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Biosynthesis and characterization of α-FeOOH nanoparticles using Isabella grape (*Vitis labrusca* L.) extract

Huseyin Ozcan[1], Aslihan Dalmaz[2], Mesut Ozdincer[3], Kubra Zenkin[1], Sefa Durmus[4*]

[1] Düzce University, Graduate Education Institute, Department of Chemistry, 81620, Düzce, Türkiye
[2] Düzce University, Graduate Education Institute, Department of Natural and Herbal Products/Cosmetic Products, 81620, Düzce, Türkiye
[3] Düzce University, Graduate Education Institute, Department of Composite-Materials, 81620, Düzce, Türkiye
[4] Düzce University, Faculty of Art and Science, Department of Chemistry, 81620, Düzce, Türkiye

Abstract

The advancement of environmentally sustainable and eco-friendly approaches to nanoparticle synthesis has gained significant importance in analytical chemistry. This research examined the green synthesis of iron oxyhydroxide nanoparticles, utilizing *Vitis labrusca* L. (Isabella grape) extract as both a reducing and stabilizing agent. The application of this natural extract offers an environmentally friendly alternative to conventional chemical synthesis techniques and is expected to meet the growing demand for sustainable applications. The synthesized iron oxyhydroxide nanoparticles were characterized using advanced techniques, including X-ray diffraction, scanning electron microscopy, energy-dispersive X-ray analysis and fourier transform infrared spectroscopy, to verify their composition and structure. The findings reveal the successful synthesis of iron oxyhydroxide nanoparticles with a uniform size distribution and excellent stability.

Keywords: *Vitis labrusca* L., α-FeOOH, nanoparticle, biosynthesis

1. Introduction

Nanotechnology has made great advances in recent years, with applications in medicine, chemistry, biotechnology, and has become a field that is attracting attention [1,2]. Nanoparticles are one of the basic units of nanotechnology, and have great potential in various applications [3]. Nano-sized particles are particularly attractive thanks to their high surface-to-volume ratios. Nanoparticles can be more reactive than other materials due to the fact that the atoms on the surface are more active than those in the center. With these unique and unusual physical and chemical properties, metal oxide nanoparticles offer new opportunities in nanoscale science [4–10]. Among metal oxide nanoparticles, iron-based nanoparticles are of great interest given the wide range of applications [11,12]. With applications as diverse as magnetic recording devices [13], ferrofluids [14], drug delivery systems, magnetic resonance imaging [15,16], and paint pigments, iron oxides are remarkable. In addition, it is desirable for nanoparticles that are used in biological applications to have superparamagnetic properties. Magnetic properties of particles change depending on particle size. In this sense, the synthesis of particles of the desired size gains importance in terms of application areas.

Among iron compounds, α-FeOOH nanoparticles (goethite, iron oxyhydroxide) are used in various technical applications such as pigment industries, environmental remediation and medical supplements. The α-FeOOH nanoparticles can be particularly effective in the removal of metallic cation pollutants such as arsenic and chromium [17,18]. Additionally, α-FeOOH nanoparticles have the ability to remove fluoride from contaminated aqueous media. In addition to their high adsorption capacity, these particles can be used as an effective nanocatalyst in chemical reduction reactions. The increasing use of α-FeOOH nanoparticles in a wide range of applications has led to a growing demand for the sustainable production of these nanostructures. α-FeOOH nanoparticles are usually synthesized at high temperature, and this process can become energy consuming, and non-economical. These nanoparticles can be synthesized by physical [19], chemical [20] or

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***Author of correspondence:** sefadurmus@duzce.edu.tr
Tel: +90 (380) 541 24 04
Fax: +90 (380) 541 24 03
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biological methods [21]. Various physical and chemical methods such as hydrothermal [22], sol-gel [23] synthesis may require special equipment, and qualified work force. In addition, they have toxic effects harmful to health. However, it has been observed that nanoparticles obtained by the green synthesis method are cost-effective, non-toxic, and biodegradable in nature [24–27]. For this reason, the production of metal oxide nanoparticles using the principles of green chemistry has become an important area of research.

