



Research article

Effect of bentonite on fly ash and bottom ash based engineered geopolymer composite

Muhammad Amin^{1*}, Sudibyo¹, David Candra Birawidha¹, Asnan Rinovian^{1*}, Bagus Dinda Erlangga², Muhammad Al Muttaqqi³, Ediman Ginting Suka⁴, Sherintia Pratiwi⁴

¹ Research Center for Mining Technology, National Research and Innovation Agency, Indonesia

² Research Center for Geological Resources, National Research and Innovation Agency, Indonesia.

³ Research Center for Advanced Chemical, National Research and Innovation Agency, Indonesia.

⁴ Department of Physics, FMIPA, University of Lampung, Indonesia.

Keywords:

Geopolymer,
Fly Ash,
Bottom Ash,
Bentonite,
Compressive Strength.

Corresponding author:

Muhammad Amin
Email Address: muha041@brin.go.id;
asna002@brin.go.id

Article history

Received: 19 September 2022

Revision: 26 May 2023

Accepted: 4 June 2023

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National Research and Innovation Agency
BRIN

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ABSTRACT

Fly ash, bottom ash, and bentonite have potential to be used as geopolymer precursors, because they contain high silica and alumina. Until now there has been no research that combines these three materials as geopolymer materials. This research aims to incorporate bentonite as an aluminosilicate source in the fly ash and bottom ash based geopolymer. Geopolymer concrete was made by mixing precursors, alkaline activator, aggregate (gravel), superplasticizer, and water. The characterization of geopolymer concrete was carried out using XRD, XRF, and SEM-EDS. Then the compressive strength test was carried out. The SEM-EDS results show that the elements contained in geopolymer concrete are dominated by Si, Al, and O. The XRF results, the constituent compounds of geopolymer concrete are dominated by silica and alumina compounds. The XRD phase results formed are Quartz, Albite, and Hematite. The sample with code K6, which did not contain bentonite, had the highest compressive strength value of 9.57 MPa and 8.92 MPa at a drying time of 18 hours and 24 hours, respectively. This can happen because the addition of bentonite can reduce the retraction process. This also causes the porosity of the concrete to increase, thereby reducing its compressive strength.

INTRODUCTION

The construction industry is the fastest growing industrial sector in the world. A large amount of concrete is used in construction around the world. One of the main ingredient of concrete is Portland cement. In the process of producing portland cement (OPC), a large amount of carbon dioxide (CO₂) is generated in nature which significantly contributes to greenhouse gas emissions (Nwankwo et al., 2020; Turner and Collins, 2013). Geopolymer concrete is an innovative building material, usually produced from the chemical reaction of inorganic particles that has great potential to reduce greenhouse emissions by up to 80% (Almutairi et al., 2021; Amran et al., 2021). In general, geopolymers are inorganic and

alumina-silicate (Si-O-Al) based ceramic materials similar to zeolites (Davidovits, 2017). Geopolymer formation is a rapid reaction of alkaline-activated solutions with silica-alumina minerals which then form long chains of three-dimensional polymers from amorphous covalent bonds (Turner and Collins, 2013).

Geopolymers are formed by three main ingredients, namely precursors, activators, and aggregates. Several studies have examined geopolymer concrete using fly ash, bottom ash, and bentonite as precursors (Adelizar et al., 2020; Hosseini et al., 2021; Yang et al., 2020). These three materials can be used as geopolymer precursors because they contain high silica and alumina, which are the constituent compounds of geopolymers. However, there is no research that combines these three materials into geopolymer concrete precursors.

In this study, geopolymer concrete was made using three precursors, namely fly ash, bottom ash, and bentonite. This study aims to study the effect of the composition of the three precursors on the mechanical strength of geopolymer concrete. The research was carried out in several stages, namely the manufacture of geopolymer concrete, characterization of the crystalline phase structure using XRD, characterization of the chemical composition of the material using XRF, morphological and elemental characterization using SEM-EDS, and mechanical strength testing. From this research, it is hoped that the composition of geopolymer concrete that has the best mechanical strength using fly ash, bottom ash, and bentonite as precursors is obtained.

