



Enhanced Photocatalytic Degradation of Organic Dyes in Water Using Oak Leaf-Synthesized Iron Nanoparticles: A Study on Environmental Remediation and Antioxidant Potential

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Abstract This study applies green chemistry principles to promote sustainable and environmentally friendly chemical processes by utilizing plant extracts, which are rich in bioactive compounds, as a sustainable resource for synthesizing nanoparticles. Iron nanoparticles (FeNPs) were synthesized using ferric chloride (FeCl_3) and kermes oak (*Quercus coccifera* L.) leaf extract. The synthesized FeNPs were characterized using UV–Vis spectroscopy, Scanning Electron Microscopy (SEM), X-ray Photoelectron Spectroscopy (XPS), Fourier Transform Infrared Spectroscopy (FTIR), Dynamic Light Scattering (DLS), and Zeta potential measurements. The antioxidant and dye degradation capabilities of the FeNPs were evaluated. The results revealed that the extract is rich in phenolic and flavonoid compounds. According to SEM analysis, FeNPs appeared aggregated and granular. The FeNPs exhibited ABTS and DPPH radical scavenging activities with values of $4.95 \pm 0.52 \mu\text{g/mL}$ and $1.54 \pm 0.014 \mu\text{g/mL}$, respectively. The FeNPs demonstrated exceptional photocatalytic activity in degrading Methylene Blue, Crystal Violet, Congo Red, and Methylene Orange dyes. Experiments conducted over 180 min showed that FeNPs degraded Methylene Blue, Crystal Violet, Congo Red, and Methylene Orange dyes by 89.8%,

73.1%, 50.7%, and 37.9%, respectively. This study highlights the potential of FeNPs for the photocatalytic degradation of organic dyes, emphasizing their importance in wastewater treatment applications.

Keywords *Quercus coccifera* L · Green chemistry · Water pollution · Photocatalytic degradation · Antioxidant

1 Introduction

Water is one of the most vital components for all living organisms on Earth, playing a crucial role in maintaining ecosystem health and supporting life. Over time, water quality has been continuously impacted by various factors, including population growth, environmental changes, and industrial activities. These factors have contributed to the increasing pollution of water resources, posing significant threats to both human health and aquatic ecosystems. One of the primary sources of water pollution is the extensive use of synthetic dyes in various industries, which introduces hazardous chemicals into water bodies. These chemicals reduce the solubility of oxygen in water, disrupting aquatic life and altering the ecological balance. Additionally, synthetic dyes are associated with numerous health risks, including skin irritation, respiratory issues, and potential carcinogenic effects (Bhuiyan et al., 2020). To mitigate water pollution caused by synthetic dyes, various water

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purification methods have been developed. However, many of these methods suffer from limitations such as low efficiency, high energy consumption, and the production of toxic by-products. Consequently, there is a growing need for more effective, sustainable, and eco-friendly alternatives for water treatment (Katata-Seru et al., 2018). In recent years, the synthesis and application of nanomaterials have attracted significant attention due to their unique physical and chemical properties. Among these, iron nanoparticles (FeNPs) have emerged as a promising solution for environmental remediation because of their exceptional catalytic, magnetic, and optical properties. Moreover, the green synthesis of nanoparticles using natural resources such as plants offers a sustainable approach to nanomaterial development. Unlike microbial methods that can be slow and yield low-dispersity nanoparticles, plant-based synthesis provides a faster, more efficient, and more stable alternative (Korkmaz et al., 2024; Saif et al., 2016). Plant-mediated green synthesis utilizes various plant parts such as leaves, roots, stems, or seeds as reducing agents in the formation of metal nanoparticles. These plant materials are rich in polyphenolic compounds, which possess inherent antioxidant properties, making them ideal candidates for nanoparticle synthesis. Additionally, the widespread availability and low cost of plant materials further enhance their appeal as a source for eco-friendly nanoparticle production (Ahmad et al., 2024; Godeto et al., 2023; Roy & Das, 2015). Among the diverse plant species, the *Quercus* genus, commonly known as oak trees, includes around 450 species that thrive in both temperate and tropical climates (Jaber, 2024; Tejerina et al., 2011). *Quercus coccifera*, native to Turkey and the Mediterranean Anatolian region, has been studied for its neuroprotective, antioxidant, antibacterial, and anti-inflammatory properties (Kocazorbaz et al., 2021). To the best of our knowledge, this study represents the first application of *Quercus coccifera* leaf extract for the green synthesis of iron nanoparticles (FeNPs), utilizing it both as a reducing agent and as a stabilizer in nanoparticle formation. Additionally, this is the first investigation into the photocatalytic degradation of synthetic dyes using FeNPs synthesized from *Quercus coccifera* leaf extract, as well as their antioxidant potential. This novel approach contributes significantly to the field of green chemistry and provides new insights into sustainable water treatment solutions. In this study,

Quercus coccifera leaf extract was utilized as both a reducing agent and a stabilizing agent in the green synthesis of iron nanoparticles (FeNPs), negating the need for additional chemical stabilizers or capping agents. This research aims to synthesize FeNPs using *Quercus coccifera* leaf extract, characterize these nanoparticles, and evaluate their effectiveness in the photocatalytic degradation of various synthetic dyes, including methylene blue (MB), methyl orange (MO), congo red (CR), and crystal violet (CV), as well as their antioxidant potential.

