

# Heavy metal recovery from electric arc furnace steel slag by using hydrochloric acid leaching

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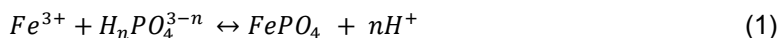
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**Abstract.** Electric Arc Furnace steel slag (EAFS) is the waste produced in steelmaking industry. Environmental problem such as pollution will occur when dumping the steel slag waste into the landfill. These steel slags have properties that are suitable for various applications such as water treatment and wastewater. The objective of this study is to develop efficient and economical chlorination route for EAFS extraction by using leaching process. Various parameters such as concentration of hydrochloric acid, particle size of steel slag, reaction time and reaction temperature are investigated to determine the optimum conditions. As a result, the dissolution rate can be determined by changing the parameters, such as concentration of hydrochloric acid, particle size of steel slag, reaction time and reaction temperature. The optimum conditions for dissolution rates for the leaching process is at 3.0 M hydrochloric acid, particle size of 1.18 mm, reaction time of 2.5 hour and the temperature of 90°C.

## 1 Introduction

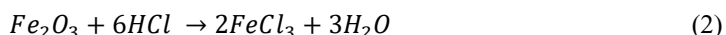
Steelmaking is the process of manufacturing steel by using iron ore and other elements. Two commonly used steelmaking process are basic oxygen and electric arc furnaces (EAF). BOF uses iron ore from the mining as the raw material for the process while the main raw material for EAF is steel scrap [1]. In Malaysia, EAF is widely used in steel mills. EAF slags are formed from the manufacturing of molten steel [2]. Ferric Chlorides could be extracted from EAF steel slags that are used to remove phosphate from wastewater which cause the eutrophication. When excessing phosphate enters the water, it can cause the microorganisms to grow faster because phosphate is a type of nutrients. With the increase in microorganisms such as algae, the oxygen composition in the water will decrease and therefore killing fish and reducing water quality [3].



Another uses of  $FeCl_3$  are to absorb moisture from the air when in crystalline form and it is usually supplied as a solution.  $FeCl_3$  was sold economically in water treatment

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industries. It is used as a coagulant for the turbidity removal and the removal of organic contaminants in drinking water.  $FeCl_3$  as a drinking water coagulant is inexpensive if maintain at a pH values around neutral pH [4]. Research shows that the presence of Ferric oxides, 33.34% have the highest weight composition in EAF steel slag followed by Calcium oxides, 27.22% and silicon oxides, 20.80% by using X-ray fluorescence (XRF) technique [5]. One of the effective ways to extract iron from EAF slag is leaching process by using hydrochloric acid.



## 2 Materials and methods

### 2.1 Materials

EAF steel slags are collected from Southern Steel Berhad, which is a steel manufacturing company in Malaysia. The samples were crushed and sieved into different size (0.3 mm, 0.5 mm, 0.6 mm, 1.0 mm and 1.18 mm). Sieved EAFS were washed with distilled water to remove impurities. The, EAFS were dried in oven at 110°C for 24 hrs and stored in heavy duty plastic bags until used [6]. For the chemical reagents preparation, distilled water was used to dilute and prepare all the hydrochloric acid solutions.

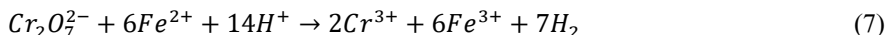
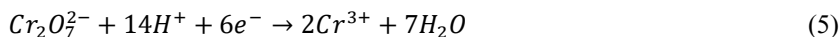
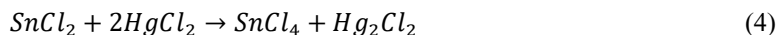
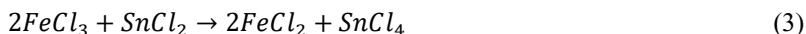
### 2.2 Methods

#### 2.2.1 Steel slag leaching with using Hydrochloric Acid

In order to determine the effect of Hydrochloric acid concentration on steel slag leaching, each sample of weight 1.0 gram is mixed with hydrochloric acid with the concentrations of 0.5 M, 1.0 M, 2.0 M, 3.0 M and 4.0 M for 24 hours. About different sample particle sizes, the sizes of the particles of 0.3 mm, 0.5 mm, 0.6 mm, 1.0 mm and 1.18 mm in 1 gram were tested using hydrochloric acid concentration of 4.0 M for 24 hours. Particle size of 1.0 mm in 1 gram is tested using hydrochloric acid concentration of 4.0 M with different heating temperatures of 60°C, 70°C, 80°C and 90°C for 1 hour each. Particle size of 1.0 mm in 1 gram was tested using hydrochloric acid concentration of 4.0 M with different reaction time of 0.5 hour, 1.0 hour, 1.5 hour, 2.0 hour and 2.5 hour. Therefore, all the sample solutions were placed on the orbital shakers for 2 hours to ensure the thorough mixture. Then, filter paper is used to filtrate the residue from each sample. Therefore, the residue is dried in oven for 60°C in 2 hours. The percentage of weight loss of EAF steel slag is measured and calculated. After that, dichromatic titration process was conducted to quantify the weight of ion  $Fe^{3+}$  presence after the leaching process.