METHOD

Tools and Materials

Tools used in this study include a 325 mesh sieve, oven (SHARP Electric Oven EO-18L, Japan), cube mold size (5x5x5) cm³, mixer B10 capacity 10 liters, and rotation 360/164 rpm, iron impact, Universal Testing Machines (UTM) Type HT2402, X-Ray Fluorescence (XRF) Pan Analytical Type minipal 4, X-Ray Diffraction (XRD) Pan Analytical Type X'Pert 3 Powder, and Scanning Electron Microscopy (SEM) Thermo Scientific Quattro 5.

The materials used in this study include fly ash and bottom ash (fine aggregate) from the Tarahan power plant (South Lampung), bentonite from Central Java, gravel (coarse aggregate) from Tanjung Bintang (South Lampung), The alkaline solution was combination of NaOH 8M, Na₂SiO₃, and water. NaOH with 98% purity comes from PT. Asahimas Chemical Banten, Na₂SiO₃ comes from PT. Brataco Bandung (West Java), superplasticizer comes from CV. Citra Additive Mandiri Jakarta, the oil fibers comes from PTPN VII Bekri Unit, Central Lampung, aquades and water come from PRTPB-BRIN Lampung.

Procedure

The procedure for mixing the ingredients was conducted using mechanical mixer for 10 minutes in total. Firstly, the solid materials (fly ash, bottom ash, bentonite, and gravel) with the composition as shown in Table 1 were dry mixed in the mixer apparatus and subsequently alkaline solution was added into the mix (Dong et al., 2020). Superplasticizer were gradually added into the mix while stirring until homogeneous and a dough was formed. The dough was put into a cube mold measuring 5x5x5 cm³. The dough was compacted by using an iron hammer in a mold. The sample was left in the mold for 24 hours at room temperature. The geopolymer concrete was removed from the mold and left at room temperature for 24 hours. Geopolymer concrete was cured in an oven at a temperature of 110°C with a varying curing time at 18 hours and 24 hours for a comparison.

Table 1. Geopolymer concrete material composition.

Sample mark	K1 (%)	K2 (%)	K3 (%)	K4 (%)	K5 (%)	K6 (%)
Fly ash	12.5	15.0	17.5	20.0	22.5	16.0
Bottom ash	22.5	20.0	17.5	15.0	12.5	25.0
Bentonite	8.5	11.0	13.5	16.0	18.5	0
Gravel	47.5	45.0	42.5	40.0	37.5	50.0
Sodium Hydroxide	1.6	1.6	1.6	1.6	1.6	1.6
Sodium Silicate	4.2	4.2	4.2	4.2	4.2	4.2
Superplasticizer	0.24	0.24	0.24	0.24	0.24	0.24
Oil palm fiber	1.8	1.8	1.8	1.8	1.8	1.8
Water	1.14	1.14	1.14	1.14	1.14	1.14

RESULTS AND DISCUSSION

Material Characterization Results

XRF results of results of fly ash, bottom ash and bentonite

Table 2 shows the XRF results of the geopolymer precursor materials used (fly ash, bottom ash, and bentonite). Based on Table 2, that the results of the XRF analysis of fly ash are dominated by oxides of SiO_2 , Al_2O_3 , and Fe_2O_3 which are more than 70% and CaO less than 10%, then the class F fly ash results are in accordance with ASTM C618-08A. Thus the fly is suitable for use in the manufacture of geopolymer concrete. While the results of the XRF analysis of bottom ash are dominated by oxides of SiO_2 , Al_2O_3 , and Fe_2O_3 . These results are in accordance with research conducted by Ul Haq et al., 2014, which states that the chemical composition of bottom ash is dominated by silica oxide SiO_2 and Al_2O_3 . The bottom ash is used as fine aggregate in the manufacture of geopolymer concrete. His bottom ash also has a small grain size of 2.705 mm. The grain size of this bottom ash meets the requirements of ASTM C330M-09 for the fine aggregate standard, which is smaller than 4.75 mm. Thus the bottom ash is suitable for use in the manufacture of geopolymer concrete.

Table 2. XRF results of fly ash, bottom ash and bentonite.

