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Geopolymer synthesis from industrials solid wastes

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بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

من اجتهد وأصاب فله أجران...ومن اجتهد ولم يصب فله أجر واحد ..

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LIST OF ABBREVIATIONS

Symbol	Description
OPC	Ordinary Portland Cement
FA	Fly ash
C-S-H	Calcium silicate hydrate
SH	Sodium hydroxide
MS	Microsilica
GBFS	Blast-furnace slag
XRD	X-ray diffraction
SEM	Scanning electron microscopy
DTA	Differential thermal analysis
TGA	Thermogravimetric analysis
SF	silica fume
W/S	water/solid ratio
KN	kilo Newton

شكر و تقدير:

مع إتمام هذا المشروع يسعني أن لا أنسى كل من وقف معي وساعدني ووجهني وقدم لي العون والمساعدة في هذا الصرح العلمي المتميز -قسم الكيمياء- في جامعة الإمام محمد بن سعود الإسلامية، ولعل على رأسهم البروفيسور/ محمد أحمد هيكل، الذي كان عوناً لي في تقديم التوجيه المستمر طيلة فترة المشروع البحثي، وكذلك لا أنسى أن أشكر سعادة الدكتور رئيس قسم الكيمياء الدكتور/ سامي بن عبدالعزيز الحسين، على توقيعه على مجموعة من الخطابات التي سهلت لي إتمام إجراء بعض الاختبارات لدى (مدينة الملك عبد العزيز للعلوم و التقنية) و(كلية الهندسة)، وكذلك أوجه شكري للمهندس/ مجدي الحجيلة، من “شركة سيكا” لتعاونه معي وتوفير مجموعة من المواد التي احتجتها في إجراء البحث العلمي.

إهداء

هذا البحث أهديه إلى جميع الأحبة والأصدقاء وعلى رأسهم والدي و والديتي -حفظهم الله- نظير تحفيزهم وتشجيعهم لي، وإلى المشرف على مشروعي البروفيسور/ محمد أحمد هيكل، وأساتذتي الذين لم يقصروا معي طيلة وجودي في الجامعة وعلى العلوم و المفاهيم التي اكتسبتها منهم وأسأل المولى أن يجازيهم على جهودهم المبذولة لرفي لمستوى الطلبة وسمعة الكلية و الجامعة، وكذلك أهدي هذا البحث لزملائي الطلاب الذين شاركوني معهم العلم والمعرفة والتشجيع المتواصل،،،،

Abstract

Geopolymers is the new knowledge-based, multi-functional materials with higher performance, reduced environmental impact. Alkali-activated binders 'geopolymers' are produced from alkaline activated aluminosilicate source. These binders exhibit technological and ecological advantages in comparison with ordinary Portland cement (OPC). Geopolymers are used in the production of high performance building materials. The preparation of eco-friendly geopolymer binders were conducted with FA-SF mixes in different alkalinity conditions (sodium hydroxide and sodium silicates).

The data show that the chemically combined water contents, bulk density and compressive strength of the alkaline activation FA-SF geopolymer pastes gradually increase with the increasing of the alkalinity condition from mix M1 to M2, then decreases with M3. M5 (97% FA + 3% SF +1% SP) is the optimum mix shows the higher values of compressive strength than other mixes. Superplasticizer improves the dispersion degree of activated FA-SF geopolymer, leading to the formation of C-S-H, C-A-H, C-(A)-S-H and N-(A)-S-(H) geopolymer gel with close compact geopolymer matrix.

المخلص العربي

الجيوپوليمر هو من المواد الجديدة القائمة على المعرفة ومتعددة الوظائف، وتعمل على انخفاض الأثر البيئي. ويتم إنتاج الجيوپوليمر من تنشيط القلوي للألومينوسيليكات. يحمل الجيوپوليمر مزايا تكنولوجية وبيئية بالمقارنة بالأسمنت البورتلاندي العادي. وتم تحضير الجيوپوليمر من التنشيط القلوي (هيدروكسيد الصوديوم وسيليكات الصوديوم) لأتربة الفحم وأتربة السليكا (FA-SF) وهما نفايات صناعية تحتوى على الومينوسيليكات الصلبة ضارة للبيئة. وتشير البيانات إلى أن محتوى المياه المتحدة كيميائياً، الكثافة النسبية وقوى التحمل للكسر للمخاليط الجيوپوليمر المكونة FA-SF تزيد تدريجياً مع زيادة كمية التنشيط القلوي للمخاليط M1 إلى M2 ، ثم يتناقص مع M3. ويعتبر المخروط المكون من M5 (97% FA + 3% SF + 1% SP) هو المخروط الأمثل الذى يعطي أعلى قيم لقوى التحمل للكسر من المخاليط الأخرى. ويعمل ملدينات اللزوجة الفائقة (Superplasticizer) على زيادة التنشيط تنشيط لتكوين C-S-H, C-A-H, C-(A)-S-H and N-(A)-S-(H). الجيوپوليمر جل المسئول عن زيادة قوى التحمل للكسر.

Chapter I

Introduction

Fly ash known as pulverized fly ash (PFA) is finely divided waste by-product that precipitated electrostatically from the combustion of pulverized coal in boilers at the thermal power plants. It is the most common artificial pozzolana. The fly ash particles are spherical and have the same fineness as cement so that the silica is readily available for reaction [1].

James [2] discussed the production and utilization of fly ash as well as the differences in material. The overall aim of the utilization of the ash from industrial boilers was to determine its characteristics, to assess their variability with boiler operation and to identify potential component outlets from these residues [3].

Characterization of fly ash from various sources shows that the mineral components of the ashes are similar, even though the chemistry of these ashes may vary widely. Based on scanning electron microscope (SEM) studies, the major mineral components in fly ash are silicates, aluminates, iron oxides, low density silicates (cenospheres) and unburned carbons. The silicates are usually present as spherical particles. They are believed to be the melted products of clays, feldspars, quartz, mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) and other common minerals in coal. The iron oxides are usually existed as spherical magnetite. Low-density silicates are frequently high alkaline silicates, which entrapped gas to yield hollow spherical particles. Unburned carbons are generally chars with irregular shapes and wide range of particle sizes [4].

