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Fabrication and characterization of fly ash-based geopolymer and its performance for immobilization of heavy metal cations

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Abstract

In this study, the geopolymer from fly ash as based-raw material has been examined on the ability of immobilization of several heavy metal ions. The fly ash analyzed for its physical and chemical properties. Fly ash and heavy metals were then mixed with an activator base until being homogeneous prior to be cast into a cylindrical mold following ASTM C 39-86 and left for 7 days. Subsequently, the geopolymer was characterized by SEM, FTIR, XRD, compressive strength test and toxicity characteristic leaching procedure (TCLP). The diffractogram of fly ash exhibited the existence of mullite, alumina and iron oxide phase, which were suitable with XRF result. From FTIR spectra, the vibration on finger print area appeared indicating the vibration of T-O-T from geopolymer network. The observation revealed that the addition of Pb2+ cations caused microcracking from SEM image and affected the compressive strength of the geopolymer. Sr2+ was an ion that was very easily leached compared to other three ions, and it caused a weak interaction between Sr2+ and geopolymer network. The higher amount of metal ions into the geopolymer network reduced the compressive strength of geopolymer. Sr2+-geopolymer had a lower compressive strength compared to Pb^{2+}, Cd^{2+}, and Co^{2+}.

Keywords: Fly ash; geopolymer; environmentally sound technologies; immobilization

1. Introduction

Fly ash is the solid waste left over from burning coal. Industries such as coal-fired power plants, cement, and paper factories are those producing the very large scale of fly ash. Of the total amount of coal combustion, approximately 7-10% is fly ash [1]. Global production of fly ash from industry will increase as long as the steam power plants still use coal as the raw material to generate the turbine. It will then bring an impact on environment due to the limitation of fly ash utilization in many power plant units

Several years ago, fly ash was categorized as toxic and hazardous waste by the Indonesian government. Due to this policy, it was less utilized in human life (only 20-30%). In 2021, Indonesia government issued a new policy under Government Regulation (PP) No. 22 that classified fly ash and bottom ash as the non-toxic and hazardous materials. As one of the environmental issues for sustainability, it is very important to expand the utilization of this industrial waste to decrease the level of discharge to the environment. Increasing the use of fly ash not only can suppress environmental hazards but also can make the ecological cycle better [2]. An alternative technique to utilize coal fly ash waste, for this reason, is required.

One way to increase the fly ash utilization is to use as raw material for geopolymer synthesis, which can be applied as a building material to replace hydraulic cement-based materials (such as Portland cement or PC) or other useful materials. Since the main components of fly ash are alumina and silica, it is possible to use it as geopolymers' raw material [3,4]. Other materials such as kaolinite and metakaolin can be used as raw materials for geopolymers as well, but the manufacture of geopolymers from fly ash provides more advantages [5–7].

The conversion of fly ash into geopolymers involves an activation step that initiates a polymerization reaction. The polymerizing components are silicate and aluminate ions. In addition to chain elongation during the polymerization process, cross-linking also occurs and increases the geopolymer strength. Ions and other particles can be bonded in the cross-linked geopolymer [8].

Iqbal et al. [3] mentioned that fly ash geopolymers can be

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utilized as the adsorbents for heavy metal cations as well as to immobilize both cations. The immobilization process may occur through the formation of chemical bonds between the metal cation with the geopolymer matrix and the physical encapsulation of the cation in the geopolymer matrix. However, it turns out that the presence of immobilized heavy metal ions in the geopolymer matrix may reduce the compressive strength [9,10,15,16].

The interaction between heavy metal ions and the geopolymer matrix varies, dependent upon the elements and its oxidation number. For example, As^{2+} ions are only slightly bound by geopolymers [11]. While, chromium ions cannot be properly immobilized in the form of Cr^{6+}, but can be immobilized very strongly by geopolymers in the form of Cr^{3+} [8,12]. Chemically, the Cs^{2+} ion is immobilized in the geopolymer - not physically encapsulated, and it does not give rise to significant changes to the mechanical properties of geopolymer [13]. The ion is also more resistant to leaching compared to other physically encapsulated metal ions. However, until now it has not been known satisfactorily how the metal ions are immobilized in the geopolymer matrix and how the heavy metal ions interact with geopolymer matrix.

