Physical and Mechanical Properties of Lightweight Aerated Geopolymer

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ABSTRACT

In this study, it is going to investigate properties of lightweight geopolymer specimens aerated by aluminium powder. It has been established well that aluminium powder can be appropriately used for foaming of traditional concrete. Reaction between aluminium powder and alkali activator in geopolymers of this study caused high porous structures based on the weight ratios of constituent materials. Different specimens were made by changing sodium silicate to sodium hydroxide, and alkali activator to fly ash weight ratios. Fly ash was substituted by aluminium powder with 1.5, 3.0 and 5.0 wt.% in different mixtures. Results indicated that substituting of 5.0 wt.% of fly ash by aluminium powder in the specimens with alkali activator to fly ash weight ratio of 0.35 and sodium silicate to sodium hydroxide weight ratio of 2.5 causes the best foamed specimen with the lowest density. Compressive strength of all aerated specimens were in the range of 0.9-4.35 MPa, which is suitable for using as bricks, fire-resistant panels, buried pipeline and so on. Finally, SEM analysis was conducted to evaluate the microstructure of successfully aerated geopolymer. It was seen that in highly aerated specimens, the foaming reaction is too fast that prevents complete alkali activation of geopolymers and many unreacted fly ash particles remains.

1. Introduction

Geopolymerization technology was proudly introduced in early 1980s by Joseph Davidovits because of its environmental friendly process [1]. Geopolymer is an adhesive aluminosilicate forming by alkaline activation of alumina and silica as starting materials at temperature slightly higher than room temperature [1, 2]. These alkali-activated materials are arranged of tetrahedral silicate and aluminate units bonded in a three dimensional structure by covalent bonds [2]. The materials used for geopolymerisation are divided into two parts: a reactive aluminosilicate material such as fly ash or calcined clays, and alkali activator solution (generally a mixture of alkali metal hydroxide such as sodium hydroxide and silicate solution such as sodium silicate) [2]. Geopolymers are usually used in building construction, bridge superstructure and deck pavements.

Geopolymers are fire-resistant materials and hence, fabricating lightweight geopolymers with enhanced thermal resistivity can be considered as an effective way of usage of these materials. Although properties and the nature of geopolymers are now clearer, their lightweight structures have not been developed well. However, some attempts have been made to introduce lightweight geopolymers through different methods. Omar et al. [1] produced geopolymers containing lightweight aggregate and tested them at elevated temperatures. Results indicated that lightweight geopolymers have better fire resistance than normal geopolymer at temperatures above 100 ^{o}C. Successful utilizing of lightweight aggregates for production of geopolymers has been reported in some other works. Aggregates from recycle lightweight block [3], oil palm shell as coarse lightweight aggregate [4-6], Cold

bonded lightweight aggregate [7], Cenospheres and expanded polystyrene (EPS) lightweight aggregates [8], palm oil clinker aggregates [9], mixture of expanded vermiculite and electrical porcelain [10, 11], refractory shale haydite [12] and artificially expanded clay granules [13, 14]. Pimraksa et al. [15] studied properties of geopolymers produced by highly porous lightweight siliceous materials including rice husk ash and diatomaceous earth. They could obtain mean bulk density of 0.88 g/cm^3 and compressive strength of 15 kg/cm^2 in the best condition. Alkali-activated fly ash has been also used as lightweight aggregate in traditional concrete [16]. Some works have been performed on production of lightweight aerated geopolymers by means of aluminium powders [17, 18].

Aerated concrete with significant reduction in density is basically a mortar with pulverized sand and industrial waste like fly ash as filler (individually or together). In these type of lightweight products, air is entrapped artificially by chemical (metallic powders like Al, Zn, H_2O_2) or mechanical (foaming agents) facilities [19]. A wide range of densities are obtained by suitable aeration of concrete and the products are used in specific applications in structural, partition and insulation grades [20]. Aerated concrete is an old concept and it is interesting that the first comprehensive review on it was made in 1954 [20-22]. Therefore, it is not convenient to review all published works in this area and just few of them is summarized here. Since the aim of this paper is development of aerated geopolymers by means of aluminium powder, a short survey has been conducted on traditional aerated concrete produced by aluminium powder.

Huang et al. [23] produced aerated concrete by utilizing skarn-type copper tailings and blastfurnace slag, and achieved compressive strength of 4 MPa and density of 610 kg/m^3. They proposed participation of most minerals available in the copper tailings in the hydration reaction during the procuring process. Kurama et al. [24] used coal bottom ash in ordinary

Portland cement (OPC) concrete and achieved compressive strengths up to 3.0 MPa.

Wongkeo et al. [25] have also reported production of aerated concrete by using bottom ash.

Although their specimens have relatively high compressive strengths (even greater than 10

MPa), the density of all produced samples was above 1000 Kg/m^3. By increasing the content

of bottom ash in their examined concrete, they achieved a slight improvement in compressive

strength, flexural strength and thermal conductivity. Not only aerated OPC concrete, but

aerated geopolymer are also produced by using aluminium powder. Arellano Aguilar [26]

produced aerated geopolymer paste and concrete by utilizing a mixture of metakaolin and fly

ash. Densities were fixed at 600, 900 and 1200 kg/m^3 and maximum compressive strength of

about 3.5, 8 and 16 was achieved by using these densities respectively. Brooks et al. [27]

produced aerated geopolymeric specimens from high strength geopolymer and while their

minimum density was 1000 kg/cm^3, compressive strength values were less than 10 MPa. This

short review indicates the relationship between compressive strength and density of OPC and

geopolymer concrete specimens and where density of specimens is below 1000 kg/cm^3,

compressive strength is normally less than 4 MPa. Liu et al. [28] studied physical and thermal

properties of aerated geopolymers made from metakaolin, α-Al_2O_3, Al powder and

phosphoric acid. Compressive strength of all specimens was more than 6 MPa while their

porosities ranged between 40 to 83 %. This relatively high strength was supposed to be due to

formation of Al-O-P bonds. Thermal analysis of the specimens revealed that those are

thermal-stable, in terms of compressive strength and shrinkage, at temperatures up to 1450

^{o}C.

