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The potential use of geopolymer containing fly ash produced in heavy fuel power plants for toxic metals fixation: Chemical and physical characteristics

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ABSTRACT

In Jordan, large amounts of fly ash (FA) are produced from heavy fuel composition in power stations. The produced fly ash is highly contaminated with heavy metals including V, Ni, Zn and Fe. Geopolymer containing different amounts of FA was fabricated for sake of minimising the mobility of toxic elements in the repository site. Using local kaolinite, fly ash and NaOH mixture, five geopolymers were prepared at room temperature and cured at 80°C at the following ratios: Si/Al: 1.68–4.71, Na_2O/Al_2O_3: 1.48–1.84, Na/Al: 0.74–0.92. The maximum compressive strength was observed for the geopolymer containing no fly ash, 28 Mpa at day 1 and dry sample. A geopolymer containing 10% FA showed promising results where a compressive strength of 23 Mpa was reported. The porosity of FA has been partially destroyed after polymerisation process as indicated from SEM pictures and XRD analysis; new phases have been created under the action of applied pressure. The leachability test indicated that the samples of higher FA are more leachable. The results proved that involving FA in geopolymerisation was significantly decreased the mobility of the toxic metals like Ni and V. Therefore, the prepared geopolymers will definitely have important industrial applications.

KEYWORDS

Geopolymers;
Fly ash;
Heavy Fuel;
Vanadium;
Nickel;
Immobilisation.

INTRODUCTION

The power generation industry that based on burning petroleum fuels (like diesel) produces a solid residue which is known as fly ash (FA)[1]. In Jordan, the total consumed power during 2009 was approximately 3.44 million toes (tone oil equivalent). This amount represents 45% of the total power consumption in our Kingdom. In 2009, the total amounts of consumed fuel in power stations were 261 thousand toes (as heavy fuel oil) and

18 thousand toes (as diesel) according to our annual reports[2]. The large consumption of fuel in power stations leaves 418.5 ton FA[2] and this number is expected to increase dramatically in the coming years due to the natural growth rate of energy demand and shortage of natural gas supplying. Having said that, it is important to mention that the biggest electric power station in Jordan (Aqaba thermal power station) was started to use heavy fuel instead of natural gas since March of 2011 and this will end up of large amounts of FA. In terms of resource recovery and environmental impact, FA is attracting much attention due to disposing problems and its harmful impact on the soil because of metals leaching[3, 4]. In fact, the previous researches on this topic is limited and indicated that FA produced in power stations is rich in C, Ni, V, Fe, Mo, and Mg and among these metals Ni and V are the most valuable[3, 4].

Depending on the nature of FA, there are number of inertisation processes to reduce the harmful influences of toxic fly ash on the environments and these including immobilisation with construction materials[5], chemical washings[6], extraction[7], incineration[8], stabilisation/ solidification technology[9] and recently geopolymerisation[8-11]. It seems that geopolymerisation is an attractive process where the FA would be entrapped within the hard and rigid silica-alumina polymers matrix[11].

The term “geopolymer” was proposed by Davidovits and it describes a cementitious binder formed by alkali activation of aluminosilicate powder which does not require the presence of cement material[12]. Generally, the inexpensive geopolymers are simply prepared from a low-cost aluminosilicate source with large amounts of SiO_2 and Al_2O_3 (80 %wt). Structurally, they have rigid 3D space containing repeating units of $(Si–O–Al–O–)_n$ and $(Si–O–Al–Si–O–)_n$ which is similar to common aluminosilicate glass[12]. The main advantage of this technology is that polymerisation occurs at moderate temperatures (40–90°C). The polymerisation reaction occurs between an alkaline solution, the chemical activator, and an aluminosilicate binder. For low cost applications, sodium is preferred than potassium or lithium hydroxide solutions. During initial mixing, the alkaline solution dissolves silicon and aluminium ions from the aluminosilicate source where the neighbouring silicon or aluminium hydroxide molecules undergo a condensation reaction to form a new Si–O–Si or Si–O–Al bond with loss of water. The final structural network consists of amorphous to semicrystalline 3D silico-aluminate structures with SiO_4 and Al_2O_3 tetrahedral linked alternatively by sharing all the oxygens. Probably, fly ash obtained from coal combustion is most applied source for geopolymers and this is attributed the high content of Si/Al in its matrix[11]. Natural kaolinite, calcined clay, mining wastes, and slag are also as a good source of aluminosilicate precursor[13]. Through polymerisation process, clay minerals are hardened and transformed into rigid geopolymers at low temperatures with minimal energy consumption[13, 14]. In the earlier process, the aluminium silicate of clay mineral (like kaolinite) reacts with alkali at low temperatures and polymerise into three-dimensional mineral phases hydroxysodalite[14]. In a similar study, Yousef and co-worker were prepared a rigid and chemically stable of good adsorption properties geopolymers from local kaolinite/sand/NaOH[10]. Jaarsveld and co-worker were prepared a stable geopolymer by mixing coal-fly ash within kaolinite in the presence of Na-silicate material[15].

