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STUDY ON PHYSICAL-CHEMICAL PROPERTIES OF POWDER FURNACE NICKEL SLAG

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Abstract

This research aims to prepare powder 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 of mapping compound of powder nickel slag samples by using XRD type Rigaku Miniflex II and SEM-EDS measurement type Tescan Vega-3. XRD analysis result indicates that the formation which similar with 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 schrrer equation. SEM measurement results showed average grain size of powder furnace nickel slag samples is less than 1μm and maximum to 4μm. Based on EDS measurement showed that the main constituent elements are Si 32.86 wt%, Mg 19.40 wt%, and Fe 32.03 wt%, respectively.

Keywords: chemical composition, grain size, nickel slag, physical-chemical properties, powder

INTRODUCTION

Many studies have conducted a nickel slag utilization, some of them are in the field of construction in various countries in the world, as considering the nickel slag production can reach thousands of ton every week [1,2,3]. In addition, in the developing countries nickel slag utilized as the construction of highways and as aggregate of concrete manufacturing [4]. Utilization of nickel slag depends on mechanical properties, electrical properties and morphology characteristics, which are largely determined by the physical-chemical composition and crystal structure [5,6]. Information of the physical-chemical composition and crystal structure of nickel slag become very important to determine of future materials applications.

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

EXPERIMENTAL PROCEDURE

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. Next, Slag size gravel rinsed with water to minimize contamination, then dried using a furnace at temperature of 170^{o}C for 30 minutes. The slag dried then crushed using a Jaw Crusher W 200 to produce shaped nickel slag sand. Slag which has been processed by Jaw Crusher W 200 then processed again by grinding mill PAL-M100M mill resulted swinging as the smooth output (this procedures also will be published elsewhere). The obtained sample from the processing chunk of nickel slag was formed at the laboratory of Material Sciences, Department of Physics, Universitas Negeri Makassar which is shown in Figure 1.

Fig. 1 Sample of powder furnace nickel slag

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 FWHM, can determine the electrical properties and crystal quality of materials [7,8]. On the other hands, the grain size and patterns of particle distribution are factor that can determine the mechanical properties of the materials. This result is obtained from SEM data analysis.

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 [9]. 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 constant Scherer (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 [10,11].

RESULT AND DISCUSSION

Characteristics of Powder Furnace Nickel Slag Samples. Based on the sample size aggregate of the nickel slag that can be set in the range of 0 – 30 mm, then the utilization of nickel slag furnace options were expended. For instance the slag with a grain size between <9.5 – 4.75 mm can be use 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 [12]. While the nickel slag in the form of powder provide 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 of 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, and HV is 15 kV. The grain size of nickel slag which had been treated has a size of more than 1500 µm. it appeared bright white dots on the surface of grain were the Ti elements with percentage of 0.20% and constituent element was sampled 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 can be seen a grain of powdered nickel slag have a variety of different size ranging between <1 µm– 4 µm. It can provide a wide option for processing nickel slag furnace in micro scale particles . The result of EDS data indicates that the main elements of a sample is 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, which caused Fe elements has a higher atomic number than Si, where the smaller of their atomic number caused the lower of their reflection causing a darker area on the image of SEM characterization.

