Original Article

Synthesis and Characterization of α-Fe2O3 Nanoparticles Using the Precipitation and Eco-Friendly Methods

Mostefe Khalid Mohammed[1], Abdulqadier Hussien Al khazraji[2*]

[1,2] Department of Chemistry, College of Education for Pure Science, University of Diyala, Iraq

E-mail: abdulqadier.niama@uodiyala.edu.iq

Abstract

Iron oxide (α-Fe2O3) nanoparticles were fabricated by chemical precipitation and Eco-Friendly routes, using Capparis Spinosa leaves extract, sodium hydroxide and iron sulphate heptahydrous. The calcination temperature for α-Fe2O3 nanoparticles was at 600 °C. All prepared samples were investigated using different physical methods such as XRD, FT-IR, FESEM, EDX and DLS. The XRD analysis confirmed that all the prepared nanoparticles consist only hematite phase with the size of 31.95 nm and 26.92 nm for chemical precipitation and Eco-Friendly method respectively. The α-Fe2O3 nanoparticles in the FESEM image showed a spherical nanoparticle structure. The EDX results presented the elements content (Fe = 64.3, O= 24.6) by green synthesis and (Fe = 66.3, O= 29.3) through chemical method. The distribution of iron oxide nanoparticles prepared via the Eco-Friendly method was greater as compared to the chemical precipitation method according to DLS analysis.

Keywords: α-Fe2O3 nanoparticles; Precipitation and Eco-Friendly methods

INTRODUCTION

Nanotechnology is one of the most important recent discoveries in various modern fields of science. It is possible to define this technique as converting large-size materials to others at the nano-size level (1-100 nanometers) with a significant change in the physical and chemical properties of these materials (Sridhara et al., 2012 and Abdul Majeed Lasoud et al., 2017). The iron oxide nanoparticles have captivated much consideration due to their many potential applications. (Siyaram Sankadiya et al,2016). The FeO,Fe2O3 and Fe3O4 are present as a main types of iron oxide nanoparticles, due to their low-cost, catalytic activity, biocompatibility, environmentally friendly nature and non-toxicity therefore, it is an important material.

Access this article online

Quick Response Code:

Website: www.pnrjournal.com

DOI: 10.47750/pnr.2022.13.04.104

The hematite (α-Fe2O3 and β-Fe2O3) and magnetite (γ-Fe2O3 and ε-Fe2O3) are the four crystalline ferric oxide phases. Fe2O3 has two types of structures: the γ-phase with cubic structure called magnetite and the α-phase with rhombohedra structure called hematite. Upon calcination (around 400 °C), a phase transition occurs from γ- Fe2O3 to α-Fe2O3, hence, lead to transform the α-Fe2O3 powder which has undergone considerable aggregation and grain growth. (Benyang Wang et al,2013), (Julian Morales et al.1989),(W Feitknech et al.1967).

Address for correspondence: Abdulqadier Hussien Al khazraji , Department of Chemistry, College of Education for Pure Science, University of Diyala, Iraq,
E-mail: abdulqadier.niama@uodiyala.edu.iq

Received date: 11 August 2022 Accepted: 13 September, 2022
Published: 10 October, 2022

How to cite this article: Mohammed K M, Al khazraji H A, Synthesis and Characterization of α-Fe2O3 Nanoparticles Using the Precipitation and Eco-Friendly Methods, J Pharm Negative Results 2022;13(4):782-789

The stable form of iron oxide nanoparticles present in rhombohedral crystal units and has an important role in research as in the case of hematite (α-Fe2O3).(Majd Abusalem et al ,2019). Various methods such as polyol, sol-gel , pyrolysis in addition to chemical co-precipitation methods, which is the most attractive method because it produces good crystal samples of hematite as a powder or thin film with high purity, low cost and short preparation time (Ali Boumejdad et al., 2011). The aim of this reserch is to synthesize α-Fe2O3 NPs through two methods, firstly using capparis spinosa plant leaf extract and secondly chemical precipitation method then study the properties of the product by different physical methods.

Experimental part

Materials

Chemicals of analytical purity (BDH) iron (II) Sulphate heptahydrate (FeSO4, 7H2O) (99%), sodium hydroxide (NaOH), ethanol , deoxygenated distilled water and Capparis Spinosa leaves were used to synthesized hematite (α-Fe2O3).

