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Characteristics of Coal Fly Ash and Bottom Ash-based Geopolymers in Unfavorable Environment

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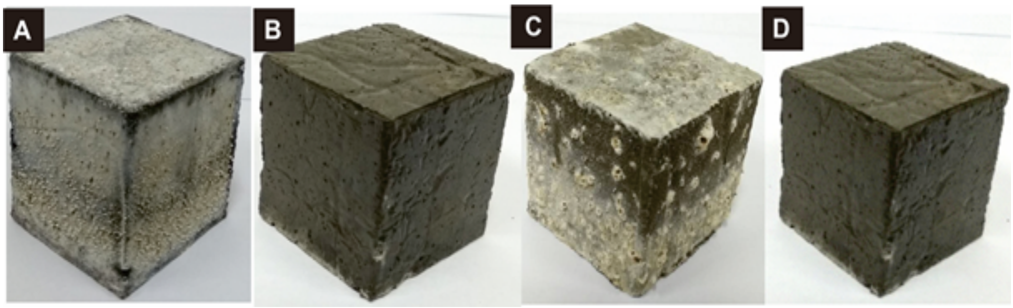
Abstract *Coal is considered a heavy polluter, a source of carbon and greenhouse gas emissions, which is a major cause of climate change. However, the energy crisis left European countries with no choice but to restart coal-fired steam power plants (PLTU) to overcome high prices and limited gas supply, which is one of the main energy sources besides NRE. Including China, which is actually one of the leading countries in NRE development, with a total installed capacity of wind and solar plants reaching 581 gigawatts (GW). Along with the concept of green utilization technology, coal ash could utilize as concrete materials. Fly ash and bottom ash can be combined to create geopolymer concrete. Geopolymer concrete making involves a polymerization process occurring between the alkali activators in combination of sodium hydroxide and water glass with Si – Al minerals resulting three - dimensional polymeric chain. In this case, heavy metals are very important to be immobilized to increase the mechanical strength of the geopolymer. The amount of heavy metal added to the geopolymer matrix does affect its compressive strength. Experimental studies were conducted to test the resistance of geopolymer bonds to immobilize of heavy metals on geopolymer concrete. Two types of geopolymer concrete samples were used with fly ash: bottom ash ratio of 9:1 and 1:1 which is then added with NaOH solution. The sample was immersed into a solution of sulfuric acid solution pH 1, sodium hydroxide pH 12, sea water and aquadest. XRD, SEM-EDS, XRF, EDX and compressive strength tests were performed on liquid and solid samples. It was found that the 9:1 concrete composition has higher compressive strength than 1:1. The result of the compressive strength test shows that 41%; 21%; 27% and 10% reduction when it immersed in sulfuric acid, sodium hydroxide, sea water, aquadest respectively. Prior to the leaching process, the results of XRF analysis showed that the main components in the raw materials for making geopolymers were* SiO_2 *and* Al_2O_3*. After leaching, the results of XRF analysis on geopolymer solid samples after 90 days of leaching in all solutions showed that the levels of Si and Al remained constant but decreased compared to the initial levels. This research aims that geopolymer concrete can be utilized as a construction material in unfavorable environment, such as geothermal area, sea and oil rig.*

Keywords*: coal, concrete, geopolymer, heavy metal, leaching.*

Graphical Abstract



1. Introduction

In the midst of limited supply of oil, gas, and renewable energy sources, coal is again an option. Even though this black mineral is starting to be abandoned because of commitments for decarbonization and net zero emissions in the energy sector, especially by developed countries in the blue continent region. Coal is considered a heavy polluter, a source of carbon and greenhouse gas

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emissions, which is a major cause of climate change. However, the energy crisis left European countries with no choice but to restart coal-fired steam power plants (PLTU) to overcome high prices and limited gas supply, which is one of the main energy sources besides NRE. Including China, which is actually one of the leading countries in NRE development, with a total installed capacity of wind and solar plants reaching 581 gigawatts (GW). Even China has retired up to 297 gigawatts (GW) of steam power plants since 2007. Just like Europe, the production of renewable energy plants that are below expectations has made this country fall into an energy crisis and rely on steam power plants. In fact, data from Bloomberg New Energy Finance (NEF) shows that the country, led by President Xi Jinping, plans to build 247 GW of new coal-fired power plants by 2025 to meet increasing energy demand as the economy recovers.

Nowadays coal ash waste just disposed in landfill sites around the factory. Coal ash waste combustion which consist of heavy metal oxide could affects human health, which is related to the emergence of chronic and non-specific respiratory tract diseases, pneumoconiosis, and can poison human nerves, fly ash also has an impact on the health of the surrounding environment (Harrianto, 2009). If it just left and accumulate without being controlled, coal ash waste will be harmful to the environment due to toxic substances that are easily carried away by the wind and mixed in underground water (Faradilla et al., 2016). The management of Fly Ash and Bottom Ash (FABA), classified as both hazardous (B3) and non-hazardous waste under Government Regulation (PP) Number 22 of 2021 on Environmental Protection and Management, remains subject to obligations to ensure it meets established standards and technical requirements (Peraturan Pemerintah No 22 Tahun 2021).

Special treatment is needed to overcome the problem of coal ash waste. On the other hand, Intergovernmental Panel of Climate (IPCC) data shows cement making production which has been used as a concrete material produces 930 million tons/year CO_2 emissions or about 7% of total CO_2 emissions. Therefore, one of the concepts of sustainable development known as Green Construction has been widely applied to the manufacture of concrete modifications (Setyowati, 2014). Fly ash consists of fine, powdery particles that are predominantly spherical in shape, either solid or hollow. They are considered to be a ferro-alumina silicate made up of glass spheres of very small particle size ranging from 20 to 80 μm with elements Si, Al, Fe, Ca, K and Na being predominant within the matrix (Petrus et al., 2021; 2022).

