RASĀYAN *J. Chem.*
Vol. 14 | No. 3 |1985-1989| July - September | 2021
ISSN: 0974-1496 | e-ISSN: 0976-0083 | CODEN: RJCABP
http://www.rasayanjournal.com
http://www.rasayanjournal.co.in

SYNTHESIS AND CHARACTERIZATION OF FeO NANOPARTICLES BY HYDROTHERMAL METHOD

K. Gurushankar[1, 2, ✉], K. Chinnaiah[1], Karthik Kannan[3], M. Gohulkumar[4] and P. Periyasamy[5]

[1]Laboratory of Computational Modeling of Drugs, Higher Medical and Biological School, South Ural State University, Chelyabinsk-454 080, Russia.

[2]Department of Physics, Kalasalingam Academy of Research and Education, Krishnankoil-626126, Tamilnadu, India.

[3]Brain Pool Program Postdoctoral Fellow, School of Advanced Materials Science and Engineering, Kumoh National Institute of Technology, 61 Daehak-ro, Gumi-si, Gyeongbuk, Republic of Korea.

[4]Vivekanandha College of Arts and Science for Women, Tiruchengode, Tamil Nadu, India

[5]Department of Physics, Nehru Institute of Engineering and Technology, T.M. Palayam, Coimbatore 641105, Tamilnadu, India

✉Corresponding Author: gurushankar01051987@gmail.com

ABSTRACT

FeO nanoparticles were synthesized via hydrothermal method and calcinated at diverse temperatures (400 °C, 450 °C, and 500 °C). The prepared samples are characterized by various techniques. Crystal size obtained for different annealing temperatures is 21, 14, and 8 nm, respectively. With an increase in temperature for annealing, the intensity of peaks has increased. SEM images note that with normal morphology, the samples have spherical. The average size of particles has obtained by SEM, which are matching with crystalline size obtained by XRD. UV absorbance results at 388, 392, and 399 nm are confined to the blue emission of wavelength. The edge of the optical bandgap towards the region of moving blue wavelength, which can be recognized at higher temperatures to decrease the FeO nanoparticles bandgap.

Keywords: FeO Nanoparticles, Hydrothermal Method, XRD, SEM with EDX, UV-DRS

RASĀYAN *J. Chem.*, Vol. 14, No.3, 2021

INTRODUCTION

The nanomaterials level is the best progressive at present, both in scientific knowledge and in commercial applications.[1-3] The enhancement of properties with potential applications, iron oxide is one of the significant pivotal roles in conducting materials today. Due to their applications, several ways have been proposed for the fabrication of FeO nanoparticles. In recent years, the methods like sol-gel method,[4] chemical coprecipitation methods,[5] flow injection methods,[6] sonochemical deposition methods,[7] electrochemical method[4] and hydrothermal method[7] are the prominent deposition methods, which are effectively useful for the synthesis of FeO. On the other hand, the significance of FeO nanostructure morphology in optical properties, and surface characteristics of nanostructure interfaces that determine nano-trapping levels. Therefore, the design and synthesis of FeO nanoparticles with different structures are very significant. Up to now, enormous research for FeO nanostructured materials with different morphology and structures synthesized in various forms such as nanoparticles,[8] nanorods,[9] and nanosheets[10] and their features, prospective applications studied intensive manner. Most of the above-mentioned synthesized techniques are comparatively expensive and usually need at high temperature, high sensitivity, and application of complex procedures, except the hydrothermal technique, which is more effective, low cost, and rewarding. In the present study, an attempt to made for the synthesis of FeO nanoparticles by hydrothermal method using Ferric chloride hexahydrate ($FeCl_3.6H_2O$) as a precursor and tested at various annealing temperatures for studying different characteristics.

http://doi.org/10.31788/RJC.2021.1436299

EXPERIMENTAL

All of the glassware employed in this experiment was acid-washed. The chemical reagents utilized were of the analytical reagent quality, without further purification. Ultra-pure water is employed for dilution and sample fabrication. Ferric chloride hexahydrate ($FeCl_3.6H_2O$) and NH_4 were attained from Loba Chemie Pvt. Ltd., Mumbai. All the chemicals have a purity of greater than 98 percent.