Fansuri et al. [6] mentioned that the immobilization of metal ions into geopolymers is determined by two factors: size and valence of metal cations. Metal cations with a larger size tend to be better immobilized in the geopolymer and to be more difficult to remove from the geopolymer. Meanwhile, the greater the valence to radius ratio, the greater the bond strength of the metal ions in the geopolymer matrix.

As previously explained, the strength of the geopolymer can be reduced because of the interaction between heavy metal ions and the geopolymer matrix [14]. For instance, Fansuri et al. [6] suggested that the $Pb(NO_3)_2$ addition into geopolymer causes a decrease in compressive strength. The lowering compressive strength due to the interaction of heavy metal ions and geopolymer has also been reported by Fansuri et al. [6]. The decrease in strength causes the geopolymer unable to be used as a high-quality building material. Therefore, it is necessary to find an appropriate way to prevent a decrease in geopolymer strength caused by the immobilization of heavy metal ions. This works observed the effects of immobilization of cations $Co^{2+,}$ Cd^{2+}, Pb^{2+}, and Sr^{2+} into geopolymer matrix on its physical and chemical properties. This research can be one of options for fly ash utilization and its potential used as building materials in the future.

2. Materials and Methods

2.1. Materials

The materials used in this work included fly ash from PT. IPMOMI Indonesia, NaOH pellet (Merck, 99%), Na_2SiO_3 supplied by PT. Brataco Surabaya, demineralized water, $Cd(NO_3)_2.4H_2O$ (Aldrich, 98%), CH_3COOH (Aldrich, 99.8%), HNO_3 (Aldrich, 90%), $Al(OH)_3$ (Aldrich, 99.5%), $Sr(NO_3)_2$ (Aldrich, 99%), $Pb(NO_3)_2$ (Aldrich, 99%), and $Co(NO_3)_2.6H_2O$ (Aldrich, 97%).

2.2. Preparation of fly ash

Fly ash from PT IPMOMI, Indonesia was sieved using a Retsch 120 mesh sieve and dried in an oven at 105°C. The fly ash was then analyzed using XRF Panalytical Minipal 4, XRD PANalytical Expert Pro, and SEM Zeiss Evo MA 10. Meanwhile, particle size and size distribution of fly ash were determined using a Malvern Mastersizer.

2.3. Analysis of sodium silicate solution

The sodium silicate (water glass) used in this study was technical grade, so it was necessary to identify the chemical composition and to know whether there were heavy metal ions such as $Co^{2+,}$ Cd^{2+}, Pb^{2+}, and Sr^{2+} that could affect the calculation of leaching with ICP-AES. The measurement of SiO_2 used the gravimetric method, while the quantitative measurement of other elements was carried out by XRF.

2.4. Synthesis of geopolymer

Geopolymers were made from a mixture of fly ash, activating solution composing Na_2SiO_3 and NaOH, and sodium silicate solution. The geopolymer composition was determined by the ratio of Si/Al, Na_2O/SiO_2, and H_2O/Na_2O as reported by Fansuri et al. [6]. The geopolymers were cast into a cylindrical shape following the ASTM C 39-86 standard.

2.5. Heavy metal immobilization into geopolymer

Geopolymer synthesis was carried out for the immobilization of Co^{2+}, Cd^{2+}, Pb^{2+}, and Sr^{2+} with the different concentrations of 1000, 2000, 4000, 8000, and 16,000 ppm relative to fly ash mass. All cations were added from their nitrate salts ($Co(NO_3)_2$, $Cd(NO_3)_2$, $Pb(NO_3)_2$, and $Sr(NO_3)_2$).

The geopolymers synthesis was carried out with the same methods as reported by Fansuri et al. [6]; however, the addition of the above metal nitrate solution was carried out at 10 seconds after the $Al(OH)_3$ suspension was mixed into the fly ash and base activator. The geopolymer was synthesized based on 260 g fly ash. The stirring time was adjusted to the geopolymer phase formed after the addition of heavy metal ions.

