

Effect of Various Factors on the Manufacturing of Geopolymer Mortar

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Abstract

In the recent years, research focus on the sustainable construction development. Due to high range of carbon dioxide emissions associated with manufacturing process of ordinary Portland cement that is finding alternatives procedures. This research studies the possibility of geopolymer mortar manufacturing from fly ash and Metakaolin then studies the fresh and hardened properties of this product. Two activating solutions were prepared for this purpose. The first is a mixture of sodium hydroxide/sodium silicate solution, fly ash, sand and water while the second is a mixture of sodium hydroxide/sodium silicate solution, Metakaolin, sand and water. Test results indicate that the compressive strength is directly affected the fly ash and Metakaolin content and significantly affected the curing condition. A total of ten mixtures were evaluated by considering the effects of aggregate content, alkaline solution to fly ash and Metakaolin ratio, sodium silicate to sodium hydroxide ratio, and curing method. Three optimal mixtures (M6, M7 and M8) were identified. Results show that the geopolymer mortar can be produced with compressive strength of 19.4 MPa at 28 days.

Keywords: Fly ash; Geopolymer mortar; Compressive strength; Sodium silicate activating solution

Introduction

Geopolymer is reaction of aluminosilicate materials with hydroxide or silicate solution (aqueous alkali) to produce alkali aluminosilicate compound, the term inorganic polymer is more common than geopolymer [1]. Geopolymer gives equivalent performance to normal cementitious binders in various ranges of utilizations, yet with more favorable position in bringing down emission of greenhouse [2]. Aluminosilicate materials show a wide differing of characteristics, upon on selection of raw materials, mixing procedure, curing methods and its temperature, alkaline solution concentration. Today, geopolymer is common reaching in an industry segment and considered an option and has been utilized as a part of various field applications like precast beams, boat ramp, pavement, bricks, retaining wall, water tanks, precast bridge decks [3]. Geopolymer concrete has specialized advantages on ordinary concrete, earlier gaining strength and higher compressive strength, less hydration heat, higher chemical resistance, good sulfate attack resistance, also good resistance to acid. Every one of these properties make geopolymer best and generally utilized [4].

Numerous materials can be utilized for assembling geopolymer as Metakaolin, fly Ash, slag, rice husk ash. Metakaolin as a source for aluminosilicate is utilized for production geopolymer. Metakaolin being produced by calcination of kaolinite clay at (500-800°C) relying on purity of this material [5]. Geopolymer binder action similarly as Portland cement. At room temperature its set and harden, and can increase sensible strength in brief time. Some proportions of geopolymer binders have been verified to be effective in construction, infrastructure applications with good properties as high mechanical performance, hard surface, thermal stability, good durability, and high acid resistance. Each present building part for example bricks, ceramic tiles and cement could be supplanted with geopolymer. Geopolymer does not needs extraordinary high production temperature, just low temperature handling of normally happening or specifically manual is applied to Kaoline or FA, mixture gives appropriate geopolymeric raw materials. These prompt critical lessening in the vitality utilization and emission of CO₂. Around (0.6) less energy is required and (80-90%)

less CO₂ is created for generation of geopolymer than Portland cement. Therefore, it is great significance for environmental protection [5].

Yip et al. [6] studied the effect of the addition of calcium to Metakaolin Geopolymer concrete. The addition of 20% of calcium as a replacement improves in compressive strength, while by increasing calcium amount more than 40% compressive strength decreases. Within two days, strength development was close to complete. After 7 days, no significant increase in strength is shown.

Khater [7] studied the effect of Silicafume on the Geopolymer concrete produced from Metakaolin. Silicafume replacement started from 0% up to 10%. The specimen was cured at room temperature for (1, 7, 28 and 90 days). By increase curing time, the compressive strength evidently increased because hydration was progressive to form CSH in addition to produce Geopolymer gel lead to fine and homogeneous structure. Strength improving by increasing SF up to 7% then the strength decreased after this percentage up to 10%. Pozzolanic activity of Silicafume that have ability to enhance the properties like compressive strength, abrasion resistance and bond strength. Silicafume have ability to produce C-S-H (by consuming calcium hydroxide).

Al Bakri et al. [8] reported the effect of curing time and curing temperature on compressive strength of Fly ash based geopolymer concrete. When curing temperature and time increase, compressive strength increases. The maximum strength value optioned with curing temperatures range from 60-90°C within a period from 24-72 hrs.

Wang et al. [9] studied the effect of NaOH concentrations on Metakaolin geopolymer paste. NaOH concentrations effect on

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Received November 20, 2017; Accepted November 29, 2017; Published December 05, 2017

Citation: Hameed AM, Rawdhan RR, Al-Mishhadani SA (2017) Effect of Various Factors on the Manufacturing of Geopolymer Mortar. Arch Sci 1: 111.