FeO nanoparticles were synthesized by using Ferric chloride hexahydrate as a precursor. Initially, 4.75g of Ferric chloride hexahydrate ($FeCl_3.6H_2O$) was taken to dissolve in 50 mL of double-distilled water (DDW) and then stirred by magnetic stirring apparatus at room temperature. 40 mL of aqueous ammonia solution was then progressively added drop-wise into the solution. Further, the resultant solution was continuously stirred for 24 h. At the final stage, the precipitate turned to green color. The ensuing precipitate was filtered and washed numerous times with DDW and ethanol, while green precipitate has dried at 100° C for 12 h in a hot air oven. Samples were annealed for 5 hours at 400° C to get the black FeO nanoparticles. For different annealing temperatures, such as 400° C, 450° C, and 500° C, the same procedure was repeated.

General Procedure

Synthesis of FeO Nanoparticles

$FeCl_3.6H_2O$ (4.75 g) + ammonia solution (40 mL)

↓

Stirred for 24 hrs

↓

Washed and filtered with DM water and ethanol

↓

Dried for 12 hrs in a hot air oven

↓

Annealed for 5 hrs at 400° C, 450° C and 500° C

Powder form in FeO nanoparticles

Instrumentation

X-ray diffraction (XRD), Scanning electron microscopy (SEM), Energy dispersive absorption X-ray analysis (EDAX), and UV-Vis were used for the prepared sample characterization. For identification of the crystalline phase and size of the particles was measured by XRD. Morphological information has been obtained by SEM. EDAX was used to confirm the various elements present in the sample. Absorption and bandgap measurement, UV-DRS spectrum was used. XRD of the samples was recorded with the support of an X-ray diffractometer (Model-D 5000 using with CuKα) consuming wavelength (λ=0.1540 nm). The morphological studies were performed by SEM (JSM 6390 JEOL) and the elemental composition was analyzed by EDX. UV-Vis-NIR spectrophotometer (Perkin-Elmer Lambda 35) was used for recording the optical absorption spectrum.

RESULTS AND DISCUSSION

XRD Analysis

Figure-1 explains the XRD patterns of FeO nanoparticles at varying temperatures (400°C, 450°C, and 500°C). The XRD patterns display that all the samples are in a single phase and the analyzed structures corresponded to the rhombohedral phase structure (JCDPS card no: 898104). The crystalline intensity peaks are (012), (104), (111), (113), (024), (116), (219), (300), respectively.

The crystalline sample size was determined with the Debye - Scherer formula:

$$D = \frac{k\lambda}{\beta \cos\theta} \qquad (1)$$

In this case, k is the dimensionless shape factor of 0.9, λ is known as X-ray wavelength, β is line broadening at half the maximum intensity (FWHM), and θ is known as angle of Bragg. FeO nanoparticles have mean crystalline sizes of 22 (400^{O} C), 14 (450^{O} C), and 8 (500^{O} C) nm respectively. The crystalline size was decreased when the annealing temperature was increased.