Chemical composition	Fly Ash (%)	Bottom Ash (%)	Bentonite (%)
SiO_2	63.25	46.12	64.38
Al_2O_3	13.36	13.46	17.87
Fe_2O_3	8.35	12.60	9.29
K_2O	1.16	2.19	0.98
CaO	4.49	7.93	2.76
MgO	5.15	8.44	0
TiO_2	0.48	0.59	0.33
Na_2O	1.69	1.98	0
LOI	2.08	6.70	0
MnO	0	0	2.90
P_2O_5	0	0	1.49

Compressive strength test results of geopolymer concrete samples

The compressive strength of concrete is the magnitude of the load per unit area which can cause the test object to crumble when given a load with a certain force (Bachtiar et al., 2020; Tampi et al., 2020). In general, bentonite would detrimentally influence the compressive strength on geopolymer materials (Chakkor and Altan, 2022; Waqas et al., 2021). Based on Table 3, K3 was found to be the lowest compressive strength at 2.16 Mpa and 2.12 Mpa (in category IV for solid concrete brick SNI 03-0349-1989). K6 with no addition of bentonite achieved highest strength at 9.57 MPa for curing time of 18 hours and curing temperature of 110 °C (in category I for solid concrete brick SNI 03-0349-1989). However, the sample of K4 and K5 experienced an increase in compressive strength. This phenomenon may be due to the influence of more fly ash used in K4 and K5 as the main precursor in this geopolymer system. The most optimum compressive strength value in sample K6 with curing time of 18 hours and curing temperature of 110 °C of 9.57 MPa using a variety of material compositions 16% fly ash, 25% bottom ash, 50% gravel, and does not use bentonite at all. This is because the addition of bentonite can inhibit the retraction process. Retraction is the loss of water by chemical reactions or evaporation in the concrete mix. This also causes the porosity of the concrete to increase, thereby reducing its compressive strength (Hou et al., 2019; Yang et al., 2020). This is because both materials contain a lot of silica and alumina which affect the compressive strength, this is also in accordance with the results of XRF analysis where fly ash and bottom ash are dominated by SiO₂ and Al₂O₃ compounds.

Based on the results of the most optimum compressive strength obtained by sample K6 at a curing temperature of 110 °C and curing time of 18 hours and the results of the minimum compressive strength of sample K3 at a curing temperature of 110 °C and a curing time of 18 hours, the XRF, XRD, and EDS characterizations were carried out on both samples.

Table 3. Geopolymer concrete compressive strength test results.

Temperature and Curing Time	Compressive strength (MPa)					
	K1	K2	K3	K4	K5	K6
110 °C 18 Hours	3.96	3.58	2.16	3.41	4.17	9.57
110 °C 24 Hours	4.24	2.96	2.12	2.09	4.26	8.92

XRF characterization results of geopolymer concrete

Table 4 shows the results of the XRF characterization of the geopolymer samples of K3 and K6. Based on Table 4. showed that the K3 and K6 samples were dominated by the oxides of SiO₂, Al₂O₃, and Fe₂O₃. The results of the characterization of samples of K3 on SiO₂ were 56.346%, Al₂O₃ was 15.404%, and Fe₂O₃ was 13.140% while K6 in SiO₂ was 55.766%, Al₂O₃ was 15.660%, and Fe₂O₃ was 13.073%. These results are in accordance with the XRF analysis of fly ash, bottom ash and bentonite where the results of the analysis are dominated by SiO₂, Al₂O₃, and Fe₂O₃.

Table 4. XRF characterization of geopolymer concrete samples at K3 and K6.

Chemical Composition	Rate (%)	
	K3	K6
SiO ₂	56.346	55.766
Al ₂ O ₃	15.404	15.660
Fe ₂ O ₃	13.140	13.073

Chemical Composition	Rate (%)	
	K3	K6
P ₂ O ₅	1.011	1.010
K ₂ O	2.090	2.068
CaO	9.312	9.466
TiO ₂	1.234	1.463
MnO	0.190	0.299
MgO	0.512	0.269
SrO	0.123	0.145
SO ₃	0.638	0.781

XRD characterization results of geopolymers concrete samples at K3 and K6

Based on Figure 1. It can be seen that the K3 sample is dominated by the Quartz phase (SiO₂) according to *file number* ICCD 00-046-1045 on peak $2\theta = 26.618^\circ$, the other phase is the Albite phase (Al₂O₁₆) corresponds to the ICCD file number 96-900-1631 at the peak $2\theta = 27.971^\circ$ and Hematite phase (Fe₂O₃) corresponds to the ICCD file number 01-089-0599 at the peak $2\theta = 29.475^\circ$. This is in accordance with the results of previous research conducted by Yang et al. (2020) where the results of XRD analysis using fly ash and bentonite are dominated by the Quartz (SiO₂) phase.

