Processing and Characterization of Fly Ash-Based Geopolymer Bricks

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Utilization of fly ash as a raw material for geopolymer brick production seems to be a logical solution that allows for the conservation of natural resources, abates further pollution and preserves the environment. Fly ash-based geopolymer have been studied by several researchers worldwide for several decades due to their excellent mechanical properties. This study has been conducted to produce fly ash-based geopolymer bricks by means of pressure forming without firing procedure and low energy consumptions. The experiments were conducted on fly ash-based geopolymer bricks by varying the ratio of fly ash-to-sand (1:2 - 1:5, by mass of ratio), curing time (1 - 24 h) and curing temperature (room temperature - 80°C). Compressive strength up to 20.3 MPa was obtained by curing at 70 °C for a period of 24 hours at 60 days of ageing. The density of geopolymer bricks ranged between 1800 Kg/m^3 to 1950 Kg/m^3. The microstructure properties of fly ash-based geopolymer bricks were investigated by using XRD and SEM analysis.

Keywords: fly ash, bricks, geopolymer, mechanical properties, microstructure

Every year, millions of tons of fly ash are generated from thermal power stations as well as the petrochemical industry all over the world. The abundant availability of fly ash is creating problems in disposal operations and tremendous environmental concerns. For this reason, the utilization of this waste material will be beneficial when treated as valuable resources in the production of good quality building materials. Class F fly ash has considered as pozzolanic materials and can be activated by high alkaline solutions to act as a binder through chemical polymerization reactions [1]. This reaction transform aluminosilicate materials (fly ash) into aluminosilicate polymers known as geopolymers. Geopolymer is one of new material and have been investigated for some decades by several researchers throughout the world. Geopolymer was first used by Prof Joseph Davidovits in St. Quentin, France, in the 1970s [2].

The geopolymer-based material involves a chemical reaction known as geopolymerization process yields polymeric Si – O – Al bonds. The geopolymerization process involves a substantially fast chemical reaction under alkaline solutions on silica-alumina materials that results in a three-dimensional polymeric chain and ring structure [3]. The exact mechanism of geopolymerization is not yet quite understood and clear, although it is thought to be dependent on the source material and alkaline activator used. Most researchers agree that the majority of the proposed mechanisms indicates an initial phase of silica dissolution, followed by the phases of transportation, and polycondensation. However, those reactions occur almost simultaneously, preventing their analysis in an individual mode [4].

Brick technologists are gradually finding applications in using of fly ash as a raw material for producing greenest brick which is free from environmental pollution. Uses of fly ash in making bricks have many advantages over conventional clay bricks as they do not emit any pollutant and greenhouse gas during and after manufacturing, requires much less energy consumption, and it costs about 20% less than manufacturing clay bricks [5]. Comprehensive utilization of fly ash in the production of geopolymer brick, which is a kind of green material contributes to social benefits and economic benefits advance together, as well as to the development of new brick with better performances.

This research presents a design and performance evaluation of fly ash-based geopolymer brick. It proposes new technology of brick production in Malaysia and widening the possibilities to recycle waste (fly ash) to useful products, especially building materials which can contribute to the environmental and economic benefits. The ultimate goal of this research is to measure, evaluate as well as to compare the performance of fly ash-based geopolymer bricks with common bricks in Malaysia based on the result obtained through this research.

Experimental part

Materials

In the present experimental work, low calcium, class F [6] dry fly ash obtained from the Manjung Power Station, Lumut, Perak, Malaysia was used as the raw material. The

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chemical compositions of the fly ash as determined by X-Ray Fluorescence (XRF) analysis are given in table 1. The natural Malaysia sand was used as fine aggregate to combine with fly ash and alkaline activator. The natural sand was selected based on the standard specification for aggregate for masonry mortar stated in ASTM C 144-11. Sieve analysis for natural sand used was done to confirm the fine aggregate has the range size below than 4.75 mm by passing through a 4.75 mm sieve.

