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Green Synthesis and Characterization of Iron Oxide Nanoparticles Using *Aronia melanocarpa* Extract

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ABSTRACT

Aronia melanocarpa (Michx.) Elliott, known for its high antioxidant content, can play an effective role in biological reduction processes. Iron oxide nanoparticles (IONPs) are effective drug carriers due to their biocompatibility and biodegradability. They are widely studied for targeted delivery in cancer therapy and related biomedical applications [1]. Nanoparticles synthesized using plant extracts with the green method offer a more sustainable production route and cause less damage to the environment compared to traditional chemical reducing agents. The present study proposes an innovative method for the synthesis of iron oxide nanoparticles (FeO NPs) using lyophilized A. melanocarpa fruit extract as a low-cost and eco-friendly reducing agent. The nanoparticles were characterized by UV–Visible spectroscopy, Raman spectroscopy, scanning electron microscopy (SEM), Energy Dispersive X-Ray analysis (EDX) and optic microscope images of green synthesised FeO NPs. The UV–Visible spectrum of FeO NPs showed the surface plasmon resonance (SPR) band at 295 nm. Functional group and morphology were shown by Raman spectroscopy. The SEM analyses demonstrated that the spherical shape of the nanoparticles with the uniform distribution of the grains. EDX results showed the percentage of elements in synthesized FeO NPs using A. melanocarpa extract. The greenness of the developed UV method was obtained using the analytical GREEnness (AGREE) approach. For densitometric analyses, the AGREE scale was estimated as 0.8. The green synthesized ¬ FeO NPs nanoparticles from A. melanocarpa extract with potential for use in the application of biomedical and environmental applications.

Keywords: Green synthesis, *Aronia melanocarpa,* Iron oxide, Nanoparticles, Plants extract.

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Introduction

Nanotechnology is a research field that is gaining increasing importance in pharmaceutical sciences, as particles reaching nanometer dimensions exhibit higher reactivity compared to their original forms [2]. Nanoparticles (NPs), which range in size from 1 to 100 nanometers, act as an intermediary link between atomic and molecular structures and bulk materials. Due to their small size, large surface area, and free bonds, these structures exhibit higher reactivity than their bulk counterparts and demonstrate remarkable physicochemical properties [3, 4]. The green chemistry approach, which should play a leading role in the development of safe and environmentally friendly nanoparticles, is crucial for the sustainable advancement of nanomaterials and, therefore, should be widely adopted in the field of nanotechnology [5]. Nanomaterials generally exhibit unique and significant differences in their physical, chemical, and biological properties compared to their macroscopic counterparts. These distinctive characteristics grant them a broad range of applications [6]. The synthesis of metal nanoparticles remains a subject of continuous interest for various applications in fields such as catalysis, electronics, optics, environment, and biotechnology [7, 8].

Gold, iron, silver, zinc, titanium, and nickel are commonly used in the form of metal nanoparticles (MNPs) are widely applied not only in the medical and pharmaceutical industries but also in various consumer products [9]. Iron nanoparticles, in addition to possessing excellent dimensional stability, exhibit high magnetic properties, a large surface area, and superior electrical conductivity. These naturally non-toxic iron nanoparticles also have a strong tendency to oxidize easily [10].

The use of plants for the synthesis of nanoparticles (NPs) offers an advantage over other biological methods because it doesn't require a long harvesting period for the reduction of metal ions [11]. As a result, research has increasingly focused on biosynthetic green processes utilizing plant extracts, which are non-toxic and environmentally friendly [12, 13]. Various factors such as temperature, pH, and time play a crucial role in influencing the properties of nanomaterials produced from plant extracts. These factors can affect the size, shape, stability, and functionality of the nanoparticles, making plant-based synthesis an appealing and sustainable alternative for nanomaterial production. They additionally encompass a range of phytochemical constituents, including phenolic compounds, terpenoids, polysaccharides, and flavonoids, all of which exhibit redox properties. These compounds assist in the reduction of metal ions, contributing to the synthesis of nanoparticles [14, 15].

