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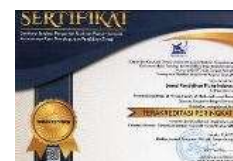
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STUDY ON PHYSICAL-CHEMICAL PROPERTIES OF FURNACE-NICKEL-SLAG POWDER FOR GEOPOLYMER APPLICATION

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ABSTRACT

This research aims to prepare powder of nickel slag from furnace machine, identify, and analyze of the elements or compounds containing in the sample. The data retrieval was done with the analysis of elemental composition, phase microstructures, crystal size, distribution, and composition mapping of the samples by using XRD, Rigaku Miniflex II, and SEM-EDS, Tescan Vega-3. XRD result indicates that the formation which the similar to amorphous phase was identified and the formation at peak $2\theta = 28.01°$ is identified as the low quartz (SiO_2). FWHM 0.18° was obtained using microcal origin 6.0 and average crystal size 53.37 nm was obtained by applying Scherer equation. SEM results show average grain size of samples which is less than 1μm and maximum to 4μm. Based on EDS result, the main constituent elements are Si 32.86 wt%, Mg 19.40 wt%, and Fe 32.03 wt%, respectively.

ABSTRAK

Penelitian ini bertujuan untuk menyiapkan bubuk slag nikel yang berasal dari furnace, yang dilanjutkan dengan mengidentifikasi dan menganalisis unsur atau paduan yang terkandung di dalamnya. Data diperoleh dengan melakukan analisis terhadap komposisi unsur, fasa mikrostruktur, ukuran Kristal, distribusi dan komposisi paduan dari sampel bubuk slag nikel furnace dengan menggunakan XRD tipe Rigaku Miniflex II dan SEM-EDS tipe Tescan Vega-3. Hasil analisis XRD menunjukkan bahwa formasi yang dihasilkan menyerupai fase amorf dan formasi yang terbentuk pada puncak $2\theta = 28.01°$ diidentifikasi sebagai low quartz (SiO_2). Dengan menggunakan microcal origin 6.0 diperoleh FWHM 0.18 ° dan menggunakan persamaan Scherer diperoleh rata-rata ukuran kristal 53.37 nm. Hasil pengukuan SEM menunjukkan bahwa rata-rata ukuran butir dari sampel bubuk slag nikel furnace bervariasi antara minimal 1μm dan maksimum 4 μm. Berdasarkan pengukuran EDS diperoleh secara berturut-turut bahwa unsur utama penyusun paduan slag nikel furnace adalah Si 32.86 wt%, Mg 19.40 wt%, dan Fe 32.03 wt%.

INTRODUCTION

Many studies conducted a nickel slag utilization, some of them were in the field of construction at many countries, as considering the nickel slag production can reach thousands of ton every week (Prederiy, 2011; Huang, Bird, Heidrich, 2007; and Motz & Geizeler, 2010). Addition, at the developing countries, nickel slag is utilized in the construction of highways such as for granular and hot-mix asphalt aggregate (engineered fill, sub base and base material, and asphalt concrete), aggregate of concrete manufacturing (fine and coarse aggregate) and cementations application (blended cement and raw material for cement manufacture) (Wang & Thompson, 2011). Nickel slag also used in industrial blast cleaning as ultra blast with a high density disposable blasting slag which has several advantages such as high production rates, non-reactant, and non-detectable

crystalline. Utilization of nickel slag depends on its mechanical properties, electrical properties and morphology, which are largely affected by the physical-chemical composition and crystal structure (Zhang & Chou, 2012; and Ghosh, Krishnamuthy, Sankaranayanan, 2010). Information of the physical-chemical composition and crystal structure of nickel slag become very important to determine its future material applications.

In this paper, the samples of nickel slag powder come from the smelting process of nickel ore after furnace processing. The analysis was focused on the physical-chemical properties of materials based on the characterization data by using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS).

METHOD

The procedure in this research is started from sample preparation of furnace nickel slag in solid form that processed into a powder, followed by analysis of micro structures through characterization by XRD and SEM and mineral composition analysis using EDS.

Sample Preparation

Hard chunks of furnace nickel slag, caused the process of nickel slag into a fine aggregate without using a machine becomes very difficult. Furnace nickel slag procedures into powder form begin with destruction to gravel size by using Jaw Crusher W 200 machine. Slag size gravel is rinsed with water to minimize contamination, and then dried in a furnace at temperature 170 °C for 30 minutes. The slag is dried then crushed using a Jaw Crusher W 200 to produce nickel slag sand. Slag which is processed by Jaw Crusher W 200, then processed again by milling, PAL-M100M, and resulted smooth output. The obtained sample from the processing chunk of nickel slag is formed at the laboratory of Material Sciences, Department of Physics, Universitas Negeri Makassar which is shown in Figure 1.

