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https://doi.org/10.5109/4794182

出版情報：Evergreen. 9 (2), pp.519-523, 2022-06. 九州大学グリーンテクノロジー研究教育センター
バージョン：

EVERGREEN Joint Journal of Novel Carbon Resource Sciences & Green Asia Strategy, Vol. 09, Issue 02, pp519-523, June 2022

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(Received February 11, 2022; Revised June 20, 2022; accepted June 20, 2022).

Abstract: Nickel is the main alloying element of stainless steel. Along with the increasing demand for stainless steel, the demand for nickel will also increase. Currently, the Indonesian government plans to add several smelters. Along with the increase in the number of smelters, this will have an impact on the production capacity of nickel and other metals. The positive impact that can be felt is the increase in the country's foreign exchange in terms of investment and also exports and imports. However, in the nickel extraction process, there is more slag was produced than the product itself. Then the waste in the form of slag in production will also increase. So the authors research and prove that this ferronickel slag can still produce a much more useful and useful output. This study aims to make refractory material from ferronickel slag with the addition of MgO which is compacted and through a roasting process. The magnesia additive variables used were 10, 20, 30, and 40 wt%. The roasting process was carried out at a temperature of 1200, holding time for 30 minutes, and oven heat rate of 5°C/minute. The results of the roasting process carried out two characterizations and 1 test. For characterization using SEM-EDS (Scanning Electron Microscope – Energy Dispersive X-Ray) and XRD (X-Ray Powder Diffraction). The test carried out is a compressive strength test. In the production of refractory raw materials, it is found that forsterite (Mg_2SiO_4) and spinel phases are increasing along with the addition of MgO additives. In terms of compressive strength, there is a maximum point obtained when adding 30 wt% additives.

Keywords: Ferronickel Slag; Magnesia; Roasting; Forsterite; Spinel Phase; Compressive Strength

1. Introduction

The increase in the number of smelters will have an impact on increasing ferronickel production capacity. On the other hand, the number of slags will increase dramatically. The production of each ton ferronickel generates 8 tons of slags[1)]. Many research activities focused on using slag or the ferronickel slag for recovery the precious compounds or valuable metals by applying various methods such as selective recovery or other treatment[2-4)]. Other studies showed that slag or ferronickel slag could be used as a raw material for specific purpose for example in construction materials[5-6)].

More than 65% of nickel is used in the stainless-steel industry, and about 12% is used in the manufacturing of superalloys or nonferrous alloys[7-8)]. Indonesia is one of the world's nickel-producing countries of the lateritic type, as well as world nickel reserves based on the United States Geological Survey report in January 2015[9)].

Laterite Nickel is a nickel resource derived from laterite nickel ore. The formation of laterite nickel ore is from a collection of several serpentine and goethite minerals, the type of rock that experienced the earliest weathering of the Earth's mantle[10)]. The composition and characteristics of laterite nickel vary in Indonesia. These differences can be influenced by the type of physical appearance on the surface, including the type of laterite, lithology, growing vegetation, and morphological conditions[11)]. Laterite nickel from the Palangga area, Southeast Sulawesi has limestone as a cover for laterite nickel deposits. Then under the limestone, there are new lateritic nickel deposits which are divided into three zones, namely the limonite, saprolite, and bedrock zones[12)]. Then in the Pomalaa area, Southeast Sulawesi. The highest levels of nickel are also found in the saprolite layer. That contains about 1.5 - 3%. The saprolite zone in this area has a thickness of 2-7 meters[13]. Indonesian laterite ore mainly been used for ferronickel production and in the future several number of smelters will be constructed; the number of ferronickel slags will also increase. Pyrometallurgical, hydrometallurgical by-products or any other products produced by mining-extractive metallurgy processing may lead environmental issue for example heavy metal pollution caused by industrial wastewater discharged[14]. Therefore, it is important to use environmentally friendly process to extract valuable metals from ores for example the implementation of bioleaching to process low grade ore[15]. The other way is the utilization of these by-products for other applications.

In recent studies that have been carried out on the beneficiation of ferronickel slags, such as: as an addition to geo-polymers, it has been shown to improve mechanical properties of geo-polymers and have less discontinuities[16], as a cement mixture that can increase workability and delays the hydration process so that the cement strength increases by more than 90%[17], as a secondary source of several precious metals[18], and as a refractory material by adding magnesia which results in good effects in terms of thermodynamics[19, 20].