2 Material and Methods

2.1 Preparation of *Quercus coccifera* Leaf Extract

The fresh kermes oak (*Q. coccifera*) leaves were initially identified with the assistance of the Biology Department, at Ege University, Izmir, Turkey. The leaves were then washed multiple times with tap water followed by deionized water. Subsequently, the leaves were air-dried at room temperature and later processed into a powder using a laboratory grinder (Retsch 200). The plant leaf extract was obtained by heating 10 g of *Quercus coccifera* leaves in 250 mL of Milli-Q water at 60 °C for 12 h. The extract solution was left to cool at room temperature and centrifuged at 9000 g for 30 min then was filtered through Whatman filter paper. The leaf extract was designated as 'Quercus Coccifera Leaf Extract' (QCLE) and stored at 4°C for further use.

2.2 Phytochemical Analysis of *Quercus coccifera* Leaf Extract

The determination of total phenolic groups was conducted using Folin–Ciocalteu assay, with gallic acid serving as a reference, as outlined by Molole et al., 2022. Total flavonoids in QCLE were determined following the protocol described by Alighiri et al., 2020.

2.3 Preparation of QCLE-FeNPs

The environmentally friendly synthesis of Fe₂O₃-NPs using QCLE was carried out according to Katata-Seru et al., 2018. For the preparation of Fe₂O₃-NPs, a mixture of 0.1 M FeCl₃ solution and the QCLE in a 1:2 (v/v) ratio was stirred for one hour on a mechanical

stirrer. The formation of green FeNPs was marked by the appearance of a black color. Subsequently, the nanoparticles were centrifuged at 9,000 rpm for 30 min. and the supernatant was discarded. To purify the nanoparticles from impurities, they were washed twice with deionized water and finally once with ethanol. The products were kept in a hot air oven (80 °C) and dried.

2.4 Characterization of QCLE-FeNPs

Particle characterization was carried out employing various analytical techniques. UV–Vis spectroscopy (Perkin Elmer Lambda 35 UV/VIS Spectrometer) was utilized to obtain absorption spectra in the range of 300 to 800 nm. The size and distribution of FeNPs synthesized based on QCLE were determined using Dynamic Light Scattering (DLS, Malvern Zetasizer Nano). The structure of the plant extract and the changes in the functional groups involved in the reduction reaction were examined using FTIR analysis (Perkin Elmer Spectrum One). Nanoparticle surface compositions were analyzed by XPS (Thermo K-Alpha XPS). The size and morphology of the iron oxide nanoparticles were determined using a Scanning Electron Microscope (SEM) EVO 40 LEQ.

2.5 Antioxidant Potential of QCLE-FeNPs

The antioxidant potential of the synthesized iron nanoparticles was assessed using two distinct assays: the 2,2-diphenyl-1-picrylhydrazyl (DPPH) assay and the 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) assay.

2.6 DPPH Assay

A 0.1 mM stock solution of DPPH radical was prepared in absolute ethanol. Various concentrations of synthesized iron nanoparticles were diluted accordingly. Each nanoparticle solution (2 mL) was mixed with 2 mL of the DPPH radical solution and vortexed. After 30 min of incubation in darkness at room temperature, the absorbance was measured at $\lambda=517$ nm using a UV–visible spectrophotometer (Pavithra & Vadivukkarasi, 2015). Absolute ethanol served as a blank, while Trolox was used as a standard antioxidant for comparison. In this study, the scavenging activity of the sample was quantified using the 50%

effective concentration (EC_{50}), which indicates the sample concentration ($\mu\text{g/mL}$) required to inhibit 50% of the DPPH radical activity.

2.6.1 ABTS Assay

The ABTS radical cation was generated by mixing 7 mM ABTS solution with 2.45 mM potassium persulfate solution in equal volumes and incubated in darkness at room temperature for 16 h. Before the assay, the ABTS radical cation solution was diluted to achieve an absorbance of 0.7 ± 0.02 at $\lambda=734$ nm. Various concentrations of synthesized iron nanoparticles were prepared. Each nanoparticle solution (100 μL) was added to 100 μL of the diluted ABTS radical cation solution and incubated at room temperature for 5 min. Absorbance was then measured at $\lambda=734$ nm using a UV–visible spectrophotometer (González-Palma et al., 2016). In this study, the scavenging activity of the sample was quantified using the 50% effective concentration (EC_{50}), which indicates the sample concentration ($\mu\text{g/mL}$) required to inhibit 50% of the ABTS•+ activity.

2.7 Fe-NPs Based Dye Decolorization

The decolorization efficiency of the synthesized iron nanoparticles (Fe-NPs) was evaluated using four synthetic dyes: Methylene Blue (MB), Methyl Orange (MO), Congo Red (CR), and Crystal Violet (CV). In each experiment, 1 mg of Fe-NPs was added to 1 mL of dye solution (12.5 mg/mL, pH 7.0). The mixtures were incubated at 37°C for various time intervals ranging from 1 to 3 h. Following incubation, the solutions were centrifuged at 10,000 rpm for 1 min. The decolorization progress was monitored by measuring the absorbance changes of the dye solutions at different time points using a spectrophotometer. The percentage of dye removal was calculated using the formula:

$$\text{Percentage Dye Removal} = (A_0 - A_t/A_0) \times 100$$

Where A_0 is the initial absorbance of the dye and A_t is the absorbance at time t of the dye (Nathan et al., 2019).

The following equation was used for the photocatalytic degradation experiment.

$$\text{Degradation rate(\%)} = (C_0 - C_t) / C_0 \times 100$$

In the equations; C_0 is the initial concentration before irradiation, C_t is the concentration at the relevant irradiation time.