The aim of the present paper is to produce aerated fly ash-based geopolymer pastes by

using different amount of aluminium powder. Density, compressive strength, macro- and

micro-structure of the produced samples are studied. Different sodium silicate to sodium

hydroxide (NaOH) and alkali activator to fly ash ratios are investigated, and the effect of percentage of aluminium powder on foam-ability is surveyed.

2. Experimental procedure

Class F fly ash was used to produce aerated geopolymers. Chemical and mineralogical of fly ash were analysed by XRD and XRF techniques. Bruker D8 Advance X-ray diffractometer was used for XRD analysis while scans collected between 5-70 ° of 2θ with a step size of 0.02 ° and a scan rate of 5 sec per step. An internal standard (10 wt.% corundum, Al_2O_3) was added to allow quantitative analysis using Rietveld refinement. Phase identification was completed using Materials Data, Inc., Jade 9.3 software and Quantitative Rietveld analysis using Bruker Diffrac[plus] Topas software. Chemical composition and mineralogical phases of the used fly ash have been given in Tables 1 and 2 respectively. Particle size distribution of the materials was obtained by using Cilas laser diffraction particle analyser and the result is illustrated in Fig. 1. SEM micrograph of fly ash has been given in Fig. 2. SEM micrographs of fly ash (and geopolymeric samples) were obtained using a Gemini apparatus with 5.0 kV energy and secondary electrons. The specimens were first coated by gold and then analysed by SEM.

D-Grade sodium silicate solution (29.4 SiO_2 and 14.7 % Na_2O by weight) from PQ Australia and analytical grade NaOH solid from Sigma Aldrich were used as alkaline activators. NaOH flakes were dissolved in distilled water to achieve 8 M NaOH solution and then the resultant solution was left in the environment to cool down. Sodium silicate was used as-received for mixing by NaOH and making alkali activator. Two ratios of sodium silicate to NaOH solution (2.5:1 and 1:1) were used in this study.

For aerating the specimens, fly ash was partially substituted by commercially pure aluminium powder (Al powder) with 1.5, 3.0, and 5.0 wt.% were appropriate. The average particle size of Al powder was 50 μm. Table 3 illustrates mixture proportions of the produced aerated samples.

For production specimens, fly ash was dry mixed by Al powder for 5 min and then alkali activator was added to the mixture. Specimens then were poured into cubic moulds with the dimensions of $50 \times 50 \times 50$ mm^3. Mixtures raised freely in few second, and the volume of produced bubbles as a result of reaction of Al powder and alkali activator depended on the ratios of materials used. After pouring the mixtures into the moulds, they were placed left for 24 hours in ambient temperature, while covered by plastic bags. After demoulding, they were oven cured for another one day at 60 ^{o}C. Afterwards, the samples were taken out and placed in the lab environment for two continuous days. Bulk density of specimens was achieved by weighting of specimens and dividing the achieved weight to the volume of specimens. Compressive strength of specimens was acquired in accordance to the ASTM C109 standard. SEM images of the specimens were provided as well. The flowchart of Fig. 3 shows experimental procedure conducted in the present work.

3. Results and discussion

Fig. 4 illustrates some of geopolymer mixtures aerated by Al powder. From specimen observations, it is evident that mixtures with alkali activator to fly ash weight ratio of 0.35 (high content of liquid) have the highest foam-ability. Although sodium silicate to NaOH ratio is important (compare Figs. 4a and 4b), but the most important factor seems to be the amount of liquid rather than its type. Reaction between Al powder and alkali materials

produces H_2 which causes high porous geopolymers. The interesting point is that successful aerating of geopolymers can be done without requiring autoclave treatment while this success for OPC concrete can only be achieved in autoclave conditions [23-25, 27]. It seems that viscosity of the liquid in geopolymer is higher than OPC concrete and hence air and H_2 bubbles are trapped easier. Fig. 4 shows the effect of percentage of Al powder on foam-ability of the considered mixtures and it is obvious that by utilizing higher contents of Al powder, more porous foam is produced. Fig. 4d illustrates that Al powder has positive effect only if the content of liquid is enough and in this case, raising of mixtures due to production of low content of H_2 bubbles is not of high performance. Therefore, successful aerating of geopolymer pastes depends on two dependent parameters: the amount of alkali activator and percentage of Al powder. A verification of this can be seen in macrographs of Fig. 5. In these figures, for alkali activator to fly ash weight ratios of 0.3 and 0.35, the surface structure of the foam consists of visible big pores (suitable for lightweight applications) while for alkali activator to fly ash weight ratio of 0.25 (low amount of liquid), macro-pores are not visible.

Density values of all aerated geopolymer pastes have been illustrated in Table 3. The lowest density is related to G1 specimen (403 kg/m^3) while the highest density is more than three times greater (1309 kg/m^3) and is related to G12 specimen. In a specific group (with the same sodium silicate to NaOH solution and alkali activator to fly ash weight ratios), increasing percentage of Al powder causes lower densities as expected. Effects of Al powder on density of the considered mixtures at various contents of mixtures parameters has been illustrated in Fig. 6. These figures can be considered as useful guides to illustrate the effect of each mixture parameter on foam-ability of the considered mixtures. For convenient, and from now on, densities are divided to three group and specimens having these values are known as

low density (less than 800 kg/m^3), medium density (between 800 and 1000 kg/m^3) and high density (above 1000 kg/m^3).