Application of geopolymerisation technology for sake of trapping toxic and harmful fly ash is a new type of research[8-9]. The published results indicated that adding fly ash that generated from municipal waste water into polymerisation reaction end up with a stable geopolymer which resist metals leaching and have a very stable structure with compressive strength around 7 Mpa[8].

In Jordan, there are many area rich in clay mineral; especially kaolinite which is widely distributed in El-Hiswa and Batn El-Ghoul areas[16-17]. The utilisation of local Jordanian natural minerals including zeolitic phases, Na-phillipsite, hydroxyl sodalite and natrolite is reported[10, 13, 14]. In this work, fabrication of four geopolymers based on kaolinite, sand and NaOH, however, in the presence of fly ash produced from diesel combustion will be outlined herein. The aim of this encapsulation is to a) minimise the toxicity of metals in FA by slowing their leachability, and b) prepare geopolymers for other possible applications. Number of physical and chemical tests will be carried out to assess the textural properties of the fabricated geopolymers.

MATERIALS AND TECHNIQUES

Materials

Jordanian kaolinitic clay (as a source of aluminium silicate) with a purity of 60% obtained from El-Hiswa deposit was used. The kaolinitic clay deposits are located in the south of Jordan about 45km to the east of Al-Quweira town[18]. For kaolinite conditioning, the initial sample was crushed, grinded, and sieved to obtain a grain size less than 425µm. The sample was washed with water and dried at 105°C for 24 hrs. The final sample was homogenised before use. The plasticity limit of the employed kaolinite was 22% according to the international testing standards[19]. For better mechanical strength and Si source, local sand (>99% quartz) was used. Before use, the sand was washed with distilled water and sieved to obtain a grain size range 100-400µm. NaOH pellets of high purity were used to start geopolymerisation process. All reactions were carried out in distilled water and the optimum water content was pre-selected to get the proper plasticity limit[10]. The optimum curing time at 80°C was 24 hours[10, 13]. The perfect ratios of the mixture were adjusted in which a high compressive strength is produced. The conditions of prepared geopolymers are summarised in TABLE 1.

TABLE 1 : Conditions of geopolymerisation

Sample	Kaolinite (g)	Sand (g)	FA(g)	NaOH (g)	H_2O (g)
Geo 1	100	0	100	16	24
Geo 2	100	25	75	16	24
Geo 3	100	50	50	16	24
Geo 4	100	75	25	16	24
Geo 5	100	100	0	16	24

a. polymerisation carried out room temperature and curing at 80°C for 24 hrs.

A composite FA sample (5 kg) was collected from central electricity generating company (Hussein Thermal Power Station of Jordan, Zarqa). The FA was only sieved before use. The collected ash was very rich with many toxic metals as indicated from the chemical testing as will be shown later. As shown in TABLE 1, FA was added to the reaction mixture with different amounts (0-40%). Adding FA to reaction mixture will interfere with geopolymer matrix and will affect the final properties of the product.