Most of the research that has been done so far only uses fly ash from coal ash waste (Setyowati, 2014; Petrus et al., 2021; Fernández-Jiménez et al., 2005). Meanwhile, more problematic waste such as bottom ash also needs to be utilized as well as possible. Therefore, in this study, bottom ash was used as a filler. Fly ash is good to be used as a binder based on its main constituent materials are silica oxide (SiO_2), aluminum (Al_2O_3) and iron oxide (Fe_2O_3) (Petrus et al., 2021; 2022). While bottom ash can be used as a sand substituent (filler) in making concrete. So, fly ash and bottom ash waste can be combined to make geopolymer concrete that 100% (overall composition) does not use cement. Geopolymer concrete making involves the polymerization process which includes some chemical reactions between alkali and Si-Al minerals resulting in a three-dimensional polymeric chain and a consistent Si - O - Al - O structure (Davidovits, 2020; 1991; Nogara and Zarrouk, 2018). This polymerization process is assisted by alkaline solvents which function as activators. The alkaline solution commonly used is a combination of Na_2SiO_3, NaOH and KOH (Davidovits, 2020; 1991; Palomo et al., 1999; Nasvi et al., 2012).

The use of coal ash as geopolymer concrete has significant environmental benefits, namely: improving the strength and durability of concrete, reducing energy use, reducing the amount of ash that must be disposed of in landfills, and utilizing natural resources and toxic adsorbing materials in concrete manufacturing materials (Adelizar et al., 2020; Fatikhin et al., 2019). In addition, geopolymer concrete is resistant to sulphate and chloride attacks. Through this process, metals in fly ash can be immobilized (Petrus et al., 2019).

Each year, millions of tonnes of heavy metal containments such as fly ash are generated across the worlds. These wastes are accumulated to such an extent that 'giga-scale disposal' is becoming a common phenomenon. Immobilization treatment is an effective solution to address the potential hazards that SWCHM pose to the environment. This technology can reduce the potential migration of heavy metals by changing the physical and chemical properties of the wastes (Guo et al., 2017). Thus, studies on the immobilization of heavy metals in geopolymer are very important for testing a resources of heavy metal solidification materials (Zhang et al., 2008; Abdelaal et al., 2020). The properties of the geopolymer produced has similarities to conventional concrete in general (Davidovits, 1991). Although the microstructure, chemistry and mechanical properties of fly ash-based geopolymers have been extensively studied, less attention has been paid to the leaching behaviour (Fernández-Jiménez et al., 2005; Li et al., 2023). However, testing is needed to determine the resistance of the geopolymer properties resulting from the above polymerization bond (Fukui et al., 2009; Izidoro et al., 2012; Kuncoro and Fahmi, 2013).

In this study, testing was carried out by immersing geopolymers into alkaline solutions, acidic solutions, and seawater to determine the ability of polymerization bond in metal immobilization and provide geopolymer strength. The leaching properties have been assessed mainly on heavy metal material under unfavorable condition such ash acidic, neutral and base conditions. It could be expected that the unfavorable conditions will release the metal and reduce its strength due to leachated metals. As for the environment, the leaching behaviour of fly ash-based geopolymer binders should be addressed with a view to its application in unfavorable area condition such as geothermal area, sea, and oil rig.

The main purpose of this study was to determine the effect of adding fly ash and bottom ash to the compressive strength of geopolymer concrete. In addition, this study aims to determine the optimum conditions for the manufacture of geopolymers with fly ash and bottom ash. It is hoped that the research that has been carried out can be a reference for developing geopolymer technology from coal ash.

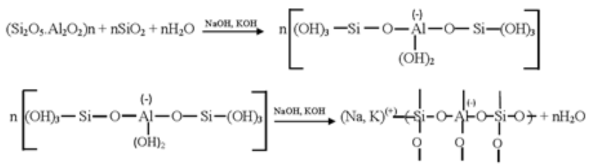

Figure 1. Reactions of geopolymerization (Singh, 2018)

2. Experimental

2.1. Preparation fly ash and bottom ash samples

Fly ash and bottom ash samples were obtained from coal-fired power plants in Tanjung jati, Jawa Tengah. Fly ash and bottom ash were crushed using a ball mill until smooth. Fly ash and bottom ash that have been smooth then were sieved to obtain a particle size of -300 mesh. Fly ash and bottom ash sample then were characterized by XRF PANalytical Epsilon 4 analysis to determine its component.

2.2. Polymerization

Sodium silicate (Na_2SiO_3) 20 mL solution and 12M NaOH solution were mixed with ash mixture with a ratio of fly ash and bottom ash of 9:1 and 1:1 (w/w). The ratio used can be said to be quite far because the researcher wants to know the effect of the ratio of fly ash and bottom ash on a larger scale. In addition, by using a large enough ratio difference, the differences in the compressive strength of the concrete from each ratio can be seen clearly. Geopolymer paste was stirred until homogeneous and put into a cube mold with a size of 5x5x5 cm^3. Geopolymer paste was then left for 28 days in room temperature. After 28 days, geopolymer samples were tested using universal testing machine to determine its initial compressive strength.

