloride permeability. Then, there was a gradual increase in RCPT values in mixes beyond 40% SMF substitution levels. The pore refinement in the microstructure in the replacements up to 30% SMF can be accredited for the improvement in RCPT values in comparison with the control mix. An increase in the amount of C-S-H gel is expected in these mixes that reduces the porosity and densifies the concrete. On the other hand, mixes of higher content of SMF exhibited poor performance in RCPT due to porous microstructure and cracks that allow the movement of charges. Moreover, the pore solution chemistry due to the presence of alkaline ions such as Na^+ , and K^+ influences the results of RCPT.

Figure 4-8 illustrates the rapid chloride permeability of the SCC mixes along with red dotted lines showing the ASTM limits. Most of the mixes were classified under the category of moderate chloride permeability at 28 days of curing according to ASTM C1202 criteria [112]. However, the concrete mixes of replacement levels of 10-30% have better performance and are reported as a low class. The charges passed through the control mix were about 2344 while charges of SMF10 to SMF30 ranged between 1287 and 1725, respectively. The values of RCPT for mixes containing 40% to 70% SMF increased by 5.6% to 32.7% compared with the control mix. A strong linear correlation exists between strength and RCPT with $R^2 = 0.8757$ as shown in Figure 4-9.

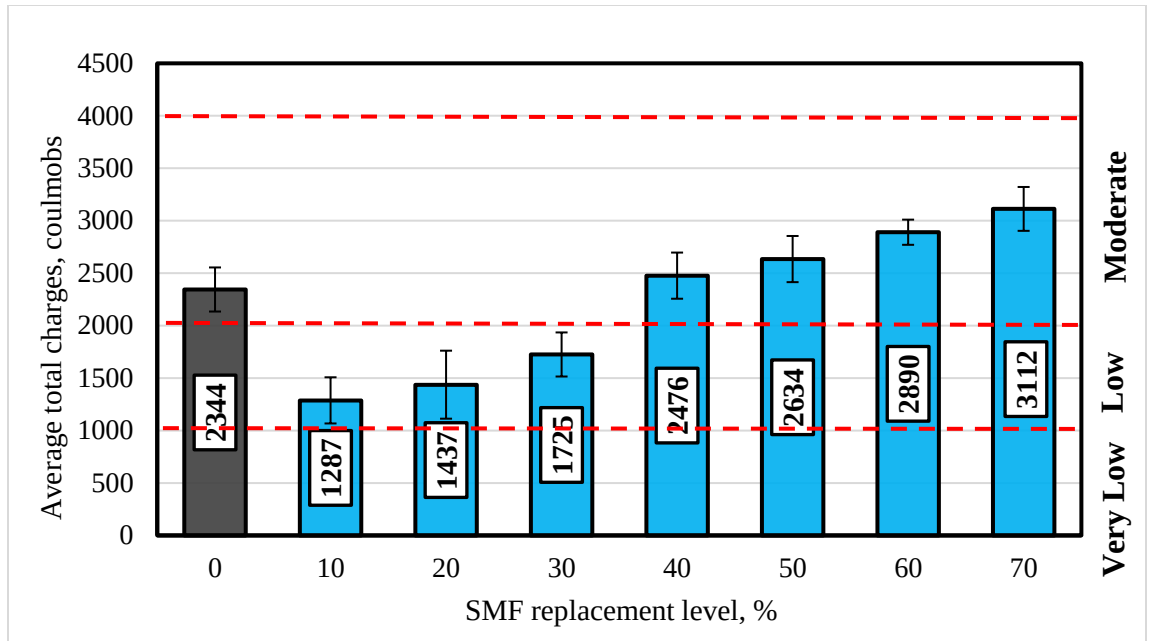


Figure 4-8: Influence of SMF on the chloride permeability of SCC mixes

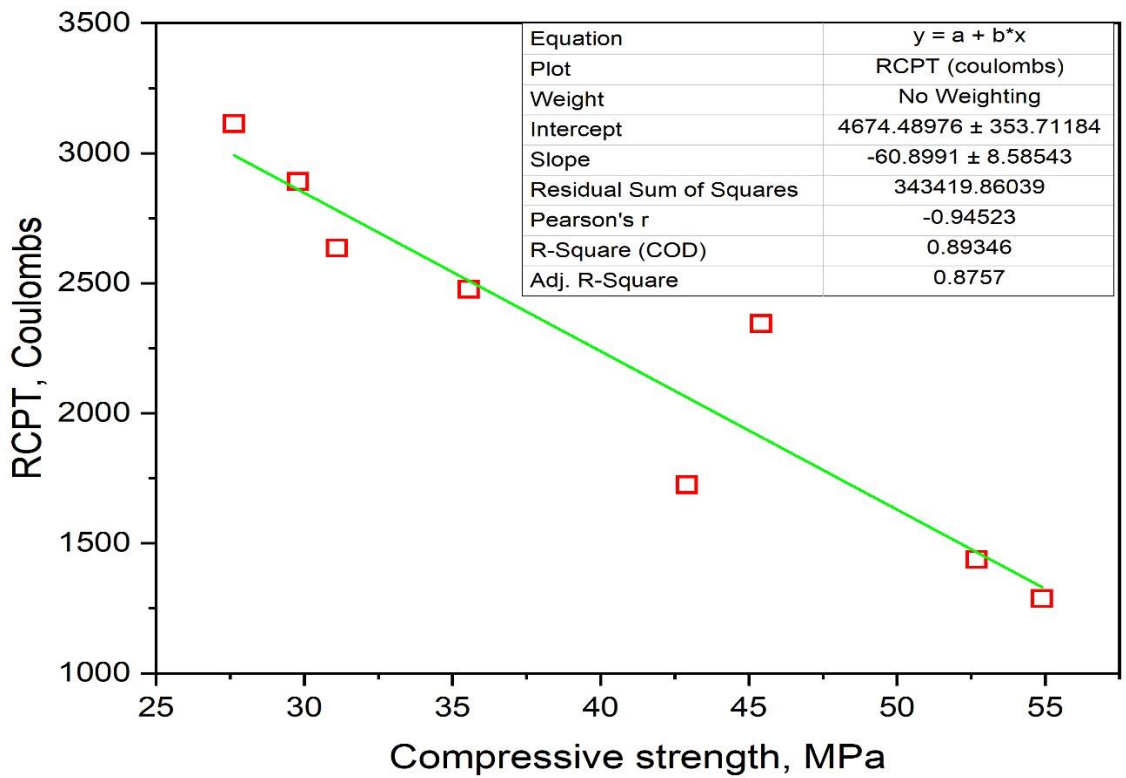


Figure 4-9: Correlation between strength at 28 days and RCPT

4.6 Electrical Resistivity (ER)

The resistivity of concrete has a significant influence on the flow rate of ions through reinforced concrete. Therefore, the measurement of electrical resistivity gives an indication of the possibility of corrosion occurrence [9] and it's used to assess concrete's durability properties. Moreover, electrical resistivity is related to the pore interconnectivity and the pore size distribution. Several intrinsic factors affect the results of electrical resistivity such as w/c ratio, size, the connection of voids, and type and size of aggregates [85]. Moreover, factors that are related to measurements such as moisture content, temperature, and geometry are also playing a significant role in the variation of the electrical resistivities [85]. The bulk electrical resistivity of concrete is mainly the ratio between the applied voltage and the resulting current in a unit cell.

The bulk electrical resistivity of concrete specimens cured in water for 28 days was measured after the specimens were saturated in water for 48 hours. Figure 4-10 depicts the measured electrical resistivity of concrete mixes at different moisture contents using the two-probe Wenner method. For all SCC mixes, there is an inverse relationship between electrical resistivity and moisture content. The developed exponential relations between electrical resistivity and moisture content are presented in Table 4-2. Then, the electrical resistivity was computed at 3% moisture content which is shown in Figure 4-11. The values showed a similar trend to the RCPT test in which concrete prepared with 10% to 30% SMF showed higher electrical resistivity compared to the control sample. The maximum value of ER of 74.78 k Ω .cm was obtained at SMF 10, whilst the lowest was 27.69 k Ω .cm at SMF 70. Replacing OPC with SMF at 10-30% ratios led to an 11-27% increment in the electrical

resistivity. This can be attributed to the limited pozzolanic reaction of SMF which produces secondary C-S-H gel, resulting in the densification of the microstructure. Moreover, its physical property as a micro-filler helps in increasing the particle packing density. However, lower calcium hydroxide produced in higher SMF content is not sufficient to produce a denser structure. On the other hand, mixes of higher SMF content were reported to exhibit lower electrical resistivity values. To define the relationship between RCPT and ER, a strong correlation developed with R^2 equal to 0.9543 as shown in Figure 4-12.

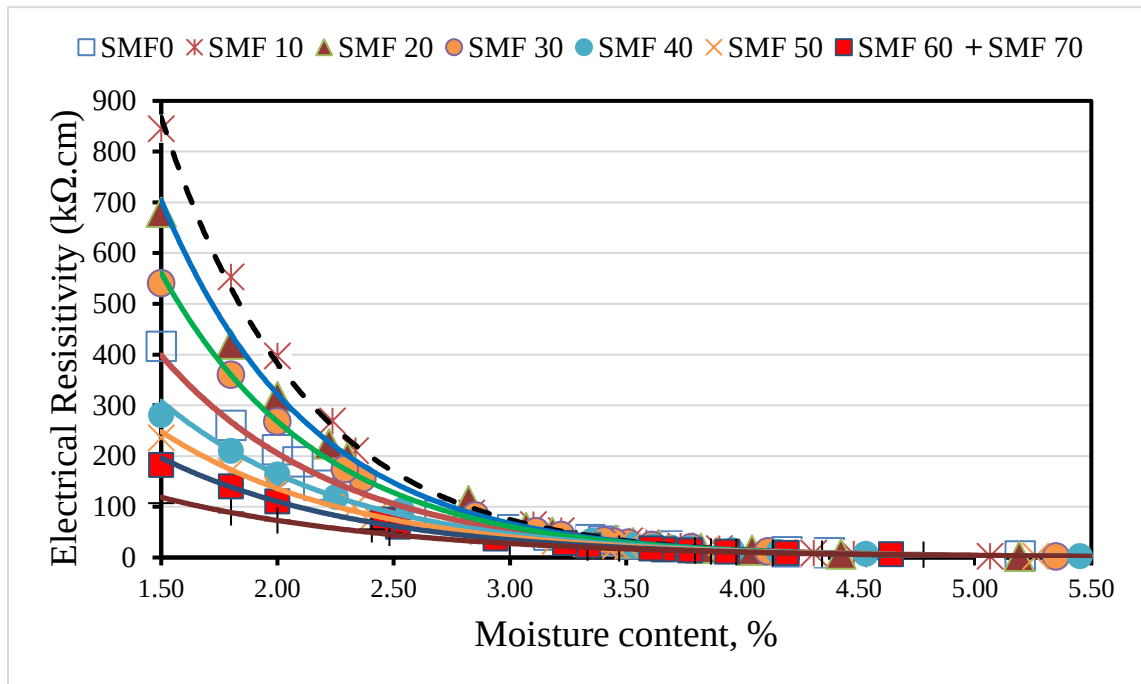


Figure 4-10: Electrical resistivity of concrete mixes vs moisture content

Table 4-2: Correlation parameters and electrical resistivity at 3% moisture content

MIX ID	SMF 0	SMF 10	SMF 20	SMF 30	SMF 40	SMF 50	SMF 60	SMF 70
A	2913.8	10092	7352	5149.3	2030	1511.9	1105.5	508.3
B	1.327	1.636	1.564	1.478	1.258	1.205	1.152	0.97
$\rho(3\%) = A \cdot e^{-3B}$	54.39	74.78	67.41	61.1	46.61	40.7	34.88	27.69

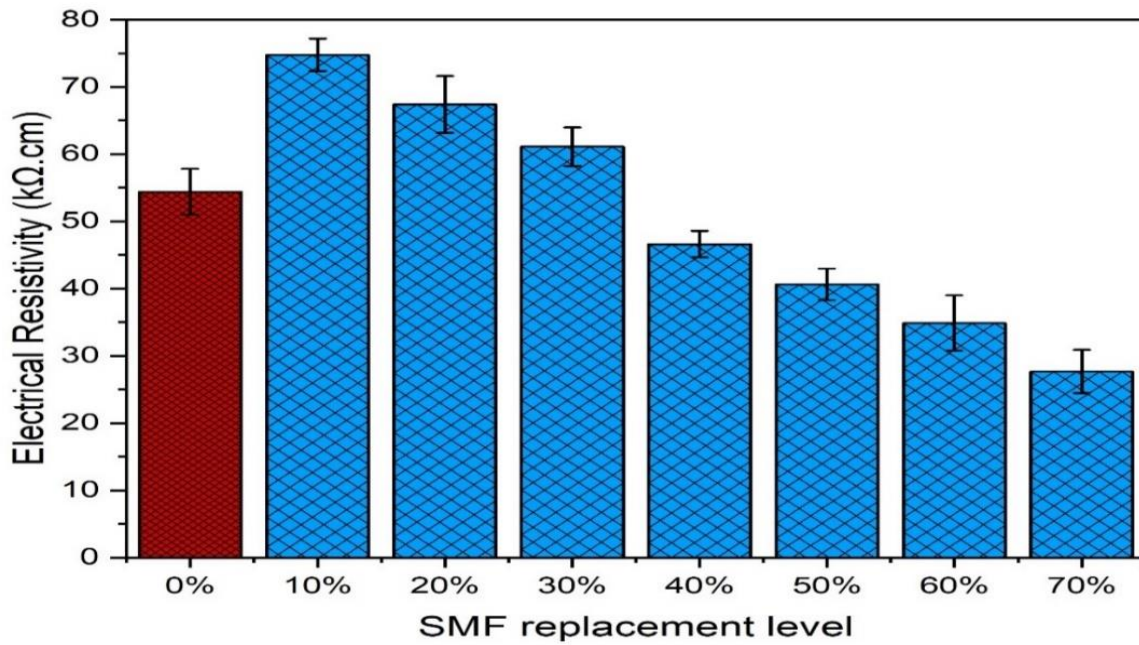


Figure 4-11: Electrical resistivity of SCC mixes at 28 days curing measured at 3% moisture content