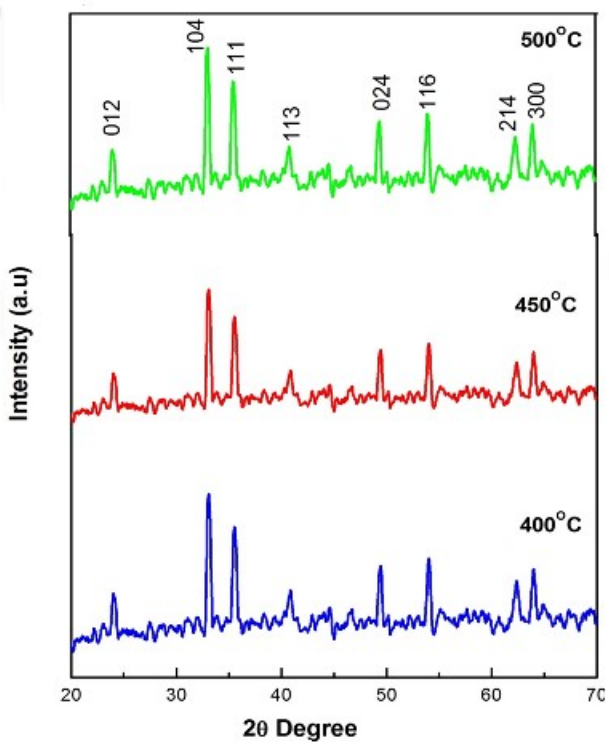

Fig.-1: XRD Spectrum of FeO Nanoparticles at Different Temperature

SEM with EDAX Analysis

To study morphology and elemental presentation in the samples, SEM and EDAX were used. Fig.-2(b) shows well-oxidized and dense agglomerations reacting to iron oxide nanoparticles. Fe and O elements were observed in conserved peaks seen in Fig.-2(d). It indicates that the formation of pure FeO nanoparticles confirmation, without any impurities. The samples are composed of spherical nanoparticles with a standard morphology (Fig.-2(a,c)) and a good correlation between the mean size and the crystalline size obtained by XRD.

UV- Vis Spectroscopy

Figures-3 and 3a display the UV-Vis spectrum of prepared FeO nanoparticles. The absorption values were noticed in the range of 300-400 nm. This range of wavelength is confined to blue shift emission. The absorption values were located at 388 (400°C), 392 (450°C), 398 nm (500°C). Furthermore, the maximum absorption peak value, due to the existence of a given bandgap, concludes that valence electrons merely consume a particular energy package to overcome the concern-insulated barricade. This may be the logical outcome of expanding the bandgap because it occurs because of the particle size reduction. As well, as the meaning of decreasing particle size and the density of states within the particle is inclined to be distinct, it tends to be discrete to decrease the number of atoms within a particle. Therefore by absorbing at an exact wavelength, valence electrons will mainly be excited than or else, whose energies appear too small to show in the far-infrared region or too large to fall within the X-ray section. This results in a definite peak in the absorbance spectrum of FeO nanoparticles (300-800 nm). In addition, the absorbance value is significantly higher than that of bulk iron oxide, which means that nanoparticles have a reduced particle size.

The direct optical band gap value of the samples analyzed through the following equation has been correlated with photon energy as well as absorption.

$$E\ (gap) = h\upsilon / \lambda \qquad (2)$$

and

$$(\alpha\ h\upsilon) = A\ (h\upsilon - Eg)n \qquad (3)$$

Where, h known as Planck constant, υ is defined as frequency, α referred as absorbance, λ represents the wavelength of absorption and n represents electronic transition occurred in various forms.