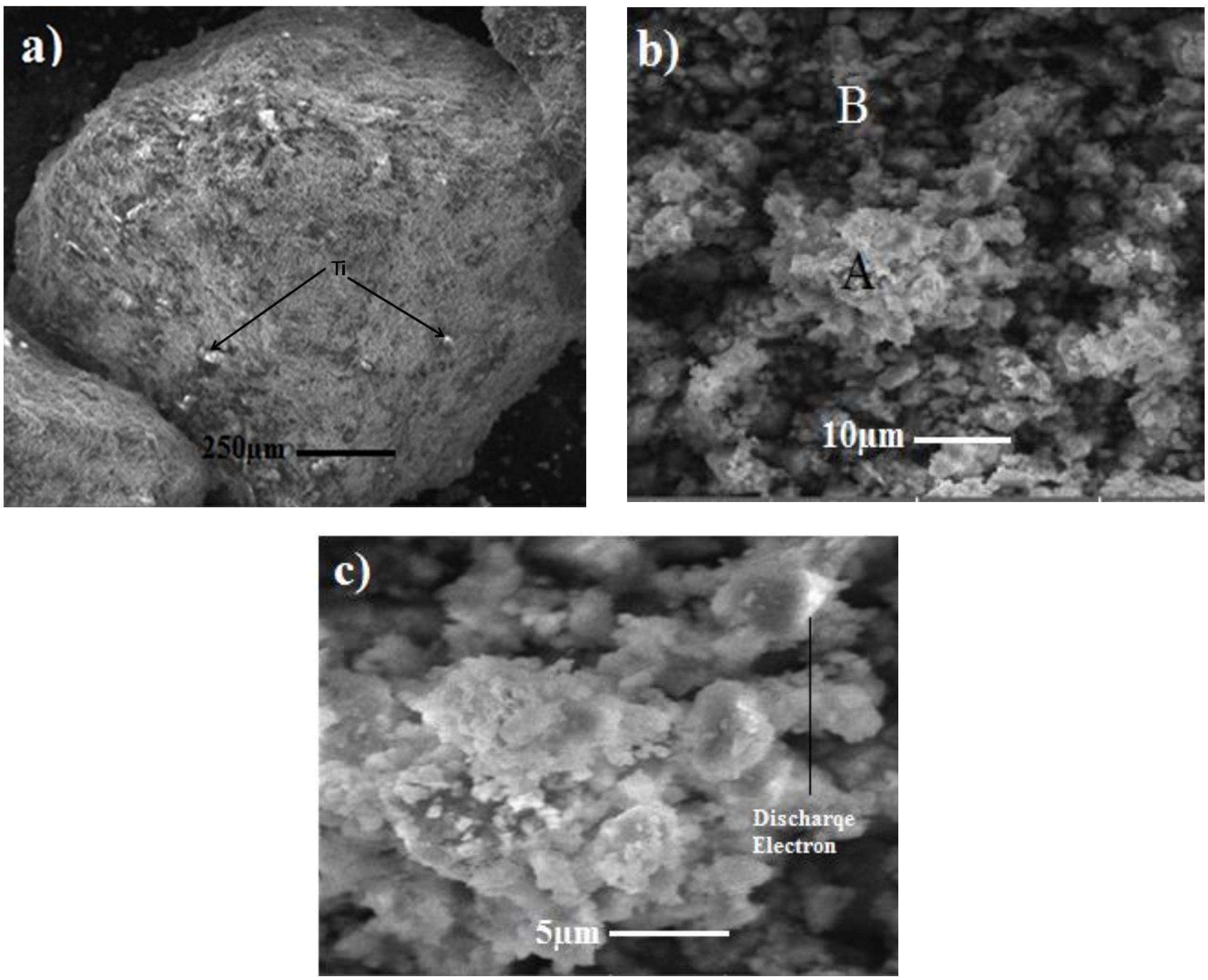

Fig. 2 a) Nickel slag furnace with 81 times magnification (Ti showed by bright dots). b) Nickel slag furnace with 2000 times magnification (A=Fe rich area; B= Si rich Area). c) Nickel slag furnace with 5000 times magnification.

Figure 2.c showed nickel slag furnace powders with 5000 times magnification, while the scale of 5 µm is focused on the element Fe, which is in the picture it looks that the grain size for Fe element also varied from small to large, in some part of Fe element in the granules is also seen some discharge electron because on the grain boundary.

Chemical properties. Characterization result of x-ray diffraction (XRD) from powder furnace nickel slag shows the content or composition of the constituents in the form of compounds. Crystal phase was indicated by the presence of sharp peaks while the other phase resemble the amorphous phase that was indicated by the formation of the hump with an irregular intensity, which is a characteristic for sample in powder form.

X-ray diffraction pattern in Figure 3 shows the diffraction patterns from nickel slag powder which showed the emergence of some of the top of the crystal phase and amorphous like phase of nickel slag. Data retrieval at the angle of 2θ starts from 20^o-80^o with λ = 1.54 nm at 30 kV voltage and current of 15 mA.

