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The Influence of Nickel Slag Aggregate Concentration to Compressive and Flexural Strength on Fly Ash-Based Geopolymer Composite

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Abstract. Fly ash-based geopolymer with nickel slag aggregate has been successfully produced. Fly ash and nickel slag were obtained from Bosowa Jeneponto Power Plant and PT. Vale Indonesia, respectively. This research aims to investigate the influence of nickel slag concentration to compressive strength, flexural strength, and microstructure of geopolymer composite. The increment of nickel slag aggregate on fly ash was relative to the weight of samples. Geopolymer composite were synthesized by using alkali activated method, cured at temperature of 70 °C for 1 hour. The resulting composites were left at room temperature for 14 days, before compressive and flexural strength were performed. The results showed that the addition of nickel slag aggregate was found to increase the compressive strength of the material. The optimum compressive strength was 14.81 MPa with the addition of 10% aggregate. The optimum flexural strength was 2.63 MPa with the addition of 15% aggregate.

INTRODUCTION

Portland cement is the most dominant binder to produce pasta, composite, and concrete for structural applications. Production of 1 ton portland cement generates 1 ton of carbon dioxide to the air [1,2]. Portland cement production contributes around 7% to the total greenhouse emissions in the world [3].

Many studies have been conducted to develop friendly composite, such as fly ash-based geopolymer composite. Geopolymer composites were introduced as eco-friendly composite in order to reduce CO_2 emissions. Geopolymer composite has a potential as a replacement for Portland cement because it has good physical and mechanical properties such as high early compressive strength, heat and fire resistant, and low water absorption [4,5]. Geopolymer composites were commonly produced from aluminosilicates mineral having Si/Al ratio around 2, activated with alkaline solution [6]. Several research was reported of producing geopolymer composite by using combination of nickel slag and fly ash as raw materials [7,8]. The results indicate that combination of nickel slag and fly ash at certain percentage ratio can be increasing the compressive strength.

PT. Vale Indonesia produces 3000 tons nickel slag in nickel refining process in solid form every week [9]. The waste materials in the form of nickel slag generated a big problem to the environmental equilibrium. Combination of both nickel slag and fly ash becomes a solution to create environmental friendly materials that reveals good mechanical properties.

This paper aims at experimentally produce fly ash-based geopolymer composite with nickel slag aggregate. In addition, the effect of varying weight percent of nickel slag on the compressive and flexural strength of the samples and their microstructure were reported.

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EXPERIMENTAL

Material

Fly ash used as the raw materials was obtained from Bosowa Energi Power Plant, Jeneponto Regency, South Sulawesi. The chemical compositions of fly ash is shown in Table 1, and it is categorized as type C (ASTM C 618-03 $SiO_2 + Al_2O_3 + Fe_2O_3$ up to 59%). The slag used in the present study was obtained from the furnace Converter of PT. Vale Indonesia, East Luwu, South Sulawesi. Sodium Silicate ($Na_2O.3SiO_2$), sodium hydroxide (NaOH), and Aquadest (H_2O) were supplied from CV. Intraco Makassar, Indonesia.

TABLE 1. EDS results of chemical compositions of Fly Ash (weight %).

Oxide	SO_3	MgO	Na_2O	K_2O	CaO	TiO_2	Cr_2O_3	MnO	FeO	P_2O_5	SiO_2	Al_2O_3	NiO	ZnO
Fly Ash	2.21	13.24	1.77	1.82	18.60	1.29	0.35	0.33	15.33	0.35	27.26	16.60	0.30	0.54

The powder of nickel slag was produced by the following steps. First, chunks of nickel slag were cleaned by using aquadest, and then kept to dry at room temperature. The second, nickel slags were hammered manually to obtained gravel nickel slag. The third step, nickel slag were crushed by using mortar and pastel (particle size ranges from 75 < µm) [10]. The last process was the addition of nickel slag aggregate on fly ash by varying the weight of nickel slag percentage namely 0%, 5%, 10%, 15%, and 20%, respectively. The chemical composition of nickel slag with EDS is shown on Table 2.