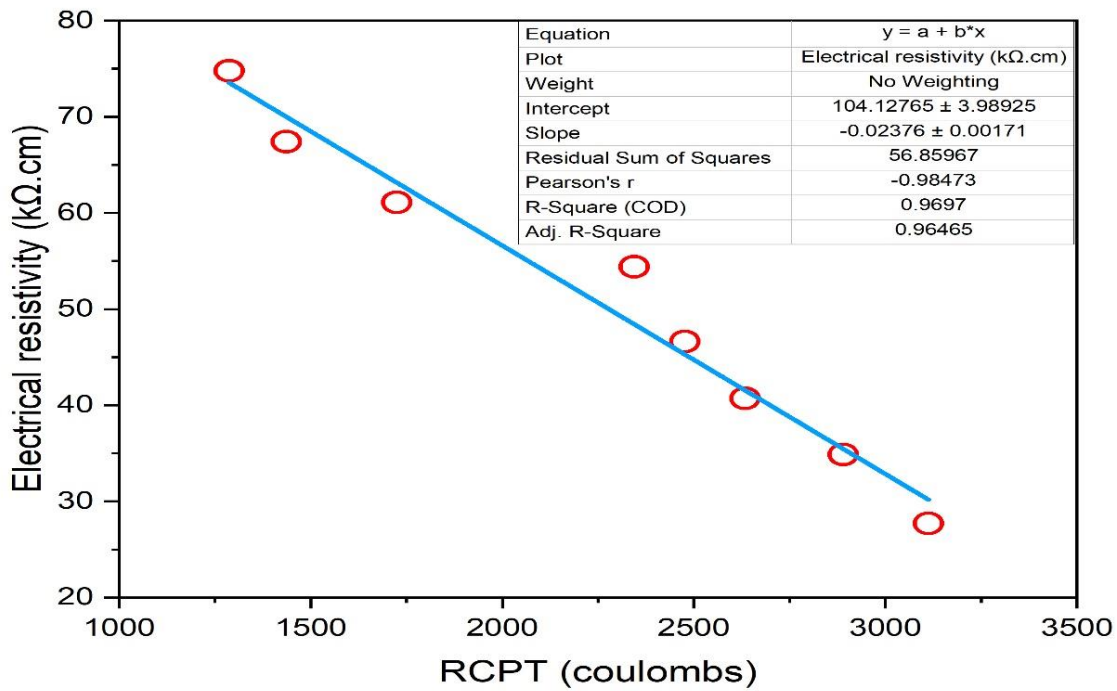


Figure 4-12: Correlation between electrical resistivity and RCPT of SCC mixes

4.7 Acid Attack

Exposure of conventional concrete to acidic environments presents a major strength and durability problem that eventually affects the maintenance cost, lifetime, and performance of structural infrastructures [92,94]. The durability of concrete due to the chemical attack by acid ingress is an implication of its ability to survive cracking, breakdown, spalling, and deterioration. OPC concrete is vulnerable to acid attacks due to its alkaline nature [119]. The mechanism of an acid attack includes two main reactions. In the first reaction, sulfuric acid reacts with calcium hydroxide to form gypsum which results in more than two times volumetric expansion. However, the reaction of gypsum and tricalcium aluminate can produce ettringite that causes 7 times more expansion. Following the formation of ettringite, inner stresses and expansion are induced due to volumetric expansion, creating some cracks. As the concrete erosion continues, discoloration and softening lead to further deterioration and cracks to reach ultimate destruction. In this study, the effect of 2% sulfuric acid attack on SCC blended with SMF was determined by the variation of the compressive strength values and visual examination of concrete specimens.

4.7.1 Visual Inspection

Figure 4-13 and Figure 4-14 present the images of concrete specimens before and after immersion in 2% sulfuric acid solution for a period of 3 and 6 months. In general, all concrete mixes suffered from severe deterioration such as softening, spalling, and disintegration of the surface layers at the end of 90 days and 180 days. The surface layer of all concrete mixes was noticed to be completely deteriorated due to the washing away

of the paste around aggregates as shown in the taken pictures. However, the cubical specimens still retained their shape even after 180 days. The discoloration or formation of pale-yellow color on the outer periphery was noticed in the control mix as well as in blended mixes up to 30% SMF which substantiate the formation of gypsum and other corrosion products. The growth of gypsum crystals has induced internal disintegrating stresses which accelerates the total rate of deterioration and caused spalling of surface layers. The higher surface area of SMF with lower specific gravity lowers the density of the concrete and increases the porosity, contributing significantly to the apparent damage in concrete cubes. At the end of 180 days, concrete specimens showed relatively higher damage when compared with their counterparts at 90 days, which could be the result of a higher amount of unremovable gypsum deposits. This damage is also quantified in terms of strength losses in the next section.

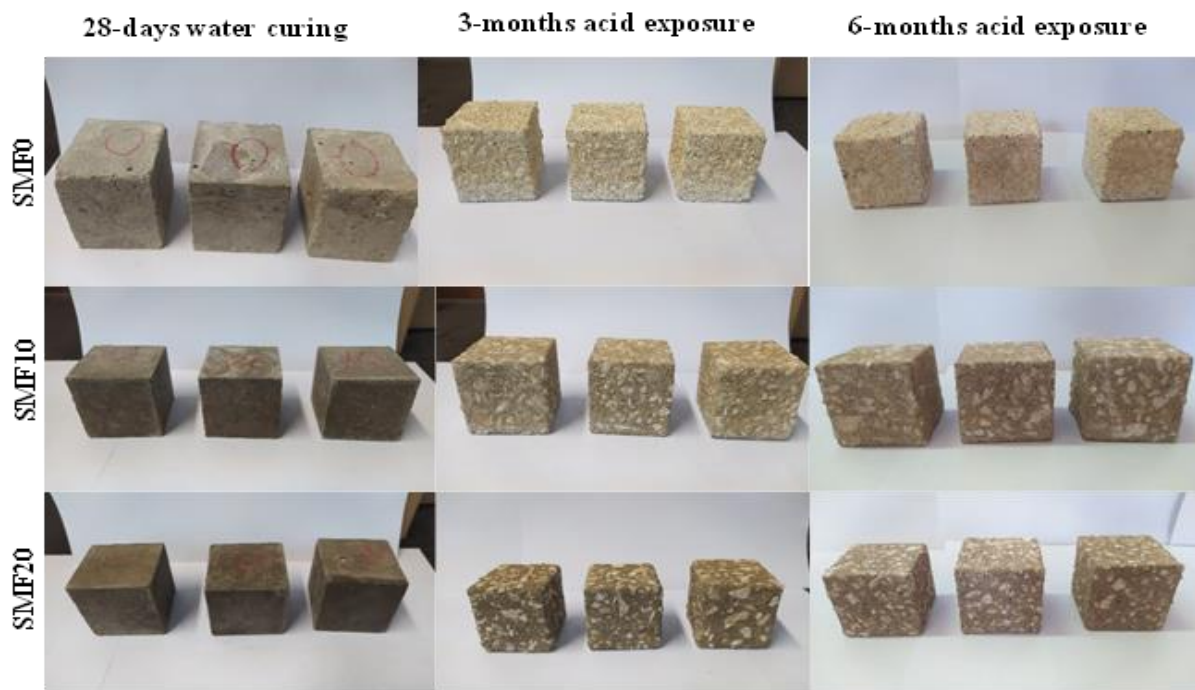


Figure 4-13: Images of SCC mixes (SMF0- SMF20) before and after exposure to 2% sulfuric acid

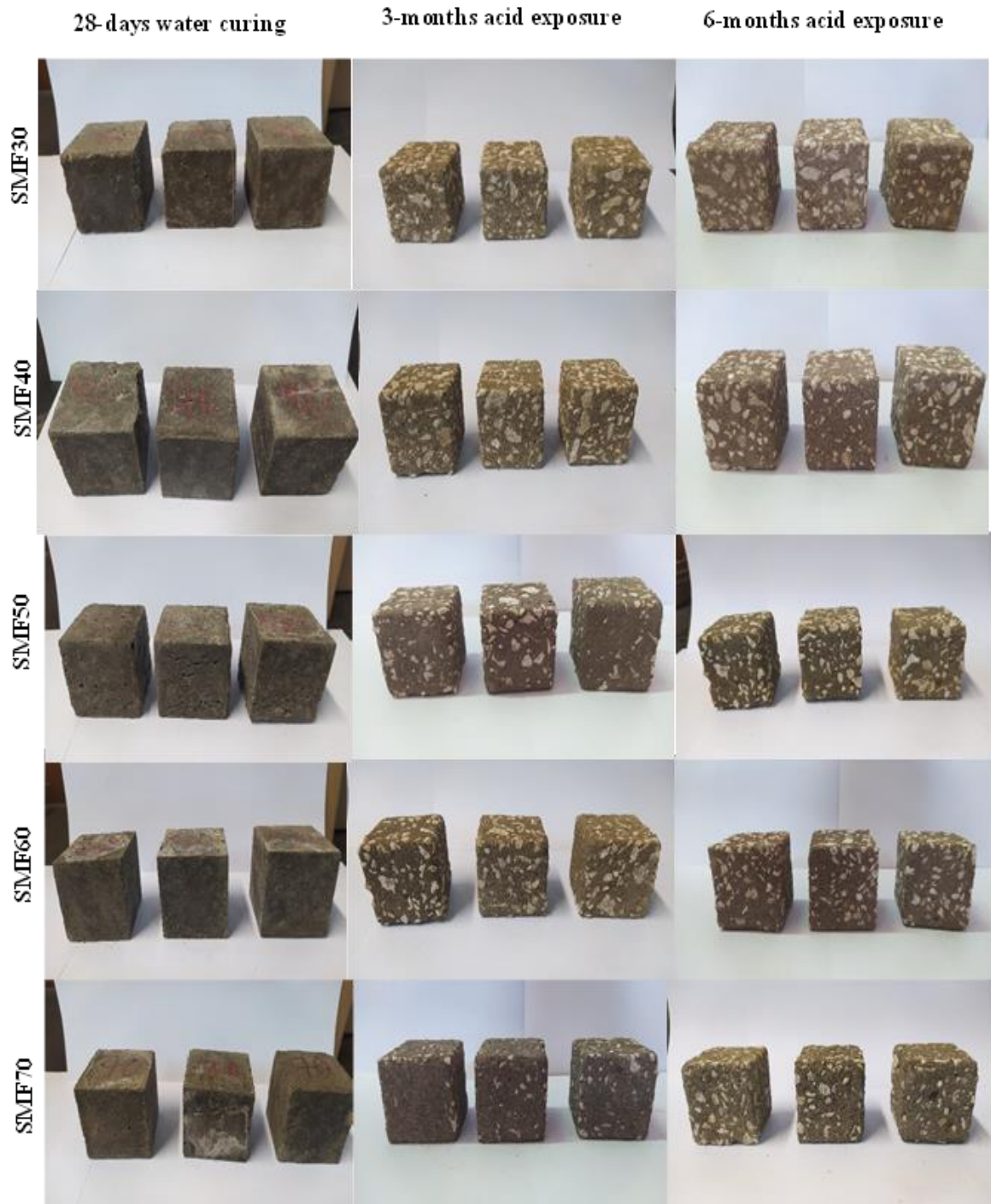


Figure 4-14: Images of SCC mixes (SMF30- SMF70) before and after exposure to 2% sulfuric acid

4.7.2 Compressive Strength Loss

Figure 4-15 depicts the compressive strength variation of SCC mixes at 28 days of water curing and 3 and 6 months of acid exposure. Compressive strength values of unexposed specimens increased with the increase in SMF content to reach its maximum at 20%, followed by a gradual decrement in strength. Approximately similar trends were observed for the compressive strength values at 3- and 6-months of acid exposure. All mixes showed a higher reduction in compressive strength with the progress of exposure. The inferior compressive strength of concrete specimens due to acid exposure can be attributed to their erodible surfaces that get deteriorated faster. The reduction in 3-months compressive strength ranged between 23.80% and 46.13% while the strength loss after 6-months exposure was between 32.7% and 59.19% as presented in Figure 4-16. Moreover, specimens of higher SMF content experienced the highest loss of strength at 3-months with 37.18%, 43.31%, and 46.13% in SMF 50, SMF60, and SMF 70, respectively. The significant decrement of strength at SMF10 could be explained by the higher calcium content that leads to faster leaching of calcium from C-S-H gel and calcium hydroxide due to decalcification. The porous microstructure and the formation of gypsum in the last three mixes could justify their poor performance against acid attack. From the compressive strength loss of these samples, the optimum mixes that showed good performance were SMF 20 and SMF 30.