Based on these characterization results, a separation process has been developed to yield high quality fly ash materials. A schematic flow sheet of the separation process is shown in Fig. 1. This process consists of a gravitational separation process to separate the cenospheres, a

magnetic separation process to separate the iron oxide spheres and a forth flotation process to separate the unburned carbon [5]. The material left after these separations are designated as clean ash. For example, a forth flotation circuit may be needed for ash processing if the material is used only for cement replacement. The experiments have been conducted in cooperation with **Detroit Edison, Consumers Power and HolmanCement** they have shown that clean ash can be used successfully in cement and concrete applications [6].

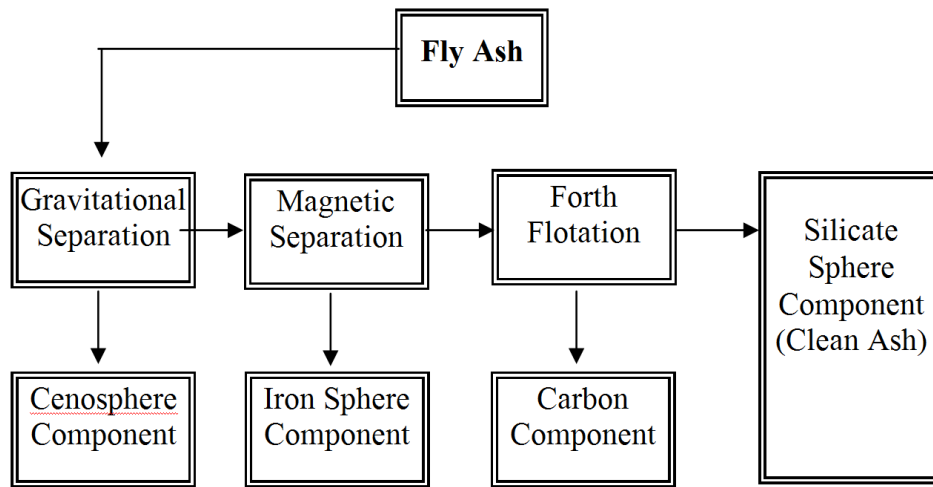


Fig. 1: Fly ash separation processes

The properties of fly ash are variable and depend upon several factors, such as the type and origin of coal (bituminous, sub-bituminous and lignite coals), degree of coal pulverization, flame temperature, oxidation conditions and pretreatment during or prior to burning for SO_x removal, method of collection, storage of fly ash and carbon content [7].

At present, the biggest market for fly ash is a replacement for cement in concrete applications for its pozzolanic properties. ASTM Standard C-618 has set a maximum limit of 6% LOI (primarily carbon) for fly ash in this application. In practice, a 3% LOI is commonly recognized by the market [4].

The efficiency level of the coal burning power plant is also reflected in the quality of fly ash. Thus, better plant efficiency means getting a better fly ash for use in concrete mixes [8]. Cement and concrete are currently the largest user of fly ash. It is known that up to 50%, of the

cement in concrete can be replaced by fly ash without compromising concrete performance. This is due to the pozzolanic and/or cementitious properties of the ash. It is possible to obtain concrete with fly ash replacing the portland cement that provides a significant cost savings and energy extensive material for concrete applications. In addition, the use of good quality ash in concrete offers benefits, such as improved strength and workability (water requirements for consistency), less heat of hydration, increased durability, improved resistance to alkali and sulphate attack as well as permeability reduction. There is a clear economic and environmental advantage for choosing of fly ash as an essential ingredient of structural concrete [4].

Chapter II

LITERATURE REVIEW

1. Fly ash (FA):

The activation of fly ashes is a process very different from the OPC cement hydration, but resembling the chemical principles involved in the formation of several types of zeolites with an alkaline aluminosilicate as the main reaction product. This reaction product involves the tetrahedral coordination of silica and aluminum in polymeric chains in which the Al^{3+} replace the Si^{4+} , with the negative electric charge being compensated by alkali cations. During the alkali attack of the aluminosilicate material, an initial nucleation phase takes place where the aluminosilicate species are dissolved. When the nuclei reach a critical size, they start to crystallize, where this process is a very slow so it may be complete after a long time [9]. When fly ashes are treated with activated alkaline solution, a dissolution process of the Al and Si takes place [10]. The alkaline solution attack opens the spheres exposing small spheres on the inside which will be also dissolved until the spheres, became almost dissolved with the formation of reaction products inside and outside the sphere Fig. 2. The activation of fly ashes is a process that may be considered as a zeolitization in which the last phase does not occur, since the experimental conditions lead to very fast dissolution and condensation reactions but a lower one when the hardening take place [11].The reaction products are generated both inside and outside the shell of sphere until the ash particle is completely or almost completely consumed (Fig.2,a-c). At the same time, precipitations of reaction products occur as the alkaline solution penetrates the larger sphere and fill up the interior space with reaction products, forming a dense matrix (Fig.2,b).

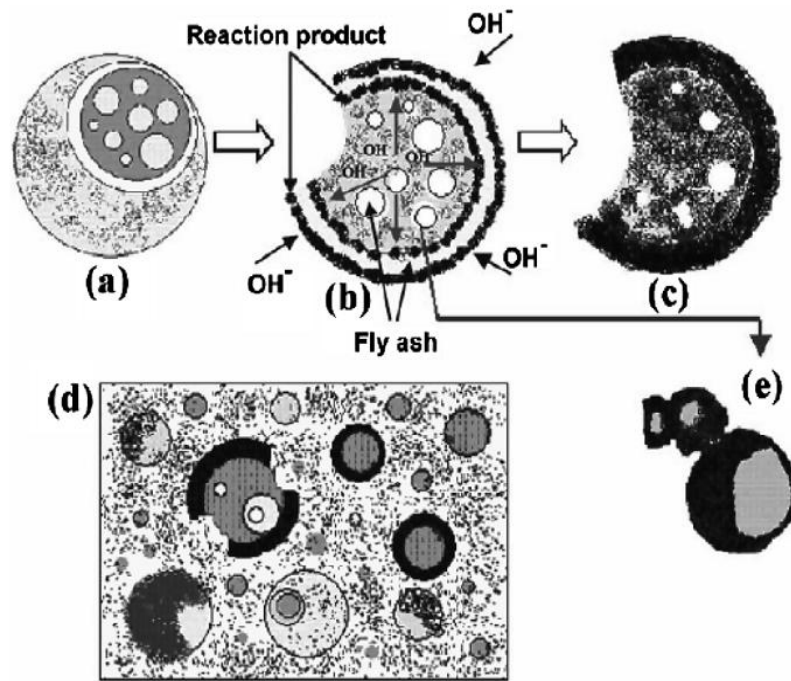


Fig. 2: Descriptive model of the alkali activation of fly ash [10].

