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Review Article

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Hydrothermal Synthesis of Mixed Nanocomposite of α-[Fe_2O_3-FeOOH] from Iron Nitrate Salt in Teflon Autoclave

John Godwin

Department of Chemistry, Kogi State College of Education (Technical), P.O.B 242 Kabba, Nigeria

Corresponding author
John Godwin, Department of Chemistry, Kogi State College of Education (Technical), P.O.B 242 Kabba, Nigeria.

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ABSTRACT
Alpha type Fe2O3-FeOOH nanocomposite was synthesized by hydrothermal method and characterized using some spectroscopic methods of analysis. X-ray diffraction (XRD) scanning electron microscopy SEM), elemental diffraction X- ray (EDX) and transmission electron microscopy (TEM) methods were used to characterize the synthesized nanocomposite. XRD analysis shows that the single-crystalline phase of the nanomaterial was formed while the zeta potential analysis indicated 20.0 mV respectively. The morphology and shape of the synthesized mineral was studied with SEM and TEM while the elemental analysis was done with EDX. SEM and TEM analysis show that the synthesized alpha Fe_2O_3-FeOOH nanomaterils were rod-like in shape while EDX analysis gave a good composition of the elements of the synthesized material. The average crystal size of the synthesized α-[Fe_2O_3-FeOOH] estimated by Debye-Scherer equation to be 56.78 nm which confirms the nanosize of the material.

Keywords: Synthesis, Characterization, Hydrothermal, Nanocomposite, Alpha

Introduction

Nanotechnology and fabrication of materials have become research areas that are presently receiving a great attention by many researchers around the world. This area is still undergoing exploitation because of the enhanced chemical and physical properties of nanomaterials compared to the bulk materials. The properties of materials that have been enhanced when they are brought to the nanoscale include particle size, shape and surface chemistry [1]. Iron based nanomaterials such as α-Fe_2O_3 and α-FeOOH have found applications in various ways due to their exceptional physical, chemical and biological properties. These have made these materials of great interest and value in nanotechnology, science and medicine [2]. Some of the areas of applications of iron-based nanomaterials include nanodisc, electrochemical sensors, photocatalyst, adsorption and many others [3-7]. Both α-Fe_2O_3 and α-FeOOH have been applied in various fields as separate entities and in mixed forms to form composite in divers fabricated materials and have shown excellent properties and functionalities in comparison with their separate or individual applications [8-10]. Separate consideration of α-Fe_2O_3, an n-type semi-conductor with a band gap of 2.1 eV, has shown that it has been applied in gas sensing applications [10]. This because of its low cost, high resistance to corrosion, chemical stability and low toxic nature and has shown selectivity and sensitivity towards various volatile and toxic gases. On the other hand, α-FeOOH is a stable oxohydroxide of iron thermodynamically at ambient temperature and is often the end member of transformation of other iron oxides [11]. α-FeOOH has been applied in much research work in adsorption of organic pollutants and metals. In other to further enhance both the physical and chemical properties of α-Fe_2O_3 and α-FeOOH to improve their selectivity and sensitivity, surface modifications have been devised and employed by many researchers and still counting. To do this, various synthesis methods have been applied to achieve individual nanoparticles of α-Fe_2O_3 and α-FeOOH and a good composition of nanocomposite which including coprecipitation, forced hydrolysis, surfactant assisted method etc [12-14].

In this research work, facile hydrothermal synthesis was employed to prepare nanocomposite of alpha α-[Fe_2O_3-FeOOH]

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from iron nitrate salt using sodium hydroxide in a Teflon autoclave and characterized using spectroscopic method.

Material and Methods

Nanocomposite of α-[Fe_2O_3-FeOOH] with $Fe(NO_3)_2.9H_2O$ (98.0%) and NaOH (97.0 %). A 10 ml of ferric nitrate solution (0.5 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, it was transferred immediately to the Teflon autoclave and double distilled water was added to make up about 70 % 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 150 0C for hydrothermal reaction for 16 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 oC for 24 hours and the synthesized nanomaterial was stored in a reagent bottle while a little quantity was taken for XRD, FESEM, EDX, TEM and Zeta potential analysis.