The alkaline liquid used was a combination of sodium silicate solution and sodium hydroxide solution. The Sodium silicate (Na_2SiO_3) solution was supplied by South Pacific Chemicals Industries Sdn. Bhd. (SPCI) Malaysia. The chemical composition of the Na_2SiO_3 solution was SiO_2 = 30.1%, Na_2O = 9.4% and water = 60.5% by mass with a SiO_2/Na_2O modulus of 3.2, specific gravity at 20°C = 1.4g/cc and viscosity at 20°C = 400cP. The sodium hydroxide (NaOH) solution was prepared by dissolving the NaOH powder with 99% purity in distilled water. The NaOH solution was prepared with a 12 M concentration [7, 8] and cooled down to room temperature. The alkaline liquid was prepared at least 24 h prior to use by mixing a Na_2SiO_3 solution.

Table 1
CHEMICAL COMPOSITION OF FLY ASH

Chemical composition	Percentage (%)
SiO_2	52.11
Al_2O_3	23.59
Fe_2O_3	7.39
TiO_2	0.88
CaO	2.61
MgO	0.78
Na_2O	0.42
K_2O	0.80
P_2O_5	1.31
SO_3	0.49

Mixture Proportion

The mixture proportions of fly ash-based geopolymer bricks were determined using the trial and error method and also based on the previous work done by several researchers [7, 9] in our group of CEGeoGTech team. The ratio of fly ash to sand was found by trial mixing and varied from 1:2 to 1:5 ratio, by mass. Then the ratio was fixed in 1:3 ratio, by mass for most of the mixtures based on their good workability and highest strength development with lower water absorption. The ratio of sodium silicate solution to sodium hydroxide solution, was fixed at 2.5 ratio, by mass and the ratio of solid to liquid (fly ash to alkaline activator) was fixed at 2.0 ratio, by mass, for all the mixtures based on studying done by Mustafa et al. [9] which give highest compressive strength for geopolymer-based materials.

Mixing Process

The materials (fly ash, sand, sodium hydroxide solution and sodium silicate) were weighed according to the given ratio. The materials were put into the mixer followed the sequence. Fly ash and sand is mixed first for 5 min before the alkaline activator is added to the mixes. After the alkaline activator was added, the mixture was mixed for 10 min or until integrated all mixtures. Then the mixes were weighed approximately 2.5 kg for each sample of brick, then poured into the mould and compressed with the pressure of 10 MPa to get the compact sample bricks. The timing for mixtures achieved their homogeneity must not be greater than 20 min and not less than 10 min because it will affect the geopolymerization process and the workability of the samples. The mould has been greased to avoid the samples stick to the mould during removing the samples. Table 2 shows the size and dimension of bricks according to the British Standard, BS 3921:1985 [10].

Curing Process and Testing

The bricks were cured in the oven after removing the samples from the mold at 70°C for 24 h. Then, the bricks were taken out and air dried at room temperature until they are ready to be tested.

Testing program on fly ash-based geopolymer bricks consists of mechanical test and microstructure test. The compressive strength of fly ash-based geopolymer bricks were tested by imposing the bricks to compression load until failure. The compressive strength test for geopolymer bricks were carried out according to ASTM C67-11 [11] by using Hydraulic Compression Testing Machine VU 2000 at the rate of load speed 0.6 $N/mm^2/s$. The effects of compressive strength of geopolymer bricks were only tested on their stretcher face and tested at the ages of 1, 3, 7, 28 and 60 days. Five samples of bricks are tested at each age to evaluate the average strength. The maximum load was recorded and the strength calculated by dividing the maximum load with the area of the face subject to loading (length x height). This area used in the calculation was based on the smaller of the two opposite faces. The strength was recorded in N/mm^2 to the nearest 0.1 N/mm^2. The equation for obtaining the compressive strength is as follows:

$$\sigma_c = F / A$$

where:

σ_c = Compressive strength;
F = Applied load (N);
A = Area of stretcher face (mm^2).