Due to the massive precipitation of reaction products, some portions of smaller particles are covered with the products providing crust which prevents the contact with alkaline solution (Fig. 2,e), resulting an unreacted FA particle. As a consequence, several morphologies may co-exist in a single paste: unreacted particles, particles attacked by the alkaline solution but which maintain their spherical shape, reaction product and so on (Fig. 2,d)

Rashad [12] developed new binders, as an alternative to OPC by alkaline activation; this trend is a current research interest. Alkali-activated FA (AAFA) binder is obtained by a manufacturing process less energy-intensive than OPC and involves lower greenhouse gasses emission. AAFA resists aggressive acids, sulfate attacks, aggregate alkali reaction elevated temperatures.

Ryu et al., [13] developed cementless alkali-activated concrete using 100% FA as a binder. The results showed that chemical changes of the alkaline activators had a significant effect on the early strength. Analysis of the structure with SEM and EDS verify the compactness

of the structure of the alkali-activated mortar. Study on the engineering properties of geopolymer concrete made from alkali activated FA has been presented by **Joseph [14]**. The curing temperature was varied from ambient (30-120 °C). Major conclusions could be summarized as follows:

1. The compressive strength of geopolymer concrete increases in curing temperature up to 100°C, then decreases.
2. Modulus of elasticity and Poisson's ratio of geopolymer concrete higher than that of the corresponding OPC.
3. The tensile strength of geopolymer concrete increases.

2. Alkaline Activators:

Fly ash, slag and other aluminosilicate materials activated using different alkaliactivators to form hydrated binding material. Typically, caustic alkalis or alkaline salts are used as alkaline activators.

Of all these activators, NaOH, Na₂CO₃, Na₂O·nSiO₂ and Na₂SO₄ are the most widely available and economical chemicals. Some potassium compounds have been used. However, their potential applications will be very limited due to their availability and costs [15]. On the other hand, the properties of sodium and potassium compounds are very similar.

Chemical compounds composing of an alkali metal cation and the hydroxide anion (OH⁻) are called alkali hydroxides. They are the most widely used as activating agents. Sodium and potassium hydroxides are the most commonly used in the production of alkali activated binders. NaOH is reasonably cheap when compared to KOH and largely available making it an obvious pick for activation of aluminosilicate materials. The use of high concentrations of NaOH or KOH as the activating agent has been reported to the formation of zeolitic structures after an extensive period of moist curing or a brief period of heat curing[16]. The dilution of NaOH releases a large amount of heat, thus requiring special precautions to be taken after mixing. The carbonation and

leaching of the reaction products activated using the alkali hydroxides is a concern due to the high alkalinity in the system.

Chapter III

MATERIALS AND EXPERIMENTAL TECHNIQUE

3.1. Materials

3.1.1. Fly Ash (FA):

Class F fly ash (FA) was provided from Sika Company, Riyadh, KSA, for Construction Chemicals. The chemical oxide composition is given in Table 1. XRD patterns and SEM images are shown in Figs.3 and 4. The Blaine surface area of class F FA was 3570 cm²/g. The XRD pattern of FA is shown in Figure 3. It revealed that FA has a small vitreous phase content (halo registered between 2θ =20° and 2θ=35 °) and therefore the major crystalline phases are quartz (SiO₂) and mullite (3Al₂O₃.2SiO₂). The vitreous phase contains the Fe₂O₃.

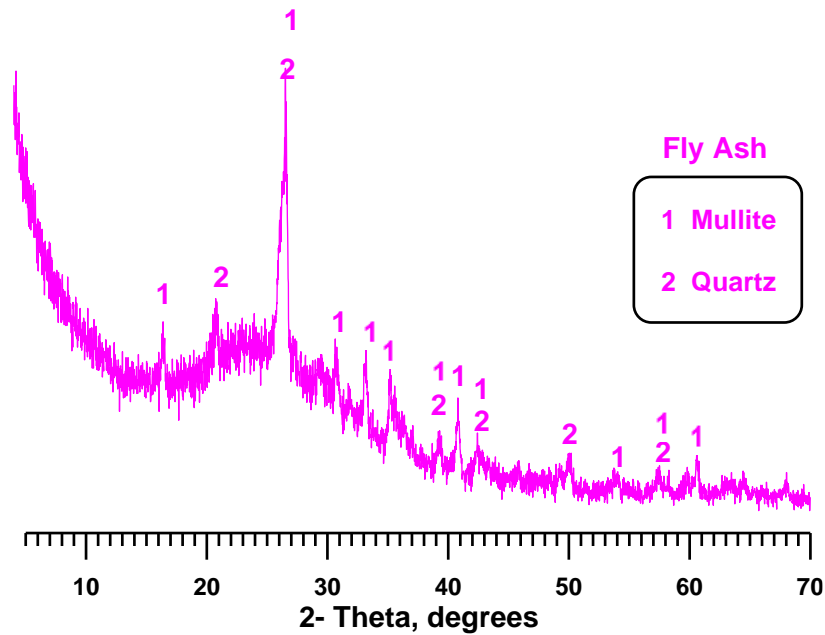


Fig.3: XRD pattern of Fly ash (FA)

Scanning electron microscopy (SEM) was used to investigate the morphology of FA as shown in Fig. 4. The micrograph reveals the presence of spherical ash particles with much brighter surface which are identified as cenospheres ranging in size from 2 to 20 μm . Moreover, the micrograph shows the presence of agglomeration of fine particles and sticking of finer particles to the coarser ones.