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geopolymer pastes was investigated using different concentration of NaOH (8, 10, 12, 14 and 16 mol/L). Compressive strength of geopolymer increased with increasing NaOH concentration for 7 days age specimen. There results indicated that the compressive strength increases with NaOH concentration up to the studied value of 16 mol/L.

Kamarudin et al. [10] studied the effect of different concentration of NaOH on Kaolin Geopolymer concrete. This study focused on the development of compressive strength of Geopolymer from NaOH of different concentrations (6, 8, 10, 12 and 14M) at age of 1, 2 and 3 days curing in an oven at a temperature of 80°C. After, curing of the specimen for 1 day and 2 days, there were no steady strength gains for different concentrations of NaOH solutions. Compressive strength is highest when NaOH concentration was 12M for 3 day curing. The strength improved with an increase in NaOH concentration in solutions, the activation of binder become stronger and quicker. With increasing NaOH concentration solubility of aluminosilicate enhanced.

Demie et al. [11] studied the effect of curing temperature on compressive strength. The result referred to the compressive strength increased when the specimens were cured in oven up to 70°C for 48 hrs, while the specimens cured at curing temperature above 70°C results in a decrease in the compressive strength.

Rovnaník [12] studied the effect of curing temperature and curing time for Metakaolin based geopolymer on the development of dense structure. First group of specimens were cured at temperatures (10, 20, 40, 60 and 80°C) using electrical oven for 4 hr and then kept at ambient temperature 20°C until the day of testing, only one specimen was stored in the fridge at (10°C) for the whole period before testing. The second group of specimens dealt with the effect of curing period at different temperatures. The specimens were cured at different time for 1, 2, 3 and 4 hrs at temperatures 40, 60 and 80°C. After that, the specimens are stored at an ambient temperature of 20°C until tested. Increasing temperature leads to accelerates formation of dense structure and accelerate geopolymerization reaction at early ages. The specimens cured at 60 and 80°C reached to final stage of strength at 24 hrs only, but there is reduction in strength as compared with specimens cured at ambient temperature (20°C), on the other hand the specimens cured at 10°C reach the same strength at 28 days.

Nath and Sarker [13] reported the effect of ambient curing at 23°C on compressive strength development of different geopolymer mixtures. Geopolymer manufacturing by using Fly ash only showed slowly to develop strength when cured in ambient condition (20-23°C). When granulated blast furnace slag (GBFS) was incorporated in the mixture as a part of total binder, the strength increased clearly. Compressive strength in 28-day increased up to 10 MPa for every 10% increment of slag content from control concrete. The increase in compressive strength by adding slag may be because of increase the calcium bearing compound in the dissolve binder which produced reaction product from both alkali activated fly ash and slag.

Liew et al. [14] reported the effect of curing regimes on Metakaolin geopolymer pastes. The results showed that curing at room temperature was unfeasible therefore, heat is relatively important in Metakaolin geopolymer synthesis. Low curing temperature, make the dissolution of the Metakaolin slow and thus the geopolymerization process was slow. The chemical reaction speeded up at higher temperature. The compressive strength improving at moderate elevated curing temperature (40 and 60°C). High temperature leads to quick

polymerization and forming Geopolymer gels with poor structure, therefore compressive strength decreases.

Mechanism of geopolymerization

Geopolymerization is complex process accompanied by an exothermic production, stages of geopolymerization are [5]:

- (a) Destruction to coagulation
- (b) Coagulation to condensation
- (c) Condensation to crystallization

Generally, two main stages are carried out to produce geopolymer concrete: first stage includes aluminosilicate dissolution and formation species of polymer, second stage includes growth of polymeric particles through nuclei achieve critical size also crystals start to create [15] as shown in Figures 1 and 2. Dissolution stage begins when Si-Al from raw materials contacts alkaline solution, to produce Si and Al species. Many variables influence formation of Si and Al species, like alkaline metal type (Na⁺ or K⁺), alkaline solution concentration, rate, time mixing. Reorientation stage includes Si and Al diffused into the oligomers after dissolution. Oligomers into aqueous phase form many networks by condensation to gel formation, filtration of reactive Al and Si species from the raw materials is happening at Al⁺³ and Si⁺⁴ dissolving on surface of source Si-Al materials are removed. Solidification stage incorporates for formation of continuous gel including rearranging and reorganization, three stages, lead to produce geopolymer with amorphous, or semi-crystalline, three-dimensional aluminosilicate, network [15]. Table 1 shows the applications of geopolymeric materials based on silica-to Alumina [16].

The purpose of this research is to produce new binding material (Geopolymer) that can bind components of conventional concrete and mortar (sand and gravel) instead of Portland cement depending on local materials (Metakaolin) in addition to imported materials (Fly ash).