Aronia melanocarpa, also known as chokeberry, belongs to the Rosaceae family and is one of the therapeutic and aromatic plants [16]. *A. melanocarpa*, commonly consumed as fresh fruit, in biscuits, or as tea for colds, originated in North America and spread globally, especially to Central and Southeastern Europe and Southeastern Asia in the 20th century [17]. It is rich in bioactive compounds such as procyanidins, anthocyanins, flavonoids, and phenolic acids, which contribute to its numerous health benefits [16]. The strong antioxidant potential of *A. melanocarpa*, driven by its polyphenol content, contributes to a wide range of health benefits, including antimicrobial, antiviral, antihypertensive, anticoagulant, antilipidemic, antidiabetic, cardioprotective, gastroprotective, hepatoprotective, anti-inflammatory, immunomodulatory, radioprotective, and anticancer effects [16, 18].

Anthocyanin exhibits remarkable antioxidant and anti-inflammatory properties; however, its limited biocompatibility and insufficient stability hinder its broader application [19]. Although anthocyanin is a powerful antioxidant with health-enhancing attributes, its stability is influenced by various factors, including pH, temperature, light exposure, oxygen, enzymatic activity, and the presence of other chemical substances. These factors can degrade its effectiveness and limit its potential applications. NPs offer an effective method for enhancing the stability of anthocyanin by protecting them from environmental factors (light, oxygen, temperature), thus preventing their degradation. Additionally, surface modifications of nanoparticles can create stronger interactions with the chemical structures of anthocyanins, further improving their stability. These modifications can help anthocyanins remain active for a longer period, allowing them to exhibit biological effects more efficiently and maintain their efficacy in therapeutic applications. As a result, controlled release of anthocyanins can be achieved, ensuring their activity is preserved over a desired time frame [20, 21].

As far as we know, no research has been conducted on the green and eco-friendly synthesis of FeO NPs using *A. melanocarpa* extract. UV–Visible spectroscopy, Raman spectroscopy, SEM, EDX, and optical microscope properties have been used to characterize the synthesized green FeNPs.

Materials and Methods

Plants and Chemicals

Aronia fruits used in the analyzes were supplied by a local market in Yalova. Iron chloride were obtained from Sigma Aldrich (Hamburg, Germany). Sodium hydroxide (NaOH) and methanol (CH_3OH) were purchased from Isolab Chemicals (Eschau, Germany). Distilled water was provided by Ankara Medipol University Analytical Chemistry Laboratory.

Instruments

The optical properties of the sample will be analyzed using Shimadzu 1601 double beam path spectrophotometer (Kyoto, Japan). Spectra will be analyzed and evaluated at the appropriate absorbance value using UV-probe software. SEM (ZEISS, Supra 50 V) was utilized to obtain high-resolution images of the surface morphology. EDX (Oxford Instruments, INCA Energy) was used to determine the composition and elemental distribution of the sample. Raman spectroscopy (WITec alpha-300R instrument) was employed to investigate molecular vibrational modes, offering valuable insights into the interactions of chemical bonds within the material. Laboratory Comparison SIF500R (Seoul, South Korea) model incubator shaker was used for incubation. ISOLAB Laborgeräte GmbH (Eschau, Germany) digital pH meter was used for pH parameters and magnetic stirrer. Nüve NF200 (Ankara, Turkey) model centrifuge was used for centrifugation. Weighing was carried out using Shimadzu ATX224 model weighing device (Kyoto, Japan).

Organic Extract Preparation from Aronia melanocarpa

The *Aronia* fruits stored at -40°C before being lyophilized and ground. Extraction was performed according to the method with slight modifications, using a solution of 2% citric acid and 80% ethanol [22]. The mixture was sonicated at 50°C for 60 minutes, then filtered, evaporated, and the yields of the extracts were determined.