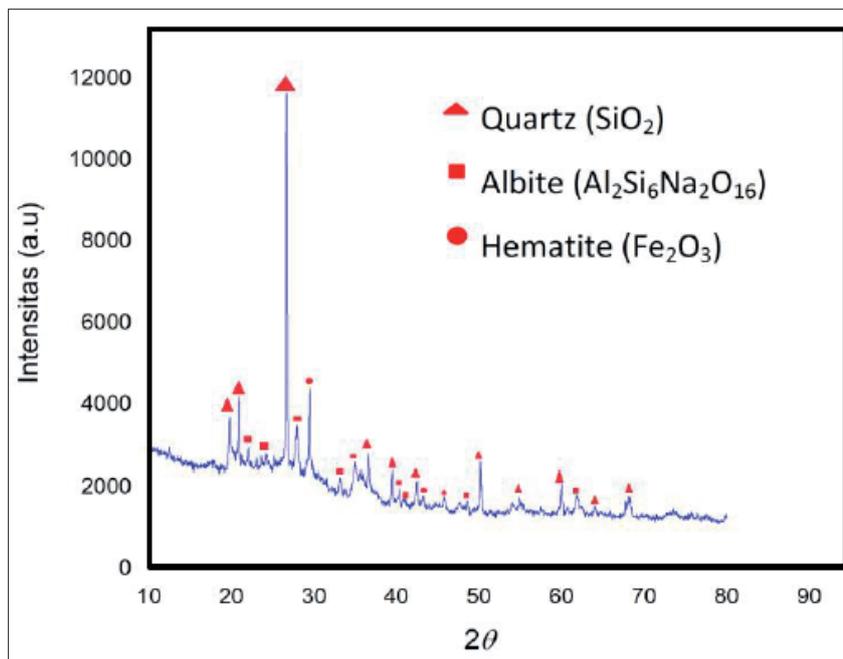


Figure 1. K3 sample XRD diffractogram.

Results of XRD characterization of geopolymers concrete samples at K6

Based on Figure 2. It can be seen that the K6 sample is dominated by the Quartz phase (SiO₂) according to *file number* ICCD 00-046-1045 on peak $2\theta = 26.688^\circ$, the other phase is Albite phase (Al₂O₁₆) corresponds to the ICCD file number 96-900-9664 at the peak $2\theta = 27.971^\circ$ and Hematite phase (Fe₂O₃) corresponds to the ICCD file number 01-089-0599 at the peak $2\theta = 29.475^\circ$. This is in accordance with Mehta et al. (2017) and Ryu et al. (2013) where the results of XRD analysis using fly ash are dominated by the Quartz (SiO₂) phase.