Fig. 6a illustrates the effect of fly ash content on density of geopolymers at different percentages of Al powder. It is obvious that in any percentage of Al powder, the higher content of fly ash causes the higher density. As it was stated previously, the higher

content of fly ash (lower content of liquid) results in lower amount of H$_2$ bubbles and hence foam-ability of concrete decreases. For very high fly ash content, Al powder has the lowest effect on decreasing density.

Fig. 6b illustrates the effect of NaOH solution content on density of geopolymers containing different amounts of Al powder. There are two different behaviours in this figure in NaOH solution contents of less than 140 (lower band), and higher than 140 kg/m^3 (medium band). In lower band and with any content of Al powder, densities are same and relatively high. In upper band, densities decrease by increasing percentage of Al powders; however, in higher content of NaOH solution and low percentages of Al powder, densities are higher. From the figure it can be seen that medium levels of NaOH solution are more suitable to attain lower densities. Success in alkali activation of fly ash and producing geopolymers strongly depends on the ratio of sodium silicate to NaOH solution. While this ratio has a wide range of usage, a ratio of 2.5 has been illustrated to have better efficiency on production of geopolymers. While the content of NaOH solution is very low (in this paper it was occurred when the liquid content was low), aeration is hard and therefore densities are higher. On the other hand, in high contents of NaOH solution (for sodium silicate to NaOH solution weight ratio of 1), viscosity of the liquid is not high enough to effectively trap H$_2$ bubbles and hence, density is supposed to be higher.

Fig. 6c shows the effect of sodium silicate content on density of aerated geopolymers at different percentages of Al powders. The effect of sodium silicate content on density values can be considered in two ranges. The first range is for sodium silicate contents of less than 320 kg/m^3 where aeration of geopolymers depends on the percentage of Al powder (which decreases by increasing the percentage of Al powder). The second range is for contents above 320 kg/m^3 where densities are independent of the content of Al powder. The former range is occurred when the content of liquid is low or the content of NaOH solution is high. Therefore, successful aeration depends on the percentage of Al powder and subsequent formation of H$_2$ bubbles. In the latter range, the amount of viscose sodium silicate is high enough to trap most of the formed H$_2$ bubbles during aeration and hence the percentage of Al powder just contributes in the kinetic of aeration. Visual observations during producing of specimens revealed that by using higher percentage of Al powder, foaming is done faster. This figure also shows that by increasing sodium silicate content in the latter range, densities decreases.

Although some discussions on the effect of NaOH solution and sodium silicate contents was presented, Fig. 6d illustrates that the effect of sodium silicate to NaOH solution weight ratio on density of aerated geopolymers depends on the percentage of Al powder. It should be noted that in production of specimens with alkali activator to fly ash weight ratio of 0.25, we just could produce specimens with sodium silicate to NaOH solution weight ratio of 2.5 and production of specimens with sodium silicate to NaOH solution weight ratio of 1 was not possible because of flow difficulties and geopolymerization possibility. Therefore, high densities obtained here (in absence of weight ratio of 1) cause this problem. Therefore, Fig. 6d cannot be considered as an efficient illustration of the fact. Instead, Fig. 6e shows that densities of specimens are more dependent on alkali activator to fly ash weight ratio than the

percentage of Al powder. Therefore, as indicated above, the content of liquid can be considered as the most important parameter determines densities of aerated geopolymers.

Compressive strength values of all aerated geopolymer pastes have been illustrated in Table 3. The lowest strength is related to G1 specimen (0.90 MPa) while the highest strength is near five times greater (4.35 MPa) and is related to G12 specimen. In a specific group (with the same sodium silicate to NaOH solution and alkali activator to fly ash weight ratios), increasing percentage of Al powder causes lower strengths. From the table and Fig. 7, which illustrates the relationship between density and compressive strength values at different percentages of Al powder, it is obvious that specimens with lower densities have lower compressive strength which is an expected result due to higher content of porosities formed during aeration of geopolymers with lower densities. Same as the way followed for densities, the effect of different parameters on compressive strength of aerated geopolymers at different percentages of Al powder has been illustrated in Fig. 8.

Fig 8a shows the effect of fly ash content on compressive strength of geopolymers at different percentages of Al powder. It is evident that higher compressive strengths are achieved by higher content of fly ash. This is an acceptable result where the content of fly ash act as the cement content in OPC concrete and higher strengths are achieved by higher binder contents. The effect of NaOH solution and sodium silicate content on compressive strength as illustrated in Figs. 8b and 8c respectively are similar to the effect of these parameters on density values. This is an acceptable behaviour that shows density and strength values behave in same manner and one decreases by decreasing the other. Finally Figs. 8d and 8e show that compressive strength of specimens depends on the content of liquid and where the content of liquid is low, higher strengths are achieved.

SEM micrographs of some aerated geopolymeric samples have been illustrated in Fig. 9. It is evident from all pictures that many unreacted fly ash particles are remained in the aerated sample. This is due to the rapid reaction between Al powder and alkali activator. While geopolymerization required long times, the reaction is completed in few seconds and therefore, some part of alkali activator is consumed. Additionally, stoichiometry of alkali activator is damaged as a result of this reaction. Due to the high porosity of specimens, circulating of air inside the specimens is easier as well (it was tried to minimize this circulation by putting the specimens in plastic bags) and hence carbonation may cause lower strengths. All of these effects as well as high porosity of specimens causes low strength in aerated specimens. An evidence for damaging stoichiometry of alkali activator can be observed in Figs. 9d and 9c. In these figures alkali activator to fly ash weight ratio is low and hence aeration has not been done well. However, the change in stoichiometry of alkali activator has caused remaining of alkali activator as glue on the surface of fly ash particles. This glue which may gain its strength through carbonation causes weak bonds between fly ash particles and although it seems that the strength of the resultant specimen is between aerated specimens, it is not an acceptable specimen for engineering application of geopolymers.