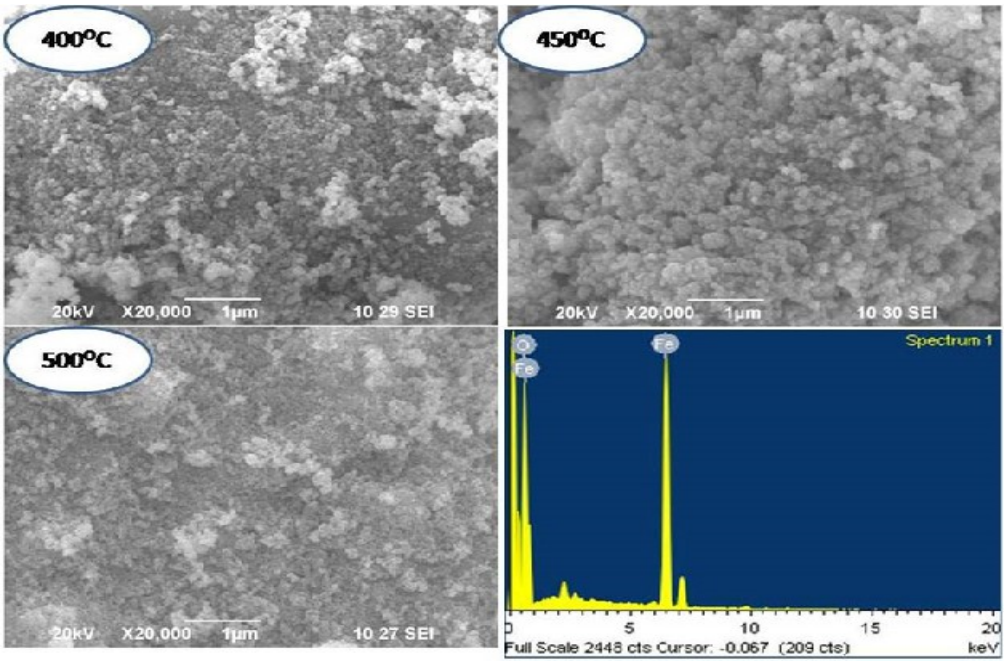

Fig.-2: (a, b, c) SEM images of FeO Nanoparticles at Different Temperatures (d) EDAX study of FeO Nanoparticles

The optical direct bandgap of FeO nanoparticles was 2.8, 2.7, 2.6 eV (400^{O} C, 450^{O} C, and 500^{O} C). The absorption is generally inversely proportional to the nanoparticles' bandgap value. As seen from Fig.-3(a), if the temperature increases, on the other hand, bandgap values decreased. If the size of the particles is also smaller, the quantum confinement effect is found to be close to the electron's wavelength. Therefore, the reduction in the confining dimension separates the energy levels until the nano-scale range reduces the size of a particle, and these increases or enlarges the bandgap and eventually increases the energy of the bandgap. Because with a decrease in size, the bandgap and wavelength have inversely related to each other the wavelength decreases and the emission of blue radiation is the proof. In the present study, the estimated bandgap of 450^{O} C and 500^{O} C, 2.7 & 2.6 eV, respectively. This value is slightly higher than the annealing temperature at 400^{O} C, which indicates the reduced particle size of synthesized materials.

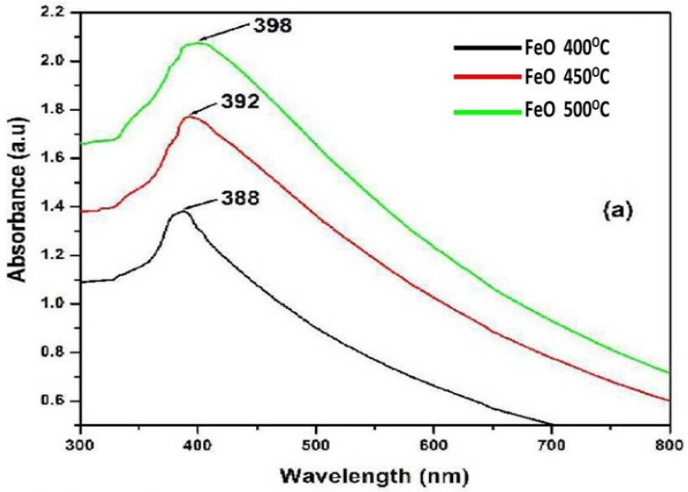

Fig.-3: UV Visible Spectra of FeO Nanoparticles at 400^{O} C, 450^{O} C, and 500^{O} C

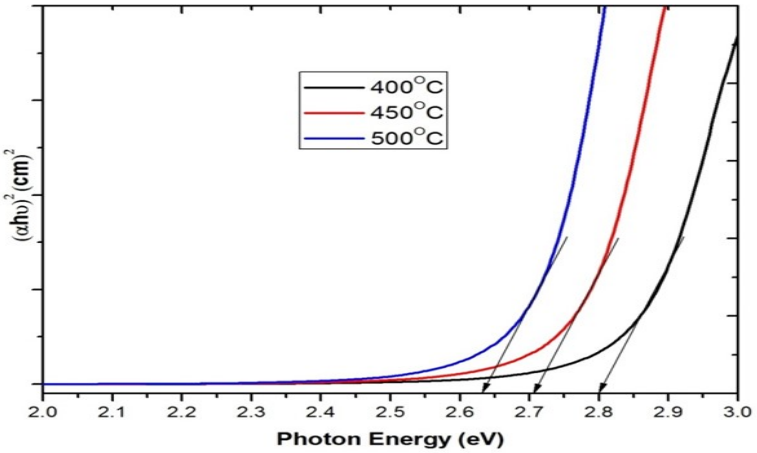

Fig.-3(a): Bandgap Energy of FeO Nanoparticles at 400^{O} C, 450^{O} C, and 500^{O} C

