Republic of Iraq
Ministry of Higher Education and Scientific Research
University of Babylon
College of Materials Engineering
Department of Ceramics Engineering and Building Materials

Synthesis of Self-Leveling Geopolymer Material

A Thesis
Submitted to the Council of the College of Materials Engineering/University of Babylon in Partial Fulfillment of the Requirements for the High Diploma Degree in Materials Engineering/Ceramic

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2022 A.D. 1443 A.H

بسم الله الرحمن الرحيم

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Dedication

To my family, friends, and anyone who inspired me.

Zaid 2022

Acknowledgments

First of all, profusely all thanks be for **allah** lord of all creations who enabled me to achieve this work.

I would like to express my deep thanks to the college of materials engineering/ university of babylon for giving me the chance to complete my study.

I would like to express my deep gratitude and appreciation to my supervisor **prof. Imad ali disher al-hydary** (ph.d.) For his advice, support and encouragement they gave me during the course of work. His suggestions and moral support in difficult times have been essential for completing this work. I am very proud to achieve my work under his supervision.

I want to express my appreciation and gratitude to the head of department, **prof. Shaker j. Edrees (ph.d.)**, all academic staff, and support staff in the ceramics department.

My deep appreciation goes to the support staff of ceramic materials laboratories, especially **eng. Mahmood, eng. Ibtisam, tech. Ashwaq, eng. Khansaa, eng. Homam, eng. Ghaida, eng. Rehab** for their help.
I would also like to thank all my friends and colleagues during the study in the college, for creating an unforgettable environment in which my research was carried out, especially, support staff in libraries, college of materials engineering, University of Babylon

Abstract

Geopolymer is a relatively new material that has attracted the attention of numerous academics due to its lower environmental pollution than traditional portland cement concrete, the production of Portland cement is a high energy consumption material that emits a large amount of carbon dioxide (CO2) into the environment. Self-leveling material is mostly applied on the ground surface, it provide more level, high-performance, and easy-to-apply flooring. Where it may solidify into a flat, wear-resistant surface without the need for vibrating or plastering. The self-leveling materials also reduce the damage that may occur as a result of using traditional methods of leveling floors.

Usually, epoxy is used as a self-leveling material, or superplasticizers with a polymeric basis are added to concrete mixtures to increase workability and reduce viscosity, and the concrete becomes self-leveling concrete. These materials (epoxy, self-leveling concrete) are characterized by their relatively high cost.

In this study, we investigate the possibility of using geopolymer as a low cost self-leveling material without using any additives.

Geopolymer have been prepared from Metakaolin as a source of alumina (Al_2O_3) and silica (SiO_2), Sodium hydroxide (NaOH) and aqueous sodium silicate (Na_2SiO_3) as an alkaline solution. XRD and particle size analysis test were performed for the started materials (MTK). Several mixtures have been designed, Samples were prepared for two groups (G1, G2) that are equal in the amount of added water and different in amount of sodium silicate. In addition, each group contains 4 mixing ratios with different water amount used to determine the optimum mixing proportion of reactant materials. In addition, doing the fluidity, setting time and density tests of the geopolymer paste Compressive strength, density and shrinkage tests were performed after geopolymer solidification.

The flow value has been obtained for the G1 samples within range (150 mm, 200mm) and the spread % was ranged (294.7%, 426.3%). The flow value has been obtained for the G2 samples within range (117.5 mm, 175mm) and the spread % was

ranged (209.2%, 360.5%). Self-leveling properties were selected mixtures having a flow of ≥ 150mm.

The self-leveling characteristics were obtained and the highest compressive strength of the geopolymer paste (65.39 MPa at 28 days old) was relatively high. In comparison to the sample (G1.3) containing the same amount of water of (G2.3), the results showed that sample (G2.3) had a higher compressive strength, higher density and lower shrinkage rate. Which indicates a higher degree of polymerization reaction.

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List of Abbreviations

Abbreviation	Meaning
BA	Coal bottom ash
DTA	Differential Thermal Analysis
FA	Flay ash
GSLM	Geopolymer self-leveling material
ICDD	International Center of Diffraction Data
M	Molarity
MTK	Metakaolin calcined at 750 ˚C
NMR	Nuclear magnetic resonance
OPC	Ordinary Portland cement
PAC	Phosphor anhydrite from the calcination
PG	Phosphor gypsum
PTFE	Polytetrafluoroethylene
R&D	Research &development
RHA	Rice husk ash
S/L	Solid/liquid
SLC	Self-leveling concrete
SLM	Self-leveling material
SLU	Self-Leveling Underlayments
SS/SH	Sodium silicate/ sodium hydroxide
TG	thermogravimetric analysis
XRD	X-Ray Diffraction

CHAPTER ONE

INTRODUCTION

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INTRODUCTION

1.1 Overview

Cement is one of the most popular building materials in the construction industry. The creation of Portland cement is leading extensive use of energy material that emits a significant quantity of CO_2 into the environment. The expenses of meeting these energy requirements are high. [1]

1 ton Portland clinker manufacture produces (0.55 ton) of CO_2 directly and the carbon fuel that uses to production an extra (0.40 ton) of CO_2.
to simplify: 1 ton from Portland cement production 0.95 ton from CO_2.
$5CaCO_3 + 2SiO_2 \rightarrow 5CO_2 + (3CaO,SiO_2)(2CaO,SiO_2)$ [2]
As a result, more research into cementitious products is required to decreased environmental consequences and enhanced economic benefits.

An novel substance is geopolymer cement and it is used as a substitute for ordinary portland cement for use in the basic structure of transportation, construction and off coast applications. Whether to use industrial materials or minimally processed natural materials by products to considerably limit its carbon's effects, while also being very resistant to possible problem deterioration in durability occur in traditional concrete.

Geopolymer cement is an amorphous substance composed of molecular units (monomers) linked together by a covalent bond. Geopolymer is regarded as novel material with distinct features. [3]

There are two major types of aluminosilicate materials, commercial by-products such as blast furnace slag, fly ash (FA), and rock-based minerals such as kaolin and metakaolin, among others, have been used to make geopolymer cement. The recycled use of these materials in building will minimize the cost of disposal elsewhere and, as a result, the overall cost of concrete making. [1]

Davidovits invented the term "geopolymer" the prefix "geo" refers to an aluminosilicate that is inorganic and based on geological components that interacted creating a binder using an alkaline solution via polycondensation

reaction. This is followed by a curing procedure at room temperature or somewhat higher temperatures in the 20–100 ℃ range. The geopolymerization reaction is the process by which geopolymers are created. [4]

Geopolymer, in other terms creation entails the production of solid-liquid combinations (figure 1). The solid consists of powdered aluminosilicate sources containing an appropriate percentage of silica and alumina with higher reactivity. While the liquid of alkaline solution to form binder through polycondensation reaction. [3]

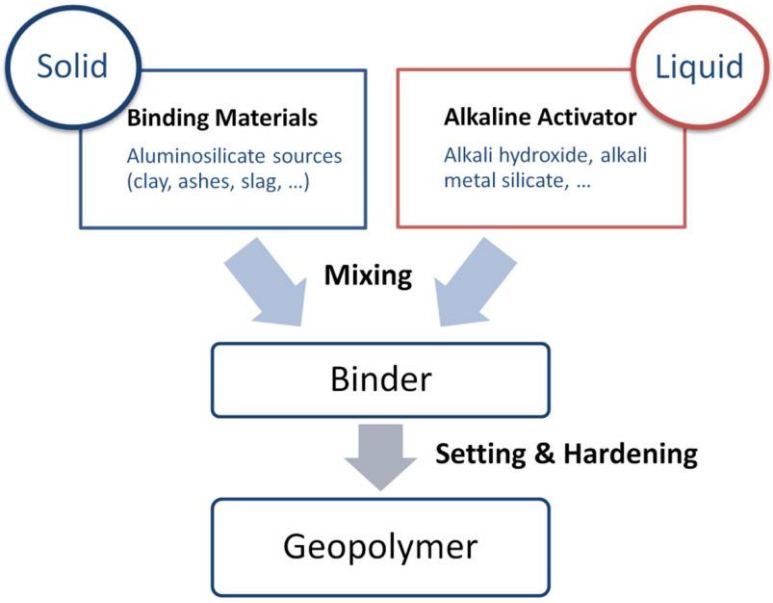

Figure 1. Geopolymer formation schematic diagram. [3]

1.2 Self leveling materials

Over time, concrete flooring can gain flaws that cause damage, obstructing smooth transit, industrial features, and some residential disadvantages Furthermore, defects and cracks are advancement is well-known over time and with use, causing more significant harm. [5]

In self-levelling cement (SLC), prior to applying floor coverings, substrates (mostly concrete slabs) are leveled using methods that give flat horizontal surfaces. [6]

A polymer-based cement with high flow properties is referred to as self-leveling cement. When it is laied, it comes as a very thick liquid. As it dries, it compresses so that its strength is comparable to – and often higher than – traditional concrete. [7]

One of the high-performance concretes made possible by superplasticizers is (SLC). A distinct property of SLC is its very low (plastic) viscosity. This enables for a very quick and massive flow, resulting in (SLC). Because of the low viscosity, the concrete becomes unstable, resulting in segregation and bleeding. Concrete stabilization is important, but it should not obstruct flow qualities by raising viscosity. Therefore, superplasticizers used to reduce viscosity and increase flow ability. [8]

Self-leveling cement underlayment, like tile, possesses the ability to resist a variety of environmental conditions. It is strong (durable), heat- and fire-resistant, moisture-resistant, non-combustible and requires no maintenance after it has been cured.

1.3 Scope of the current study

The current work is an attempt to prepare self-leveling material using geopolymer based on the controllable flow proportion of the geopolymer paste in room temperature without the addition of superplasticizers, which are usually expensive, with different compound proportion. Metakaoline (MTK), commercial sodium silicate and hydroxide were used as starting materials in order to reduce the cost. The final product is desired to be green product with low cost.

1.4 The aim of the study

The current work aimed to prepare geopolymer paste with properate flow proporties to be a candidate material for self-leveling applications

1.5 The structure of the dissertation

There are five chapters in this dissertation. An introduction to the subject is provided in Chapter 1, its importance and the current working technique. Chapter 2 is divided into two parts the first part is focused to revising some theoretical ideas on the study's themes. The second part is a literature review. Chapter 3 involves experimental methods utilized to fabricate the samples and the instrumentations used for testing. Chapter 4 shows the results obtained in the study and the discussing each result. The different conclusions derived from the present work and more scopes of the future work were shown in chapter 5.

CHAPTER TWO

THEORETICAL ASPECTS & LITERATURE REVIEW

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2.1 introduction

Two sections are into this chapter: the theoretical aspects of the current project is the first part, and a review of prior studies' literature is the second part.

2.2 theoretical aspects

2.2.1 Overview

Because it has outstanding cementitious and compressive strength capabilities, geopolymer is a novel binder material that is employed in structural applications. It is made up of a chain of mineral molecules joined by a covalent bonds. Davidovits invented the term geopolymer in 1978 [9]. The prefix "geo" indicates aluminosilicate materials based on geological resources such kaolinite clay. The geopolymer manufacture technique involves polycondensing the aluminosilicate material with an alkali solution to create geopolymer. The reaction takes place at a low temperature (below 100 ℃) or at room temperature. Alumina (Al_2O_3) and silica (SiO_2) content in the aluminosilicate material must be high. Kaolinite, industrial byproducts such as granulated blast furnace slag, fly ash are some of the materials that can be utilized as an aluminosilicate source.

Similar hydrothermal synthesis conditions are needed to produce geopolymeric binders, which are amorphous analogues of zeolites. However, reaction rates are much faster, resulting to amorphous to semi-crystalline matrices as compared to highly crystalline and regular zeolitic structures. [10]

The geopolymer has the following chemical formula

$$x\,(Na2O, K2O).\ Al2O3.\ y\ SiO2.\ z\ H2O \qquad (2.1)$$

where "x" is a value in range of about 0.9 to 1.6, "y" is a value in the range 3.3 to about 4.5 and "z" value at the most equal to 4.

As shown in figure. 2.1, the proposed geopolymer structural terminology divides the geopolymers depending on their Si:Al proportions into three basic geometries: poly(sialate), poly(sialate-siloxo), and poly(sialate-disiloxo). [11]

Geopolymer has a semi-crystalline to amorphous structure with Si-O-Al 3D polymeric networks. By sharing the coordination of Al in IV-fold with all of the oxygen atoms, the tetrahedral of AlO_4 and SiO_4 are connected to each other as shown in figure.2.1. [9, 12]. The IV-fold coordinated Al has a negative charge, which is balanced by cations such as $H3O^+$, Nh^{4+}, Ba^{2+}, Ca^{2+}, Li^+, K^+, and Na^+. However, it is thought that in addition to playing a role in charge balancing, cation inclusion is critical in determining the final product structural integrity. [13]
Geopolymer is made via two different synthetic methods: [14]

1- Alkaline route: the geopolymer is manufactured using an alkali medium containing one or two or more hydroxide solutions (Na+, Ca++, K+, Cs+).

2- Acidic route: in order to make poly alumino-phospho geopolymer, phosphoric acid (H_3PO_4) is employed as a medium.

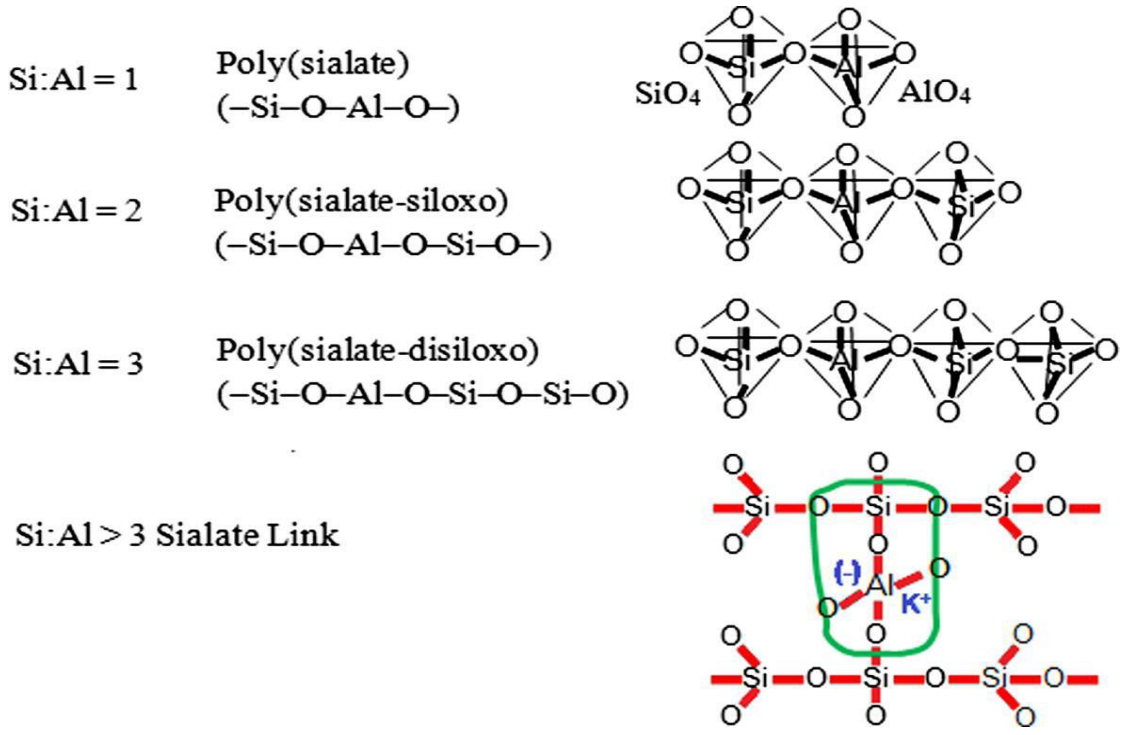


FIGURE 2.1. Various systems of geopolymer depending on the Siloxo number of Si-O units. [11]

The alkaline route is the most important in commercial applications, and research and development (R&D). At low temperatures, below 100 °C, or at room temperature, hardening, setting, or geopolymerization occurs. Geopolymer has good mechanical and thermal qualities below 1000°c, making it suitable for use in construction applications, brick, radioactive and toxic waste encapsulations, heat resistant composites in the 200°C - 1000°C range, and industrial sealants in the 200°c-600°c range. [1]

2.2.2 Geopolymer and alkali-activated materials

The word geopolymer is often used synonymous with alkali-activated materials in books, research articles, and other sources of information. The latter is made with the same precursors that are used to make geopolymer, and both geopolymer and alkali-activated materials are made in the same way [15]. However, according to davidovits, the product spectra for the ^{27} Al NMR should include a peak at around 55 ppm. Al might exist solely in the product structure and completely in the coordination 4. If this is not the case, the product may be referred to as an alkali activated substance rather than a geopolymer [16, 17]. This extremely brief description fits substantially just the materials obtained by pure metakaolin alkali activator [18].