Instruments and Apparatus

All analysis of the samples were worked at the College of Science/University Tehran-Iran. The XPERT-Pro diffractometer operating at 30 mA and 40 kV for the scanning angles from 20 to 80° is used to get the XRD pattern via irradiating the samples with Siemens model D500. For the surface studies, Scanning Electron Microscope ZEISS model: Sigma VP are used. FTIR spectra of α-Fe2O3 synthesized nanoparticles are obtained in Diyala University/College of Education for Pure Sciences from Schimadzu Spectrophotometer/Japan. The size distribution studies of the samples are carried out by DLS malvern zetasizer nano zs and elemental analysis of α-Fe2O3 NPs studies by EDX.

Preparation of aqueous leaves extract:

The Capparis Spinosa leaves (5.0 g) were taken and washed with distilled water, then ground it into fine powder. Put the fine powder in distilled water (200 mL) and boiled at 80 oC for 30 min. Then the extract was filtered through filter paper (Whatman filter paper 1) and stored in refrigerator at 4 ºC for further studies.

Green synthesis of α-Fe2O3 NPs using leaves extract:

Iron salt solution was prepared by dissolving (0.3g, FeSO4.7H2O) in the deionized water (50 mL). Plant extract solution was added gradually on the precursor solution under stirring at(30°C), raise temperature of solution to (80 °C), NaOH (0.1M) solution was added dropwise to the mixture (salt solution and leaves extract), to obtain pH at 14 approximately under continuous stirring for 1 hour. Subsequently, the mixture was filtered; the precipitate was collected and dried in an oven at 80 °C for 4 hours. Then, the mixture was filtered, the precipitate collected (Fe(OH)2 NPs) and dried in an oven at 80°C for 4 hours. Calcination the precipitate product at 600 °C, 6 hours to obtain iron oxide nanoparticles (α-Fe2O3).

Chemical synthesis of iron oxide nanoparticles by precipitation method:

α-Fe2O3 nanoparticles were synthesized using Ferrous Sulphate heptahydrous (FeSO4.7H2O). Iron salt solution (5.0 g) dissolved in the deionized water (50 mL). NaOH (0.1M) solution was added dropwise to salt solution, to obtain pH at 14 approximately under continuous stirring. After a few minutes, a black precipitate is formed from iron hydroxide nanoparticles (Fe(OH)2NPs), which is collected and washed with distilled water several times until we get pH 7. After that, black precipitate dried at 80 degrees for 16 hours. Iron oxide nanoparticles obtained after calcination of α-Fe2O3 NPs at 600 ° C for a period of 6 hours.

Result And Discussion

FTIR analysis

Figures 1 (a and b) represent FT-IR spectra of the iron hydroxide nanoparticles samples were synthesized by green and chemical synthesis, respectively. In figure 1a the peak at 3420 cm−1 refer to O–H stretching vibration bond, while FT-IR spectrum in figure 1b, showed a new band with two frequencies at 3240 cm−1 and 3420 cm−1 represented bond O-H stretching vibration.

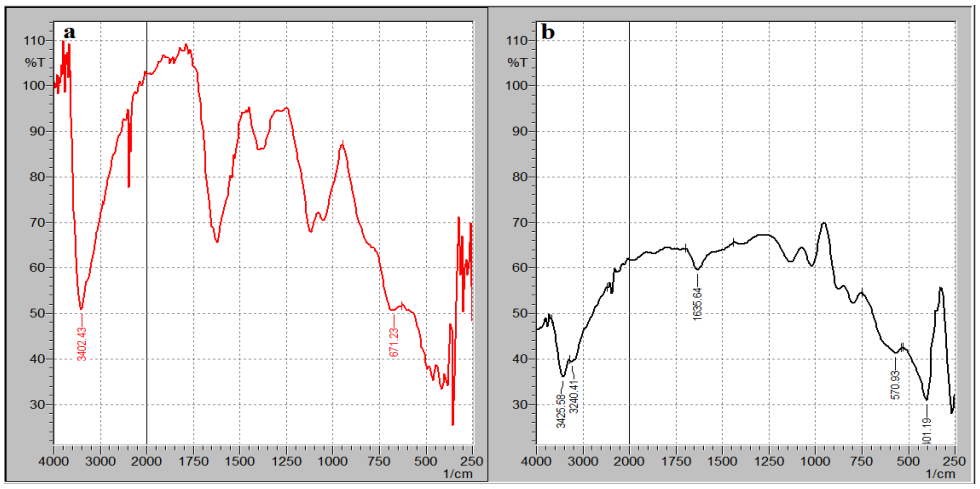

Figure 1. FTIR analysis of Fe(OH)2 nanoparticles synthesized by (a) green method and (b) chemical precipitation method.