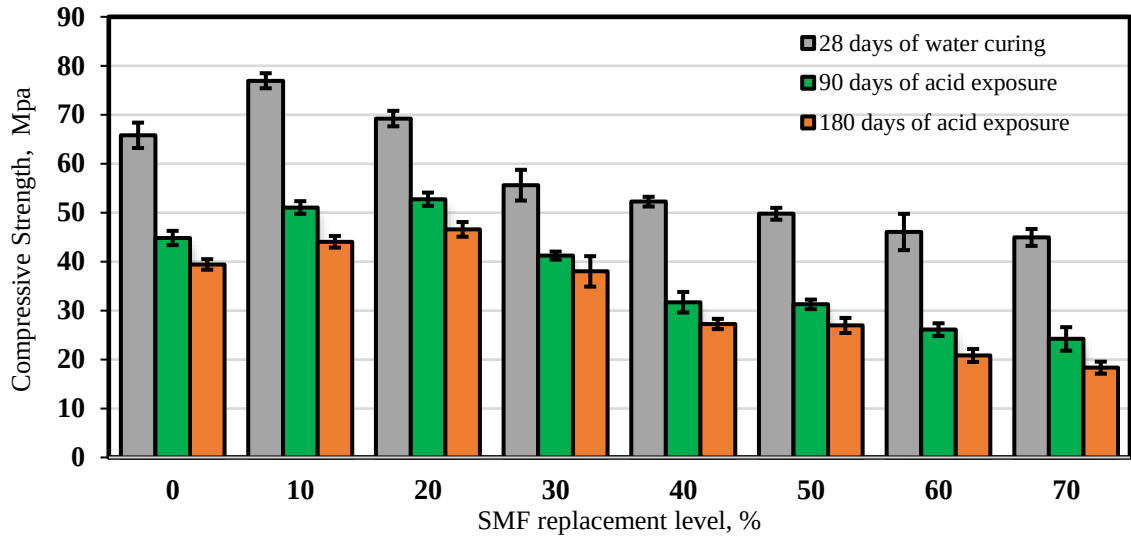


Figure 4-15: Compressive strength of SCC mixes before and after exposure to 2% sulfuric acid

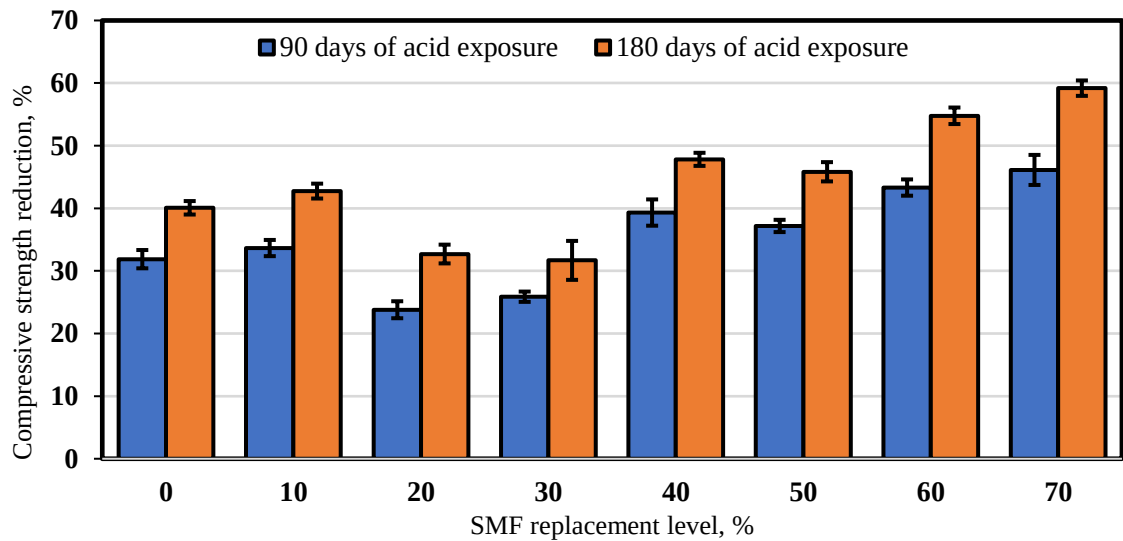


Figure 4-16: Compressive strength loss of SCC mixes after 90 and 180 days of acid exposure

4.8 Sulfate Resistance

Concrete undergoes a series of chemical reactions between hydrated cement paste and sulfate ions brought by external sources [102]. Moreover, higher powder content of 400-600 kg/m³ is very vulnerable to chemical attacks [80,102]. The sulfate attack occurs when

sulfate ions penetrate concrete and react with calcium hydroxide and alumina-bearing phases to produce ettringite [95]. The adverse effects of ettringite and gypsum result in the expansion and cracking of hardened concrete. Moreover, calcium silicate hydrate (C-S-H) can react with cations such as Na^+ and Mg^{2+} leading to decalcification of the gel and loss of strength and cohesiveness of the cement matrix. In the case of magnesium attack (MgSO_4), layers of gypsum and brucite formed on the surface and in the transition zone after the reaction of sulfate with hydration products, depleting Ca in C-S-H [120]. OPC concrete exhibited several deteriorations when subjected to sulfate such as expansion, strength loss, spalling, and disintegration [95–97]. However, the durability of concrete admixed with pozzolanic materials is improved as reported in some of the literature [120]. In this study, cement paste samples molded in 25-mm cubes and 25×25×285 mm prisms were exposed to 2.1% magnesium and sodium sulfates, respectively. Compressive strength loss and expansion were determined for specimens exposed under magnesium and sulfate solutions, respectively.

4.8.1 Visual Examination

Inspection of paste samples exposed to 2.1% magnesium sulfate for 3 and 6 months showed no remarkable signs of physical deterioration as presented in Figure 4-17. However, there were some changes in the appearance of cubes due to the covering of white deposits on their surface layers. This whitish deposit at 6 months of exposure is quite evident as compared with 3 months, particularly at higher content of SMF. Some studies reported these deposits to be $\text{Mg}(\text{OH})_2$, SiO_2 , and the silicate of calcium and magnesium compounds, however, gypsum has been proved using SEM/EDS to appear at the surface [120]. Based

on the physical appearance, it is expected to have a mass gain in most of these mixes which depends on the amount of deposits in each mixture and at each exposure period. A closer look reveals that higher levels of SMF exhibited higher weight gain compared with other mixes. This is assigned to the ingress of sulfate solution into these mixes, forming recrystallized salts [120]. Mixes containing high SMF content are expected to be porous and permeable due to high fineness and specific surface area. Therefore, their performance is predicted to decline when exposed to sulfate solution.

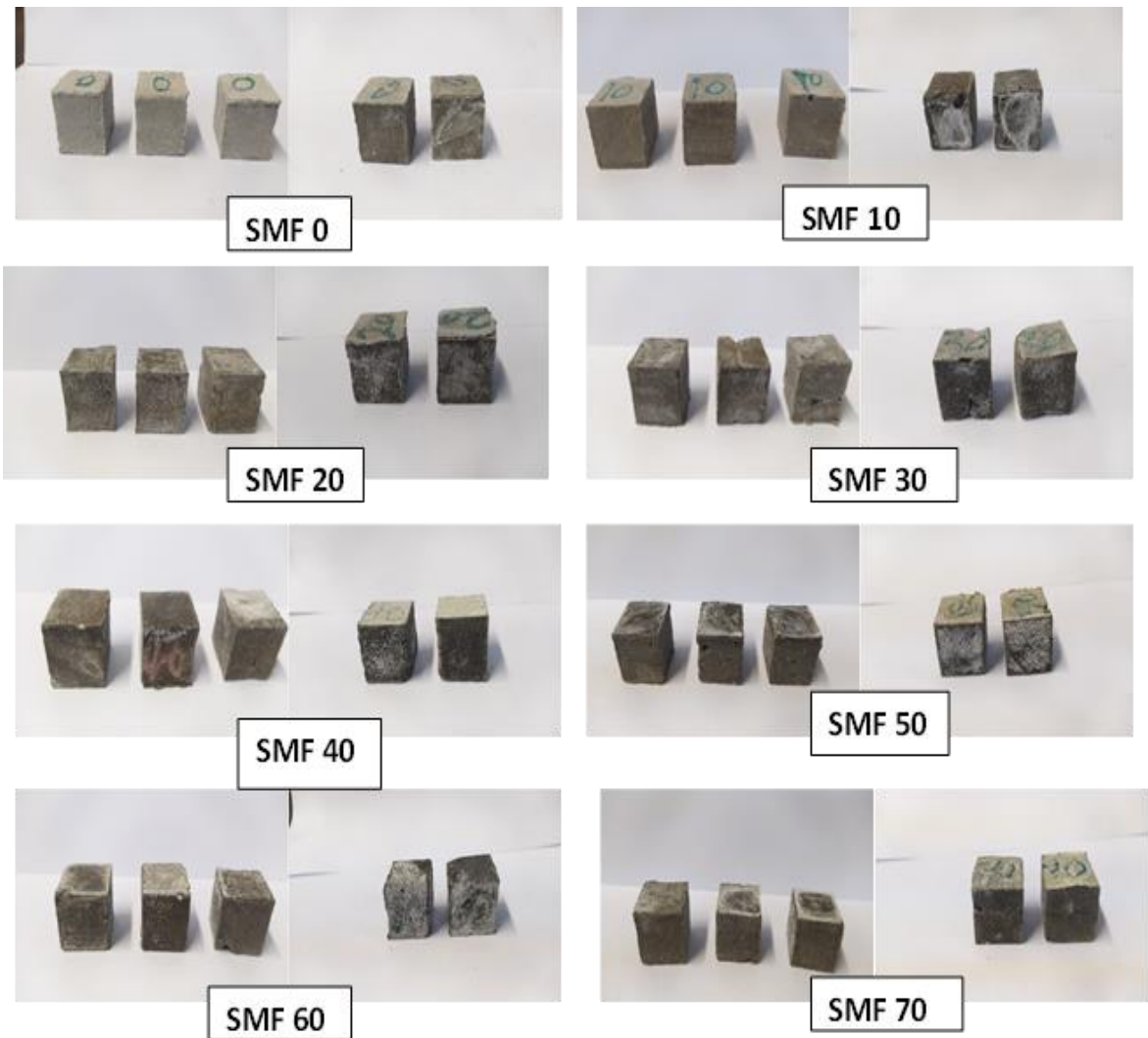


Figure 4-17: Photos of Paste samples after 3 and 6 months of sulfate exposure

4.8.2 Effect of Magnesium Sulfate on Strength Loss

Figure 4-18 presents the compressive strength loss of paste samples immersed in 2% magnesium sulfate solution for 3 and 6 months. From the figure, there was a strength loss between 20 and 56% at 3 months while it was in the range of 33-57% at 6-months. The minimum strength loss occurred at 10% SMF in comparison with the control mix. Also, the high content of SMF showed lower strength losses compared with the control mix.

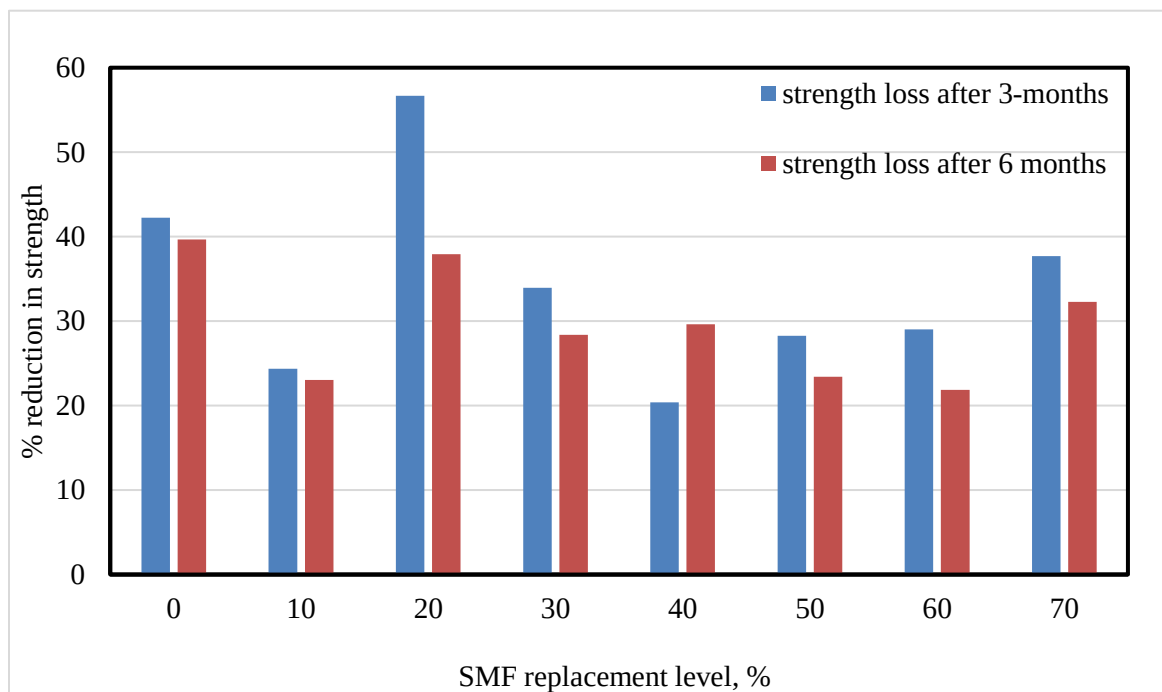


Figure 4-18: Compressive Strength loss due to sulfate exposure

4.8.3 Effect of Sodium Sulfate on Expansion of Prisms

The expansion values were increasing as the time prolongs as well as with SMF content as shown in Figure 4-19. At 10 days of exposure, small values of expansion were reported between 28.07 and 84.21 micro strains. As the exposure proceeds, expansion values became more pronounced, especially at higher replacement levels of SMF. For instance, at

206 days, values were in the range between 169.43 and 456.34 micro strains. It is also observed that higher SMF content exhibits more expansion in comparison with the control mix. The expansion noted in concrete mixes containing SMF can be attributed to the high alkalinity of the pore solution of these mixes that promotes the production of more ettringite. Moreover, SMF has high sulfate content that reacts with CH to produce gypsum to further reacts with C_3A to form ettringite.