2.2.3 Materials for making geopolymer cement

The materials used to produce geopolymer cement are classified into two categories: aluminosilicate precursor material and alkaline solution, as shown in figure 2.2.

2.2.3.1 Aluminosilicate precursor materials

For the preparation of geopolymers, a variety of materials are utilized as precursors. Kaolinite was widely used in the synthesis of geopolymers [1, 11, 19], the research was soon expanded to include additional raw materials, such as calcined clays [20, 21], waste materials produced as a by-product of industry (e.g.,

copper mine tailings) [22], waste glass [23], slag [24, 25], ashes [26, 27], and several other artificial and natural silicoaluminates like zeolite [28], pure powder of Al_2O_3–$2SiO_2$ [29], minerals containing magnesium [30].

By acting as a significant source of ions (Si^{4+} and Al^{3+}) in the binding system, these materials play a significant role in the development of geopolymers. If at all possible, Materials having a total composition of more than 70% must be in the reactive phase of amorphous materials, including SiO_2 and Al_2O_3. [9, 31].

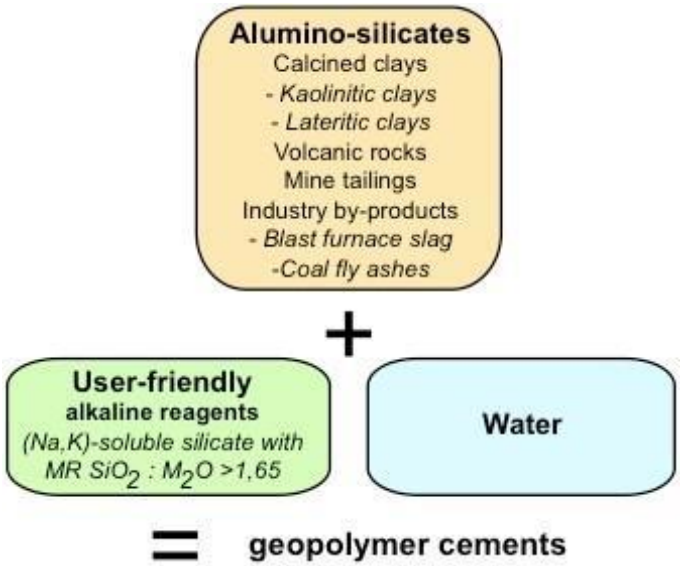


FIGURE 2.2 Material for geopolymer synthesizes process [1].

Clay is commonly used as a basis material for geopolymers because of its ease of interpretation of results. The analysis of the results might be affected by the high percentages of impurities found in composite raw materials like slag and fly ash. Kaolinite is the most common clay mineral used in the production of geopolymers. Kaolinite has a 1:1 uncharged dioctahedral layer structure, as seen in (figure 2.3), with the chemical formula $2SiO_2.\ Al2O_3.\ 2H_2O$. This layer consists of a sheet of $(Si_2O_5)\ n^{-2}$ and a sheet of (gibbsite) $Al(OH)_3$ that are joined by sharing oxygen atoms [32]. geopolymers are often made from calcined raw minerals such as metakaolin. The heating treatment helps in increasing the kaolinite's reactivity to the geopolymerization reaction. Effective kaolinite calcination results in a large

number of pozzolanic amorphous phases. Crystalline phases are changed by heating into reactive amorphous phases. [33]

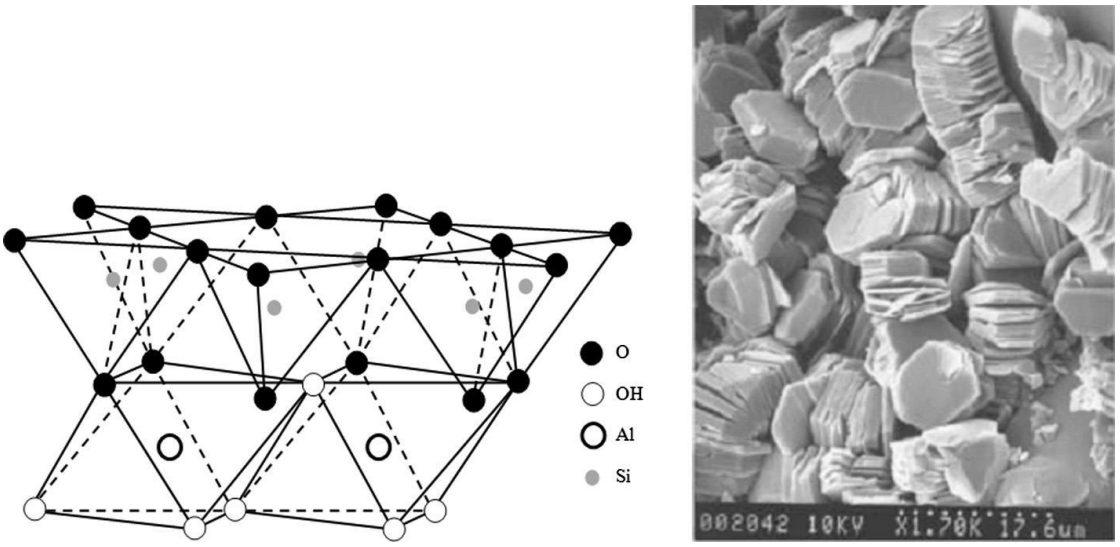


FIGURE 2.3 (a) kaolinite structure & (b) kaolinite microstructure [32].

Metakaolin

The active constituent, which defines the final geopolymer strength, is mostly given on by the amorphous structural phases. During kaolinite treatment by heating at 550–800 ℃, the layer of Al-dehydroxylate constitutive's by water loss is strongly bound by the hydroxyl ions. [34].

This kaolinite transforms into the metastable disordered metakaolin phase. After passing through the thermal treatment, the metakaolin remained in a layerd structure. The layered structure of metakaolin, on the other hand, appears to contain more gaps than kaolinite [35].

Usually the temperature of calcination kaolinite is between 600 and 900℃. In the work by rowles et al, kaolinite can be heated in air for one day at 750℃ to produce the reactive metakaolin. Metakaolin produced poor geopolymer product strength at temperatures above 900℃. Over calcination may have caused a phase of reactive amorphous conversion into phases of mullite-crystalline, which are not reactive and dead burned [36].

2.2.3.2 Alkalis reactant

A soluble potassium or sodium-based alkali metal is used as an alkali reactant. Carbonates, hydroxides, alkali silicates, and other additives such as cement kiln dust or sodium aluminates are used. A strong alkaline media work to rapidly dissolve of the aluminosilicates, promoting the dissolved species and releasing SiO_4 and AlO_4 tetrahedral units for polycondensation [37, 38]. The alkaline reactant solution is a combination of hydroxides (KOH or NaOH) and silicates (K_2SiO_3 or Na_2SiO_3) [33, 39].

K and Na alkali solution

The most often used alkali metal in the geopolymer manufacturing process is NaOH or KOH basic alkaline solution. The dissolving rate of aluminosilicate rises as the molarity of alkaline solution increases, resulting in increased mechanical characteristics, especially compressive strength. Because there is a positive relationship between geopolymer molarity and compressive strength, aluminosilicate is dissolved more rapidly in NaOH than in KOH solution, regardless of the fact that the dissolution rate in NaOH is higher than KOH. However, the compressive strength of KOH base geopolymer is greater than that of NaOH base geopolymer [40]. Panagiotopoulou et al. studied the capacity of several varieties of aluminosilicate to dissolve in 10 M NaOH and 10 M KOH. At the same molarity, the result shows that NaOH has a faster dissolve rate than KOH as shown in figure (2.4) [41].

The formation of a tinny oligomer is made easy by the Na+ bonding with the silicate anion. The bigger K+ forms a larger oligomer with the silicate anion. As a result, compressive strength of K-based geopolymer is 42% higher than that of Na-based geopolymer. In any scenario, a Na-based geopolymer is employed because of its low cost [40].

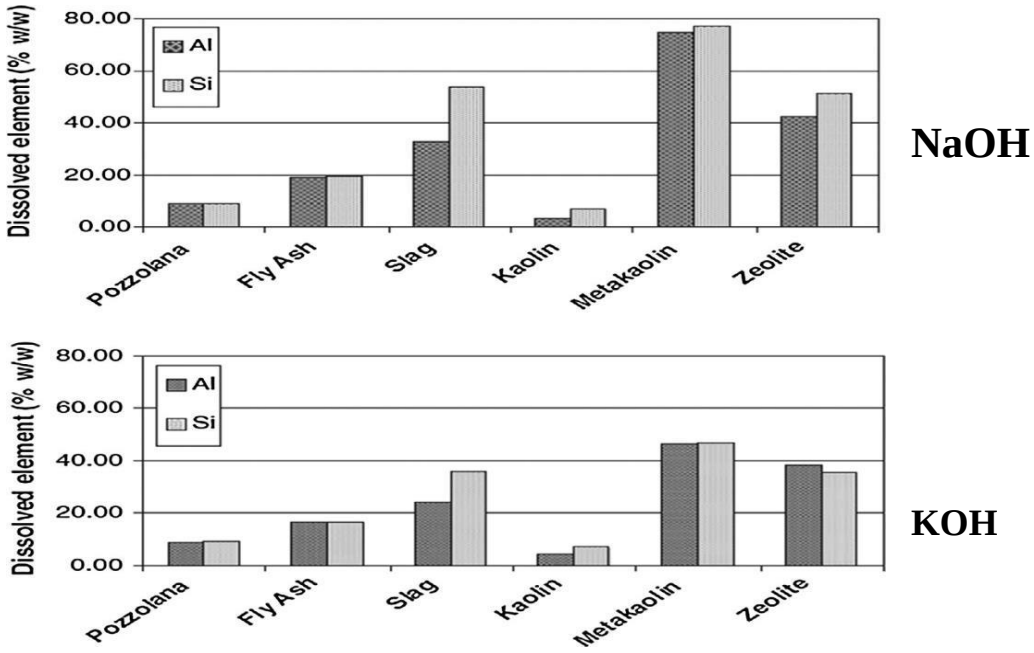


FIGURE 2.4 Dissolution of Al and Si in 10M of NaOH and KOH [41]

2.2.3.3 Alkali silicate and hydroxide mixture

Alkali hydroxide is required to promote the release of silica and alumina from aluminosilicate into the solution, which is required for the geopolymerization process (a multi - step process that includes leaching, diffusion, reorientation, polymerization, and condensation). To make an alkali mixture solution, mixing the alkali hydroxide with an alkali silicate is required rather than using alkali hydroxide alone, several research have been undertaken using a blend of alkali silicate and hydroxide. The results show that mixing alkali solutions enhances microstructure and strength. When NaOH was used as an alkali solution, the compressive strength was lower than that of an alkali solution including an alkali hydroxide and a silicate. In addition, the alkali solution promotes a densely packed structure with fine grains. This indicates a high rate of geopolymerization process when sodium silicate and sodium hydroxide are combined [42]. Alkali silicate works as a plasticizer or dispersant, alkali reactant, and binder [43]. Silica fume can be used as a metal silicate additive or as an alternative to potassium/sodium silicate solutions. They've been employed to improve the silica content of the mix on a few occasions and promoting the precipitation and gelation of silicates [13].

Most experiments confirm the hypothesis that the presence of alkali silicate in the solution of alkali reactant is significant and results in increased mechanical strength. A specific amount of SiO_2 soluble in an alkali silicate solution favors the formation of oligomers, dimmers, and monomers [44].

Solutions containing alkali reactants in the geopolymerization process, a combination of K-silicate/KOH, Na-silicate/NaOH, and K-silicate/NaOH was also used [3].

2.2.4 Reaction of Geopolymerization

Geopolymerization is a chemical reaction in which the amorphous aluminosilicate sources rapidly transforms into 3-D polymer networks. The alkalination chemistry is dependent on the type of alkali reactant and the sources of aluminosilicates.

Depending of the basic ingredients used, synthesis of geopolymer uses a similar procedure. It is important to know the reaction that leads to the production of geopolymers. The exact response of geopolymerization is not fully known at this time [42]. Nonetheless, many academics agree the dissolution of Si and Al species from the surface of aluminosilicates is necessary for the production of geopolymers, the gel is created by the polymerization of soluble types and active surfaces, and the gel then hardens to create a solids. Instead, use the graphic schematic which shows the Al-substituted silicate formation layers in figure(2.5) after being attacked by NaOH solution. After being chemically attack, the deformed Al sites structure was created and changed into tetra coordinated Al sites. [45].

Geopolymerization a reaction that produces heat (exothermic), and it is expected that oligomers would be used in the synthesis (trimer, dimer), It will provide 3-D macromolecular structures its real unit structures. As seen in figure(2.6), davidovits proposed the creation of geopolymer equations. (Si_2O_5, Al_2O_2) refers to the Al coordination IV-fold, whereas SiO_2 is generated from a solution of silicate. The finished product contained Si-O-Al structural core. [9, 10].