Many efforts have been made to use various plant extracts for the preparation of nanoscale metal oxides [28,29]. Secondary metabolites such as phenolics, polysaccharides, and flavonoids, which possess redox capacities [29,30], play a crucial role in the synthesis of metal oxide nanoparticles. In the field of nanotechnology, the use of natural capping agents, such as *Vitis labrusca* L., has gained interest due to their perceived eco-friendliness, and biocompatibility compared to traditional synthetic surfactants, and reducing agents. *Vitis labrusca* L. extracts contain a variety of organic compounds, including flavonoids, and tannins [31,32], which have been shown to be effective in stabilizing, and reducing metal ions during the synthesis of metal oxide nanoparticles. However, it is important to acknowledge that the efficacy of *Vitis labrusca* L. as a sequestrant and reducing agent in the synthesis of metal oxide nanoparticles depends on several factors, including the extraction method, the extract concentration, and the specific type of metal oxide nanoparticle under consideration. Further research is needed to fully understand the potential of *Vitis labrusca* L. as a capping, and reducing agent in metal oxide nanoparticles synthesis. Notably, some studies have reported the successful synthesis of metal oxide nanoparticles using *Vitis labrusca* L. extract as a green reducing agent [33]. For example, Raota et al. demonstrated the synthesis of silver nanoparticles using grape pomace extract, which was evaluated for its phenolic compound content, and subsequently used as a stabilizing reducing agent [34]. The researchers also investigated the application of silver nanoparticles in the disinfection of raw wastewater.*Vitis labrusca* L., a fragrant grape found only in the Black Sea region, was chosen for its affordability, accessibility, and sustainable nature. In this work, we present a new method for the synthesis of α-FeOOH nanoparticles using *Vitis labrusca* L. as a coating, and reducing agent. The resulting nanoparticles were then characterized using a variety of techniques, including scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), and fourier transform infrared spectroscopy (FT-IR) analysis.

2. Materials and Methods

2.1. Chemicals and Instrumentations

All of the chemical materials used in the study were of analytical grade purity and were used without any purification. $Fe(NO_3)_3.9H_2O$ and NaOH were obtained from Merck company. In this study, the *Vitis labrusca* L., which was used as a reducing agent, was obtained from Duzce at the time of harvest. The image of *Vitis labrusca* L. is shown in Fig. 1.

Figure 1. Image of *Vitis labrusca* L.

FT-IR spectroscopy results were recorded using a Perkin Elmer Spectra Two UATR FT-IR spectrophotometer. Scanning electron microscopy (FEI Quanta FEG 250) was employed to determine the size, and morphology of nanoparticles, while energy dispersive X-ray analysis was utilized to determine their elemental composition. The X-ray diffraction pattern of nanoparticles was obtained using a Bruker D8 Discover instrument with Cu-Kα radiation (1.5406 Å). The maximum peaks in the XRD patterns of the nanoparticles were matched with JCPDS cards.

The pH control of the reaction mixture was measured using an Isolab brand pH meter. A VWR brand centrifuge device was employed to separate the obtained product from the supernatant. The drying process of the obtained product was conducted using an Elektromag M5040P brand oven. A Heidolph brand MR Hei-Standard model magnetic stirrer was utilized to complete the dissolution process, and ensure the mixing of the solutions until the reaction was complete.