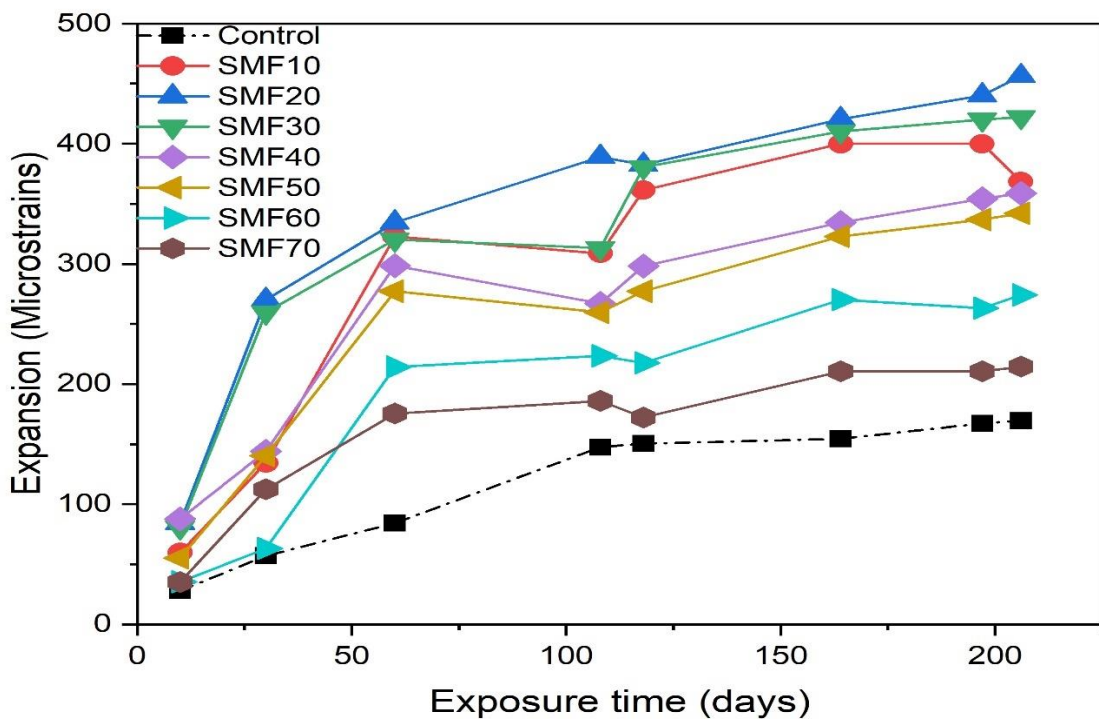


Figure 4-19: Expansion of prisms exposed to sodium sulfate solution

4.9 Reinforcement Corrosion

4.9.1 Corrosion Half-cell Potential

Reinforced concrete specimens prepared with cement and SMF were partially immersed in a 5% sodium chloride solution and the corrosion potential has been recorded for 243 days

using a saturated calomel electrode (SCE) as a reference electrode. Figure 4-20 shows the variation of half-cell potentials for SCC mixes with exposure time. The dotted red line shows the threshold limit of ASTM C876 [113] of corrosion potential (-270 mV) which represents 90% probability of initiation of corrosion. At the beginning of exposure, the corrosion potentials on steel rebars were in the range of -22 to -165 mV, more negative in specimens with higher content of SMF. The early age corrosion initiation in SCC mixes can be attributed to the higher permeability of the concrete matrix that permits the ingress of oxygen and moisture to initiate the corrosion. As the exposure proceeds, more negative values were recorded especially at later ages. For instance, the magnitude of corrosion potential on steel rebars of specimens prepared with 0,50,60 and 70% was -469.60, -450.30, -511.20, and -530.53 mV, respectively, after about 240 days. These values indicate a probability of more than 90% that the steel rebar was in a state of active corrosion.

The corrosion potential curves have been utilized to determine the time to initiation of reinforcement corrosion according as shown in Table 4-3. The time to initiation increased with an increase in SMF content up to 40%, reaching its maximum value of 99 days at SMF 40. Moreover, a substantial reduction in initiation time when partial replacement of cement exceeds 50%. The lower porosity of these mixes along with slow pozzolanic reaction could be accredited for this significant decline in their resistance against corrosion.

Table 4-3: Time to initiate reinforcement corrosion for SCC mixes

Mix ID	SMF 0	SMF 10	SMF 20	SMF 30	SMF 40	SMF 50	SMF 60	SMF 70
Time to initiate reinforcement corrosion, days	60	84	90	74	99	57	51	45

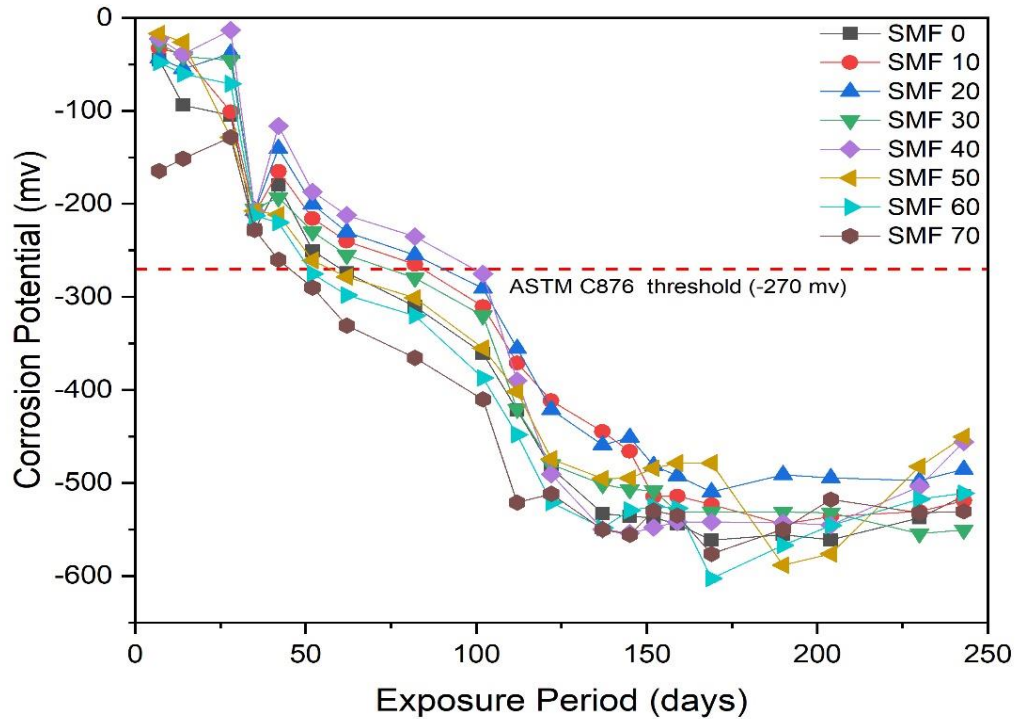


Figure 4-20: Variation of corrosion potential with exposure time for SCC mixes

4.9.2 Corrosion Current Density

The corrosion current density, I_{corr} , on steel rebars in SCC mixes immersed in 5% NaCl solution is plotted against the exposure period in Figure 4-21. The results showed a steady increase in corrosion current density with time, especially at later ages. All blended mixes attained lower values in comparison to the control mix. The maximum corrosion current density of $0.452 \mu\text{A}/\text{cm}^2$ was obtained in the control mix after 162 days of chloride exposure while the lowest occurred at SMF 30 with $0.024 \mu\text{A}/\text{cm}^2$. From the graph, it's clear that substitution levels of 10%-40% SMF exhibited better performance as compared to replacement levels of 50%-70%. This can be attributed to the less porous and dense concrete where SMF effectively filled the gaps between the aggregates and cement.

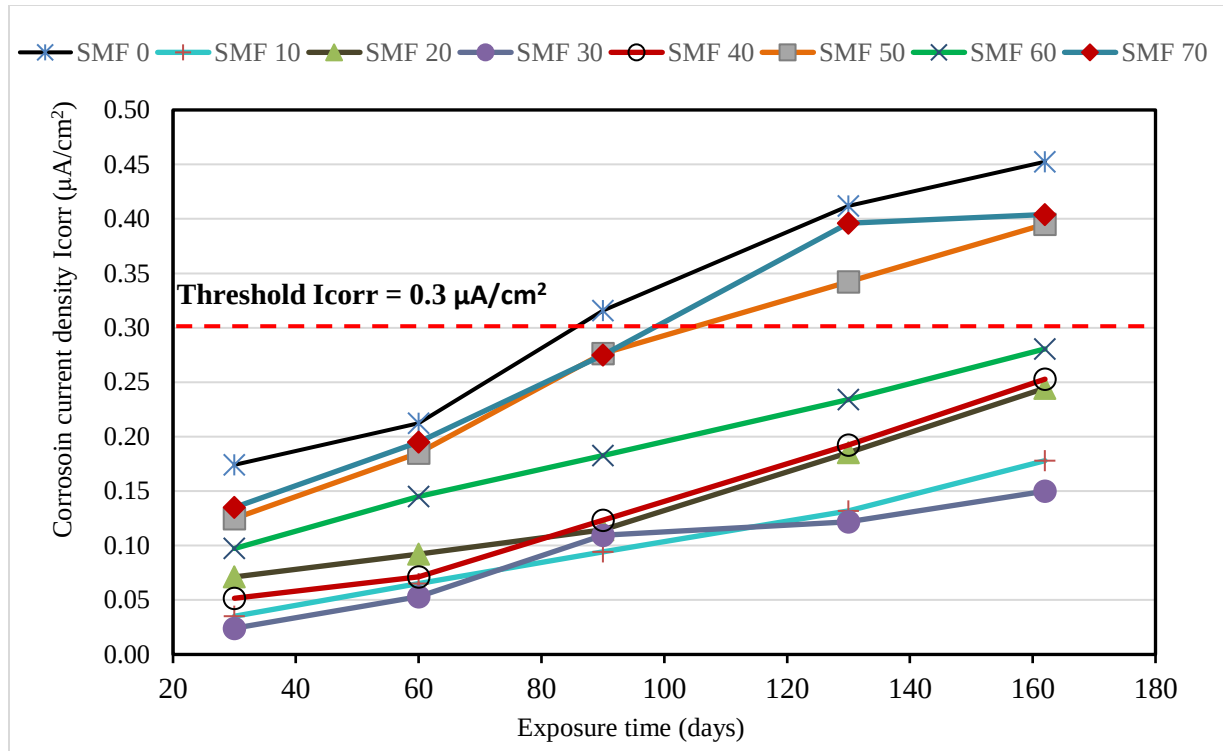


Figure 4-21: Variation of Icorr in SCC mixes

4.10 Chloride Diffusion

The chloride profiles for the concrete mixes prepared with and without SMF are shown in Figure 4-22. The chloride concentrations in terms of weight of concrete were determined using the absorbance values after exposing the specimens for 3-months to 5% NaCl solution. After that, the chloride profiles were employed to find the diffusion coefficients based on Fick's second law of diffusion: $C(x, t) = C_s - (C_s - C_i) \cdot \text{erf}\left(\frac{x}{2\sqrt{D_a t}}\right)$

Where $C(x, t)$: the chloride concentration at time t and depth x

C_s : the chloride concentration on the surface of the concrete

C_i : the initial chloride concentration before exposure to chloride

D_a is the coefficient of bulk diffusion (m^2/s) and erf is the gauss error function

The values of the chloride diffusion coefficient of concrete mixes obtained from Fick's second law of diffusion are depicted in Table 4-4. The results showed that all concrete mixes have chloride content near the surface above 0.6% by weight of concrete. The control sample and SMF 10 showed higher initial chloride concentration near the surface compared to other mixes. However, they reach lower values at higher depths. Contrary to the previous trends, the inclusion of SMF in SCC mixes increased the coefficient of chloride diffusion. All mixes except SMF 50 showed higher values of chloride diffusion coefficient compared to the control mix.