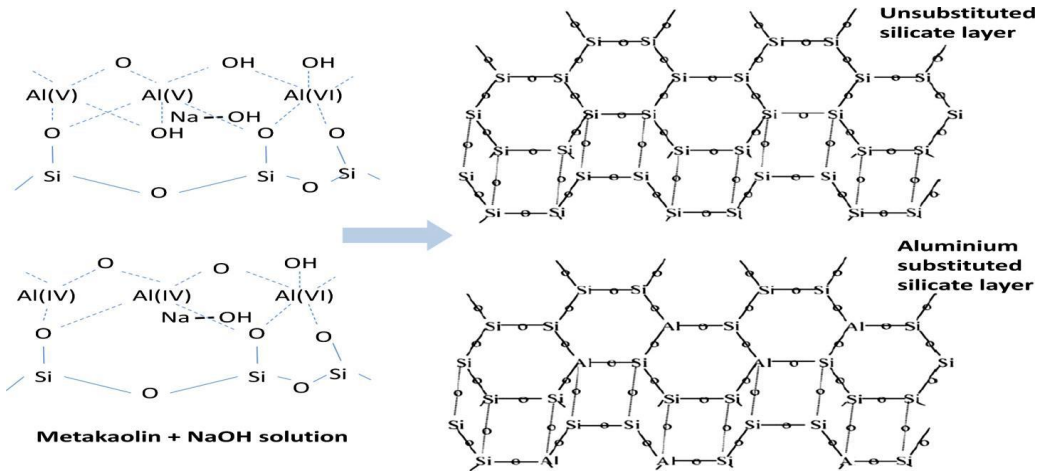


FIGURE 2.5 Diagram scheme demonstrating the substituted of aluminium and silicate layer unsubstituted if metakaolin responds with solution of NaOH. [45]

$$n\,(Si_2O_5,\ Al_2O_2) + 2nSiO_2 + 4nH_2O \rightarrow n\,(OH)_3{-}Si{-}O{-}Al^{(-)}(OH)_2{-}O{-}Si{-}(OH)_3$$

(Geopolymer Precursor)

$$n\,(OH)_3{-}Si{-}O{-}Al^{(-)}(OH)_2{-}O{-}Si{-}(OH)_3 \rightarrow (Na^+,\ K^+){-}(-Si(O)(O)-O-Al^{(-)}(O)(O)-O-Si(O)(O)-O-) + 4nH_2O$$

(Geopolymer Backbone)

Figure 2.6 Process of geopolymerization diagram scheme [9, 10]

The presence of hydroxyl in the alkali reactant starts the alumino-silicates dissolving process in alkali media, releasing aluminate and silicate types for further polymerization reactions [43, 46].

The degree of dissolution is determined by the concentration of alkalinity solution, the structure of the precursor materials, ion exchange capacity and fineness. The reaction of geopolymerization is thought to occur in many phases that occur at the same time, including [47-49],

a- The dissolving of aluminosilicates in a high-alkalinity reactant.

b- The diffusion and the formation of fine coagulated structures together with the rearrangement of dissolved ions.

c- Polycondensation to produce phases of aluminosilicate gel.

d- Solidification and hardening to generate hard solids.

These steps are shown in figure (2.7).

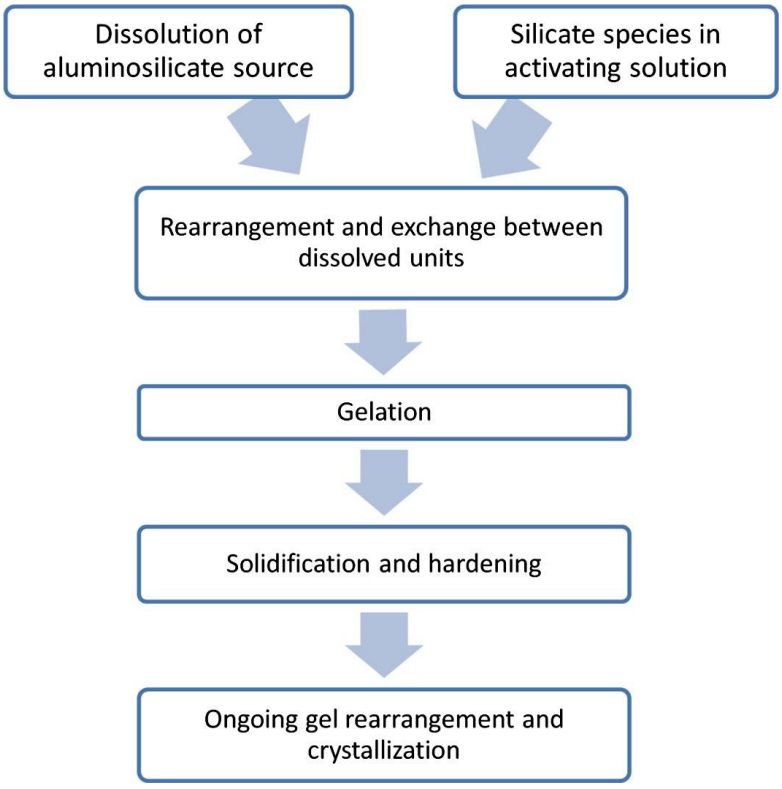


FIGURE 2. 7 Reaction steps during the polymerization process [50].

Provis [50] proposed a model for the geopolymerization of fly ash (FA) and metakaolin (MTK). This model includes more information about the oligomers of silicate that may be present in alkali solutions. Based on the percentage of Si/Al, these oligomers were separated into two stages: amorphous structure and nano-crystalline structure. Other from the zeolite phase, another chemical route for direct aluminosilicate gel formation was also given. figure 2.8 shows the response of the geopolymerization sequence.

According to provis et al. [50, 51], the original phase of geopolymer gel differs from the final phase of gel following increased healing, depending on their model. Incessant gel phase rearrangement occurs during healing, resulting in increased

cross-linking, the exclusion of released water, and the formation of zeolitic crystallites. Gel phases are described by the "hardening and solidification" and "crystallization and rearrangement of continuing gel" in this model, respectively. In the final result, several ordered phases have been shaped.

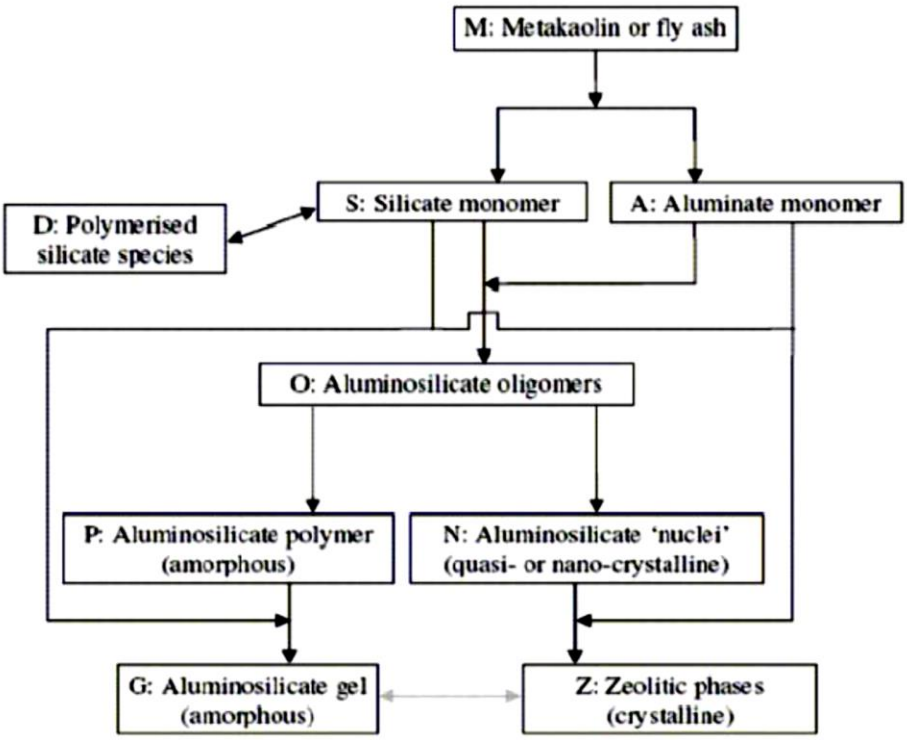


FIGURE 2.8 Geopolymerization reaction process [50]**.**

According to a recent research [12], throughout the polymerization process, there are two types of gels: initial gel G1 and final gel G2. When the reaction starts, an initial gel G1 is generated, which is then arranged into a more cross-linking structure after curing. The molecules' expulsion of free water results in the development of a zeolite-like structure. Solidification is represented by G1 and hardness is represented by G2, which relates to rearrangement and crystallization, respectively. In the final structure, the orderly phase is present. During polymerization, the covalent bonds between (Si-O-Al) and (Si-O-Si) collapse into a contact phase and come into touch with the alkaline solution. According to most research, the dissolved species react to produce a curd structure [52]**.**

The reaction continues with the initial gel G1, which has a high aluminum Al content, and then transforms to the final gel G2, which has a higher Si content, as illustrated in figure (2.9). The final gel G2 eventually grew and developed a three-dimensional structure.

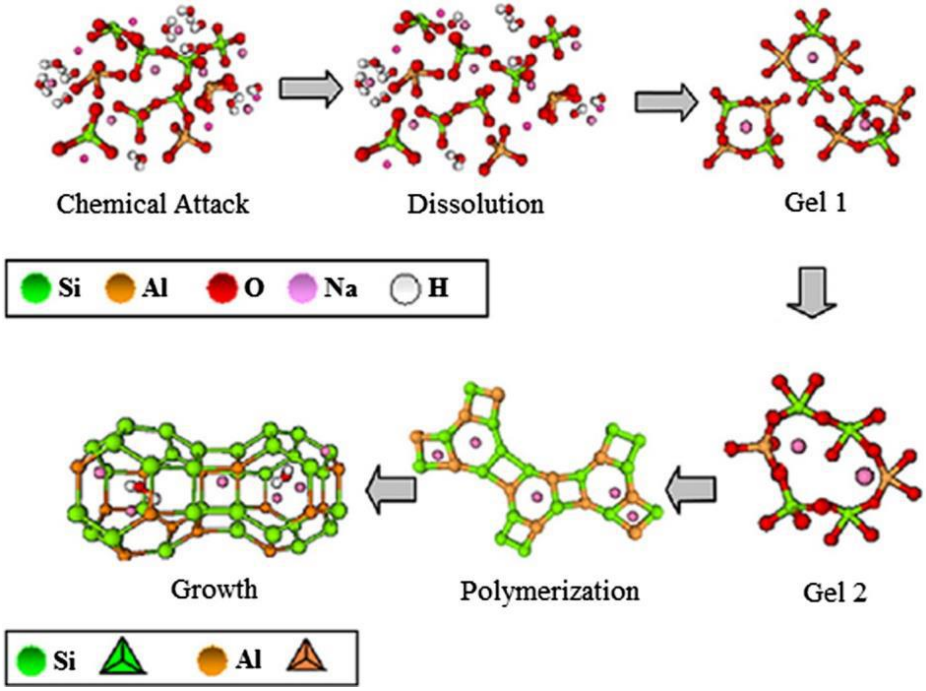


FIGURE 2.9 Graphical sketch of alkination of geopolymer [42]

2.2.5 Factor effected on geopolymers characteristics

Particle size, initial liquid or solid content, phases of reactive materials, metal silicate types, chemical composition and aluminosilicates types, alkali concentration, curing methods, fillers or additive content, and water content all have an influence on geopolymer manufacture.

2.2.5.1. Proportions of solids/liquid (S/L)

The importance of the S/L ratio because it controls of liquid amount and solids in a homogeneous mixture, this directly affects on final product's ultimate strength, geopolymerization reaction, dissolution, and workability.

2.2.5.2 Water Amount

water has an effect on the properties, structure, and development of geopolymers. It's an important geopolymer component. Water is responsible for dissolution, the mobility of dissolved ions, the hydrolysis of oligomers, and their polycondensation. During geopolymer synthesis the water roles are depicted in figure(2.10) [53].

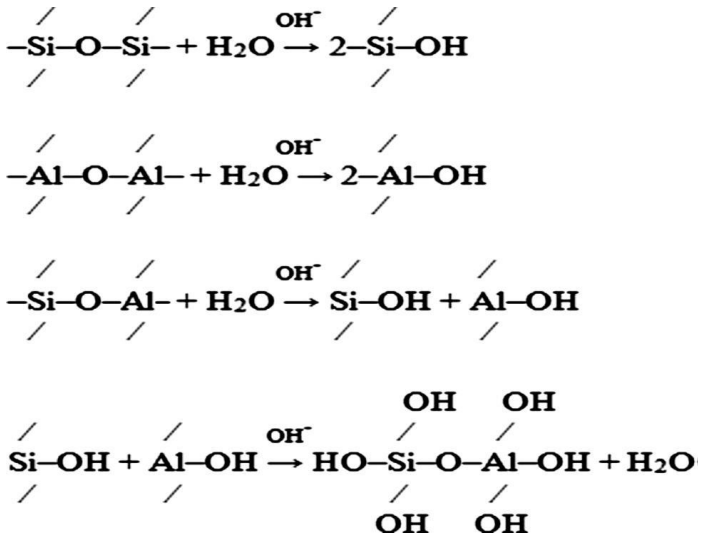


FIGURE 2.10 Role of water in geopolymerization [53].

Furthermore, the flowability of geopolymer mixes is caused by water. A sufficient amount of water promotes mixing and provides an ion transport mechanism [21]. There has always been worry about adding more water to the geopolymer manufacturing process. Extra water is thought to dilute the system's alkalinity while transferring ions away from the reaction zone [1]. Because this reaction is considered a procedure for releasing water, more water might slow down the geopolymerization process [39]. Furthermore, the amount of water in the geopolymer system has an effect on the final product's open porosity and density. The presence of a lot of water resulted in a lot of open porosity [54]. However, the amount of water required is determined by the properties of the raw materials used. Furthermore, other mixing parameters such as alkali concentration, percentage

S/L, and alkali reactant proportions must be mixed with the amount of water in the geopolymer system [3].

2.2.5.3 Curing system

after conducting the mix, geopolymers are usually exposed to ambient or higher ambient temperatures to cure. A healing temperature of less than 100°C is often desired. The majority of researchers concur with this data. For example, davidovits indicated that curing temperatures range from 60 to 95 ℃. Despite the fact that increasing the curing temperature improves strength, the strength may decrease if the temperature is too high or the exposure duration is too lengthy. Heat improves reaction speed by speeding up the dissolution of alumina and silica from aluminosilicates and simplifying the geopolymer matrix hardening and polycondensation process [55, 56]. In other words, to initiate the geopolymerization reaction, heating must be highest than the activation of the thermal reaction.

2.2.5.4 PH of liquid phase

The PH of the alkaline solution is the most significant controlling factor for compressive strength. If the PH of the solution is increased, the setting time of the cement is decreased. The geopolymer combination remained viscous cement at lower PH magnitudes, however at higher PH, the mixture achieved an extra fluid gel composition that is more workable and less viscous. If the concentrations are equivalent, NaOH estimates lower monomer solubility in comparison to KOH due to decreased alkalinity. As the PH increases, the amount of chain oligomers and monomeric silicate available to react with soluble aluminum decreases. When the PH rises, more soluble aluminum is produced, which reacts with existing calcium for reaction.