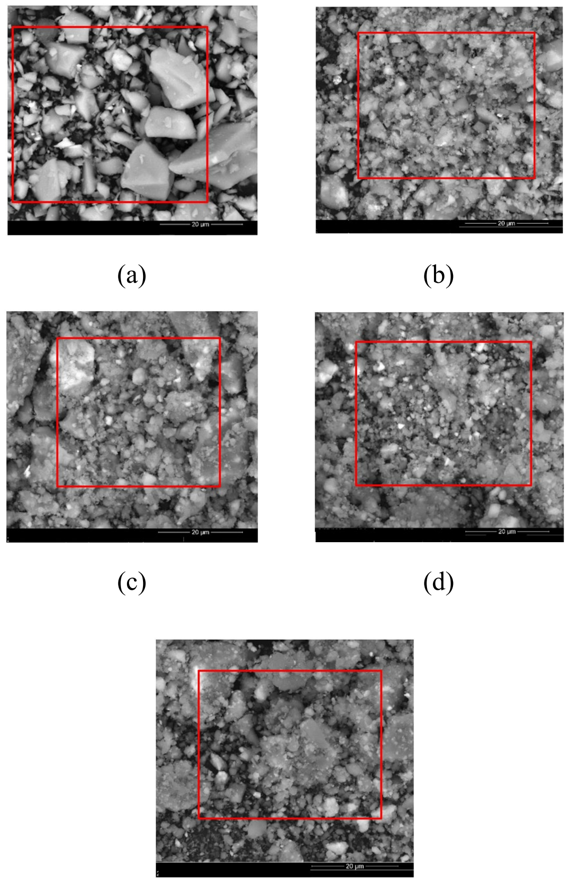
2. Methods

This research uses ferronickel slag from Sulawesi as the main raw material. Its purpose is to make refractory material. In the first stage, the slag is subjected to a drying process, then crushing and grinding to a size of 200 mesh. After the size is uniform, several gram samples were characterized to obtain initial composition and morphology data using SEM-EDS (scanning electron microscope – energy dispersive X-ray), and XRD (X-ray powder diffraction). Then the other slag samples were divided and added with magnesia additives with 4 percentage variables and each had 3 samples. Those variables are 10, 20, 30, and 40 wt% addition of MgO. Furthermore, the 12 samples were each compacted with a pressure of 200 MPa Each sample was compacted with a gradual increase in compressive strength until it reached 200 MPa. The goal is that the contact between the particles is getting closer and closer so that when removing the sample from the briquette, the pellet is in the perfect shape without defects.

The result of compaction in the form of solid pellets is reheated in a tube furnace with inert conditions, with a temperature of 1200℃ for 30 minutes, and a heat rate of 5℃/minute. This process is to sinter the particles uniformly and make a strong bonding between each particle[21]. After held the samples for five minutes at the specific temperature, immediately turn off the oven to start cooling down the samples. Those samples were kept in the oven until the atmosphere of the oven is back to room temperature. Then one roasted product is taken from each variable, a compressive test is carried out to determine its compressive strength. The rest of the roasted product was then milled to 200 mesh and characterized using SEM-EDS and XRD.

3. Results and Discussions

After the sintering process is carried out in the furnace, the sample is again subjected to the milling process to a size of 200 mesh. The milling process aims to make the SEM-EDS and XRD characterization process runs well. The SEM-EDS characterization of the slag sample refers to the morphology of the structure and also the elements contained in the sample according to the area being observed. EDS observations are in the area that is inside the red box area.



(a) (b) (c) (d) (e)

Fig. 1 (a) SEM picture of pure ferronickel slag sample **(b)** SEM picture of 10 wt% sample **(c)** SEM picture of 20 wt% sample **(d)** SEM picture of 30 wt% sample **(e)** SEM picture of 40 wt% sample

As seen from **Fig. 1(a)**, there has not been much bonding between particles. The particle size is still not uniform. This proves that the XRD observations are correct, showing that only a few compounds have been detected. The majority of these compounds are hematite (Fe_2O_3), forsterite, and SiO_2[22]. In terms of grain size, it tends to be non-uniform and looks large. The results obtained from the SEM-EDS observations are attached in **Table 1**. If you look at the table, the effect of adding MgO to the 10 wt% sample is seen in the significant increase in Mg levels. In addition, Si and O contained also increased. This may refer to the increased formation of forsterite (Mg_2SiO_4).