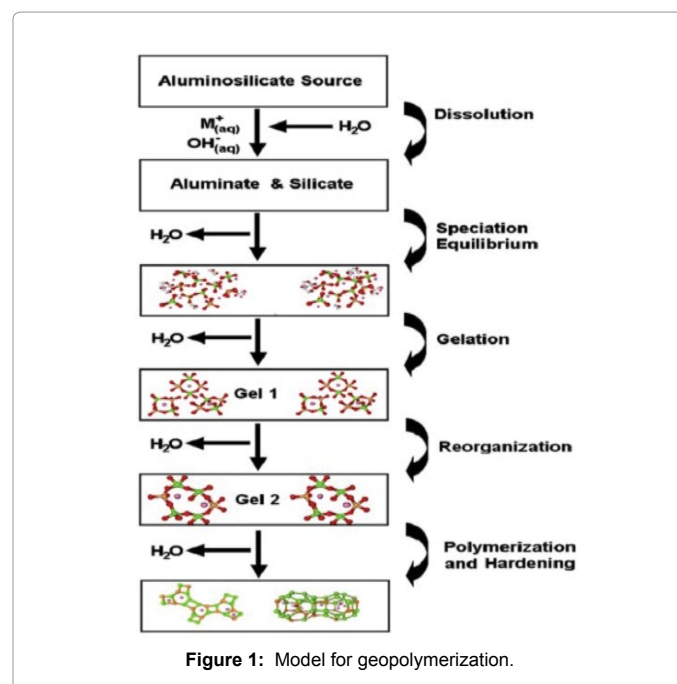


Figure 1: Model for geopolymerization.

Materials and Methods

The experimental work devoted to manufacture of geopolymer mortar by using metakaoline and fly ash. Many trial mixes were made to find out the optimum proportions. Many variables effect on this type of mortar are:

- (a) Fly ash, Metakaoline content
- (b) Ratio of Fly ash, Metakaoline to alkaline solution, NaOH and Na₂SiO₃
- (c) Extra water content
- (d) Ratio of NaOH to Na₂SiO₃
- (e) Type and dose of superplasticizer
- (f) Ratio of fine to total aggregate content
- (g) Methods of curing: sunlight curing, laboratory curing, heat curing

Materials and their properties

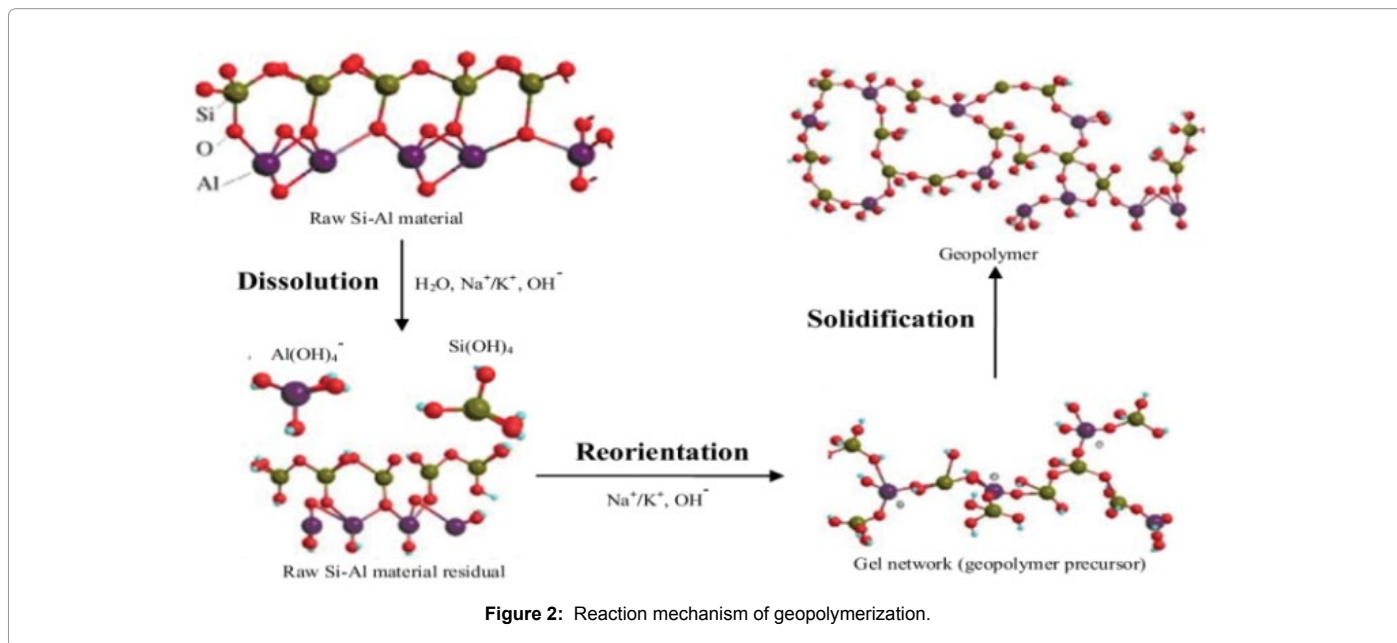
Fine aggregate: AL- Ekhaider sand was used as fine aggregate in

this work with ultimate size of 4.75 mm and grading limited zone II. Sieve analysis and grading curve of fine aggregate show in Table 2 and Figure 3. Results demonstrate that fine aggregate grading and sulfate content were within requirements of I.Q.S (No. 45/1984) [17]. Table 3 demonstrates the chemical and physical properties of sand.

