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Recovery of iron from electric arc furnace slag: effect of heating temperature and time

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Abstract. This work presents the iron oxide recovery from electric arc furnace slag with different temperature and time using acid leaching method. The results showed that formation of iron oxide from electric arc furnace (IO-EAFS) was influenced by the temperature and time of heating. This is evidenced by constant acid concentration and liquid to solid ratio, 5 hours of heating time was exhibited the highest amount of iron ion in extracted solution. The maximum of iron extract in heating temperature was at 100 °C. Throughout the characterization using Scanning Electron Microscopy (SEM), the iron oxide illustrated the microstructures were agglomerates predominantly with irregular shapes and XRD results further corroborated the presence of hematite combined with other metal oxide exists in the IO-EAFS. From the TGA analysis, it was identified that the recovered iron oxide was totally decomposed at 525°C. This results indicate that electric arc furnace slag is potential material of iron oxide for the future application.

1. Introduction

Malaysia is a growing developed country with population of 31.19 million in 2016 where demand of development is high [1]. Therefore, the demand of the raw materials for building and architecture constructions such as ceramic, polymers and steel are also on the rise [2]. Hence, it will increase the waste generation. It was assessed around 2 to 4 tons of wastes are being created per ton of steel delivered [3]. The assortment of wastes such as slags and sludges that are generated from steel plants are known as blast furnace flue dust (BFFD) and sludge (BFFS), blast furnace slag (BFS) and electric arc furnace slag (EAFS). Ca-silicates, Ca-Al-ferrites, and molten oxides of calcium, magnesium, manganese and iron are some profitable metals that contain in the sludges [1]. The increment of production of steel every year leads to inflation in slag generation and usually they are disposed via landfilling [4].



Landfill is necessary for Malaysia and waste production is predicted to increase from 292 kg/capita to 511 kg/capita within year 2000 to 2025 [5]. Furthermore, most landfills in Malaysia are operating with lacking legitimate defensive measures, for example, lining frameworks, gas venting and leachate treatment [6]. In recent time, landfilling turns out to be less positive as it will involve a considerable measure of terrains and subsequently required a huge expense [3]. Nonstop opening another land for landfill likewise will ultimately cause long-lasting corruption to the natural flora and fauna in encompassing region [2] [7].

Hence, reusing of the wastes for different profitable item or product is regards as an attractive and environmental-friendly solution for legitimately deal with the yearly enormous measure of the wastes. Recovering these wastes to be reused as auxiliary crude materials or other application in industry are vital for managing metals and mineral assets as well as for ensuring the earth sustainability. To recover the EAFS, some physical or chemical mineral processing techniques may be applied such as crushing, grinding, classification, hydro cyclone, magnetic separation, flotation, roasting or leaching [3].

In this research, leaching process are used to recover iron oxide from EAFS. Iron oxide is chosen as the metal that are going to recover because the EAFS are an iron oxide rich product and iron oxide can be used as secondary raw material in steel production [7] [8]. By utilizing the iron oxide in EAFS, the disposal and production cost for steel production will be reduced others than reducing the waste.

2. Methodology

2.1. Experimental procedure

The crude EAFS in the form of rock were grinded by using jaw crusher, cone crusher and ring mill and then was sieved to obtain a specific range of 40 to 60 μm . The sieved EAFS was weighted for 100 g before soaked in 300 mL of 50wt% sulfuric acid at a liquid to solid ratio of 3:1. The mixture was then heated at 80 °C for 4 hours. After heating process, the mixture was filtered and the filtrate was collected for the precipitation process. A few drops of hydrogen peroxide were added slowly into the filtrate in order to quench the reaction until it is completely oxidised and turn from greenish to brownish solution. To adjust the pH at 3, ammonia solution was added into the solution. The precipitated of iron oxide will form and filtered before drying it at 80°C for 24 hours. The dried iron oxide was then calcined by using muffle furnace at temperature of 400°C for 2 hours before further analysis. The process was then repeated by varying the temperature during acid leaching process for 90°C, 100°C and 110°C.

2.2 Characterization

The recovered iron oxide from EAFS was characterized to obtain its physical properties by using TGA, SEM and XRD.

3. Results and Discussion

3.1. Effect of heating temperature

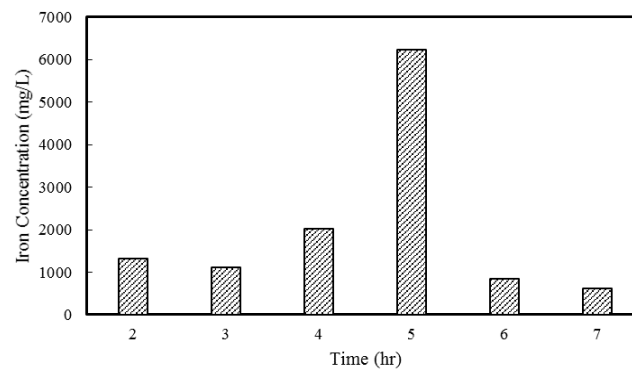
A variation of temperature in the range of 80°C to 110°C with 10°C temperature interval during sulfuric acid leaching were conducted. The weight of iron oxide recovered at each temperature were weighted and tabulated in Table 1. The optimum reaction temperature for the leaching process is found to be 100°C which 0.56 g of iron oxide was leached. According to F. Kukurugya et al., 95°C is the optimum temperature in sulfuric acid leaching in recovery of iron oxide [9]. This claim can also be supported by similar research done by Biao et al.[10] and Lim et al. [11]. However, these claims can be contended by T. Havlik et al. who indicated that the optimum temperature is at 100°C [12].