2.7.1 Reusability of Fe-NPs

In this study, the reusability of Fe-NPs was investigated by adding fresh MB, MO, CV and CR dye solutions to the reactor while maintaining the same reaction conditions. In the first step, Fe-NPs were centrifuged, then purified by washing with water and ethanol and dried in an oven at 70°C for four hours. The recycled nanoparticles were used as photocatalysts during five replicate experiments. After each run, the same amount of fresh MB, MO, CV and CR dye solutions were added to evaluate the stability of the photocatalyst.

3 Results and Discussion

3.1 Synthesis of Fe-NPs Using Quercus coccifera Leaf Extract

The kermes oak leaves (*Quercus coccifera* L.) aqueous extract (QCLE) revealed the presence of flavonoids at 14.93 ± 0.11 mg/g and phenolics at 48.0 ± 2.8

mg/g. QCLE was observed as a light-yellow solution. The slow addition of FeCl_3 solution induced a gradual color change from light yellow to intense black (Fig. 1). The color change is an indicator that ferric (Fe^{+3}) ions are being reduced, leading to the formation of iron nanoparticles. Iron nanoparticles consist of a metallic core surrounded by an oxide layer. The oxide layer is formed through the reaction of the metallic core with oxygen or water (El-Kassas et al., 2016; Ibrar et al., 2024).

3.2 UV–Vis Spectrum of QCLE-FeNPs

The components forming QCLE-FeNPs synthesized through the green synthesis method were characterized by UV–VIS spectroscopy with an area scan in the wavelength range of 200–800 nm. The maximum peak absorbance value of the nanoparticles was read at 288 nm, indicating a high energy absorption. The UV–vis spectra of QCLE-FeNPs (Fig. 1) indicate the absorption band at 288 nm as reported elsewhere (Lesiak et al., 2019). It is known that the bands arising from resonances in the surface plasma of iron electrons are in the range of 205 nm and 350 nm (Gierschner et al., 2012). It validates the production of QCLE-FeNPs, with the robust width of the absorption peak indicating a uniform and well-distributed particle size (Pathan et al., 2022).

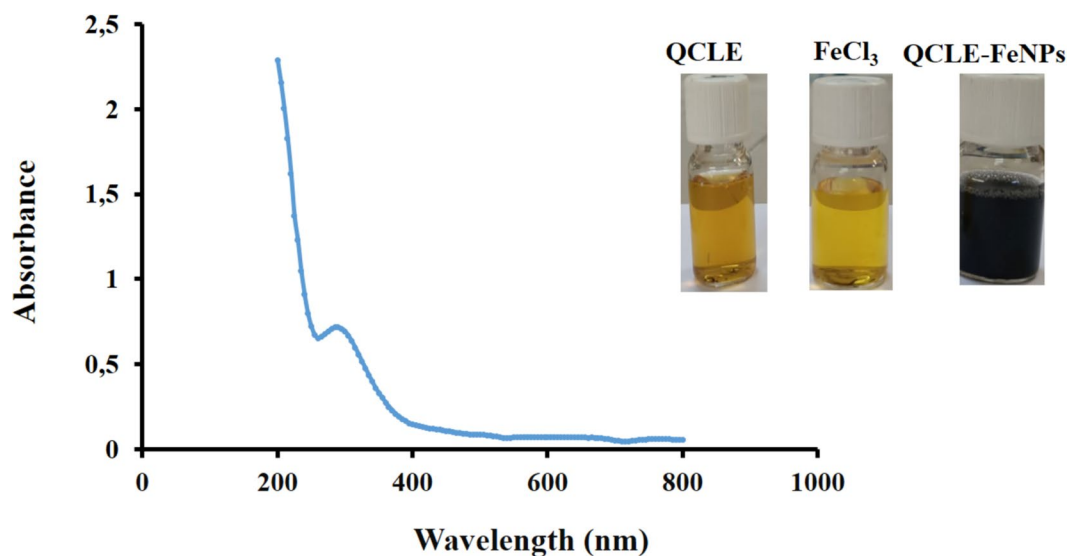


Fig. 1 UV–Visible spectrum of QCLE-FeNPs

3.3 DLS and Zeta Potential of QCLE-FeNPs

Dynamic Light Scattering (DLS) analysis was employed to determine the hydrodynamic diameter of the synthesized iron nanoparticles. The obtained data indicated an average size of 514.9 nm (± 107.9) for the iron nanoparticles. These results provided valuable insights into the size distribution and stability of the nanoparticles (Fig. 2).

Zeta potential measurements were conducted to assess the surface charge of the synthesized iron nanoparticles. The obtained data revealed a zeta potential of -23.7 mV for the iron nanoparticles. These results offered significant information regarding the surface charge and dispersion behavior of the nanoparticles (Fig. 2).

3.4 FTIR Analysis of QCLE-FeNPs

The FTIR spectrum of iron oxide nanoparticles is shown in Fig. 3. The peak observed at 3345.35 cm^{-1} represents the frequency of vibration movements belonging to C-H groups. The peaks at 1612.80 cm^{-1} and 1337.45 cm^{-1} arise from the asymmetric and symmetric stretching vibrations of carbonyl groups

(C=O). These peaks likely originated from the carboxylic acid groups present in the kermes leaf extract used in the synthesis of FeNPs. The peak at 1047.74 cm^{-1} is attributed to the protein bonds (C–O–C and C–OH) in the extract. The observation of bands at 565.68 cm^{-1} suggests the formation of Fe–O bonds and supports the synthesis of $\text{Fe}_2\text{O}_3\cdot\text{Np}$. The observed bands closely resemble those reported for iron nanoparticles (Madhu et al., 2019).