2.3. Leaching

Natrium hydroxide (NaOH) solution with pH 12, sulfuric acid (H_2SO_4) solution with pH 1, sea water from Jungwok beach and aquadest, each 250 mL were put into a 500 mL beaker glass. Each solution was analyzed using Shimadzu EDX-8000 to determine its initial composition. Each composition (9:1 and 1:1) of geopolymer samples were put inside each solution for total 90 days. During the process, 10 mL samples were taken at 1 day, 3 days, 7 days, 14 days and 90 days. Liquid samples after immersion then were analyzed using EDX to obtain leachable contents of geopolymer. Solid samples of geopolymer each ratio was tested using universal testing machine to determine its compressive strength after leaching. Solid samples were also analyzed using XRD PANalytical x'pert 3 powder and XRF PANalytical Epsilon 4 to obtain elemental and mineral components after being immersed.

2.4. Microstructure

Microstructure and distribution of major elements over the geopolymer concrete were investigated by A JSM -6510 LA Scanning Electron Microscope coupled with EDS micro-analysis. SEM-EDS allowed us to determine the quantitative composition of the geopolymer matrix.

2.5. The Compressive Test

The compressive test is one of the tests that have been carried out in this study with the aim of knowing the effect of the ratio of the ash mixture on the strength of the concrete. The compressive test has been carried out using a series of universal testing machine presses. The principle of the tool is quite simple. The tested concrete is placed in the test container. Then the concrete is pressed continuously until it causes cracks. At the same time, the compressive strength of the concrete can be seen on the pressure gauge attached to the tool.

3. Results and Discussions

3.1. Raw material analysis

The results of fly ash and bottom ash analysis using XRF are shown in Table 1. The results of XRF analysis indicate that the main components in the geopolymer manufacturing raw materials are SiO_2 and Al_2O_3. Type C fly ash is used because it contains more than 35% SiO_2 and Al_2O_3, and 20% CaO.

Table 1. Concentration of a number of elements in sample fly ash and bottom ash

Compounds	Concentration (%wt)	
	Fly Ash	Bottom Ash
SiO_2	37.6	52.1
Al_2O_3	12.5	15.7
Fe_2O_3	20.8	18
CaO	20.7	6.37
K_2O	1.97	2.64
SO_3	2.07	0.449
TiO_2	1.28	1.47
P_2O_5	1.89	2.02
NiO	0.116	0.09
V_2O_5	0.09	0.08
Other	0.983	1.081

3.2. Microstructure of geopolymer after leaching study

Surface morphology of geopolymer after being leachate in sulfuric acid, sodium hydroxide, sea water and aquadest shows in figure 2 and figure 3. From figure 2 can be concluded that 1:1 geopolymer have more crack and ruptures compare to 9:1 geopolymer. This phenomenon related to its Si-O-Al geopolymer bond which was disturbed by alkali and acid solution. This will impact the compressive strength of geopolymer concrete. In addition, fly ash is a material that is good enough to be used as a binder based on its main constituent materials. While bottom ash can be used as a sand substituent (filler) in the manufacture of concrete. Thus, the more fly ash, the stronger the bond in the concrete and this will also increase the strength of the concrete. In the surface of geopolymer that was immersed in seawater and sulfuric solutions formed some salts crystal. It's due the reactions between the minerals in concrete and acidic solutions. It is also shown in XRD results there are some mineral salts detected and affect its compressive strength.

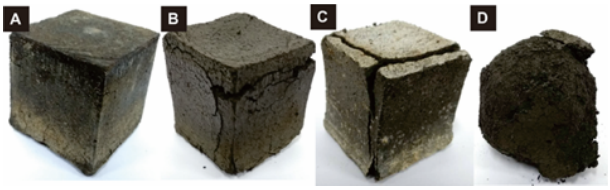

Figure 2. Concrete 1:1 after leaching in (a) seawater, (b) aquadest, (c) H_2SO_4, (d) NaOH.

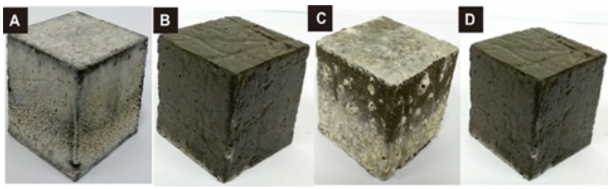

Figure 3. Concrete 9:1 after leaching in (a) seawater, (b) aquadest, (c) H_2SO_4, (d) NaOH.

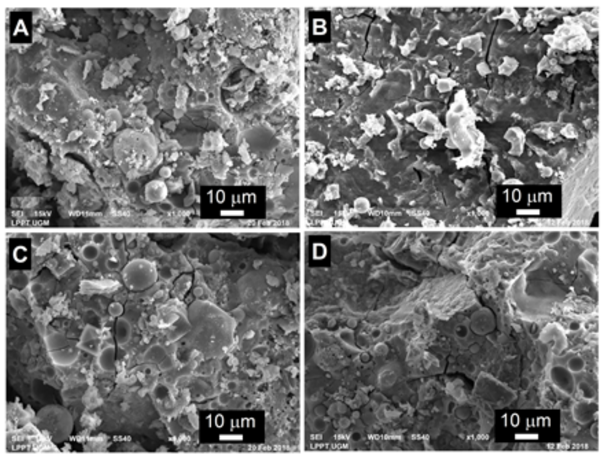

Figure 4. Scanning Electron Microscopy 1:1 result in (a)seawater, (b) aquadest, (c) H_2SO_4, (d)NaOH.

