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RESEARCH ARTICLE - PHYSICS

Sol-gel synthesis of IONPS for photocatalytic activities

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Abstract

Iron chloride ($FeCl_2$+$FeCl_3$) salt and Gardenia extract were combined using a sol-gel way to Create iron oxide nanoparticles (IONPs). The biomolecules in Gardenia extract, according to the study, can convert iron salts to IONPs. The technique modifies the phases, shape, size and purity of iron oxide NPs from $FeCl_2$+$FeCl_3$ to ε-Fe_2O_3. Methylene blue removal from water requires ε-Fe_2O_3 NPs in water treatment (MB). For the identification of IONPs, techniques such as XRD, Photoluminescence spectroscopy, ultraviolet microscopy, and scanning electron microscopy were all used. Results from XRD indicated that the average crystal size of 78.85 nm for ε-Fe_2O_3NPs synthesised using the Sol-gel technique. The diffraction results showed tetragonal peaks, and the crystal quality was outstanding. A range of particle sizes (9.04 to 54.65 nm) and an average grain size of 23.67 nm were used in the sol-gel process.1.94eV to 3.12eV, a shift in sol gel way's energy gap was seen in UV-VIS studies. The emission from the near band boundary of ε-Fe_2O_3 NPs by sol-gel method was around 2.74 eV, according to PL spectroscopy. Photocatalytic activity was demonstrated in this work, with The degradation of MB dye transpiring when there was average level of light exposure for the NPs. At 75 minutes for 3 mg and 150 minutes for 5 mg, the degrading efficiency of ε-Fe_2O_3 NPs reached 90.3 percent and 94 percent, respectively, with significant photocatalytic efficacy, according to the results of this study.

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Keywords: Gardenia extract; IONPs; Photocatalytic Activity; Sol-gel procedure; MB dye.

1. Introduction

Nanotechnology is the pitch of science that studies produces at the nanoscale, generally among 1 and 100 nanometers. This technology brings diverse fields of science such as pharmaceuticals, dentistry, and bioengineering[1].Preparation and characterization of iron oxide NPs for photocatalytic activity using different methods: Simple Chemical method, Sol gel method Because of its ability to fabricate electronics like microprocessors, emitting diodes, transistors, and sensors, new nanomaterial development has gained a lot of attention in the physical and chemical sciences. They are used to make antimicrobial and antibacterial medications in addition to cancer treatments. Essential applications such as pollution control catalysts and gas detectors rely on their utilization. Heavy metals, organic and inorganic compounds, and other contaminants can be removed from polluted water by using nanomaterials[2-7]. It has recently been attempted to create ways for monitoring metallic oxide nanoscale structures [8]. Iron oxide (Fe2O3), copper oxide, and zinc oxide are an instances of metallic oxides.[3, 9]. The crust of the planet contains iron oxides in rocks, bedrock, and water and the bodies of a living organisms (animals and plants)[10]. Under normal temperature and pressure, stable Fe2O3 NPs are widespread in nature. Their magnetic field is weak [10] and in most cases, hand magnets have little effect[11]. An entirely new area of nanotechnology has emerged, known as "green synthesis." This technique makes use of a variety of organisms, including bacteria, algae, and plants [12-16]. You may manufacture environmentally-friendly metal NPs using metal oxide nanoparticles, such as iron oxide nanoparticles (IONP). Numerous green manufacturing methods for NPs can employ plants to reduce or eliminate harmful chemicals using metal oxide nanoparticles, such as iron oxide nanoparticles (IONP). Numerous green manufacturing methods for NPs can employ plants to reduce or eliminate harmful chemicals. [11]. Iron oxide (Fe_2O_3) nanoparticles have small band gaps, chemical stability, and magnetic characteristics in addition to other characteristics that produce them a suitable materials for environmental or medical uses [12-15]. The environmentally friendly green synthesis of Fe nano particles is said to be long-term stable, according to the claims. Fe nano particles show excellent antibacterial and toxic cells capabilities, in addition to photocatalytic activity in the degradation of dyes MB [16]. In 2021, E. Paulson, et al., [17] Sol-gel synthesis was used to make hematite (α-Fe_2O_3) nanoparticles. The effect of temperature on hematite nanoparticles produced and calcined was studied. X-ray diffraction spectroscopy was used to characterise produced chemicals (XRD). The arrival of temperature assured that the surface morphological investigation was able to confirm the form, size, and particle homogeneity. Iron oxide nanoparticles, dubbed "Hematite," were made using the simple sol-gel process. At 300 rpm, vigorous stirring was maintained for 15 minutes to dissolve the precursor 0.3 M $FeCl_3$ Ferric chloride anhydrous. A phase transition from a single phased rhombohedra α-Fe_2O_3 nanocrystalline structure with an average crystallite size of 12.7 nm was demonstrated through structural research. Recently, Chauhan etal[17] chemically synthesised iron oxide NPs from Lawsonia inermis gardenia extract . In (2019), Sammy I., etal [18], Galinsoga parviflora, Conyza bonariensis and Bidens pilosa extracts were used to construct IONP catalysts to degrade MB degradation (chemical ways). Sol-gel process is example about bottom-up tactic for producting NPs. Dye MB degradation in the normal light conditions is still a novel notion, however it has been tested with the Sol-gel. Sol-gel can be used to create non-toxic, eco-friendly, and reasonably priced nanoparticles (NPs) with excellent crystalline structures. These nanoparticles are safer for the environment and have a better purity. The green synthesis is more economical and More ecologically friendly than chemical and physical procedures and It can be easily scaled up for large-scale synthesis without using a lot of energy or hazardous chemicals. Green synthesis allows for more control over the crystal formation process. Green nanoparticles are cheap, helpful as well as environmentally friendly [11, 19, 20]. Gardenia extract combined with iron ($FeCl_2$+$FeCl_3$) chloride were utilized to create iron oxide NPs using the sol-gel technique. This work used SEM and high resolution X-ray diffraction to investigate the crystallinity and morphology of the materials, respectively. Photoluminescence instruments and UV-VIS spectrophotometers were utilised to examine these characteristics. Examined was the MB dye's deterioration in typical lighting.