4. Conclusions

In the present paper, the effect of Al powder's content on density, compressive strength and microstructure of aerated geopolymer pastes was studied. Different geopolymer pastes were made by changing sodium silicate to NaOH solution (1 and 2.5) and alkali activator to fly ash (0.25, 0.30 and 0.35) weight ratios. These specimens were aerated by

different percentages of Al powder (1.5, 3.0 and 5.0 wt.% of fly ash). In total, 12 different aerated mixtures were investigated and the following conclusions were arisen from this study:

- The lowest density was 403 kg/m^3 for a specimen with sodium silicate to NaOH solution weight ratio of 2.5, alkali activator to fly ash weight ratio of 0.35 and percentage of Al powder of 5.0. The highest density was more than three times greater (1309 kg/m^3) and was related to a specimen with sodium silicate to NaOH solution weight ratio of 2.5, alkali activator to fly ash weight ratio of 0.25 and percentage of Al powder of 1.5. It was shown that the content of alkali activator (liquid) is the most important parameter affecting density values of aerated geopolymers and lower densities are achieved by utilizing more liquid content.

- The lowest compressive strength was 0.90 MPa for a specimen with sodium silicate to NaOH solution weight ratio of 2.5, alkali activator to fly ash weight ratio of 0.35 and percentage of Al powder of 5.0. The highest density was near five times greater (4.35 MPa) and was related to a specimen with sodium silicate to NaOH solution weight ratio of 2.5, alkali activator to fly ash weight ratio of 0.25 and percentage of Al powder of 1.5. Both strength and density values behave in the same manner and strength of specimens decreased when the density was lower. Specimens with lower liquid content revealed higher strength.

- Microstructure analysis of the specimens showed that many unreacted fly ash particles remained during aeration of geopolymers as a result of fast reaction between Al powder and alkali activator. Additionally, specimens with low liquid gained their strength through a weak adhesion between fly ash particles. Aerating of geopolymers causes damages in stoichiometry of alkali activator which causes incomplete Geopolymerisation of fly ash particles.

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Table 1. Chemical composition of fly ash

Chemical composition	SiO_2	Al_2O_3	CaO	MgO	Fe_2O_3	Na_2O	P_2O_5	K_2O_5	MnO	SO_3	TiO_2	L.O.I.
Content (wt.%)	51.1	25.6	4.30	1.45	12.5	0.77	0.89	0.70	0.15	0.24	1.32	0.57

Table 2. Mineralogical phases of fly ash

Material	Quartz	Mullite	Hematite	Magnetite	Amorphous (SiO_2 + Al_2O_3+ CaO+ other oxides)
Content (wt.%)	13.0	14.6	4.17	1.83	66.0 (32.3+16.8+4.30+12.6)

Table 3. Initial mixture proportion of geopolymer pastes (non-aerated condition)

Sample designation	Sodium silicate/NaOH solution	AA/FA	Percentage of Al Powder (wt.% of FA)	Content of materials (Kg/m^3)				Density (Kg/m^3)	Compressive strength (MPa)
				FA	NaOH solution	Sodium silicate	Al powder		
G1	2.5	0.35	5.0	1401	142	356	21.3	403	0.90

G2	2.5	0.35	3.0	1375	142	356	46.7	635	1.35
G3	2.5	0.35	1.5	1351	142	356	71.1	712	1.80
G4	1	0.35	5.0	1401	249	249	21.3	545	1.20
G5	1	0.35	3.0	1375	249	249	46.7	739	1.95
G6	1	0.35	1.5	1351	249	249	71.1	915	3.00
G7	1	0.3	5.0	1455	222	222	22.2	685	1.50
G8	1	0.3	3.0	1433	222	222	44.4	963	2.85
G9	1	0.3	1.5	1403	222	222	73.9	1102	3.45
G10	2.5	0.25	5.0	1513	110	274	23.0	1126	3.60
G11	2.5	0.25	3.0	1490	110	274	46.1	1235	3.90
G12	2.5	0.25	1.5	1459	110	274	76.8	1309	4.35