The water absorption test was carried out according to ASTM C140 [12] at the ages of 1,3,7, 28 and 60 days. Three samples have been tested for each age for their absorption test. The samples were immersed in water at room temperature for 24 h. After 24 h , the samples were removed from the water, and then visible surface water was removed with a damp cloth and allowed to drain for 1 minute. The saturated weight (W_s) was recorded. Next, the samples were dried in oven at 100°C for not less than 24 h and until two successive weights at intervals of 2 h show an increment of loss not greater than 0.2% of the last previously determined weight of the samples. The oven-dry weight was recorded as W_d. Water absorption was determined by the equation below:

$$\text{Absorption (\%)} = (Ws-Wd)/Wd \times 100$$

where:

W_s = Saturated weight of samples (g);
W_d = oven-dry weight of samples (g).

The dimensions test of the geopolymer bricks was measured from the respective length, width and height of overall dimension of 24 bricks and individual brick dimension. Test procedure was conducted on 24 bricks which were selected randomly from 80 bricks prepared for testing program in accordance to BS 3921: 1985. The

Coordinating Size (mm)			Work Size (mm)		
Length	Width	Height	Length	Width	Height
225	112.5	75	215	102.5	65

Table 2
SIZE OF BRICKS

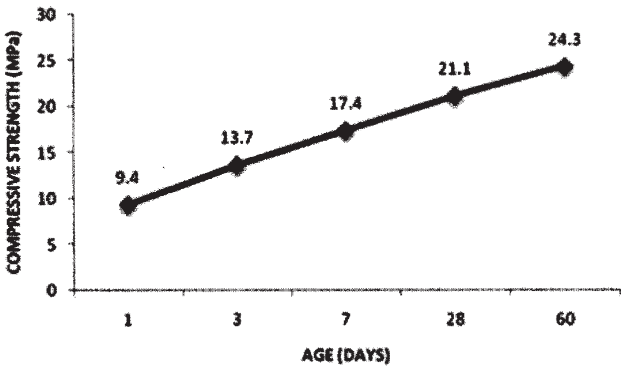


Fig. 1. Compressive strength of different aging time

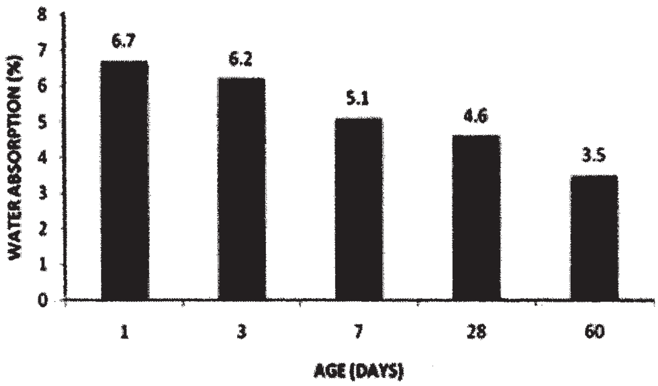


Fig. 2. Water absorption of different aging time

overall dimension (length, width or height) for 24 bricks was measured to the nearest millimeter using a measuring tape.

Density analysis was determined on air dry. Bricks samples were cured in the laboratory at room temperature for 1, 3, 7, 28 and 60 days after oven curing 24 hours. The density of the geopolymer bricks at different ages were measured by dividing the weight of the bricks with the volume of the bricks. The weight and the volume of the bricks were determined by calculating the average value of five bricks to be tested for every age.

Scanning Electron Microscopy (SEM) analysis in this study was performed using a JEOL JSM-6460LA Scanning Electron Microscope (SEM). The samples to be characterized was placed onto an aluminum sample stub via double sided carbon tape as the adhesive and gold coated before analysis.

X-Ray Diffraction (XRD) measurements were performed for phase analysis of the starting materials and to investigate the degree of crystallinity of the resulting geopolymer bricks. The sampling for this analysis was made by grinding the sample with a pestle and mortar to obtain very fine powder before the sample spread on the surface of the sample holder using pieces of glass to minimize the effect of preferred orientation on the XRD pattern. Random powder samples of fly ash were prepared by lightly pressing powder samples into aluminium holders. XRD powder diffractograms of geopolymer specimens are collected on a XRD-6000 Shimadzu X-Ray Diffractometer with CuKα radiation generated at 30 mA and 40 kV. Step scans are performed from 10 - 70° 2θ at 0.02° 2θ steps, integrated at a rate of 1.2 s per step.