Table 4-4: Chloride diffusion coefficient of SCC mixes

MIX ID	SMF 0	SMF 10	SMF 20	SMF 30	SMF 40	SMF 50	SMF 60	SMF 70
$D_a (\times 10^{-12} \text{ m}^2/\text{s})$	14.7	17	46.9	19.9	54.9	3.54	47.2	100
Cs (%)	1.09	1.277	0.798	0.871	0.613	1.31	0.943	0.501

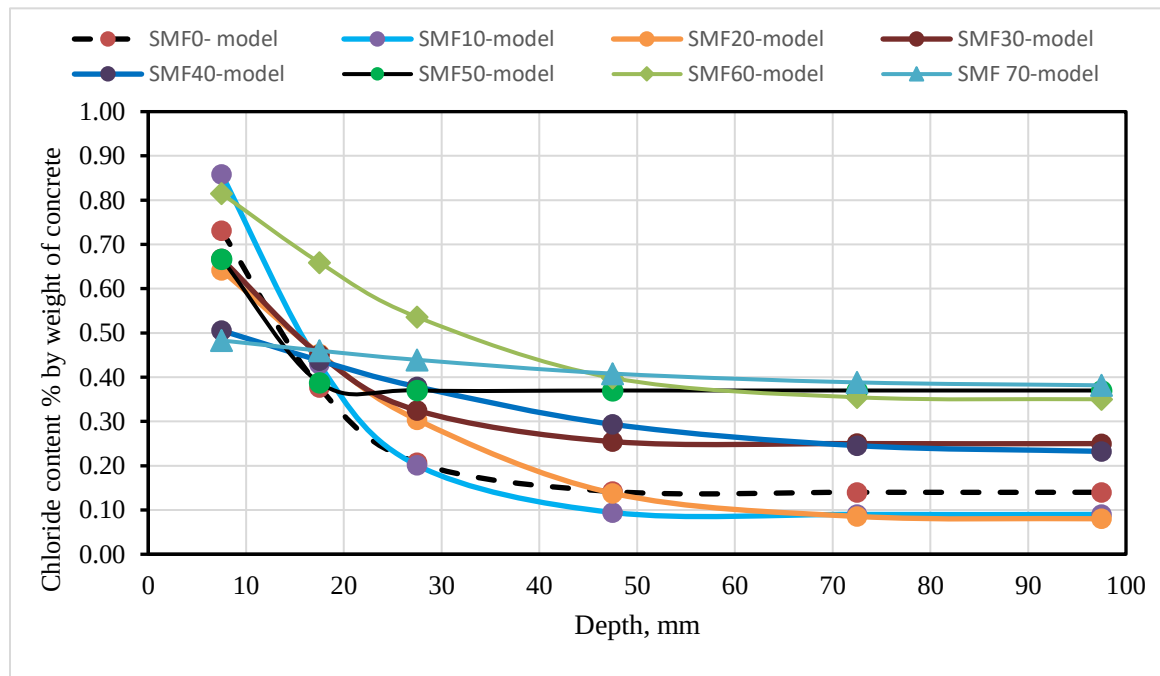


Figure 4-22: Chloride Profile of SCC mixes

4.11 Thermal Conductivity

The thermal conductivity of concrete disks was evaluated based on the steady-state hot plate method [121]. In this method, a heat stream flows through a unit thickness perpendicular to the surface area of the sample that is fixed between hot and cold plates to measure its thermal conductivity. In concrete, conduction is the primary mode of heat transfer at normal operating temperatures [121]. According to kim et al. [122], many factors are affecting the thermal conductivities of concrete, mortar, and cement paste such as age, amount of cement, type of admixtures, moisture content, and temperature. Moreover, the density and porosity of concrete have a strong correlation with thermal conductivity. The results of the 28-day thermal conductivity of all SCC mixes are schematically shown in Figure 4-23. The thermal conductivity (λ -value) of mixes blended with SMF from 10% to 20% has higher values (1.69 and 1.70 W/m.k) compared to the control mix (1.52 W/m.k). However, higher substitution levels of SMF beyond 40% resulted in a general reduction of thermal conductivity reaching the lowest value of 1.13 W/m.k at 70% SMF. This is attributed to the porous and less densified microstructure of mixes with the content of SMF above 50% compared to the mixes with lower percentages of SMF. In addition, concrete replaced with high content of SMF has lower density due to the low specific gravity of SMF which also explains the reduction in thermal conductivity of these samples. The thermal resistance of concrete mixes mainly depends on the concrete thermal conductivity and member's thickness. Here we consider a screed concrete in a roof slab having 10 cm thickness. The thermal resistance for the solid member is computed using the following equation [123]:

$$R_{slab} = \frac{L}{\lambda_{slab} \times A} \text{ (K/W)}$$

where A is the net area of the member

L is the thickness of the solid member (here it's a roof slab of 10 cm)

λ_{slab} is the thermal conductivity of the mix.

Now, the thermal resistance was determined assuming an area of meter square so that it

$$\text{becomes: } R_{slab} = \frac{L}{\lambda_{slab}} \text{ (m}^2 \cdot \text{K/W)}$$

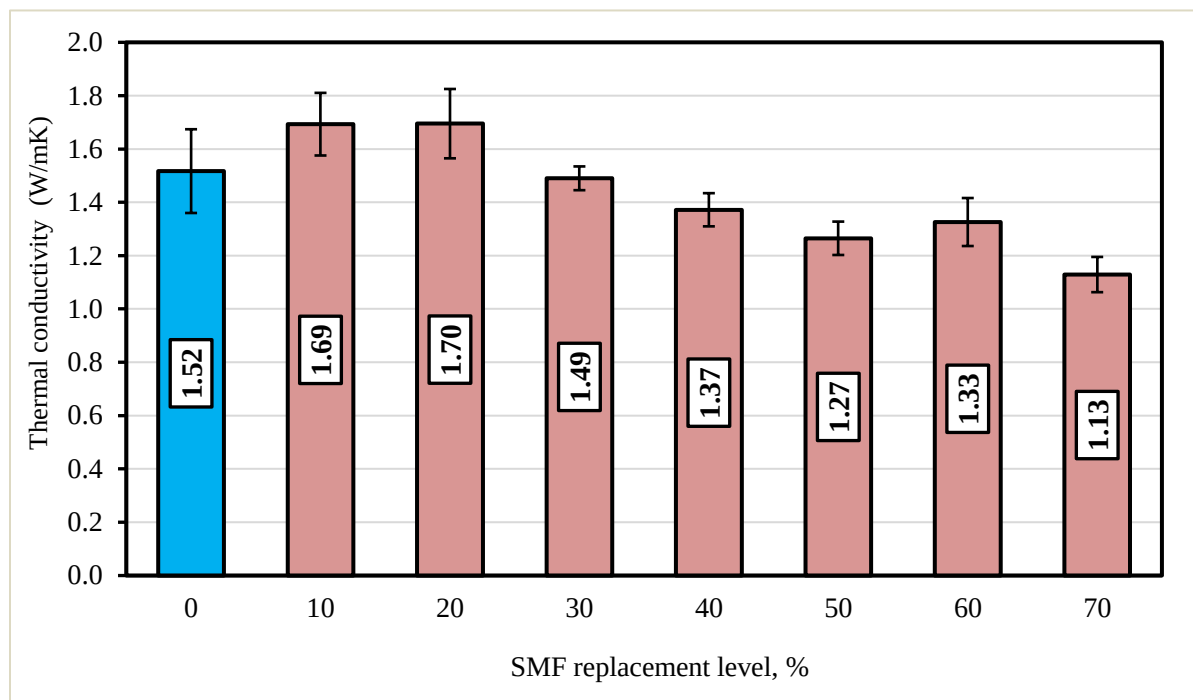


Figure 4-23: Influence of SMF on Thermal Conductivity of SCC mixes

Figure 4-24 shows that the thermal resistance of all concrete mixes is varying between 0.059 and 0.089 m². K/W. Compared to the control mixture, SMF content beyond 50% increases the thermal resistance by about 1.1 to 1.35 times. The thermal resistance of a

solid member is also depending on the member thickness. If the slab thickness has increased to 20 cm, the thermal resistance will reach 0.16 and 0.18 m². K/W in SMF 50 and SMF 70, respectively.

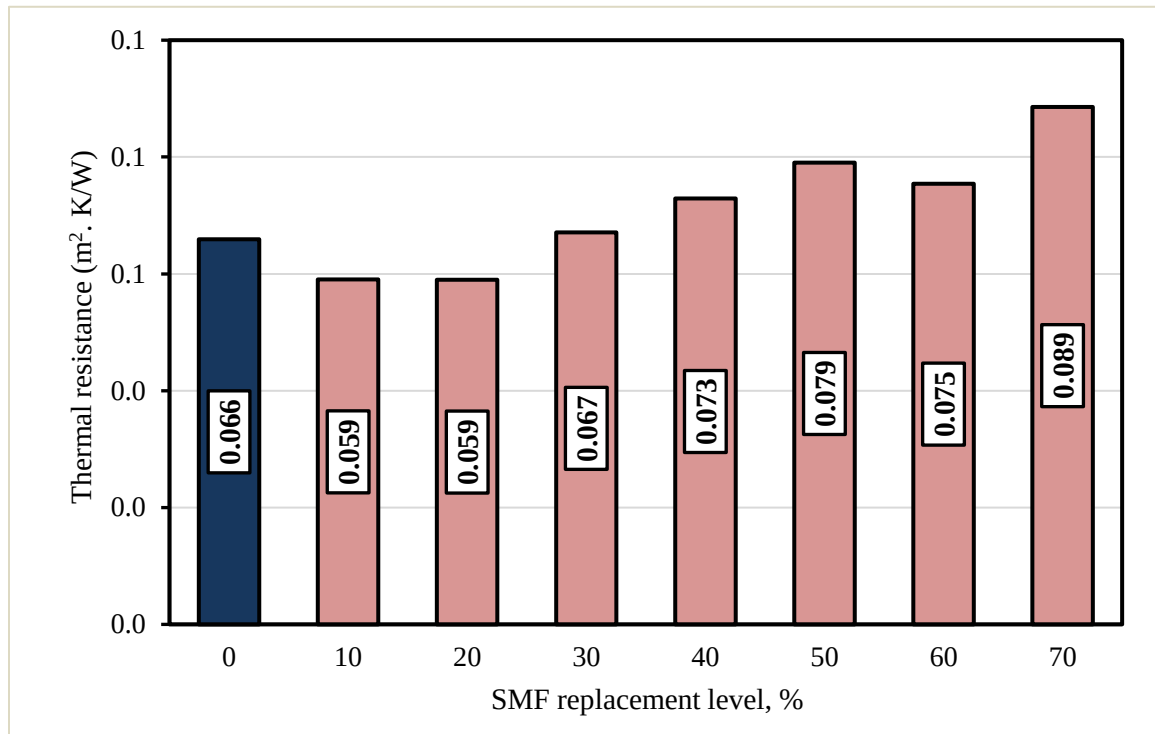


Figure 4-24: Influence of SMF on Thermal Resistance of SCC mixes

CHAPTER 5

CHARACTERIZATION OF PASTE SAMPLES

5.1 SEM

5.1.1 At 28 Days of Water Curing

The morphology of Paste samples cured in water for 28 days was examined using Scanning electron microscope (SEM) as shown in Figure 5-1 and Figure 5-2. At curing of 28 days, the control sample showed a denser microstructure with some hydration products such as portlandite, C-S-H, calcite, and ettringite. However, small pores and cracks manifest in their microstructure. When SMF is incorporated in paste samples at 10% and 20%, a better microstructure was observed with minimum numbers of voids and pores. This is due to the micro filling effect of SMF in which it closes the pores and voids. Moreover, SMF could participate in the chemical reaction to produce secondary C-S-H gel to densify and refine

the microstructure. The inclusion of high content of SMF resulted in a less densified microstructure with the presence of more voids, cracks, and pores in their morphology.