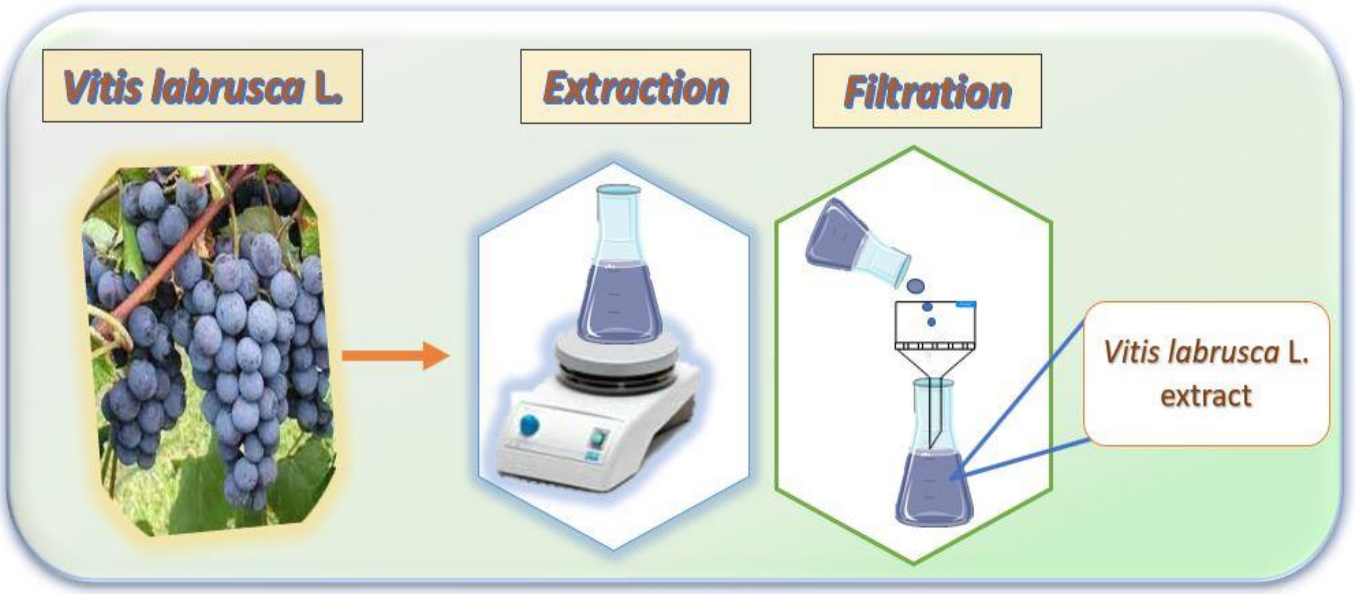


Figure 2. Representative illustration of *Vitis labrusca* L. extract preparation

The filtration processes of *Vitis labrusca* L. extract, and the product obtained were carried out using an Isolab brand vacuum pump. All weighing processes during the study were conducted using Radwag brand electronic scales.

2.2. Preparations of *Vitis labrusca* L. Extract

Vitis labrusca L. was rigorously cleaned by washing several times with deionized water to remove dust and other impurities. Then *Vitis labrusca* L. extract was prepared by boiling 20 g grapes in 250 mL deionized water in a magnetic stirrer for 90 minutes. The prepared extract was cooled, filtered through Whatman filter paper and stored in a refrigerator at 4 °C to be used in the synthesis of α-FeOOH nanoparticles (Fig. 2).

2.3. Green Synthesis of α-FeOOH Nanoparticles

A solution of 2,8 g $Fe(NO_3)_3.9H_2O$ salt in deionized water was added to 25 mL of *Vitis labrusca* L. extract, the temperature of which was kept constant at 60 °C. Then the pH of the solution was adjusted to 8 with 0.1 M NaOH. It was mixed with a magnetic stirrer for one hour at 60 °C and a completely homogeneous mixture was obtained. The obtained homogeneous mixture was centrifuged at 5000 rpm for 5 minutes, and then washed several times with deionized water. The obtained product was left to dry in an oven at 70 °C for 24 hours and then calcined at 550 °C for 3 hours. The resulting dark brown α-FeOOH nanoparticles were stored in a desiccator.