Preparation of geopolymers

Five geopolymers were prepared using Kaolinite-Sand-NaOH-FA combination as clarified in TABLE 1. Silica sand was used as a filler to provide high mechanical properties of the final geopolymer and a secondary source for Si. The alkaline NaOH-solution was used for initial dissolution of oxides prior to polymerisation process. FA was added in different amounts into reaction mixture to replace sand while the amount of kaolinite was maintained constant (TABLE 1). Reaction mixtures were homogenised using a controlled speed mixer. Mixing speed was 10 rpm for the first 2 min and increased to 200 rpm over the next 10 min. The produced geopolymers were compared against a reference one containing no FA (sample Geo 5 in TABLE 1). Good mixing was necessary to obtain homogeneous mixtures and to avoid the agglomeration of the mixtures. Each mixture (series) was divided into different specimens of 50 g. The mixture was moulded immediately after weighing to avoid drying and to decrease the workability of the mixture. The paste was moulded in a stainless steel cylinder (dimensions 25mmx45mm) at an external pressure of 15 MPa using Carver Hydraulic Laboratory Press. The moulded specimens of each mixture were cured by heating at 80°C for 24 h. After curing, the samples were cooled down at room temperature and subjected to physical and chemical testing. The applied tests include XRD, compressive strength and leachability test. The compressive strength of geopolymers was carried out using CONTROLS testing machine (Model T106

Figure 1 : Three specimens of geopolymer number 4 (Geo 4).

modified to suit with standard testing) where the load was applied on a surface area (12.5×12.5×π) mm^2 and height 50 mm, and increased by a displacement rate of 2 mm/min. The physical properties of geopolymers were measured using standard protocols[13, 19]. Figure 1 depicts three specimens of geopolymers number 4 (Geo 4) and this sample is one of the best samples as will be shown later.

Spectroscopic techniques

The prepared geopolymers were tested for their electrical conductivity, compressive strength and density. Testing was carried out on three different specimens of the geopolymer and an average value is provided. The electrical conductivity is measured as a function with time using WTW-pH meter. The scanning electron microscope/ energy-dispersive X-ray spectroscopy (SEM/EDX) techniques (FEI- INSPECT-F50 of SEM/ EDX, Netherlands) were applied to study the chemical, mineralogical and textural details of the geopolymers. X-ray diffraction (XRD) analysis was applied to identify the crystalline phases of the prepared geopolymers. Representative portions of geopolymers were X-rayed using Philips 2KW model, Cu Kα radiation ($\lambda = 1.5418$ Å nm) with a scan rate of 2°/min and the X-ray diffractograms were provided for assessment.

Metals leachability

The total metal contents in FA or geopolymers were determined after leaching using inductively coupled plasma atomic emission spectroscopy (ICP OES, model Alan 5600, PerklinElmer SCIEX, USA) or by Inductively Coupled Plasma - Mass Spectrometry (ICP MS model Alan DRC-e 9000, PerklinElmer SCIEX, USA) depending on the metal level in the extract. FA or geopolymer was mixed with 0.01 M $Ca(NO_3)_2.4H_2O$ (1:10) and the final mixture was agitated using horizontal shaker for 24 hrs. Before analysis, a sample of the mixture was centrifuged for 5 min at 2500 g and then filtered using filter paper and was stored in plastic bottles[20].

RESULTS AND DISCUSSION

Geopolymers are an old technology that has recently received attention in various forms one of which is waste stabilisation. They function similarly to cement binders in terms of encapsulation however have improved chemical and physical properties, such as acid resistance, compressive strength, durability and thermal resistance[11].

Fly ash

Chemical analysis revealed that FA obtained from the fuel is a toxic matrix where large levels of metals were detected: V (3.7%), Ni (0.8%), Mo (105 ppm), and Mg (9.5%). It is important to mention that this large level of metals was obtained using concentrated acidic medium and much lower amounts are obtained when using softer eluent (TABLE 4). Having said that, this material is dangerous and should not be directly exposed to the environment. In this work, inertisation of FA will be carried out using geopolymer technology. In fact, the FA would be industrially useful material for obtaining V and Ni and this is under investigation in our laboratory.