From **Fig. 1(b)**, it can be seen that the grains at the electron shooting location look darker than the SEM image in **Fig. 1(a)**. The grain size is also smaller than the sample in **Fig. 1(a)**. With the change in the size of the existing grains, it indicates that more compound bonds occur between MgO and the compounds contained in ferronickel slag. In the sample of 20 wt%, the elements detected by the EDS tool are as shown in **Table 1**. In these data, there is not too much increase related to the addition of MgO which is now 20 wt%. From **Fig. 1(c)** it can be seen that the grain size of the sample becomes smaller and tends to be more homogeneous in size.

In **Fig. 1(d)**, it can be seen that the particle size of the particles is smaller and more uniform and the bonds between the particles appear darker. From this, it indicates that the darker color is the forsterite compound formed. The reaction occurs as the MgO increases following the reaction in **Equation 1**.

Table 1. EDS Data

Elements	**MgO Addition (wt%)**				
	0	**10**	**20**	**30**	**40**
O	41.27	23.76	24.81	28.29	29.3
Mg	0.92	5.36	7.11	15.25	26.14
Al	8.29	4.04	3.5	3.98	4.07
Si	16.32	8.24	7.19	8.85	11.26
P	4.4	2.66	6.22	3.61	2.47
Ca	13.57	18.08	19.17	14.53	9.97
Ti	7.43	15.45	16.03	10.71	6.66
Fe	7.62	16.32	12.83	11.55	8.09
Others	0.18	6.09	3.14	3.23	2.04

In the 40 wt% sample, there was a significant increase in Mg levels compared to other samples. And there is an increase in levels of Si and O elements leading to the formation of a more dominant forsterite phase. When viewed from the SEM results, the visible bond colors tend to be darker than the images in the other samples. This further indicates that the more dominant phase in this variable is forsterite[20]. The grain size is also smaller and uniform when compared to other samples.

In order to determine the chemical composition and compounds contained in the sintered sample, XRD testing was carried out. According to **Fig. 2(a)** regarding the XRD results of the slag samples used. In samples that have not been added with MgO additives, the high intensity of hematite (Fe_2O_3) which is the strongest absorber of heat energy, makes MgO react with hematite and Cr_2O_3 to form magnesium iron chromate spinel[20]. This reaction is as shown in **Equation 2**.

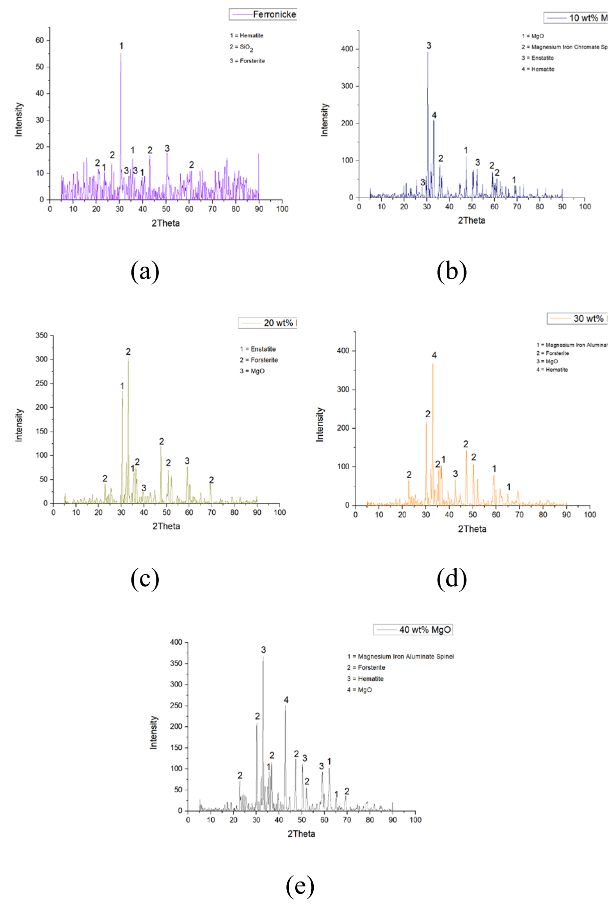


Fig. 2 (a) XRD pattern of pure ferronickel slag sample **(b)** XRD pattern of 10 wt% sample **(c)** XRD pattern of 20 wt% sample **(d)** XRD pattern of 30 wt% sample **(e)** XRD pattern of 40 wt% sample