Characterization of α-[Fe_2O_3-FeOOH]

The phase identification of the synthesized nanocomposite was characterized by X-ray diffractometer (XRD). 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.

Table 1: XRD data for α-[Fe_2O_3-FeOOH] synthesized by hydrothermal method

Pos. [°2θ]	Height [cts]	FWHM Left [°2θ]	d-spacing [Å]	Rel. Int. [%]	Tip W-+idth	Matched by
21.2123	759.98	0.2040	4.18512	28.82	0.2448	98-007-1808
24.0898	672.94	0.1632	3.69133	25.52	0.1958	98-008-2136
33.0763	2637.35	0.2040	2.70609	100.00	0.2448	98-008-2136, 98-007-1808
34.6047	242.72	0.2040	2.58999	9.20	0.2448	98-007-1808
35.5188	1890.37	0.1836	2.52540	71.68	0.2203	98-008-2136, 98-007-1808
36.5907	578.67	0.2040	2.45384	21.94	0.2448	98-007-1808
40.7292	604.98	0.2244	2.21355	22.94	0.2693	98-008-2136
49.3085	779.20	0.2244	1.84661	29.54	0.2693	98-008-2136
53.0681	269.83	0.1632	1.72431	10.23	0.1958	98-007-1808
53.9101	1230.74	0.2040	1.69935	46.67	0.2448	98-008-2136
57.4535	247.18	0.1632	1.60268	9.37	0.1958	98-008-2136, 98-007-1808
58.8013	221.35	0.1632	1.56911	8.39	0.1958	98-007-1808
62.2138	577.59	0.2244	1.49099	21.90	0.2693	98-008-2136
63.7531	743.27	0.2244	1.45865	28.18	0.2693	98-008-2136, 98-007-1808
71.6698	222.33	0.1836	1.31573	8.43	0.2203	98-008-2136, 98-007-1808
80.7108	59.52	0.5712	1.18960	2.26	0.6854	98-008-2136
88.2374	116.97	0.4896	1.10652	4.44	0.5875	98-008-2136

The morphology of the synthesized material was characterized with scanning electron microscopy couples with elemental diffraction x-ray (SEM-EDX) with type zeiss evolution 200 series (25 kV) and transmission electron microscopy (TEM) with type Zeiss EM-900, (80 kV). The Fe and O elemental analysis of the samples was performed by EDX. All the spectroscopic analyses were carried out at room temperature.

Results and Discussion

XRD for the synthesized α-[Fe_2O_3-FeOOH] material by hydrothermal method at 45 Kv and 40 Am was used to identify crystalline phases of the sample. Figure 1 shows the XRD pattern of the powdered nanomaterial which show that α-Fe_2O_3 (blue peaks) and α-FeOOH were the only phases present in the product. The XRD peaks at 2θ (presented in Table 1) correspond to the crystal planes of alpha α-Fe_2O_3 and α-FeOOH which were identified using the standard data file No. 98-008-2136 and 98-007-1808 (JCPDS) respectively. The plots of the identified phases of the prepared nanomaterials are also represented in figure 1. The peaks in the identifier plot correspond to peaks of pure phase of α-Fe_2O_3 and α-FeOOH [15-20].

The average crystal size of the synthesized nanomaterial was calculated using Debye-Scherer formula according equation 1.

$$K = \frac{0.9\lambda}{\beta \cos \theta}$$

Where; the value 0.9 was taken as the particle shape factor and λ is the wavelength of the CuKα radiation while β is the full width at half maximum (FWHM) of the diffraction peak in radian which corresponds to the Bragg diffraction angle (2θ). The crystal size of the synthesized α-[Fe_2O_3-FeOOH] estimated by the equation above to be 56.78 nm which confirms the nanosize of the material.