Characterisation of geopolymers containing FA

As mentioned earlier, the main aim of this study is reduce the potential dangerous effect of FA by binding it in a suitable geopolymer. If this achieved, the obtained material would have very low leachability and the final geopolymer will find many industrial applications[10].

The general features of FA are viewed at different magnification powers (5000x and 12000x) for more assessment (Figure 2). At 5000x magnification, the picture (Figure 3A) indicates the porous structure of the FA that created that the high temperatures upon fuel burning. Carbonaceous large particle of diameter more than 30 μm is shown in Figure 3A, Figure 3B which taken at higher magnification power indicated the presence of needle-like structure of average length 10 μm which may back to metal oxides of Ni and V that created at high temperatures. In general, both Figure 3A and 3B indicated that carbonaceous nature of FA. AS expected, the EDX spectrum of FA proved the presence of C, V, Na, Mg, S, and O. The high intensity of C atom is reflected the carbonaceous nature of FA.

Accordingly, FA is not a good starter for geopolymer where both Si and Al atoms are absent; however, this porous material contains large fractions of toxic heavy metals which upon leaching can harm the environment.

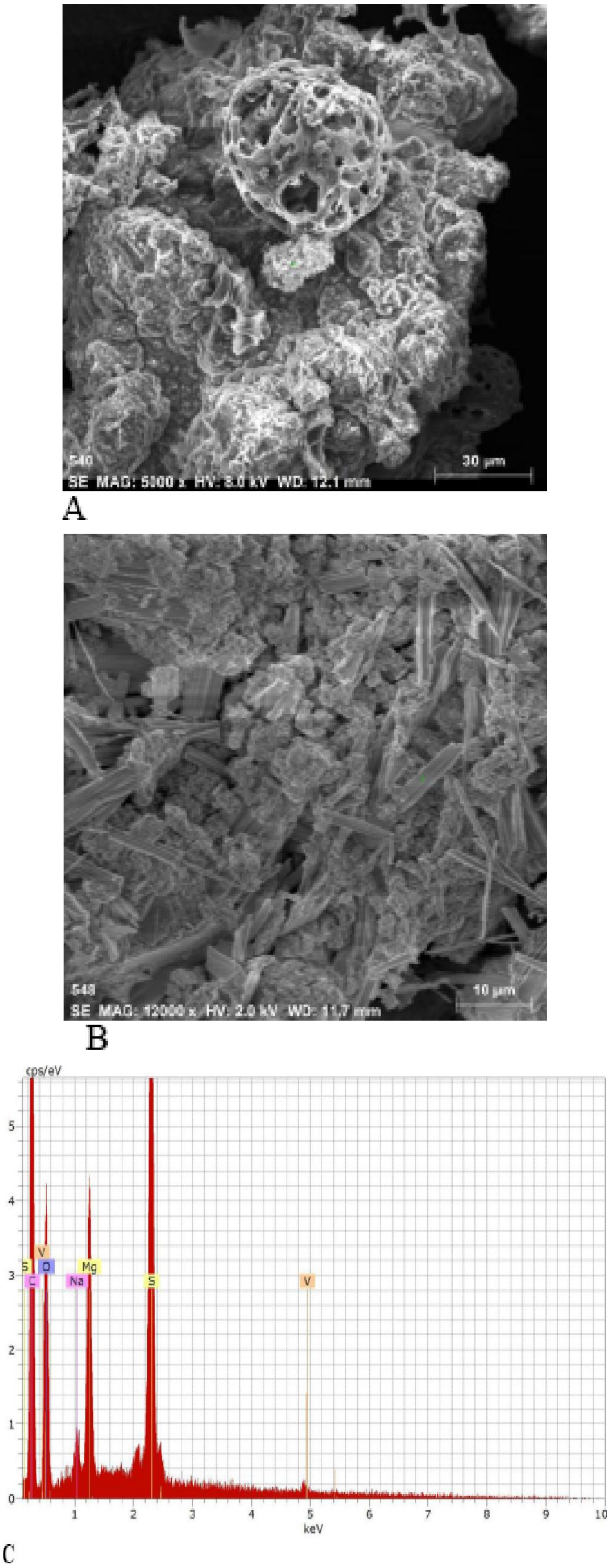

Figure 2 : SEM of FA magnified at 5000x (A) and 12000x (B) and EDX of FA (C).