TABLE 2. EDS results of chemical composition of nickel slag (weight %)

Oxide	SO_3	MgO	CaO	Cr_2O_3	FeO	SiO_2	Al_2O_3
Nickel Slag	0.29	23.60	0.86	2.27	29.75	38.85	4.38

Procedure

Geopolymer composites were prepared by alkali activation method and then cured at 70°C temperature for an hour. Samples were kept for 14 days old before compressive and flexural strength measurement were conducted. Compressive strength were performed on cylindrical samples with a diameter of 4 cm and height 8 cm (Figure 1), and samples for flexural strength test were 12 x 3 x 1.5 cm^3. Each composition was comprised of 3 samples to obtain the average of the measurement.

TABLE 3. The composition of a mixture of materials made from nickel slag geopolymer with fly ash binder.

Compositions	*Fly ash* (g)	NaOH (g)	Sodium Silicate (g)	Water (g)	Nickel Slag (%)
S. 1	30	2.5	7	4.25	0
S. 2	30	2.5	7	4.25	5
S. 3	30	2.5	7	4.25	10
S. 4	30	2.5	7	4.25	15
S. 5	30	2.5	7	4.25	20

(S.1: sample. 1, S.2: sample 2, S.3: sample 3, S.4: sample 4, S.5: sample 5)

Scanning electron microscopy (SEM) coupled with energy dispersion spectroscopy (EDS) was used to investigate the surface morphology (cracks formation), the interface zone between fly ash (matrix) and the aggregate. Phase is obtained by using XRD (X-Ray Diffraction) (CuKα radiation, λ = 0,154 nm).

Compressive strength was measured by using Hydraulic Servo Universal Testing Machine (WAW-500E Models), and flexural strength was measure by using Testometric M500-25CT.

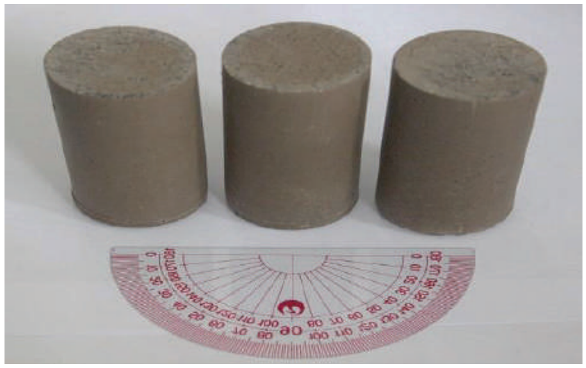
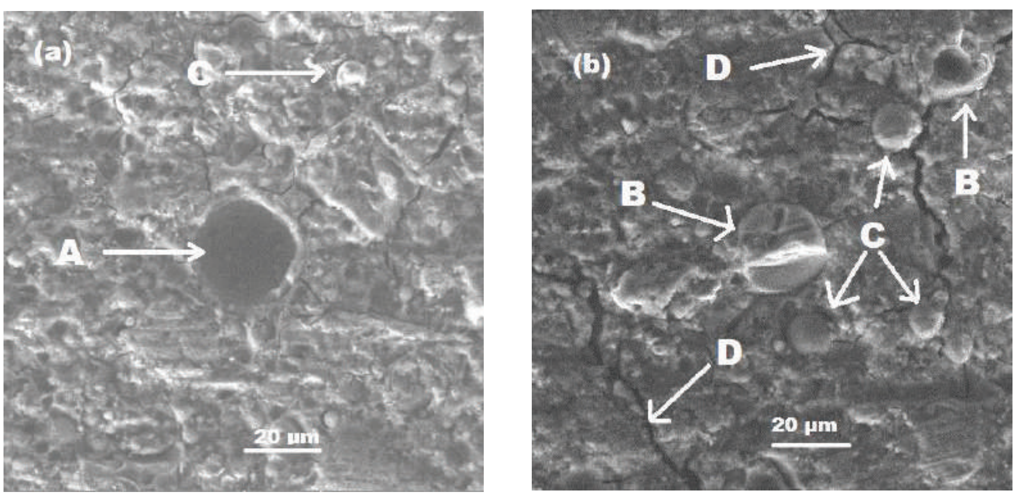


FIGURE 1. Geopolymer composite for compressive strength test produced in this study.