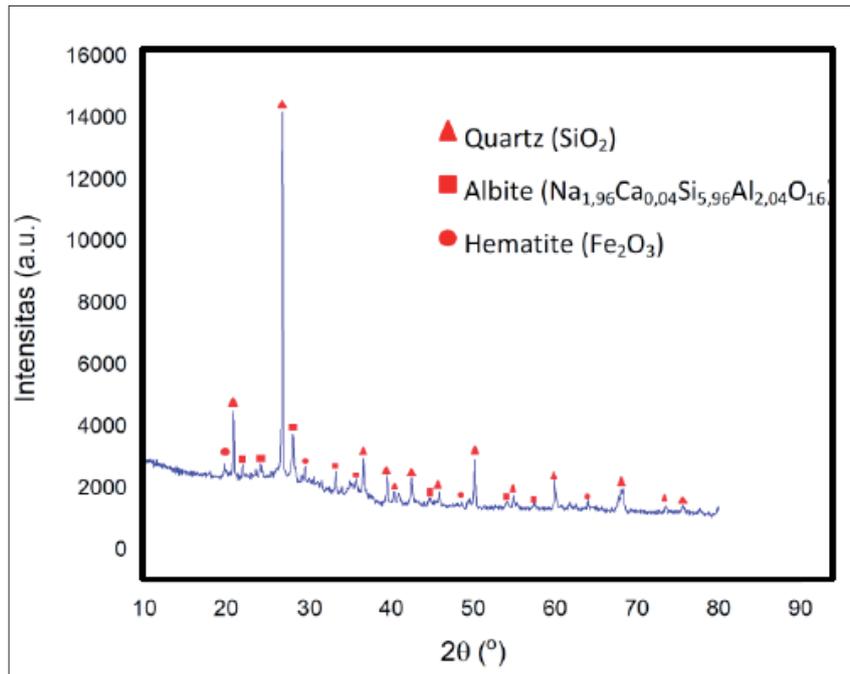


Figure 2. K6 sample XRD diffractogram.

Results of SEM-EDS characterization of geopolymer concrete samples at K3 and K6

Based on Figure 3. It can be seen that the elements formed in the two samples are Si, O, Al, Fe, Ca, Mg, Na, K, C, and S. Where the most dominating distribution is blue, namely Si elements, dark blue the element Al, and the red color is the element O.

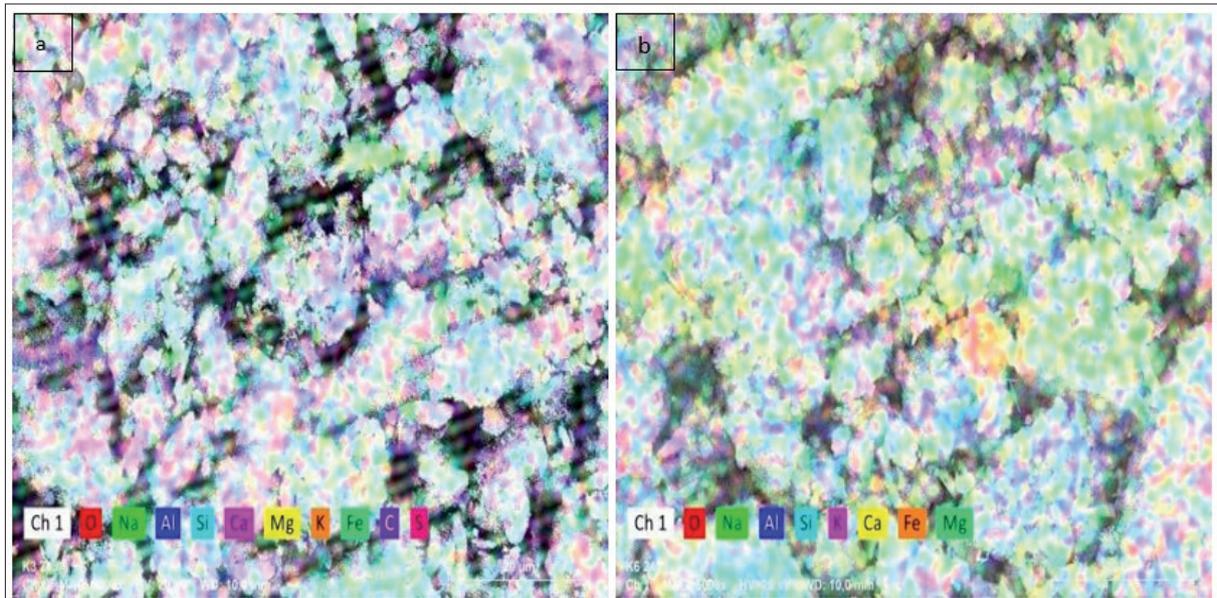


Figure 3. (a) The morphology of the SEM-EDS results on the K3 sample (b) Morphology of SEM-EDS results on sample K6.

Based on Figure 4. It can be seen that the two samples show that the EDS spectrum with the highest peak is dominated by Si, Al, and O. This is in accordance with XRF and XRD analysis. Where the highest phase is dominated by the quartz phase (SiO_2).