As will be shown soon, Geo 4 which containing 10% FA exhibited good mechanical properties and poor metals leachability so it was reasonable to view its textural properties (Figure 3).

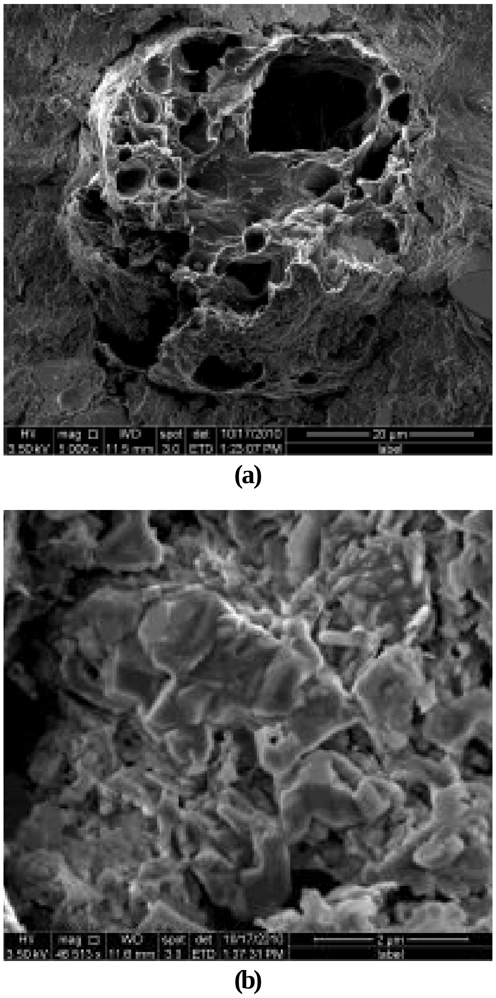

Figure 3 : SEM microphotographs showing Geo4 containing 10% FA at 5000x (A) and at 46500x (B) magnification powers.

As shown in Figure 3A, the geopolymer matrix contain FA that do not chemically involved in polymerization process and this is expected because the ash has no Si/Al in its structure. The porous structure of Geo 4 will give this material more importance in filtration systems. Figure 3B shows the crystalline nature of the prepared geopolymer.

Dissolution or creation of new phases upon polymerization could be studied using XRD technique. Again, the XRD scans of Geo4 received more attention and the results are shown in Figure 4. Figure 4 illustrates that most of the crystalline phases in FA have been disappeared upon geopolymerisation, possibly dissolved in the alkaline solution, and their components were physically attached with the geopolymerised rigid matrix. For example, FA showed characteristics XRD peaks with good intensity with the range (2θ: 28-33), after making the geopolymer, those XRD peaks have been disappeared as viewed in Figure 4.