Results and discussions

Compressive Strength

Figure 1 shows the results on the compression test. As seen in figure 1, the compressive strength was increased as the age of geopolymer bricks increased up to 60 days. The least compressive strength is 9.4 MPa and this satisfies the requirements of BS 6073 for compressive strength ($\geq$ 7 MPa). The highes strength of geopolymer bricks was 20.3 MPa in the samples cured at 70°C for 24 h on 60 days of ageing. This indicated that long curing period does affect compressive strength development of geopolymer bricks. It can be attributed to the continuous formation of aluminium silicate hydrate/calcium silicate hydrate gel. The minimum one day strength attained is sufficient for handling and transportation of geo-polymer bricks and the minimum seven days strength attained is sufficient for early masonry work [13]. This result also shows another advantage of geopolymerc materials, represented by strength-gaining at long-term ages in addition to the advantage of the high early strength reported as early as three and seven days [14].

Table 3 below shows the comparison between fly ash-based geopolymer bricks with those of other bricks available in the market. Two types of bricks comprising of the common clay bricks and cement bricks used for construction in Malaysia were chosen randomly from the factory output. Five samples were tested for compressive strength, three samples were tested for water absorption and density analysis prior for comparison with the fly ash-based geopolymer bricks.

The results obtained shows that the geopolymer bricks performed better characteristics and properties due to high compressive strength (5 to 25 MPa), low water absorption (3.5 to 7%) and the range of density between 1800 to 1950 kg/m^3 is suitable for use in construction compared to the other two types of conventional bricks. This is also of great significance because geopolymer bricks may become the main load bearing elements that would be able to carry several floors more than allowed for the conventional bricks.

Water Absorption

From the data obtained in figure 2, the water absorption of geopolymer bricks could be decreased as the age of geopolymer bricks increased. The water absorption of fly ash-based geopolymer bricks ranged between 3.5% - 6.7%. The water absorption at 1 day (6.7 %) of ageing was higher while at 60 days (3.5 %) of ageing was the lowest percentage of water absorption. Most of the bricks available in the market contain pores which will permit the water to pass through pore space. This result obtained was due to the capillary effect on the pores of the bricks, the pores will absorb water from the mortar that lay on the bricks [15].

Dimensional Tolerances

Table 4 below shows the results obtained from the measurement of 24 bricks and individual measurement of brick. The results tabulated in table 4 were compared with the work sizes stated in the BS 3921: 1985. The work sizes were as given in the BS for length, width and height i.e. 215 mm, 102.5 mm, and 65 mm respectively. The mean measurement for individual length was 215.9 mm,

Table 3
PROPERTIES OF GEOPOLYMER BRICKS COMPARED TO COMMON BRICKS USED IN MALAYSIA

	Geopolymer bricks	Clay bricks	Cement bricks
Compressive strength (MPa)	5 to 25	4.5 to 8.5	2 to 5
Water absorption (%)	3.5 to 7	5 to 15	5 to 10
Density (kg/m^3)	1800 to 1950	1750 to 1900	1800 to 1900

Table 4
DIMENSIONS OF BRICKS MEASURED

Dimensions	Total measurement for 24 bricks (mm)	Mean measurement for individual brick (mm)
Length, L	5182	215.9
Width, W	2395	99.8
Height, H	1545	64.3

Table 5
DENSITY OF GEOPOLYMER BRICKS MEASURED

Age (Days)	Density (Kg/m^3)
1	1813.5
3	1824.0
7	1828.2
28	1887.5
60	1915.4

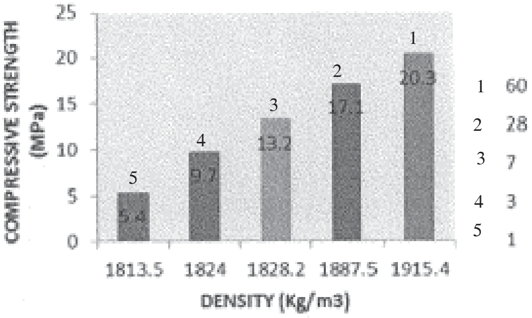


Fig. 3. Relation of density and compressive strength of geopolymer bricks

Table 6
PROPERTIES OF GEOPOLYMER BRICKS COMPARED TO COMMON BRICKS USED IN MALAYSIA

	Geopolymer bricks	Clay bricks	Cement bricks
Compressive strength (MPa)	5 to 25	4.5 to 8.5	2 to 5
Water absorption (%)	3.5 to 7	5 to 15	5 to 10
Density (kg/m^3)	1800 to 1950	1750 to 1900	1800 to 1900