2. Experimental details

2.1 Materials and Processes

Baghdad, Iraq, provided the $FeCl_2$+$FeCl_3$ (iron chloride) and the Gardenia were purchased on the local market there. Eating this plant will provide you with a wealth of Minerals, minerals, amino acids, phenolic acids, and glycosides.

2.2 Preparation the Gardenia extract

To keep away any pollutants, Gardenia employ in this study was sweep up and air dried for a week. It was made into a powder by using stainless steel blender that was utilised by professionals. This extract was created by using 10 grammes with Gardenia powder with 100 mL of deionized water entirely. For two hours, the mixture was heated to 80°C with magnetic stirrer. After bringing the finished product to room temperature and passing it through Whatman paper, It was ready for operation [21].

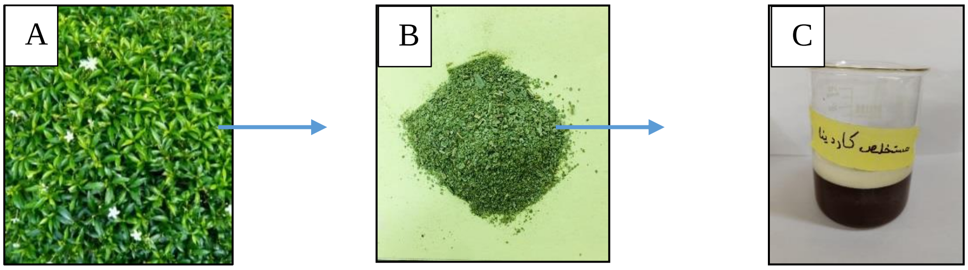

Figure 1: The way for converting the Gardenia to extract for two a hours at 80 oC, a) Gardenia , b) Gardenia powder, c) Gardenia extract.

2.3-Creation of iron oxide nanoparticles utilising extract from Gardenia

$FeCl_3$+ $FeCl_2$ solution, 100 mL Gardenia extract, (0.2 M (2.7 mg) + 0.1 M (0.8 mg), 100 mL) the ratio was 2:1 M with continuous stirring, at 40 °C for 1 hour and then 60 °C for 0.5 hour with constant stirring. After centrifuging the solution for 20 minutes at 4000 rpm, Turbidity resulted by scattered particles appearing in the solution. After the supernatant was collected, the precipitates were filtered and dried at 200 °C. Gardenia extract with $FeCl_2$ and $FeCl_3$ can be utilized to make IONPs, as shown in Fig. 2 [22].

