International Journal of Applied Science and Engineering, 8(1): 11-18, June 2020

DOI: 10.30954/2322-0465.1.2020.2

Hydrothermal Synthesis and Spectroscopic Characterization of one-dimentional Rod-like Hematite (α-Fe_2O_3) Mineral in Alkaline Medium

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ABSTRACT

Rod-like α-Fe_2O_3 was synthesized by hydrothermal method and characterized using spectroscopic methods of analysis. X-ray diffraction (XRD), Fourier transform infrared (FTIR), X-field emission scanning electron microscopy (FESEM), elemental diffraction X- ray (EDX), transmission electron microscopy (TEM) and dynamic light scattering (DLS) methods were used to characterize the synthesized mineral. XRD analysis shows that the single-crystalline sample indexed as the pure rhombohedra α-Fe_2O_3 material was formed while the DLS and zeta potential analysis indicated average particles sizes of about 1300.9 nm and 31.2 mV respectively. The morphology and shape of the synthesized mineral was studied with FESEM and TEM while the elemental analysis was done with EDX. FESEM and TEM analysis show that the synthesized **α-**Fe_2O_3 minerals were rod-like in shape while EDX analysis gave a good composition of the elements of the synthesized material. Therefore, this method may be applied to synthesize other forms of iron minerals or other inorganic materials at industrial scale especially when rod-like materials are needed.

Keywords: Synthesis, Hematite, characterization, rod-like

Iron based minerals have been used for centuries in many applications such as in science, technology and engineering. The use of iron oxides, hydroxides and oxohydroxides in different scales or sizes has been applied in medicine, as coloring pigment and in environmental remediation. Other areas where iron minerals have been utilized include microbial sciences, imaging systems, pharmaceutical sciences, biological products, civil engineering, immobilization techniques, and chemical industries. Among these

Received: 17 Jan., 2020 **Revised:** 21 Apr., 2020 **Accepted:** 23 May, 2020

iron based minerals, α-Fe_2O_3 is a major component of various types of sediments, soils and iron ores and has gained prominent use as catalysts, electrodes, gas sensors, magnetic materials, photo catalyst, corrosion protective paints and for red pigment (1). Fe_2O_3 has shown a special property in the adsorption of metal ion wastewater in different research work (2). As a result of its wide areas of applications, efforts have been made by various researchers to find the easiest and fastest method to synthesize it. Some of the methods that have been applied to synthesize α-Fe_2O_3 include hydrolysis (2), co-precipitation [3, 4, 5], electrical explosion of iron [6], sol-gel method [7] and others. These synthesis methods have been used as templates by many researchers with many disadvantages including low yield of pure α-Fe_2O_3 and multi-dimensional synthesized materials.

In this research work, we have designed an easy and simple hydrothermal approach for synthesizing pure rod-like α-Fe_2O_3 in an alkaline medium using a hydrated salt of iron without any hard template or surfactants.

MATERIALS AND METHODS

Rod-like Fe_2O_3 was synthesized with $Fe(NO_3)2.9H_2O$ (98.0%) and NaOH (97.0 %). A 20 ml of ferric nitrate solution (0.2 M) with was prepared in a beaker with constant stirring at 600 rpm to a clear solution. Similarly, a 10 ml solution of 2.5 M NaOH was prepared in a beaker and stirred till the NaOH pellets completely dissolved in solution. The alkaline solution was gradually added to the nitrate solution in the beaker with continuous stirring at the same speed. After complete addition of the NaOH solution, the stirring is allowed to continue for 5 minutes and then transferred to the autoclave and water was added to make up about 80 % of the volume of the autoclave. The autoclave and its contents were transferred to an oven that was earlier put on to attain a temperature of 160 °C for hydrothermal reaction for 46 hours including the 1 hour that the autoclave was allowed to attain the temperature of the oven environment. After the reaction time, the autoclave was brought out and allowed to cool naturally to prevent change in the composition and morphology of the content. After cooling to the ambient temperature, the precipitate was filtered and washed for several times with ethanol and doubly distilled water thoroughly till free of nitrate ions and other impurities and then dried in an oven at 100 °C for 20 hours and the synthesized material was stored in a reagent bottle while a little quantity was taken for XRD, FTIR, FESEM, EDX, TEM, DLS and Zeta potential analysis.