Table 1: Chemical composition and phase composition OPC

Oxides, %	FA	SF
SiO ₂	63.10	94.39
Al ₂ O ₃	26.54	0.48
Fe ₂ O ₃	5.40	2.80
CaO	2.33	0.90
MgO	0.00	0.00
SO ₃	0.09	0.00
K ₂ O	0.85	0.64
Na ₂ O	0.52	0.00
L.O.I	2.40	0.50
Total	99.97	99.71

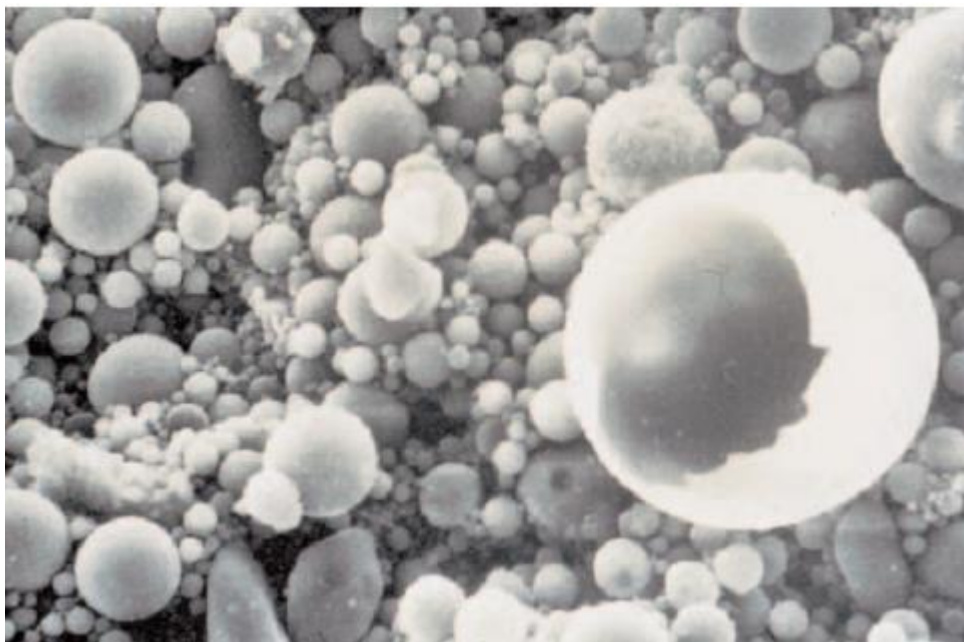


Fig. 4:SEM of FA

3.1.2. Condensed silica fume (SF):

Condensed silica fume (SF) is a by-product of silicon or ferrosilicon alloys industries. It is obtained from Sika Company, Riyadh, KSA. Its particles have an average diameter of $\cong 0.1\mu\text{m}$. Its oxide chemical composition is given in Table 1. SEM of SF was shown in Fig. 5. Fig.5 shows SEM micrograph of. SF particles are densely packed with sphere like particles. The surface area of the agglomerate is $20.90\text{ m}^2/\text{g}$.

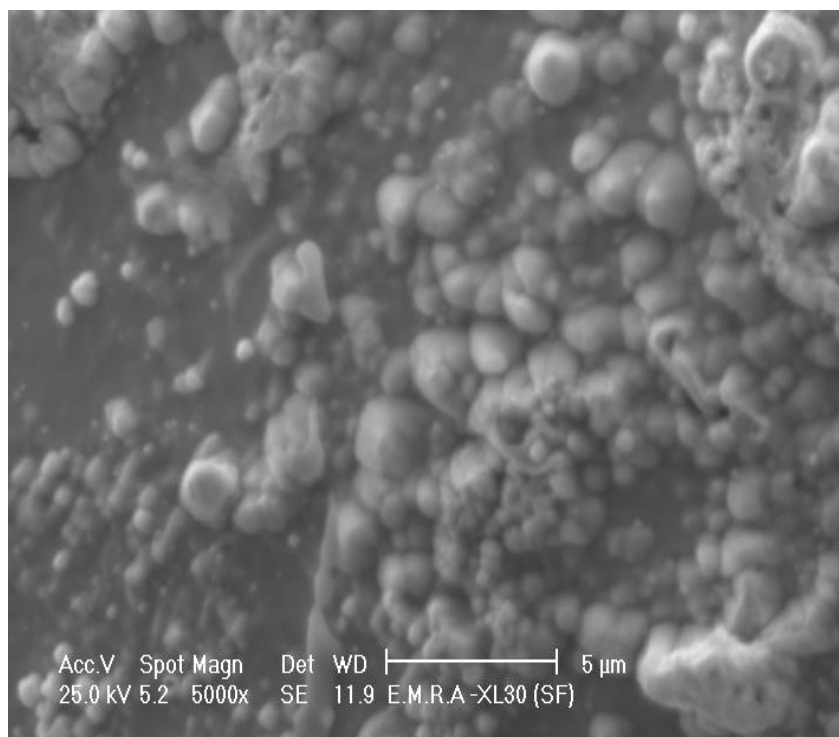


Fig. 5:SEM of Silica fume (SF)

3.1.3. Sodium hydroxide pellets (SH):

Sodium hydroxide (SH) pellets analytical grade, 98.01% purity was provided from PanreacChemical Company, Barcelona, Spain.

3.1.4. Sodium silicate liquid (SSL):

Sodium silicate liquid consists of 30.7% SiO₂, 10.3% Na₂O and 59% H₂O, with silica modulus SiO₂/Na₂O equal 2.98 and density 1,380 g/cm³ was used. Sodium silicate liquid was provided from AppliChem Chemical Company, GmbH, Darmstadt, Germany.

3.2. Experimental Techniques:

3.2.1. Preparation of dry mixes:

Five blends were prepared by mixing each sample as presented in Table 2. The dry constituents of each mix were mechanically mixed for one hour in a porcelain ball mill using three balls to attain complete homogeneity. The mixes were kept in airtight container.

Table (2): Mix composition, designations and water/solid (W/S) ratio

Mix no.	FA, %	SF, %	SH, %	SSL, %	SP, %	W/S ratio
M1	100	0.0	2	3	0.0	0.35
M2	100	0.0	4	3	0.0	0.35
M3	100	0.0	6	3	0.0	0.35
M4	97	3	4	3	0.0	0.35
M5	97	3	4	3	1.0	0.35

3.2.2. Mixing:

The required amount of sodium hydroxide (SH) pellets and sodium silicate liquid were mixed in W/S ratio = 0.35. The temperature of the mixture was initially quite high then left to reach room temperature before proceeding. Alkali activated blend was obtained by the addition of both FA and SF to alkali-activation solution (SH:SSL) followed by mechanical mixing to ensure homogeneity.

The paste was placed in the mould and pressed into the corners along the surface of the mould until homogeneous specimen was obtained. After the top layer was compacted, the surface of the paste was smoothed by the aid of thin edged trowel.

Freshly prepared cement paste was placed in 2 cm cylinder moulds into two approximately equal layers. Each layer was compacted and pressed until homogenous specimen was obtained then manually vibrated for a few minutes to remove any air bubbles to give a better compaction of the paste.

3.3. Curing:

Immediately after moulding, the specimens were cured in a humidifier (100% RH) for first 72 hours at 40°C. At the end of the moist curing period, the cubes were moulded in 2 cm cylinder moulds until the required time of testing 14 and 21 days of curing. The cylindrical specimens prepared shown in Figure 6.