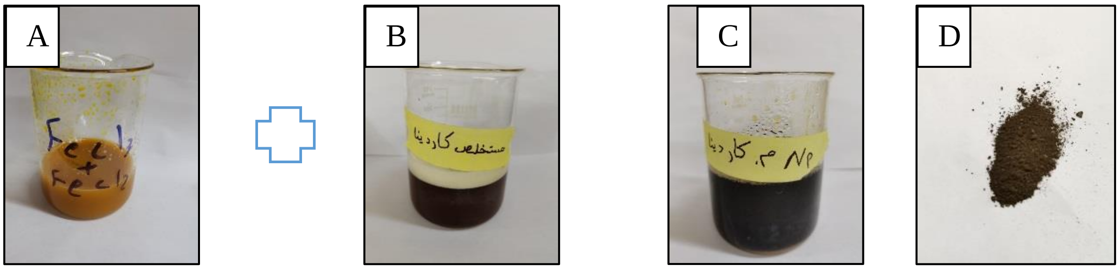

Figure 2: Steps in the sol-gel process that turn the mixture into iron oxide nanoparticles A) $FeCl_2$ + $FeCl_3$ solution, B) Gardenia extract, C) ε-Fe_2O_3 NPs solution, D) ε-Fe_2O_3 NPs powder**.**

2.4 Description of ε-Fe_2O_3 NPs Made up from Gardenia extract.

The specimen in question was identified by XRD analysts with the use of data from the JCPDS card. Measurements in the degree Celsius range from (20–70)°C were made using a step-by-step investigation model (XRD-6000, Shimadzu), using 30 mA and 40 kv of XRD power. The PL spectrum was analysed using a double-beam spectrophotometer (Jobin Yvon HR800UV).

2.5 Iron oxide nanoparticle photocatalytic activity utilising Gardenia extract in ambient light

A predetermined quantity of MB dye liquid (1 mg, 3×10^{-5} M) was combined with 100 mL of ionised water to evaluate the photocatalytic ability of the IONPs. to get an ending level of 10 mg/L in the MB dye liquid. To retain a suspension at a constant temperature and preserve equilibrium, the mixture was stirred using a magnetic stirring device for five minutes in complete darkness after the addition of 3 mg of iron oxide NP powder. The mixture was exposed to direct normal (115 mW/mm2 intensity, as measured by the Ana solar power metre SM 206) after five mins. It is 0.15 metres from the source of light. Following centrifugation for 20 minutes at 4000 rpm, the supernatant from the 5 mL of solution was tested by UV–vis spectrophotometers (Shimadzu, UV-1800). A greatest rate of absorption can be recorded at a 664 nm wavelengths.Changes to the surface of the adsorbent material and dye molecule have a major impact on the interactions between adsorbed substances and dye molecule particles (the adsorbent substance) [23]. Every 10 minutes, the experiment was repeated with a dose of 5 mg.

MB dye's the effectiveness of degradation was computed with the following formula:

Using this procedure, Eq. (1) was used to obtain the MB dye degradation percentage.

$$\text{Percentage of degradation (\%)} = \left[\,1 - \frac{Cfin}{Cini}\right] \times\ 100\ \% \qquad (1)$$

Cfin is the final dye concentration and Cini is original (MB) dye concentration.

Equation (2) was used to determine the kinetic constant rate (Kph) of the degradation of the MB dye.

$$\ln[Cini/Cfin] = Kph \times t \qquad (2)$$

Where t is the radiation time and Kph is the constant rate of MB dye.

The formula for determining the deteriorating efficiency of MB dye is as follows:

Degradation effectiveness (%) =$[\,Cini - \frac{Cfin}{Cini}\,] \times 100\ \%$ (3)

3. Results and discussion

3.1 produce and Description of iron oxide NPs by using Gardenia extract.

IONPs are made by reacting Gardenia extract with iron salt under a variety of circumstances. The characteristics of the Gardenia extract can control the synthesis, field, and stability of iron oxide nanoparticles. Iron can be replaced in a short period of time by phytochemicals in Gardenia extract. It also has a crucial role in the reduction and stabilisation of several variables in simple formation of iron oxide nanoparticles.