Mechanism of Iron Oxide Nanoparticle Synthesis Using Aronia melanocarpa Extract

The biosynthesis of iron oxide (FeO) nanoparticles using plant extract obtained from *A. melanocarpa* plant offers high sustainability as an environmentally friendly method. In this process, first of all, an iron salt such as iron(III) chloride dissolves in aqueous medium and releases Fe^{3+} ions. Natural reducing agents such as polyphenols, flavonoids and anthocyanins in the extract reduce these Fe^{3+} ions to Fe^{2+} ions or directly to FeO nanoparticles. During the reduction, phytochemicals are oxidized and provide a stabilization that prevents agglomeration by binding to the surface of the nanoparticles. The reduced iron species formed undergo nucleation and growth stages under supersaturation conditions and form nanoparticles with homogeneous size and shape. The natural compounds in the extract also play a role in the coating of the nanoparticles and provide long-term colloidal stability. This method provides a green synthesis approach that is both environmentally and economically advantageous by providing energy efficiency without the need for toxic chemicals [23].

Synthesis of Aronia melanocarpa -Mediated Iron Nanoparticles

The biosynthesis of iron oxide nanoparticles (FeO NPs) was accomplished by optimizing the protocols used by previous authors for the environmentally friendly synthesis of FeO NPs using different plant extracts [24-28]. For the synthesis of iron oxide nanoparticles, 10 mg of *A. melanocarpa* extract was dissolved in 30 ml of methanol:water (60:40) mixture. Then, iron chloride was weighed and dissolved so that there was 50 mmol of iron chloride in the mixture. The pH of the solution was adjusted to 10 and incubated at 90°C for 4 hours. After incubation, it was centrifuged 3 times for 15 min at 5000 rpm. The resulting precipitate was dried at 40°C.

Results and Discussion

UV-Vis Spectrophotometer Analysis

In the synthesis of FeO NPs from *A. melanocarpa* fruit extract, the extract amount was successfully optimized. During these optimization studies, 10, 20 and 30 mg of extract were used and the best UV-Visible spectrometer results were observed in nanoparticles obtained from 10 mg of extract (Fig.1). The absorption of FeO NPs was determined at 295 nm wavelength [29].

Raman Spectrophotometer Analysis

Raman spectrum illustrate the Raman spectroscopy of FeO NPs synthesized from *A. melanocarpa* extract (Fig.2). Raman peaks related to FeO NPs were stated at about 414 cm^{-1} E_g, 614 cm^{-1} E_g, in close agreement with previous studies [30, 31]. The peak at 520 cm^{-1} was stated 300 nm SiO_2 coated Si substrate [32].