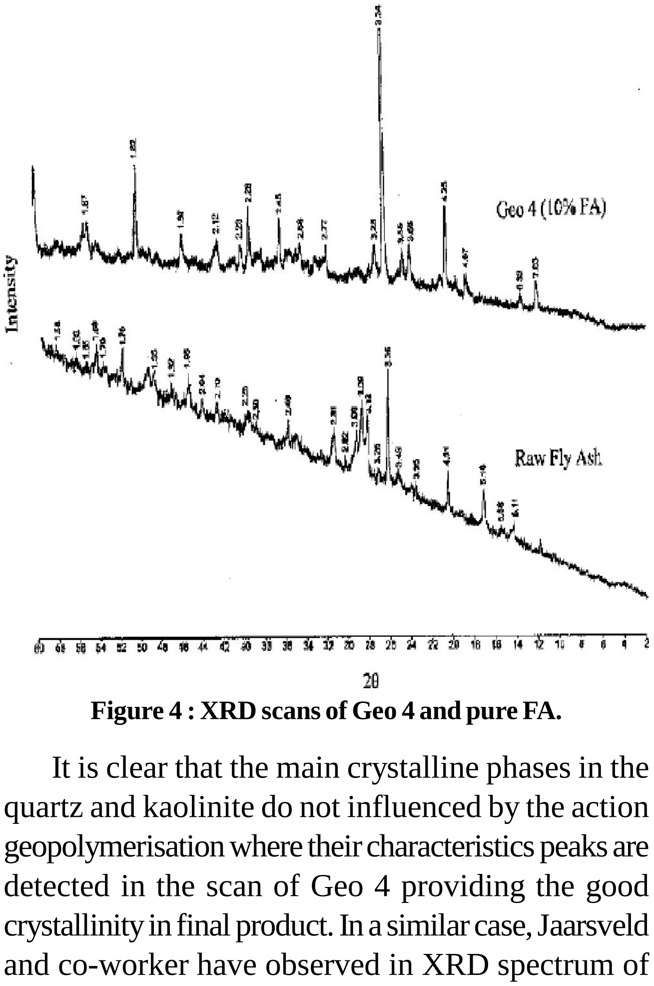

Figure 4 : XRD scans of Geo 4 and pure FA.

It is clear that the main crystalline phases in the quartz and kaolinite do not influenced by the action geopolymerisation where their characteristics peaks are detected in the scan of Geo 4 providing the good crystallinity in final product. In a similar case, Jaarsveld and co-worker have observed in XRD spectrum of kaolinite-based geopolymer some of characteristics peaks of kaolinite and concluded that kaolinite has partially involved in geopolymerization process[15]. Bankowski and co-workers have prepared many geopolymers from coal-fly ash which has a crystalline structure (not like the ash used in our study) and the authors showed that the XRD peaks of raw fly ash do not greatly changed even after geopolymerisation which reflect that the ash was partially involved in the process[21]. A note point to mention, the rigid nature of prepared geopolymers has trapped the particles of FA and this will prevent (or lower) metals leaching as will be shown next.

Physical and chemical testing of the prepared geopolymers

The final influence of adding FA into geopolymer mixtures could be evaluated via studying the chemical and physical properties of geopolymers and this is a general trend in this area (10-11, 15, 21). Before discussing the physical and chemical results, is it important to calculate the following ratios of Si/Al, Na/Al, and H_2O/Al because they would affect the property of the final geopolymers. TABLE 2 summarizes the molar ratios of atoms in the geopolymers.

TABLE 2 : Molar rations of Si, Al, Na, H_2O in the geopolymers

Geopolymer[a]	Si/Al ratio	Na/Al ratio[b]	H_2O/Na ratio[b]	SiO_2/ Al_2O_3[c]	Na_2O/ Al_2O_3[c]
Geo 1	1.68	0.92	2.65	1.62	1.84
Geo 2	2.37	0.87	2.78	2.39	1.74
Geo 3	3.10	0.83	2.93	3.28	1.65
Geo 4	3.93	0.79	3.09	3.93	1.57
Geo 5	4.71	0.74	3.27	4.70	1.48

a. See Table 1 for polymerisation conditions; b. The mass ratio of Na in FA is 2.2%; c. Average value for three different portions.

The XRD-chemical analysis revealed that kaolinite (the main principal source for geopolymers) contains (%wt): SiO_2 52.26, Al_2O_3: 27.40, Fe_2O_3: 6.22, Na_2O: 0.20, K_2O: 1.49, and LOI: 10.44. As indicated from chemical analysis, kaolinite is a good starting material from producing geopolymers (beside silica sand) where the total content of SiO_2 and Al_2O_3 is 80% which necessary for producing organized 3D geopolymer structure[9]. Si/Al ratio, in fact, is the most important parameter that should be adjusted to get a hard geopolymer and the perfect ratio as recommenced in literature is 3:1[9]. Accordingly, geopolymers 3 and 4 are expected to be the strongest polymers where the Si/Al ratios are 3.1 and 3.93, respectively.