Microstructure Analysis

Information of the microstructure sample gathered through the XRD and SEM data analysis. Microstructure properties of materials obtained from XRD such as crystal ability of material, the average of crystal size, and full width half maximum (FWHM), can determine the electrical properties and crystal quality of ma-

terials (Wickenden & Kisthemnacer, 1994; and Sujiono, Sani, Saragi, Arifin, Barmawi, 2001). On the other hands, the grain size and patterns of particle distribution are factor that can determine the mechanical properties of the materials. This data obtained from SEM analysis.

Figure 1. Sample of furnace nickel slag powder.

Mineral Composition

Analysis of EDS data results was used to determine the element of nickel slag samples. This result can be used to identify the dominant constituent element of materials. This data is very important as the basis of utilization of the materials based on the characteristics of the element constituting, and the basic separation of nickel slag elements by using the appropriate method such as reduction under vacuum condition (Deng, Liu, Yao, Ma, 2014). Average particle size can be calculated from the top of the highest intensity by using the Debye-Scherer:

$$t = k\lambda / B \cos\theta \qquad (1)$$

where, t is the average of crystal size, k is the Scherer constant (0.89), λ is the wavelength of the X-rays, B is the FWHM in radiant, and θ is the angle of Bragg's diffraction, respectively (Khalil, Saad, Wahsa., 2012; and Kumar, Kumar, Narayan, Manoranjan, 2013).

RESULT AND DISCUSSION

Characteristics of Furnace Nickel Slag Powder

Based on the aggregated sample size of the nickel slag is in the range of 0 – 30 mm, so that the its advantages are wider. For instance, the slag with a grain size between 9.5 – 4.75 mm can be used as coarse aggregate and slag with a grain size between 4.75 – 0.075 mm can

be served as a fine aggregate such as in application for prestressed concrete, concrete block, etc. (ASA, 2011). While the nickel slag in powder provides wide opportunities of utilize the nickel furnace slag and further research on mixing the powdered sample with other powder materials to improve the quality of materials in order to maximize its application.

Physical Properties

Analysis of SEM result was used to analyze the surface morphology, size, and form of granules, pattern of granules distribution, mapping elements and compounds of nickel slag furnace constituents. Figure 2.a shows the result of SEM characterization of nickel slag furnace with 81 times magnification, using an SE detector at 15 kV. The grain size of nickel slag has a size of more than 1500 μm, the bright-white dots on the surface of grain are Ti at 0.20% and constituent element was taken with the highest atomic number that has the highest level of brightness.

Figure 2.b shows the result of SEM characterization of nickel slag powder furnace with 2000 times magnification. A grain of nickel slag has various size, 1 μm – 4 μm. It can provide a wide option for processing nickel slag furnace in micro scale particles. The EDS data indicates that the main elements of a sample are Si and Fe. The Si rich area on the SEM image (Figure 2.b) is marked with "B" on darker part, whereas the Fe rich area is marked with "A" on the brighter part because Fe has a higher atomic number than Si. It is so because the smaller of their atomic number leads to the less reflection appears as a darker area on the image of SEM characterization.