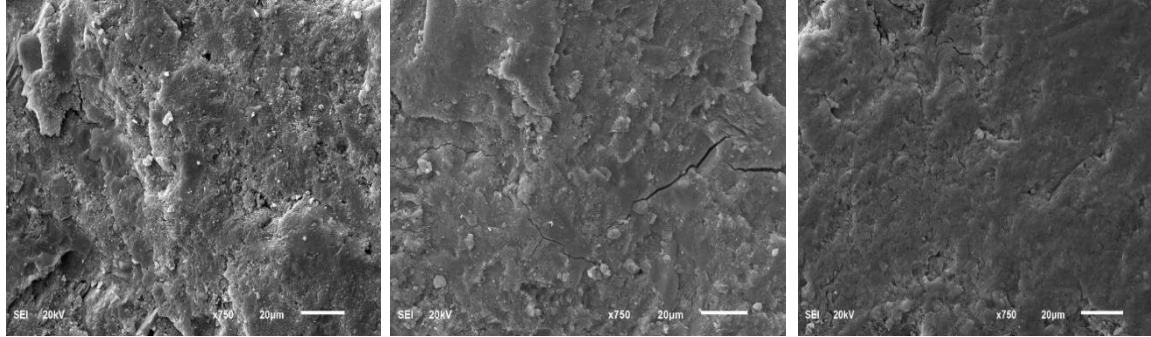


Figure 5-1: SEM image of control (left), SMF 10 (middle), SMF 20 (right) at 28 days of water curing

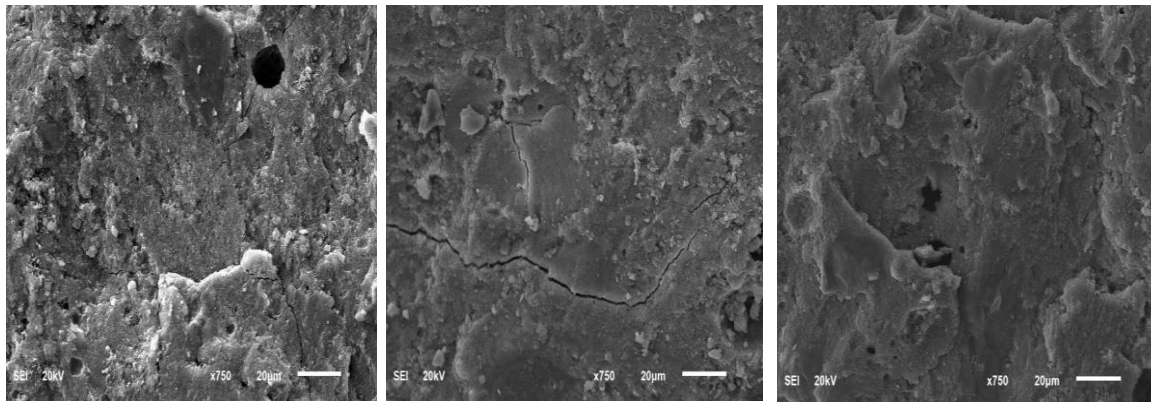


Figure 5-2: SEM images of SMF 40 (left), SMF50 (middle), and SMF70 (right) at 28 days of water curing

The elemental composition at the selected points in some samples are shown in Figure 5-3, Figure 5-4, and Figure 5-5. Main elements identified in the control mix are Ca, Si, O, and Al while in blended mixes Si, Ca, Mn, and K are embedded in the gel matrix. Minor or trace quantities of Mg, Al, Na, and Fe were also detected in the developed microstructure of these samples. EDS analysis was carried out on eight different spots in two regions of paste samples to give information about the available elements in these samples. The possible formation of gel products was studied by plotting ternary diagrams where each

axis represents the molar ratio of either Mn, K, and Si or Si, Al, Na normalized with respect to Ca as shown in Figure 5-6. In these plots, the spectrum of control paste samples only existed at the vertex of Si/Ca due to the absence of Mn and K in their elemental composition. The addition of SMF shifted the points from the vertex to the region between the borders of Mn/Ca and Si/Ca axes. The average molar weights of Ca showed a decreasing trend with the increase of SMF due to the reduction in cement content while the molar weights of Mn showed an increasing trend as depicted in Table 5-1.

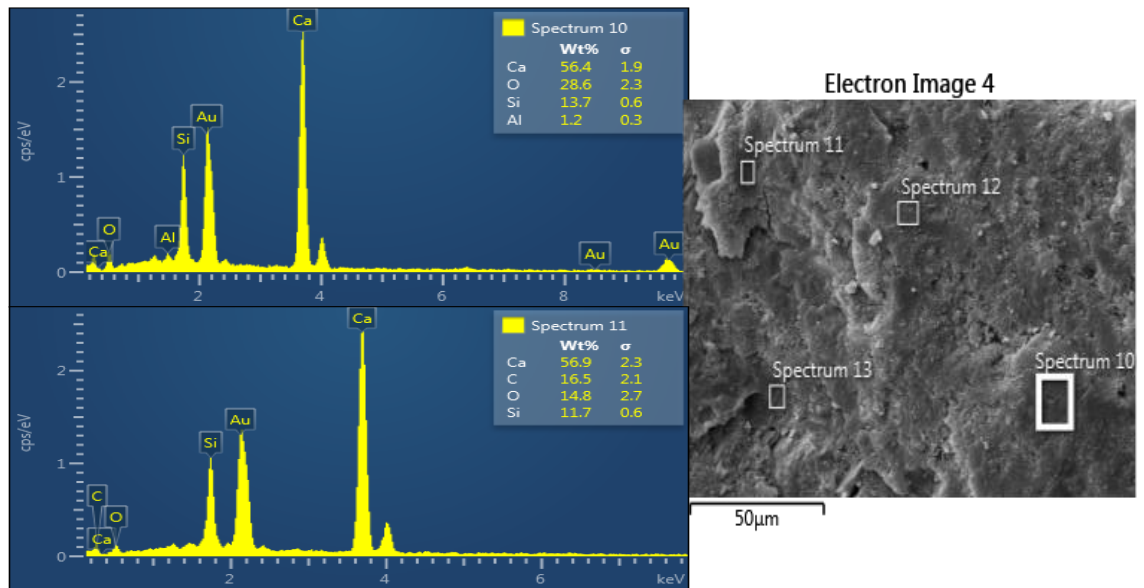


Figure 5-3: SEM image and EDS spectra of SMF0

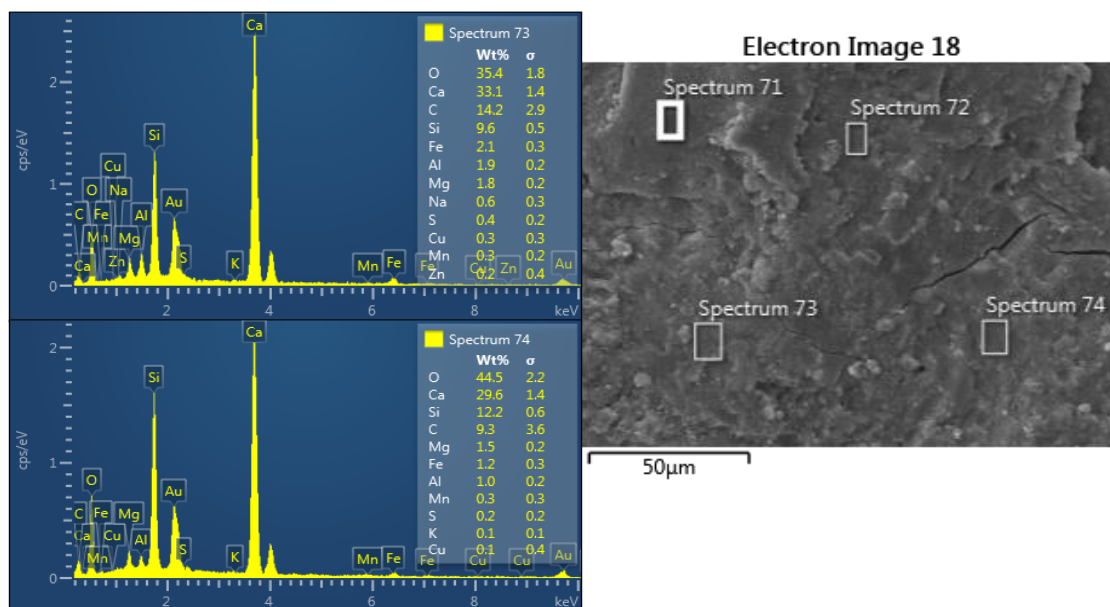


Figure 5-4: SEM image and EDS spectra of SMF 10

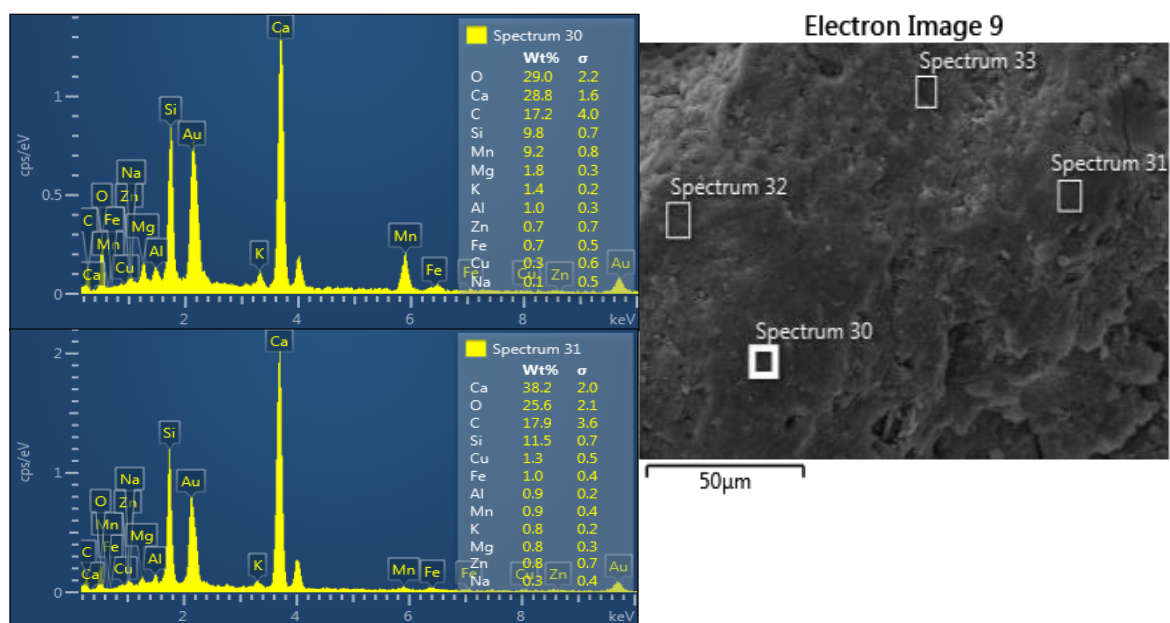


Figure 5-5: SEM image and EDS spectra of SMF 20

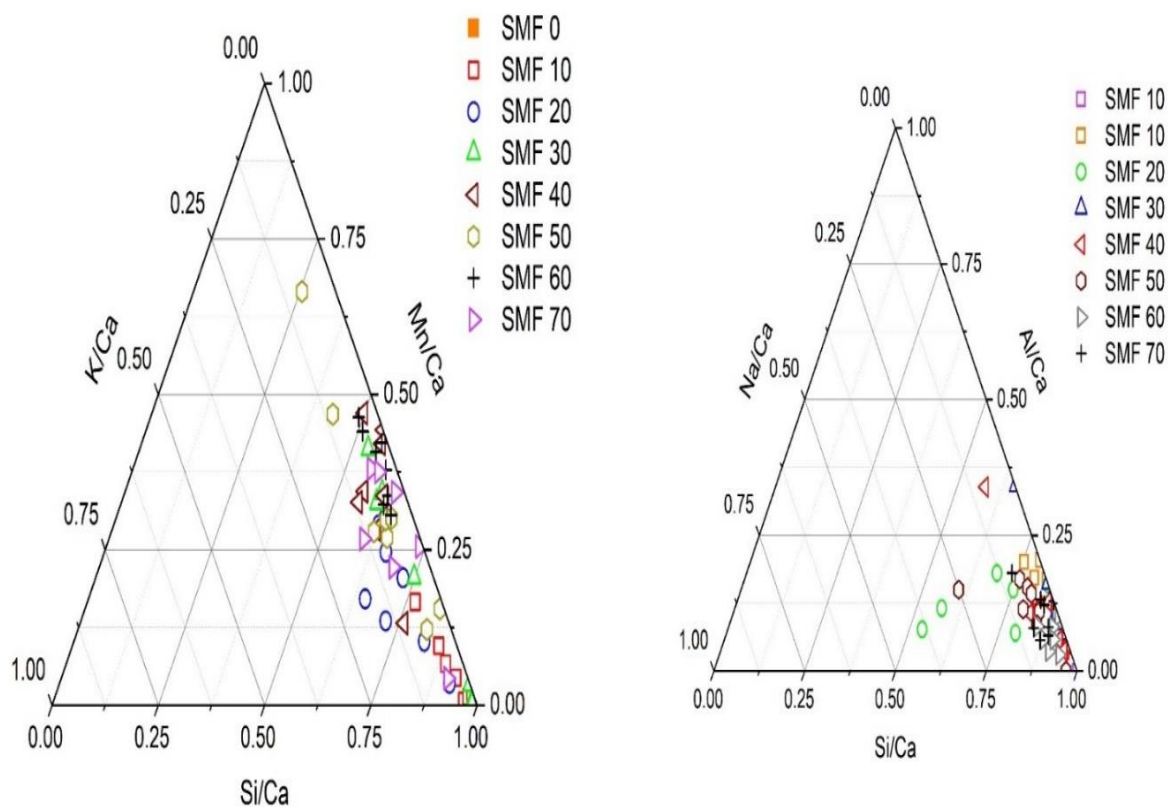


Figure 5-6: Ternary diagrams of molar ratios of paste samples

Table 5-1: Average molar weights and ratios of elements in paste samples

Molar weights and ratios	SMF 0	SMF 10	SMF 20	SMF 30	SMF 40	SMF 50	SMF 60	SMF 70
Si	9.59	7.26	6.45	7.03	8.53	5.16	9.09	10.90
K	0.00	0.22	0.63	0.47	0.56	0.68	0.53	0.94
Ca	25.84	18.63	23.45	17.98	14.91	11.03	10.35	15.58
Mn	0.00	0.36	1.48	2.79	4.94	2.59	5.52	5.51
Al	0.88	1.13	1.00	1.23	1.02	0.72	0.72	1.38
Na	0.00	0.17	1.23	0.00	0.35	0.50	0.46	0.86
Si/Ca	0.37	0.39	0.27	0.39	0.57	0.47	0.88	0.70
Mn/Ca	0.00	0.02	0.06	0.16	0.33	0.23	0.53	0.35
Al/Ca	0.03	0.06	0.04	0.07	0.07	0.07	0.07	0.09
Na/Ca	0.00	0.01	0.05	0.00	0.02	0.05	0.04	0.06
k/Ca	0.00	0.01	0.03	0.03	0.04	0.06	0.05	0.06

5.1.2 After 1 Month of Acid Exposure

The morphology of Paste samples after exposure to 2% of sulfuric acid for 1 month was studied using SEM as shown in Figure 5-7 and Figure 5-8. In these figures, the surface manifested to be highly porous with the presence of voids and cracks. A noticeable amount of C-S-H gel was noticed to be decomposed into smaller particles. The presence of gypsum in the morphology is a clear sign of its formation due to its possible reaction between acid and portlandite.