The microstructure of geopolymer after immersed 90 days in several solution is shown in Figure 4 and Figure 5. Solid surface samples after leaching were analyzed with XRF and XRD to determine it components and structures. SEM-EDS microstructure and morphology analysis shows that metal ion such ash Si, Al, Mg, Ca, Fe in 9:1 geopolymer more evenly distributed compared to the geopolymer 1:1. This shows the geopolymer 9:1 polymerization bond is stronger than geopolymer 1:1 and minerals are remained immobilized in geopolymer bond. The compressive strength of the concrete was also analyzed. Compressive strength test result shows that geopolymer immersed in sodium hydroxide solution have the smallest compressive strength reduction. The more evenly distribution in geopolymer the higher compressive strength result in geopolymer concrete.

The results of XRF analysis on geopolymer solid samples after 90 days of leaching in all solutions showed that Si and Al levels remained but decreased compared to the initial levels. This shows that geopolymer bonds are still present in concrete although not strong as before it was leachated. High concentration of Si, Al, Ca, Fe in both types of geopolymers provide more strength to the concrete. Presence of Ti, Ni, Mn components indicates that the metal is still in geopolymer concrete bonding.

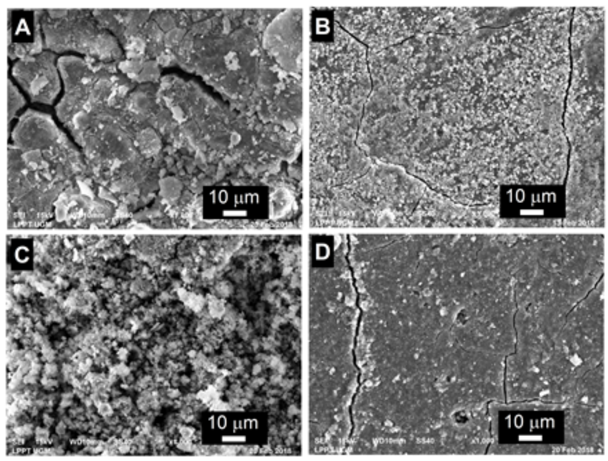

Figure 5. Scanning Electron Microscope 9:1 result in (a) seawater, (b) aquadest, (c)H_2SO_4, (d)NaOH.

Figure 8 and figure 9 show XRD results of 1:1 geopolymer and 9:1 geopolymer after immersing for 90 days. The quartz (COD# 96-153-6390) and hematite (COD# 96-152-8613) were appeared in each sample. It shows that the geopolymer bond was still in the geopolymer after being immersed. Presence of some salt crystal, such as alite ($SiCa_3O_3$, COD# 96-154-0706), aegirine (NaFeSi2O6, COD# 96-901-3275), magnesioferrite ($MgFe_2O_4$, COD# 96-101-1246) and other that was found contribute to the strength of concrete. From the figure, it indicates the presence of salt minerals from seawater that are attached to the concrete surface which causes some elements in the geopolymer concrete to be released.

Table 2. Concentration of geopolymer after leaching

Component	Concentration after leaching (ppm)							
	Aquadest		Sea Water		H_2SO_4		NaOH	
	9:1	1:1	9:1	1:1	9:1	1:1	9:1	1:1
Si	32.67	32.47	32.43	33.29	32.77	32.43	32.77	32.43
Al	11.73	10.06	11.63	8.94	11.84	10.02	11.84	10.02
Fe	30.52	32.37	30.23	32.62	30.14	32.54	30.14	32.54
Ca	13.89	11.85	14.14	12.26	13.91	12.08	13.91	12.08
K	2.73	2.57	2.79	2.64	2.77	2.49	2.77	2.49
S	0.29	1.14	0.30	0.87	0.28	0.83	0.28	0.83
Ti	1.16	1.06	1.17	1.13	1.19	1.30	1.19	1.30
Na	3.97	5.59	4.02	5.03	4.04	5.13	4.04	5.13
Ni	0.06	0.07	1.35	1.19	0.06	0.07	0.06	0.07
Mn	0.18	0.17	0.19	0.18	0.18	0.19	0.18	0.19
Others	2.80	2.65	1.75	1.85	2.79	2.92	2.79	2.92