On the other hand, Rowles and O'Connor have recommended that Si/Al ratio should be within the range 1.5-2.5 to end up with a high compressive strength for the geopolymer[22]. For better results, the perfect Na/Al ratio should be within 1.0-1.29 and this was almost achieved in Geo 1 & 2 where the reported Na/Al is closed to 1.0. Finally, H_2O/Na ratio was not optimum for all geopolymers according the values reported in literature[9]. Undoubtedly, the compressive strength is the most targeted property in the geopolymers and the obtained results are depicted in TABLE 3. For more assessment, the compressibility test was carried out on dry and wet specimens. The wet samples were prepared before testing according to the ASTM standards of concrete[23].

TABLE 3 : Compressive strength (in Mpa) for FA-containing-geopolymers

Geopolymer-Dry	FA content (%)	Compressive Strength (Mpa)[a]		
		Day 1	Day 7	Day 28
Geo 1	0	28	29	33
Geo 2	10	23	22	9
Geo 3	21	8	7	10
Geo 4	31	7	6	10
Geo 5	42	3	3	4
Geopolymer-Wet				
Geo 1	0	15	18	20
Geo 2	10	13	11	3
Geo 3	21	4	2	3
Geo 4	31	3	1	3
Geo 5	42	2	-	-

a. The reported values were averaged of three tests on three specimens (RSD<10%).

The following conclusions would be obtained from TABLE 3: a) compressive strength increased with time for both dry and wet systems. The test was carried out on different days for Geo 1 because it has the maximum strength. It is expected that compressive strength will decreases after wetting because many ions will leach and this will reduce the overall strength, b) as the content of FA increase, the compressive strength decrease to become less than 5 MPa indicating a weakness in the geopolymer infrastructure and FA do not chemically interact with geopolymer matrix. The highest compressive strength of the prepared geopolymers was 23 Mpa (13 Mpa for wet sample) and reported for Geo 2 which contains 10% FA, c) It seems that FA has replaced the strong quartz matrix and this reflected badly on the compressive strength of the final geopolymer. However, in this case Geo 2 is still having a high mechanical strength and the most important point it trapped toxic FA. Beside it high mechanical strength, quartz gains ay involved in polymerization reaction and form stable chemical bonds with Al atom of kaolinite and this is possible due to the high compressive strength of Geo 1, and d) A proper explanation for the large variations in compressive strength of geopolymers is attributed to the Si/Al and H_2O/Na ratios. Geo 1 has a perfect Si/Al ration (1.68, TABLE 2) to get high compressibility according to Rowles and O'Connor[22], on the other hand, this ratio is not optimum in Geo 5 while the amount of Si is 5 times higher than Al. As reported in many studies, higher strength was observed when ratios SiO_2/Al_2O_3 and Na_2O/Al_2O_3 were 3.0-3.8 and 1.0, respectively[24-27]. From TABLE 2, SiO_2/Al_2O_3 ratios of most samples (except Geo 3) do not fall in the typical 3-3.8 range and Na_2O/Al_2O_3 ratio of all geopolymers is higher than 1.0. Finally, the reported compressive strength values for dry Geo 1 and Geo 2 samples are high and unusual if compared to other reported values. Galiano and co-worker reported only 7 Mpa for their geopolymers[8] and using kaolinite – based geopolymers Jaarsveld and co-workers reported 11.4 Mpa as compressive strength[15]. On the other hand, a compressive strength of 100 Mpa for metakaolin-geopolymer (Si/Al ratio 1.9) was reported by Duxson and co-worker[26]. However, Stevenson and Sagoe-Crentsil reported compressive strengths close to these reported in our study using metakaolin and sodium silicate solution[27].