#### 2.2.2 Titration process

Potassium dichromate is chosen for the oxidation of  $Fe^{2+}$  ion in the titration process. The samples were heated until boil and then  $SnCl_2$  solution is added into each of the sample until the yellow color disappears. After that, the solution is cooled and 10 ml of  $HgCl_2$  was added immediately. A white precipitate should appear. Furthermore, 5 ml of concentrated  $H_2SO_4$  and 7 ml of  $H_3PO_4$  was added to pre-reduced the solution. The solution is then diluted with distilled water and 8 drops of diphenylamine sulfonate indicator was added. After that, the solution is titrated with  $K_2Cr_2O_7$  solution until it turns from a color of blue-green to violet. The volume of the titrated  $K_2Cr_2O_7$  solution was recorded. The experiment is repeated two times to get the average value.



### 3 Results and discussions

#### 3.1 Material Characterization

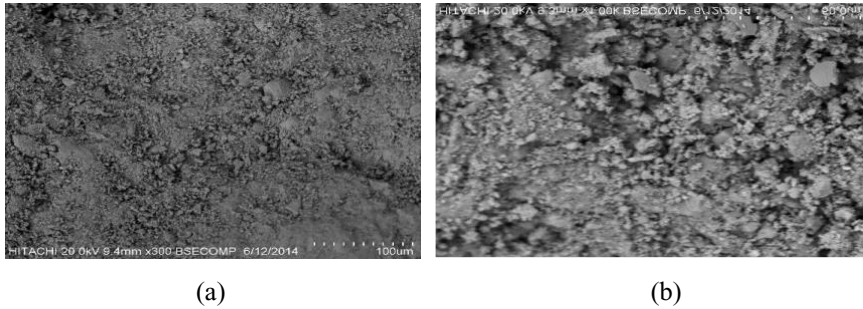
##### 3.1.1 Scanning Electron Microscopy (with Energy Dispersive X-ray Spectroscopy)

SEM is the sample scanning method that using focused beam of electron with high amount of energy in order to observe the topographical, morphological and compositional information of material in solid form [7]. A test on sample of EAF slag was conducted by using a scanning electron microscope (model S -3400N, HITACHI, Japan) to collect the morphology, crystalline structure and orientation of based material creating the EAF slag sample at an accelerating voltage of 20.0 kV, beam current of 50 mA and working distance of 9.0 mm – 10.0 mm with utilization of backscattered electrons (BSE) set objectively with aperture of 3 [7]. Images at low magnification (300×) and high magnification (1000×) were captured and the micrographics of the non-reacted EAF slag are presented in Fig. 1. Areas with lighter color contain a higher proportion of Fe, Mg, Mn and Al while the shady areas contain a higher proportion of Ca and Si [8].

EAF steel slag of 2 samples was analysed by using energy-dispersive x-ray spectroscopy. The primary elements found in the sample A included 29.93% CaO<sub>2</sub>, 22.01% Fe<sub>2</sub>O<sub>3</sub> and 21.41% SiO<sub>2</sub>. On the other hand, sample B included 23.29% Fe<sub>2</sub>O<sub>3</sub>, 22.76% SiO<sub>2</sub> and 21.1% CaO<sub>2</sub>. It is concluded that the main element in EAF steel slag is Ca, Fe and Si.

##### 3.1.2 X-Ray Diffraction

The studies on mineralogical composition on steel slag were conducted by several researchers. Many overlapping peaks appear in XRD patterns indicates the presence of crystalline phases in the steel slag [9]. The more crystalline structured compound has its XRD peaks more easily to be identified [10]. The common mineral phases present in steel slag include two- and three-component compositions of the type CaO-SiO<sub>2</sub>, CaO-Fe<sub>2</sub>O<sub>3</sub>, CaO-MgO-SiO<sub>2</sub>, MgO-FeO-SiO<sub>2</sub> and CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> [9,11], and the most highly represented minerals in slag are dicalcium and tricalcium silicates, while different aluminates and silicates are likely to appear as well [12].