Spectroscopic Characterization of α-Fe_2O_3

The phase identification of the synthesized mineral was characterized by X-ray diffractometer (XRD) with type empyrean series 2. The XRD pattern were recorded with 2θ in the range of 10 – 90 °C with panalytical X-Pert high score (POM) equipped with Cu-Kα (λ = 1.54606 Å) at a scan rate time of 24.765 seconds with a generating set of 40 mA and 45 Kv at a temperature of 25 °C. The morphology of the synthesized material was characterized by field emission scanning electron microscopy (FESEM) with type ZEISS evolution series 2000, 25 kV and transmission electron microscopy (TEM) with type tecnai G2 20 twin, FEI Netherland, 80 kV. The Fe and O elemental analysis of the samples was performed by energy dispersive spectroscopy (EDS) type Anton paar Lenovo litesizier 500. All the measurements were carried out at room temperature.

Table 1: List of XRD peaks for the synthesized rod-like α-Fe_2O_3 by hydrothermal method

Pos. [°2θ]	Height [cts]	FWHM Left [°2θ]	d-spacing [Å]	Rel. Int. [%]	Tip Width	Matched by
17.7153	5.66	0.4488	5.00260	0.38	0.5386	98-016-1283
24.2008	402.68	0.2856	3.67464	27.36	0.3427	98-016-1283
33.1810	1471.92	0.2652	2.69779	100.00	0.3182	98-016-1283
35.6822	1174.09	0.2448	2.51421	79.77	0.2938	98-016-1283
40.8855	429.13	0.2448	2.20544	29.15	0.2938	98-016-1283
49.4759	564.92	0.2856	1.84075	38.38	0.3427	98-016-1283
54.0560	845.28	0.2652	1.69510	57.43	0.3182	98-016-1283
62.4567	486.36	0.2448	1.48577	33.04	0.2938	98-016-1283
64.0193	539.70	0.2856	1.45322	36.67	0.3427	98-016-1283
71.9109	123.88	0.4896	1.31192	8.42	0.5875	98-016-1283

RESULTS AND DISCUSSION

X-ray diffraction spectroscopy (XRD) of the synthesized α-Fe_2O_3 material by hydrothermal method at 45 Kv and 40 Am was used to identify crystalline phases of the sample. Fig. 1a shows the XRD pattern of the powdered material which shows that α-Fe_2O_3 was the only phase present.