Figure 2.c showed nickel slag furnace powders with 5000 times magnification, while the scale of 5 μm is focused on the Fe. The Fe grain size is various, where some discharge electron can be seen at Fe granules because of its position, on the grain boundary.
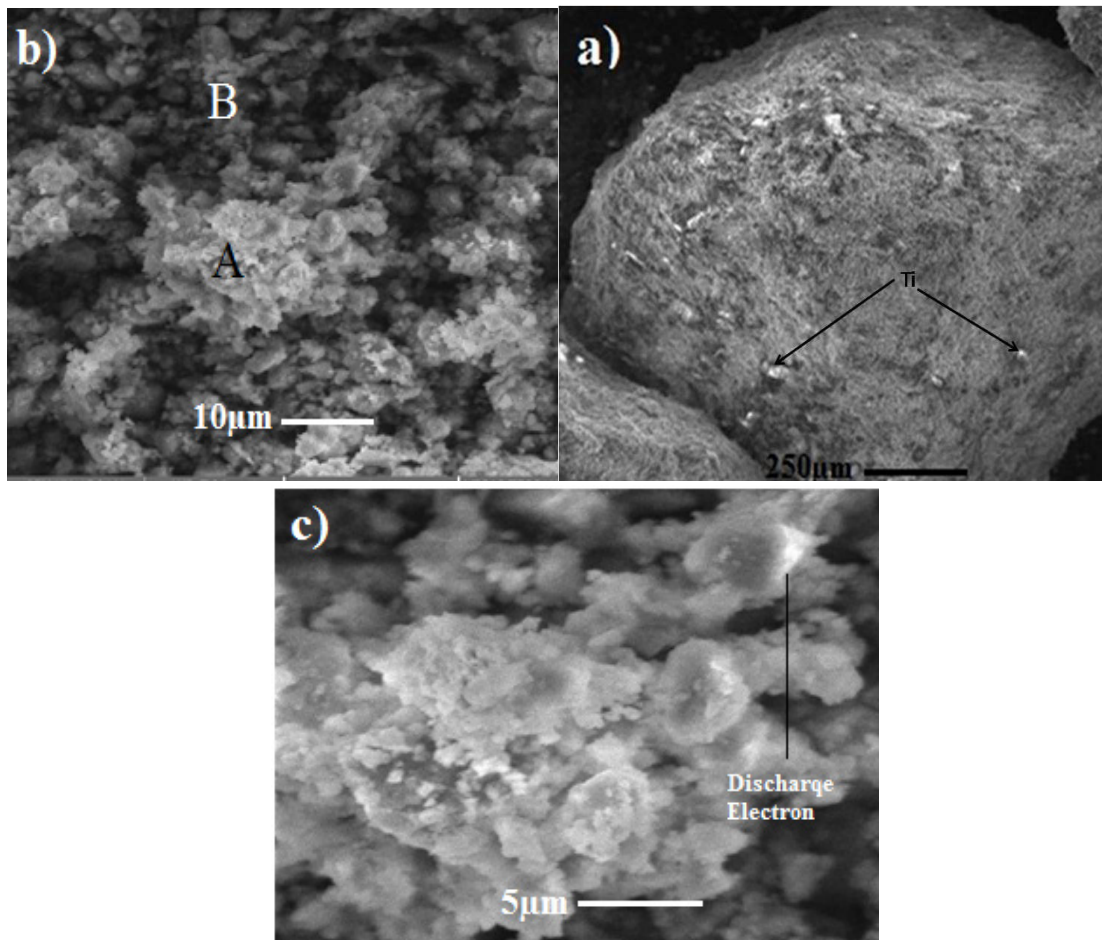

Figure 2. SEM image of: a) Nickel slag furnace with 81 times magnification (Ti appears as bright dots). b) Nickel slag furnace with 2000 times magnification (A=Fe rich area; B= Si rich Area). c) Nickel slag furnace with 5000 times magnification.

Chemical properties

The XRD data shows the compounds with its crystal phase are indicated by the presence of sharp peaks while the other phase resemble the amorphous phase that is indicated by the formation of the hump with an irregular intensity, revealing a characteristic for sample is in powder form.

XRD pattern in Figure 3 shows the patterns of nickel slag powder with some emergence of the crystal phase and amorphous like phase of nickel slag. Data retrieval at the angle of 2θ starts from $20°$-$80°$ with $\lambda = 1.54$ nm at 30 kV and 15 mA.

The most dominant peak is on (113) field with the intensity of the 1866 counts at the angle of 2θ is $28.01°$. The field shows the phase hypothetical silica (SiO_2) with 32.86 wt% is similar with the dominant mineral in powder furnace nickel slag from literature (Pan *et al.*, 2013) but this sample has a larger FeO, so its possible to make a new alloy material rich with FeO by adding powder nickel slag as an aggregate. FWHM value obtained from the Microcal Origin 6.0 was $0.18°$, this value is quite small refer to literature, that mean this phase has a good crystal structure and electrical properties (Wickenden and Kisthemnacer, 1994; and Sujiono *et al.*, 2001) and by Scherer equation acquired crystal size of 53.37 nm. Next on the field of (221) and intensity of the 482 counts at an angle of 2θ $29.69°$ and in field (310) with the intensity of 1470 counts at an angle 2θ is $30.99°$ show the clinoenstatite ($Mg(SiO_3)$) phase. In the field of (122) and the intensity of the 178 counts at an angle 2θ of $42.36°$ shows the phases of silimanite $Al_2(SiO_4)O$. In the field of (110) and the intensity of 1169 counts at an angle of 2θ $36.36°$ shows the phases of magnetite (Fe_2O_3). Whereas in the field of (200) and the intensity of the 849 counts at an angle of 2θ is $35.58°$ shows the phases of calcium peroxide (CaO_2). The formation of both clinoenstatite ($Mg(SiO_3)$) and silimanite $Al_2(SiO_4)O$ phase show that Si can bound with Mg and Al. The absence of compound between Si and Fe or Ca indicated that these elements cannot bound together or the diffraction is quite small to detect by XRD detector.