The FTIR spectrums of produced α-Fe2O3 nanoparticles are shown in figures 3 (a and b) in the range of 400 - 4000 cm-1 (M. Farahmandjou et al,2015). Figure 1a showed strong band at 540.07cm-1 refer to Fe-O stretching bond and the in the figure 1b observed bands at 547 cm-1 refer to same bond. The presence of hematite α-Fe2O3 NPs was identified by the presence of two absorption bands approximately at of the bulk hematite (α-Fe2O3) (Majd Abusalem et al,2019). In the region from 2800 to 3500 cm−1, H2O in the air and hydrated α-Fe2O3 sample are responsible for existence of such peaks. It is confirmed through the FTIR spectrum results that synthesized α-Fe2O3 nanoparticles green compared to Fe(OH)2 and FeSO4.7H2O present pure.

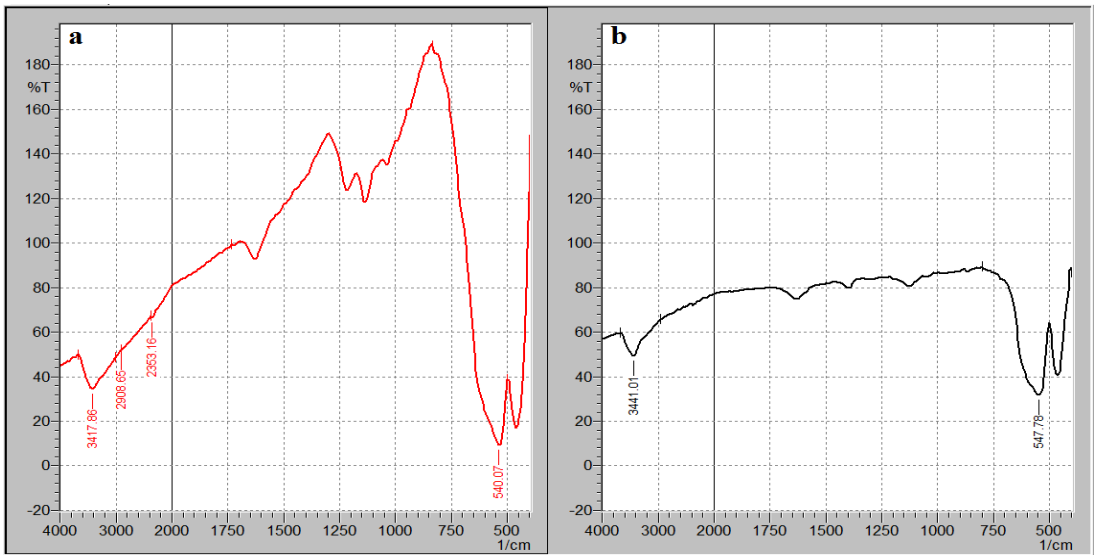

Figure 3. FTIR analysis of α-Fe2O3 nanoparticles synthesized by (a) green method and (b) chemical precipitation method.

XRD analysis

The XRD data as shown in Figure 5 (a and b) indicates that the 2 theta at 24.36°, 33.38°, 33.89°, 41.09°, 49.67°, 54.29°, 62.63° and 64.19 indicates the formation of α-Fe2O3 nanoparticles with the average crystallite size at 26.92 nm and 31.95 nm.