Fig.6: Specimens prepared

3.4. Methods of Investigation:

3.4.1. Bulk density measurements:

The bulk density was carried out before the specimens subjected to compressive strength determination. Bulk density was determined through the weighing out the samples of hardened pastes (suspended in water) and in air (saturated surface dry). Each measurement was conducted on three similar cubes of the same age as shown in Fig.7.

The following equations were used for calculating the bulk density:

$$\text{Bulk density (dp)} = \frac{\text{saturated weight}}{\text{Volume of sample}} \text{ g/cm}^3$$

$$\text{Volume of sample} = \frac{\text{saturated weight} - \text{suspended weight}}{\text{Density of water}}$$

$$\text{Bulk density (dp)} = \frac{\text{saturated weight}}{\text{Saturated weight} - \text{suspended weight}} \text{ g/cm}^3$$

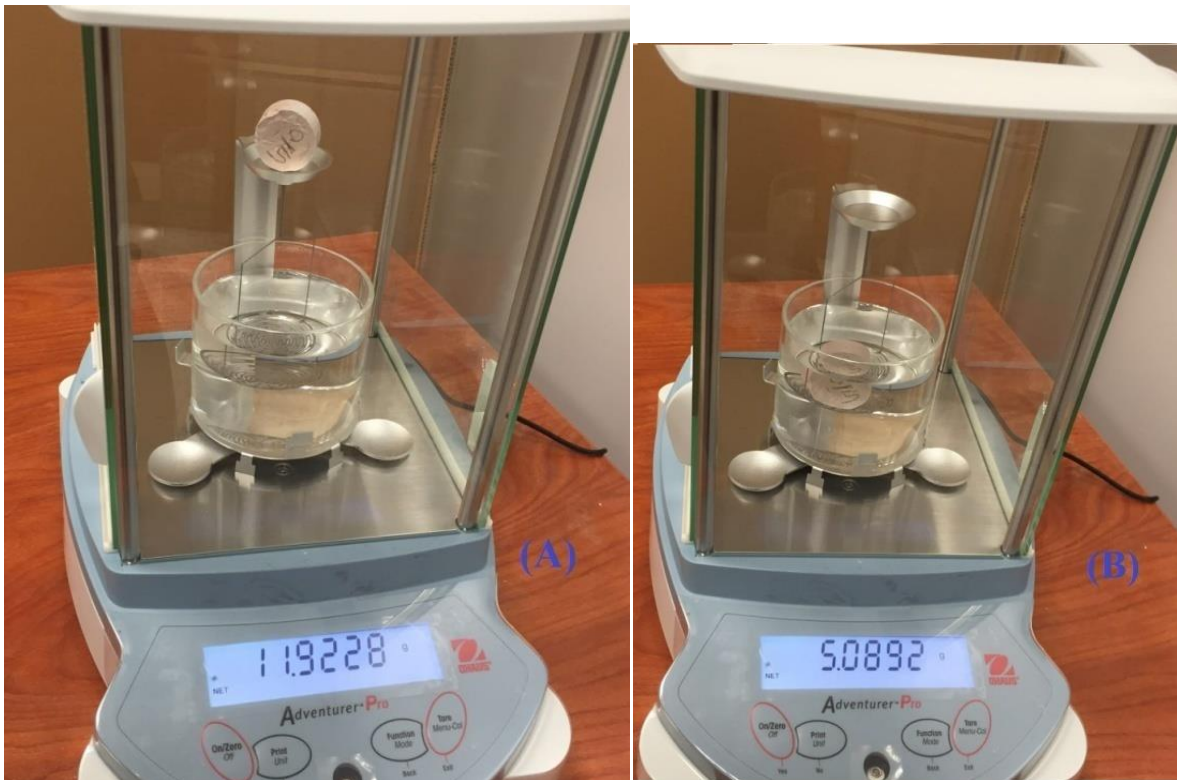


Fig.7: Determination of Bulk density, a) saturated surface dry, b) suspended in water

3.4.2. Determination of compressive strength

The compressive strength and weight loss were determined. Three cubes were used for the compressive strength determination of cement paste (ASTM Designation: C-150, 2007). The compressive strength was done on a compression machine with maximum capacity of 600 KN

force as shown in Fig. 8. The compressive strength was carried out on three samples as described by ASTM Specifications C-150, 2007.



Fig. 8: The compression testing machine.

3.4.3. Stopping of the hydration:

The stopping of hydration was performed on the crushed pastes after the compressive strength determination. The combined water W_n , is used as an indication for the degree of

hydration of the hydrated samples after a given period. W_n , is determined after the free water is removed. The stopping solution was prepared (1:1 by volume) of methyl alcohol and acetone. A representative sample of about 10 g was taken from the broken cubes and ground in alumina mortar under the surface of 75 ml from the stopping solution, then filtered through sintered glass funnel (G4) and washed three times with the stopping solution. Finally, the residue was washed with 50 ml of fresh diethyl ether then dried at 70°C in a drying for one hour, and kept in airtight containers.

3.4.4. Determination of chemically combined water contents:

The chemically combined water contents (W_n), was determined in a similar manner to (W_t). It is the weight loss of the dried paste on ignition at 800°C. Two representative samples of the dried specimen about 1g, each were exactly weighed in a porcelain crucibles and ignited for one hour at 800°C in a muffle, cooled in a desiccator, then weighed. The combined water content was calculated as W_n using the following equation:

$$W_n = \left[\frac{(W_1 - W_2)}{W_2} \right] \times 100$$

where:

W_n is the non-evaporatable water,

W_1 is the weight of sample before ignition,

W_2 is the ignited weight of specimen,

3.5. Phase identification:

The phase composition of the formed hydrates was investigated by means of differential scanning calorimetry (DSC), X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FT-IR).

3.5.1. X-Ray Diffraction (XRD):

XRD technique was carried out on some selected hardened cement pastes to show the hydration products. The XRD technique was carried out using Philips, Pw 1390 channel control, (Copper target-Nickel filter), 40 kV-25 mA. The sample was finely ground to pass a 200-mesh sieve. The identification of all samples was confirmed by computer-aided search of the PDF database obtained from the Joint Committee on Powder Diffraction Standards-International Center for Diffraction Data (**JCPDSICDD**), **2001**.