The surface morphology of the synthesized nanomaterial was studied using SEM method of analysis. The images of the α-[Fe_2O_3-FeOOH] nanomaterial as represented in figure 2 shows that samples prepared by hydrothermal method show some spherical images with little agglomeration.

TEM analysis was employed after the synthesis of α-[Fe_2O_3-FeOOH] to confirm the growth pattern and the distribution of the crystallites sample. Figure 3 shows that the TEM image of the synthesized nanomaterials by hydrothermal method are nanorod in nature.

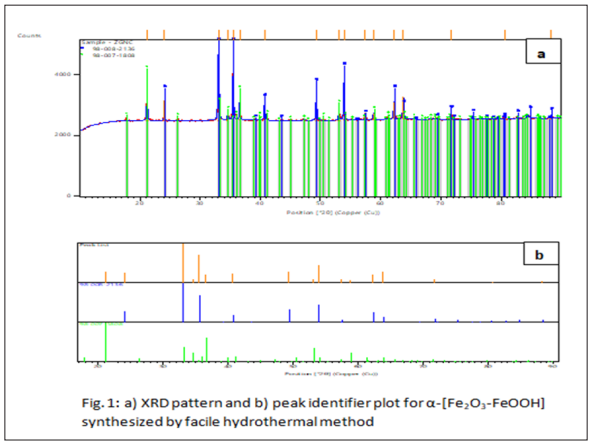

Fig. 1: a) XRD pattern and b) peak identifier plot for α-[Fe_2O_3-FeOOH] synthesized by facile hydrothermal method

Figure 1: XRD pattern and b) peak identifier plot for α-[Fe_2O_3-FeOOH] synthesized by facile hydrothermal method

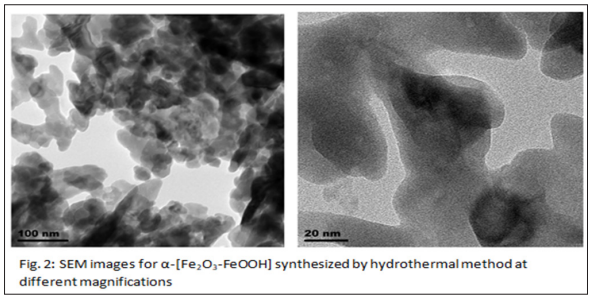

Fig. 2: SEM images for α-[Fe_2O_3-FeOOH] synthesized by hydrothermal method at different magnifications

Figure 2: SEM images for α-[Fe_2O_3-FeOOH] synthesized by hydrothermal method at different magnifications

EDX was used to analyze the elemental composition of the nanomaterials prepared by hydrothermal method under SEM analysis. Figure 4 shows the EDX analysis of the synthesized α-[Fe_2O_3-FeOOH] which confirms the presence of Fe and O with weight percent. The EDS result shows the peaks of iron and oxygen with carbon (C) and Aluminum (Al) peaks due to contamination from the materials used to prepare the samples for analysis. It can also be noticed that peaks due to hydrogen atom is absent because EDX cannot detect lightest elements below the atomic number for detectors. The EDX result shows the degree of purity of the prepared nanomaterials.

The data output for the zeta potential measurements for the synthesized nanorod α-[Fe_2O_3-FeOOH] prepared by hydrothermal method is represented in table 2 and figure 5. The value of the zeta potential was found to 20.0 mV.