3.5 XPS Analysis of QCLE-FeNPs

XPS measurements were carried out to examine the chemical states of Fe and O elements in iron oxide nanoparticles. As shown in Fig. 4c, the O1s spectrum reveals a primary peak at 533.2 eV. This peak is ascribed to lattice oxygen, suggesting the possible presence of oxygen (O_2) in the iron oxide nanomaterials. As shown in Fig. 4b, the binding energies for the $\text{Fe}2p_{1/2}$ and $\text{Fe}2p_{3/2}$ signature peaks are observed around 712.76 eV and 739.1 eV, respectively, aligning well with reference values for Fe_2O_3 (Ahmmad et al., 2013). The carbon peak at 285.94 eV (Fig. 4d) indicates the presence of aliphatic or alkyl carbon, confirming that the plant bioactive molecules are

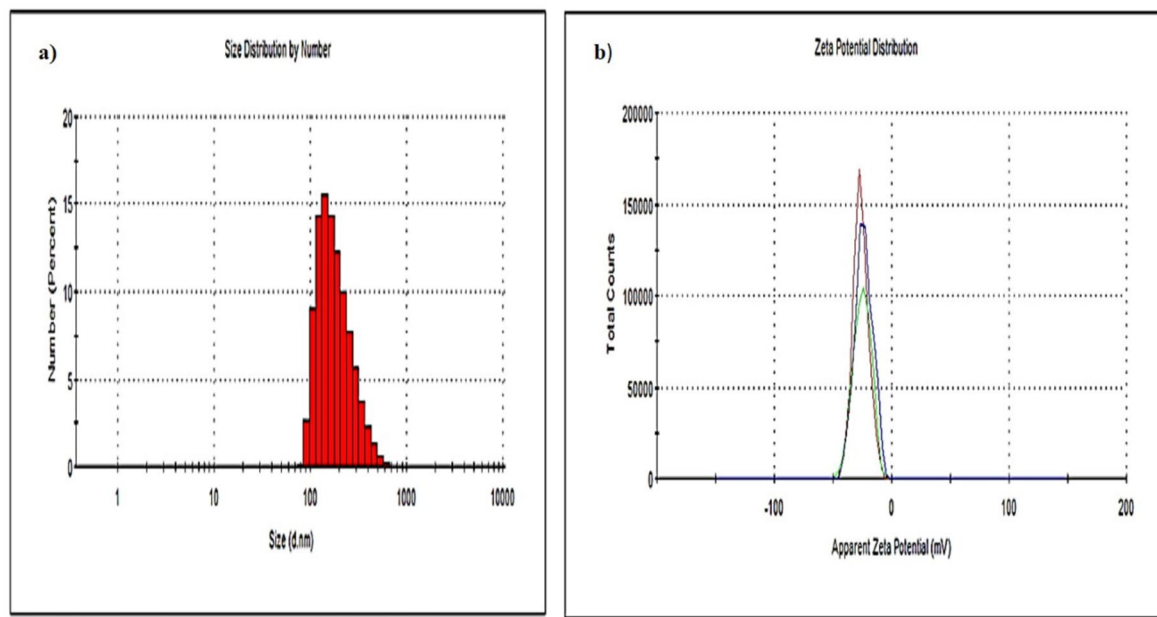


Fig. 2 a) Dynamic light scattering analysis and b) zeta potential analysis of QCLE-FeNPs