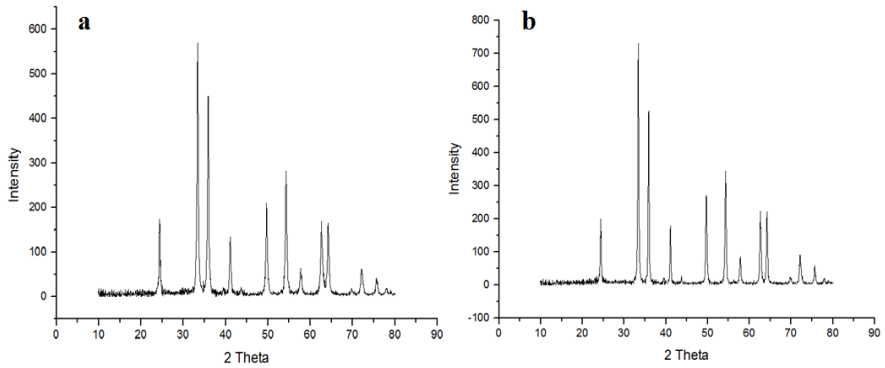

Figure 5 .XRD pattern α-Fe2O3 NPs (a) green method and (b) chemical method

FESEM

FESEM images of the Fe(OH)2 and α-Fe2O3 nanoparticles are shown in Figures (6-9) revealed spherical and oval nanoparticles. The spherical nanoparticles belong to the Fe(OH)2 and α-Fe2O3 nanoparticles prepared by the green method with average diameters 57.15 nm and 75.21nm , respectively. While in the case of Fe(OH)2 and α-Fe2O3 nanoparticles prepared by the chemical precipitation method, we noticed an almost oval shape with average diameters, respectively.

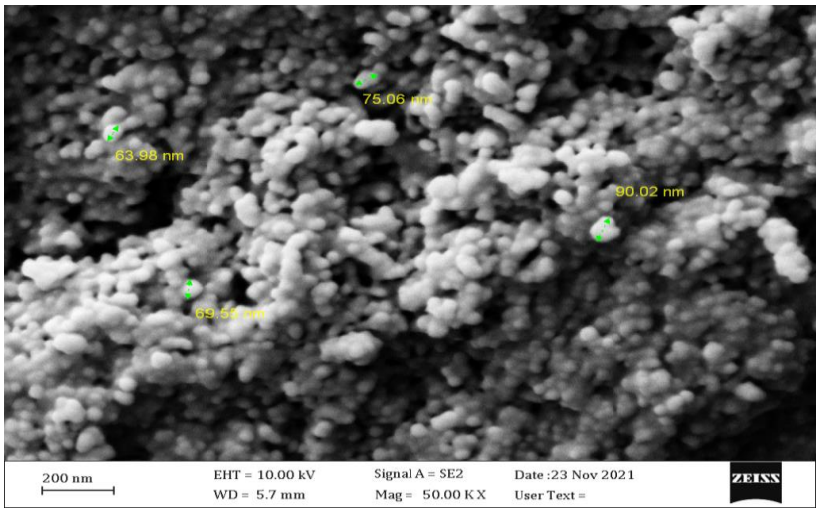

Figure 6. FESEM image of Fe(OH)2 nanoparticles (green method)

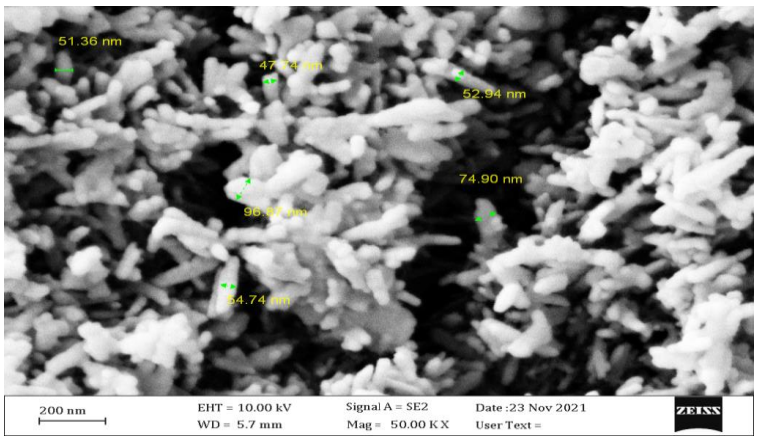

Figure 7. FESEM image of Fe(OH)2 nanoparticles (chemical method)