3.2 The XRD analysis of iron oxide NPs (ε-Fe2O3) by using Gardenia extract.

XRD testing may be used to examine material composition, structure, and orientation. Sol-gel synthesis of IONPs was achieved by combining Gardenia extract with FeCl2+FeCl3 for two an hours at 200 oC. In a sol-gel way the crystalline peaks (γ-Fe2O3) phase (JCPDS card no. 00-016-0653)) is (220) corresponding to (202), (031), (003), (420), (040), (521), (622) and (352) millers indices, as shown in fig. 3[24, 25]. Table 1 displays the results for IONP (ε-Fe2O3) phases and crystallite dimensions. The crystallite diameter (D) was calculated. applied the following: Scherrer's formula [26, 27].

$$D\ (nm) = \frac{k.\lambda}{\beta\cos\theta} \qquad (5)$$

λ is wavelength (0.15418) nm (CuKα), β is full width at half maximum (FWHM), k is shape factor (0.9) and θ is diffraction angle[27].

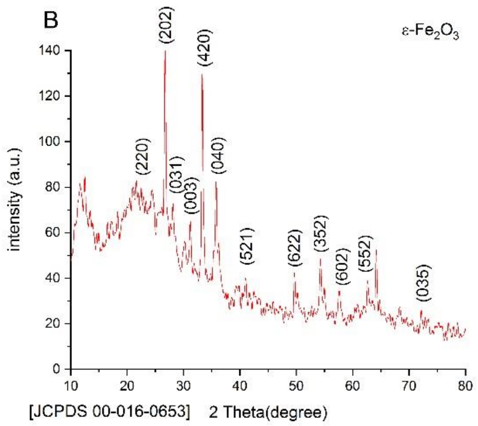

Figure 3: XRD pattern of IONPs extracted by Gardenia employing FeCl2+ FeCl3 salt for 2 hours at 200 °C via the sol-gel technique.

Table 1: results XRD for ε-Fe2O3 NPs by Gardenia extract using FeCl2+FeCl3 for 2h , 200 °C with sol-gel method.

Method	Plant extract	Material	FWHM (deg.)	Plane (hkl)	Crystallite size D (nm)
Sol-gel	**Gardenia**	**ε-Fe2O3**	**0.1557**	**202**	**52.9896**
			0.1852	**420**	**43.8612**
			0.1365	**040**	**60.8562**

3.3 FESEM pictures of IONPs (ε-Fe2O3) created from Gardenia extract.

We used 200 ^{o}C FESEM imaging to investigate the size and surface morphology distributions of eco-friendly IONPs created from Gardenia extract and iron (II+III) chloride. According to the sol-gel process, the typical grain size ranges from 9.04 to 54.65 nm, and Fig. 4 shows the morphology of ε-Fe2O3 NPs (wustite), which has an average grain size of 23.67 nm and a nano rode with a nano spherical shape at 200 ^{o}C. [28].