Mixing water: Tap water was utilized for washing, mixing and curing during the experimental work that provided from water supply network system.

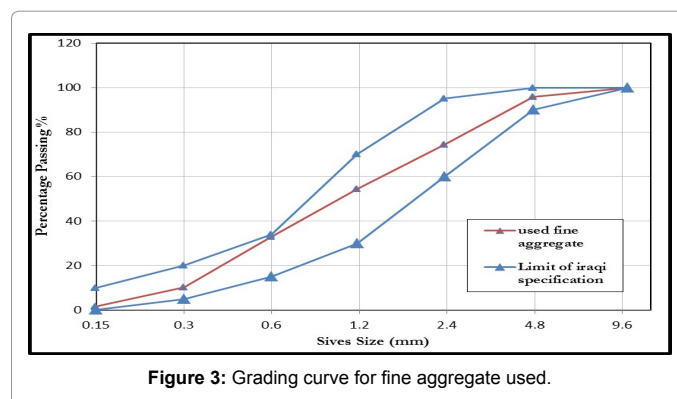
High range water reducing admixture (Superplasticizer): GLENIUM 54 superplasticizer produced by BASF construction chemicals UAE LLC. It relies upon modified polycarboxylic ether which was utilized into test work. Dosage recommended by manufacturer is from 0.5 to 2.5 liters per 100 kg of cementitious materials. This type of admixture is according to ASTM C494- 05 (Table 4) [18].

Fly ash: Fly ash (FA) is fine and glassy powder that is recovered due to coal combustion through production of electricity from ISKENment-Turkey power station. It is viewed as coal combustion waste. Composition of FA relies on source, however FA incorporates significant amounts of (SiO₂). It also contains (CaO), (Al₂O₃), (Fe₂O₃), Mg, K, Na, Ti, S which are also present in lesser amounts. FA atoms are



(Si:Al) ratio	Applications
1	Bricks Ceramics Fire protection
2	Low CO ₂ cements and concretes Radioactive and toxic waste encapsulation
3	Fire protection fibre glass composite Foundry equipments Heat resistant composites, 200°C to 1000°C Tooling for aeronautics titanium process
>3	Sealants for industry, 200°C to 600°C Tooling for aeronautics
20-35	Fire resistant and heat resistant fibre composites

Table 1: Applications of geopolymeric materials based on silica to alumina atomic ratio.



spherical shape particle size ranged (0.5-100) μm. Two principle types of FA: Class F, Class C. Class F have been examined, it contains less than 20% CaO. Chemical composition of FA utilized in this examination is exhibited in Table 5, physical requirements are recorded in Table 6. Results demonstrate that FA utilized as a part of this examination adjusts requirement of ASTM C 618 [19], as appeared in Table 5. Some proportion about 2 ml of Glenium 54 was added into mix to determine % flow equal 110%. Casting of specimens and cured in the lab, then were subjected into airtight container for cured in oven, at (60 ± 5°C) for six days as appeared in Figure 3. Pozzolanic strength activity index was calculated by equation 1.

$$P.I = (A/B) \times 100 \tag{1}$$

Where:-

P.I: pozzolanic strength activity index,

A: compressive strength, of 6 fly ash specimens, MPa,

B: Average compressive strength, of 6 reference specimens, MPa,

Sieve size (mm)	Cumulative passing %	Limits of Iraqi Specification No.45/1984, zone (2)
10	100	100
4.75	96.6	90 -100
2.36	92.4	60 -90
1.18	85.8	30 – 70
0.6	74.4	15 -34
0.3	40.3	May-20
0.15	6.9	0 -10

Table 2: Grading of fine aggregate.

Physical properties	Test Results	Limits of (IQS No.45/1984)
Specific gravity	2.6	-
Bulk Density (kg/m ³)	1729	-
Sulfate content %	0.343	≤ 0.5%
Absorption %	2	-

Table 3: Physical properties of fine aggregate.

Property	Description
Form	liquid
Color	light brown
Relative density	1.07gm/cm ³ at 20°C
PH	05-Aug
Viscosity	128+/-30 cps at 20°C
Transport	not dangerous
Labeling	not hazard
Storage	stored in original containers above 5°C
Packaging	available in 208 litter drums

Table 4: Properties of Glenium 54.

Oxide	Content %
SiO ₂	57.63
Al ₂ O ₃	19.17
Fe ₂ O ₃	5.32
CaO	1
MgO	0.98
SO ₃	0.07
L.O.I	2.77

Table 5: Chemical analysis of fly ash.

Procedure of mixing and compressive test of specimens were performed according to ASTM C305-02 [20] and ASTM C109/C 109M-05 [21] respectively.