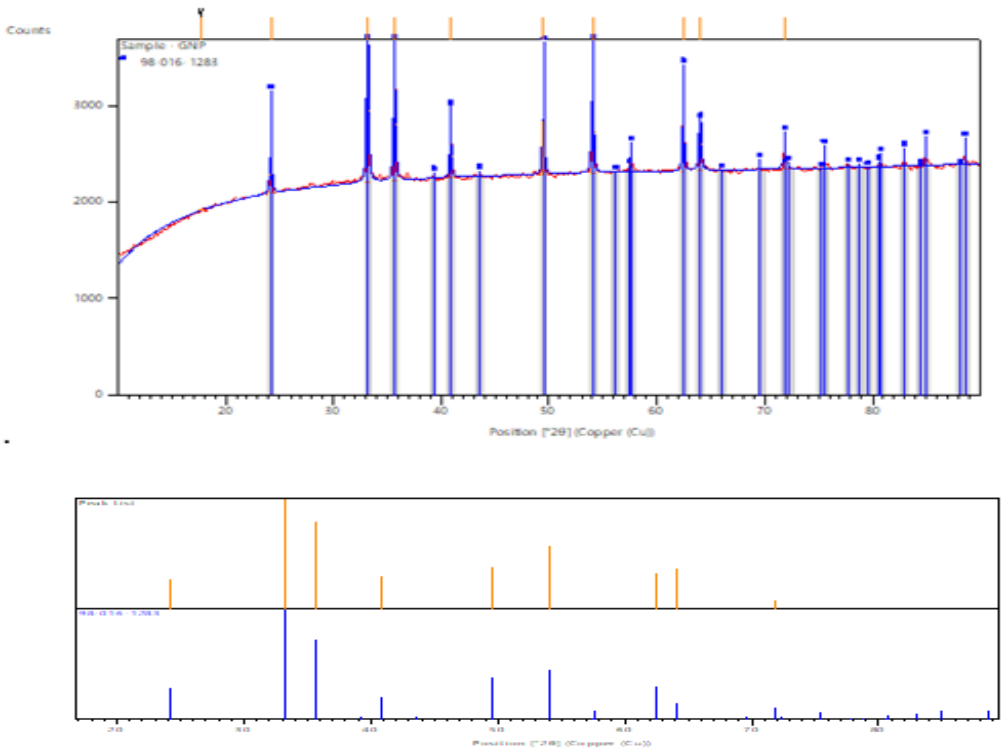

Fig. 1: (a) XRD pattern **(b)** Plot of identified phases of rod-like α-Fe_2O_3 synthesized by hydrothermal method

The XRD peaks at 2Θ = 17.7153, 24.2008, 33.1810, 35.6822, 40.8855, 49.4759, 54.0560, 62.4567, 64.0193 and 71.9109 represented in Table 1 correspond to the crystal planes (012), (104), (110), (113), (024), (116), (018), (214), (300) and (101) indexed as pure rhombohedra structure of α-Fe_2O_3 (*a* = 5.0380 A°, *c* = 13.7720 A°) which is identified using the standard data file No. 98-016-1283 (JCPDS). The plot of the identified phases of the prepared material is also represented in Fig. 1b. The peaks in the identifier plot correspond to peaks of pure phase of α-Fe_2O_3 mineral [8, 9, 10].

The surface morphology of the synthesized material was studied using field emission scanning electron microscopy (FESEM) method of analysis. The images of the α-Fe_2O_3 material as represented in Fig. 2 shows that α-Fe_2O_3 samples prepared by hydrothermal method are rod-like in nature with little agglomeration.

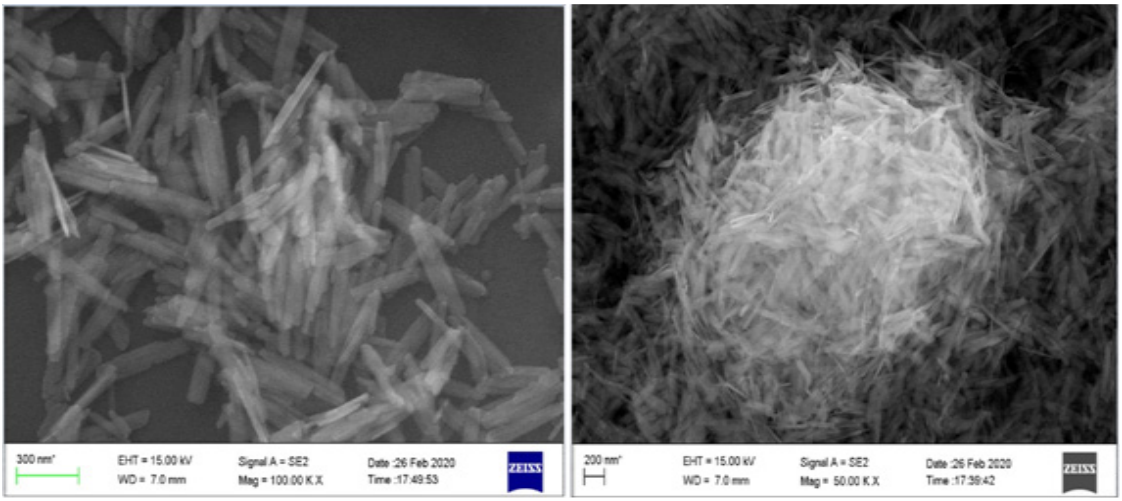

Fig. 2: FESEM pattern for the rod-like α-Fe_2O_3 synthesized by hydrothermal method