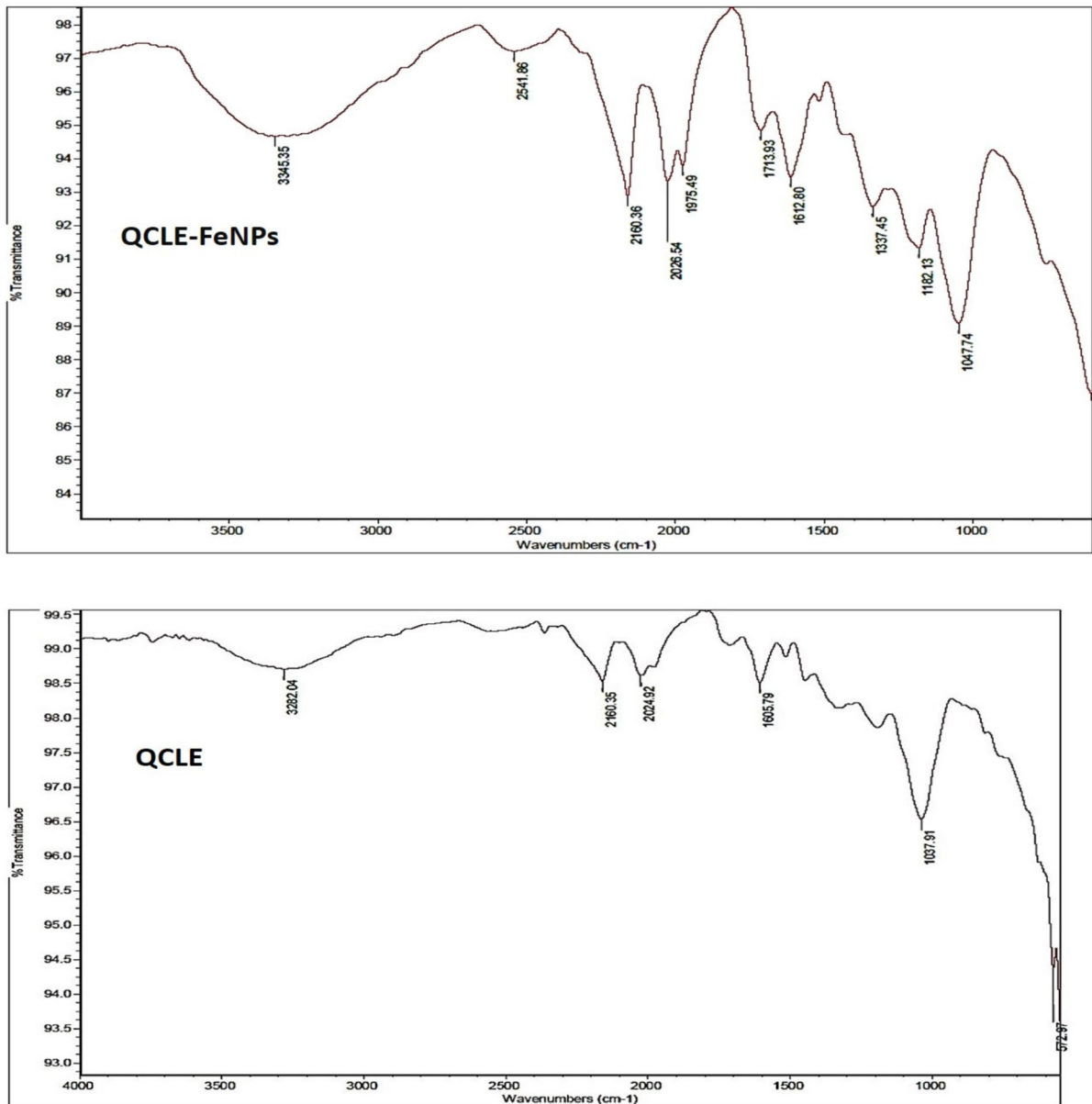


Fig. 3 FTIR spectrum of QCLE (top panel) and FTIR spectrum of QCLE-FeNPs (bottom panel)

attached to the surface of the QCLE-FeNPs (Vinayagam et al., 2020).

3.6 SEM Analysis of QCLE-FeNPs

The iron nanoparticles' morphology was characterized by the use of a scanning electron microscope

(SEM). The produced nanoparticles show a non-homogeneous distribution, as shown in Fig. 5 above. The average particle diameter, it was discovered, was less than 50 nm. Furthermore, there is variation in the size of nanoparticles. Dlamini et al. (2020) reported the non-homogeneous size distribution of green synthesized nanoparticles.