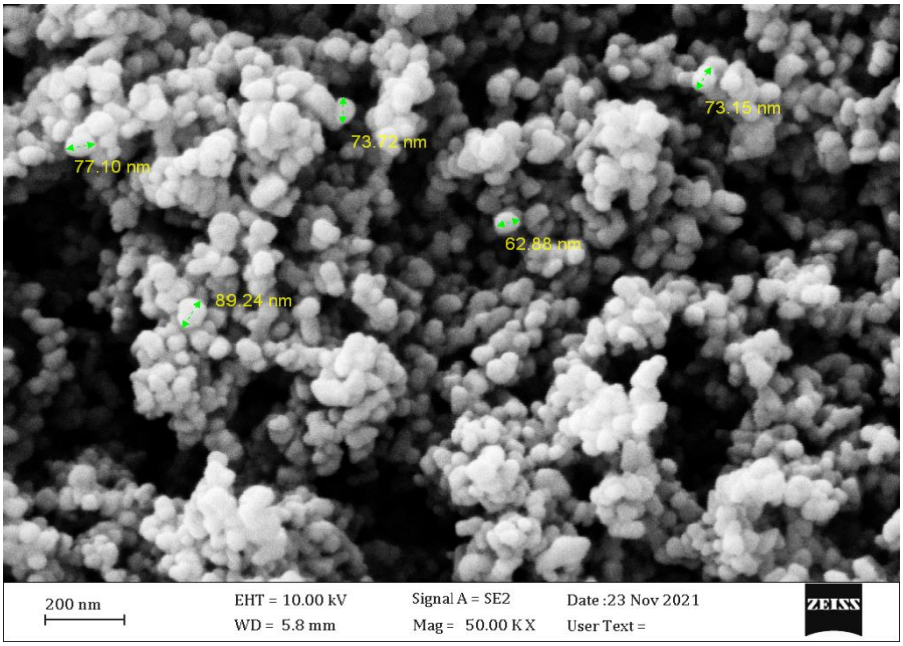

Figure 8. FESEM image of α-Fe2O3 nanoparticles (green method)

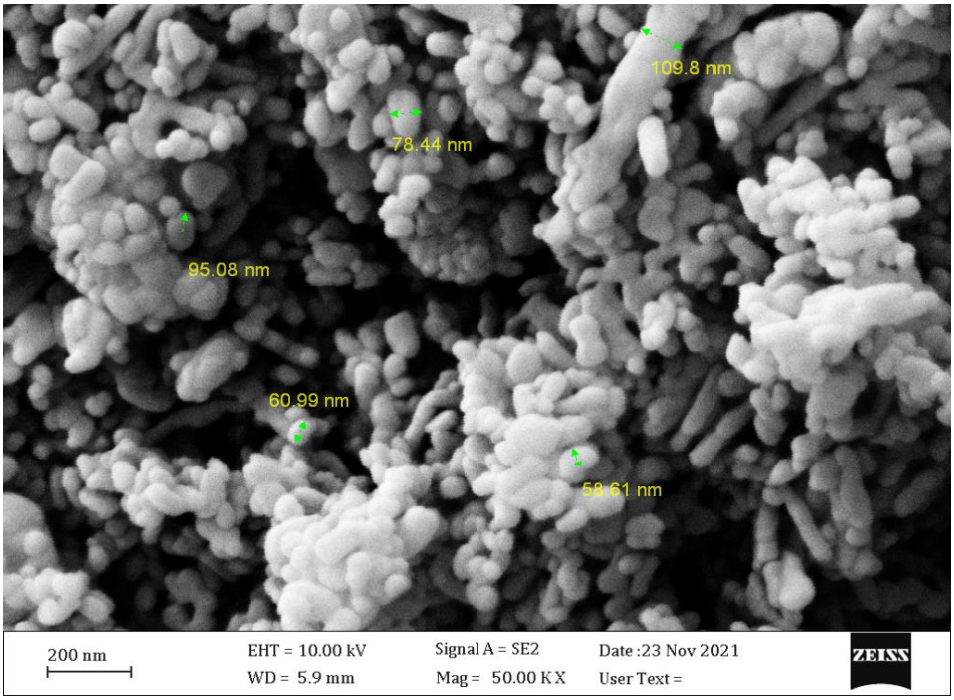

Figure 9 . FESEM image of α-Fe2O3 nanoparticles (Chemical method)

EDX Analysis

EDX results of α-Fe2O3 NPs that synthesized by eco-Friendly and chemical precipitation methods are shown in Figures 10 and 11. The element percentages obtained from EDX were (64.3% and 66.3% of Fe, 24.6% and 29.3% of O) for eco-Friendly and chemical precipitation methods, respectively, which showed the α-Fe2O3 formation with acceptable purity.