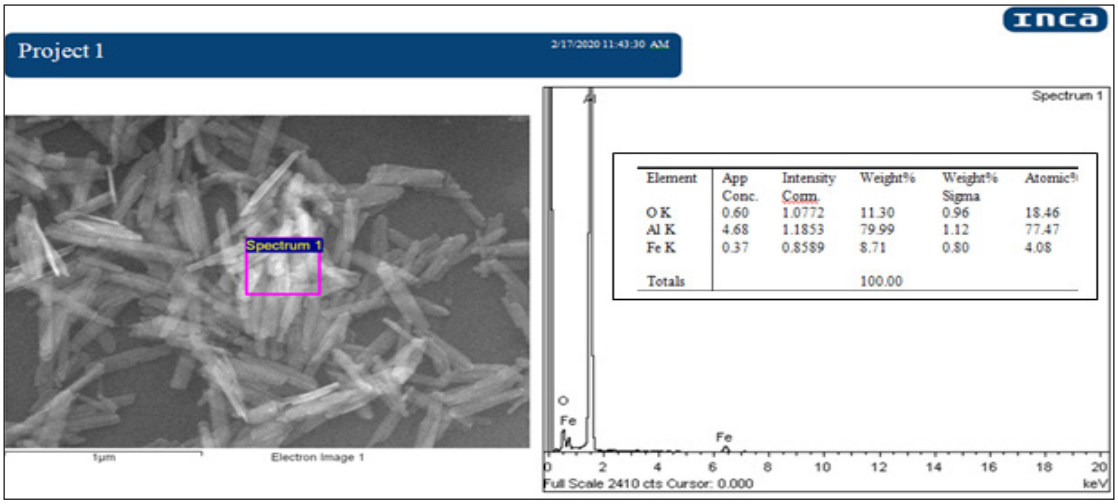

Fig. 3: EDS analysis for the rod-like α-Fe_2O_3 synthesized by hydrothermal method

The transmission electron microscopic (TEM) method of analysis was carried out after the synthesis of α-Fe_2O_3 mineral to confirm the growth pattern and the distribution of the crystallites sample. Figure 4 shows that the TEM image of the synthesized material by hydrothermal route is rod-like in structure which confirms the result obtained by FESEM analysis above. It can also be seen that the materials were prepared with less aggregation.

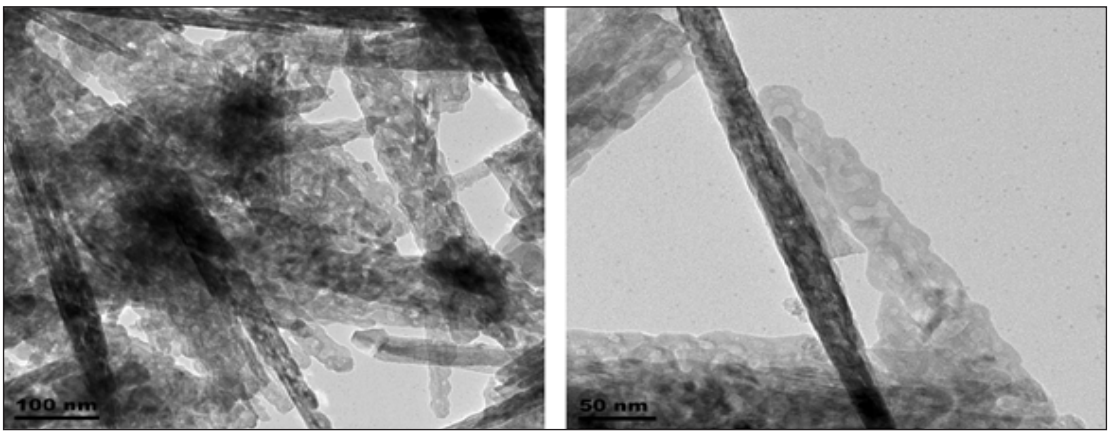

Fig. 4: TEM images for the rod-like α-Fe_2O_3 synthesized by hydrothermal method

Elemental diffraction spectroscopy (EDS) was used to analyze the chemical composition of the material prepared by hydrothermal method under FESEM analysis. Fig. 3 shows the EDS analysis of the synthesized rod-like α-Fe_2O_3 which confirms the presence of Fe and O with weight percent. The EDS result shows the peaks of iron and oxygen without any other elements present except the peak due to aluminium as a result of aluminium foil used to prepare the samples before analysis. The EDS result shows the degree of purity of the rod-like α-Fe_2O_3 prepared by hydrothermal method.

The mean size of the ordered rod-like α-Fe_2O_3 prepared by this method as represented in Fig. 5 was determined by using dynamic light scattering (DLS) method and was found to be about 1300.9 nm with a zeta potential of 31.20 mV as represented in Fig. 6.