FA: fly ash; AA: alkali activator (NaOH solution + sodium silicate)
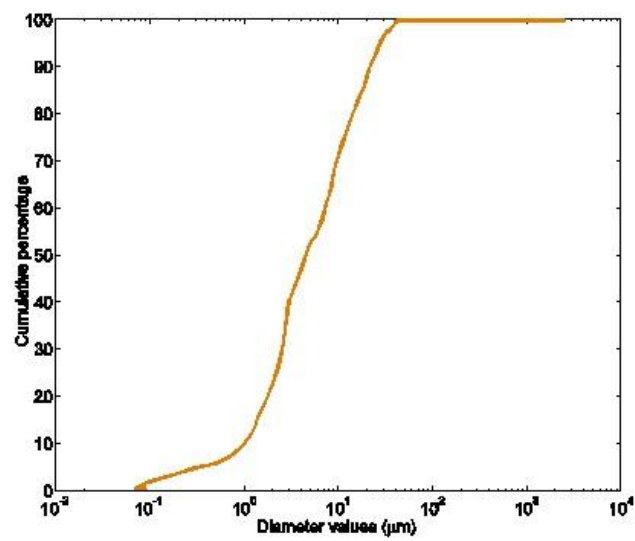
Fig. 1. Particle size distribution of fly ash sample
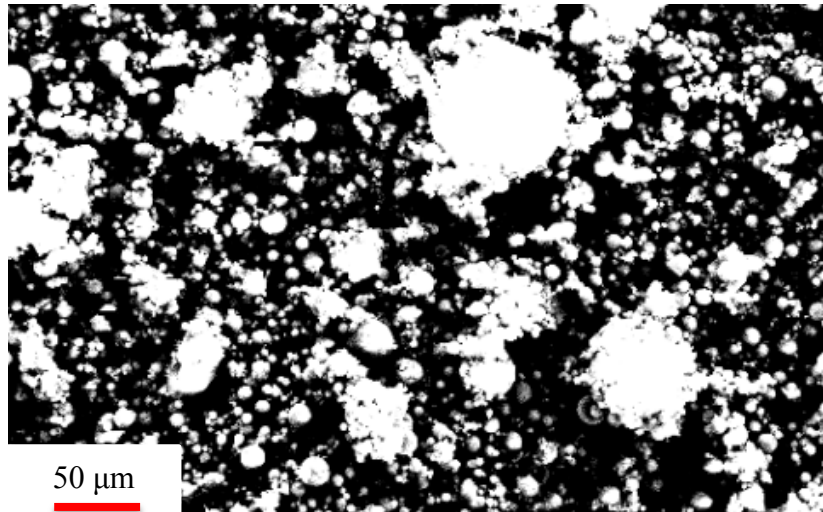
Fig. 2. SEM micrograph of fly ash powder

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                    ┌─────────────────────────┐
                    │   Characterized fly ash  │
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| Alkali activator (sodium silicate and NaOH solution) | | Al powder (1.5, 3.0 and 5.0 wt.%) |

Production of aerated geopolymers

| Compressive strength | Density | Microstructure |

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                          ┌───────────────┼──────────────────────┐
                          ▼               ▼
                 ┌─────────────────────────────────┐
                 │        Analyzing the results     │
                 └─────────────────────────────────┘
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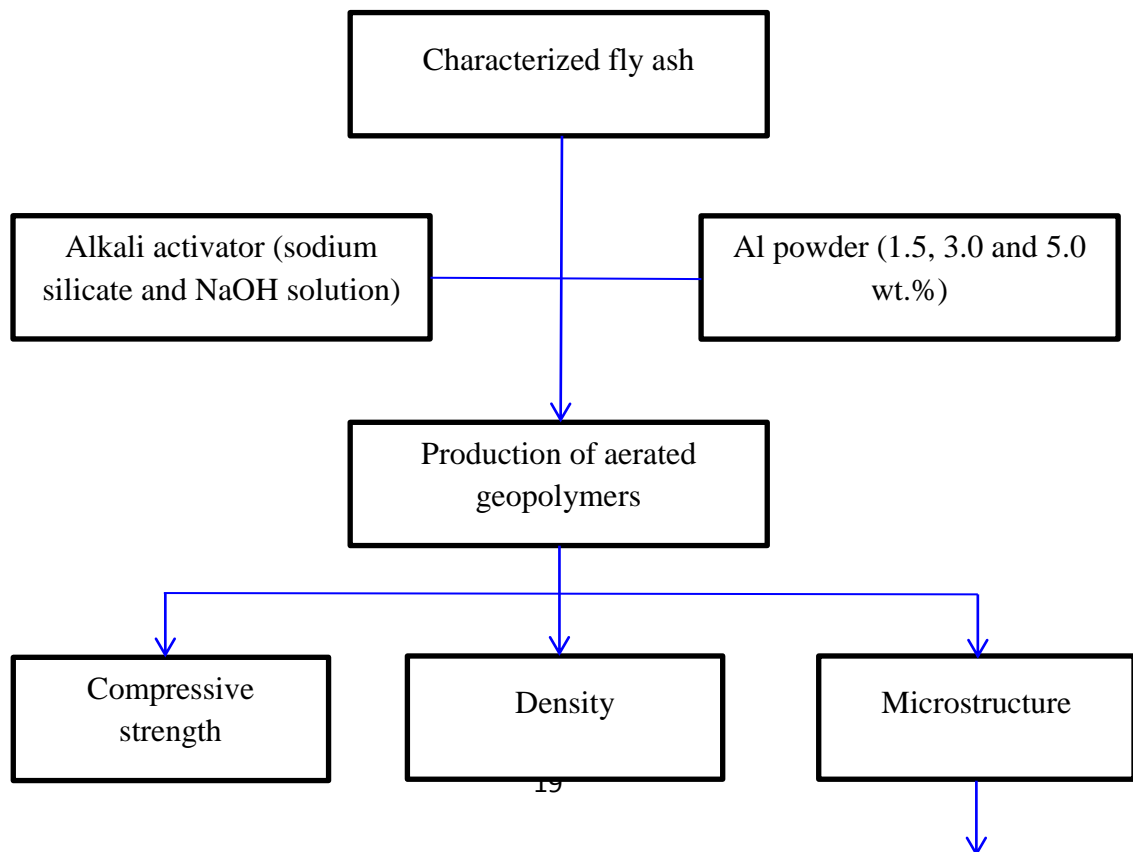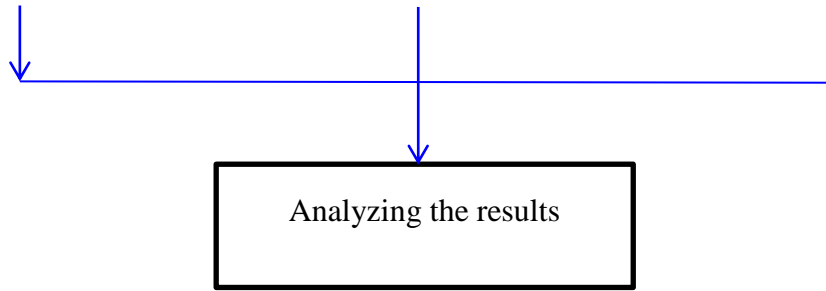
Fig. 3. Experimental procedure of the current work