Metakaolin: Kaolinite is a clay mineral with the chemical composition of Al₂Si₂O₅(OH)₄. Metakaolin is a dehydroxylated form of the clay mineral kaolinite associated with the reaction [22]:



Between 100-200°C, kaolinites lose most of their adsorbed water. In the range of 500-700°C, kaolinites become calcined by losing water through dehydroxilation. This process of changing kaolinite to Metakaolin is an endothermic process due to the large amount of energy required to remove the chemically bonded hydroxyl ions, which breaks down the crystal structure producing a transition phase (silica and amorphous alumina in reactive form) with high surface area.

Iraqi kaolin clay brought from Dewekhla region (Al-Anbar) was used. Kaolin was ground by air blast then burned in furnace up to 700 ± 20°C, for one hour, after that metakaolin was cooled for 24 hrs to the room temperature, depending on procedure mentioned by [5] and [23]. Table 7 shows the chemical analysis of metakaolin while Table

Physical properties	Fly ash	
Physical form	Powder	
Color	Grey	
Specific gravity	2.33	
Surface area, m ² /kg	773	
Chemical properties	Fly ash	Pozzolan class F
Oxide composition		
SiO ₂ +Al ₂ O ₃ +Fe ₂ O ₃ , min. percent	70	89.73
SO ₃ , max. percent	5	0.67
Loss on ignition, max. percent	6	2.77

Table 6: Physical and chemical properties of fly ash.

Oxide	Content %
SiO ₂	51.59
Al ₂ O ₃	38.11
Fe ₂ O ₃	1.82
CaO	0.45
MgO	0.23
SO ₃	0.14
Na ₂ O	0.11
K ₂ O	0.43
L.O.I	6.12
	Σ=99.00

Table 7: Chemical analysis of Metakaolin.

Physical property	Result
Physical form	Powder
Color	Off-white
Specific gravity	2.61
Surface area, m ² /kg	1650, 1960 and 2300

Table 8: Physical properties of Metakaolin.

Oxide composition	Pozzolan class N	Metakaolin
SiO ₂ +Al ₂ O ₃ +Fe ₂ O ₃ , min. percent	70	91.52
SO ₃ , max. percent	4	0.14
Loss on ignition, max. percent	10	6.12

Table 9: Chemical requirements of natural Pozzolan according to the ASTM C618.

8 illustrates physical properties of metakaolin and Table 9 focuses chemical requirements of pozzolan ASTM C618 [19].

Strength Activity Index for Metakaolin: The strength activity index for Metakaolin is conducted according to the ASTM C311-05 [24].

Manufacturing of metakaolin geopolymer mortar:

A. Preparation alkaline solution for geopolymer mortar

Solution of geopolymer mortar is constituted of sodium hydroxide and sodium silicate. Sodium hydroxide high purity, more than 98%, can be solved into distilled water to produce solution with appropriate concentration. NaOH concentration was varying from 5 to 16 Molar, mass of sodium hydroxide solids in solution varies relied upon solution concentration. The sodium silicate solution is commercially obtainable in various types. Throughout this work using solution of sodium silicate has a ratio of SiO₂ to Na₂O by mass which equals 2.4. The proportions, by mass of components, were SiO₂=32.5%, Na₂O=13.4%, and water=55.1%. After preparing NaOH as a solution, it was added to the Na₂SiO₃ solution. Alkaline liquid was combination of (NaOH) solution and Na₂SiO₃ solution, before using, must be alkaline liquid is prepare by mixing both solutions together at least 24 hrs. Extra water was usually used in geopolymer with different contents to improve workability. For more improvement, high range water reducing superplasticizer can be added which could result in strength improvements as well. The mixing of extra water with the superplasticizer was continued until a homogenous solution was produced. Finally, the alkaline liquid was added to the mixed extra water and superplasticizer together and mixing continues for not less than 2 minutes. Fifty trial mixes were made to select the optimum mix, depending on changing the components of mortar mixture for reaching to the best mix that giving higher compressive strength. Fine aggregate content, was kept constant for all mixes. The concentration of NaOH was 10 Molar, the mass ratio of NaOH to Na₂SiO₃ was 1:3.5 and the fineness of Metakaolin was 1650 m²/kg. The method of curing used for these trials was sunlight in August, when the temperature was ranging from 36 to 48°C as shown in Table 10 trial mixtures of geopolymer mortar.

B. Studying the effect of different curing systems on compressive strength of geopolymer mortar

Type of curing is considered very important factor effecting on the strength of geopolymer mortar. After mixing, casting, Metakaolin

geopolymer mortar specimens were demolded after 24 hrs and then the curing was started until the age of test. The followings are the used curing systems:

Sunlight curing: This method of curing was carried out by subjecting the specimens to the sunlight outside the laboratory after demolding. In Iraq, there is a high difference in temperature between winter and summer. To find out the effect of temperature of sunlight curing on strength of metakaolin geopolymer mortar; two groups of specimens were cast. The first was cured in January while the second were cured in August. The difference of temperature ranged from 12-19°C in January but changed from 36-48°C in August.