2.2.5.5 Concentration of alkali

The alkali content has a significant effect on the mechanical and physical properties of geopolymers. Alkali promotes aluminosilicate dissolution and

solubility, as well as speeding up the geopolymerization reaction rate [57]. The concentration of alkali is based on the magnitude of PH, and it specifies the quantity of ions to assist in the dissolving process. With an increase in alkali content, the reaction rate and heat evolution increases. The appropriate alkalinity for the source materials dissolution is suggested by a faster rate of heat evolution [53]. In addition, the workability of geopolymers changes based on the alkali concentration used. An increase in alkali content generates a faster setting rate, which is linked to the paste's workability.

2.2.5.6 The rate of alkali reactant

Increasing the reactant alkali often lead to a strong geopolymers. Increasing the quantity of silicate improves the polymerization process, resulting in a resultant reaction with increased mechanical strength [58].

The alkali reactant proportions calculate the amount and rate of geopolymerization process [59]. Until now, the paste workability has been limited at a specified high percentage, resulting in a later reduction in strength.

For metakaolin geopolymers with a 59MPa ultimate compressive strength, wang et al.[40] proposed a percentage of Na_2SiO_3/NaOH of (0.24).

Pelisser et al [60], on the other hand, used a wide variety of Na_2SiO_3/NaOH proportions (1.0, 1.6, and 2.2) to make geopolymers based on metakaolin. At 1.6, the greatest strength (64 MPa after 7 days) was reached. The lowest strength was reached with a 1 wherein the geopolymer matrix is porous in proportion.This result was observed by poowancum and horpibulsuk [61] for geopolymer made of sedimentary clay that has been calcined. However, The optimum fraction for sedimentary clay was found to be 0.5, with a strength of 27 MPa.

2.2.5.7 Molar proportions of (H2O, Na, Al, and Si amounts)

NaOH solution and liquid Na_2SiO_3 describe the Na quantity in a geopolymer system. Liquid Na_2SiO_3 and aluminosilicates contribute to the quantity of Si, but only aluminosilicates contribute to the amount of Al. The H2O comes from liquid Na_2SiO_3, a NaOH solution, and free water that was added while process of mixing.

Changing mixing parameters (for example, NaOH concentration, S/L ratio, and Na2Sio3/NaOH ratio) results in different atoms or proportions of oxide molar in geopolymersystem. However, the quantity of each component contributing to the geopolymerization reaction is dependent on the reactivity or reactive phases of aluminosilicates, as well as the duration and how effectively they are incorporated to build a stiff network. To conclude, the quantities of Na, Al, and Si in the final geopolymer have a massive effect on its properties. If different aluminosilicates other than clay-based were used, the quantities differed. Most researchers believe that the extent of reaction, rather than the initial mixture composition, would determine the final features since different raw materials have varying reactive phases and the potential of each component to bind in the system to create a rigid network [3].

2.2.6 Applications of geopolymers

Geopolymer can be used in a wide range of applications, including: sciences, history and archaeology, cultural heritage, decoration and arts, geopolymer coating, toxic waste and radioactive waste containment, high-tech resin systems, high-tech composites for automobiles and internal aircraft, composites for infrastructure reinforcement and repair, concretes and cement, foundry industry, biotechnology (materials for medical uses), thermal shock refractories, items of refractory, and ceramic tiles with low-energy [15].

2.2.7 Advantages and disadvantage of geopolymer

2.2.7.1 Advantages of geopolymer

There are many advantages for geopolymer including [62]:

1- In certain cases, there is a significant increase in compressive strength.

2- Low CO2 emissions.

3- Excellent corrosion resistance, especially when coupled with PTFE filler.

4- Fire resistance (1200°c) and no hazardous gases released when heated.

5- At room temperature, geopolymer cement was able to form strong connections to old and new substrates, steel, boro-silicate ceramics, and glass.

6- Excellent resistance to chloride chemical attacks, including sulphate, various acids, and sea water.

7- Low permeability, good isolation, and no bleeding.

8- Low shrinkage and low thermal conductivity

2.2.7.2 Disadvantage of geopolymer

The main disadvantage of geopolymers is the necessity for specific handling due to alkaline materials' excessive alkalinity. Furthermore, the cost of alkaline solution is high, and there are practical problems with conducting heat healing/high temperature healing treatments [62].

2.2.8 Self-leveling material (SLM)

Over time, concrete flooring can develop flaws that cause damage, preventing smooth transportation, industrial features, and some residential disadvantages. Furthermore, flaws and cracks are known to develop through time and use, resulting in more serious damage. Self-leveling mortar has been utilized on a small scale around the world to provide more level, high-performance, and easy-to-apply flooring. However, there has been a shortage of knowledge on how to utilize them and what the best practices are [5].

Before applying floor coverings, the substrates (often concrete slabs) are leveled using SLM that give flat horizontal surfaces. SLM is mostly applied on the ground surface, where it may solidify into a flat, wear-resistant surface without the need for vibrating or plastering [63].

Meanwhile, in recent years, introducing SLM, a brand-new kind of functional construction material. that has been identified as a material with a high value added, has become increasingly popular. It's usually found in public buildings like offices, hospitals, schools, and factories. In a fresh condition, SLM under its own weight, has great fluidity and self-leveling capabilities, and in a hardened state, it

has a smooth surface, steady volume change and high early strength. The popularity of SLM is owing to its unique properties, as well as the cost and labor savings it provides [64, 65].

SLM are building materials that are used to fix surface flaws and level floors. with a new flooring system, it is intended to prevent a variety of problems in traditional cementitious slabs, such as self-leveling, reduced cracking risk, pumpable, self-leveling, curing rapidly, and application speed, which can decrease dead load due to the ability to manufacture thin coatings [66].

SLM has three primary uses [67].

A. Underlayment is a material that is utilized to smooth out any surface and correct any imperfections in the concrete. This is done before any form of flooring is installed.

B. Covering is the process of applying self-leveling mortar from the start of the project to act as the real finished floor without the requirement for a floor covering.

C. In applications such as bridges and highways, self-leveling material is used as a repair material for damaged concrete. Furthermore, self-leveling substances can be utilized to create a smooth and durable new surface for decorative treatments.

Leveling and smoothing substances are clearly described by the following layer thickness ranges:

1- fine leveling compounds, applied in a single pass to a layer thickness of approximately 3 mm.

2- smoothing substances, applied in a single pass, can also be used in thicker layers (approx. 3 to 10 mm).

3- fillers and leveling chemicals are applied in a single pass, with layer thicknesses ranging from 5 mm to more than 10 mm.

The flowability of self-leveling material reduces as it becomes thicker. Because of its high flow-ability, it can't be used on vertical surfaces. There is no requirement for vibration or compaction with self-leveling material. As a result, a quick team is needed to spread the mortar all over the required area before it hardens. An

aluminum mob is the only piece of equipment that may be used to speed up the process of spreading mortar over a large slab [68]. Another feature of self-leveling materials its fluidity; as already stated, it has a high fluidity and strong segregation resistance. Self-leveling material has a density range of 2000 to 2200 Kg/m3, which is lower than normal mortar, which has a density range of 2400 to 2600 Kg/m3. As a result, the dead load is reduced. It might come in a variety of colors and be used as a final layer without any additional material [69].

Admixtures are added to self-leveling material to make it more workable and reduce viscosity. Its flow-ability is especially apparent as a result; it may easily spread all over the surface. Furthermore, polymers in such mortar mixes unite the product's viscosity, ensuring that the composition remains consistent from top to bottom without any segregation. Engineers are also using self-leveling material as a floor covering for industrial spaces, and they sometimes coat it with an epoxy coating to provide color and shine, as shown in (figure 2.11) furthermore, people have started utilizing it as a decorative item in their homes. It may also be used as a decorative layer on top of bricks. It may, on the other hand, be used to repair things like road pavements and bridge cracks [70].

A) Covering floor for industrial areas by SLM

b) garage area floor covering with epoxy layer

FIGURE 2.11: Uses of SLM in floor covering. [70]

The use a superplasticizer such as a co- or ter-polymer of a carboxylic acid, polyacrylate, phosphoric acid, or sulfonic acid and polyethyleneglycol monoallyl ester sulfate in cementitious materials that has no negative impact on the materials' mechanical characteristics and achieves the above properties [71].

2.2.8.1 Properties of self-levelling concrete

These characteristics are achieved via the self-leveling concrete's design, which includes: [72]

1. Stability
2. High flowability
3. Low viscosity plastic
4. Low bleeding
5. Low segregation

2.2.8.2 Advantages of self-levelling concrete

The following are some of the most significant advantages of self-leveling concrete: [73]

1. Ease of application.
2. There is less work to be done.
3. The result is a leveled and smooth surface.
4. It is possible to obtain a water-resistant surface.
5. Resist growth of microorganisms.
6. Concrete is homogenously hardening as it forms.
7. It is possible to achieve a compressive strength that is greater than that of ordinary concrete.
8. SLC produces a concrete surface that is flat and smooth.
9. SLC creates a solid foundation that resists bleeding and segregation.

2.3 literature review

2.3.1 Geopolymer literature review

Barbosa, V. F. et al. (2000). Combined metakaolinite with sodium silicate in a 1:2 Al: Si ratio to create an inorganic polymer that was cured at room temperature or 65 ℃. The compressive strength achieved was 48.1 MPa. When the concentration of Na is high enough, a mechanism for charge balancing is available for the tetrahedral replacement of si by Al, which has insufficient excess to form sodium carbonate by air carbonation. The cured geopolymers are amorphous and have 3-D structures (glassy), with si occurring in a range of settings and al is fundamental to all tetrahedral geopolymers [1].

Song et al. (2005). Conducted an experiment to test the durability of geopolymer concrete. Fly-ash based on geopolymer concrete was employed. The material was soaked in a 10% H2SO4 sulphuric acid solution for eight weeks to conduct the test. In the manufacture of geopolymer concrete, a combination of Na_2SiO_3 and NaOH is used as an alkali solution. After a day of casting, all specimens were healed in a furnace at 23°c or 70°c for one day. At 28 days, the compressive strength was in the range of (33-42 MPa). The mass reduction and compressive strength of the sample were evaluated after soaking it in a 10% H2SO4 sulphuric acid solution for 7, 28, and 56 days. The results showed that geopolymer concrete has a very high sulfate resistance, as measured by mass loss (less than 3 percent). Also, even when the sample was completely immersed in H2SO4 sulphuric acid solution, the geopolymer concrete exhibited a high load capacity [74].

Sofi et al. (2007). As an alkaline solution, they employed sodium silicate, sodium carbonate, and sodium hydroxide. The test resulted in a result that was similar to the australian code for regular portland cement. The poisson's ratio, compressive strength, splitting tensile strength, elasticity modulus, and flexural strength were all determined throughout their research. All of the samples were steam cured for 24 hours. The coarse aggregate utilized was 14 mm crushed basalt

rock and ground granulated blast slag (GGBS) in a single size aggregate. Gp concrete has structural qualities that are similar to those required by the australian code for regular portland cement mixes in all instances [75].

Reddy et al. (2010). Made geopolymer concrete with fly ash as an aluminosilicate source and the alkalin solution reactant used the sodium silicate Na_2SiO_3 with sodium hydroxide NaOH. It was discovered that increasing the molarity of alkali solution increased compressive strength (27MPa at 10 M and 32MPa at 16 M). It was also discovered that when the molarity of the alkali solution increases, the geopolymer workability decreases [76].

A. M. Al Bakri et al. (2013). Geopolymer based on fly ash has prepared as a replacement for ordinary Portland cement (OPC) in concrete have increased in significance. Geopolymer concrete does not need the presence of Portland cement as a binder. Instead, an activating geopolymer can be utilized with an alkaline activator. Moreover, the use of fly ash is more environmentally friendly due to the reduced CO2 emissions and costs compared to OPC, which requires the burning of large quantities of fuel and the decomposition of limestone and can result in significant CO2 emissions. The compressive strength increases with the optimum NaOH molarity, fly ash/ alkaline activator ratio, Na2SiO3 /NaOH ratio used, and curing process handled. Fly ash-based geopolymer also provides superior performance givens its better resistance to aggressive environments compared to normal concrete also has greater performance, with a strength of 71 MPa, making it more resistant to harsh climates than conventional concrete [77].

Y.M. Liew et al. (2013). Prepared metakaolin geopolymer paste and study the effect of curing temperature (room temperature, 40°C, 60°C, 80°C and 100°C) and curing time (6h, 12h, 24h, 48h and 72h) on the geopolymer pastes produced from geopolymer powder. The results showed that curing at room temperature was unfeasible. Heat was required for the geopolymerization process, where strength increased as the curing temperature was increased. Moderate elevated curing temperature favored the strength development of geopolymer pastes in comparison with those treated with extreme elevated curing temperature. The microstructure

of geopolymer paste cured at moderate curing temperature showed obvious densification of structure. In contrast, the structure formed was weak and less compact at very high elevated curing temperature [78].

Francesco et al. (2013). Use of commercial epoxy-based organic resins and geopolymeric slurry to produce the specimens. In this method, geopolymer-based hybrid mortars were created that have a homogenous dispersion of the organic resin and a different quantity of normalized sand (up to 66 percent by weight). Due to the organic polymer's ability to create a more cohesive microstructure with fewer microcracks, these new materials exhibit increased compressive strength and toughness once hardened in comparison to both geopolymer and the hybrid pastes. A similar interfacial transition zone to that found in cement-based mortars and concretes may be detected thanks to the microstructural characterisation. Additionally, it has been studied how microstructural characteristics relate to mechanical characteristics [79].

Puput risdanareni et al. (2015). Investigated the effect of alkaline activator percentage (Na_2SiO_3/NaOH) on geopolymer concrete mechanical properties. The mechanical properties of geopolymer concrete, such as setting time, splitting tensile strength, and porosity, are examined. Fly ash was used as a filler in trass and as a cement substitute. Alkali activators such as natrium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) are used. The results showed that the concentration of NaOH and the fraction of alkaline activator had no influence on the geopolymer concrete's split tensile strength, porosity, and setting time. Because it has a high compressive strength of around 30.97 MPa, geopolymer concrete with a concentration of NaOH ten molars and an activator mass ratio of Na_2SiO_3/NaOH 2.5 can be utilized for structural concrete [80].

z. Zayer et al. (2020). Made the geopolymer cement based at room temperature, metakaolin as a source of aluminosilicate, free silica from silica gel, and sodium silicate, potassium hydroxide, and sodium hydroxide as alkaline solution precursors. To manufacture geopolymer cement, the taguchi technique for the design and analysis of experiments was utilized to determine the optimum mix

fraction of starting materials. Carefully controlling the silica concentration, water content, SS/SH ratio, and mixing time is required to produce geopolymers with compressive strengths more than 100 MPa. It was discovered that, a geopolymer paste with a compressive strength of (146 mpa) may be created using the formula (Al_2O_3. $3.8SiO_2$. 0.5 K_2O. $0.5Na_2O$. W H_2O). The alkali silicate to alkali hydroxide molar ratio should be set between (3.25 and 3.02). When compared to conventional portland cement concrete, the modified geopolymer concrete's compressive strength at 28 days was (46 MPa), which is too high. [81].

2.3.2 Self leveling material literature review

M. A. A. w. Ali et al.(2020). Geopolymer concrete technique using fly ash as cementitious material, sodium silicate solution (water glass) and hydroxide sodium as alkali-activated solutions and lightweight artificial coarse substances as aggregates enabling to produce lightweight tile. The produced tiles of research results have 35%, 50% less in weight, and thermal conductivity respectively than the normal tile which improved in iraqi tiles and can be used for roof flatness [82].