RESULT AND DISCUSSION

Microstructure of Geopolymer Composite before Mechanical Testing.

Microstructure of fly ash-based geopolymer was analyzed by using SEM. The presence of pores on the surface of geopolymer was also reported several study [11,12]. Figure 2. a and 2. b also shown the presence unreacted fly ash particles during geopolymetization process, but some of them has been distorted.



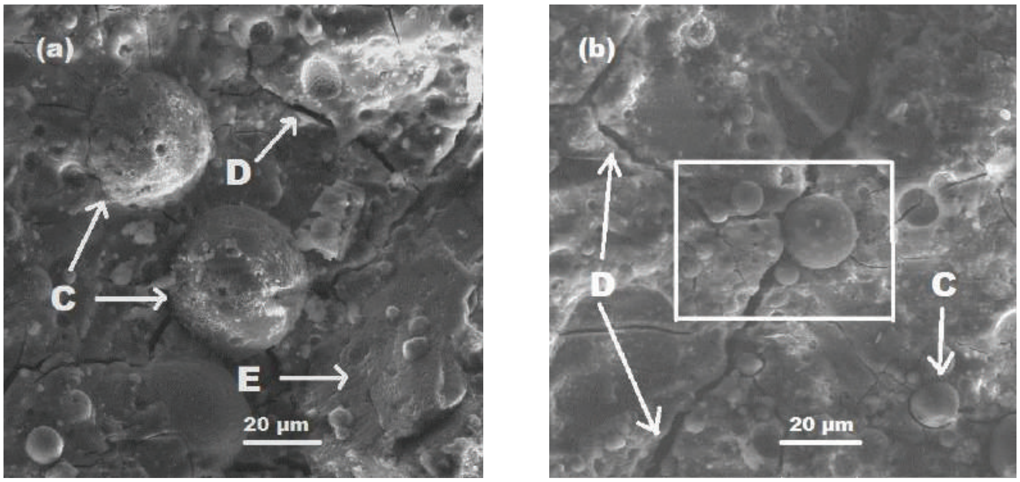
(a) (b)

FIGURE 2. SEM Micrographs of Fly Ash-Based Geopolymer Composite (a) S.1 (0% of nickel slag), (b) S.2 (5% of nickel slag). (A- Pores, B- Unreacted fly ash particle and nickel slag bound, C- Unreacted fly ash particle and alkali solution, D- Crack).

Figure 2.(a) and 2.(b) show SEM micrograph of geopolymer composites revealing pores and cracks in the surface of the sample. The cracks in figure 2 (a) and figure 2 (b) were probably as a results polishing process during SEM sample preparation. Unreacted fly ash particles and nickel slag acting like aggregate in the geopolymer composites. The bound between aggregate and matrix geopolymer appear weak as indicate by unreacted fly ash and nickel slag bound as shown in figure 2 (b) marked with “B”. Pores and air bubbles initiated the cracks as reported elsewhere [10].

Geopolymer composite microstructure after mechanical testing

The SEM characterization was also conducted to investigate the microstructure and cracks pattern of samples after mechanical testing. Figure 3. (a) and 3. (b) show the micrographs of sample surface after compressive strength testing was performed.



(a) (b)

FIGURE 3. SEM image of geopolymer composite after compressive strength testing (a) S.2 (5% of nickel slag), (b) S.5 (20% of nickel slag). (C- Unreacted fly ash and alkali solution, D- Crack, E-Geopolymer paste).

The presence of spherical fly ash particles with diameter of 10 – 40 µm were observed as shown figure 3 (a) and figure 3 (b) with marked "C" and geopolymer paste with marked "E" were observed on sample surface indicating that some of fly ash was remain with unreacted alkaline solution. The cracks pattern formed around the fly ash particles shows that the bound between particles fly ash and geopolymer pasta are weak with marked using rectangular area as shown Figure 3 (b).