(a)

(b)

(c)

(d)

Fig. 4. Foam-ability of aerated geopolymer samples by aluminium powders; a) Sodium silicate/NaOH solution = 2.5, AA/FA = 0.35, b) Sodium silicate/NaOH solution = 1, AA/FA = 0.35, c) Sodium silicate/NaOH solution = 1, AA/FA = 0.3 and d) Sodium silicate/NaOH solution = 2.5, AA/FA = 0.25. In all parts of picture (a, b, c and d), from left to right, percentage of Al powder is 1.5, 3.0 and 5.0.

5 mm

(a)

5 mm

(b)

5 mm

(c)

5 mm

(e)
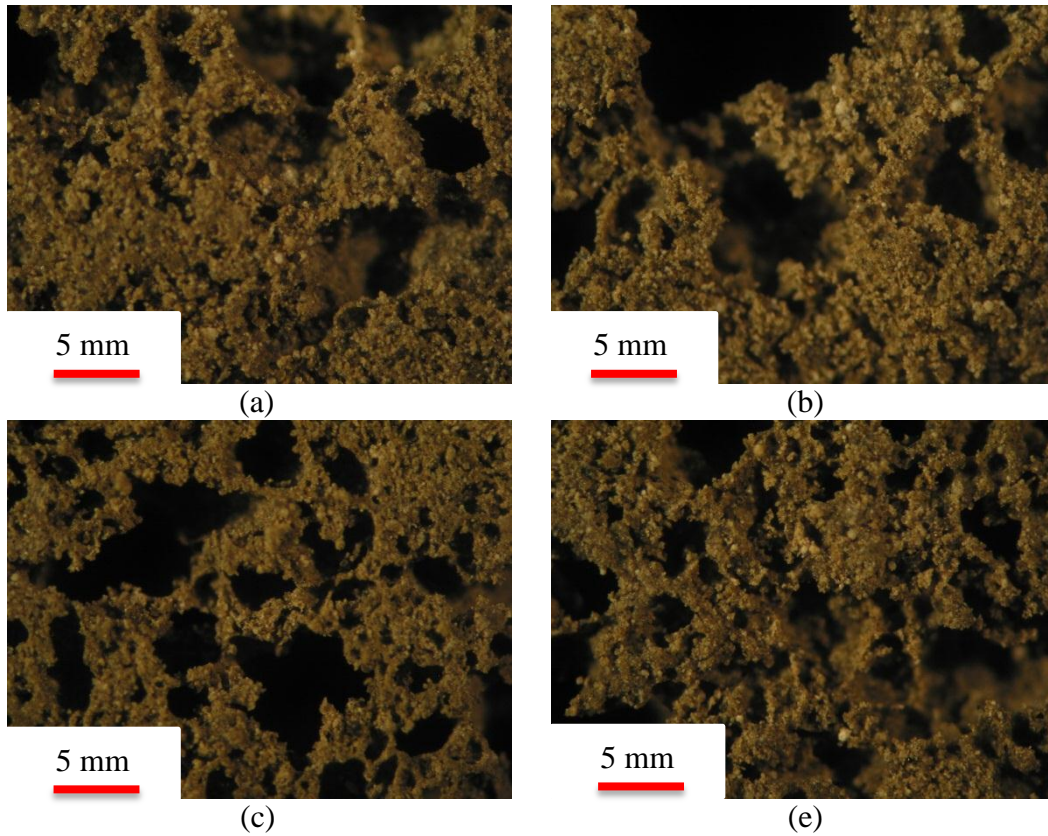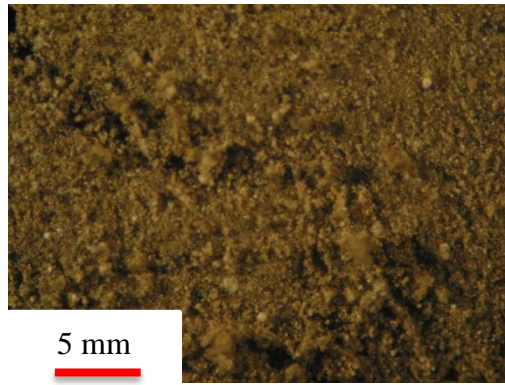
(e)

