Solid State Science and Technology, Vol. 24, No 2 (2016) 45-51
ISSN 0128-7389 | *http://journal.masshp.net*

ELEMENTAL ANALYSIS AND IR BAND CHARACTERISTICS OF α-Fe_2O_3 AND $BaFe_{12}O_{19}$ STEEL WASTE PRODUCT BASED

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ABSTRACT

This project focused on the elemental analysis and IR band characteristic of α-Fe_2O_3 derived from recycled steel waste product. The steel waste flakes were ball milling for several hours to form a fine powder. The steel waste powder had been purified by using impurity separation technique and magnetic separation technique. The purified steel waste powder then oxidized at 500 °C to form hematite (Fe_2O_3). The hematite were used to synthesize $BaFe_{12}O_{19}$ by using salt-melt method. The samples were characterized using X-ray Fluorescence (XRF), Fourier Transform Infrared spectroscopy (FTIR), X-ray diffraction (XRD) and energy-dispersive X-ray analysis (EDAX). The XRF and FTIR results show the formation of Fe_2O_3, the IR characteristic bands of Fe_2O_3 and single phase $BaFe_{12}O_{19}$ is obtained from recycled steel waste product.

Keywords: mill scales; hematite (α-Fe_2O_3); barium ferrites ($BaFe_{12}O_{19}$); XRF; XRD; FTIR;

INTRODUCTION

There are about 500 kg/ton of solid wastes of different nature are produced in steelmaking process[1]. Some of the wastes are high with iron content, such as mill scales. Mill scales are useful byproduct of steel making process due to its richness of iron contents (~72%)[2], stable chemical composition and low impurities[3]. Mill scales contain three different types of iron oxides such as hematite, wuestite and magnetite [2][4]. Primarily, the mill scales consists of magnetite, Fe_3O_4, the characteristic blue-gray steely color. The thin outer mill scales film is a form of hematite, Fe_2O_3, and the inner portion of the magnetite contains fine metal grains of black wuestite, FeO, which contribute to the roughness of descaled metal. The mill scale flakes image is shown in Figure 1. Mill scale is found on hot-rolled steel products processed in an atmosphere. At hot rolling temperatures, an inner layer of wuestite, FeO, exists between the steel and magnetite. At elevated temperatures, FeO constitutes about 85 % of the scale thickness,

Fe_3O_4 about 10 to 15% and Fe_2O_3 about 0.5 to 2%. During slow cooling (below 833.15 °K) of hot-rolled coils of hot-rolled strip and heavy shapes, most of the FeO is transformed to Fe and Fe_3O_4, and the latter oxide is predominant after cooling.

Barium ferrites ($BaFe_{12}O_{19}$) is one of fascinating materials due to its high magnetic anisotropy, high coercivity, excellent chemical high saturation magnetization, and resistance properties[5][6][7]. These materials have been broadly used in various applications as magnetic recording media, permanent magnet, magnetic tapes, microwave devices and motor [8]. There are many methods used to synthesis $BaFe_{12}O_{19}$, such as high energy ball milling [9], sol gel[10][11], hydrothermal technique [12] and co-precipitation [13]. In this study we present ammonium nitrate salt melt technique [14] to synthesis $BaFe_{12}O_{19}$. The method is based on formation of metal nitrates by dissolving metal oxide in melting ammonium nitrates. An oxygen rich precursor for the formation of complex oxides is obtained by heating of the mixture of ammonium nitrate solution and iron oxide at 260 °C. Here, the separation of iron oxide from mill scales and the synthesis of $BaFe_{12}O_{19}$ powder using ammonium nitrate salt melt technique will be discussed.

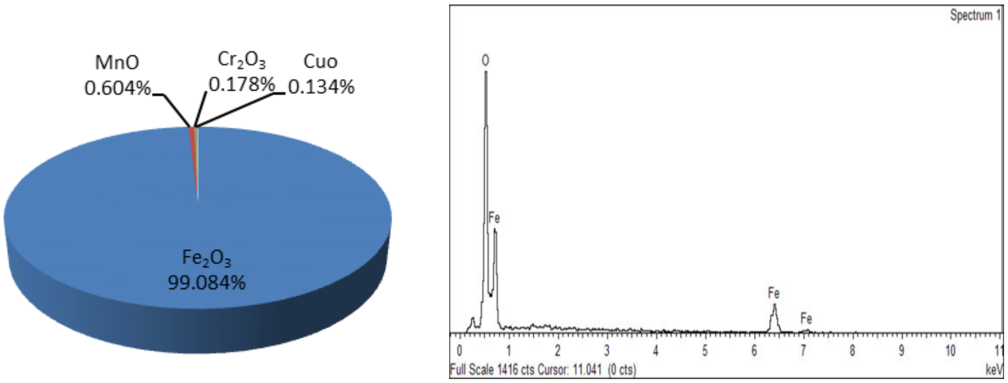
Figure 1: Mill scales flakes from steel industry