Chemical composition of powder furnace nickel slag from XRD analysis as shown in Figure 4 shows that the highest phase is clinoenstatite 68%. In addition there is another compound like hypothetical silica 13%, iron (III) oxide 12%, silimanite 4% and calcium peroxide 3%.
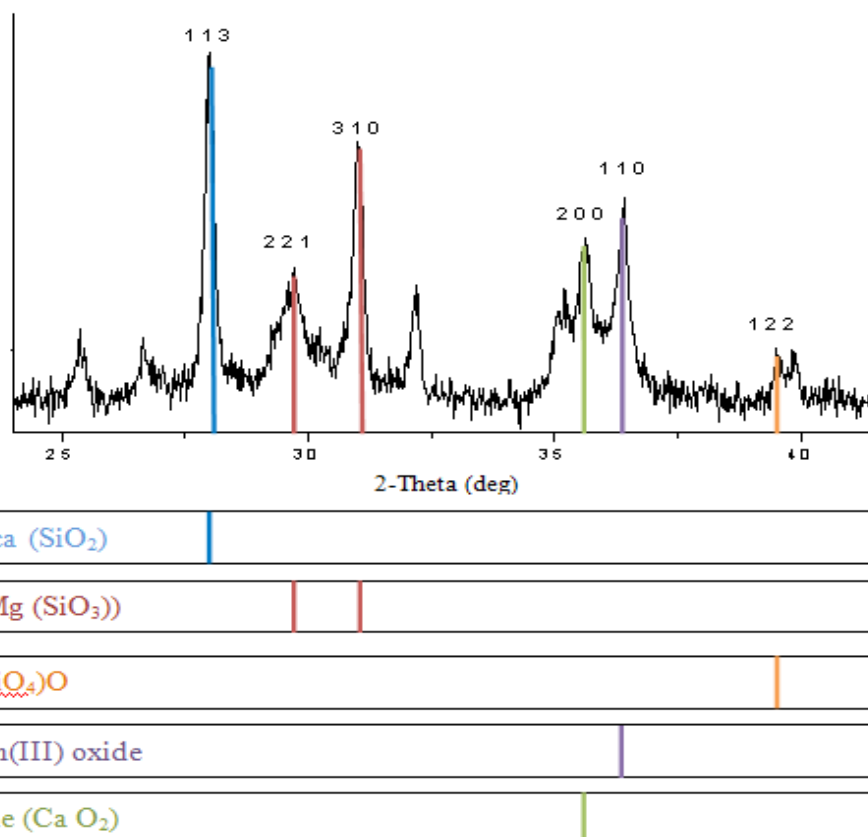

Figure 3. XRD pattern of powder furnace nickel slag.

Table 1. The EDS data of furnace nickel slag powder.

Symbol	Si	Al	Na	Fe	Mn	Cr	Ca	Mg	S	Ti	Co
Percentage [%]	32.86	4.02	0.25	32.03	0.50	2.07	7.82	19.40	0.24	0.20	0.60

Chemical Composition

The EDS result provides information on the chemical composition of furnace nickel slag as shown in the Table 1. There are three dominant elements Si, Fe, and Mg. These elements could be extracted from powder furnace nickel slag for further application, such as to extract Mg using carbonation process (Wen-ning, Yu-chun & Yan, 2010) and to extract Fe element with precipitation method.

Furthermore, the presence of Si and Al with ratio larger than 8 in powder furnace nickel slag give a possibility to use it as a raw material in synthesis geopolymer (Davidovits, 1994; Rangan, 2010), which has chain structure from Si and Al ions (Cheng & Chiu, 2003; Maragkos, Giannopoulou & Panias, 2009). Based on these results of the compounds contained in samples of powdered, furnace nickel slag can be separated using physical chemical methods whose results are expected to support the acceleration of the Indonesia economic development master plan.
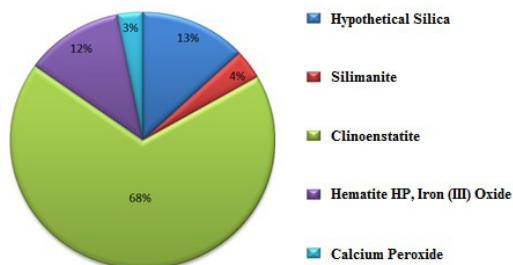

Figure 4. Percentage of compound in powder furnace nickel slag sample

CONCLUSION

Sample powder of furnace nickel slag with varied grain size on the first stage has successfully produced. SEM measurement data analysis result shows grain size for sample furnace slag powder on average 1 µm – 4 µm. XRD data analysis indicated resemble an amorphous phase formation was characterized by the presence of hump, with its highest peak at $2\theta = 28.01°$ which is a phase of low quartz (SiO_2), with the average FWHM of 0.18 and crystal size 53.37nm. The analysis of EDS indicates that major elements of nickel furnace slag powder are Si 32.86 % wt, Mg 19.40 %wt and Fe 32.03 %wt, respectively. The presence of Si and Al element with ratio larger than 8 is possible to use as a raw material for synthesis of a geopolymer.

ACKNOWLEDGEMENT

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