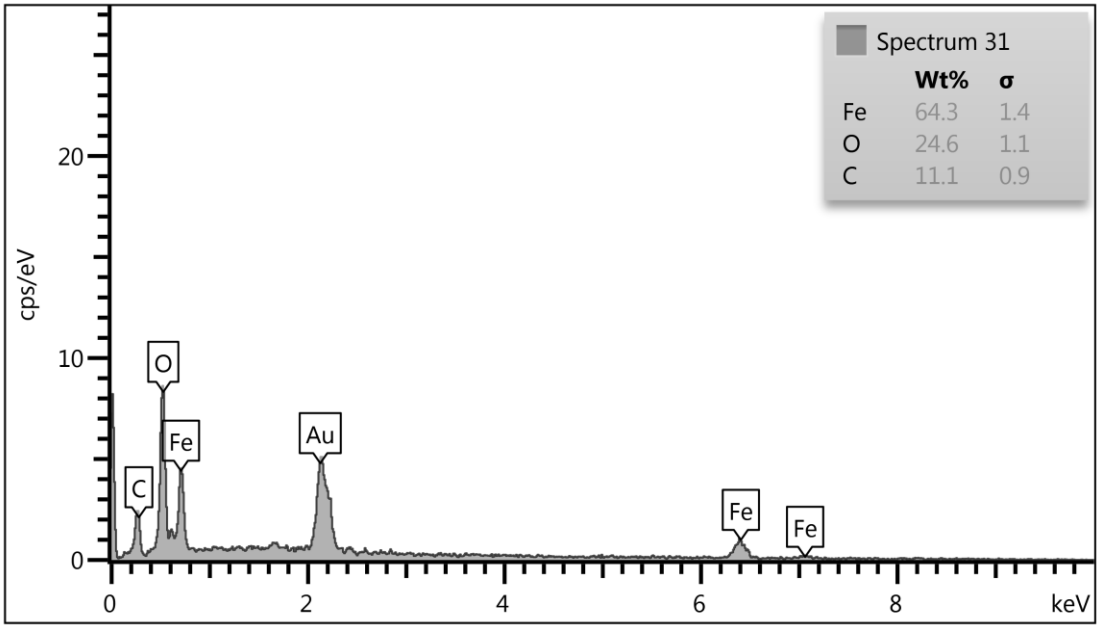

Figure 10. EDX Analysis of α-Fe2O3 nanoparticles according to intensity (green method)

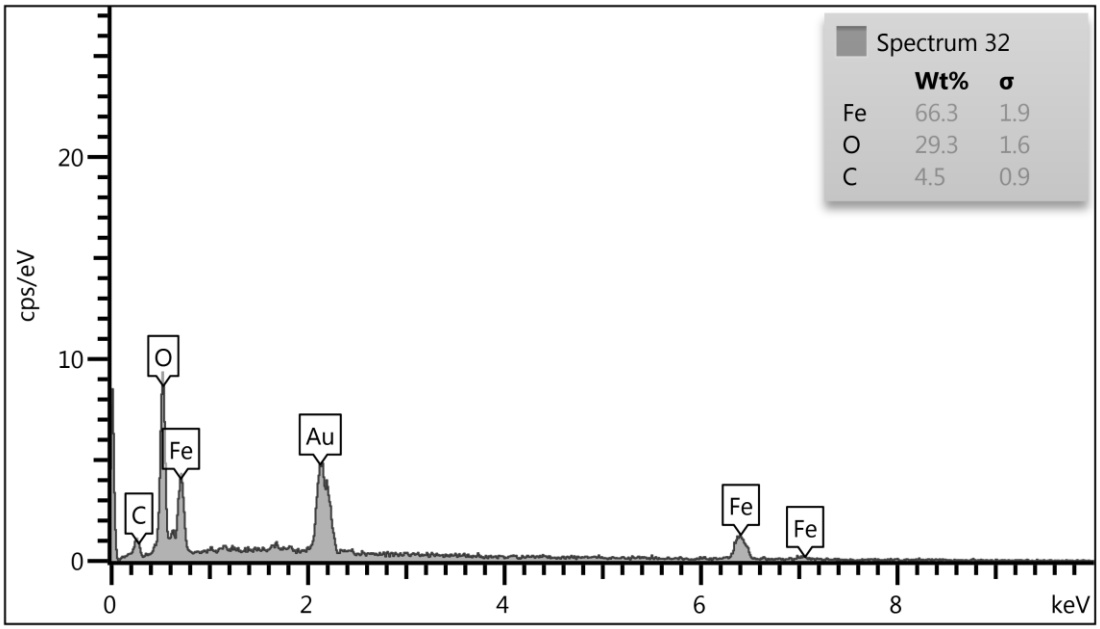

Figure 11. EDX Analysis of α-Fe2O3 nanoparticles according to intensity (chemical method)

DLS analysis

Distribution size of α-Fe2O3 nanoparticles by DLS technique were obtained at ranges approximately from 400 nm to 1000 nm in case the green method Figure 12, and 45 nm to 60 nm in case chemical method with mean particle size of 66 nm Figure 13. On other hand, we noticed that the α-Fe2O3 nanoparticles prepared by the chemical deposition method gave a narrower nanoparticle size distribution compared to the environmentally friendly method.