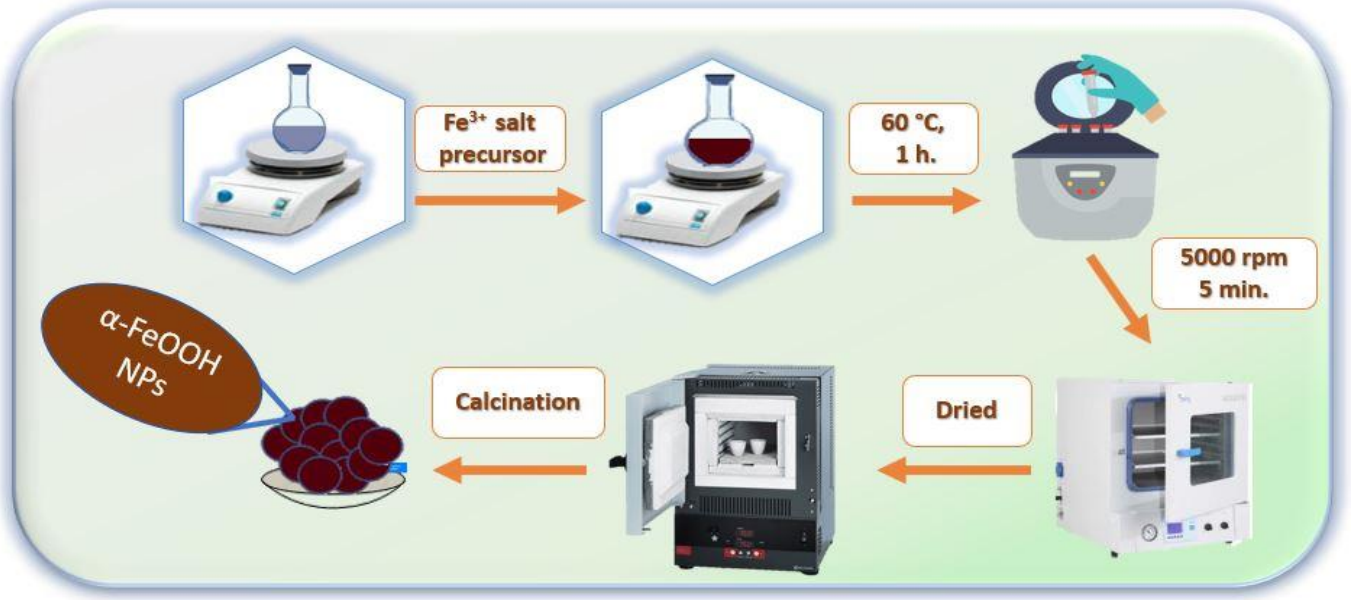


Figure 3. Schematic representation for green synthesis of α-FeOOH nanoparticles

3. Results and Discussion

3.1. FT-IR spectra

3.1.1. Vitis labrusca L. Extract

FT-IR spectroscopy analysis was performed to investigate the presence of functional biomolecule groups in *Vitis labrusca* L. extract and synthesized metal oxide nanoparticles. When the FT-IR spectrum of *Vitis labrusca* L. shown in Fig. 4 is examined, the band seen at 3283 cm^{-1} is due to O-H stretching vibrations in the structure of polyphenols. The asymmetric C-H stretching vibration of -CH_3 groups in the structure of biomolecules was observed at 2932 cm^{-1} and the peak of the carbonyl (C=O) functional group was observed at 1720 cm^{-1}. The peak observed at 1604 cm^{-1} in the FT-IR spectrum indicates the presence of the O-H group in the structure of water. Furthermore, the peaks corresponding to aliphatic C-H stretching and CH_3 symmetrical bending vibrations are observed at 1408 cm^{-1} and 1343 cm^{-1}, respectively. The C-O stretch vibration of hydroxy flavonoids in the biostructure of *Vitis labrusca* L. is observed at 1251 cm^{-1}, while the band at 1026 cm^{-1} corresponds to the C-O stretch of primary alcohols. Besides, the vibration of the C=O bending band in the structure of fatty acids was observed at 917 cm^{-1}[35]. In addition, the absorption bands shown at 865, 815 and 776 cm^{-1} can be assigned to the =C-H flexure, which is consistent with the polyphenolic or flavonoid compounds present in the extract [36].

3.1.2. α-FeOOH Nanoparticles

The FT-IR spectrum of α-FeOOH nanoparticles is given in Fig. 4. In the FT-IR spectrum of α-FeOOH nanoparticles, vibrations at 3168, 1590, 1375, 800, 700, 548 and 397 cm^{-1} were noted. The 3168 cm^{-1} vibration is attributed to the O-H group stretching vibrations of polyphenols found in the *Vitis labrusca* L. plant extract, while the 1575 cm^{-1} vibration corresponds to carbonyl group vibrations of the same polyphenols. Additionally, the in-plane bending vibration at 891 cm^{-1} and the out-of-plane bending vibration at 794 cm^{-1} are specific to the Fe-OH bending vibration of the goethite particles. It is worth noting that the thermal transformation from goethite to hematite results in hematite characteristic vibrations at 533 and 454 cm^{-1}, which have been previously reported for similar findings in goethite [37].