2.6. Leaching test

The leaching of metal ions was tested using the TCLP method. The test was carried out on geopolymers that had been cured for 7 days. The geopolymer specimen had a mass of between 37 and 38 grams. Each specimen was immersed in 1 L of 0.01 M acetic acid solution and the ratio of the weight of the geopolymer to the mass of the acetic acid solution was 1:25. Immersion was carried out while stirring with a magnetic stirrer at 300 rpm for 32 hours. 10 mL of geopolymer soaking acetic acid solution (leachate solution) was taken at 1, 2, 4, 16 and 32 hours since the immersion started. The solution was put into a vial and one drop of concentrated nitric acid was added to keep the pH of the solution less than 2. The concentration of metal ions in the leachate solution was analyzed by ICP-AES to determine the number of heavy metal ions leached.

2.7. *Characterization*

Geopolymer characterization included mechanical properties measured by Universal Testing Machine, microstructure was observed by XRD PANalytical Expert Pro and SEM Zeiss Evo MA 10, and functional group used FTIR Shimadzu. The compressive strength of geopolymer (after 7 days of curing time) was determined using a Universal Testing Machine by applying an increasing compressive force at a rate of 100 $Kg.s^{-1}$ until the geopolymer being tested began to break. The pressure applied when the geopolymer was just about to break was recorded as the compressive strength of geopolymer in tons. Compressive strength measurements were carried out on three specimens from each sample. The conversion of the compressive strength value from tons to MPa was done by dividing the compressive force (in tons) and the cross-sectional area of the geopolymer specimen measured according to equation 1.

$$P = F.A^{-1} = (m \text{ x } g) \text{ x } (\pi \text{ x } r^2)^{-1} \qquad (1)$$

where g is gravitational force of 10 m s^{-1}, m is the result of the compressive strength as indicated on the tool (kg), π refers to 3.14, r = radius (m), P = compressive strength (Pa) = 10^{-6} MPa.

3. Results and Discussion

3.1. *Chemical composition of fly ash*

The results of XRF analysis (fable 1) showed that fly ash of PT. IPMOMI contained SiO_2, Al_2O_3, and Fe_2O_3 as much as 76.42% and 12.70% CaO. According to ASTM C 618, the fly ash is categorized as type F if the composition of SiO_2+Al_2O_3+Fe_2O_3 more than 70%. According to Kumar et al. [17], fly ash that contains Si and Al at that concentration can be used as a raw material for the manufacture of geopolymers.

Normally, Al_2O_3 content in fly ash is in the range of 25-45% [2], while the Al_2O_3 content in PT. IPMOMI was 13.76%. Fly ash with Al_2O_3 content below 25% is classified as low Al_2O_3 fly ash. This type of fly ash was attractive but has received less attention [2] and some additional Al_2O_3 source is needed when it will be used as a raw material for geopolymer synthesis.

3.2. *Particle size of PT. IPMOMI's fly ash*

Figure 1 shows the measurement results in which it showed that the fly ash of PT. IPMOMI contained many fine particles less than 43 μm. Leong et al. [1] reported that fly ash can produce geopolymer with good compressive strength if it contains fine particles below 43 μm more than 68.5% with compressive strength reaching 42.9 MPa after 7 days of curring. The particle size of PT. IPMOMI's fly ash contained 87.04% particles with a size of 30μm or lower. Based on the particle size analysis result, fly ash from the PT. IPMOMI has met the classification as a raw material that can produce geopolymers with high compressive strength. Based on PSA data, it can be seen that the fly ash has the heterogeneity in particle sizes. It might be caused by the coal burning producing the particles with various sizes in which the heaviest ones will fall first into the fly ash shelter; as a consequence, the particles do not have a good size distribution.