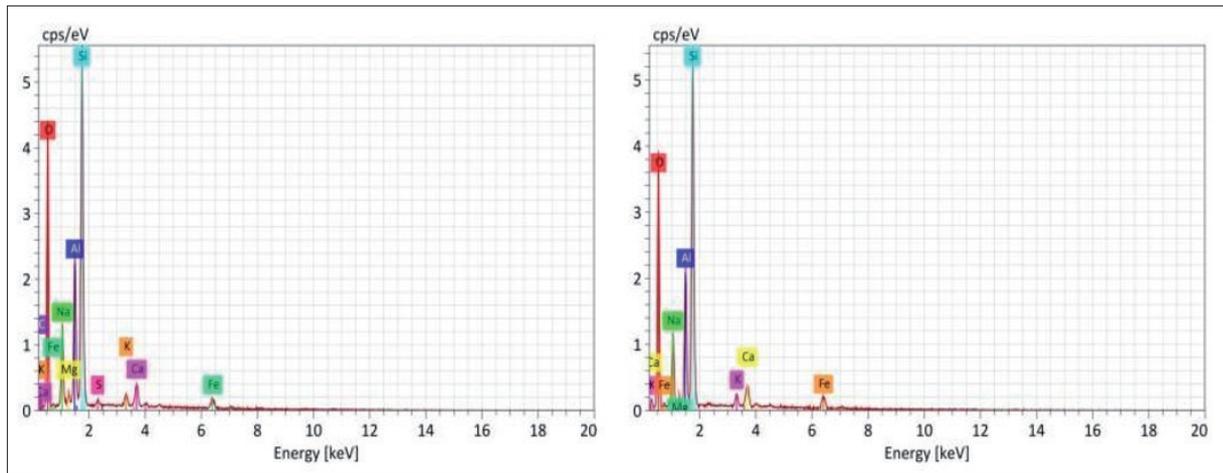


Figure 4. (a) EDS spectrum of K3 samples (b) EDS spectrum of sample K6.

Based on Table 5. It can be seen that the most dominating elements in the K3 sample are Si at 17.27% and Al at 8.21%. And other formed elements are C, O, Na, Mg, S, K, Ca and Fe. It can also be seen that the K3 sample is quite evenly distributed in the number of elements formed. While in K6, it can be seen that the most dominating element in the K6 sample is Si at 22.60% and Al at 9.83%. And other formed elements are O, Na, Mg, K, Ca and Fe. It can also be seen that the K6 sample is uneven because the carbon and sulfur elements are not formed. There was no significant difference found on the XRD analyses. Meanwhile, it was clearly showed by the SEM pictures that K3 with bentonite addition had more voids than K6, which is detrimental to the compressive strength.

Table 5. Number of elements of geopolymer concrete samples at K3 and K6.

Element	K3 Mass (%)	K6 Mass (%)
C	9.50	0
O	51.72	51.82
Na	7.00	8.25
Mg	0.63	0.48
Al	8.21	9.83
Si	17.27	22.60
S	0.36	0
K	0.94	1.24
Ca	2.21	2.66
Fe	2.15	3.11

CONCLUSIONS

Based on this study, it can be concluded that bentonite can negatively affect the compressive strength on fly ash and bottom ash based geopolymer materials. Even though bentonite can participate in the geopolymer reaction as showed in EDS, the appropriate proportion should be considered as it reduces the strength. SEM-EDS results show that the elements contained in geopolymer concrete are dominated by Si, Al, and O. The XRF results, the constituent compounds of geopolymer concrete are dominated by silica and alumina compounds. The XRD phase results formed are Quartz, Albite, and Hematite. The sample with code K6, which did not contain bentonite, had the highest compressive strength value of

9.57 MPa and 8.92 MPa at a drying time of 18 hours and 24 hours, respectively. Further research on the physical and chemical properties of geopolymer concrete with a similar composition can be carried out to determine the potential for mixing bentonite with fly ash and bottom ash. Although the compressive strength is not as good as geopolymer concrete that does not use bentonite, the addition of bentonite may produce better chemical and physical properties than the other side.

ACKNOWLEDGEMENTS

The authors would like to thank the Head of P RTPB BRIN, Head of the Non-Metal Laboratory, Head of the Preparation Laboratory and Head of the Chemical Analysis Laboratory of the Mining Technology Research Center – BRIN Lampung.

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