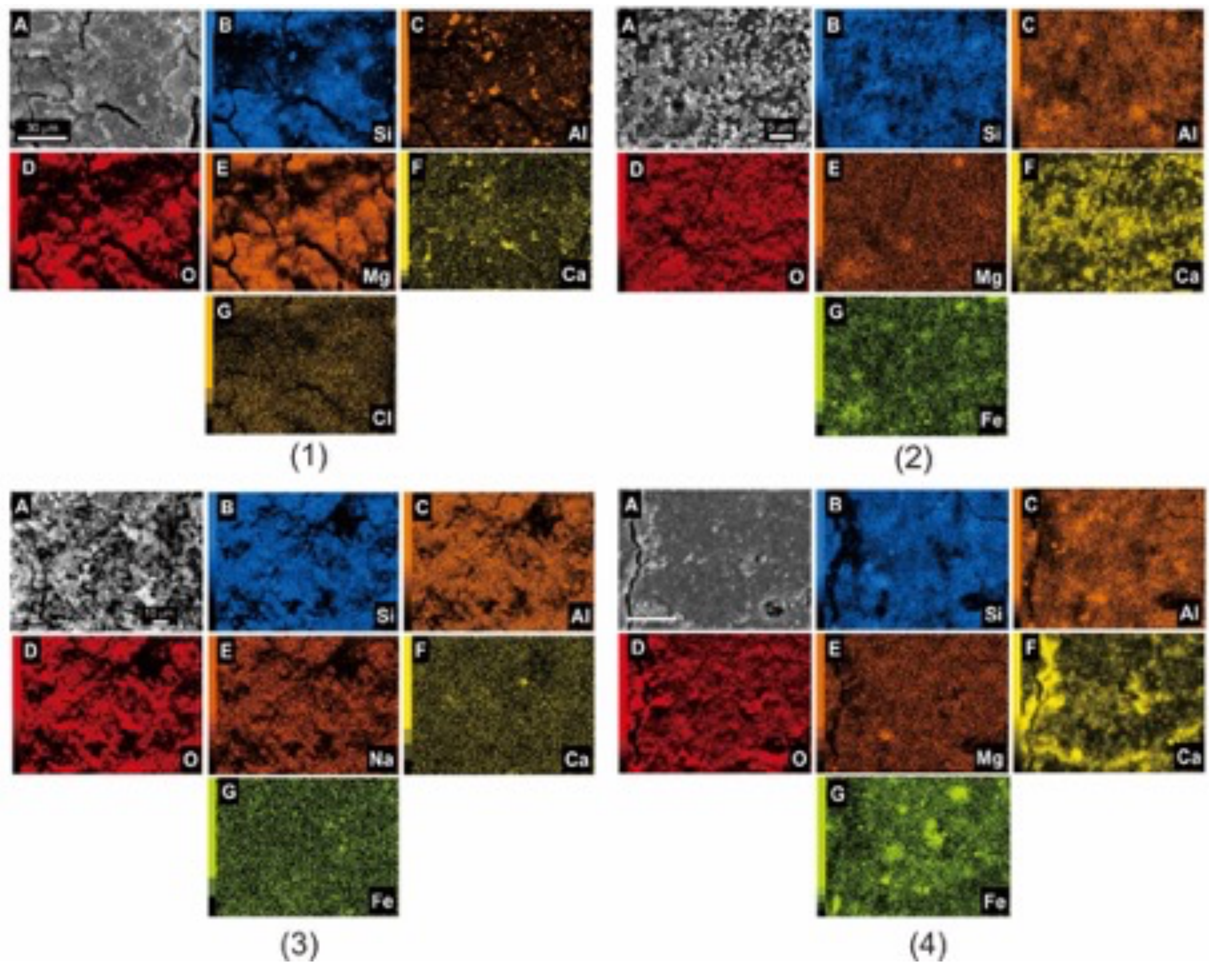

Figure 6. EDS 9:1 result in (1) seawater, (2) H_2SO_4, (3) aquadest, (4) NaOH.