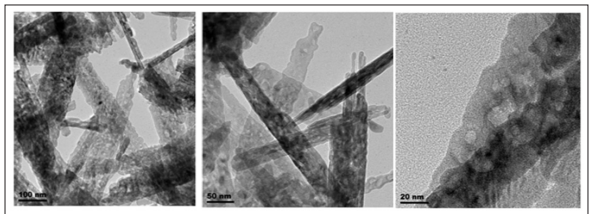

Fig. 3: TEM images for α-[Fe_2O_3-FeOOH] synthesized by hydrothermal method at different magnifications

Figure 3: TEM images for α-[Fe_2O_3-FeOOH] synthesized by hydrothermal method at different magnifications

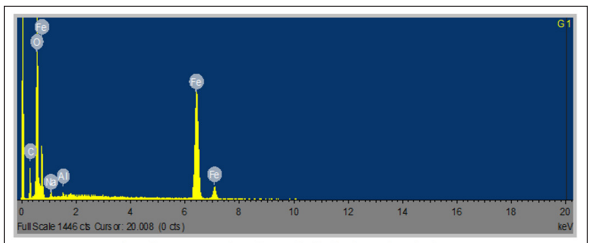

Fig. 4: EDX pattern for α-[Fe_2O_3-FeOOH] synthesized by hydrothermal method

Figure 4: EDX pattern for α-[Fe_2O_3-FeOOH] synthesized by hydrothermal method

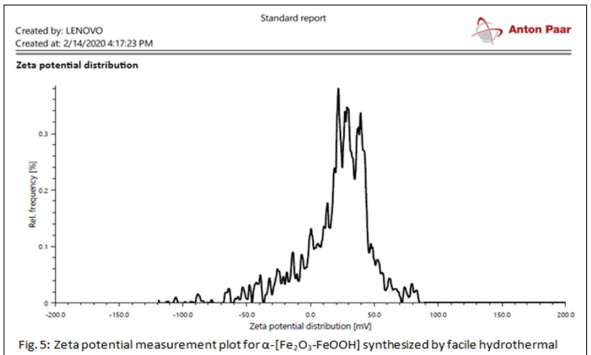

Fig. 5: Zeta potential measurement plot for α-[Fe_2O_3-FeOOH] synthesized by facile hydrothermal method

Figure 5: Zeta potential measurement plot for α-[Fe_2O_3-FeOOH] synthesized by facile hydrothermal method

Table 2: Out data for zeta potential for measurement for synthesized α-[Fe_2O_3-FeOOH] by hydrothermal method

Mean zeta potential	20.0 mV	Electrophoretic mobility	1.5624 µm*cm/Vs
Distribution Peak Value	21.9 mV	Filter optical density	2.4131
Processed runs	100	Conductivity	0.198 mS/cm
Adjusted voltage	37.9 V	+/- Standard deviation	4.1 mV
Mean intensity	958.6 kcounts/s	Transmittances	51.0%

Conclusions

Facile synthesis via hydrothermal method with Teflon autoclave was successfully employed to synthesize α-[Fe_2O_3-FeOOH] which was confirmed by XRD analysis to be in a pure form without other phases. The results obtained from SEM image at different magnifications show that the morphology of the synthesized product with less agglomeration of the nanomaterials. The TEM image shows that the synthesized α-[Fe_2O_3-FeOOH] has an average particle size of 56.78 nm and a zeta potential of 20.0 mV with less aggregation. The EDX result shows peaks due to iron (Fe) and oxygen (O_2) in the synthesized nanorod α-[Fe_2O_3-FeOOH].