Fig. 5. Macrographs of aerated geopolymer samples; a) Sodium silicate/NaOH solution = 2.5, AA/FA = 0.35, wt.% of Al powder = 5.0, b) Sodium silicate/NaOH solution = 2.5, AA/FA = 0.35, wt.% of Al powder = 3.0, c) Sodium silicate/NaOH solution = 1, AA/FA = 0.3, wt.% of Al powder = 5.0, d) Sodium silicate/NaOH solution = 1, AA/FA = 0.3, wt.% of Al powder = 1.5 and e) Sodium silicate/NaOH solution = 2.5, AA/FA = 0.25, wt.% of Al powder = 5.0.
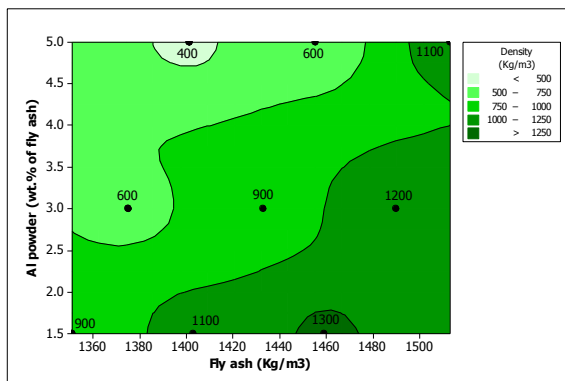
(a)
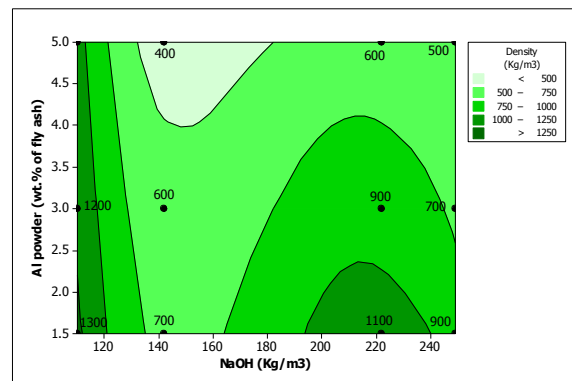
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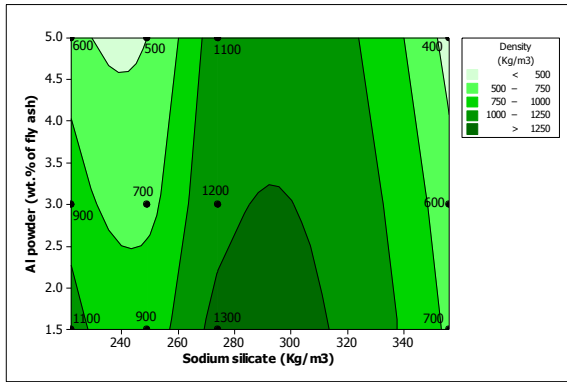
(c)
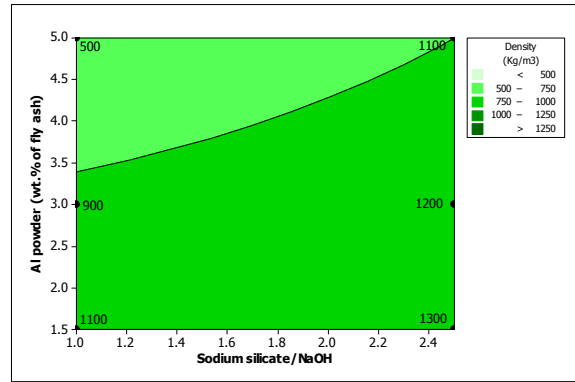
(d)
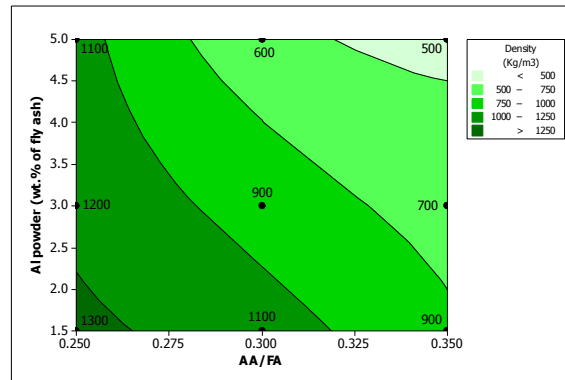
(e)

Fig. 6. Effects of different parameters on density of aerated geopolymer pastes
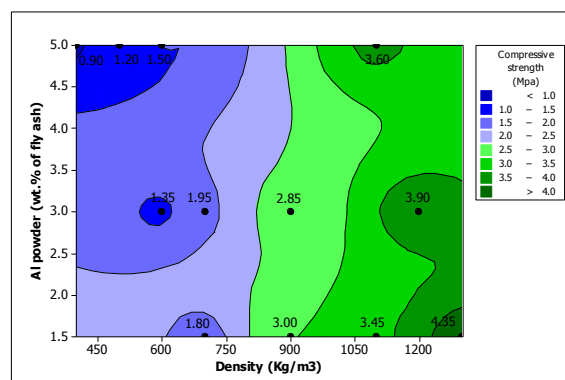
Fig. 7. Relationship between compressive strength and density of aerated geopolymer