Yu chen et al.(2012). Achieving the possibility of using the waste can completely replace natural sand for the production of self-leveling mortar this research studied the effectiveness of self-leveling mortar since the advantages of better fluidity, less water consumption, smaller time loss and high strength. The strength decreases with the addition of mineral admixtures, but it can still satisfy the requirements of standard JC/T 985-2005 "Cementitious self-leveling floor mortar , Moreover, the method effectively reduced pollution and production costs. [63].

Padilla et al. (2015). The creation of self-leveling material utilizing micro bubbles composed of aluminium silicate with a density of 0.25 g/cm3. Taguchi with 8 variables and 3 states was used to create the formulas. The taguchi technique of result analysis allows the identification of the preponderant influence of each variable on the stated properties. More than 250 percent fluidity, a compression

strength of around 100 kg/cm2, and a low volumetric weight were all characteristics of some formulations. When compared to the weight of commercial self-leveling mortars, the volumetric weights achieved are 20% lighter [69].

Liang chen et al. (2016). Investigated the features of geopolymer-based alkali-activated metakaolin, Water, sodium silicate, sodium hydroxide, and metakaolin, in weight proportions of 41.6, 6.7, 45.0, and 6.7 percent, were found to generate the best geopolymer. In contrast, the optimal curing state was found to be 60 ℃ for 7 days (168 hours), where the geopolymer-based alkali-activated metakaolin displayed the highest compression strength of 52.26MPa. The mechanical properties of alkali-activated metakaolin-based geopolymers are mostly determined by the phase of amorphous gel [83].

Rakngan, B.E. et al. (2016) For the purpose of increasing the workability of geopolymer pastes. production of geopolymers, three sources of high calcium, class C fly ash were utilized as aluminosilicate sources. The mini slump test and rheological tests were used to examine the workability of the geopolymer pastes. The workable time of the geopolymer pastes was found to be increased by the inclusion of two chemical agents (sodium gluconate and a commercial hydration stabilizer marketed under the brand name "recover") as chemical admixtures. When compared to pastes without chemical admixtures, the compressive strength of the geopolymer pastes was decreased. When compared to pastes without chemical admixtures, the compressive strength of the geopolymer pastes was decreased [84].

Ezzat h. Fahmy et al. (2016). created mortar with self-leveling flow properties. A number of mixes have been created utilizing different ingredients and a moderate 28-day strength of 35 MPa. Limestone has been used with chemical and mineral admixtures to increase performance by enhancing flow and cohesiveness. The results indicate that self-leveling mortar with similar qualities to ready-to-use market products may be successfully produced. These combinations were examined for their qualities in terms of performance and economic merits [5].

Cecília Schaefer et al . (2017). created self-leveling underlayments using phosphogypsum (PG). The work was in two parts. Initially, Heated the phosphogypsum at 350°c, 450°c, 550°c, and 650°c to form phosphoanhydrite (PA). A total of two heating periods were used (2 and 4 h). The production of anhydrite was confirmed by X-ray diffraction and thermogravimetric analysis (DTA-Tg) (XRD). The study showed that anhydrite II was produced at temperatures over 450 °C and that greater calcination temperatures reduced the solubility of PA. In the 2nd section of this study, pa was used in self-leveling underlayments as the main binder in a ternary system made up of calcium sulfate, calcium aluminate cement, and portland cement. The greatest mechanical strength performance was displayed by self-leveling mortar screeds made with PA (550 C/4 h) and PA (650 C/4 h), and no deterioration was seen following immersion and immersion-drying testing [85].

Tambara Júnior et al. (2018). They studied the reactive properties of self-leveling alkali activated hybrid cements such as ordinary portland cement (OPC) and its leftover precursors, rice husk ash (RHA) and coal bottom ash (BA). Due to weak reactivity of BA, binary mixes containing 2.5–30% of the treated BA samples were made using OPC. Additionally, ternary mixtures employing RHA as a replacement for OPC in percentages of 25%, 50%, and 75% were produced. (in mixture with 90%:10% BA:OPC). Mortars containing 30% OPC were found to have a 33.5 MPa compressive strength and a 7.53 MPa flexural strength. Alkali activation OPC 10 produced the best results its strong workability attributes, compressive strength (28.21 MPa), dynamic modulus (16.05 GPa), lowest sorptivity (0.0084 cm P min), lowest curling screed (100 m), and least amount of mass loss to acid attack. [86].

Jiayu yang et al. (2019). putting forward the shrinkage control measures for evaluating the effect and mechanism of shrinkage compensation of self-leveling mortar by analyzing the shrinkage characteristics of cementitious self-leveling mortar (SLM). The drying shrinkage, autogenous shrinkage and mechanical properties of the self-leveling mortar are analyzed by comparing it with the ordinary mortar. The difference between the two kinds of matrix was explained by

the result of hydration and pore structure. The internal curing of self-leveling mortar was carried out by superabsorbent polymer. The variation of drying shrinkage and autogenous shrinkage of self-leveling mortar were tested with the content at 0%wt, 0.2% wt, 0.4%wt and 0.6%wt. The pore structure and pore size distribution were also investigated. The experimental results show that the self-leveling mortar has larger the drying shrinkage and smaller the autogenous shrinkage than those of ordinary mortar. The main reasons for this are the hydration delay and the change of pore structure of self-leveling mortar caused by the addition of organic admixture [87].

CHAPTER THREE

EXPERIMENTAL WORK

CHAPTER THREE
EXPERIMENTAL WORK

3.1 Introduction

The methodologies for synthesis of geopolymer paste, the materials employed in that process, and a brief overview of the characterisation techniques used in this work are defined in this chapter.

3.2 The Starting Materials

To make geopolymer cement, kaolin, sodium silicate, and caustic soda were utilized as starting materials.

3.2.1 kaolin

The metakaoline substance used in this study came from calcination of locally-sourced kaolin clay (Western Iraqi Desert/Dwaikhla). The kaolin was calcined in the furnace for three hours at 750°C.
The symbol used to mention the metakaolin used in this study (MTK).

Table (3.1) demonstrates the outcomes of the wet chemical analysis of the kaolin [81].

TABLE 3.1 The kaolin wet chemical analysis results [81]

SiO_2 %	Fe_2O_3 %	Al_2O_3 %	TiO_2 %	CaO %	MgO %	SO_3 %	LOI %	K_2O %	Na_2O %	Cl %	P_2O %
48.77	1.76	34.27	1.47	0.43	0.08	0.11	12.46	0.43	0.17	0.03	0.02

3.2.2 Sodium silicate

In this study, sodium silicate sourced from the UAE was used, which is characterized by its availability, cheapness and it is in the form of a solution also known as water glass, which is shown in figure(3.1). The compostion and specific gravity of this material are present in table 3.2.

To calculate the amount of dissolved pure sodium silicate and free silica in a water glass.

- Chemical composition of pure sodium silicate is Na_2SiO_3. It contains : Na_2O 50.77% , SiO_2 49.22% (Depending on the chemical formula)
- For 100gm water glass will contain:Na_2O 13.4 gm, Si_2O 32.5gm(from table 3.2)
- The amount of pure sodium silicate that needs to be dissolved to get an amount of 13.4 gm of Na_2O = 26.39 gm

  The amount of SiO_2 obtained from dissolving 26.39 gm of pure sodium silicate= 12.98 gm
- The amount of free silica= Total SiO_2 – SiO_2 from pure sodium silicate

  = 32.5 – 12.98 = 19.52 gm

  ∴ 100g of glass water will contain : 26.39 gm of dissolved pure sodium silicate

  19.52 gm free silica

  54.1 ml water

TABLE 3.2: The properties of sodium silicate

Sodium silicate	%SiO2	%Na2O	%H	specific gravity
	32.5	13.4	54.1	1.54 g/ml

FIGURE 3.1 Sodium silicate solution (glass water)

3.2.3 NaOH

In this study, commercial sodium hydroxide was used. Where it is economically priced at $1 or less per 1 kilogram.

3.3 Preparation of Geopolymer Self-leveling Material (GSLM)

3.3.1 Alkaline Solution

This study used a mixture of sodium hydroxide and sodium silicate(glass water) as an alkaline liquid. Because NaOH is a hazardous substance, it must be handled with care. To get the requisite molarity (M), NaOH was weighed and placed in a beaker, and then distilled water was added. As the NaOH pellets dissolved in the water, they released heat. After complete dissolution of NaOH, sodium silicate was added. At this time the solution is placed on a stirrer, placed magnetic capsule in the solution, and the mixture was stirred at a 600 Rpm speed. After that, The solution was then allowed to cool to room temperature for a while.

3.3.2 GSLM

The geopolymer formula used in this study was 1: 1: 3.8 sequentially Al_2O_3, Na_2O, SiO_2 [81], and ratio SS/SH is 2.722.

After alkaline solution cooled to room temperature The metakaoline (MTK) was added to it and was mix using a mechanical mixer, the mixture was mixed at slow speed of approximately 800 rpm for 60 s . Then it is mixed at a speed of 1200 rpm for three minutes. Then it is mixed at a speed of 1500 rpm for five minutes (see figure 3.2) .

Two groups (each group contains 4 mixing ratios) were used to determine the optimum mixing proportion of reactant materials as shown in table (3.3). G1 and G2 have the same amount of ingredients, but the G1 contains less sodium silicate than G2, which leads to replacing the amount of water by adding more water. The setting time and the fluidity of GSLM were measured immediately after mixing. For other tests molding the geopolymer pastes, moulds of plastic with a diameter of (21 mm) and a height of (42 mm) were used. Specimens should be kept at a

particular temperature of (2∓23 °C) in the laboratory. Samples were removed from the mold when fully solidified at the 24 hours. Then, For seven or twenty-eight days, these samples had been cured in a laboratory environment. Figure(3.3) shows Flow chart summarized the experimental work performed in the current study. Figure (3.4) showing a specimen of geopolymer cement paste.

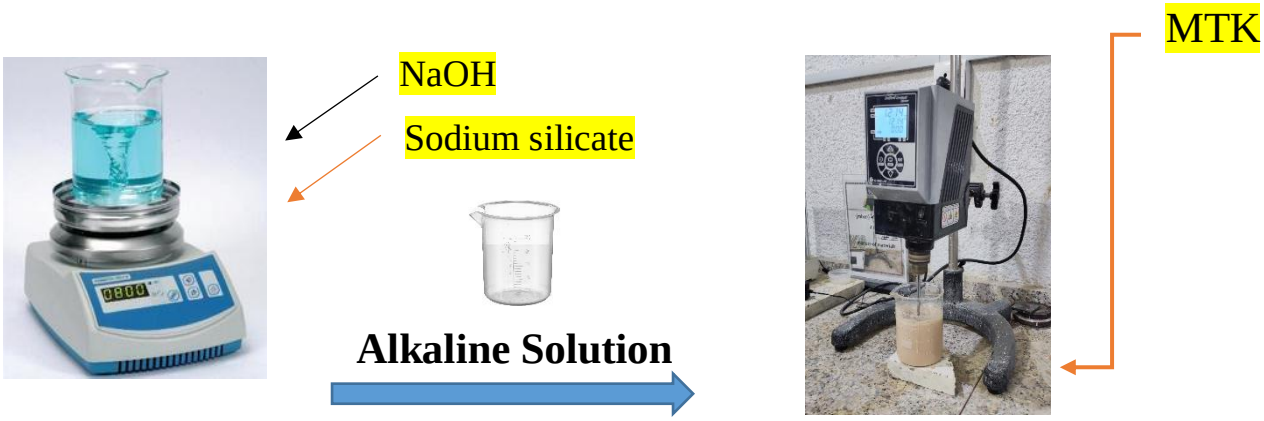


FIGURE 3.2 Scheme of the GSLM preparation procedure.

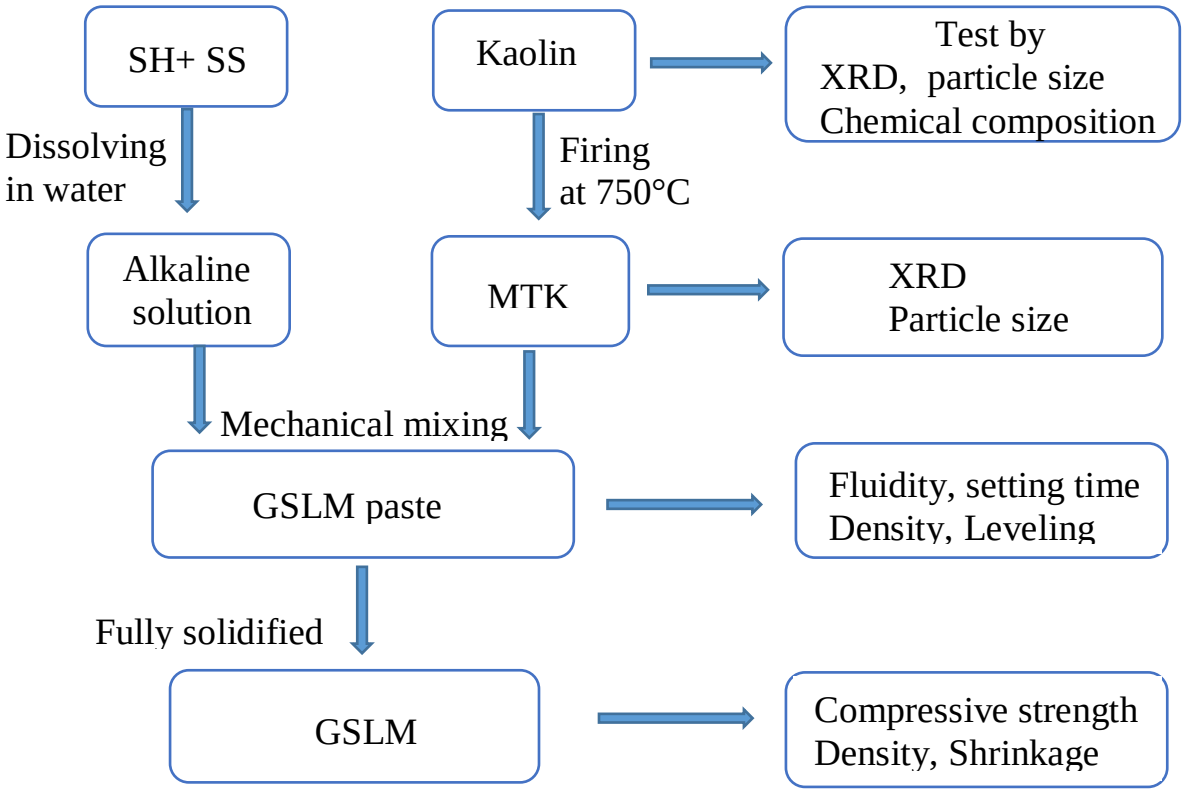


FIGURE 3.3 Flow chart of experimental work performed in the current study

TABLE 3.3 Mix proportion of GSLM

No. sample	MTK (gm)	Sodium silicate (gm)	NaOH (gm)	H2O (ml)
G1.1	70.506	45.77	7.718	31.23
G1.2	70.506	45.77	7.718	38.23
G1.3	70.506	45.77	7.718	45.23
G1.4	70.506	45.77	7.718	52.23
G2.1	70.506	79.618	7.718	12.926
G2.2	70.506	79.618	7.718	19.926
G2.3	70.506	79.618	7.718	26.926
G2.4	70.506	79.618	7.718	33.926

FIGURE 3.4 The specimen of geopolymer cement paste after 24 hours past casting

3.4 Testing Methods

3.4.1 XRD X-Ray Diffraction

The phase composition of the geopolymer and the production of amorphous metakaolin phase were determined using XRD analysis

The x-ray was created by using high voltage electricity (40kv/30mA), which fast-tracks electrons from a heated tungsten filament to impact the copper target. The x-ray is created and travels through a nickel filter before hitting the specimen, which is rotating in a circular motion at a speed of 5°/min. When the incident beam diffracts according to Bragg's law, as shown in equation (3.1), diffraction occurs.