The Effect of nickel slag percentage on mineralogical and crystal structure.

The effect of nickel slag percentage to the mineralogical and crystal structure was measured by using XRD. The results of search-match using PDXL II showing that the dominant phase on geopolymer composites is quartz, sodium aluminum silicon oxide, hematite, and periclase (Figure. 4).

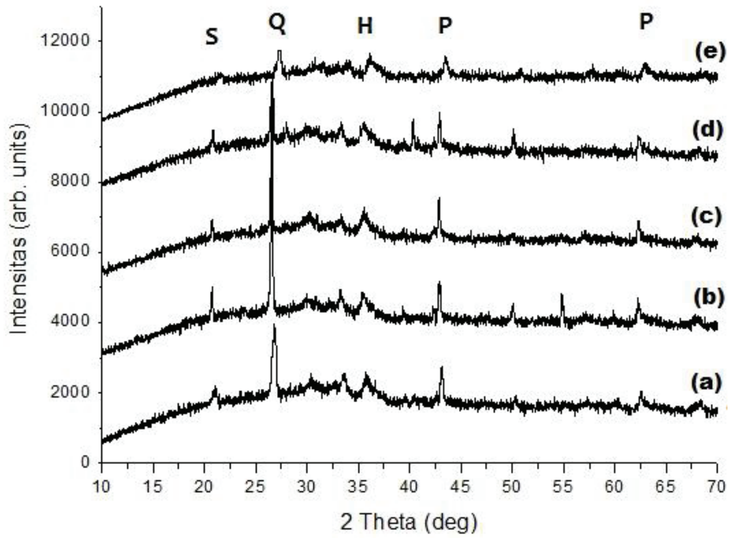


FIGURE 4. XRD Difractogram of Geopolymer Composite with nickel slag varies. (a) S.1 (0% of nickel slag), (b) S.2 (5% of nickel slag), (c) S.3 (10% of nickel slag), (d) S. 4 (15% of nickel slag), (e) S.5 (20% of nickel slag) (S = sodium aluminum silicon oxide, Q = quartz, H = hematite, and P = periclase).

The peak phase of the quartz, sodium aluminum silicon oxide, hematite, and periclase were found to increase on sample having 5% aggregate as shown figure 4 (b). The fourth phase of the peak decreased with the addition of nickel slag were observed as shown figure 4 (c), 4 (d), and 4 (e), respectively. The addition of nickel slag percentage affect the phase of geopolymer composite. The geopolymer composite diffraction patterns showed the amorphous hump at 20º-38º (2Θ) in aggrement with those reported by literature [13]. XRD pattern showed that the addition of reduce the level of crystallinity of the samples.

The effect of nickel slag percentage on compressive strength

The compressive strength results were shown in Figure 5. The average value of compressive strength for each mixture and standard deviation is shown as error bars. The total of five mix was made to investigate the effect of the nickel slag aggregate addition toward compressive strength. The detail of each mixture can be seen in Table 3.

The addition of nickel slag percentage do not have a significant effect in increasing the compressive strength of geopolymer composite. Compressive strength was found to increase in the addition of 0%, 5%, 10% of aggregate and then decreased on the addition of 15% and 20%. Geopolymer composite with 10% weight nickel slag percentage showed the highest compressive strength 14.81 MPa.

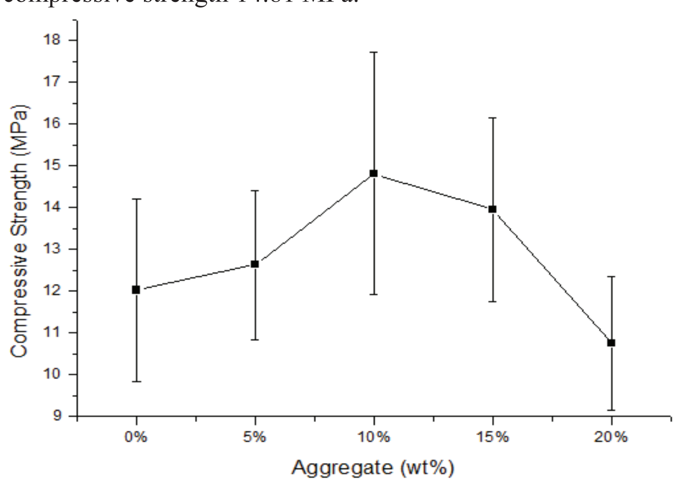


FIGURE 5. The effect of nickel slag percentage on the compressive strength of geopolymer composite.