3.5.2. FT-IR Spectroscopy

For IR spectroscopic investigation, the samples were prepared using alkali halide KBr pressed disk technique. The IR analysis was recorded from KBr disks using Genesis FT-IR spectrometer in the range $400\text{--}4000\text{ cm}^{-1}$ after 256 scans at 2 cm^{-1} resolution.

Chapter IV

RESULTS AND DISCUSSION

4.1. Chemically combined water contents:

The chemically combined water contents of alkali activated FA-SF cured at 14 and 21 days are represented in Fig. 9. Chemically combined water contents (W_n) can be used as a measure of the amount of hydration products and the degree of hydration products. Chemically combined water contents of the alkaline activation FA-SF geopolymer pastes gradually increase from 14 days up to 21 days. The increasing of the alkalinity condition from mix M1, M2 and M3, the hydration can promote the alkali activated FA geopolymer pastes, as a result of this the degree of hydrolytic destruction of the alkali activated FA increases. This is due to the continuous hydration and accumulation of geopolymer hydrated products, and then precipitated in the available open pores. The increasing of the alkalinity condition from mix M1, M2 and M3 chemically combined water contents increase with curing time as shown in Fig. 9. Fig. 9 indicates that the influence of increasing alkalinity of hydration condition chemically combined water contents increase from mixes M1, M2 and M3 FA geopolymer pastes. Mix M2 shows higher value of chemically combined water contents than those of M1 and M3.

Mix M4 (97% FA + 3% SF) and M5 (97% FA + 3% SF + 1% SP) are alkali activated FA-SF geopolymer pastes. The substitution of SF with 3 mass % SF in Mix M4 increases the combined water contents at all ages of hydration. This is mainly due to the reactivity of SF as comparison with FA. Alkali activation of FA-SF form a C-(A)-S-H and N-A-S-(H) geopolymer gel, with the formation of a higher amount of chemically combined water contents. From the chemical point of view, SF is highly reactive pozzolana which reacts with formed CH producing calcium silicate hydrates (C-S-H), this is also due to the high surface area and glass content of SF in comparison with FA.

M5 is the optimum mix shows the higher values of chemically combined water contents than other mixes. Superplasticizer improves the hydration kinetics of M5 (97% FA + 3% SF +1% SP) as shown in Fig. 8.

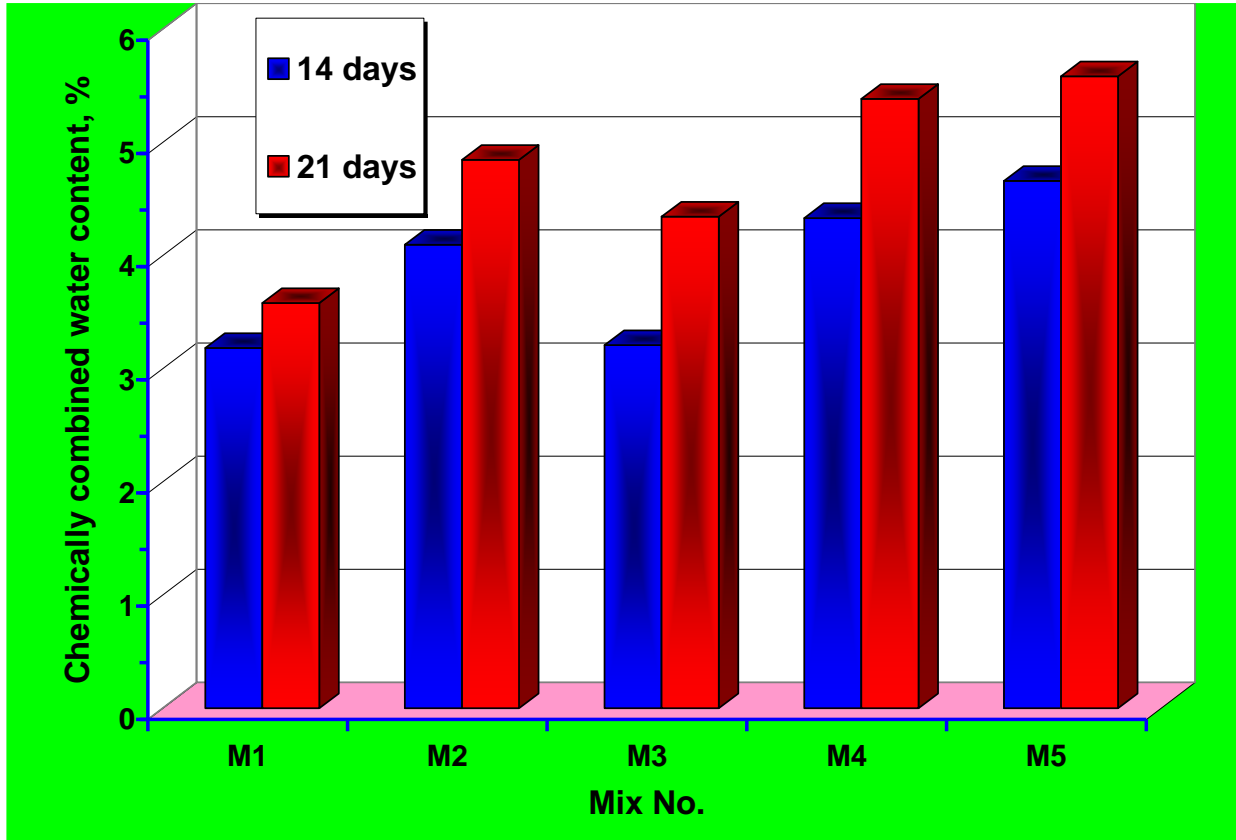


Fig. 9: Chemically combined water contents of alkaline activationFA-SF cured up to 21 days.

4.2. Bulk density

The bulk density of hardened of the alkaline activation FA-SF geopolymer pastes cured up to 21 days is illustrated in Fig. 10. The values of bulk density increase with curing time from 14 days up to 21 days. The bulk density of alkali activated FA-SF geopolymer pastes increases with curing time, this is attributed to the continuous degree of hydrolytic destruction of the alkali activated FA-SF to formation of C-S-H, C-A-H, C-(A)-S-H and N-(A)-S-(H) geopolymer gel hydrated products. These hydrated products are deposited in the open pores that increase the bulk density of the alkaline activation FA-SF geopolymer pastes.