$n\lambda = 2d \sin\theta$ ---------- (3.1) [88].

Where (λ) is the incident wavelength ray (λ=1.5406 angstrom), (d) is the inter-planar distance (A˚), the angle (θ) is located between the incident beam and the surface of specimen, and (n) is a positive integer. The rotating detector, which rotated at a speed of (2θ), receives the x-ray diffracted from the sample. Finally, a computerized system was used to collect diffraction data (the intensity of the diffracted beam and the values of diffraction angls). The d-spacing values of the tested samples were compared to the International Center of Diffraction Data's (ICDD) d-spacing standard data for ceramic materials. This test was carried out using an x-ray diffractometer (type: XRD 6000, produced in Shimadzu, Japan) available at the University of Babylon's/ College of Materials Engineering, Department of Ceramics Engineering and Building Materials,.

3.4.2 Particle Size Analysis

The purpose of this test was to measure the metakaoline powder's particle size distribution utilizing a Laser particle size analyzer available at the University of Babylon's (College of Materials Engineering/ ceramics engineering and building materials department).

Laser diffraction is based on a very basic principle: Diffraction, or light scattering on a particle, results in an angle-dependent intensity distribution behind the particle, which consists of a ring system with bright and dark parts. The bright and dark region intervals vary depending on particle size, with small particles generating broad ring gaps and big particles resulting in intensity distributions with closely bordering rings. The distances between the rings may be calculated accurately using the Mie-theory, however with sufficiently large particle

diameters, the Fraunhofer-approximation theory can be applied. This test is mixing a small amount of powder with 500 mL of distilled water, placing the solution within the device, and reading the results from the computer device connected to the device.

3.4.3 Fluidity of GSLM

The fluidity was measured using a mini slump cone, made of Teflon for the current study with dimensions (38 mm bottom diameter, 19 mm top diameter and a height of 57 mm) according to literatures [86, 89, 90] figure (3.5). In order to conduct the test, the test cone was positioned horizontally in the middle of a plate glass (30 cm 30 cm) and filled with the test material. After vertically lifting the mold, measurements were taken of the paste's spread-out diameters in two perpendicular directions, and the average value was used to determine the final spread diameter [89]. The flow feature is taken over two hours at 30 minute intervals. Based on equation (3.2), the % spread was determined using the measured value of the final diameter average.

$$\% \text{ spread } = \frac{\text{final diameter} - \text{initial diameter}}{\text{initial diameter}} \times 100\% \text{----- (3.2) [90].}$$

a- Cross section of the mold

b- Upper section of the mold during examination

FIGURE 3.5 Schematic diagram of mini-slump test cone.

By comparing the results with previous studies, table 3.4 show the reslts of previous studies for the fluidity test.

TABLE 3.4 Results from previous studies for the fluidity test

Mold dimensions	Results	Reference
height of 50 mm ± 0.1 mm, inner diameter of 30 mm ± 0.1 mm	flow values are ranged (130mm , 160 mm) and fluidity higher than 250%	[63-65, 69, 91]
cone with: height of 57 mm, top diameter 19 mm and bottom diameter is 38 mm	fluidity higher than 300%	[68]
cone with: top radius 20 mm and bottom radius 40 mm and a height of 60 mm	flow value of 158 mm was obtained	[92]
according to J-16B-103	The flow value was 220mm	[68]

As a result of the above Self-leveling specifications were obtained for mixtures having a flow of ≥150mm or spread% $\geq 295\%$.

3.4.4 The Test of Compressive Strength

At the age of 7 and 28 days, this test was done on geopolymer. To ensure that the samples are flat, the specimen should be polished with a polishing machine. The test was conducted at the university of babylon's/ college of materials engineering department of ceramics engineering and building materials. The compression strength can be calculated using the formula below [93].

$$\sigma c = P/A \quad \text{---------- (3.3)}$$

Where: σc =compression strength (MPa)

P = load that used (KN)

A= sample area (mm^2)

3.4.5 Setting Time

The test was performed at the University of Babylon's/ College of Materials Engineering/ Department of Ceramics Engineering and Building Materials. The Vicat needle which shown in Figure 3.6 wes used to calculate the setting times of geopolymer paste in accordance with ASTM C191 [94]. The penetration was measured every fifteen minutes until a penetration of 5 mm or less was achieved. The initial setting was derived via depth of interpolation at 5 mm. The final setting time, defined as the period when the not clearly the needle sink into the paste, was also determined.

FIGURE 3.6 The Vicat needle device

3.4.6 Density Measurements

3.4.6.1 Density of mortar

A material's density is defined as its mass per unit volume (volumetric mass density, which is more exact; also known as specific mass). ρ is the most often used symbol for density. The definition of density is mass divided by volume.

$$\rho = m/V$$

Where m is the mass, V is the volume and ρ is the density. In this study, density is measured to determine the weight needed for a given unit volume.

3.4.6.2 Density of geopolymer GSLM

ASTM-C373-1988 indicates that [95], the density and porosity of the synthesized samples were tested as follows:

1. All the samples were dried to 120°C for 6 h, and cooled in desiccators to normal temperature of room. The dry weight (D) was determined to the closest 0.001 g.

2. The samples were immersed in purified water and heated to 100°C for 5 hours, with the water surrounding the samples. After then, the samples are immersed in water for another 24 hours. After that, when the sample was hanging in water, the hanging mass (S) was measured to the closest 0.01 gram.

3. After measuring the (S), the surface of the samples are dried with a cloth to remove any excess water. The samples are weighed to the closest 0.01g to determine the soaking mass (M). Using the equation below, the outer volume (Ve) it calculated

$$Ve = \frac{M-S}{\rho w} \qquad \text{---------- (3.4)}$$

Where ρw represents the density of distilled water (1 g/cm3), S represents the hanging weight in grams, and M represents the soaking weight in grams.

The percentage ratio of dry mass divided by the outer volume including pores gives the sample's bulk density (B) in g/cm3.

$$B = \frac{D}{Ve} \qquad \text{---------- (3.5)}$$

The fraction of the sample's volume that is made up of open pores to its total volume is known as apparent porosity. Using the following equation, the apparent porosity was calculated:

$$P = \frac{M-D}{Ve} * 100\% \qquad \text{---------- (3.6)}$$

The following equation was used to determine water adsorption:

$$Aw\% = \frac{M-D}{D} * 100\% \qquad \text{---------- (3.7)}$$

3.4.7 Test of Shrinkage of self-leveling compounds

The length changes of bars (25x25x150 mm) were used to calculate shrinkage GSLM show (figure 3.7). The bars were examined, The specimens were kept in their molds for the first 24 hours. After 24 hours, the bars were demolded, and the measurements began immediately. After that, the bars were kept at room temperature and were monitored for 28 days. Based on equation (3.8), the % shrinkage was determined using the measured value of the length.

$$\%\text{shrinkage} = \frac{\text{final length} - \text{initial length}}{\text{initial length}} \times 100\% \qquad \text{----------(3.8)}$$

3.4.8 Leveling test

The flow-ability of the best mix was assessed in this nonstandard test by pouring it onto a surface of a concrete slab measuring 30 x 30 cm which is shown in Figure 3.8. It was placed diagonally and cracks and holes are made on its surface.

Before starting the application of the GSLM on the concrete slab, the amount of water absorbed by the concrete slab must be provided in order to ensure that water is not absorbed from the GSLM mixture and does not affect its specifications. the water absorbency of the concrete slab was taken into account. Where it was measured by the concrete slab were dry at 105°C for 6 hours, and cooled in desiccators to normal temperature of room. The dry weight (D) was determined. then soaked it for 24 hours in water and the saturated weight (S) was taken. The amount of water absorbed by the concrete slab is calculated by the equation 3.9.

$$Wa = S - D \qquad \text{……..(3.9)}$$

FIGURE 3.7 Samples shape for shrinkage test

FIGURE 3.8 The concrete slab used for testing

CHAPTER FOUR

RESULTS AND DISCUSSION

CHAPTER FOUR
RESULTS AND DISCUSSION

4.1 Introduction

The experimental work's results are presented in this chapter, and they are discussed, Includes a description of the effect of the mixing ratio of the components on rheology and compressive strength.

4.2 Result of Kaolin and Metakaolin

4.2.1 XRD Analysis

The XRD patterns of kaolin powder is shown in Figure 4.1. The crystalline structure of the powder used is confirmed by the pattern. The characteristic peaks of kaolin were observed and the peaks were compared using x-pert program to verify the components. After the analysis, it was found that the peaks match the codes (96-901-5000 kaolinite) and (01-089-8935 silicon oxide).

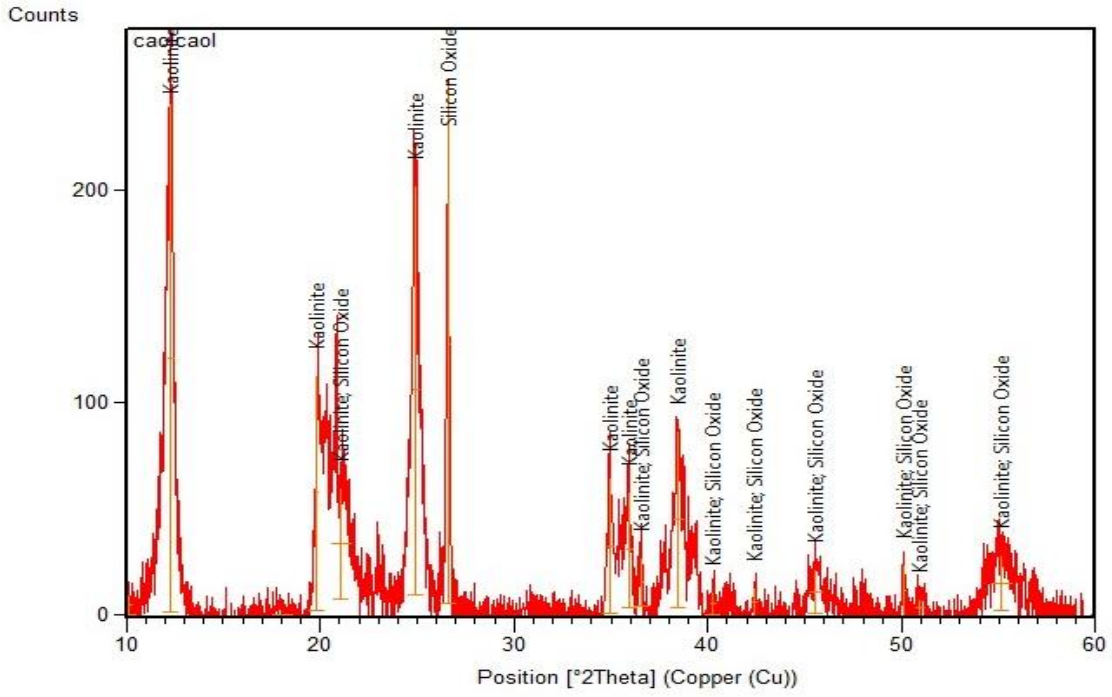


FIGURE 4.1 kaolin XRD form

Generally, the production of metakaolin (MTK) from moderate temperature heat treatment of kaolin, can be confirmed for kaolin that was heated for three hours at 750 °C by XRD results, as shown in Figure 4.2. After analyzing the peaks of metakaolin, it was found that the peaks match the code 01-089-8936 silicon oxide. The pattern asserts the amorphous structure of metakaolin powder. XRD analysis of metakaoline demonstrates that there is a sharp peak refers to quartz in the kaolin powder.

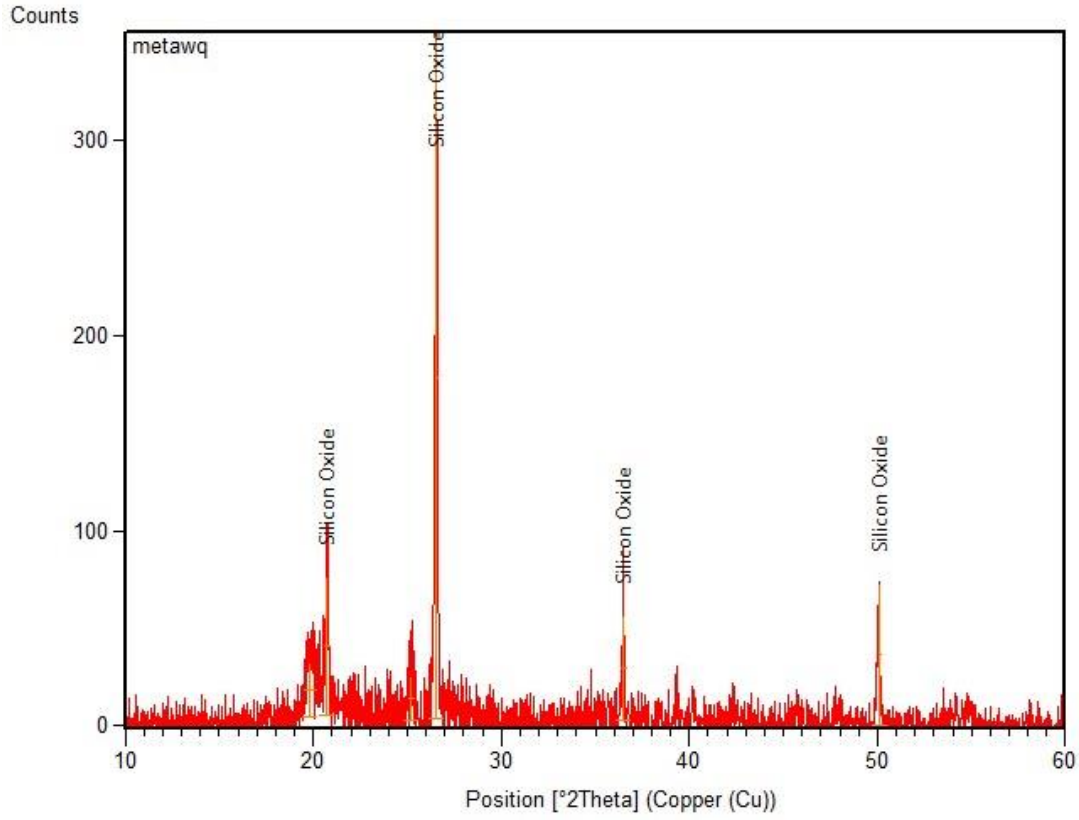


FIGURE 4.2 Metakaolin XRD form.