In **Fig. 2(b)**, the enstatite compound is the compound with the highest intensity. Next is the hematite phase as the second-highest intensity compound in the sample. With the addition of MgO additive, a forsterite formation reaction occurs where a reaction occurs between enstatite ($MgSiO_3$) and MgO[19].

$$MgSiO_3 + MgO = Mg_2SiO_4 \ldots\ldots\ldots\ldots\ldots\ldots\ldots \textbf{(1)}$$

However, the spinel phase formed tends to have low intensity. For the formation of magnesium iron chromate spinel[23], the following reaction occurs:

$$5MgO + 3Cr_2O_3 + 2Fe_2O_3 = 5MgFe_{0,8}Cr_{1,2}O_4 \ldots \textbf{(2)}$$

If in the sample of 10 wt% MgO, the enstatite compound has the highest intensity, in **Fig. 2(c)**, the compound with the highest intensity is forsterite. Forsterite is increasing because the enstatite present reacts with MgO. Furthermore, in **Fig. 2(d)**, the forsterite phase begins to tend to stabilize and the magnesium iron aluminate spinel phase begins to form. The formation of the magnesium iron aluminate spinel phase occurs due to the reaction between MgO, alumina, and hematite as in

Equation 3[20].

$$5MgO + 4Al_2O_3 + Fe_2O_3 = 5MgFe_{0,4}Al_{1,6}O_4 \ldots\ldots \textbf{(3)}$$

Samples of 40 wt% MgO, had spinel and forsterite phases which were more dominant than the previous samples. As seen in **Fig. 2(e)**. So that it can be said that the more MgO additives are added, the more dominant phases are forsterite and spinel phases. Where the two phases react with each other with MgO which makes the growth of the forsterite and spinel phases increase.

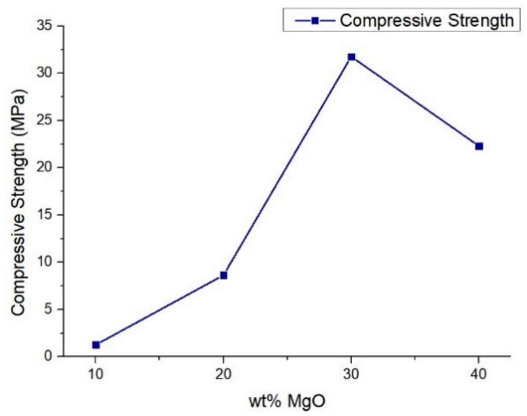


Fig. 3 Compressive Strength

One of the requirements for a material to become a refractory material is to have a high compressive strength. The compressive strength obtained from this test is shown in **Fig. 3**. Where the graph is obtained from the data from **Table 2**. It shows that the maximum compressive strength is in the sample containing the additive 30 wt% MgO. Then the compressive strength decreased in samples containing 40 wt% additives. Eventually, the maximum compressive strength with the addition of MgO for 30 wt% can reach 206.62 MPa, with some differences in additives and the slag conditions[19]. The beneficiation of slag in this study can be an alternative process to reduce the environmental problem which will be increase in the future due to smelting activities in Indonesia. In the future, it is essential that every activity related to mining, refining or processes that have a risk of environmental impact to comply with environmental regulations issued by the government[24, 25].

Table 2. Compressive Strength Data

Sample (wt % MgO	Diameter (mm)	Area (mm^2)	UTS (kgf/mm^2)	**Comp. Strength (MPa)**
10	25.3	502.93	0.128	**1.26**
20	19.4	295.7	0.88	**8.63**
30	20.4	326.98	3.24	**31.77**
40	20.5	330.2	2.27	**22.3**

4. Conclusions

There are three dominant compound elements contained in Ferronickel Slag. These compounds are hematite, SiO_2, and forsterite. Compounds produced by sintering products are diverse. With the addition of 10-40 wt% additives, the resulting compound is started from the growth of the forsterite phase, and the formation of the spinel phase. And the more additives are added, the more dominant phases are forsterite and spinel phases. One of the properties of refractory materials is that they have high compressive strength. In this case, from the variables carried out in the study, the variable addition of MgO 30 wt% was the most optimal sample. According to the results of the compression test, the variable is the maximum point of the compressive strength achieved. The compressive strength of the addition of 30 wt% MgO is 31.77 MPa.

Acknowledgements

The authors would like to express their gratitude for the funding from Hibah Publikasi Terindeks Internasional (PUTI) Prosiding 2020 Universitas Indonesia : BA-1045/UN2.RST/PPM.00.03.01/2021.

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