The alkali activator enhances the production of higher concentration of $[\text{SiO}_4]^{4-}$ which increases the rate of hydration and formation of more geopolymer gel hydrated products. Mix M2 shows higher values of bulk density of hardened alkaline activation FA-SF geopolymer pastes than those of M1, M3, M4 and M5. The specific gravity of SF is lower than FA this is the reason of the decrease of values of bulk density of hardened alkaline activation FA-SF geopolymer pastes containing SF.

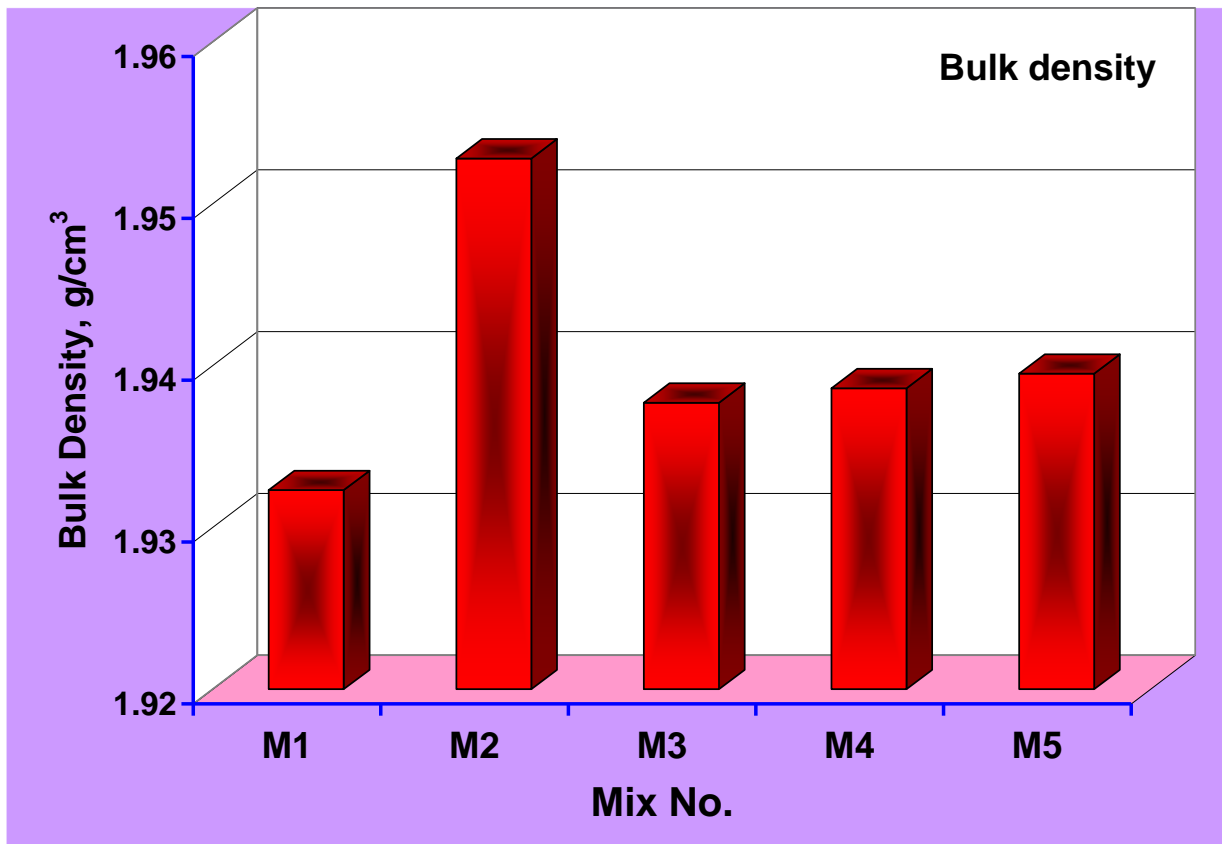


Figure 10: Bulk density of alkaline activation FA-SF cured up to 21 days.

4.3. Compressive strength

The compressive strength of hardened the alkaline activation FA-SF geopolymer pastes cured up to 21 days. The values of compressive strength are graphically represented in Figure11. The increasing of the alkalinity condition the degree of hydrolytic destruction of the alkali activated FA increases from mix M1, M2 and M3. Mix M2 shows the higher values of

compressive strength at all curing ages of hydration than those of mixes M1 and M3. As the amount of alkali activator increases, the compressive strength enhances.