Figure 5 shows that the compressive strength of geopolymer composite increase with the addition of nickel slag aggregate and reached the maximum value at the addition of 10% nickel slag relative to the weight % of fly ash. Based on this result, geopolymer matrix able to bind the aggregate perfectly up to 10%, this indicated by the declining of the compressive strength. If the deviation standard is calculated, then the compressive strength of geopoymer composite with and without aggregate approximately the same.

The bound between aggregate-matrix became weaker and the compressive strength of geopolymer composite decline as the effect of the increasing of aggregate concentration [14]. The compressive strength of geopolymer composite depends on the bond strength between the binder and the aggregate [15]. Pointed out that the decline of compressive strength geopolymer caused the ratio of sodium silicate sodium hydroxide is high, the amount of sodium hydroxide is not enough to finish the completion of dissolution process during the formation of geopolymer.

The effect of weight nickel slag percentage on flexural strength

Flexural strength of geopolymer composite was measured by using three bending point method. The addition of weight nickel slag percentage aggregate on geopolymer composite effect on the value of the flexural strength. Figure. 6 shown the flexural strength as the function of nickel percentage. Geopolymer composite sample with nickel slag aggregate of 15% obtained the maximum flexural strength of 2.36 MPa, and the lowest flexural strength obtained at the addition of 20% weight nickel slag percentage to 0.52 MPa.

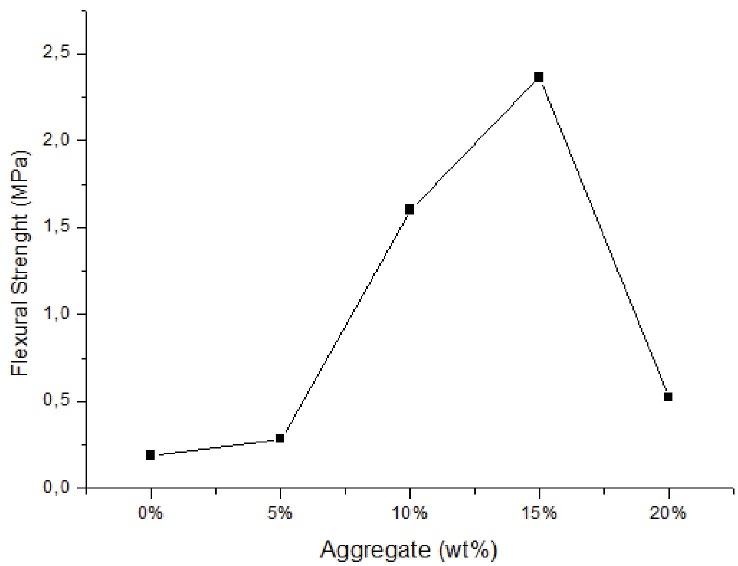


FIGURE 6. The effect of nickel slag percentage on compressive strength of geopolymer composite.

The results of compressive and flexural strength of geopolymer composite analysis, showing the greater nickel slag percentage the weaker their mechanical strength. It was found that the maximum addition of nickel slag in the range of 10-15% relative to the fly ash.

CONCLUSIONS

The effect of nickel slag percentage on compressive and flexural strength, mineralogical and crystal structure, and microstructure of fly ash-based geopolymer composite was investigated. It was found that the addition of nickel slag aggregate increase the compressive strength of composite and reached the maximum at the addition of 15% aggregate. The addition of aggregate exceeding 15% the bond between the matrix and the aggregate slag becomes weak and the mechanical strength decrease. It was also found that the addition of aggregate reduce the setting time of geopolymer and the workability was low.

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