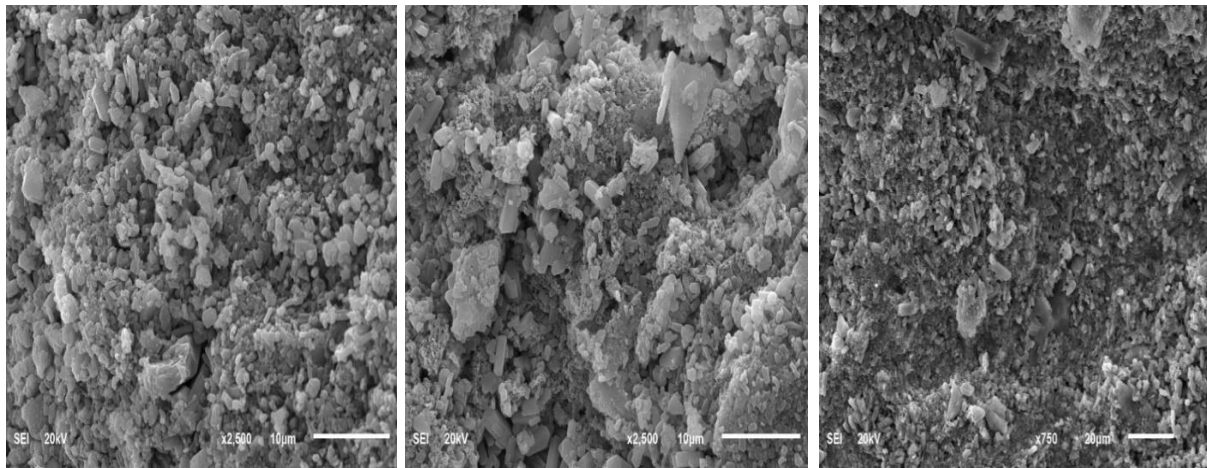


Figure 5-7: SEM images of SMF0 (left) to SMF 20 (right) samples immersed in acid for 1 month

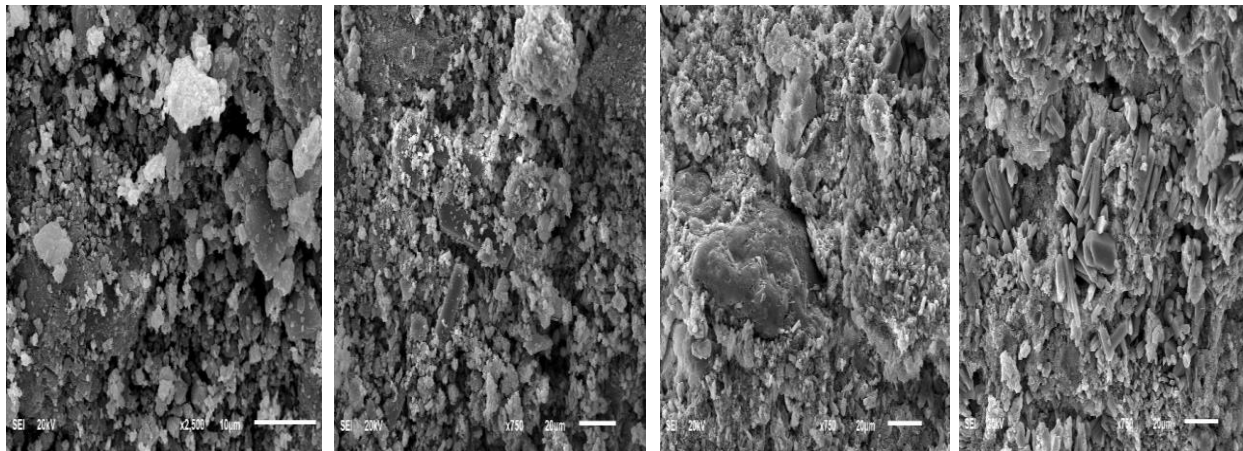


Figure 5-8: SEM images of SMF40 (left) to SMF 70 (right) samples immersed in acid for 1 month

The main elements identified in most of the paste samples exposed to 1 month of sulfuric acid include O, Ca, Si and S while minor traces of Al, Mn, Fe, and Na were also present in the EDS of some of these samples as shown in Figure 5-9, Figure 5-10 and Figure 5-11. The presence of Ca, Si, S, and O confirms the formation of gypsum due to acid attack which is the main reason for the deterioration of the samples.

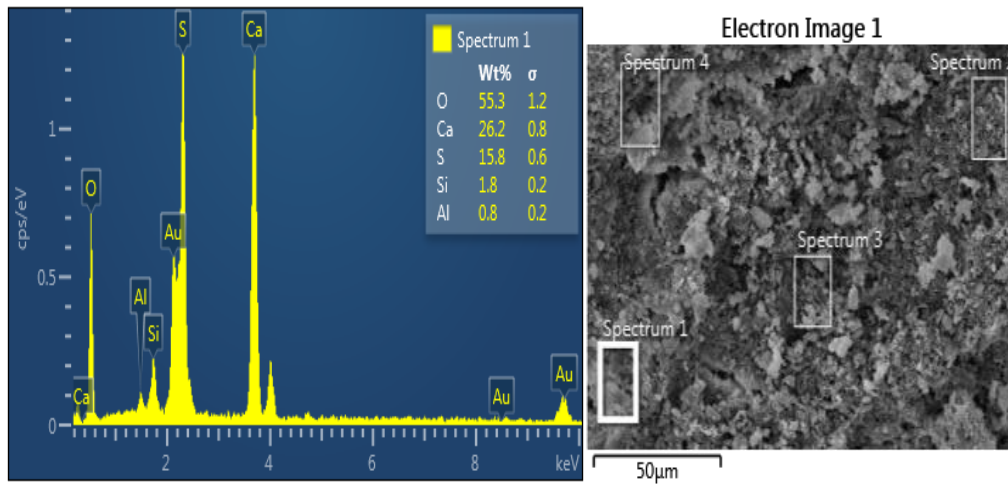


Figure 5-9: SEM image and EDS of SMF0 exposed to acid

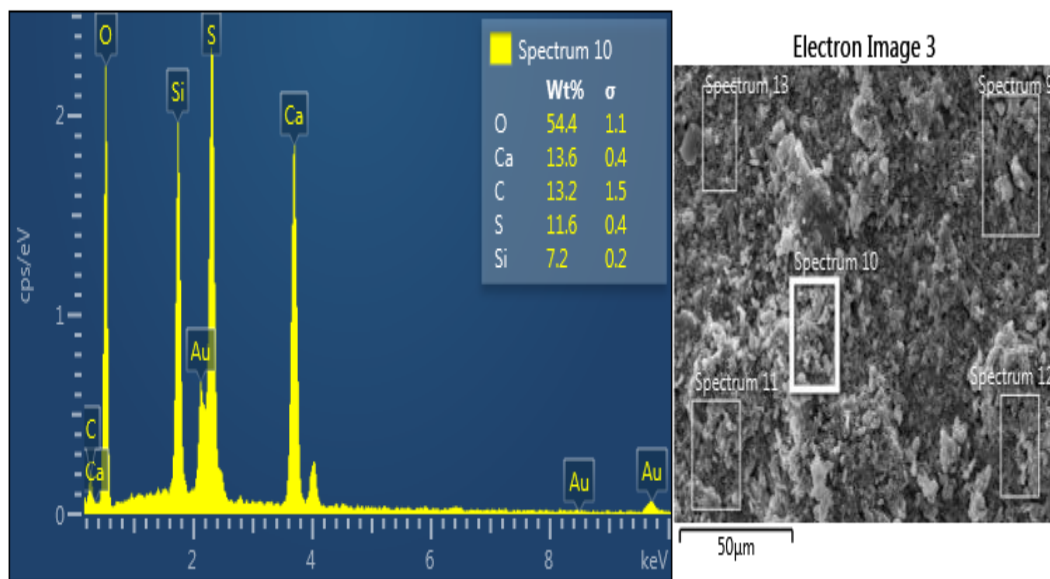


Figure 5-10: SEM image and EDS of SMF10 exposed to acid

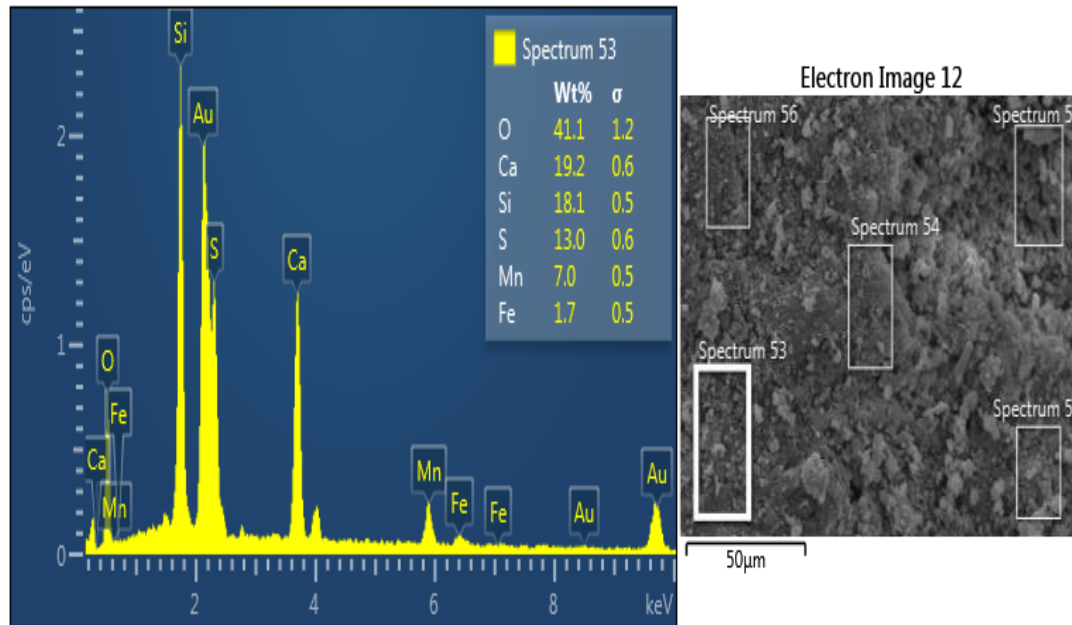


Figure 5-11: SEM image and EDS of SMF50 exposed to acid

5.2 XRD

5.2.1 At 28 Days of Water Curing

Figure 5-12 presents the XRD spectra of the paste samples which were cured in water for 28 days. The XRD spectra of hydration products of these pastes are mainly comprised of ettringite, portlandite, alite, belite, and calcite, in which portlandite is the major phase. In all these samples, peaks of ettringite (Aft) were identified at 2-theta of 9.1° , 15.83° , and 22.9° while peaks of calcite (CaCO_3) were observed at 2-theta of 29.4° , 39.4° . In addition, portlandite (Ca(OH)_2) peaks were detected at 2-theta of 18.1° , 28.6° , 34.1° , 47.1° while the peak of Calcium silicate hydrate gel (C-S-H) was manifested at 2-theta of 29.4° . Moreover, smaller peaks for Belite ($2\text{CaO} \cdot \text{SiO}_2$) were identified at 2-theta of 32.2° and 43.2° . The results showed that the peak intensity of portlandite decreases with the addition of SMF,

which indicates that the inclusion of SMF reduces the CH content in SMF-blended pastes. These can be attributed to the decrease in the content of cement when replaced by SMF which subsidizes the decrement of portlandite as shown in XRD patterns. Moreover, the low pozzolanic reactivity of SMF could play a role in further reducing the CH content in SMF-blended pastes and the formation of C-S-H as a secondary hydration reaction. Higher intensity of peaks of C-S-H was identified at 0%,10%, and 20% of SMF due to the hydration reaction and more cement content. Paste samples containing beyond 50% SMF showed lower peaks of C-S-H due to the dearth of cement required for hydration. The existence of calcite peaks in these samples is mainly ascribed to the ingress of CO₂ during the curing process to form CaCO₃. Figure 5-12 shows the variation of intensities of C-S-H peaks in paste samples at 28 days of water curing.