exceeding the limit stated in BS 3921: 1985 by 0.9 mm. While the individual measurement of width and height is still within the limits of BS 3921: 1985 which showed the value of 99.8 mm and 64.3 mm respectively. Table 4 clearly demonstrates that the fly ash-based geopolymer bricks in this research had widths and heights marginally in agreement with the BS 3921: 1985 standards but the length were oversize.

Density Analysis

Table 5 shows the density of fly ash-based geopolymer bricks at different ages. The geopolymer bricks at 60 days of ageing have highest density, whereas the geopolymer bricks at 1 day of ageing have the lowest density. An increase in the density from 1 day to 60 days of ageing is caused by the complete reactions of geopolymerization occurred in the geopolymer bricks. When geopolymerization reaction has reached completeness, the geopolymer structure becomes compact and denser hence provide higher density. This result was associated with the compressive strength test results. As shown in figure 3, the density and compressive strength of geopolymer bricks are related to each other. The higher density of the bricks will provide the higher strength of bricks.

Comparison between Geopolymer Brick and Conventional Brick

Table 6 below shows the comparison between fly ash-based geopolymer bricks with those of other bricks available in the market. Two types of bricks comprising of the common clay bricks and cement bricks used for construction in Malaysia were chosen randomly from the factory output. Five samples were tested for compressive strength, three samples were tested for water absorption and density analysis prior for comparison with the fly ash-based geopolymer bricks.

The results obtained shows that the geopolymer bricks performed better characteristics and properties due to high compressive strength (5 to 25 MPa), low water absorption (3.5 to 7%) and the range of density between 1800 to 1950 kg/m^3 is suitable for use in construction compared to the other two types of conventional bricks. This is also of great significance because geopolymer bricks may become the main load bearing elements that would be able to carry several floors more than allowed for the conventional bricks.

Microstructure of fly ash-based geopolymer bricks
Scanning Electron Microscope (SEM)

The microstructure of geopolymer bricks using fly ash cured at 70°C for 24 h was examined using Scanning Electron Microscopy (SEM). The SEM analysis of the microstructure of geopolymers bricks was analyzed under 2000x magnifications while the original fly ash was analyzed under 5000x magnifications. From the micrograph, the formation of geopolymers can be observed in between of the fly ash particles.

Figure 4 showed the micrograph of original fly ash before the activation takes place. The original fly ash consists of a series of spherical vitreous particles of different sizes, but with a regular smooth texture. These particles are usually hollow, and some spheres may contain other particles of a smaller size in their interiors.

Figure 5 shows the results of fly ash activation with alkaline solution at room temperature, at 70°C, at 7 days of ageing and 60 days of ageing respectively. The number of unreacted fly ash particles decreases as the temperature and age of geopolymer bricks increased. It was observed in figure 5 the unreacted ash particles are embedded in the binder, and display some dissolved particles of ash together with the gel formation. It is clear that there is an