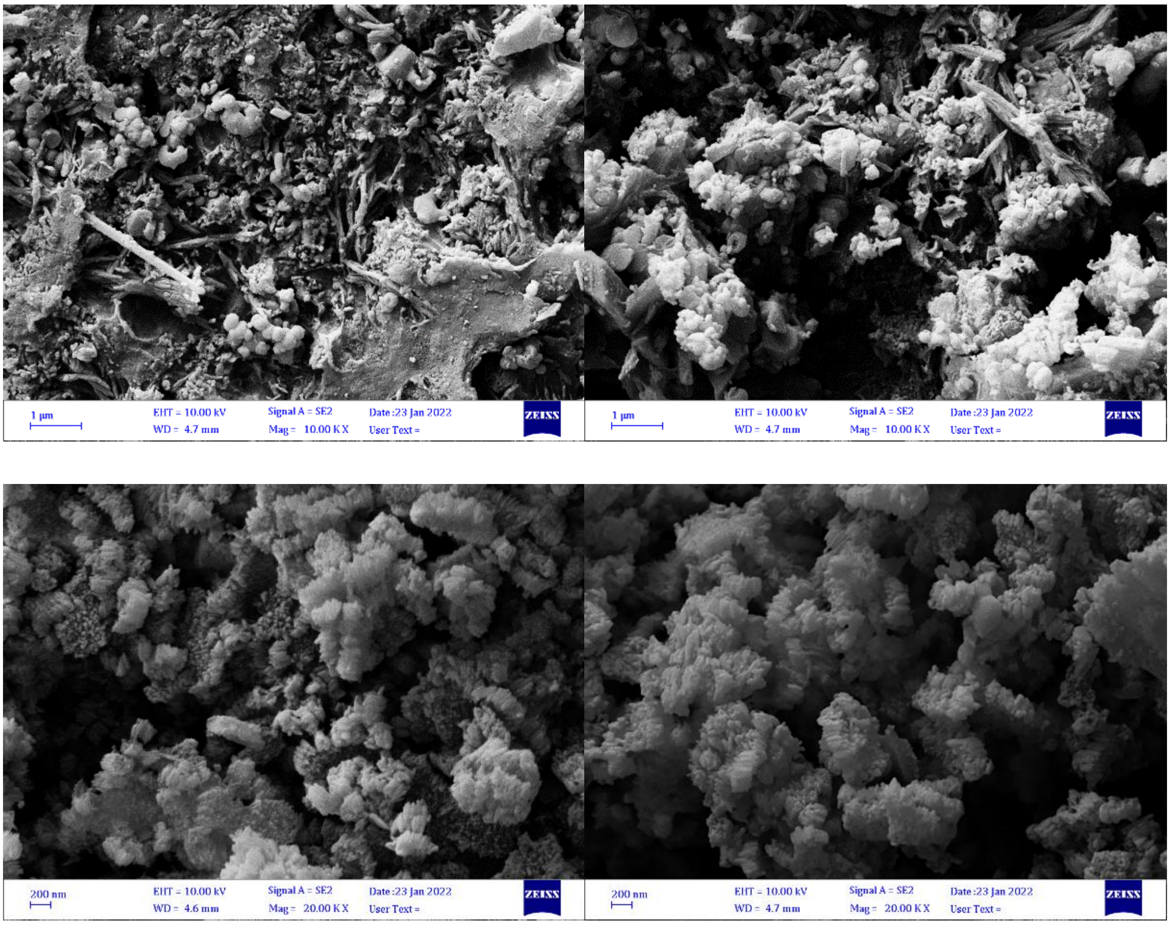

Figure 4: SEM images of iron oxide NPs made sol-gel way by using Gardenia extract with FeCl2+FeCl3 salt for 2 h, 200 °C.

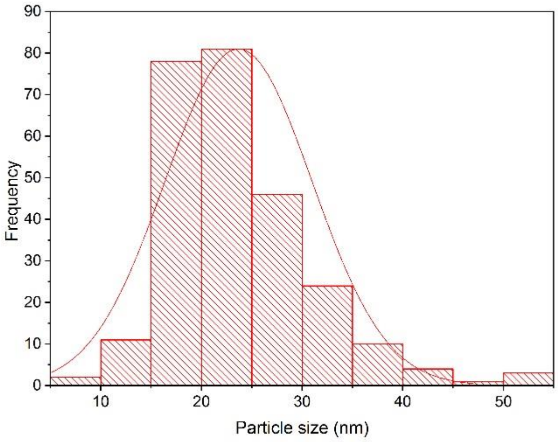

Figure 5: Particle size distribution by sol-gel way

3.4 UV–VisSpectrophotometer of Iron oxide NPs by sol-gel

Using Gardenia extract and FeCl2 + FeC3 salt, a sol-gel was used to measure spectrum of optical transmission of ε-Fe2O3 NPs, as shown in Fig. 6 [29] . It is shown in Fig. 7 that the photon energy (hυ) plotted against the square of the (αhυ)2 shows the energy band gap for ε-Fe2O3 NPs generated by the sol-gel method using Gardenia extract. To find the energy band gap, use a straight line extrapolation to (αhυ)2. The energy band gap of a powder crystal can change in a variety of ways depending on the crystal lattice's layout and distribution of atoms. According to the sol-gel process, the optical band gap for ε-Fe2O3 NPs varied from 1.94 to 3.12 eV as in Fig. 7[10]. Using the equation below, you can get the energy band gap[10, 30].

(αhυ) = A (hυ – Eg)n (6)

In this equation, A is a constant, hυ is light energy, α is the absorption coefficient, and n is a parameter determined by the kind of electron transfer [30].