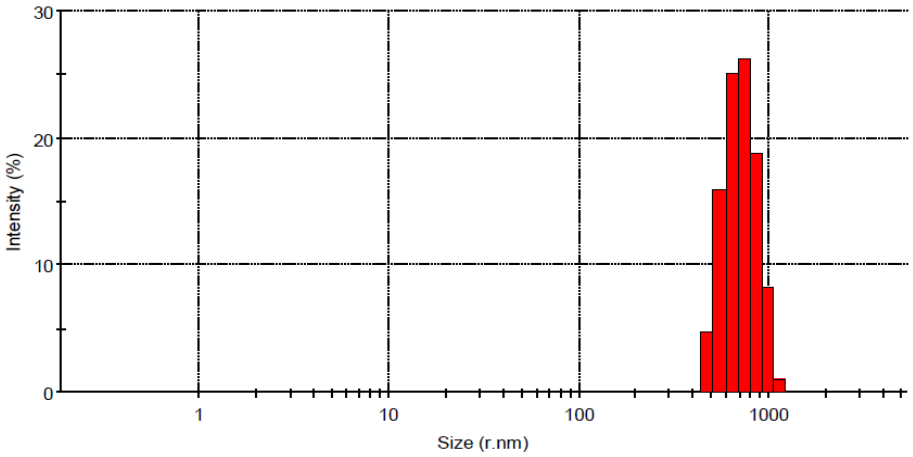

Figure 12. Size distribution of α-Fe2O3 nanoparticles according to intensity (green method)

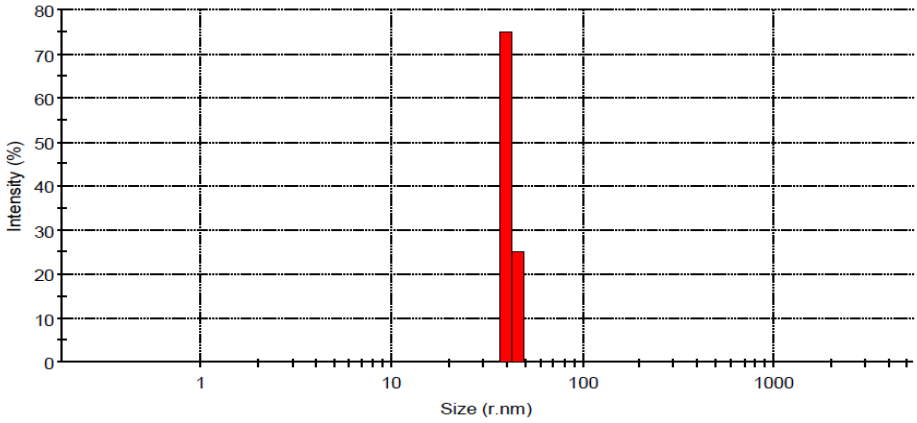

Figure 13. Size distribution of α-Fe2O3 nanoparticles according to intensity (chemical method)

Conclusions

In general, α-Fe2O3 NPs was prepared by two methods (chemical precipitation and Eco-friendly method) and studies their properties. Result of FTIR, XRD, DLS, FESEM and EDX technique demonstrated that α-Fe2O3 NPs were successfully synthesized. Formation of α-Fe2O3 NPs is confirmed from XRD, further FTIR technique determines the functional group. The strong band of α-Fe2O3 NPs was observed at below 600 cm-1. FESEM analysis reveals different particle sizes with oval and spherical shapes with average diameters 57.15 nm and 75.21nm. EDX result showed elements of α-Fe2O3 NPs. Using DLS the α-Fe2O3 nanoparticles prepared by the chemical deposition method gave a narrower nanoparticle size distribution compared to the environmentally friendly method with distribution size from 400 nm to 1000 nm and 45 nm to 60 nm .

References

Ali, H. R., Nassar, H. N., & El-Gendy, N. S. (2017). Green synthesis of α-Fe2O3 using Citrus reticulum peels extract and water decontamination from different organic pollutants. Energy Sources, Part A: Recovery, Utilization, and Environmental Effects, 39(13), 1425-1434.