CONCLUSION

Nanocrystalline FeO nanoparticles with a rhombohedral structure successfully synthesized by hydrothermal method. XRD results indicated that with an increase in annealing temperature, the intensity of peaks has increased. The spherical nanoparticle shape with regular morphology is confirmed by SEM. Moreover, the size obtained by SEM, which was in good conformity with the crystalline size computed through XRD. The obtained UV absorbance at 388, 392, and 399 nm was confined to the blue emission of wavelength. The edge of the optical bandgap moves towards the blue wavelength region, which can be attributed at higher temperatures to the decreasing bandgap of the FeO nanoparticles. In the future, the FeO nanoparticle can be considered being a suitable material for fabricating photovoltaic devices, gas sensors, and magnetic materials.

REFERENCES

1. M. Malakootian, K. Kannan, M.A. Gharaghani, A. Dehdarirad, A. Nasiri, Y.D. Shahamat and H. Mahdizadeh, *Journal of Environmental Chemical Engineering,* **7,** 103457(2019), https://doi.org/10.1016/j.jece.2019.103457
2. K. Kannan, M.H. Sliem, A.M. Abdullah, K.K. Sadasivuni and B. Kumar, *Catalysts,* **10,** 549(2020), https://doi.org/10.3390/catal10050549
3. K. Pradeeswari, A. Venkatesan, P. Pandi, K. Karthik, K.V. Hari Krishna and R. Mohan Kumar, *Mater. Res. Express* **6,** 105525(2019), https://doi.org/10.1088/2053-1591/ab3cae
4. S. Laurent, D. Forge, M. Port, A. Roch, C. Robic, L.V. Elst and R.N. Muller *Chemical Reviews,* **108,** 2064(2008), https://doi.org/10.1021/cr068445e
5. S. Wu, A. Sun, F. Zhai, J. Wang, W. Xu, Q. Zhang and A.A. Volinsky, *Materials Letters,* **65,** 1882(2011), https://doi.org/10.1016/j.matlet.2011.03.065
6. G. Salazar-Alvarez, M. Muhammed and A.A. Zagorodni, *Chemical Engineering Science,* **61,** 4625(2006), https://doi.org/10.1016/j.ces.2006.02.032
7. W. Wu, Q. He and C. Jiang, *Nanoscale Research Letters,* **3,** 397(2008), https://doi.org/10.1007/s11671-008-9174-9
8. F.C. Nalle, R. Wahid, I.O. Wulandari and A. Sabarudin, *Rasayan J. Chem,* **12,** 14(2019) http://dx.doi.org/10.31788/RJC.2019.1214082
9. R. Sun, J. Gao, G. Wu, P. Liu, W. Guo, H. Zhou, J. Ge, Y. Hu, Z. Xue, H. Li, P. Cui, X. Zheng, Y. Wu, G. Zhang and X. Hong, *Cell Reports Physical Science,* **1,** 100118(2020) https://doi.org/10.1016/j.xcrp.2020.100118
10. K. Sharma, I. Katyal, A. Srivastava, V. Raghavendra Reddy and A. Gupta, (2020) Synthesis and Characterization of GaO(OH)–FeO(OH) Nanorod Composite Prepared via Hydrothermal Method, In: V.K. Jain, S. Rattan and A. Verma, (eds) Recent Trends in Materials and Devices. Springer Proceedings in Physics, **256**. Springer, Singapore, https://doi.org/10.1007/978-981-15-8625-5_10

[RJC-6092/2020]