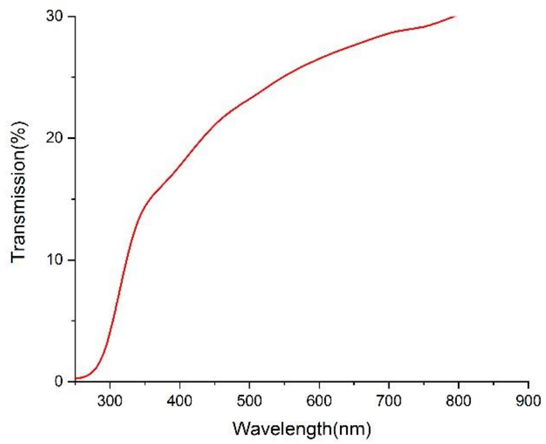

Figure 6: UV-VIS transmission spectra of iron oxide NPs produced by immersing Gardenia extract in FeCl2+FeCl3 salt for 2 hours at 200 °C using the sol-gel technique.

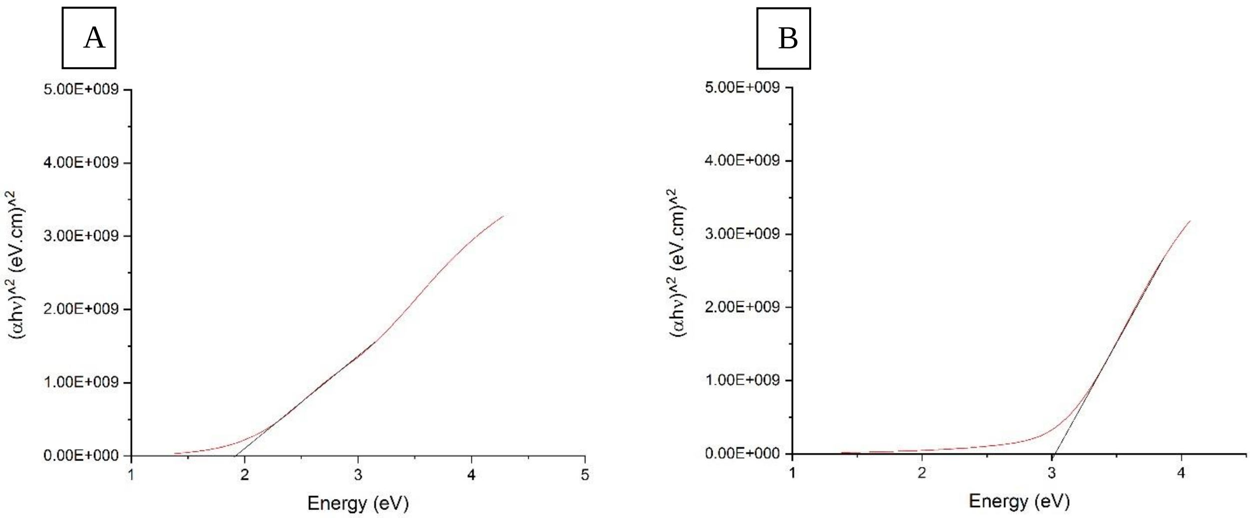

Figure 7: Energy band gap of iron oxide NPs produced from Gardenia extracted with FeCl2+FeCl3 salt using A) bulk FeCl2+FeCl3 material, and B) sol-gel method.

Fig. 6 displays the transmittance spectra of nanoparticles that were annealed at 200 °C. The spectra were recorded in the wavelength range of 810–250 nm. Band gap grew from 1.94 to 3.12 eV at these two wavelengths when iron oxide nanoparticles were annealed at 200 °C. The increase in crystallinity that results in fewer defects and improved crystal structure may be the cause of the band gap that increases in tandem with the annealing temperature [10].

3.5 The PL spectrum of iron oxide NPs (ε-Fe2O3) by using Gardenia extract.

Gardenia extract with FeCl2+FeCl3 at (200oC) may form iron oxide NPs that can be observed in the PL spectrum at the band-edge. Near band edge (2.74) eV in the sol-gel process with the exaction band of 325 nm of ε-Fe2O3 NPs (wustite, the near wavelength of (451 nm)) at 200 ^{o}C is shown in fig. 8 [31].