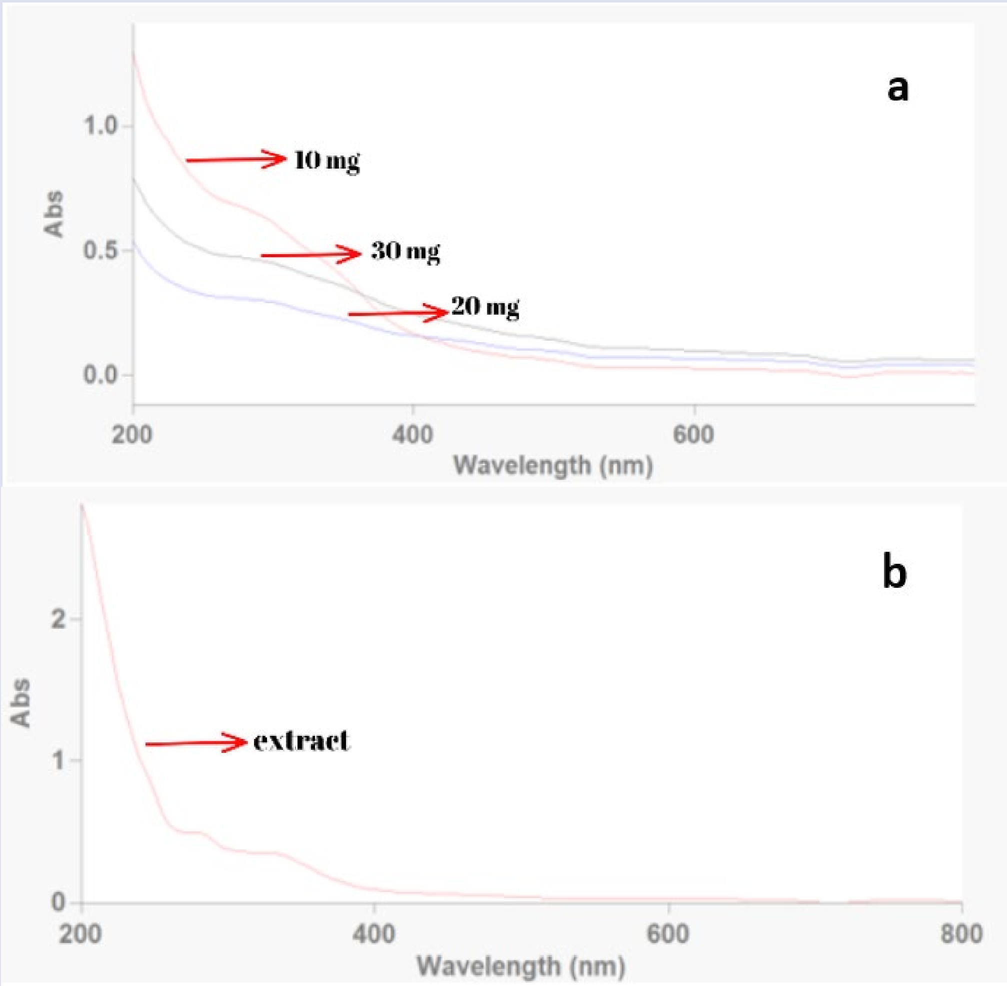


Figure 1. UV-Visible spectra of FeO NP synthesized a) 10mg, 20 mg and 30 mg *of A. melanocarpa* extract , b) *A. melanocarpa* extract

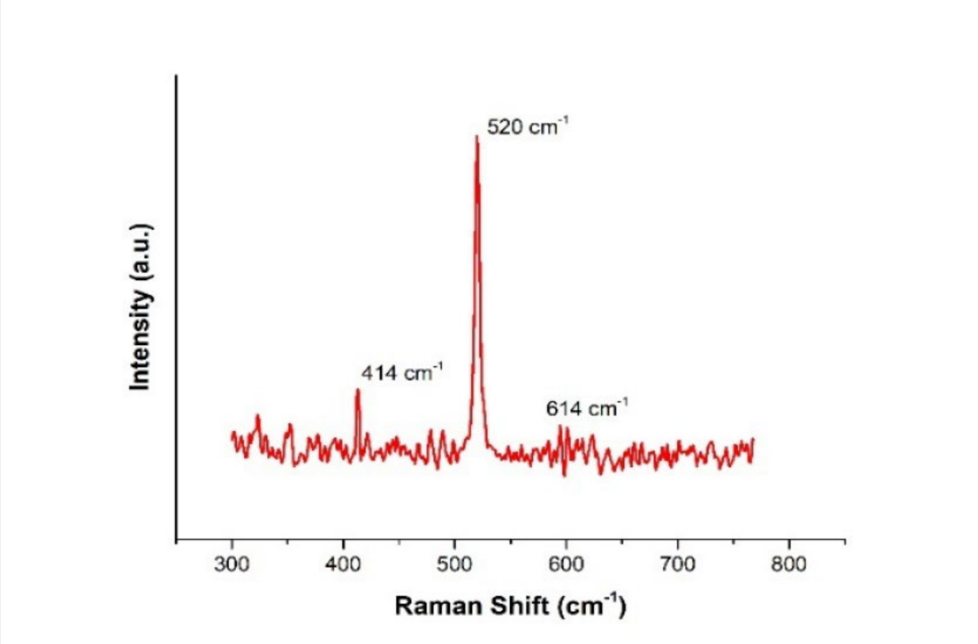


Figure 2. Raman spectra of FeO NP synthesized from *A. melanocarpa* extract

Surface Morphological Properties

SEM images demonstrated the FeO NPs synthesized from *A. melanocarpa* extract with different magnitudes ranges from 1000x to 8000x (Figure 3a-3d). SEM measurement showed that FeO nanoparticles exhibit a uniform growth pattern.