EXPERIMENTAL

To obtain fine powder, the mill scales samples were milled for a several hours. The impurities in mill scales were separated using magnetic separation technique. The magnetic materials were isolated from non-magnetic materials using Curie separation technique. To obtain α-Fe_2O_3, the magnetic powder was then oxidized at 500°C. To synthesis pure $BaFe_{12}O_{19}$, barium carbonate ($BaCO_3$), iron (III) oxide α-Fe_2O_3 obtained from mill scales and ammonium nitrate (NH_4NO_3) will be used as starting materials. The ammonium nitrate salt was weighed and poured into a transparent beaker then melted at 170°C. The mixture of the $BaCO_3$ and α-Fe_2O_3 were added and stirred until a thick solution was formed. The temperature was increased to 260 °C and the solution was left on hotplate for one day until ash like reddish precursor was produced. The powder was grind and crush using a mortar before heated 1300 ^{o}C in air using a high temperature box furnace. The powder were characterized using X-ray diffraction (XRD), fourier transform infrared (FTIR) and X-ray fluorescence (XRF). X-ray powder diffraction data were collected in a Philips Expert PW3040 diffractometer operating at 40 kV/30 mA using Cu Kα (0.154 nm) range from 10° to 80°. Infrared spectra (280–4000 cm^{-1}) is recorded using an FTIR spectrometer (Perkin Elmer model 1650). The particle morphology was examined by a field emission electron microscope (FESEM).

RESULTS AND DISCUSSION

Figure 2(a) shows the XRF analysis of the mill scales powder after oxidized at 500 °C. The results show an oxidized mill scales contain 99.084 % of α-Fe_2O_3, and the presence of small amount impurities of MnO (0.604 %), Cr_2O_3 (0.178 %), and CuO (0.134%). This results are agreed as reported by Azis *et al*[2]. The EDAX results (Figure 2(b)) also show the high contains of Fe and O indicates the element present in the sample which showed that it contains Fe_2O_3.



(a) *(b)*

Figure 2: (a) XRF analysis of mill scales after separation and (b) EDAX analysis of hematite after oxidation at 500°C

Figure 3 shows the XRD spectrum for (a) oxidized mill scale and (b) sintered $BaFe_{12}O_{19}$ powders. For powder (a), the XRD ICSD Ref. No. 98-002-8843 for standard hematite (α-Fe_2O_3). The Fe_2O_3 cystal system hexagonal *R-3c* with lattice parameters a = 5.0340 Å, b = 5.0340 Å and c =13.7460 Å. From XRD pattern, it is show the presence of small amount of magnetite, Fe_3O_4 after oxidation process. For the $BaFe_{12}O_{19}$ powder (Figure 4 (b)), the XRD spectrum was indexed according to ICSD Ref. No. 98-001-9939. The XRD results show the single phase $BaFe_{12}O_{19}$ hexagonal structure was detected without any other impurities for powders calcined at 1300 °C for 6 h, which means that no intermediate phases are appeared. It can be found that sintering temperature at 1300 °C attribute the peaks width becomes narrower, indicating that the mean crystalline size of synthesized ferrites increased.