4.2.2 Result of Particle Size Analysis For Kaolin

The distribution of particle size for kaolin powder is shown in Figure 4.3. The analysis indicated that kaolin is mostly made up of micro-sized particles less

than 20 micrometers. The particle size distribution is multimodal with D50 of $3.618\mu m$.

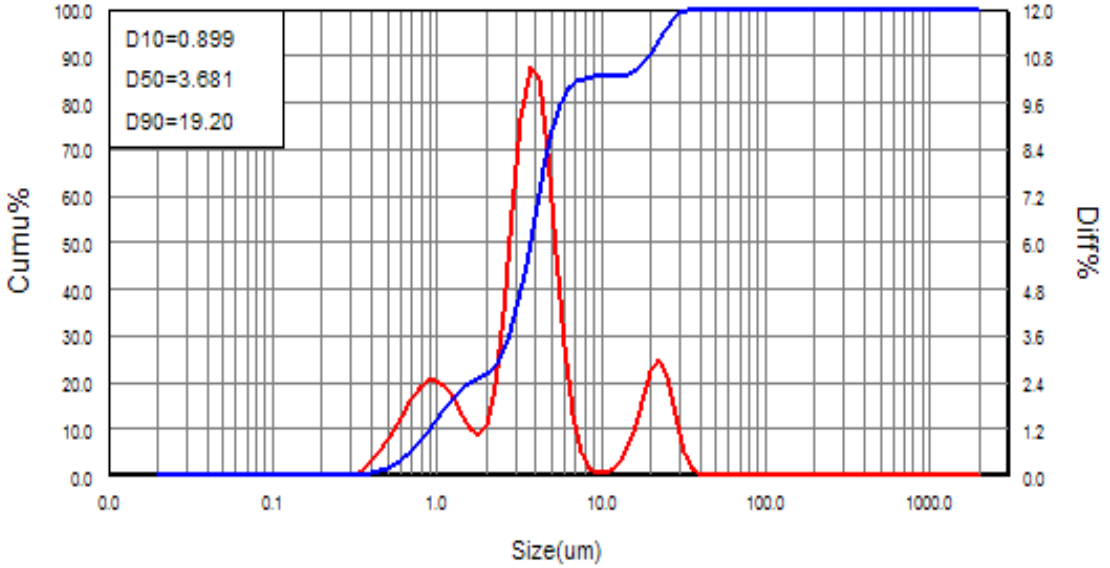


FIGURE 4.3 particle size analysis of kaolin

The distribution of particle size for (MTK) is shown in Figure 4.4, it is well known that metakaolin has a fine particle size as compound with kaolin as it is produced due to the breaking of the kaolinite structure. However, the agglomeration and aggregation lead to the formation of large secondary particles as confirmed in Figure 4.4.

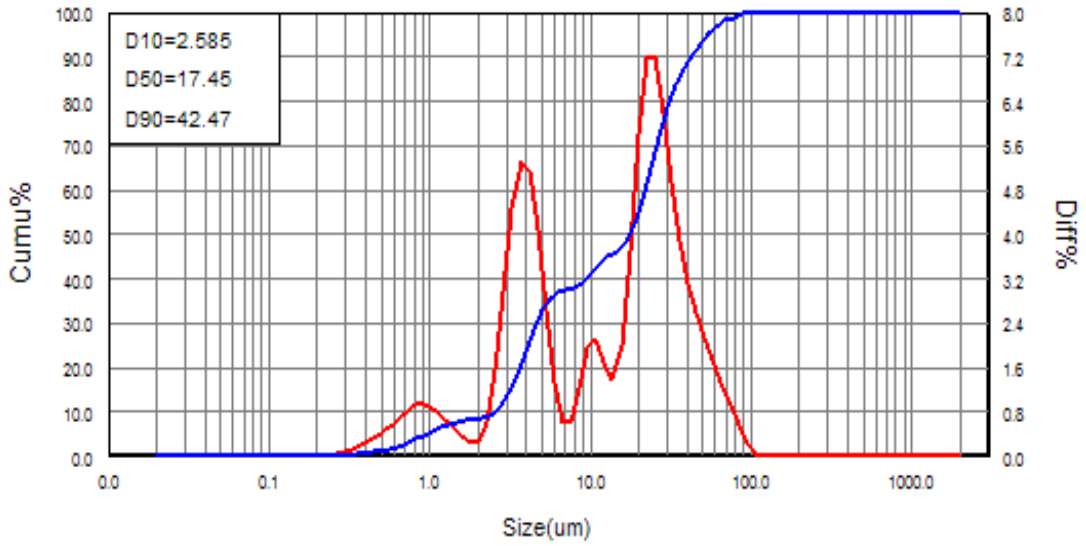


FIGURE 4.4 particle size analysis of Metakaolin

4.3 Results Of GSLM

4.3.1 Fluidity of GSLM

Results of flow test of the GSLM are given in Table 4.1. Figure 4.5 shows the values of the %spread. Self-leveling specifications were obtained for mixtures having a flow of $\geq 150mm$. As the flow increases with the increase in the amount of water added, therefore, an increase in the amount of water added to the mixture leads to a decrease in viscosity and an increase in the values of flow Except for the samples (G1.4 , G2.1 , G2.2), where sample G1.4 shows a segregation which happened due to the increase in the quantities of water and became unstable. And the samples (G2.1, G2.2) had a low flow value <150 mm, so the self-leveling specifications were not met.

The figure 4.6 shows the fluidity of GSLM for samples G2.2 and G1.4. We note that sample G2.2 has a small flow diameter, while sample G1.4 has segregation due to the amount of excess water.

TABLE 4.1 Flow and %spread of GSLM

No. sample	Flow value (mm)	% spread
G1.1	150	294.7 %
G1.2	165	334.2 %
G1.3	185	386.8 %
G1.4	200	426.3 %
G2.1	117.5	209.2 %
G2.2	130	242.1 %
G2.3	167	339.5 %
G2.4	175	360.5 %

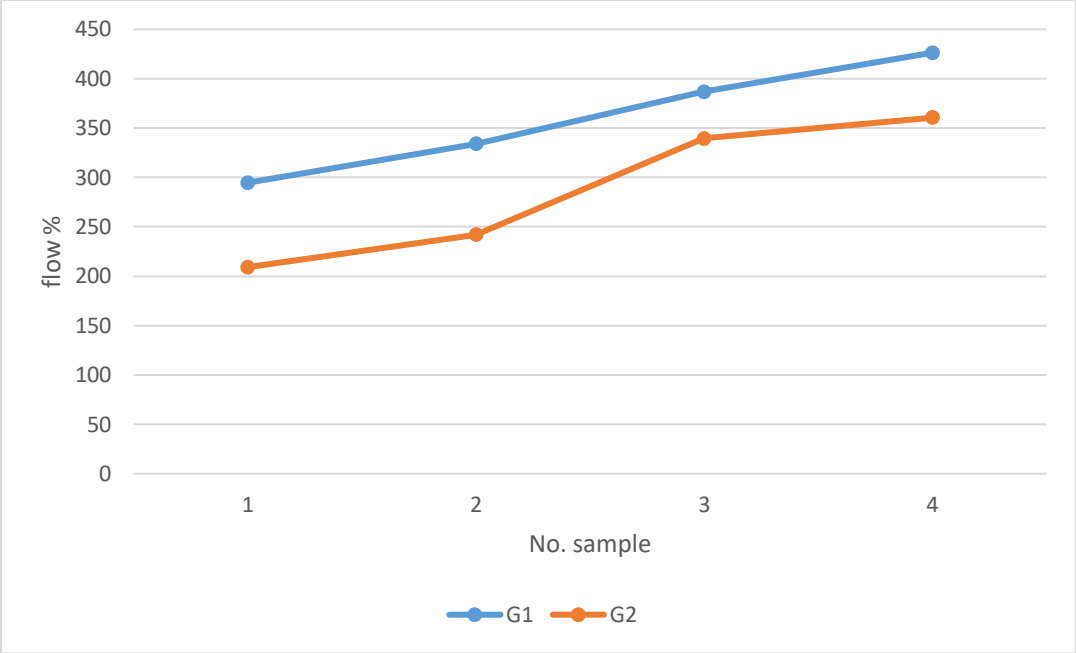
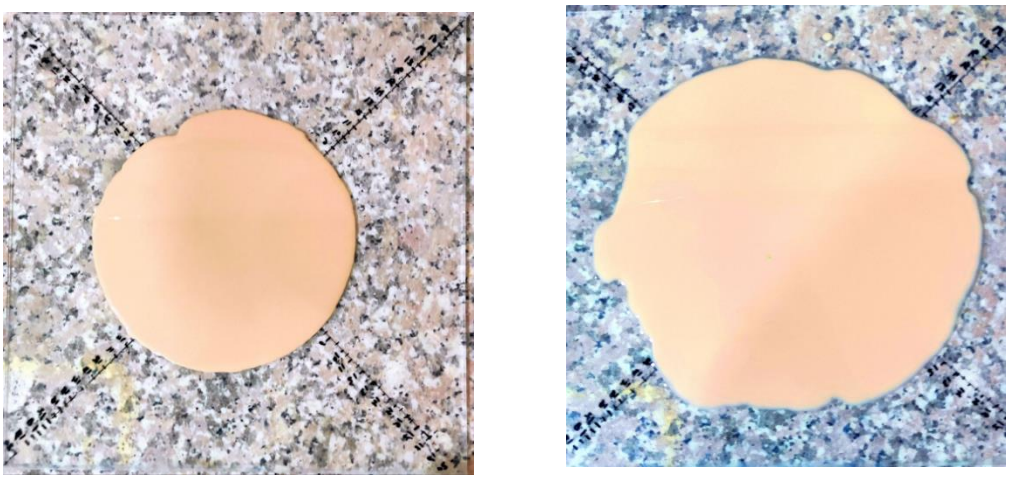


FIGURE 4.5 spread % of GSLM



a- For sample G2.2 b- For sample G1.4

FIGURE 4.6 The fluidity test using mini slump cone

4.3.2 Result of Density test

4.3.2.1 Result density of mortar

Table 4.2 shows the density of mortar. As the amount of water increases, the density values drop. It can be noticed that the sample from (G1) is very

close to density of sample from (G2), that contain the same amount of water, except the samples G1.3 and G2.3. The density of G2.3 is higher indication the higher degree of polymerization.

TABLE 4.2 The density of geopolymer mortar

No. sample	Density (gm/cm^3)
G1.1	1.81
G1.2	1.73
G1.3	1.62
G1.4	1.60
G2.1	1.82
G2.2	1.74
G2.3	1.71
G2.4	1.66

4.3.2.2 Result of density, porosity and water absorption of Geopolymer

Table 4.3 shows the bulk density, porosity and water absorption of GSLM. Results are the average of three measurements. The density values ranged from (1.21 – 1.49), since an increase in water causes an increase in the material's porosity, it is clear that the density drops as the amount of water increases. It can be noticed that the (G2) samples have higher density as compound with (G1) samples that have the same water content.

4.3.3 Result of Shrinkage test

Figure (4.7 and 4.8) shows the shrinkage test results. It can be noticed that the (G1) samples shrank more than the (G2) samples, due to the (G2) samples have highest polymerization. In addition to the shrinkage of G2.4 represents the highest

shrinkage value because it contains more water. Because there are no specified shrinkage performance standards, even the items with the largest shrinkage might be acceptable. However, the chance of shrinkage cracking increases as shrinking value increases.

TABLE 4.3 The porosity, density and water absorption of GSLM at 28 days.

No. sample	Bulk Density (gm/cm^3)	Porosity %	water absorption %
G1.1	1.37	38.24	27.95
G1.2	1.29	44.73	34.69
G1.3	1.25	44.92	36.04
G1.4	1.21	48.05	39.78
G2.1	1.49	31.74	21.27
G2.2	1.36	41.57	30.57
G2.3	1.33	44.28	33.33
G2.4	1.28	42.66	33.33

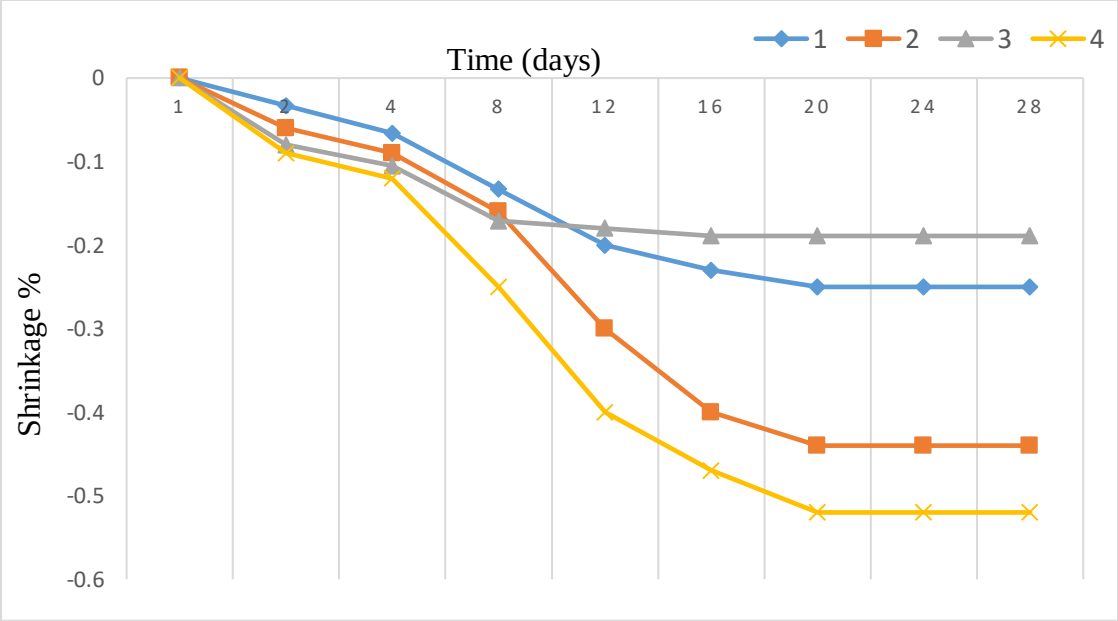


FIGURE 4.7 Results shrinkage test of G1 samples

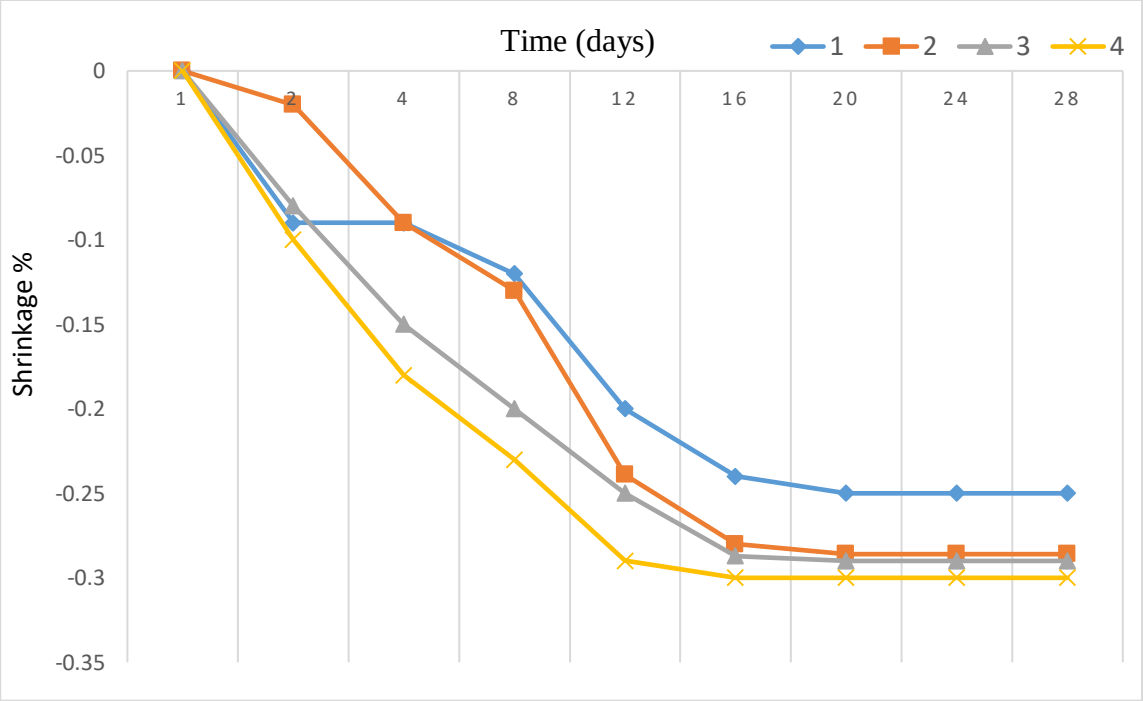


FIGURE 4.8 Results shrinkage test of G2 samples

4.3.4 Result of Compressive Strength

4.3.4.1 Compression Strength at Seven Days

The compressive strength of a different sample of geopolymer cement at 7 is shown in Table 4.4. The results reveal that the compressive strength values were good. As the samples give good compressive strength at early ages. This is important for flooring application.