3.2. XRD Analysis

In the XRD pattern of α-FeOOH nanoparticles as shown in Fig. 5, distinct peaks were observed at 2θ=22.42°, 34.08°, 36.78°, 39.72°, 44.82° and 59.39°. These peaks were interrelated to (110), (130), (111), (200), (131) and (151) hkl planes. All these diffraction peaks are consisted to orthorhombic goethite compatible with JCPDS Card No. 29-0713 and space group $P2_1nm$. The cell parameters of the α-FeOOH nanoparticles a= 4.61 Å, b = 9.95 Å and c = 3.02 Å as shown in Table 1. Since there are no different peaks in the substance, the product does not contain impurities [38].

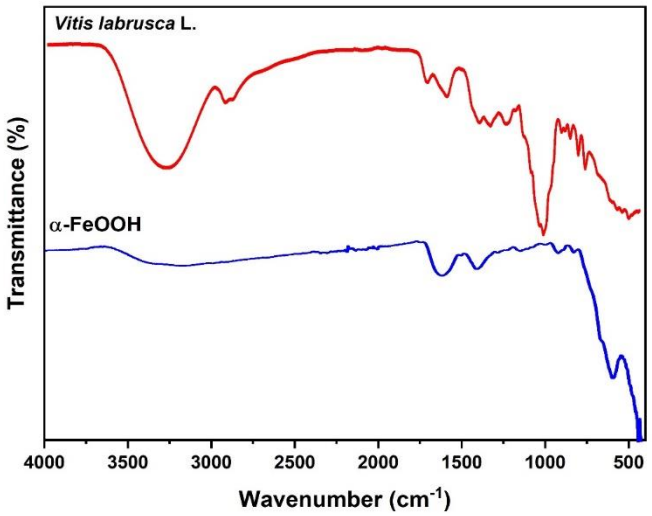
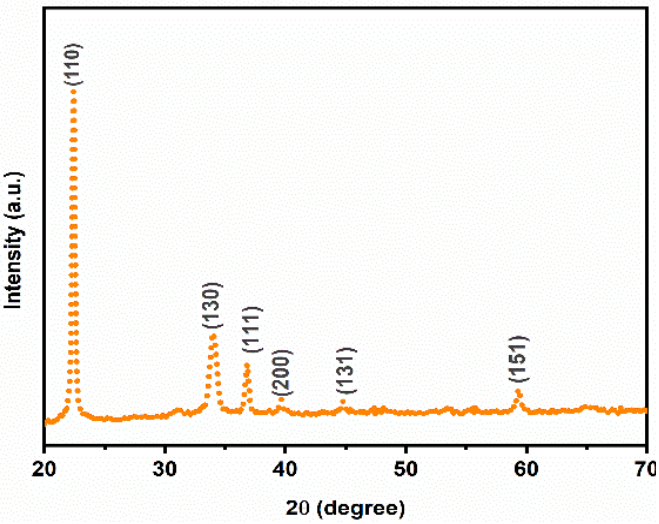


Figure 4. FT-IR spectra of dry *Vitis labrusca* L. extract and α-FeOOH nanoparticles



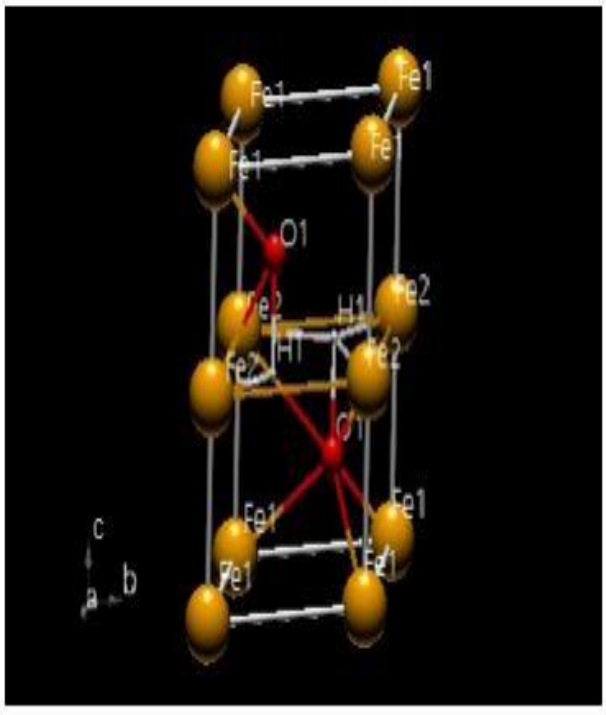


Figure 5. Powder XRD pattern and illustration of the crystal structure of α-FeOOH nanoparticles