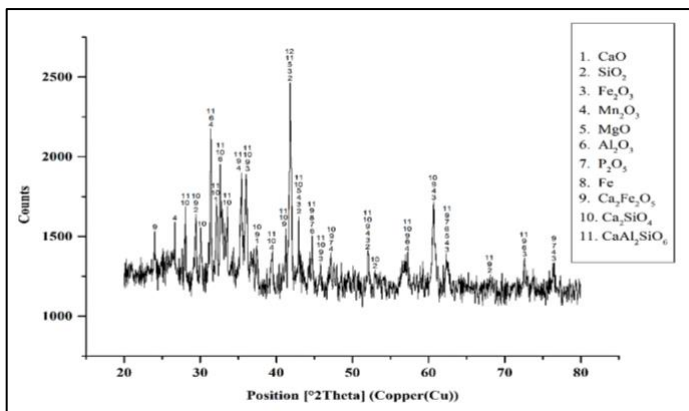
**Fig 1.** SEM image on unreacted EAFS at magnification of (a) 300x and (b) 1000x.

The XRD analysis of unreacted EAFS is shown in Fig. 2. The diffraction pattern is heterogeneous, consisting of a mixture of crystalline phases [13]. The analysis identifies calcium oxide (CaO), silicon oxide (SiO<sub>2</sub>), hematite (Fe<sub>2</sub>O<sub>3</sub>), manganese (III) oxide (Mn<sub>2</sub>O<sub>3</sub>), magnesium oxide (MgO), aluminium oxide (Al<sub>2</sub>O<sub>3</sub>), phosphorus oxide (P<sub>2</sub>O<sub>5</sub>), metallic iron (Fe), srebrodolskite (Ca<sub>2</sub>Fe<sub>2</sub>O<sub>5</sub>), dicalcium silicate (Ca<sub>2</sub>SiO<sub>4</sub>) and pyroxene (CaAl<sub>2</sub>SiO<sub>6</sub>).

### 3.2 Effect of hydrochloric acid concentration

The effects of HCl concentrations on the dissolution EAF slag were studied at varying concentration of HCl. It is observed that the dissolution of EAF slag increased with increasing concentration of HCl at constant temperature and time. When the concentration of HCl increased from 0.5 M to 4.0 M, the dissolution increased 17.06 %. The optimum concentration of HCl for the leaching process is found to be 3.0 M which 77.41 % was leached. The dissolution rate of EAF slag can be calculated by equation (9).

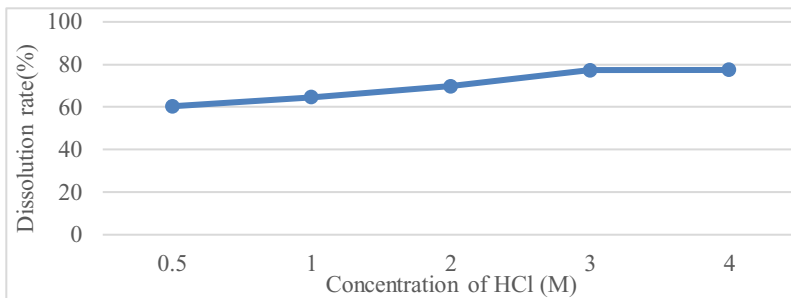
$$\alpha (0.5 \text{ M}) = \frac{\Delta \text{mass of EAFS (gram)}}{\text{initial mass of EAFS (gram)}} \quad (9)$$



**Fig. 2.** XRD pattern on unreacted EAFS.

**Table 1.** Dissolution percentage and weight of Fe<sup>3+</sup> on varying HCl concentrations.

Concentration of HCl (M)	Dissolution (%)	Weight of Fe <sup>3+</sup> (gram)
0.5	60.35	0.14559
1.0	64.58	0.16886
2.0	69.74	0.15975
3.0	77.41	0.20674
4.0	77.22	0.18837



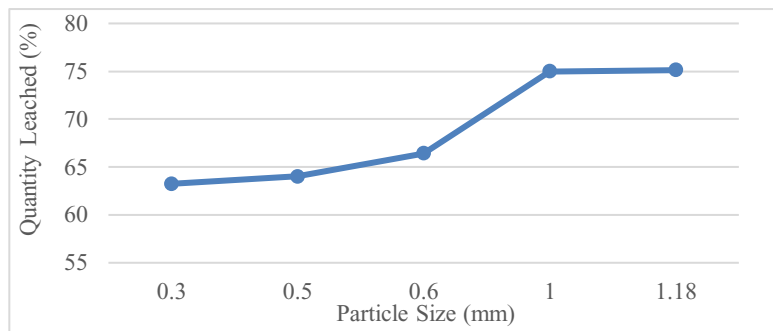
**Fig. 3.** Dissolution rate against concentration of HCl.