References

1. Enlei Z, Guosheng W, Xiaozhu L, Zhumin W. Synthesis and Influence of Alkaline Concentration on α-FeOOH Nanorods Shapes. Indian Academy of Sciences. 2014. 37: 761-765.
2. Ghanbariasad A, Taghizadeh SM, Show PL, Nomanbhay S, Berenjian A, et al. Bioengineere. 2019. 10: 390-396.
3. Jurkin T, Štefanić G, Dražić G, Gotić M. Synthesis Routeto FeOOH Nanodiscs. Material letters. 2016. 173: 55-59.
4. Marinho JZ, Montes RHO, de-Moura AP, Longo E, Varela JA, et al. Rapid preparation of a-FeOOH and α-Fe2O3 Nanostructures by Microwave Heating and their Application in Electrochemical Sensors. Material research bulletin. 2014. 49: 572-576.
5. Sun M, Senthil RA, Pan J, Osman S, Khan A. Facile Synthesis of Visible-Light Driven Rod-on-Rod like α-FeOOH/AgVO3 Nanocomposite as Greatly Enhanced Photocatalyst for Degradation of Rhodamine B. Catalyst. 2018. 8: 392-406.
6. Sarkar ZK, Sarkar FK. Selective Removal of Lead (II) Ion from Wastewater Using Superparamagnetic Monodispersed Iron Oxide (Fe3O4) Nanoparticles as an Effective Adsorbent. International Journal Nanoscience and Nanotechnology. 2013. 9: 109-114.
7. Hafez H. A Study on the use of Nano/micro Structured Goethite and Hematite as Adsorbents for the Removal of Cr(III), Co(II), Cu(II), Ni(II), and Zn(II) Metal ions from Aqueous Solutions. International Journal of Engineering Science and Technology. 2012. 4: 3018-3028.
8. Montes-Hernandez Z, Renard F, Chiriac R, Findling N, Ghanbaja J, Toche F. Sequential precipitation of a new goethite-calcite nanocomposite and its possible application in the removal of toxic ions from polluted water. Chemical engigeering Journal. 2013. 214: 139-148.
9. Fusheng YZ, Yu LS. Preparation and Photo-catalytic Activities of FeOOH/ZnO/MMT composite. Applied surface science. 2015.
10. Kaneti YV, Quadir MD, Zakaria D, Zhang Z, Chen C, et al. Solvothermal Synthesis of ZnO-decorated α-Fe2O3 Nanorods with highly enhanced Gas-sensing Performance toward n-butanol. Journal of material chemistry. 2014. 2: 13283-13292.
11. Alexandra B, Iveta S, Zuzana D. Sorption of Copper and Zinc by Goethite and Hematite. Archives for Technical Sciences. 2015. 12: 59-66.
12. Farahmandjou M, Soflaee F. Synthesis and Characterization of α-Fe2O3 Nanoparticles by Simple Co- Precipitation Method. Phyical Chememistry Research. 2015. 3: 191-196.
13. Fu D, Keech PG, Sun X, Wren JC. Iron oxyhydroxide nanoparticles formed by forced hydrolysis: dependence of phase composition on solution concentration. Physical chemistry. 2011.
14. Zhang J, Zhu X, Yu K. Surfactant-assisted nanorod synthesis of a-FeOOH and its adsorption characteristics for methylene blue. Journal of Material Research. 2014. 29: 509-517.
15. Khalil M, Yu J, Liu N, Lee RL. Hydrothermal synthesis, characterization, and growth mechanism of hematite nanoparticles. Journal Nanoparticles Research. 2014. 16:2362.
16. Qin W, Yang C, Yi R, Gao G. Hydrothermal Synthesis and Characterization of Single-Crystalline α-Fe2O3 Nanocubes. Journal of Nanomaterials. 2011.
17. Morales-Morales JA. Synthesis of Hematite α-Fe2O3 Nano Powders by the controlled precipitation method. Ciencia en Desarrollo. 2017. 8: 99-107.
18. Krehula S, Popovic S, Music S. Synthesis of acicular a-FeOOH particles at a very high pH. Materials Letters. 2002. 54: 108-113.
19. Ghosh MK, Jai Poinern GE, Issa TB, Singh P. Arsenic adsorption on goethite nanoparticles produced through hydrazine sulfate assisted synthesis method. Korean Journal Chemical Engineering.
20. Gilbert F, Refait P, Le´veˆque F, Remazeilles C, Conforto E. Synthesis of goethite from Fe(OH)2 precipitates: Influence of Fe(II) concentration and stirring speed. Journal of Physics and Chemistry of Solids. 2008. 69: 2124-2130.