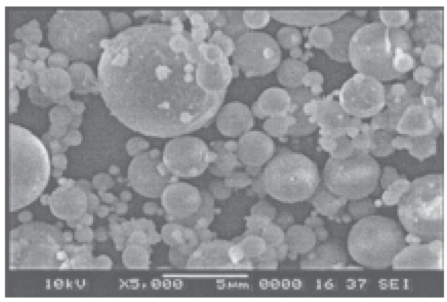


Fig. 4. SEM micrograph of class fly ash with amplify x 5000 magnifications

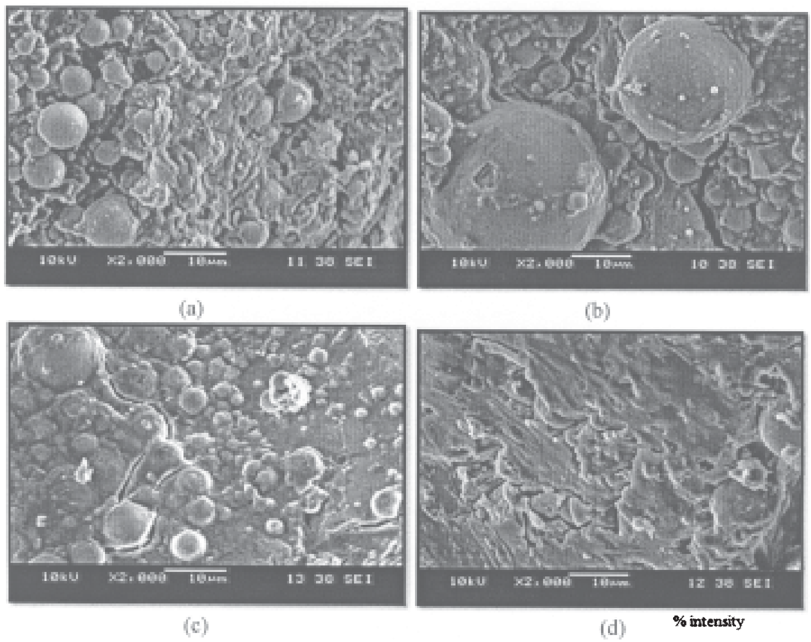


Fig. 5. (a) cured at room temperature for 24 h; (b) cured at 70°C for 24 h; (c) 7 days of ageing; (d) 60 days of ageing

intimate bond between the aggregate and geopolymer binder. A measly formed micro cracks were also found on the surface of geopolymer bricks structure.

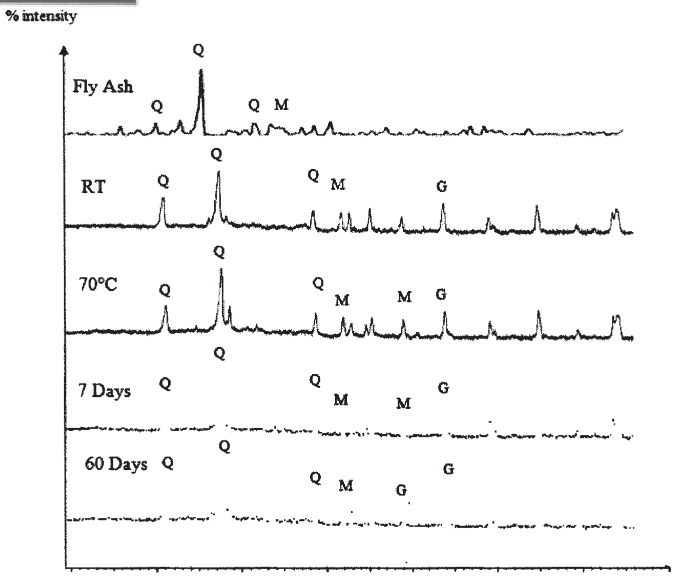
Curing at room temperature (fig. 5(a)) presents a scarcely compact structure in which numerous unreacted spherical fly ash particles are clearly visible. On the other hand, when curing is carried out at 70°C for 24 h (fig. 5(b)), unreacted spherical ash particles are embedded in the matrix and irregularly shaped micro cracks are clearly seen between the unreacted fly ash particles and the geopolymeric matrix. As seen in figure 5 (b), the microstructure of geopolymer brick cured at 70°C for 24 h would be denser than geopolymer bricks cured at room temperature (fig. 5(a)). These results explained its ability to perform in higher strength and lower absorption.

The microstructure of geopolymer bricks at 60 days of ageing (fig. 5(d)) shows the disappearance of sphere-shaped fly ash particles and the microstructure of geopolymeric brick became relatively compacted compared to the microstructure of geopolymer bricks at 7 days of ageing (fig. 5(c)). The compactness of geopolymer materials improved by duration of activation attributed to the formation of greater amount of alumino-silicate gel.