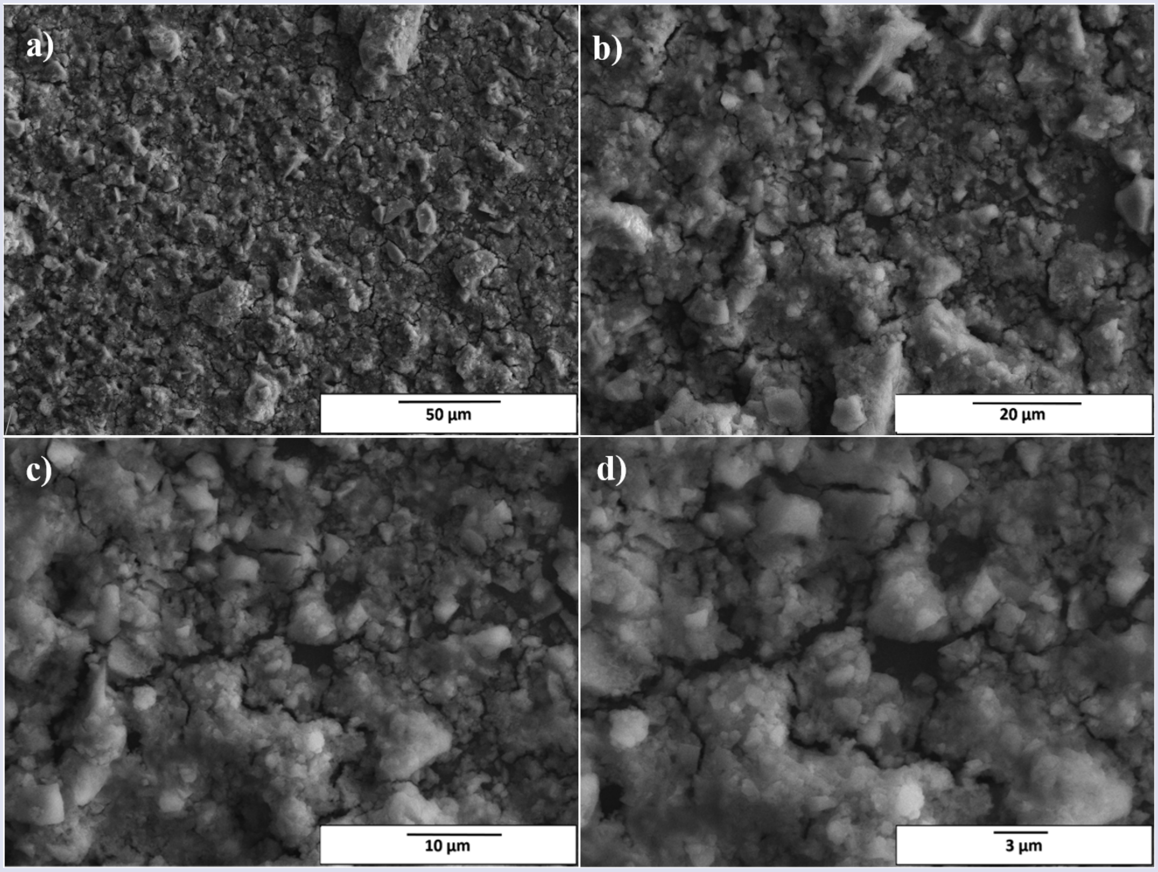


Figure 3a-3d. SEM measurements of FeO NPs synthesized from *A. melanocarpa* extract a) 1000x magnitude, b) 3000x magnitude, c) 5000x magnitude, d) 8000x magnitude

Energy Dispersive X-Ray Analysis

EDX anaysis showed the FeO nanoparticles on SiO_2/Si substrate that the weight percentages are approximately 2.83% C (carbon), 14.74% O (oxygen), 4.38% Na (sodium), 0.23% Si (silicon), 14.66% Cl (chlorine), 0.15 Ca (calcium) and 63% Fe (iron), while the atomic percentages are approximately 8.13% C (carbon), 31.76% O (oxygen), 6.56% Na (sodium), 0.29% Si (silicon), 14.25% Cl (chlorine), 0.13% Ca (calcium) and 38.88% Fe (iron) (Figure 4).