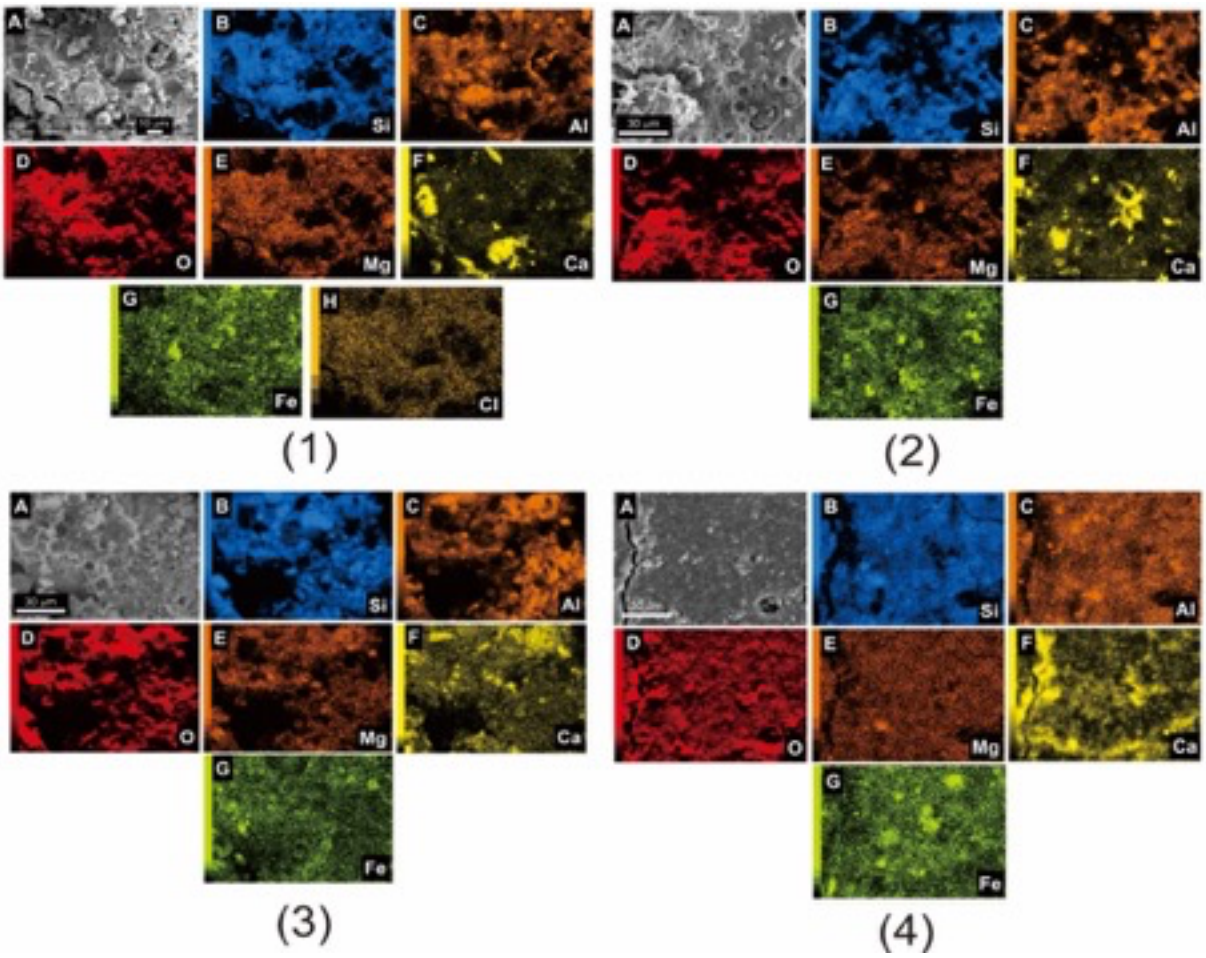

Figure 7. EDS 1:1 result in (1) H_2SO_4, (2) aquadest, (3) seawater, (4) NaOH

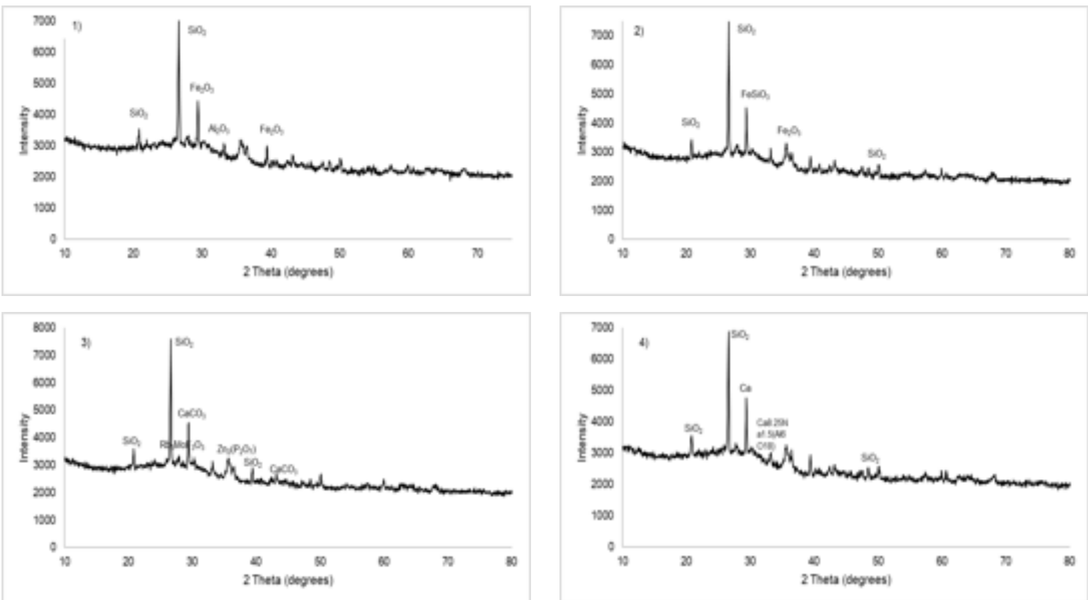

Figure 8. XRD results of 1:1 geopolymer in (1) H_2SO_4, (2) NaOH, (3) Seawater, (4) aquadest.

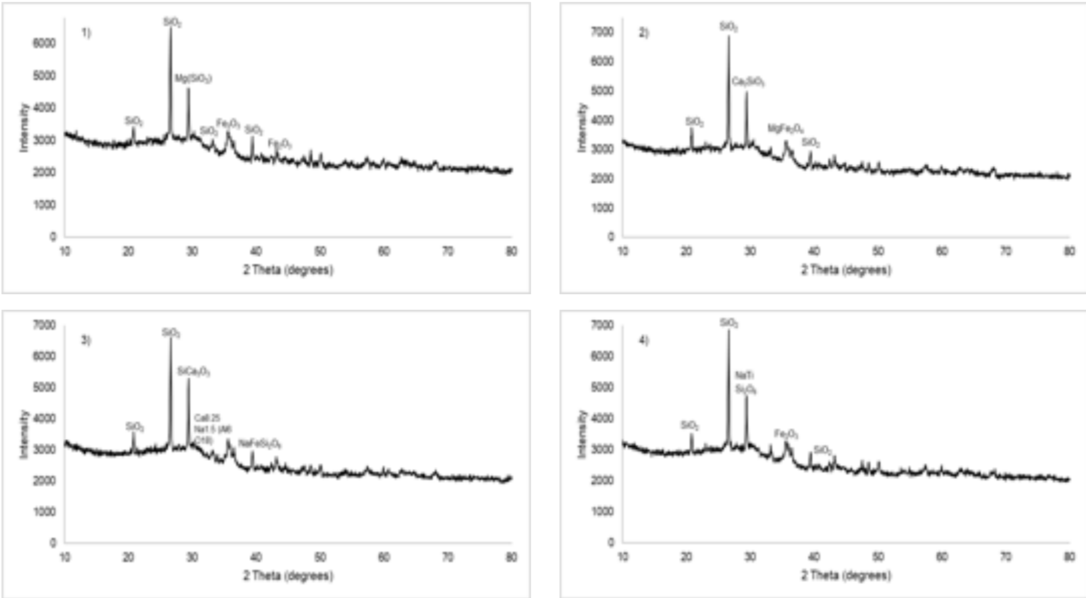

Figure 9. XRD results of 9:1 geopolymer in (1) H_2SO_4, (2) NaOH, (3) Seawater, (4) aquadest.