Table 1. Effect of acid leaching temperature

Leaching temperature (°C)	Weight of iron oxide after dry (g)
110	0.28
100	0.56
90	0.41
80	0.11

3.2. Effect of heating time

Six samples were carried out with different heating time during leaching process such as 2, 3, 4, 5, 6, and 7 hours respectively. The iron content in the leaching solution was analysed using ICP-AAS accordingly. Figure 1 illustrated the iron recovered from the leaching process. Li et al stated as the time increases, more iron being leached out from the slag in sulfuric acid solution [13]. It can be seen the highest optimum condition for the iron recovery is at 5 hours sample with constant 90°C. After 5 hours of leaching at fixed temperature, an abrupt decrease of iron concentration in the solution can be observed followed with 7 hours accordingly. According to studies from Jinping et al, the efficiencies of Fe recovery will increased from 2 to 24 hours at 80°C with the ratio of 1:5 [13].

**Figure 1.** Recovery of iron from EAFS at Different Time

3.3. Iron Oxide Characterization

The thermal behaviour of recovered iron oxide from EAFS was shown in Figure 2. The declining TGA thermal curve indicates the weight loss of recovered iron oxide occurred after being operated at temperature range of 25°C to 600°C. Referring to the TGA thermal curve, it shows that the decomposition step of recovered iron oxide occurred within the temperature range of 25°C to 525°C. The major weight loss as much as 40% takes place at temperature region of 300°C to 400°C and it is complete decomposed at 525°C. According to Divine et al, the major weight loss occurs at temperature 225°C and iron oxide decompose completely at temperature 500°C [14]. This finding had identified that the recovered iron oxide was totally decomposed at 525°C. Figure 3 illustrated the agglomerates predominantly with irregular shapes with low sphericity and sharp edges. The particles examined where extremely rough surface texture with platy, crystalline structure. Moreover, Yao et al studied the microstructure of the particle is in irregular shapes after leaching process. The pores that are found within agglomerates indicate the particle of EAFS have been leached by H₂SO₄ during leaching process [15]. Figure 4 represents the XRD patterns of phases in iron oxide. Besides, peaks appear overlapping in XRD pattern that represents the crystalline phases in the sample. It was stated that the more peaks appear that showed its crystalline structure and presents mascagnite ((NH₄)₂SO₄),

manganese iron phosphide ($\text{Mn}_{0.1}\text{Fe}_{0.9}\text{P}$), hematite (Fe_2O_3), and iron aluminium silicate ($\text{Fe}_3(\text{Al}_{0.2}\text{Fe}_{1.8})(\text{SiO}_4)_3$).

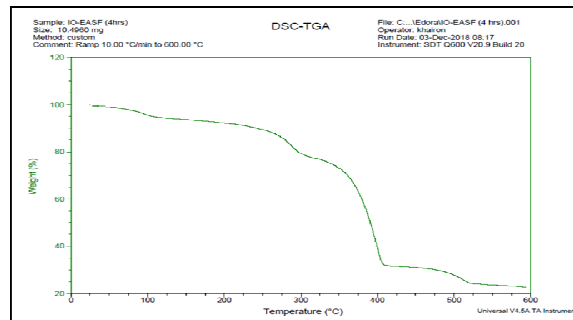


Figure 2. TGA Thermal Curve of IO-EAFS

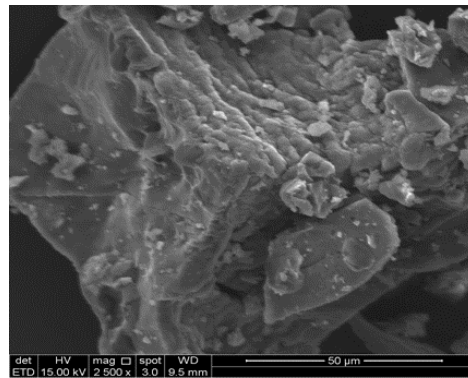


Figure 3. SEM image of IO-EAFS

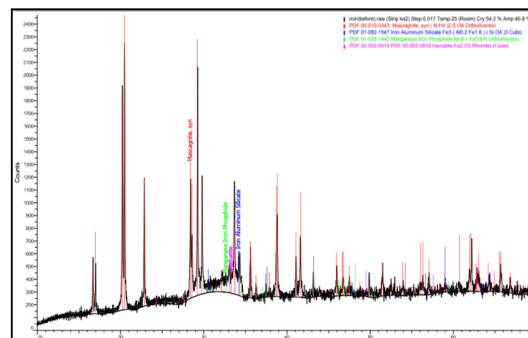


Figure 4. XRD pattern of IO-EAFS

4. Conclusion

In this study, the effect of heating temperature and time were carried out. The best results in iron recovery were obtained when the condition for acid leaching at temperature of 100 °C for 5 hours. Regarding to the characterization it is observed that iron oxide existed in form of hematite and other

metals oxide. Thus, it can be concluded that iron oxide from EAFS via method of acid leaching can be maximised with appropriate of conditions.

Acknowledgements

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