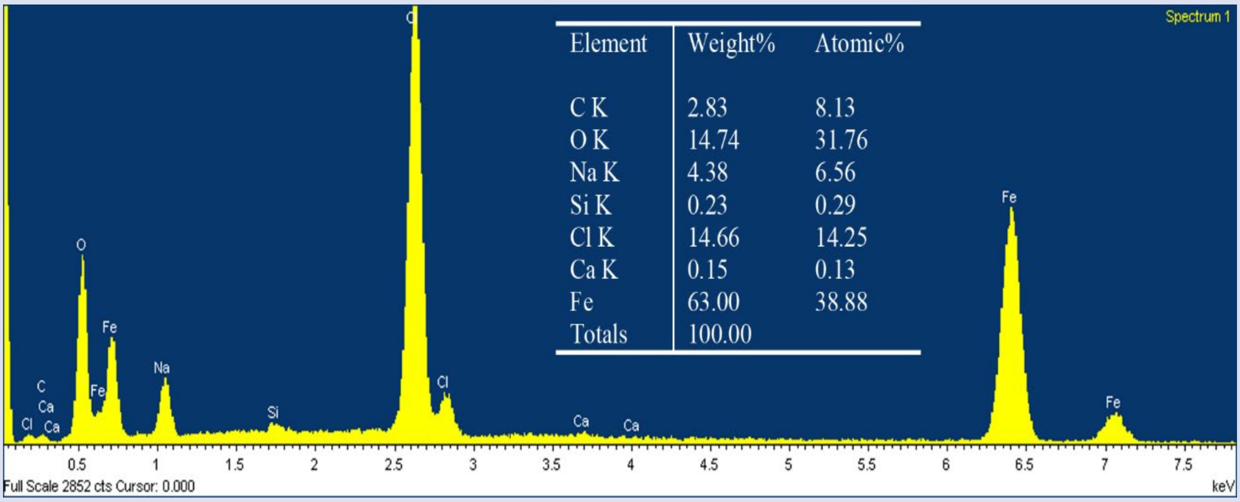
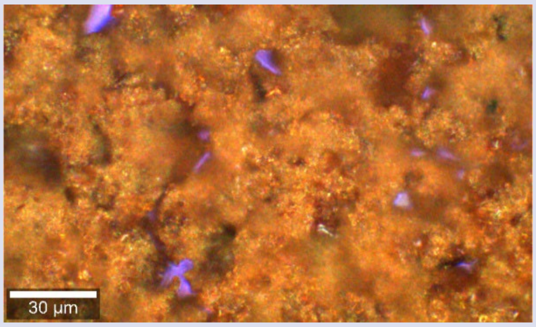


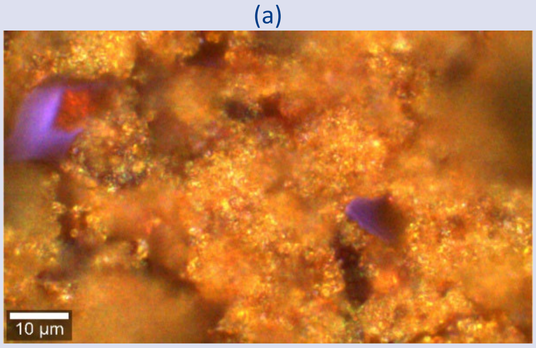
Figure 4. EDX analysis of FeO NP synthesized from A. melanocarpa extract

Optical Microscope Images

The optical microscope images of the synthesized materials at low magnification illustrate the influence of various catalysts on the morphology, size, and size distribution of the FeO nanoparticles. As seen in between Figure 5a and 5b, produced FeO NPs are homogeneously distributed on SiO_2/Si substrate. A decrease in particle size leads to an increase in surface area, which is an important factor in various applications.