Table 1. Chemical contents of PT. IPMOMI Fly Ash

Compound	Percentage (%)
SiO_2	50.67
Al_2O_3	13.76
CaO	12.70
MgO	6.26
Na_2O	0.19
K_2O	1.28
TiO_2	0.76
Fe_2O_3	11.99
MnO_2	0.14
S_2O	0.63
Se_2O_3	0.12
P_2O_5	0.08
Ignition loss	1.42
Total	100 %

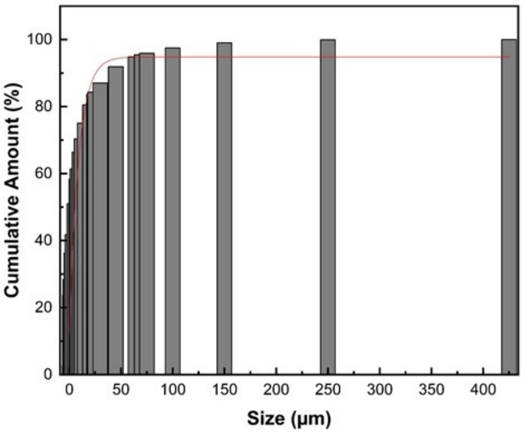


Fig. 1. The particle size distribution of fly ash

3.3. *Mineralogical structure of fly ash*

The fly ash was characterized using XRD. As seen in Figure 2 wherein the diffraction peak at 2θ = 26.5° exhibited the presence of mulite and quartz phases. The presence of a hump or mound in the 2θ region between 20° to 40° exhibited the presence of an amorphous phase of quartz. The peak at 2θ = 50° indicated the presence of mullite phase, and the peak from 60-80° represented the diffraction from iron oxide phase [19]. The existence of this amorphous phase easily leads to the formation of the geopolymerization network.

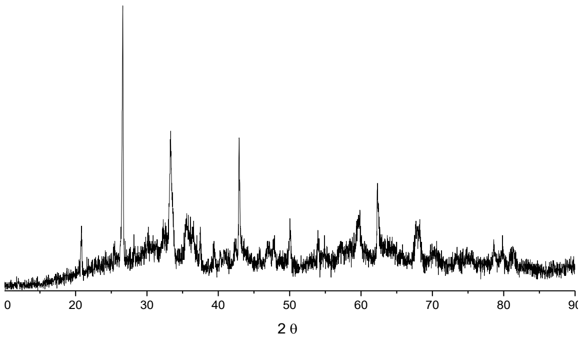


Fig. 2. Diffractogram of fly ash from PT. IPMOMI

3.4 Microstructure analysis of PT. IPMOMI fly ash

The image of the SEM analysis shows the shape and particle size of PT. IPMOMI fly ash in which it was dominated by sizes below 6 µm, like fine and very fine particles. The results were supported by particle size analysis and size distribution data with Malvern Master Sizer S. Because it contained a lot of fine particles, it was expected that the resulting geopolymer had physical and mechanical properties which was suitable for the immobilization of heavy metal ion through the geopolymerization process.

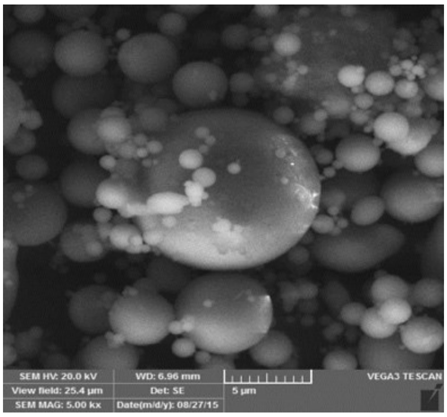


Fig. 3. Microstructure of fly ash from PT. IPMONI

3.5. Composition of Na_2SiO_3 technical grade

Based on the results of the characterization of the Na_2SiO_3 used in this study, the compositions of SiO_2, Na_2O, and H_2O were figured out as presented in Table 2. The technical grade of sodium silicate was used considering that it was easy to obtain and more affordable compared to the similar pro-analytical quality material. The selected sodium silicate material was very suitable for the geopolymerization process.

Table 2. The contents of Na_2SiO_3 technical grade

Content	wt%	Analysis Method
SiO_2	19.16	Gravimetry
Na_2O	37.99	AAS
H_2O	23.07	
PbO	0	XRF
CdO	0	XRF
Fe_2O_3	0.15	XRF
NiO	0.03	XRF
CuO	0.07	XRF
BaO	0.06	XRF
Others	19.47	

3.6. Compressive strength of heavy metal-geopolymer

Figure 4 presents the compressive strength of geopolymers that have been immobilized with heavy metal cations in various variations. In general, the immobilization of heavy metal cations impacts the compressive strength at low concentrations, and the addition of heavy metal cations can increase the geopolymer compressive strength [13]. However, the compressive strength will decrease when the number of metal ions immobilization exceeds the limit as the maximum acceptable for the geopolymer. The bond of S-O-Si or Si-O-Al, which is known as T-O-T bonding determined the compressive strength of geopolymer.