Laboratory curing: Specimens were placed inside the laboratory after removing from molds until the day of test. Also, two groups of specimens were made, one for winter and other for summer. The range of temperature inside the laboratory changed from 8-15°C in winter and ranged from 32-37°C in summer.

Oven curing: In this method of curing, the specimens were put into oven with temperatures ranged (70-100°C) and the curing process was continued until completing the age of test.

Water curing: Casted specimens were placed into a water tank after demolding and curing remained to the age of test. The temperature of water curing was kept at the range of 20 ± 2°C. This method is similar to that used for curing Portland cement.

Heating in oven and laboratory curing: After demolding the specimens heating in an oven was started. Temperature inside the oven was maintained at 60°C for 6 hrs, specimens were cured in laboratory temperature until the age of test. This system was adopted for curing only in winter when temperature deficiency.

Heating in oven and sunlight curing: In this method, heat curing process was started at first to accelerate the reaction of geopolymer mortar at 60°C for 6 hrs, then the specimens were put under sunlight in summer until the test day.

C. Mixing procedure of geopolymer mortar

Silica and aluminum oxide in metakaolin react with alkaline liquid solution (Na₂SiO₃ and NaOH) to form a paste of geopolymer that binds fine aggregate (sand) with other materials together to produce geopolymer mortar. Procedure of mixing have main effect

Mix type	FA g/cm ³	MK, g/cm ³	Fine Agg. g/cm ³	CaO %	NaOH to Na ₂ SiO ₃	M of NaOH	Water by wt. of FA, MK	Alkaline solution, by wt. of FA, MK	HRWR, ml/cm ³
M1	300	-	600	-	0.4	8	0.2	1:2	-
M2	300	-	600	-	0.4	8	0.2	1:2	-
M3	300	-	600	-	0.4	12	0.2	1:3	-
M4	270	-	600	30	0.5	12	0.2	1:3	-
M5	270	-	600	30	0.45	12	0.2	1:3	-
M6	240	-	600	60	0.45	8	0.2	1:2	-
M7	210	-	600	90	0.45	10	0.2	1:2.5	12
M8	300	-	600	-	0.45	12	0.1	1:3	12
M9	300	-	600	-	0.45	12	0.1	1:3	10
M10	300	-	600	-	0.45	12	0.3	1:2.5	14
M11	300	-	600	-	0.45	8	0.3	1:2	12
M12	-	300	600	-	0.45	10	0.1	1:2.5	12
M13	-	300	600	-	0.45	8	0.1	1:2.5	12
M14	-	300	600	-	0.45	8	3	1:2	-
M15	-	270	600	SF 10%	0.45	8	0.3	1:2	-

Table 10: Trial mixtures of geopolymer mortar.

on workability and strength of geopolymer mortar. Metakaolin, fine aggregate first were mixed together in dry form in a pan mixer for 3 min. Sand are prepared in saturated surface dry, superplasticizer, water was mixed with alkaline liquid for not less than 2 min. Then, liquid solution of mixture was added to the dry compounds into mixer pan for mixing continued usually for another 4 min. Casting in molds and compaction of geopolymer mortar by using vibrating table are then done. Compaction needed high skill because of the metakaolin based geopolymer mortar having zero flow while the compaction by rod was inappropriate technique.

Many trial mixes were conducted in this research to find out the optimum mix of metakaolin based geopolymer mortar. All component of mixture studied including metakaolin content, NaOH concentration, ratio of NaOH to Na₂SiO₃, G54 dose, ratio of metakaolin to alkaline solution. The optimum mix consisted of 500 kg of metakaolin, 80L of alkaline solution (NaOH to Na₂SiO₃=1:3.5), 3L of G54 superplasticizer, 40 kg of extra water and 720 of fine aggregate. The optimum mix used to study the fresh and hard properties of metakaolin geopolymer mortar.

D. Fresh properties

Flow test of geopolymer mortar was measured as traditional mortar test. Flow test conducted directly after mixing according to the ASTM C C1437-03 [25].

E. Hardened properties

The compressive strength test was done based on (ASTM C109/C 109M-05). Cubes with dimensions of 50x50x50 mm were tested by using hydraulic compression machine of 2000kN. Test was carried out at ages of 7 and 28 days under sunlight curing in January and August.

F. Utilize silicafume and aluminum oxide to produce modified geopolymer mortar

Silicafume was used to modify the metakaolin geopolymer mortar. Metakaolin was replaced by silicafume 10%. The specimens casting in cubes and cured under sunlight when temperature ranged from 36 to 48°C, till the day of testing and the compressive strength test conducted at 28 days. Alkaline solution was kept constant for all mixes (45% of binder weight) with NaOH: Na₂SiO₃ ratio equal 1:3.5, when the concentration of NaOH was 10 molar. Extra water was 10% of binder weight and 3 mL G54 superplasticizer for all mixes while the fine aggregate mass was 720 kg.