### 3.3 Effect of particle sizes

The effect of particle sizes was studied at varying particle sizes. It is observed that the dissolution of EAF slag increases with increasing particle size at constant HCl concentration, which is 3M. When the particle size increased from 0.3 mm to 1.18 mm, the dissolution increased 11.87%. The optimum particle size for the leaching process is found to be 1.18 mm which 75.12 % was leached.

**Table 2.** Dissolution percentage and weight of Fe<sup>3+</sup> on varying particle size.

Particle size (mm)	Dissolution (%)	Weight of Fe <sup>3+</sup> (gram)
0.3	63.25	0.13213
0.5	64.01	0.13255
0.6	66.43	0.12091
1.0	74.98	0.13684
1.18	75.12	0.14045



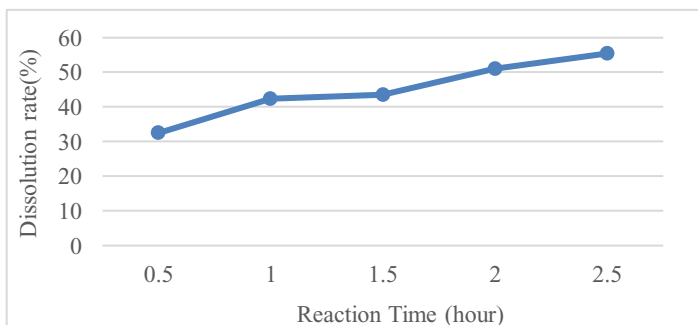
**Fig. 4.** Percentage of steel slag leached against particle sizes.

### 3.4 Effect of reaction time

The effect of reaction time was studied at varying reaction time. It is observed that the dissolution of EAF slag increases with increasing reaction time at constant HCl concentration, which is 3M. When the reaction time increased from 0.5 hour to 2.5 hour, the dissolution increased 22.95%. The optimum reaction time for the leaching process is found to be 2.5 hour which 55.45 % was leached.

**Table 3.** Dissolution percentage and weight of Fe<sup>3+</sup> on varying reaction time.

Reaction time (hour)	Dissolution (%)	Weight of Fe <sup>3+</sup> (gram)
0.5	32.50	0.05726
1.0	42.35	0.0704
1.5	43.48	0.0963
2.0	51.02	0.0698
2.5	55.45	0.0730



**Fig. 5.** Dissolution rate against reaction time.

### 3.5 Effect of reaction temperature

The effect of reaction temperature was studied at varying reaction temperature. It is observed that the dissolution of EAF slag increases with increasing reaction temperature at constant HCl concentration, which is 3M. When the reaction temperature increased from

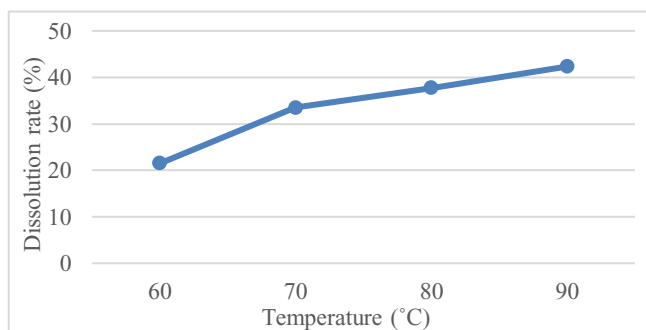
60°C to 90°C, the dissolution increased 20.84%. The optimum reaction temperature for the leaching process is found to be 90°C which 42.34 % was leached.

## 4 Conclusions

By using material characterization methods, it shows that EAF steel slag containing a suitable amount of Fe ions. It is proven that the leaching of EAF steel slag contains a suitable amount of  $FeCl_3$  for further usage such as wastewater treatment. In this experiment, different variables that affect the concentration of  $Fe^{3+}$  are chosen to be the focus when conducting the EAF slag leaching process. The highest dissolution percentage of the leaching process are 77.14%, 75.12%, 55.45% and 42.34% with HCl concentration, particle size, reaction time and reaction temperature respectively. The highest weights of  $Fe^{3+}$  leached are 0.20674 gram, 0.14045 gram, 0.073 gram and 0.07141 gram for HCl concentration, particle size, reaction time and reaction temperature respectively. It can be observed that the optimum conditions for the leaching process is at 4.0 M HCl, particle size of 1.18 mm, reaction time of 2.5 hour and reaction temperature of 90°C.

**Table 4:** Dissolution percentage and weight of  $Fe^{3+}$  on varying reaction temperature.

Reaction temperature (°C)	Dissolution (%)	Weight of $Fe^{3+}$ (gram)
60	21.50	0.0243
70	33.48	0.02841
80	37.70	0.05714
90	42.34	0.07141



**Fig 6.** Dissolution rate against reaction temperature.

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