Table 1. The structural parameters of the prepared α-FeOOH nanoparticles obtained from XRD analysis

Sample	JCPDS Card no:	Crystal Structure	Space Group	Lattice Parameters (Å)	Cell Volume (Å^3)	Distance (Å)
α-FeOOH	29-0713	Orthorhombic	$P2_1nm$	a= 4.61 b= 9.95 c= 3.02	138.52	Fe1-Fe2= 2.32 Fe1-O1= 2.04 Fe2-O1= 2.06 Fe2-H1= 1.69 O1-H1= 1.22

3.3. SEM-EDX Analysis

The basic composition of the synthesized materials is determined by EDX, a microanalytical technique used in conjunction with SEM. EDX detects X-rays emitted by the sample as a result of electrons bombarding the material surface. The measurement of density and energy provides information on chemical composition. The EDX spectrum for each energy level shows the frequency of X-rays as counts. By determining the intensity of the peak, information about the amount of the element in the sample is obtained.

A morphological analysis of goethite by SEM in Fig. 6 showed that it was composed of agglomerated pseudo-spheres with a spongy appearance, consistent with its low crystallinity [39]. The size of the nanoparticles is in the range of 20–38 nm. When the EDX analysis result of α-FeOOH nanoparticles was examined, the main iron peaks were located at approximately 0.75 keV, 6.5 keV and 7.2 keV. The peak corresponding to oxygen was observed at 0.5 keV (Fig. 7). The peak corresponding to oxygen was observed at 0.5 keV. The atomic percentages of oxygen and iron elements without any impurities were found to be 40.05/59.95 [40].