3.3. Leaching and compressive strength

Figure 10 shows the analysis results of geopolymers immersed in NaOH solution. From graphic, in the days 1-28 show fluctuations in the released components. The highest released components are silica, sulphur, and aluminium components. After 14 days, the concentration of the leached calcium and potassium components tends to be constant. This shows there is no components released from geopolymers because the polymerization bond could immobilize these components. On the other hand, silica, sulphur and aluminium component still increase until 90 day leaching experiment. In 1:1 geopolymer which is immersed in NaOH solution, the released component is more than 9:1 geopolymer. This affects the compressive strength result which 9:1 geopolymer have better strength than 1:1 geopolymer. It is because of silica and Aluminium components, which contributed in polymerization bonds, are more leached on 1:1 geopolymer. In addition, calcium which form calcium oxide compound still immobilized in geopolymer 9:1 and makes the concrete stronger.

Figure 11 shows the results of the elemental analysis of geopolymers immersed in sulfuric acid solutions. In 9:1 geopolymer immersed in sulfuric acid, silica, aluminium, iron, potassium, and calcium ions tend to be stable after 14 day and sulphur ion has a tendency increase. This result shows that geopolymer bonds can immobilize metals contained in fly ash and bottom ash. In 1:1 geopolymer, the concentration of each component is released more than 9:1 geopolymer. This phenomenon causes 9:1 geopolymer has higher compressive strength than 1:1 geopolymer. It's due to Si and Al components, which contributing polymerization bonds are less released.

In Figures 12 it can be seen that the main components of geopolymers that produce geopolymer bond (silica and alumina) are less release at 9:1 ratio compares to 1:1. This causes the 9:1 geopolymer concrete composition have high compressive strength due to strong polymerization bonds. Elemental analysis of geopolymers immersed in aquadest is shown in figure 13. In 9:1 geopolymer immersed in aquadest silica, aluminium, iron, potassium, and calcium ion tend to be stable after day 14 and concentration of sulphur ion still increase. In 1:1 geopolymer immersed in aquadest, the concentration of each component is released more than 9:1 geopolymer. This make 9:1 geopolymer has higher compressive strength than 1:1 geopolymer.

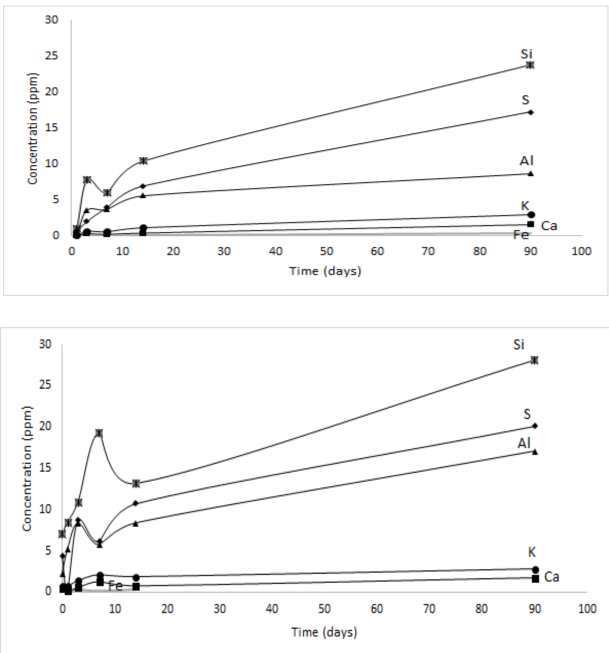

Figure 10. EDX results of (1) 9:1 geopolymer, (2) 1:1 geopolymer immersed in NaOH.

Leaching analysis found that some components such as silica, calcium, aluminium, potassium, iron and sulphur are released from geopolymers after immersed in sulfuric acid, sodium hydroxide, sea water and aquadest. It is found that 9:1 geopolymer have fewer component release compare to 1:1 geopolymer. It indicated that 9:1 geopolymer have strong polymerization bond compare to 1:1 geopolymer and causes the resulting of compressive strength test of 9:1 geopolymer have higher compressive strength. Geopolymer that immersed in sulfuric acid solution have the highest component released, due to strong sulphate ions that attacked Na-Si-O-Al geopolymer bond so the metal ions released from geopolymer bond. On the other hand, geopolymer which immersed in alkali solution sodium hydroxide has the lowest component released, due to natrium and hydroxide ion could contribute in Na-Si-O-Al geopolymer bond. Another phenomenon is heavy metals such as Titanium and Vanadium are not detected in leachate solution, it can be concluded that geopolymer bond could bind the metal and prevent it from release to the environment.

The compressive strength is a very important parameter in determining the best conditions in the study. To calculate the compressive strength of the data obtained from the tools used, the following equation is used.

$$\sigma_c = \frac{F}{A} = \frac{F}{s\,l} \qquad (1)$$

where, σ_c is compressive strength [kg/cm^2], F is compressive force received by concrete (kg.cm/s^2), A is cross-sectional area of compressive force receiver (cm^2), and s and l are base length and width (cm). The result of compressive strength analysis was shows in Figure 13. It can be seen that the geopolymer 9:1 immersed in sulfuric acid, sodium hydroxide, sea water, and aquadest, decreased by 41%; 21%; 27% and 10% respectively. The compressive strength test was not carried out on 1:1 geopolymer because after 90 days of leaching, concrete form was not cube anymore, so that compressive strength could not be determined accurately. Geopolymer which was immersed in aquadest had greatest compressive strength than other because the metal released in aquadest was smallest. The higher metal ion that released, the lower the compressive strength. It's due metal that provides compressive strength in the polymerization bond has been released (Škvára et al., 2005; Ukwattage et al., 2013; Zhuang et al., 2016).