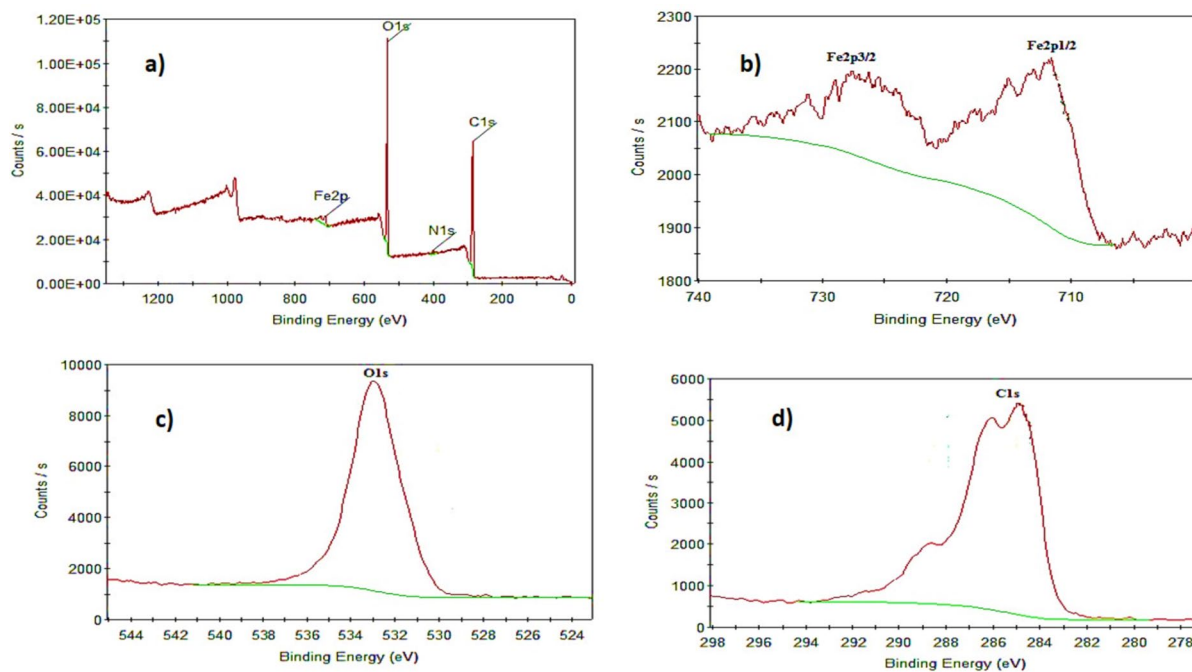


Fig. 4 XPS spectrum of (a) QCLE-FeNPs, (b) Fe 2p, (c) O 1 s, (d) C 1 s,

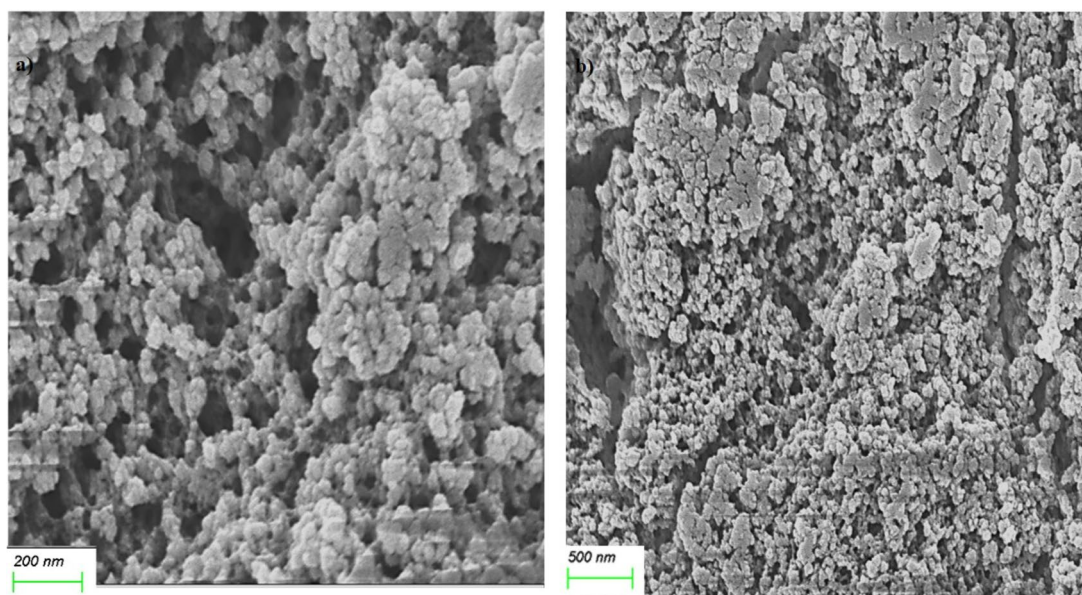


Fig. 5 SEM image of QCLE-FeNPs a)200 nm b)500 nm

3.7 Antioxidant Activity of QCLE-FeNPs

The scavenging property of the biosynthesized Fe_2O_3 -nanoparticles was assessed through both the DPPH and ABTS assays. The EC_{50} value was utilized to quantify the antioxidant activity. As shown in Fig. 6 displays the results of the DPPH and ABTS assays for various concentrations of QCLE-FeNPs, illustrating their antioxidant activity. The results demonstrate that the biosynthesized QCLE-FeNPs exhibited EC_{50} values of 1.54 ± 0.014 $\mu\text{g/mL}$ for DPPH scavenging and 4.95 ± 0.52 $\mu\text{g/mL}$ for ABTS scavenging, while the standard Trolox showed EC_{50} values of 10.0 ± 0.13 $\mu\text{g/mL}$ for DPPH scavenging and 7.43 ± 0.609 $\mu\text{g/mL}$ for ABTS scavenging. These findings indicate the antioxidant activity of both the nanoparticles and the standard trolox in scavenging DPPH and ABTS radicals. This study revealed that the positive control exhibited higher inhibition compared to the QCLE-FeNPs. Previous studies have provided evidence supporting the excellent antioxidant activities of QCLE-FeNPs (Kumar et al., 2020; Shab-bir et al., 2023). The radical scavenging activities of the synthesized nanoparticles can be attributed to

the presence of bioactive components in the extract, which possess strong antioxidant activity. As a result, the surface-functionalized iron oxide nanoparticles exhibit significant scavenging activity against free radicals (Sandhya & Kalaiselvan, 2020).

3.8 FeNPs-Based Dye Decolorization

The decolorization efficiency of Fe_2O_3 nanoparticles synthesized from *Quercus coccifera* leaves was evaluated for four synthetic dyes: Methylene Blue (MB), Methyl Orange (MO), Crystal Violet (CV), and Congo Red (CR) under standardized conditions of pH 7.0 and 37 °C. The absorbance changes were recorded at specific wavelengths in the 400 to 600 nm range following a 3-h incubation period for each dye. Treatment with QCLE-FeNPs resulted in a significant reduction in color intensity, as demonstrated in Fig. 7.

For the four dyes tested, maximum absorption peaks were observed at 665 nm for MB, 470 nm for MO, 585 nm for CV, and 510 nm for CR. Over time, these absorption peaks gradually decreased, indicating the effective degradation of MB, MO, CV, and CR due to the photocatalytic activity of the