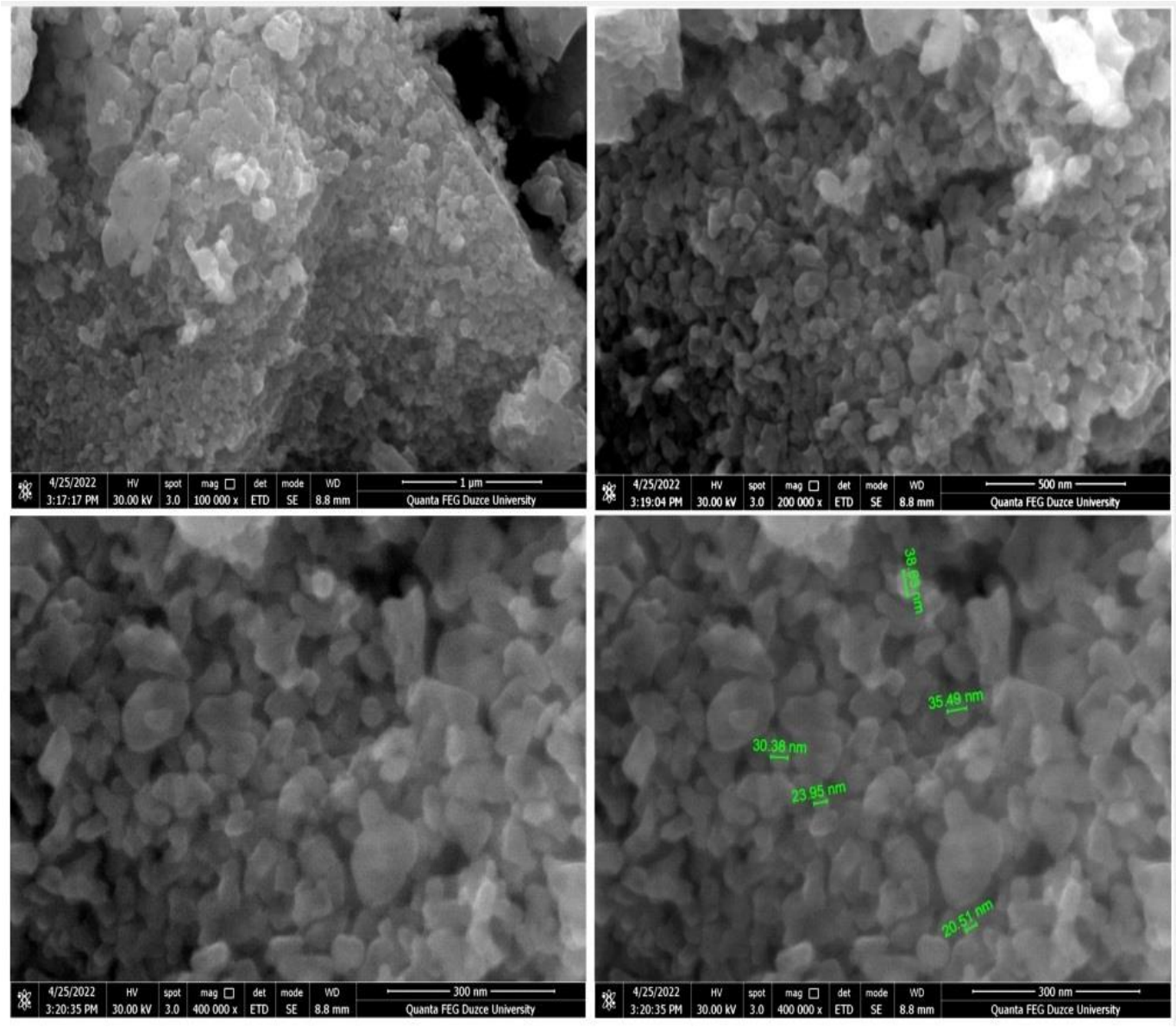


Figure 6. SEM images of the synthesized α-FeOOH nanoparticles, observed at various magnification levels

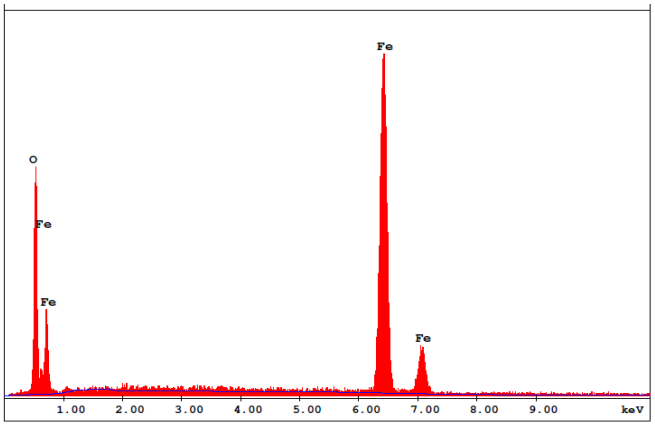


Figure 7. EDX spectrum of α-FeOOH nanoparticles

4. Conclusions

In summary, the aqueous extract of *Vitis labrusca* L. serves as an excellent reducing and stabilising agent for the synthesis of α-FeOOH nanoparticles. The growing interest in green chemistry approaches stems from their resource efficiency and eco-friendliness. Moreover, green-synthesized nanoparticles are devoid of harmful byproducts, making them suitable for biomedical and biotechnological applications. In this study, we successfully synthesized α-FeOOH nanoparticles using a green method mediated by the aqueous extract of *Vitis labrusca* L. We employed FT-IR, XRD, and SEM-EDX analyses to characterize the synthesized α-FeOOH nanoparticles. The FT-IR and XRD results confirmed the purity of the synthesized α-FeOOH nanoparticles, without the presence of other iron compounds. SEM-EDX analysis revealed that the α-FeOOH nanoparticles have a pseudo-spherical structure and fall within the size range of 20–38 nm. This study serves as a valuable reference for the preparation of environmentally friendly, cost-effective, and high-yield α-FeOOH nanoparticles with strong adsorption capacity for analytical applications.

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