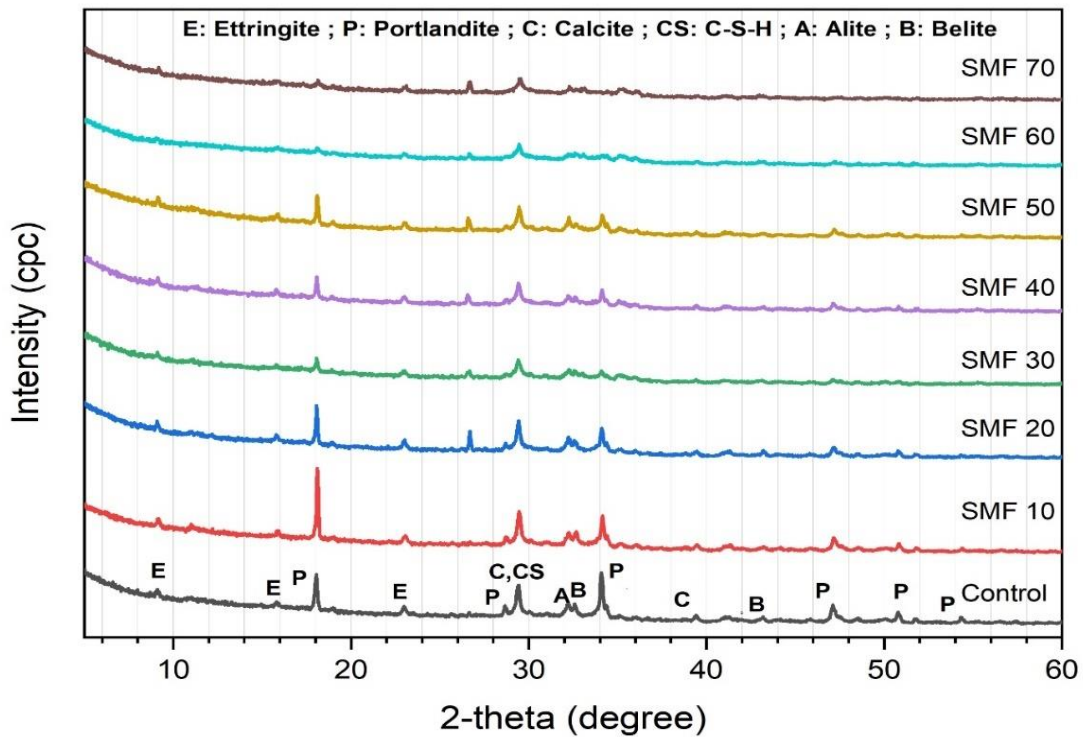


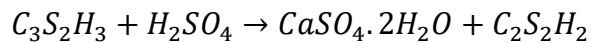
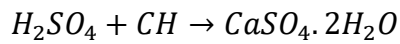
Figure 5-12: XRD spectra of paste samples at 28 days of water curing

Table 5-2: The variation of C-S-H intensity in paste samples at 28 days

MIX ID	SMF 0	SMF 10	SMF 20	SMF 30	SMF 40	SMF 50	SMF 60	SMF 70
C-S-H intensity	4095	4325.41	3826.67	2808.75	3144.17	3371.67	2473.33	24423.8

5.2.2 After 1 Month of Acid Exposure

The XRD pattern of the paste samples exposed to 2% H₂SO₄ for one month is depicted in Figure 5-13. It can be observed that peaks of ettringite (Aft) were located at 2-theta of 9.08°, 11.61°, and 22.96° while peaks of portlandite (Ca(OH)₂) were observed at 2-theta of 18.03° and 34.06°. Moreover, Gypsum (Ca(SO₄)·2H₂O) peaks were detected at many angles and specifically at 2-theta of 20.72°, 23.36°, 29.09°, 31.08°, 33.35°, 40.65°, 43.41°, 47.92° and 50.30° while the peak of Calcium silicate hydrate gel (C-S-H) was identified at 2-theta of 29.09°. Since the XRD spectrum was taken after only one month of exposure, the intensity of peaks will be more if the specimens were exposed and analyzed after 3 and 6 months. Moreover, the formation of peaks of ettringite and gypsum is the main reason behind the deterioration of concrete and the reduction in strength due to sulfuric acid attack. Gypsum can be formed due to the reaction between sulfuric acid with portlandite or sulfuric acid with C-S-H as shown below:



Moreover, the expansion of concrete due to the formation of ettringite results in more stress and cracking of concrete. The main reaction for the formation of ettringite due to sulfuric acid is shown below:

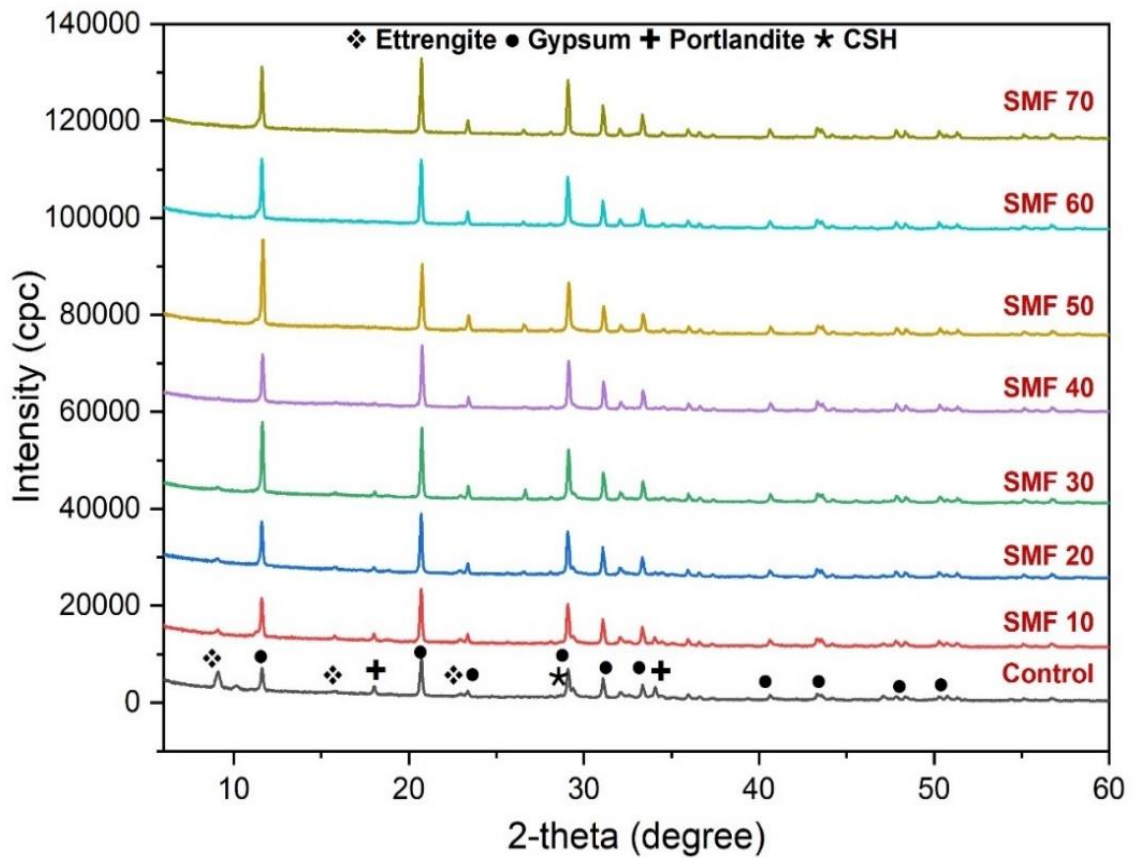
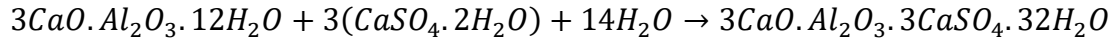


Figure 5-13: X-ray pattern of paste samples after one month of exposure to 2% sulfuric acid

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

This research was conducted to develop an eco-friendly SCC utilizing an indigenous waste material, SMF, at substitution levels of 10% to 70% of OPC by weight. This material was sourced from a local ferroalloy plant in Jubail, Saudi Arabia for the detailed investigation of its performance in SCC. The mix design was prepared using a binder content of 500 kg/m³, a water to binder ratio of 0.4, and fine to coarse aggregate ratio of 1. The following conclusions can be deduced from the previous results:

1. The agglomerated spherical particles of SMF combined with small dosages of SP had positive effects on the workability and flowability of concrete mixes. In addition, concrete mixes containing up to 70% SMF met all the requirements of flowability criteria in EFNARC.
2. Initial and final setting times prolonged with the increase in SMF content due to the slower reactivity of SMF and the delayed product formation.
3. The temperature profile of paste samples showed a clear retardation effect with the inclusion of SMF. Accordingly, the use of SMF has generally increased the dormant period, the peak temperature, and the time to reach the peak. In addition,

strong correlations between setting times with dormant periods and peak temperatures were developed.

4. The compressive strength of concrete mixes with SMF up to 20% was superior to that of the control mix at all curing ages due to the physical and chemical effects of SMF in densifying and refining the microstructure. However, the strength showed a clear retrogression when SMF content increased above 30% due to the dilution effect. The deterioration of strength in these mixes is attributed to the increase in porosity and cracks and the lower formation of gel products.
5. The concrete mixes having SMF between 10 to 30% were classified as low permeability while the control mix and mixes replaced by over 40% SMF were designated in the moderate category. The values of chloride permeability decreased by 45% and 26% for SMF levels of 10% and 30%, respectively. Bulk electrical resistivity results showed an enhancement in these samples compared to the reference mix due to the physicochemical effect of SMF. Furthermore, the electrical resistivity of concrete specimens showed a decreasing trend with the increase in moisture content due to the passage of electric current in concrete pores.
6. The use of SMF above 50% has enhanced the thermal resistance compared to the control mixture due to its porous microstructure and lower density which can be practical in the production of thermally-isolated structures.
7. All concrete mixes exhibited an increase in corrosion potential and corrosion current density with time. The time to initiate the reinforcement corrosion was delayed in replacement levels up to 40% of SMF compared to the control mixture.

8. Concrete specimens blended with SMF have experienced a visible physical deterioration and disintegration after 3 and 6 months of 2% sulfuric acid exposure as well as a higher reduction in strengths owing to the formation of corrosion products such as gypsum. Concrete mixes containing 20% and 30% performed better in acid exposure in terms of strength reduction.
9. Paste samples exposed to 2% magnesium sulfate were not affected physically and only white salt deposits were observed on their surfaces. Moreover, prisms with blended SMF that were immersed in 2% sodium sulfate exhibited higher expansion compared to the control mix.
10. There were good linear correlations between some properties like rapid chloride permeability, electrical resistivity, and strength.
11. SEM images of paste samples at 28 days revealed a dense and uniform microstructure in specimens containing 10% to 40% of SMF while the other mixes were porous and nonuniform.
12. XRD of paste samples at 28 days showed the formation of portlandite (calcium hydroxide), Calcium silicate hydrate (C-S-H), Calcite (CaCO_3), Albite, and Ettringite. Maximum intensities of C-S-H were observed in the control sample, 10% SMF and 20% SMF. Moreover, portlandite intensities are reduced at the high content of SMF due to the lower cement content.
13. XRD of paste samples exposed to acid showed the formation of gypsum in many peaks in all samples as the main contributor to the deterioration of samples. However, ettringite is mainly formed in the control sample due to the fast reaction

of available tricalcium aluminate. The intensity of Portlandite peaks were reduced due to the consumption of its content in reaction with acid to produce gypsum.

6.2 Recommendations

Based on the above conclusions, the following recommendations and practical avenues are hereby proposed:

1. Limited content of SMF (not exceeding 20%) should be employed to develop a high-strength SCC with high durability applications especially chloride permeability, reinforcement corrosion resistance, and electrical resistivity.
2. SCC mixes prepared with SMF content above 30% can be employed in normal or low strength concrete with medium or lower durability performance and higher thermal resistance.
3. It's recommended to blend SMF with other supplementary cementitious materials such as GGBS in concrete mixes to get a better synergetic effect on strength and durability properties.
4. SMF can be effectively used to replace cement in mass concrete or in an application where the heat of hydration needs to be minimized. This may help to reduce the effects of high temperature on concrete, but this needs to be more investigated in future studies.

6.3 Suggestions for Future Research

This study is the basis for further research in concrete technology and structural applications. The suggested future topics are listed as follows:

1. Study other durability properties of concrete such as water absorption, carbonation resistance, sorptivity, freeze/thaw performance, seawater resistance, fire resistance, etc.
2. Develop SCC in which SMF is blended with other local waste materials (such as fly ash, slags, metakaolin, etc.) to get the best synergistic performance in such combinations.
3. Study the rheological properties of SCC mixes and relate them to fresh and hardened properties.
4. Study the pumping of the developed SCC mixtures and the lateral pressure on formwork.

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VITAE

Name : MAZEN ANWAR SAEED ABDULLAH

Nationality : YEMEN

Date of Birth : 1/24/1994

Email : abdullahmazen1995@gmail.com

Address : KFUPM, DHAHRAN, 31261, SAUDI ARABIA

Academic Background : **B.Sc. Civil Engineering**, (2013 - 2018),
KFUPM, Dhahran, Saudi Arabia

M.S Civil Engineering, Structures, (2019 - 2022),
KFUPM, Dhahran, Saudi Arabia

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- Gawah, Q., Al-Osta, M.A., Maslehuddin, M., Abdullah, M.A., Shameem, M., Al-Dulaijan, S.U.: Development of sustainable self-compacting concrete utilising silico manganese fume. European Journal of Environmental and Civil Engineering. 1–22 (2022).