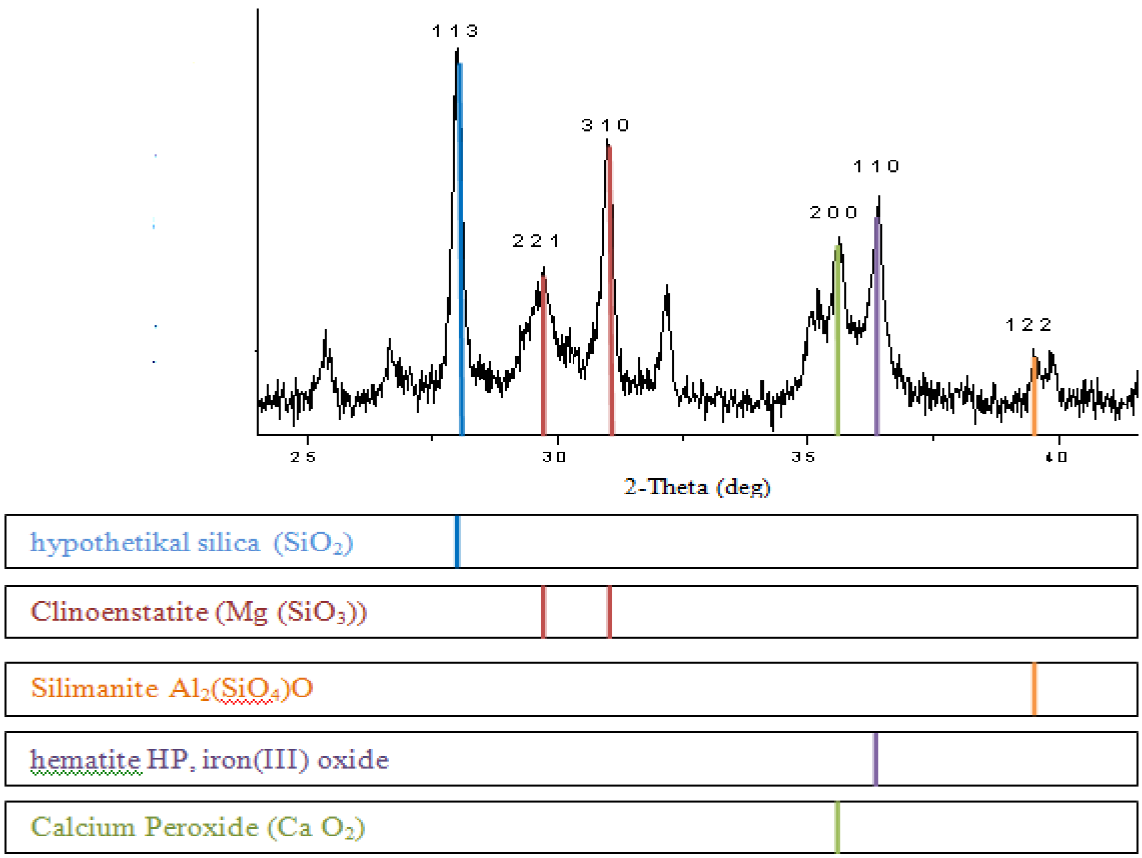

Fig. 3 XRD pattern of powder furnace nickel slag.

The most dominant peak is on hkl $d_{(113)}$ 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 [13] 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^o, this value is quite small refer to literature, that mean this phase has a good crystal structure and electrical properties [7,8] and by Scherrer equation acquired crystal size of 53.37 nm. Next on the field of hkl $d_{(221)}$ and intensity of the 482 counts at an angle of 2θ 29.69^o and in field hkl $d_{(310)}$ with the intensity of

1470 counts at an angle 2θ is 30.99^o showed shows the clinoenstatite ($Mg(SiO_3)$) phase. In the field of hkl d($_{122}$) and the intensity of the 178 counts at an angle 2θ of 42.36^o showed the phases of silimanite $Al_2(SiO_4)O$. In the field of hkl d($_{110}$) and the intensity of 1169 counts at an angle of 2θ 36.36^o, showed the phases of Magnetite (Fe_2O_3). Whereas in the field of hkl d($_{200}$) and the intensity of the 849 counts at an angle of 2θ is 35.58^0, showed the phases of calcium peroxide (CaO_2). The formation of both clinoenstatite ($Mg(SiO_3)$) and silimanite $Al_2(SiO_4)O$ phase showed that Si can bound with Mg and Al. The absence of compound between Si and Fe or Ca indicated that these elements can not 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 chemical compound like hypothetical silica 13%, iron (III) oxide 12%, silimanite 4% and calcium peroxide 3%.

Chemical Composition. The result analysis of EDS (Energy Dispersive Spectroscopy) provides information on the chemical composition of furnace nickel slag as shown in the table 1. It is shown that 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 [14] and to extract Fe element using precipitation method.

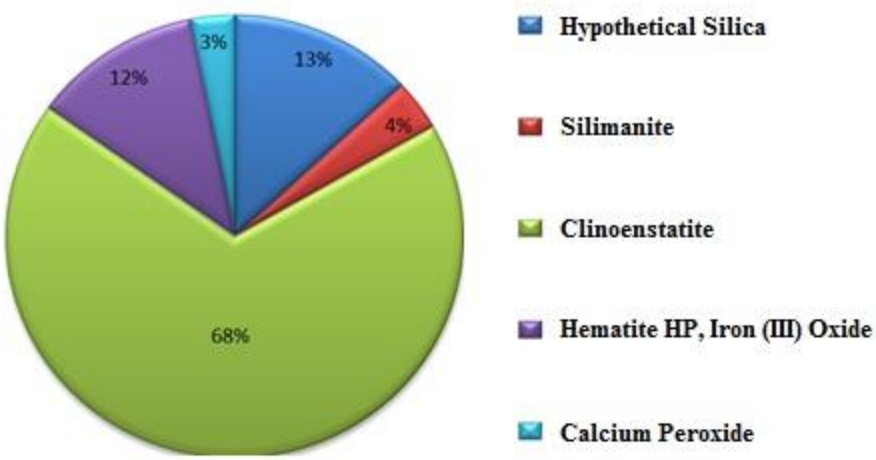

Fig. 4 Percentage of compound in powder furnace nickel slag sample

Table 1. The result of EDS characterization powder furnace nickel slag

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

Furthermore, the presence of Si and Al element with ratio larger than 8 in powder furnace nickel slag give a possibility to use it as raw material in synthesis geopolymer [15,16], which has chain structure from Si and Al ions [17,18]. 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.

CONCLUSION

Sample powder of furnace nickel slag with varied grain size on the first stage has successfully produced. SEM measurement data analysis result showed grain size for sample furnace slag powder on average <1 µm– 4 µm. Result of XRD data analysis indicated resemble an amorphous phase formation was characterized by the presence of hump, with its highest peak at $2\theta = 28.01^{o}$ 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.

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