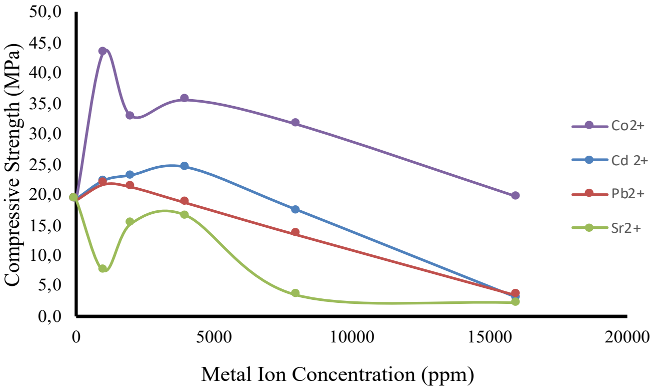


Fig. 4. The compressive strength of geopolymer and heavy metal-geopolymer

The immobilization of heavy metal cations was estimated to affect the formation of the basic structure of the geopolymer. Hence, the immobilization of these metal cations could reduce the compressive strength [10]. For the Sr^{2+} and Cd^{2+}, there was an increase after the addition of 4000 ppm of cations in which it might be caused by the replacement effect of counter ion during the geopolymerisation. The higher compressive strength of geopolymer indicated that the matrix could be immobilized effectively although there was the limitation of geopolymer matrix which led to the immobilized heavy metal ions.

3.7. Mineralogical structure of heavy metal-geopolymer

Figure 5 presents the results of this XRD analysis. It showed the crystalline phases of quartz (marked by the symbol Q), mullite (marked by the symbol M), and magnetite (marked by the symbol Ma). There was no other phase in the diffractogram.

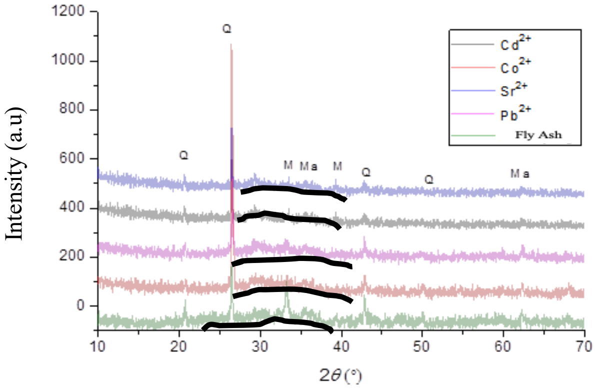


Fig. 5. XRD Patterns of fly ash and Heavy Metal-Geopolymer

The changes in the diffraction pattern from fly ash to geopolymer with the formation of a new amorphous phase, such as aluminosilicate, were indicated by a hump shift at an angle of 2θ between 20° and 37° to 22° and 41°. The presence of a small peak at 2θ around 22° indicated the presence of traces of quartz. After reacting with the activating solution, the intensity at an angle of 22° decreased showing that the amount of crystalline quartz phase was reduced [7,19]. This phenomenon, coupled with the presence of hump shift, was a characteristic of the formation of geopolymers. The width of the hump and the decrease in the intensity of the diffraction peaks indicated the formation of a more irregular or amorphous structure [20].

3.8. FTIR analysis

The signature of the infrared absorption band of geopolymers was shown by the vibration of the T-O-T bond around 957 cm^{-1} where T referred to Si or Al. The vibration at 1670 cm^{-1} indicated the presence of of H-O-H that it might be formed during geopolymerisation reaction due to the replacement position of counter ion from base activator into geopolymer network, the H_2O molecule only adsorbed as physical adsorption on the surface of geopolymer.