(a)



(b)

Figure 5. Optical microscope images of FeO NPs synthesized from *A. melanocarpa* extract a) 30 µm, b) 10 µm

Evaluation of Greenness Profile Using AGREE

AGREE (Analytical Greenness Calculator) is a tool that evaluates the environmental sustainability of analytical methods. Based on the principles of Green Analytical Chemistry (GAC), it scores each method from 0 to 1 according to 12 different principles and visualizes these scores in the form of a clock face.

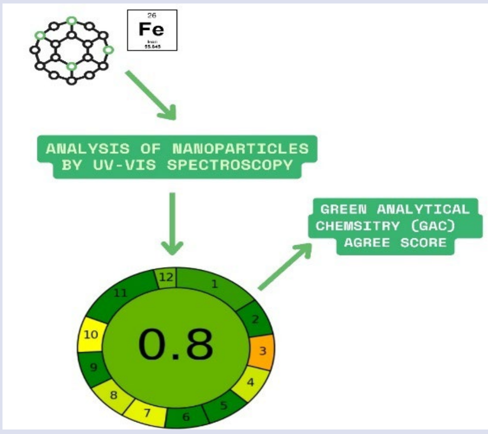


Figure 6. The representative pictogram of the AGREE scale for the proposed UV method was derived using the AGREE calculator.

The color scale, from dark red to green, indicates the environmental friendliness of the method. AGREE encourages the adoption of more sustainable laboratory practices by providing the opportunity to compare different methods [33].

In this study, nanoparticles obtained from the fruits of Aronia melanocarpa plant by green synthesis method were analyzed and characterized by UV-Vis spectrophotometer with an environmentally friendly approach. This analysis, which does not require sample preparation, was performed with only 0.01 g of sample with a direct and miniaturized method and energy consumption was kept at a very low level (0.005 kWh). Measurements were made in-line system and since the device works automatically, operator intervention and safety risks were minimized. No toxic or non-renewable reagents were used, no derivatization was applied and almost no waste was generated. Multi-parameter evaluation was performed with UV-Vis and the method was designed in accordance with the 12 green analytical chemistry principles (Figure 6.).

Conclusions

In this study, iron oxide nanoparticles (FeO NPs) were synthesized using extracts prepared from *A. melanocarpa* fruits, representing the first example of such a synthesis. *A. melanocarpa* extract has a strong antioxidant capacity due to its high polyphenol content in its structure. This enables it to be used as an effective biological agent in the reduction of metal ions. In this way, Fe^{3+} ions can be effectively converted into FeO nanoparticles by natural reducing agents (especially polyphenols such as flavonoids and anthocyanins) in *A. melanocarpa* extract. During the bioreduction process, the extract acts not only as a reducing agent but also as a natural coating/stabilizer that provides stabilization of the formed nanoparticles. In this way, a green synthesis method that does not harm the environment, does not contain toxic chemicals and saves energy is presented. In addition, the biological effects of *A. melanocarpa*, such as antimicrobial and anti-inflammatory, increase the usability of the synthesized FeO nanoparticles in potential biomedical applications.

Characterization processes were carried out using UV-Visible spectroscopy, Raman spectroscopy, SEM, EDX, and optical microscopy. The aqueous extracts of *A. melanocarpa* demonstrated a significant capacity in synthesizing FeO NPs. UV-Visible spectroscopy clearly indicated the formation of FeO NPs with an absorption peak at 295 nm. The Raman spectrum exhibited a peak at 520 cm^{-1} corresponding to a wavelength of 300 nm, confirming the presence of a SiO_2^-coated Si substrate. SEM imaging revealed the distinctive spherical and homogeneous shapes of the synthesized NPs, suggesting significant potential for further applications. The EDX spectra verified the presence of elemental signals within the FeO nanoparticles. Optical microscopy indicated that FeO NPs were evenly distributed on the SiO_2/Si substrate. All these characterization studies suggest that the FeO NPs synthesized from *A. melanocarpa* provide a green, cost-effective, safe, non-toxic, and environmentally friendly approach for mass production. With the progression of research in this subject area, the industrial application of plant-based FeO NPs could offer greater benefits compared to traditional chemical synthesis methods.

Conflict of Interest

No declaration of interests

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