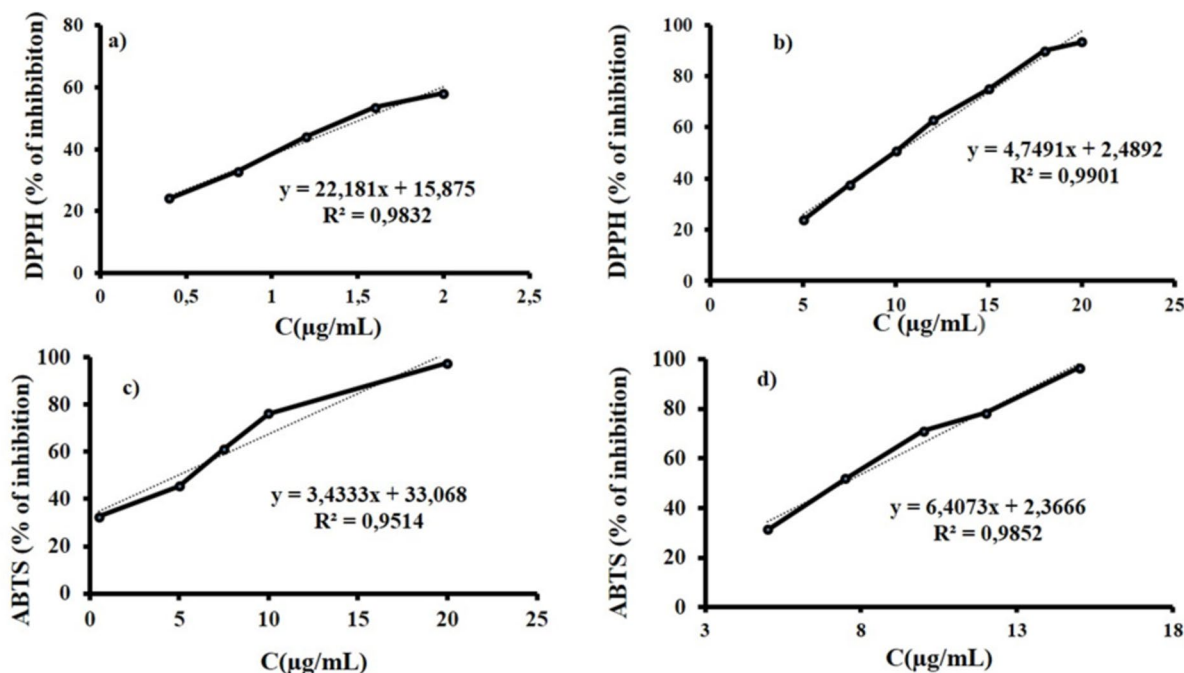


Fig. 6 Antioxidant activity of QCLE-FeNPs a) QCLE-FeNPs for DPPH assay b) Trolox for DPPH assay c) QCLE-FeNPs for ABTS assay d) Trolox for ABTS assay

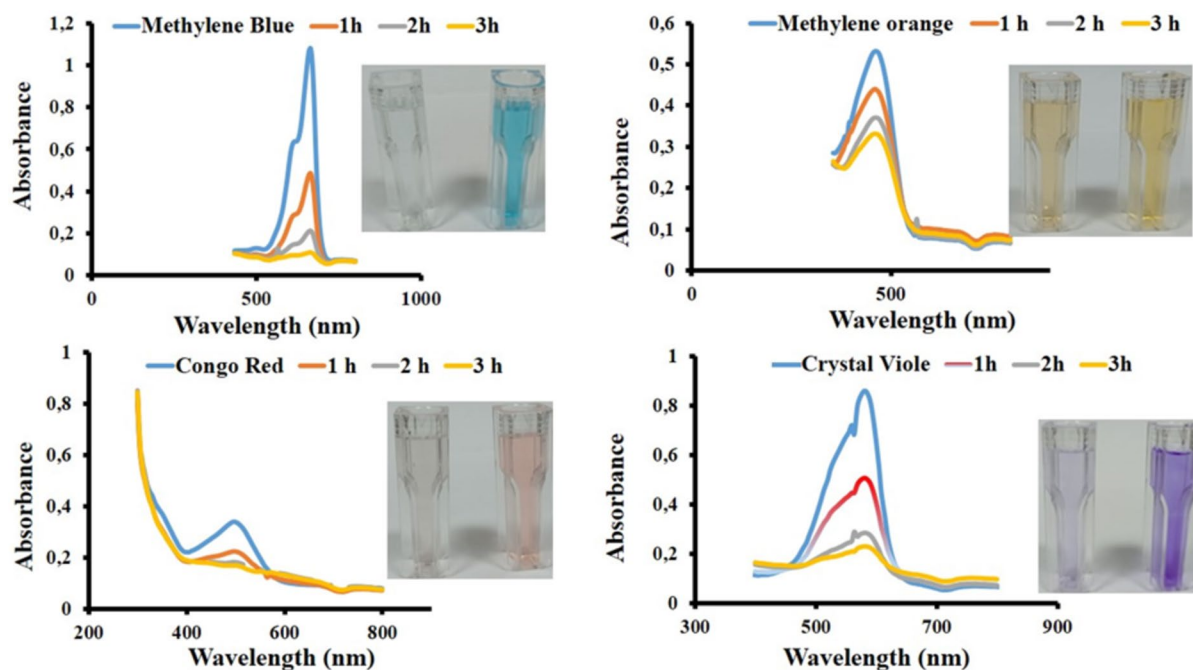


Fig.7 Decolorization analysis of QCLE-FeNPs

QCLE-FeNPs, as shown in Fig. 7. Specifically, the decolorization percentages achieved by QCLE-FeNPs were 89.75% for MB, 73.13% for CV, 50.7% for CR, and 37.9% for MO, following incubation. These results align with previous studies that corroborate the effectiveness of QCLE-FeNPs in dye degradation (Dharshini et al., 2021; Mbachu et al., 2023; Sarojini et al., 2022; Yassin et al., 2023). The findings confirm

that QCLE-FeNPs are highly effective in degrading MB, MO, CV, and CR, showcasing their potential for practical applications in wastewater treatment and environmental remediation.

In the experiments, the initial (C_0) and time interval (C_t) concentrations of each dye solution were measured to evaluate the photocatalytic degradation kinetics and the results were analyzed with half-life

Fig.8 Photocatalytic degradation kinetics of MB,MO;CV, and CR dye. $\ln(C/C_0)$ vs time plots for QCLE-FeNPs