The geopolymer spectra without or with the addition of heavy metal ions had a similar vibration band. However, it can be seen that the addition of Sr^{2+} ions did not follow the trend of increasing the atomic number of each element. The relative atomic weights of the Co^{2+}, Cd^{2+}, Pb^{2+}, and Sr^{2+} ions were getting bigger. In Figure 6, the trend of the IR absorption for Co^{2+}, Cd^{2+}, and Pb^{2+} was in accordance with the trend of atomic weight when the wavenumber increased or decreased. On the other hand, the trend of Sr^{2+} contradicted the trend of the other three heavy metal cations.

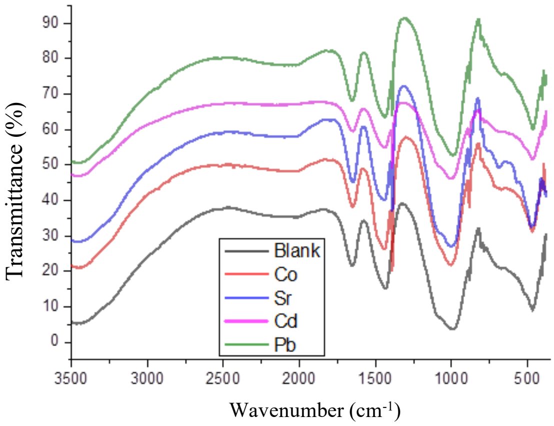
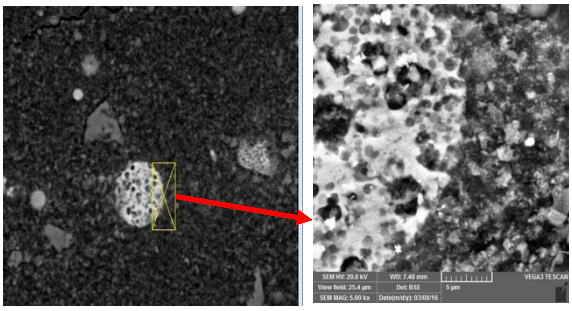


Fig. 6. FTIR spectra of geopolymers with and without the addition of heavy metal ions

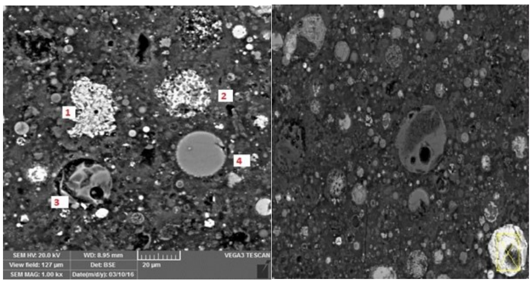
3.9. Microstructure of heavy metal-geopolymer

In Figure 7, Co^{2+} was successful to be immobilized into geopolymer network, and it seemed more homogeneous and compact. Based on the SEM image, it can be seen that there was only a small amount of fly ash that did not react (not changing its shape), and the pores formed were also not much. This supported the previous study. Then, the geopolymer added with Co^{2+} had the best compressive strength. The immobilization of Cd^{2+} into geopolymer had more pores explaining the reason for the lower compressive strength when compared to Co^{2+}-added geopolymer.

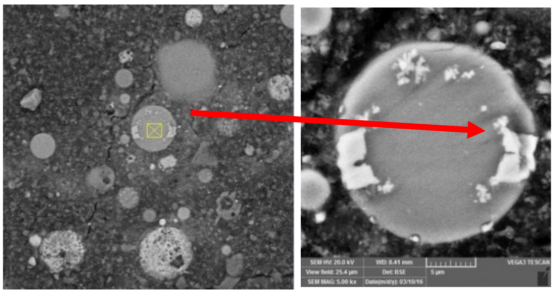
In contrast to the Cd^{2+}-added geopolymer, the Pb^{2+}-added geopolymer containing more microcracking. The more cracks, the less the strength of the geopolymer. The geopolymer added with Sr^{2+} contained many unreacted fly ash particles compared to other geopolymers making the compressive strength very low. The relationship between microstructure and strength obtained in this study was in accordance with that reported by Faradilla et al. [16] stating that geopolymers with a compact microstructure and few formed pores will have the best compressive strength.