Manufacturing of FA geopolymer mortar: Fly ash based geopolymer mortar was produced for the comparison it with local metakaolin geopolymer mortar. Sunlight and heating curing were performed in August for curing fly ash geopolymer mortar. The tests conducted on fly ash geopolymer mortar included the flow and compressive strength tests. All these tests were conducted according to the same ASTM standard and same mixing, placing and curing for metakaolin geopolymer.

Fly ash-metakaolin geopolymer mortar: Fly ash used with metakaolin for modifying the Si/Al ratio; the compressive strength of geopolymer concrete was tested at 28 days. The optimum mix was used and the specimens (50 mm³ cubes) were cured by using heating, sunlight curing system in August when the temperature ranged between 36-48°C.

Results and Discussion

An attempt was made in the current study and numerous trials were

conducted to produce geopolymer mortar see Table 1. Diverse types of curing studied in this research to find out the perfect way for curing fly ash and metakaolin geopolymer mortars such as the mixing types illustrated in Table 11. Compressive strength could be considered as scale for choosing the best type for curing that suitable with fly ash and metakaolin geopolymer mortar. Table 11 illustrates the effect of curing systems on the compressive strength of geopolymer mortar.

Effect of curing temperature

Table 11 shows that the effect of curing temperature on the 7 and 28 days strengths of all mixes which cured at 70°C gives significantly increased strengths compared to those cured at 50°C for the same time. Lower curing temperature leads to mitigation of such expansion thus reflected negatively on the strength values.

Investigations on reactivity of FA under the effect of thermal curing reported that the good curing should be worked with temperatures in the range 30°C to 85°C [26]. Reaction kinetics resulting from different curing temperature were investigated with isothermal calorimetric tests on different materials (portland cement, activated slag and fly ash, and selected fly ash-slag blends) at 25°C, 35°C and 40°C observing the development of the polymerisation.

Effect of alkali ratio

An increase in alkali dosage (M⁺) resulted in an increase of the strength up to an M⁺ of 1.5%. Beyond this 'optimum' value, the strengths decreased, which is attributed to saturation of the gel with alkali ions resulting in less free water to be available for speciation of silica and alumina oligomers from the dissolution of FA [26]. There are many types of alkali activators that can be used to activate fly ash, metakaolin. For example, sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium silicate (Na₂SiO₃), potassium silicate (K₂SiO₃), calcium hydroxide (Ca(OH)₂). The combined use of NaOH and Na₂SiO₃ is the most common practice in term of cost effectiveness to produce geopolymer with good compressive strength than the solely use of alkali silicate or hydroxide. The ratio of alkali activator/ash can be a critical parameter to the strength development. High alkali activator/ash increases the viscosity and difficulty in compaction. The workability and compressive strength decrease by further increment of the alkali activator/ash and Na₂SiO₃/NaOH.

According to the compressive strength results appeared in Table 11, sunlight curing in summer gives compressive strength equivalent to 16.9 MPa at 28 days when temperature ranged from 40 to 50°C, but compressive strength diminished obviously in winter when

Mix type	Curing system		Compressive Strength, MPa at age:	
			7 days	28 days
M1	Sunlight	Summer Temp. (40-50°C)	10.3	16.9
M2		Winter Temp. (15-20°C)	9.5	15.6
M3	Laboratory	Summer Temp. (30-35°C)	8.2	13.53
M4		Winter Temp. (8-15°C)	6.4	10.5
M5	Water		10.2	16.58
M6	Oven Temp.(70-80°C)		12.1	19.9
M7			12.06	19.4
M8			11.3	19.05
M9			10.9	16.2
M10			11.6	17

Table 11: Effect of curing systems on compressive strength of geopolymer mortar.

temperature was between 15 and 20°C. Increasing of temperature accelerates formation of hard structure particularly in early stage of geopolymerization reaction [12]. Distinction in compressive strength between summer and winter was around 8.42% at 7 days and 8.33% at 28 days. Compressive strength for laboratory cured mix was too close to sunlight curing. As was the case in sunlight curing, laboratory curing specimens had high compressive strength at high temperature (in summer) and also there was reduction in strength when temperature decreased (in winter). Curing with temperatures of (30-35°C) was viewed good mechanism for gaining strength and helping for geopolymerization that needs to heat with temperature of more than 35°C [1].

Difference in compressive strength between summer and winter was around 28.12% at 7 days and 28.85% at 28 days. Water curing for fly ash and metakaolin geopolymer mortars could be viewed as ineffectual for providing geopolymer mortar with moderate compressive strength. This type of curing was unsatisfactory and prompts to deceleration of reaction because of water inter inside mortar and makes expansion causing cracks into geopolymer structure. Optimum curing technique into oven at temperature (70-80°C) gave highest compressive strength 19.9 MPa at 28 days, increasing temperature of curing tend to increase geopolymerization process accordingly. There was increase in rate of improved strength at early ages with reduction in later ages. At low temperatures, such as 10°C, geopolymerization process was very slow, therefore compressive strength was very low in spite of only amorphous phase is available.