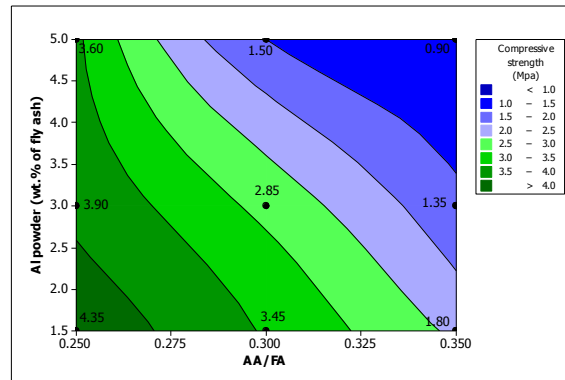pastes at different percentages of Al powder
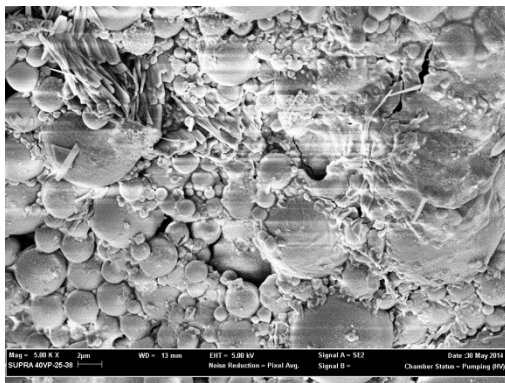
23
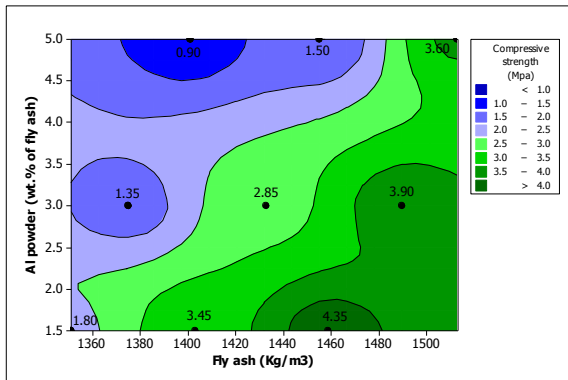
(a)
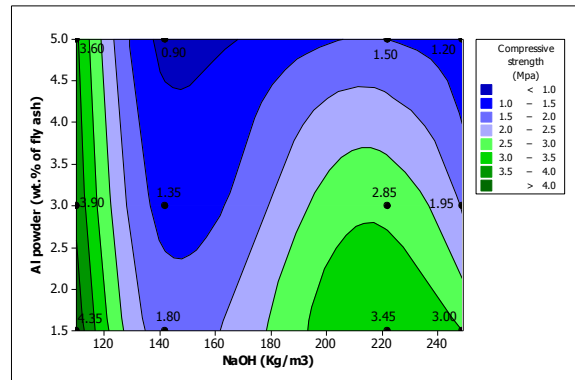
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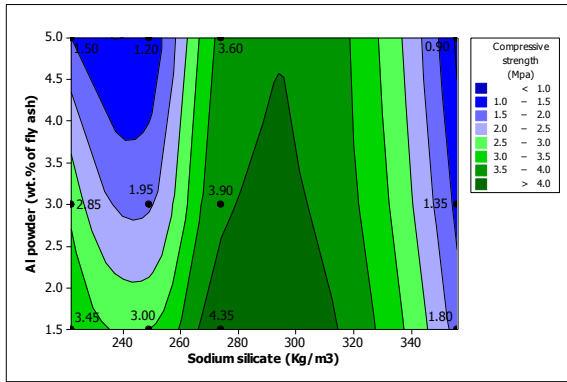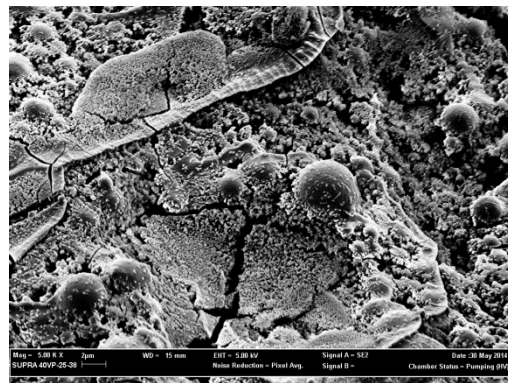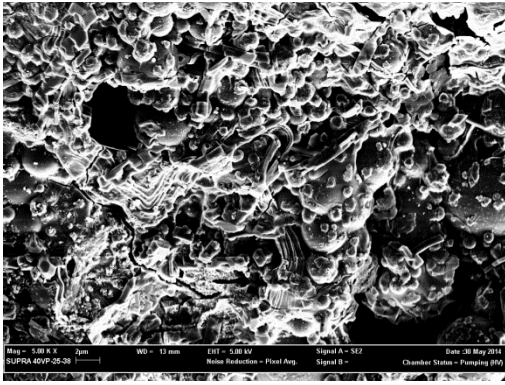
(c)
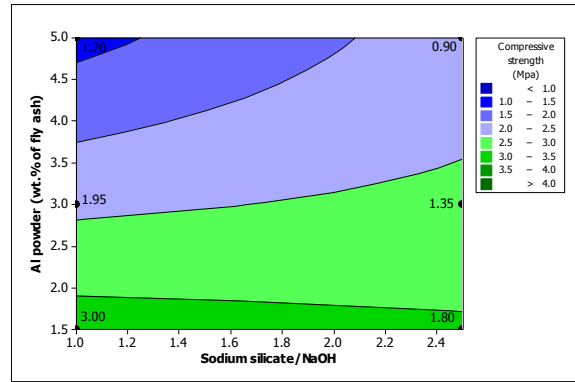
(d)

(e)

Fig. 8. Effects of different parameters on compressive strength of aerated geopolymer
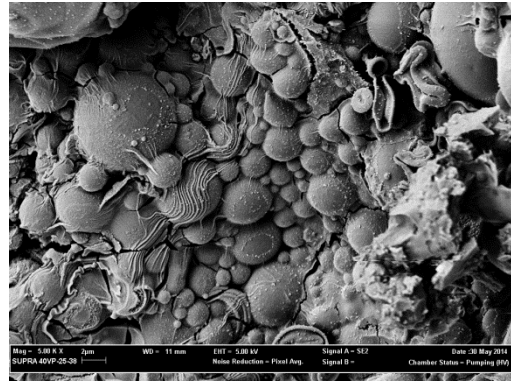
pastes

(a)

(b)

(c)

(d)

(e)

Fig. 9. SEM micrographs of aerated geopolymer samples; a) Sodium silicate/NaOH solution = 2.5, AA/FA = 0.35, wt.% of Al powder = 5.0, b) Sodium silicate/NaOH solution = 2.5, AA/FA = 0.35, wt.% of Al powder = 3.0, c) Sodium silicate/NaOH solution = 1, AA/FA = 0.3, wt.% of Al powder = 5.0, d) Sodium silicate/NaOH solution = 1, AA/FA = 0.3, wt.% of Al powder = 1.5 and e) Sodium silicate/NaOH solution = 2.5, AA/FA = 0.25, wt.% of Al powder = 5.0.