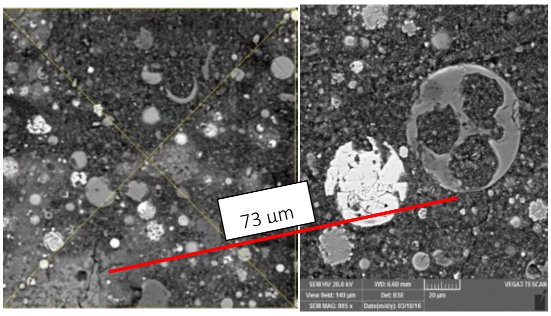
Co^{2+} addition



Cd^{2+} addition



Pb^{2+} addition



Sr^{2+} addition

Figure 7. Microstructure of geopolymer with different metal additions

3.10. Leaching test

Figure 8 presents the graph of leaching of geopolymers added with metal cations. At an additional concentration of 1000 ppm, it was only the geopolymer with Sr^{2+} added that experienced leaching although the amount was very little.

The amount of leached Sr^{2+} increased with the increase in leaching time where at the beginning of the leaching process there was an increase in leaching speed, and then the leaching speed decreased until one day, and stopped at a constant concentration because all the Sr^{2+} that could be leached have been exhausted.

Leaching that occurred in the Sr^{2+}-geopolymer proved that this cation was immobilized weakly by the fly ash-based geopolymer compared to other metal ions. According to van Almadhani et al. [10], the efficiency of immobilization was highly dependent on the valence and radius of the added ion. However, this ion was the weakest immobilized so that it was easily leached. The efficiency of metal immobilization in the geopolymer network was dominated by the mechanism of physical micro-encapsulation. Because the Sr^{2+} radius was larger, it was difficult to physically encapsulate in the geopolymer matrix. Other metals with smaller radius were better encapsulated so that they were not leached, thus the synthesized geopolymer was suitable for the immobilization of metal cations, which had a smaller radius than Sr^{2+}.

Another analysis result that supports the occurrence of encapsulation based on the cation radius is the compressive strength, as shown in Figure 3. Co^{2+}-geopolymers had the highest compressive strength compared to other geopolymers because their radii were the smallest among other heavy metal ions so all of them could fill the pores of geopolymer. The compressive strength was followed by geopolymer with Cd^{2+} and Pb^{2+} addition. The Sr^{2+}-geopolymer, which had the largest radius had the lowest compressive strength. Therefore, it can be stated that the metal encapsulation in geopolymer acts as a pore filler so that the more pores were filled, the more compact the geopolymer was formed, so that the higher compressive strength was obtained. The order of the compressive strength corresponded to the order of cation radii addition into the geopolymer network.

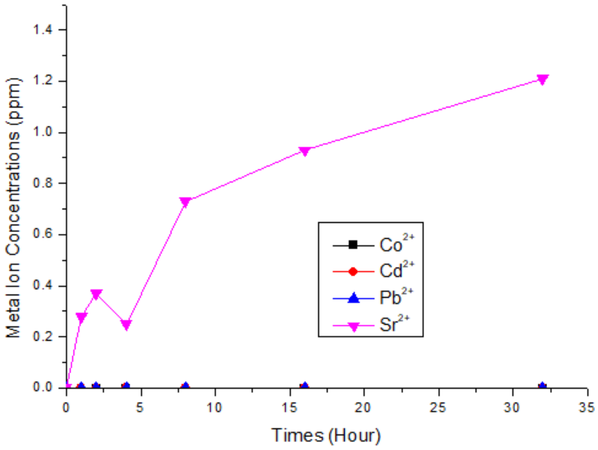


Fig. 8. TCLP result of heavy metal-geopolymer

4. Conclusion

Fly ash from PT. IPMOMI Power Plant was successfully synthesized as a geopolymer which was immobilized by several cations. The immobilization process of heavy metal cations affected the compressive strength in which the higher the metal concentration, the lower the compressive strength of geopolymer. It can be maintained by modified of the Si/Al ratio of geopolymer with addition another source of Si or Al. Then, the observation revealed that the addition of Pb^{2+} cations caused microcracking from SEM image and affected the compressive strength of the geopolymer. Sr^{2+} was an ion that very easily leached compared to the other three ions, and it caused the weak interaction between Sr^{2+} and the geopolymer network.

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