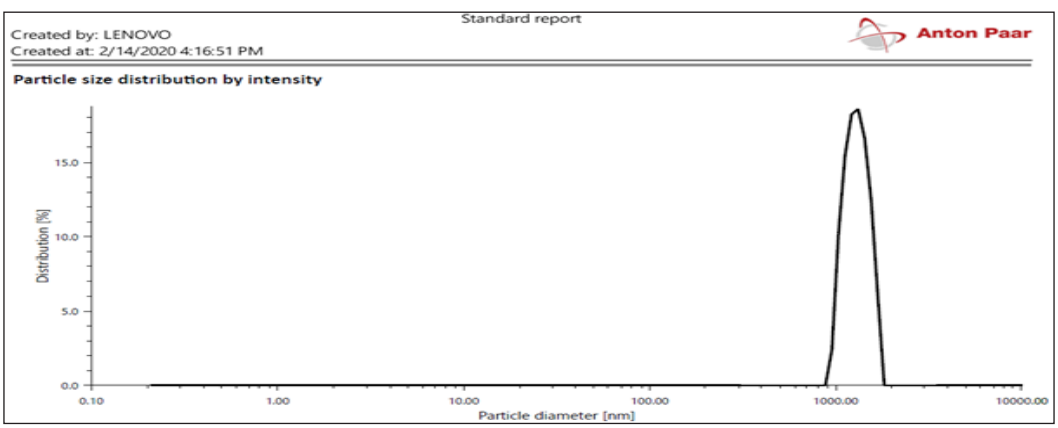

Fig. 5: DLS analysis for the rod-like α-Fe_2O_3 synthesized by hydrothermal method

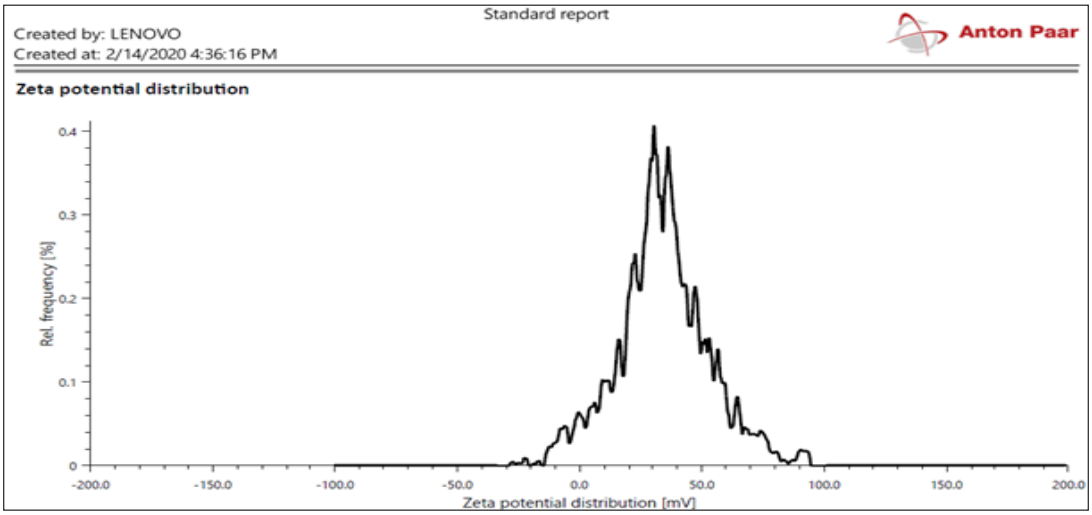

Fig. 5: Zeta potential analysis for the rod-like α-Fe_2O_3 synthesized by hydrothermal method

CONCLUSIONS

Simple hydrothermal method has been successfully carried out to synthesize α-Fe_2O_3. XRD pattern shows rod-like structure of α-Fe_2O_3. A SEM image clearly shows that the morphology of the synthesized material is rod-like in shaped with less agglomerate. The TEM image also shows that the synthesized α-Fe_2O_3 prepared by hydrothermal route has an average diameter of about 1300.9 nm and a zeta potential of 31.20 mV with less aggregation. The EDS result shows only peaks of iron (Fe) and oxygen (O_2) in the absence of other impurities in the synthesized rod-like α-Fe_2O_3.

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