In the micrograph, the presence of gel (sodium aluminosilicate hydrate) that forms the cementitious matrix could be observed alongside the spheres of unreacted ash particles [16]. Hence, the geo-polymerization without thermal activation could be regarded as a superficial reaction based on diffusion mechanisms rather than on a complete dissolution and subsequent formation of species [17].

X-ray Diffraction Analysis (XRD)

X-ray diffraction patterns for fly ash and geopolymer bricks were recorded over the 2θ angle range 10 to 80° counting for 1 second per 0.02° step. The diffractograms for all the geopolymer bricks studied at different conditions, as well as for the original fly ash, are represented in figure 6. The main phases formed in XRD pattern of the geopolymer bricks are amorphous or non-crystalline content. In a non-crystalline state, diffraction of X-rays resulted in a broad diffuse halo rather than sharp diffraction peaks. The broad peak of amorphous structure can be observed around 22° 2θ . Figure 6, shows that the major mineral components of original fly ash samples were mullite, quartz, and magnetite. The presence of the sharp peaks detected in the fly ash was due to the presence of quartz. The diffractogram for the original fly ash changed perceptibly when the ash was activated by alkaline solutions whose halo is attributed to the vitreous phase of the original ash slightly shifted from 25° to 40° 2θ values. This change indicates the formation of an alkaline aluminosilicate hydrate gel which has been identified as the primary reaction product and the characteristics diffraction patterns of geopolymeric [16-26].



{Q]: Silicon Oxide (Quartz) [SiO2]; [M]: Aluminium Silicate (Mullite) [$Al_6Si_2O_{13}$]; [G]: Magnetite [Fe_3O_4]

Fig. 6. Diffractograms for geopolymer bricks and original fly ash

The XRD pattern obtained in figure 6 shows that there is a marked shift in the halo to the higher 2θ values with increasing curing temperature and age of geopolymer bricks. The halo exhibit at least two highest intensity with low angle composition at 2θ = 20° to 2θ = 27° compatible to three-dimensional networks and a high angle composition at 2θ = 30° to 2θ = 39° compatible to low molecular weight silicate (dimer, monomer) [18]. The most intense halo peak was registered between 2θ = 20° and 2θ = 30° and was attributed to an amorphous silicate phase consisting of a SiO_4 tetrahedra sharing oxygen atoms [19]. The humps in XRD patterns of geopolymers were different from that in the original fly ash and indicated the dissolution of fly ash and formation of amorphous structure in the geopolymer [21]. The XRD analysis showed that the evolution phases geopolymer bricks play an important role for determining the interaction between fly ash particles and alkaline solution and they are only slightly different and not obvious.

Conclusions

The optimum ratio of fly ash-to-sand used in this research for the next parameters and test is 1:3 ratio, by mass due to the good performance in workability and structure which is suitable for use in production of geopolymer bricks. In this research, the optimum curing temperature was set at 70°C due to the achievement of higher compressive strength and lower absorption. Strength of 20.3 MPa was found to be the highest strength of geopolymer bricks produced when cured for 60 days at room temperature after removed from the oven treatment. The lowest percentage water absorption of geopolymer bricks is 3.5 % shown by bricks cured for 60 days.

Good quality geopolymer bricks can be produced from stipulated proportions of fly ash (raw materials), alkaline activator (NaOH and Na_2SiO_3 solutions) and sand. The manufacturing process of the geopolymer bricks is simple and does not require any firing or autoclaving. The heat treatment only requires low temperature and short time for curing. Therefore, the energy consumption will be much less as compared to the conventional burnt clay bricks and calcium silicate bricks. Sufficient strength is obtained by moulding the bricks at significantly lower pressure (10 MPa) compared to that for traditional or conventional bricks.

Acknowledgements: This study was supported by Center of Excellence Geopolymer and Green Technology UniMAP and School of Material Engineering, UniMAP. The authors of this research are very grateful to King Abdul Aziz City Science and Technology (KACST) for the financial support of this study.

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Manuscript received: 2.06.2014