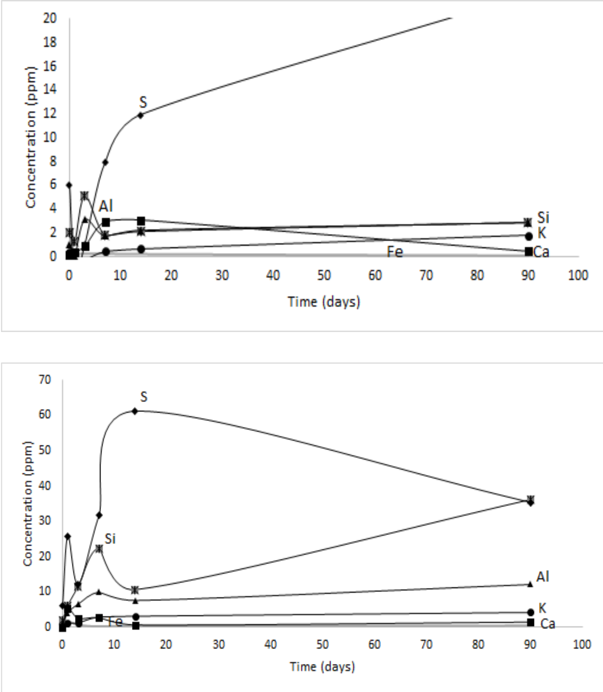

Figure 11. EDX results of (1) 9:1 geopolymer, (2) 1:1 geopolymer immersed in sulfuric acid.

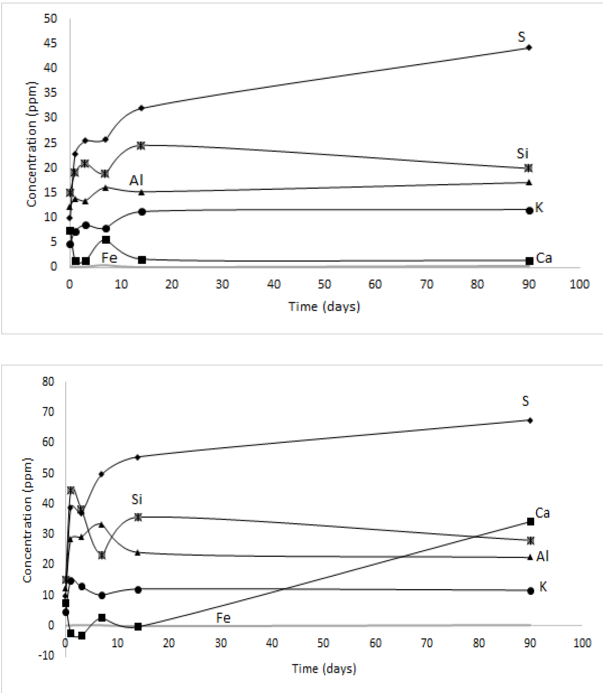

Figure 12. EDX results of (1) 9:1 geopolymer, (2) 1:1 geopolymer immersed in seawater

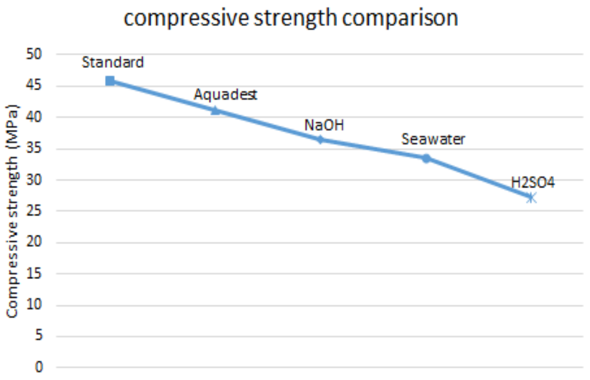

Figure 13. Comparison of 9:1 geopolymer compressive strength after leaching

4. Conclusion

After leached study of 90 days, characters of fly ash and bottom ash geopolymer concrete was changed from its initial condition. From microstructure and morphology analysis it shows that 1:1 geopolymer was cracked and changed from its initial shape, especially for geopolymer that was immersed in alkali and acidic solutions. Distribution of elements in 1:1 geopolymer also was not evenly distributed compared to 9:1 geopolymer which is have more evenly particle distribution. Elemental analysis shows Na-Si-O-Al geopolymer bond in the geopolymer after immersed in all solutions have lower amount than its initial for all geopolymer ratio. At the same time, solutions analysis shows the presence of silica, aluminium, potassium, calcium, iron and sulphur in small concentration for each solution. In addition, 1:1 geopolymer has the higher concentration for each element than 9:1 geopolymer. It is also affecting the compressive strength for all ratio of geopolymer. After immersed in acid solutions (pH 1), the compressive strength of 9:1 geopolymer decrease with the highest percentage that other solutions. No compressive strength analysis for 1:1 geopolymer, because it is cracked and changed. The released of some oxide compound affects compressive strength. Although some compound is released from geopolymer, no heavy metal found in pulverised material according to EN-12457 leaching test such as Cu, Cr, Cd and Pb. This fact that geopolymer bond are able to immobilize heavy metals on fly ash and bottom ash in various condition, such as acidic, alkaline, salt, and neutral conditions. Along with concept sustainable green concrete geopolymer concrete could be alternatives material for unfavorable conditions.

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