For mass production in precast factories sunlight curing system and oven curing in summer are viewed as reasonable systems for curing fly ash and Metakaolin based geopolymer mortars. Such a case is very convenient and economical in hot weather countries.

Effect of high-range water reducer dose

The increase in high-range water reducer content leads to an increase in compressive strength of geopolymer up to an optimum dose, 12 ml/cm³, at which strength reaches 12.1, 19.9 MPa at 7 and 28 days age respectively. After that limit, the strength decreases. By increasing the dose from 12 to 14 ml/cm³, mix M10, the reduction in compressive strength was 14.5% and that may be because of increasing of voids in the geopolymer mortar structure which have an adverse effect on compressive strength [27]. Moreover, decreasing high-range water reducer content from 12 to 10 ml/cm³, mix M9, caused the reduction by 15.5%. As the high-range water reducer dosage in the mix increases, the compressive strength increased. This was due to the more effective action of the high-range water reducer in facilitated and regulated compaction of the geopolymer mortar by enhancing workability [28].

Alkaline solution to fly ash, Metakaolin ratio

The results indicate that increasing the ratio of alkaline solution to Metakaolin from 40 to 45% of fly ash weight M5 causes an improvement in strength. For the alkaline Geopolymerisation reactions, more (Si) aids in the production of Si-O-Si bonds, and significantly increases the compressive strength of the Geopolymer. If the content of Si exceeds the suitable limit, the Geopolymerisation rate is negatively affected, leading to Geopolymer of low strength as shown in M4 when the alkaline solution to Fly ash ratio increased to 50% the compressive strength got a slight reduction that reaches to 36.3% when compared with mix M5 [10].

Alkaline solution by wt. of FA, MK	NaOH in Molarity	Compressive Strength MPa at	
		7 days Age	28 days Age
1:2	8	12.1	19.9
1:2.5	10	12.06	19.4
1:3	12	11.3	19.05

Table 12: Effect of NaOH concentration on compressive strength for fly ash and Metakaolin geopolymer.

Effect of NaOH concentration

The concentration of NaOH plays an important role in compressive strength development of fly ash and Metakaolin geopolymer mortar. Increasing NaOH concentration leads to an improve in compressive strength. Geopolymerization process needs strong alkali to activate the (Si) and (Al) in fly ash and Metakaolin. By increasing NaOH concentration ability of solution to leach (Si) and (Al) in fly ash and Metakaolin is improved due to the formation of alumino-silicate gel at an early stage that resulted from increasing NaOH concentration [29].

Formation of gel has two opposing effects. First, the depletion of ions which prompts further leaching of ions from fly ash and Metakaolin particles. Second, the thickening of solution results in a lower mobility of ions particularly at the surface of Metakaolin and retards the leaching out of ions.

Effect the ratio of Na₂SiO₃/NaOH

The effect of NaOH to Na₂SiO₃ ratio is very clear as shown in Table 12. By increasing this ratio, compressive strength increased till the ratio reaches 1:3 by mass (maximum compressive strength). The improve in compressive strength by increasing NaOH to Na₂SiO₃ ratio returned to increase dissolution of silica and alumina of Metakaolin also the increase of sodium content in the mixture. Sodium is important for the formation of geopolymer as it acts as charge balancing ions. But when increasing NaOH to Na₂SiO₃ ratio to 1:4, compressive strength decreased because of the excess of sodium silicate which hinders water evaporation and structure formation. Also excess sodium content can form sodium carbonate by atmospheric carbonation, and this may disrupt the polymerization process. Therefore the optimum ratio of NaOH to Na₂SiO₃ was 1:3 by mass for the Metakaolin based geopolymer mortar.

Conclusion

Local manufactured Metakaolin and fly ash were possible to be used for producing geopolymer mortar. Oven curing is considered the effective way for curing fly ash and Metakaolin geopolymer mortar. Sunlight curing system does not need energy, does not cause any environmental damage and without any cost. High ratio of alkali activator/ash and Na₂SiO₃/NaOH does not necessarily lead to high compressive strength. The optimum compressive strength was obtained when alkali activator/ash ratio was 0.45 regardless of the type of alkali activators. The effect of oxide molar ratios on the compressive strength was studied. Higher ratios do not lead to higher compressive strength.

The compressive strength of geopolymer using commercial grade Na₂SiO₃ was significantly higher than those using industrial grade Na₂SiO₃ regardless of the type of alkali hydroxide. Geopolymers can provide a desirable alternative to portland cement (PC) binders, not only for the environmental benefits arising from the avoidance of CO₂ emissions associated with PC production, but also in terms of their performance and durability.

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