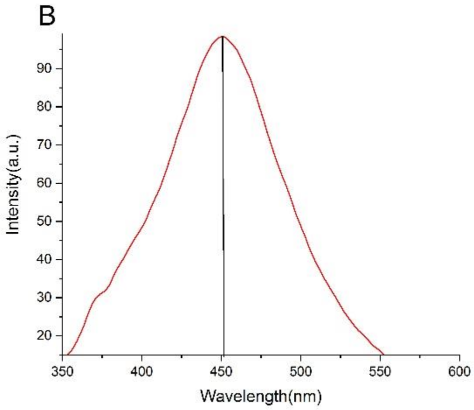

Figure 8: The PL spectra of iron oxide nanoparticles made with Gardenia extract and FeCl2+FeCl3 salt in a sol-gel process at 200 °C for two hours.

3.6 Photocatalytic activity of iron oxide NPs from Gardenia extract under normal light

Table 2: Increased time (min), weight (mg), and degradation efficiency (%) Iron oxide nanoparticles from Gardenia extract and their photocatalytic activity in ambient light

Method	Weight (mg)	Time (min)	Percentage of Degradation (%)
Sol-gel	**5**	**150**	**94**
Sol-gel	**3**	**75**	**90.3**

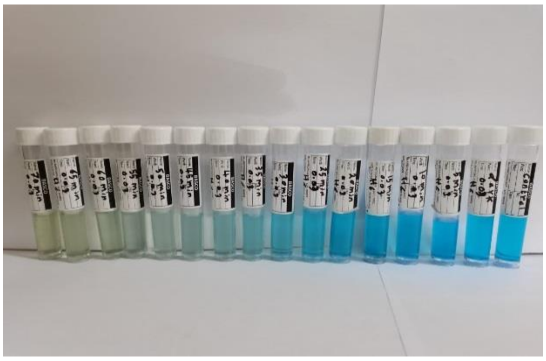

Figure 9: Illustrations showing the stages of MB dye degradation by 3 mg in 1) original dye, 2) ε-Fe2O3 (IONPs) in darkness, 3) after 5 minutes, 4) after 10 minutes, and 5) after 15 minutes 6) after 20 minutes, 7) by Sol-gel, and 8) after 25 minutes, till 75 minutes.

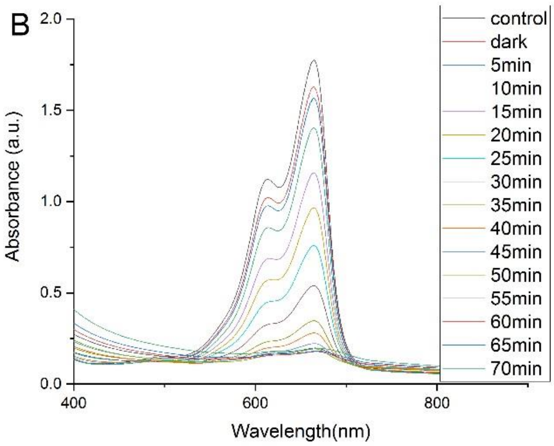

Figure 10: Degradation picture of the MB dye with iron oxide nanoparticles, sol-gel technique under ambient light from Gardenia extracted using FeCl2 + FeCl3 salt for 2 hours at 200 degrees Celsius.

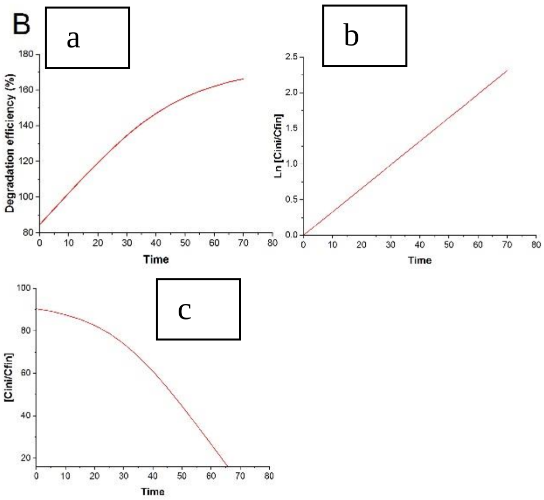

Figure 11 shows: (a) the percentage of MB dye that was degraded at 10 mg/L by iron oxide NPs made from Gardenia extract and FeCl2+ FeCl3 salt for two hours at 200 °C; and (b) the plot of MB dye degradation as a function of light intensity under the same conditions when iron oxide NPs are present. (c) The Sol-gel method's percentage of MB dye degradation.