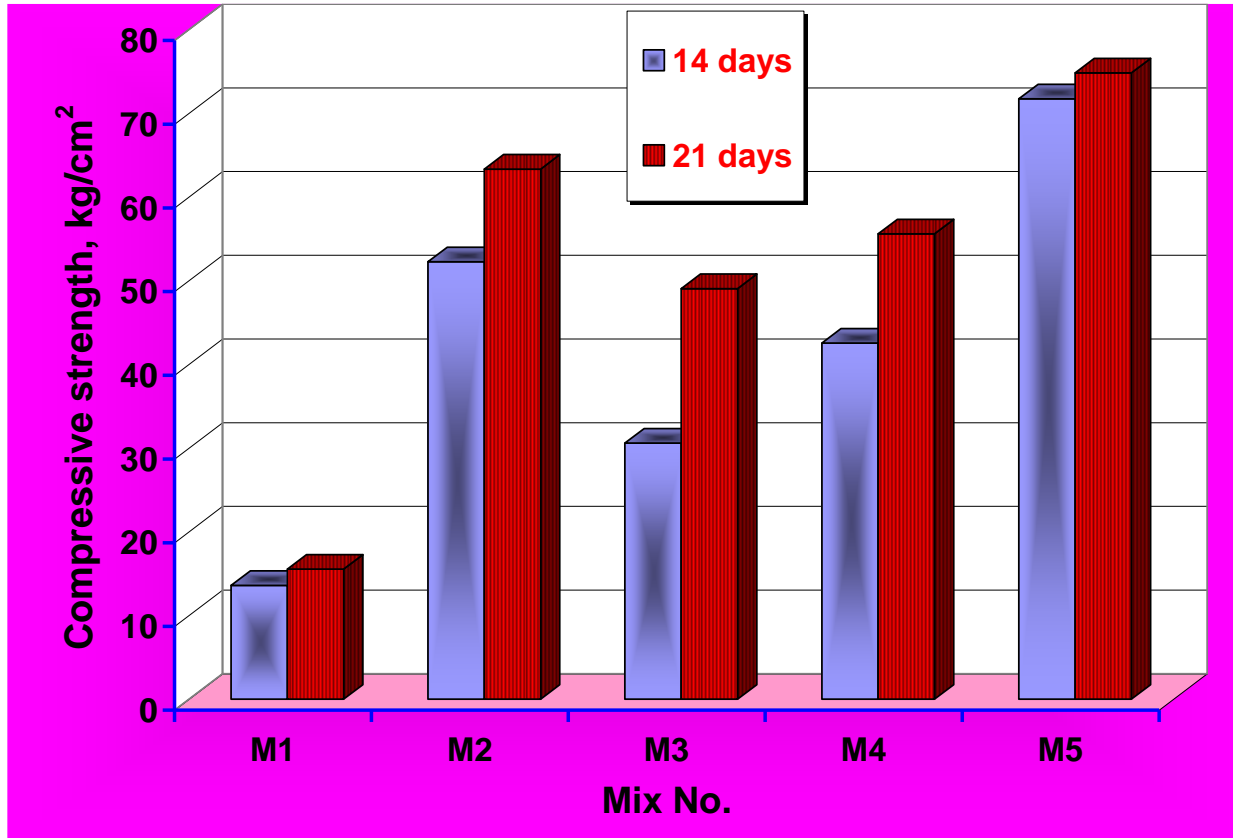


Fig.11: Compressive strength of alkaline activation FA-SF cured up to 21 days.

Fig. 11 represented that the values of the compressive strength increase with the alkaline activation, due to the higher rate of hydration and formation of geopolymer gel. The compressive strength of hardened activated FA-SF geopolymer pastes shows an increase in the presence of SP, which accelerate the activation process. Superplasticizer improves the dispersion of activated FA-SF geopolymer, leading to the formation of more efficient hydration products and higher degree of compaction to form C-S-H, C-A-H, C-(A)-S-H and N-(A)-S-(H) close compact geopolymer matrix. The improvement on compressive strength is related to the modifications of microstructure of alkali activated FA-SF geopolymer matrix as shown in Mix M5. C-S-H, C-A-H, C-(A)-S-H and N-(A)-S-(H) geopolymer matrix are responsible for the strength increase in matrices containing activated FA-SF geopolymer.

4.4. FT-IR Spectroscopy

Fig. 12 shows FTIR spectroscopy of alkali activated mix M2. Fig. 12 shows a strong stretching vibration frequency at 1424 due to the CO_3^{2-} group. The stretching vibration frequency band at 3347 cm^{-1} belongs to OH^- group of H-O-H . The band located at 1015 cm^{-1} corresponds to stretching vibration frequency of silicate (Si-O).

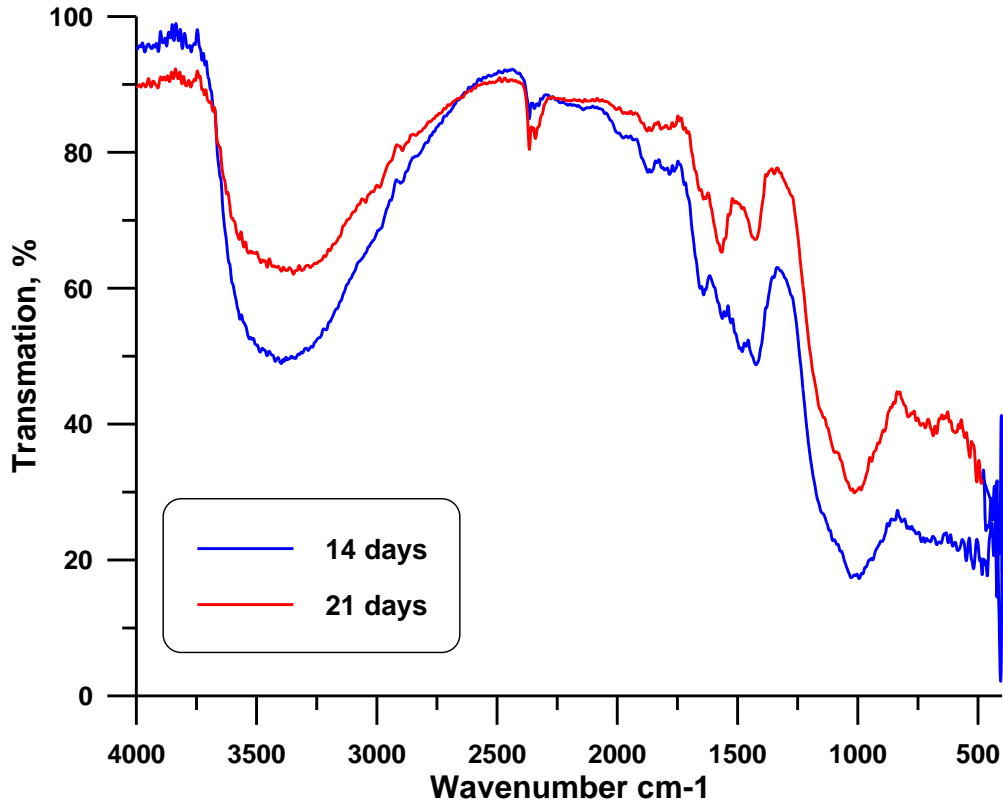


Fig. 12: FTIR of mix M2 of alkaline activation FA cured up to 14 and 21days.

5. Conclusions:

The main conclusions could be derived from this investigation are summarized as follows:

1. Chemically combined water contents of the alkaline activation FA-SF geopolymer pastes gradually increase from 14 days up to 21 days. The increasing of the alkalinity condition from mix M1 to M2, then decreases with M3. Mix M2 shows higher value of chemically combined water contents than those of M1 and M3.
2. The substitution of SF with 3 mass % SF in Mix M4 increases the combined water contents at all ages of hydration. M5 (97% FA + 3% SF +1% SP) is the optimum mix shows the higher values of chemically combined water contents than other mixes.
3. The bulk density of alkali activated FA-SF geopolymer pastes increases with curing time, this is attributed to the formation of C-S-H, C-A-H, C-(A)-S-H and N-(A)-S-(H) geopolymer gel hydrated products. Mix M2 shows higher values of bulk density of hardened alkaline activation FA-SF geopolymer pastes than those of M1, M3, M4 and M5.
4. Mix M2 shows the higher values of compressive strength than those of mixes M1 and M3.
5. Superplasticizer improves the dispersion degree of activated FA-SF geopolymer, leading to the formation of more efficient hydration products and close compact geopolymer matrix. The improvement on compressive strength is related to the modifications of microstructure of alkali activated FA-SF geopolymer matrix of Mix M5.

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