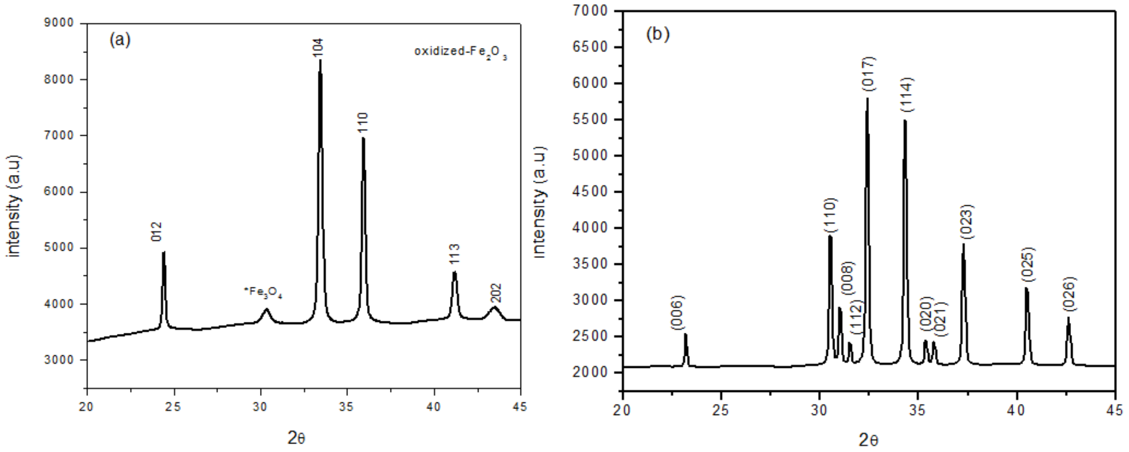


Figure 3: XRD patterns of the (a) oxidized mill scales at 500 ^{o}C and (b) BaFe$_{12}$O$_{19}$ powders sintered at 1300 ^{o}C

Figure 4(a) shows FTIR spectra for raw mill scales and oxidizes powder, while Figure 4(b) shows the FTIR spectra for the burnt precursor, and the powders calcined at 1300 ^{o}C. Assignment of bands is given in Table 1. From Figure 4(a), the FTIR show the vibration characteristic of transmittance band at 552.21 cm^{-1} and 539.23 cm^{-1} are attributed to Fe – O vibrations. The peak of 445.6 cm^{-1} of transmittance band vibration is assigned to chemical bonding of Fe^{3+}– O^{2-}. From Figure 4(b), the IR transmittance band of burnt precursor of BaFe$_{12}$O$_{19}$ shows the vibrations in the range of 325 cm^{-1}, 433 cm^{-1}, 543 cm^{-1}, 728 cm^{-1}, 815 cm^{-1}, 1328 cm^{-1}, 1413 cm^{-1}, 1497 cm^{-1} and 1775 cm^{-1}. Mahmoud et.*al* [15] reported the change in the band position ν_3-ν_1 is due to the change in the Fe^{3+} – O^{2-} inter-nuclear distance of the octahedral and tetrahedral sites [15]. Waldron [16] and Hafner[17] attributed the ν_3 to the intrinsic vibration of the tetrahedral groups, whereas ν_1 is due to is due to octahedral groups. A small band ν_1 in the range of 325.11 cm^{-1} is assigned to the divalent octahedral metal ions – oxygen ion complex [18]. As for the burnt precursor (Figure 3b), there are a series of new bands at 1497 cm^{-1}, 1413 cm^{-1}, 815 cm^{-1} and 728 cm^{-1} which come from barium carbonate and the transmittance bands of NO$_3$ ions, –NH$_2$ and COO group in the FTIR spectra of the burnt precursor. The characteristic band at 1775 cm^{-1} result from the anti-symmetrical and symmetrical stretching vibration bands of COO related to ammonium nitrate (NH$_4$NO$_3$). From Figure 3(b), all bands at ν_4 to ν_5 belong to the metal–oxygen stretching vibrations of BaFe$_{12}$O$_{19}$ hexaferrite. No other band is observed, suggesting that single BaFe$_{12}$O$_{19}$ hexaferrite powders be obtained when calcined at 1300^{o}C, which can be identified by the XRD results (Figure 3(b)). It is also observed that, ν_6 shifts disappeared with sintering temperature. Therefore, the sintering process can be described as a thermally induced anionic redox reaction of the nitrate precursor in which nitrate ions act as oxidant. Sintering process also attributed to shifting of Fe^{3+} ions toward oxygen ions with increasing the particle size, then the average bond length increases.