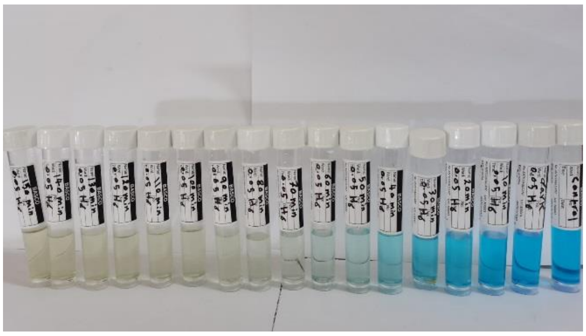

Figure 12: Illustrations of the stages involved in the 5 mg MB dye degradation: 1) using the original dye, 2) using ε-Fe2O3 (IONPs) in the dark, 3) after 10 minutes, 4) after 20 minutes, 5) after 30 minutes, 6) after 40 minutes, 7) after 50 minutes, and so on, using the Sol-gel approach.

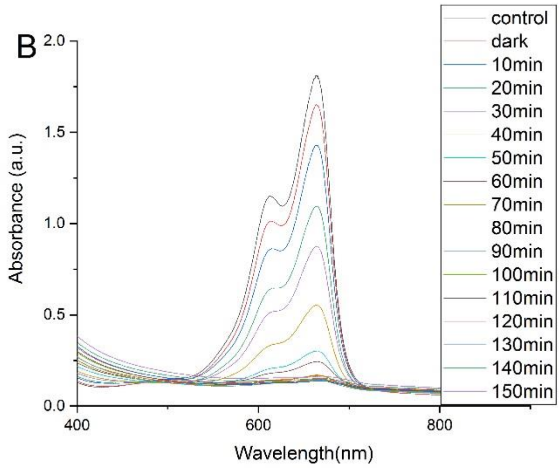

Figure 13: Gardenia extract with FeCl2+ FeCl3 salt is used in the sol-gel method with normal light to degrade MB dye for two hours at 200°C.

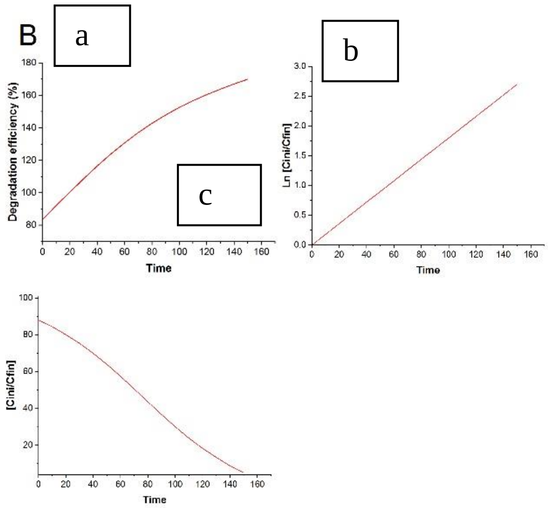

Figure 14(a) shows the percentage breakdown of MB dye at 10 mg/L by iron oxide nanoparticles (NPs) made using Gardenia extract in FeCl2+ FeCl3 salt at 200°C for two hours.(b) A linear plot showing the MB dye's breakdown under standard light irradiation with iron oxide nanoparticles present under the same circumstances. (c) The Sol-gel method's percentage of MB dye degradation.

4. Conclusion

IONPs (ε-Fe2O3) were successfully synthesised by the sol-gel method utilising Gardenia extract and FeCl2+FeCl3 without the use of any catalytic chemical substances in this study. Measurements with XRD showed that the average crystalline size of (ε-Fe2O3) NPs at 200 ◦C using Gardenia extract was (78.85) nm (wustite). Sol-gel NPs of (ε-Fe2O3) were found to have grain sizes ranging from 9.04 to 54.65 nm, with an average grain size of 23.67 nm. The optical near band edge value of ε-Fe2O3 NPs (wustite), 200 ◦C, was moved to the blue by (2.74) eV in sol-gel using the PL spectra of Gardenia. In environmental treatments, iron oxide nanoparticles (NPs) were discovered to exhibit photocatalytic abilities. For 3 mg and 5 mg, the degradation efficiency reached 90.3% at 75 minutes and 94% at 150 minutes with excellent photocatalytic efficacy, respectively. The obtained results indicate that the ε-Fe2O3 NPs produced are of high quality.

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