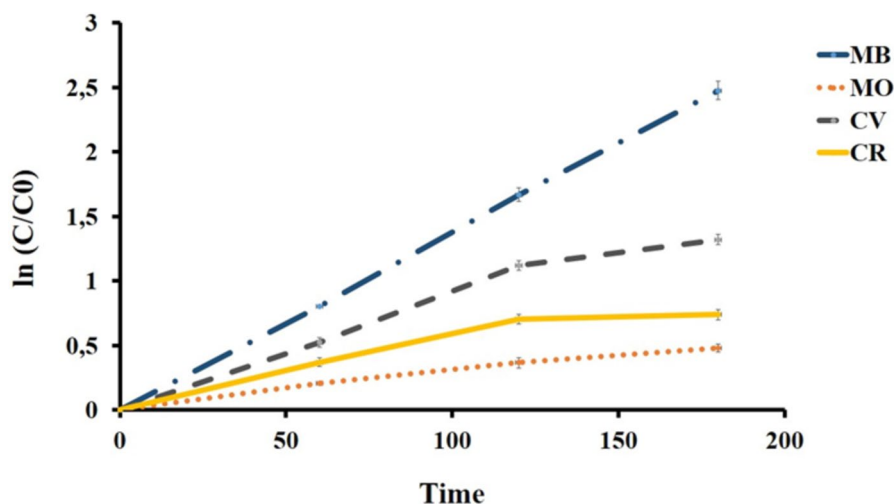
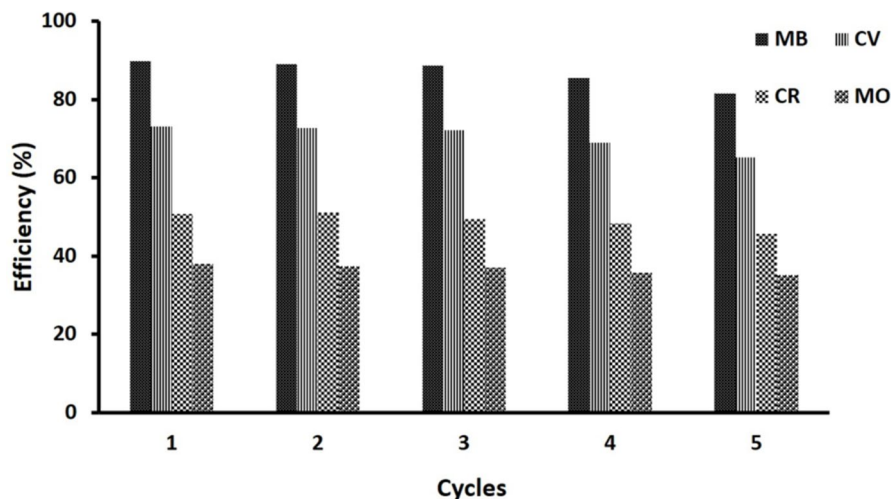


Fig. 9 The reusability test of FeNPs (B.S) against MB, MO, CV, and CR dyes



rate constants k . C_0 represents the initial dye concentration and C_t represents the measured dye concentration at a given time. C/C_0 —time plots showing the degradation rate versus time are given in Fig. 8; these plots help us to better understand the photocatalytic performance of QCLE-FeNPs.

The rate constants (k) for photocatalytic degradation kinetics were calculated as 0.0138, 0.0028, 0.008 and 0.0048 for MB, MO, CV, and CR, respectively. The k values demonstrate that silver nanoparticles display a markedly enhanced degradation efficiency towards methylene blue dye in comparison to other dyes. This indicates that the photocatalytic efficacy of silver nanoparticles may vary depending on the molecular structure of the dye and that the active sites on the nanoparticle surface interact more strongly with methylene blue molecules. In particular, the highest k value measured for methylene blue (0.0138 min^{-1}) proves that these nanoparticles offer high performance in the degradation of such pollutants. The C/C_0 ratio in the photocatalysis process indicates the concentration decrease of the dye solution with time and provides information about the kinetics of the photocatalytic activity. In our study, the data obtained fit a linear pseudo-first-order kinetic model. This result provides important information in terms of the reusability of silver nanoparticles in photocatalysis and indicates their potential to be used as an effective photocatalyst, especially for the removal of organic pollutants such as MB, CV, CR, and MO. In conclusion, this study demonstrated that biocompatible silver nanoparticles from kermes leaves offer

high efficiency in degradation of environmental pollutants and water treatment. The synthesized silver nanoparticles can be considered as a sustainable solution for the control of environmental pollution and offer an economical and environmentally friendly alternative for the photodegradation of industrial dyes. This study shows that environmentally friendly and biocompatible nanoparticle production strategies have a significant potential in environmental engineering applications.

3.9 Reusability of Fe-NPs

Reusability experiments were carried out at optimum parameters to determine whether the photocatalyst is photostable or not. The graph of the reusability experiments is given in Fig. 9.

As shown in Fig. 9, the photocatalyst showed a decrease in MB (%81.48), CV (%65.2), CR (%45.6), and MO (%35.1) activity after 5 cycles. This small decrease indicates that the stability and performance of the catalyst is good for practical catalytic decomposition of organic pollutants in wastewater treatment.

4 Conclusions

Nanotechnology involves the synthesis of nano-sized particles with diverse applications. Research has emphasized the role of nanotechnology in addressing challenges across medical science, food production, agriculture, textiles, and environmental conservation.

The significance of green nanotechnology is increasing as it eliminates harmful reagents and synthetic chemicals. In our study, we utilized the photo-synthesis method to produce Fe-NPs using *Q.coccifera* leaf extract, which proved to be a cost-effective, simple, and eco-friendly approach. This method eliminates the hazards associated with the use of harmful reducing and/or capping agents. Finally, QCLE-FeNPs can be used as an effective adsorbent for removing toxic textile dyes from wastewater with lower environmental impact. Additionally, the QCLE-FeNPs exhibited notable antioxidant activity compared to the standard Trolox, suggesting their potential for biomedical applications.

Author Contributions All stages of this study, including conceptualization, methodology, data analysis, and manuscript writing, were carried out by E.Kocazorbaz.

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Data Availability All the data generated or analysed during this are included in this article.

Code Availability Not applicable.

Declarations

Ethical Approval This manuscript does not contain any studies with human participants or animals performed by any authors.

Consent for Participate Not applicable.

Consent for Publication All authors gave their consent for publication.

Competing Interests The authors declare that there is no competing interest.

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