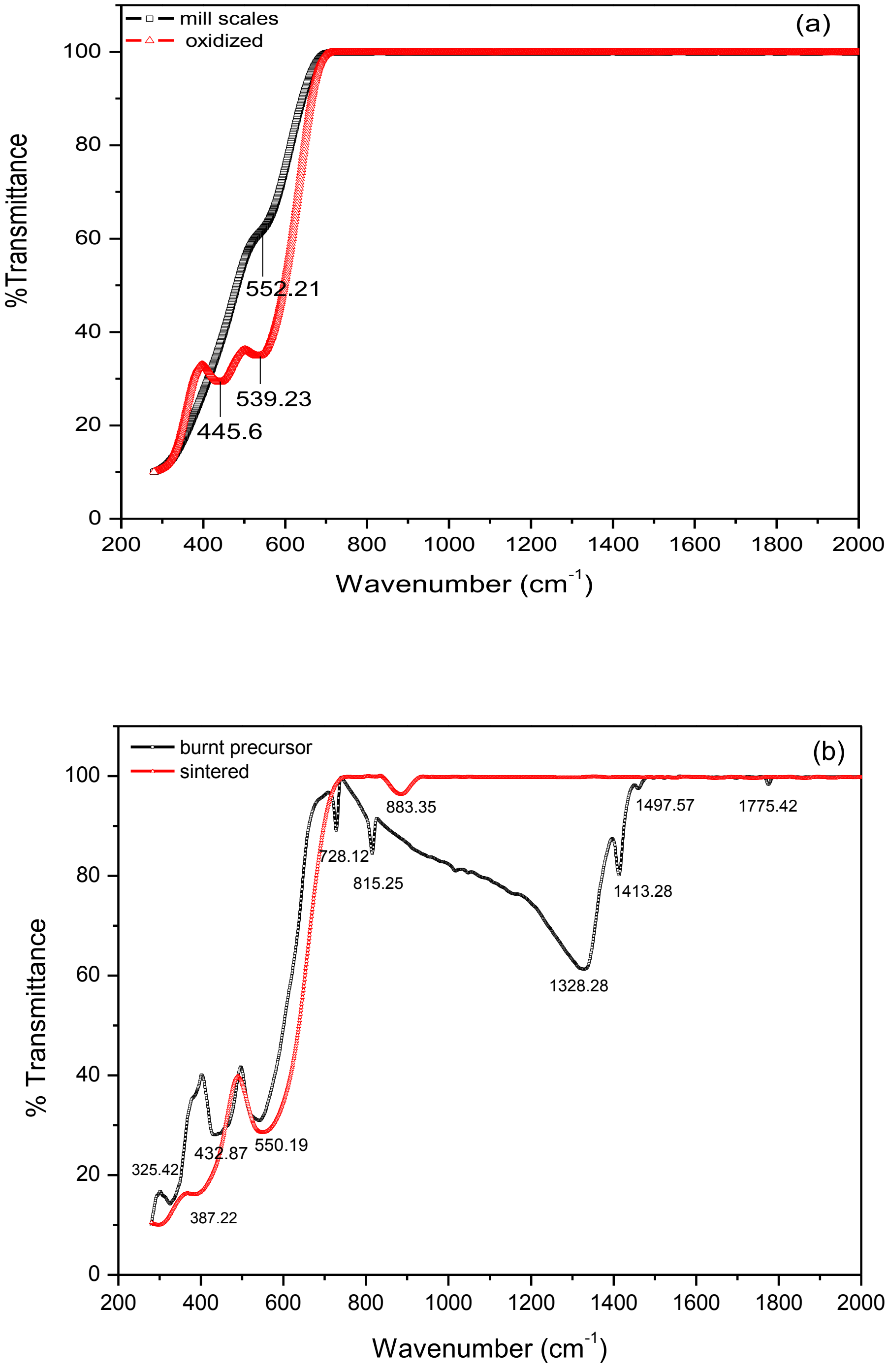


Figure 4: FTIR analysis of (a) hematite before and after oxidized; and (b) $BaFe_{12}O_{19}$ powder before and after sintered

Table 1: IR transmittance bands, ν for $\alpha-Fe_2O_3$ and barium ferrite powder

Powder	ν_1	ν_2	ν_3	ν_4	ν_5	ν_6
Raw mill scales	-	-	552.21	-	-	-
Oxidized, α-Fe_2O_3	-	445.60	539.23	-	-	-
Burnt precursor	325.42	432.87	543.75	728.12	815.25	1328.28 1413.28 1497.53 1775.43
Sintered $BaFe_{12}O_{19}$	387.22	-	550.19	-	883.35	-

CONCLUSION

The low-cost purification process of separation technique produced high purity of $\alpha-Fe_2O_3$ from steel waste product, a mill scales, as confirmed by XRF and EDAX. The barium ferrites samples were prepared derived by steel waste product and successfully characterized. The elemental analysis and infrared (IR) characteristic bands are successfully analyzed. The single phase of $BaFe_{12}O_{19}$ derived from low cost recycling steel waste product has been obtained and suitable for ferrites technology applications.

ACKNOWLEDGMENTS

The authors would like to thank to the Putra Research Grants, Universiti Putra Malaysia, vot no. 9433965 and 9438736 for the financial assistance. The authors also thank to the Department of Physics and the Materials, Synthesis and Characterization Laboratories (MSCL) ITMA, UPM for the measurements facilities.

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