Electrical conductivity and toxic-metals leaching

Electrical conductivity and leachability tests, in fact,

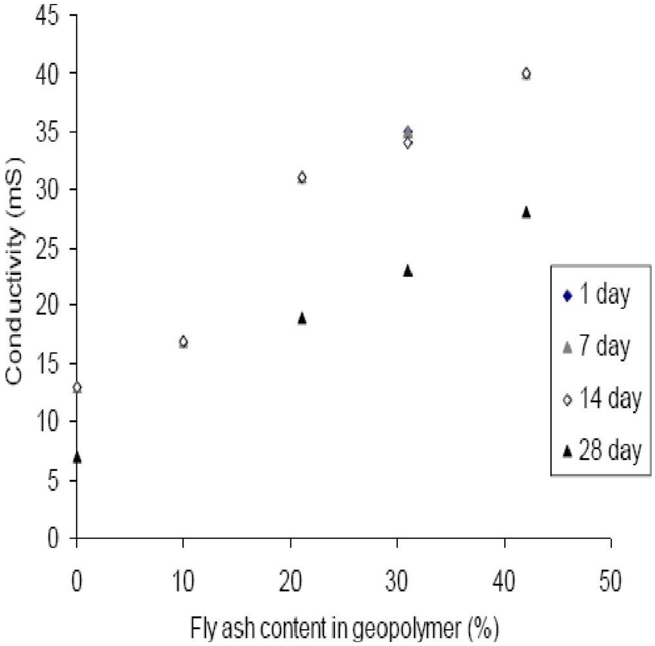

Figure 5 : Electrical conductivity of geopolymers over different time intervals.

are the most adopted tests for evaluating the chemical stability of the prepared geopolymers. Electrical conductivity test will give an idea about the amount of leached ions of high ionic mobility including H^+, OH^-, Cl^-, Na^+, and K^+, higher conductivity values indicates high leaching from geopolymer matrix. The leachability of encapsulated geopolymers is illustrated Figure 5.

It is important to mention that the leaching behaviour of fly ash alone is much higher than any of the prepared geopolymers (Geo 1 – Geo 5) and this is attributed to the immobilization of ions by the action alkali activator. According to Van Jaarsveld et al. (1998) this effect is expected to take place on an inter-tetrahedral scale. The possible explanation is that the pore openings were changed and diffusion of the metal to the leaching solution was prohibited. As shown in Figure 5, the conductivity increase by increasing the amount of FA in geopolymer, Geo 1 (which has no FA) has the poorest conductivity 12 mS. The maximum conductivity was 35 mS and observed for the geopolymer containing 42% FA. The elution of ions would be attributed to the poor geopolymeric structure, caused by presence of ions which do not participate in the formation of the aluminosilicate network. A similar observation was reported in literature[9]. TABLE 4 reveals that amount of leached heavy meals and Mg using 0.01 *M* $Ca(NO_3)_2$ solution at 35°C.

TABLE 4 : Leached metals FA and geopolymers by 0.01 M Ca(NO3)2 at 35°C.

Matrix	Mg (ppm)	Mo (ppm)	Ni (ppm)	V (ppm)
Flay Ash	55700	70.0	3700	16000
Geo 1	1887	9.5	35.9	--
Geo 2	50.77	4.74	35.12	729.0
Geo 3	--	12.73	36.83	408.2
Geo 4	49.48	5.545	35.31	755
Geo 5	28.48	6.29	35.91	1147

TABLE 4 indicated that large amounts of FA metals were interfering with the geopolymer matrix and this slow down their leachability to solution. Relatively speaking, the metals do not eluted with the same magnitude and for example Mg seems to be incorporated within polymer matrix because even at FA dose the eluted amount was very low. For V, as the content of FA increased, large amounts of V was observed in solution (Geo 5) and this indicated that V ions do not strongly involved in geopolymer building.

CONCLUSIONS

In the present study, encapsulating toxic fly ash in a suitable geopolymer is a good protocol for slow down metals leaching and production of composite materials of high mechanical strength. For better outputs, the geopolymer should not contain more than 10% of fly ash to avoid losing compressibility. Leaching of toxic Mo, Ni and Mg from the geopolymers is rather significant when compared to leaching in raw FA. As a recommendation, further research is needed on the application of FA-geopolymers in constructions and buildings. Moreover, long-term studies should investigate the longevity of the metal-immobilizing effect of the FA-geopolymers.

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