Lassoued, A., Dkhil, B., Gadri, A., & Ammar, S. (2017). Control of the shape and size of iron oxide (α-Fe2O3) nanoparticles synthesized through the chemical precipitation method. Results in physics, 7, 3007-3015.

Abusalem, M., Awwad, A., Ayad, J., & Abu Rayyan, A. (2019). Green synthesis of α-Fe2O3 nanoparticles using Pistachio leaf extract influenced seed germination and seedling growth of tomatoes. Earth Environ. Sci, 10(3), 161-166.

Akintelu, S. A., Oyebamiji, A. K., Olugbeko, S. C., & Folorunso, A. S. (2021). Green synthesis of iron oxide nanoparticles for biomedical application and environmental remediation: a review. Eclética Química Journal, 46(4), 17-37.

Phiwdang, K., Suphankij, S., Mekprasart, W., & Pecharapa, W. (2013). Synthesis of CuO nanoparticles by precipitation method using different precursors. Energy procedia, 34, 740-745.

Sankadiya, S., Oswal, N., Jain, P., & Gupta, N. (2016, April). Synthesis and characterization of Fe2O3 nanoparticles by simple precipitation method. In AIP Conference Proceedings (Vol. 1724, No. 1, p. 020064). AIP Publishing LLC.

Chandirika, J. U., & Annadurai, G. (2018). Biosynthesis and characterization of silver nanoparticles using leaf extract Abutilon indicum. Global Journal of Biotechnology & Biochemistry, 13(1), 07-11.

Farahmandjou, M., & Soflaee, F. (2015). Synthesis and characterization of α-Fe2O3 nanoparticles by simple co-precipitation method. Physical Chemistry Research, 3(3), 191-196.

Lassoued, A., Dkhil, B., Gadri, A., & Ammar, S. (2017). Control of the shape and size of iron oxide (α-Fe2O3) nanoparticles synthesized through the chemical precipitation method. Results in physics, 7, 3007-3015.

Bumajdad, A., Ali, S., & Mathew, A. (2011). Characterization of iron hydroxide/oxide nanoparticles prepared in microemulsions stabilized

with cationic/non-ionic surfactant mixtures. Journal of colloid and interface science, 355(2), 282-292.

Vasantharaj, S., Sathiyavimal, S., Senthilkumar, P., LewisOscar, F., & Pugazhendhi, A. (2019). Biosynthesis of iron oxide nanoparticles using leaf extract of Ruellia tuberosa: antimicrobial properties and their applications in photocatalytic degradation. Journal of Photochemistry and Photobiology B: Biology, 192, 74-82.

Karimzadeh, I., Aghazadeh, M., Ganjali, M. R., Norouzi, P., Shirvani-Arani, S., Doroudi, T., ... & Gharailou, D. (2016). A novel method for preparation of bare and poly (vinylpyrrolidone) coated superparamagnetic iron oxide nanoparticles for biomedical applications. Materials Letters, 179, 5-8.

Sridhara, V., Ali, B., Shaziya, K., Satapathy, L. N., & Khandelwal, P. (2012). Biosynthesis and antibacterial activity of silver nanoparticles. Res J Biotechnol, 8(1), 11-17.

Abid, M. A., Abid, D. A., Aziz, W. J., & Rashid, T. M. (2021). Iron oxide nanoparticles synthesized using garlic and onion peel extracts rapidly degrade methylene blue dye. Physica B: Condensed Matter, 622, 413277.

Wang, B., Wei, Q., & Qu, S. (2013). Synthesis and characterization of uniform and crystalline magnetite nanoparticles via oxidation-precipitation and modified co-precipitation methods. Int. J. Electrochem. Sci, 8(3), 3786-3793.

Morales, J., Tirado, J. L., & Valera, C. (1989). Preferential X-ray line Broadening and Thermal Behavior of γ-Fe2O3. Journal of the American Ceramic Society, 72(7), 1244-1246.

Feitknecht, W., & Mannweiler, U. (1967). Der Mechanismus der Umwandlung von γ-zu α-Eisensesquioxid [1]. Helvetica Chimica Acta, 50(2), 570-581.

Yuan, J. T., Wang, Z., Wang, L., & Liu, R. J. (2020). Preparation, Surface Modification, and Characteristic of α-Fe2O3 Nanoparticles. Journal of nanoscience and nanotechnology, 20(5), 3031-3037.