4.3.4.2 Compression Strength after Twenty-Eight Days

The compressive strength of a different sample of geopolymer cement after 28 days is shown in Table 4.5. Where the results of the compressive strength of the samples of group G1 ranged (65.39 - 17.02) and for samples of group G2 ranged (62.49 – 22.52). Also, the compressive strength decreases with the increase in the amount of water added, because the increase in water leads to an increase in the porosity and a decrease in the density. Figure 4.9 and Figure 4.10 show the compressive strength of (G1 and G2) samples after (7 and 28) days.

TABLE 4.4 Comressive strength at 7 days of GSLM

No. sample	Compression Strength at 7 day (MPa)
G1.1	49.18
G1.2	22.5
G1.3	19.81
G1.4	16.29
G2.1	37.45
G2.2	26.75
G2.3	35.8
G2.4	25.4

TABLE 4.5 Comressive strength at 28 day of GSLM

No. sample	Compression Strength after 28 day (MPa)
G1.1	65.39
G1.2	58.021
G1.3	25.41
G1.4	17.02
G2.1	62.49
G2.2	45.25
G2.3	30.51
G2.4	22.52

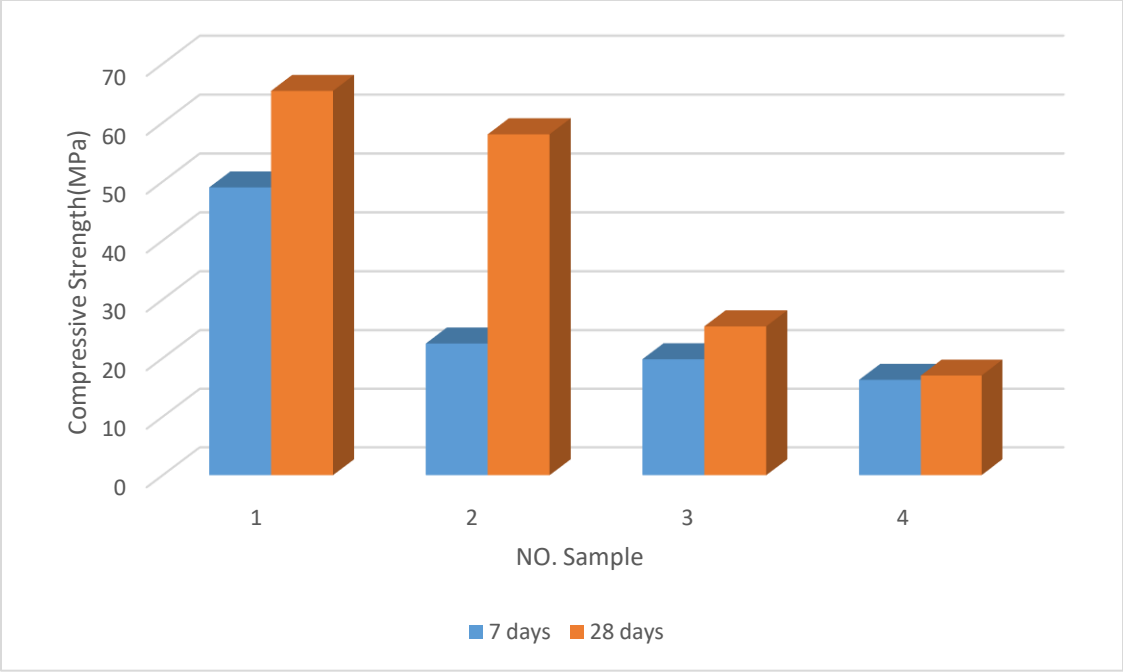


FIGURE 4.9 Compressive strength of G1 samples after 7 and 28-day

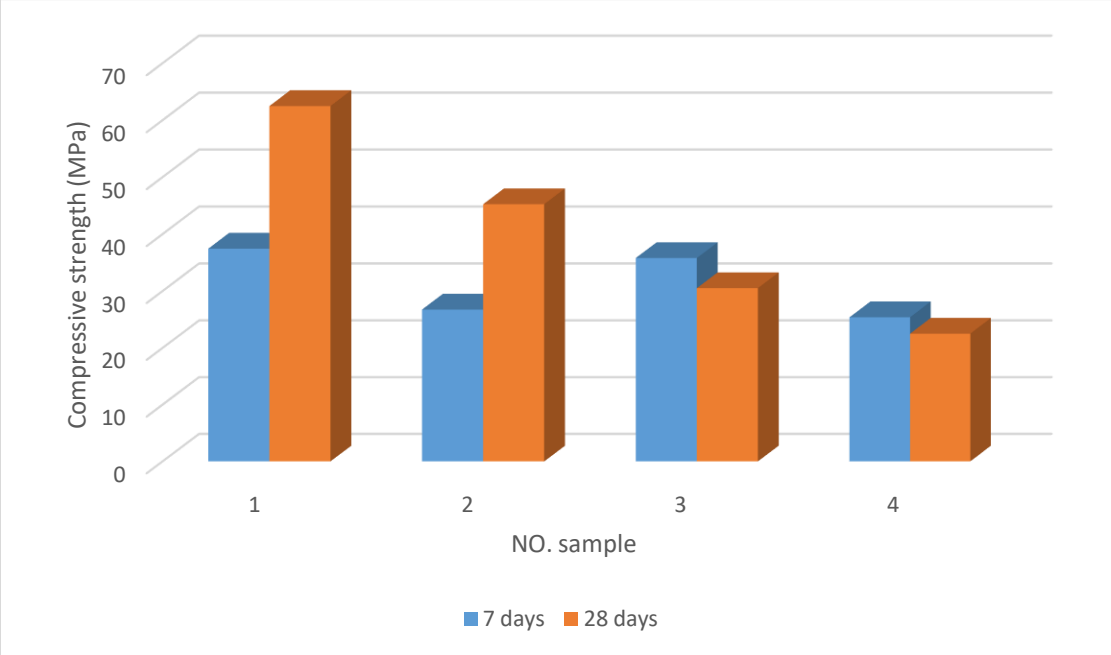


FIGURE 4.10 Compressive strength of G2 samples after 7 and 28-day

4.3.5 Result of Setting Time

The initial and final setting time as shown in (Table 4.6). As expected, more water added leads to an increase in both initial and final setting time for all batches.

TABLE 4.6 The Initial and final setting time of GSLM.

No. sample	Initial setting (Hour)	Final setting (Hour)
G1.1	11	17.5
G1.2	12.5	20
G1.3	14	21
G1.4	15	27
G2.1	18	22
G2.2	18.7	22.2
G2.3	19.3	22.5
G2.4	20	23

Since the geopolymerization process produces water, this was expected and according to Le Chateliers' principle, adding more water increases the reverse reaction. And since different amounts of water were used to obtain a suitable flow. We note a significant increase in setting time, and this time can be uses to allow flowing and surface preparation and not to harden quickly.

4.3.6 Result of leveling test

The mixture (G2.3) was selected to poured onto the concrete slab due to showed result is higher compressive strength, higher density and lower %shrinkage compared to the mixture (G1.3) containing the same amount of water added, which describes a higher degree of polymerization. And note its leveling with the leveling tool. Figure 4.11 shows the GSLM after pouring, and it can be noticed the top surface of GSLM is leveling, smooth and flat horizontal.

FIGURE 4.11 GSLM after poured on concrete slab

CHAPTER FIVE

CONCLUSIONS & RECOMMENDATIONS

CHAPTER FIVE

CONCLUSIONS & RECOMMENDATIONS

5.1 Conclusions

1. The increase in the amount of water resulted in the increase in the flow and a drop in the cable's compressive strength and density.
2. Increasing the flow within the same quantities of started materials leads to a small increase in shrinkage.
3. A decrease in the amount of sodium silicate causes shrinkage to increase and density to decrease.
4. The sample (G2.3) which contain more amount of sodium silicate showed result is higher compressive strength and density compared to the sample (G1.3) containing the same amount of water added, which describes a higher degree of polymerization.
5. Increased porosity negatively affected compressive strength.

5.2 Recommendations

Based on the results obtained in the current study, the following are recommended:

1. A study of the manufacture of self-leveling geopolymer cement paste using Metacholine (MTK) powder with particle size in nano scale, and study the effect of fine particle size on physical and mechanical properties.
2. Studying the effect of alkaline type on the fluidity of GSLM.

3. Studying the possibility of using fillers material such as fine aggregate in the manufacture of GSLM and its effect on the cost, physical and mechanical properties
4. Using SEM to study the microstructure, type of pores and their distribution
5. Study of adhesion with adhesives and floor covering materials

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الخلاصة:

الجيوبوليمر مادة جديدة نسبيًا جذبت انتباه العديد من الأكاديميين نظرًا لقلة تأثيرها على التلوث البيئي مقارنةً بالأسمنت البورتلاندي التقليدي, حيث يعتبر إنتاج الأسمنت البورتلاندي مادة عالية لاستهلاك الطاقة تنبعث منها كمية كبيرة من ثاني أكسيد الكربون (CO_2) في البيئة. يتم تطبيق مواد التسوية الذاتية في الغالب على سطح الأرضيات، فهي توفر أرضيات مستوية عالية الأداء وسهلة التطبيق. حيث يمكن أن تتصلب وتتحول إلى سطح مستوٍ ومقاوم للاهتراء دون الحاجة إلى الاهتزاز أو الترصيص. تقلل مواد التسوية الذاتية أيضًا من الضرر الذي قد يحدث نتيجة استخدام الأساليب التقليدية لتسوية الأرضيات.

عادة، يتم استخدام الإيبوكسي كمادة ذاتية التسوية ، أو يتم إضافة ملدنات الفائقة ذات الأساس البوليمري إلى الخلائط الخرسانية لزيادة قابلية التشغيل وتقليل لزوجتها ، وتصبح الخرسانة (خرسانة ذاتية التسوية). تتميز هذه المواد (الإيبوكسي ، الخرسانة ذاتية التسوية) بتكلفتها المرتفعة نسبيًا.

في هذه الدراسة ، ندرس إمكانية استخدام الجيوبوليمر كمواد ذاتية التسويه منخفضة التكلفة دون استخدام أي إضافات.

تم تحضير الجيوبوليمر القائم على الميتاكولين الذي يعتبر كمصدر للالومينا (Al_2O_3) و السليكا (SiO_2)، هيدروكسيد الصوديوم ($NaOH$) وسيليكات الصوديوم المائية (Na_2SiO_3) كمحلول قلوي. تم إجراء اختبار تحليل حجم الجسيمات و حيود الاشعة السينية (XRD) للمواد التي تم البدء بها (للميتاكولين). تم تصميم عدة مخاليط، و تم تحضير العينات لمجموعتين (G1، G2) تكون متساوية بكمية الماء المضاف و تختلف بكمية سليكات صوديوم. بالاضافه ان كل مجموعة تحتوي على 4 نسب خلط تختلف بكمية الماء, تم استخدامها لتحديد نسبة الخلط المثلى للمواد المتفاعلة. بالإضافة إلى ذلك، تم إجراء الاختبارات التالية على الجيوبوليمر: السيولة، تحديد الوقت التصلب الاولي و التصلب النهائي و الكثافة لعجينة الجيوبوليمر. وتم إجراء اختبارات الكثافة، مقاومة الانضغاط واختبار الانكماش بعد التصلب للجيوبوليمر.

تم الحصول على قيمة السيولة لعينات G1 ضمن النطاق (150 مم ، 200 مم) وتراوحت نسبة الانتشار (294.7٪ ، 426.3٪). وكانت النتيجة التي تم الحصول عليها لـعينات G2 ضمن النطاق (117.5 مم ، 175 مم) وتراوحت نسبة الانتشار (209.2٪ ، 360.5٪). تم اختيار خصائص التسوية الذاتية للخلائط التي لها سيولة $\leq$ 150 مم.

تم الحصول على مواصفات التسوية الذاتية وكانت أعلى مقاومة انضغاطية لعجينة الجيوبوليمر (65.39 ميجا باسكال بعمر 28 يومًا) والتي تعتبر عالية نسبيًا. و بمقارنة العينة (G1.3) و التي تحتوي على نفس كمية الماء للعينة (G2.3) , أظهرت النتائج أن العينة (G2.3) كانت تمتلك مقاومة ضغط وكثافة أعلى ومعدل انكماش اقل. مما يدل على درجة أعلى من تفاعل البلمرة.

وزارة التعليم العالي والبحث العلمي
جـــــامـــعـــة بـــــابـــــل
كـــليـــة هنـــدســـة الـــمـــواد
قسم هندسة السيراميك ومواد البناء

تصـنيـع مادة جيوبـولـيـمـر ذاتيـة التسـويـة

رسالة

مقدمة إلى كلية هندسة المواد /جامعة بابل كجزء من متطلبات نيل درجة الدبلوم العالي في هندسة المواد / سيراميك

من قبل

زيـد عبـد الكريـم عبـاس حسون

بأشراف

أ.د. عمـاد علـي دشـر الحيدري

1443 هـ **2022 م**