Original Article

Green synthesis of iron oxide nanoparticles using pomegranate seeds extract and photocatalytic activity evaluation for the degradation of textile dye

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\textbf{A B S T R A C T}

Iron oxide nanoparticles (Fe\textsubscript{3}O\textsubscript{4} NPs) were fabricated through green route using pomegranate (Punica granatum) seeds extract. The Fe\textsubscript{3}O\textsubscript{4} NPs were characterized by UV–vis, XRD, EDX, SEM and AFM techniques. The adopted green route furnished semi spherical Fe\textsubscript{3}O\textsubscript{4} NPs, uniformly distributed and particle size in the range of 25–55 nm. The LCMS/MS was performed for the identification of biomolecule present in the extract of pomegranate seeds and p-hydroxy benzoic acid, gallic acid, methyl gallate, catechin, kaempferol-3-O-sophoroside, 3-deoxyflavonoids, magnolol, ferulic acid, vanillic acid and pinocembrin along with other minor constituents were detected in the extracts using for Fe\textsubscript{3}O\textsubscript{4} NPs. The synthesized Fe\textsubscript{3}O\textsubscript{4} NPs showed excellent photocatalytic activity against reactive blue under UV light irradiation and maximum degradation of 95.08% was achieved with 56 min of reaction time. In view of promising activity, the Fe\textsubscript{3}O\textsubscript{4} NPs could be used photocatalyst for the degradation of dyes in wastewater and pomegranate seeds extract can be applied as eco-benign and cost effective approach for Fe\textsubscript{3}O\textsubscript{4} NPs synthesis.

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1. Introduction

Nano materials (1–100 nm) offers distinctive structural and physico-chemical properties as compared to bulk counterpart owing to the surface to volume ratio [1]. The most recent advancement in nanotechnology has led to the expansion in the synthesis of NPs by different chemical and physical methods. However, these methods have negative impact on the environment and living organisms since un-reacted chemicals are discharged in the environment. Therefore, there is need to fabricate the NPs using environmental benign techniques and NPs synthesized biogenically can be equally employed in electronics, biomedical field, material science and environmental remediation etc. [2–6]. In view of toxicity of nanoparticles, there is need to fabricate the NPs using bio-inspired agents. To date, the biosynthesis of NPs is regarded as environmentally friendly approach since no toxic agent is involved in bio-inspired approaches [4–14]. Plants are nature’s “chemical factories” and vast repertoires of secondary metabolites [15–18] that can be utilized as redox mediator and stabilizer for the NPs. It is reported that the NPs synthesized using plant products/extracts are more stable and the rate of synthesis is easy as compared to conventional techniques since green approaches are eco-benign, cost effective, simple, easy to perform and no toxic agent is involved [19–23]. To date, metal and metal oxide NPs have been prepared successfully using green route and as-prepared NPs have been applied in different field (Table 1).

In view of aforementioned facts, present study was focused on biogenic synthesis of Fe2O3 NPs using pomegranate seeds extract (PSE). The biomolecule in extracts was also identified using advanced techniques. The Fe2O3 NPs were prepared under mild conditions, characterized using UV–vis, XRD, SEM and AFM techniques and Fe2O3 NPs efficiency was studied by degrading the textile dye (reactive blue 4 dye, Fig. 1).

![Fig. 1 - Structure of reactive blue 4 dye used for PCA evaluation.](image)

Table 1 – Reports highlighting the biosynthesis of nanoparticles using plant extracts.

<table>
<thead>
<tr>
<th>S. no</th>
<th>Plant material</th>
<th>NPs</th>
<th>Properties and application</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>C. maxima peel extract</td>
<td>Iron</td>
<td>NPs with diameters of 10–100 nm</td>
<td>[24]</td>
</tr>
<tr>
<td>2</td>
<td>C. alata</td>
<td>Silver</td>
<td>Antibacterial activity against E. coli, good tensile properties</td>
<td>[25]</td>
</tr>
<tr>
<td>3</td>
<td>Lamiaceae plants (Mentha piperita, Melissa officinalis, and Salvia officinalis)</td>
<td>Gold</td>
<td>Size of the produced Au NPs was dependent on the aqueous plant extract</td>
<td>[26]</td>
</tr>
<tr>
<td>4</td>
<td>Camellia Sinensis</td>
<td>Nickel</td>
<td>Particle size in 43.87–48.76 nm range Promising photo-catalytic activity</td>
<td>[12]</td>
</tr>
<tr>
<td>5</td>
<td>Peumus boldus</td>
<td>Silver</td>
<td>Size 18 nm, spherical, stable and pure Anti-uroliithiatic activity</td>
<td>[27]</td>
</tr>
<tr>
<td>6</td>
<td>Phlogacanthus thyrsiformis Hardow (Malab) flower extract</td>
<td>Silver</td>
<td>Crystalline, monophasic Ag NPs, spherical, rod, flower and hexagonal shapes</td>
<td>[29]</td>
</tr>
<tr>
<td>7</td>
<td>T. involucrata, C. citronella, S. verbascifolium and T. ovata</td>
<td>Silver</td>
<td>Exhibited good tensile strength and thermal stability.</td>
<td>[30]</td>
</tr>
<tr>
<td>8</td>
<td>Terminalia catappa leaf extract</td>
<td>Copper</td>
<td>Exhibited good antibacterial activity against E.col.</td>
<td>[31]</td>
</tr>
<tr>
<td>9</td>
<td>H. fomes and S. apetala</td>
<td>Zinc oxide</td>
<td>Nano size, anti-inflammatory, antioxidant antibacterial, antidiabetic agents</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Rosa, Thymus, and Urtica dioica</td>
<td>Iron</td>
<td>Efficient in adsorption</td>
<td>[32]</td>
</tr>
<tr>
<td>11</td>
<td>M. indica, M. Koernigii, A. indica, M. champaca,</td>
<td>Iron</td>
<td>Efficient in adsorption</td>
<td>[33]</td>
</tr>
<tr>
<td>12</td>
<td>R. officinalis and E. globulus</td>
<td>Gold</td>
<td>Sizes range was 60.7 and 8.66 nm, respectively</td>
<td>[26]</td>
</tr>
<tr>
<td>13</td>
<td>A. scolopendrium</td>
<td>Silver</td>
<td>Antioxidant activity higher than extracts</td>
<td>[34]</td>
</tr>
<tr>
<td>14</td>
<td>A. satium, A. cepa and P. crispum</td>
<td>Zinc oxide</td>
<td>Size 14 and 70 nm, photo-active</td>
<td>[35]</td>
</tr>
<tr>
<td>15</td>
<td>Diospyros sylvatica</td>
<td>Silver</td>
<td>Crystallite size 10 nm and antimicrobial agent</td>
<td>[36]</td>
</tr>
<tr>
<td>16</td>
<td>Taxus baccata</td>
<td>Silver</td>
<td>Anticancer agent, non-toxic agent</td>
<td>[37]</td>
</tr>
<tr>
<td>17</td>
<td>Green tea extracts</td>
<td>Iron</td>
<td>Photo-active</td>
<td>[38]</td>
</tr>
<tr>
<td>18</td>
<td>Pelargonium endlicherianum</td>
<td>Silver</td>
<td>Antibacterial agent</td>
<td>[39]</td>
</tr>
<tr>
<td>19</td>
<td>Notoptyopus nimmoniana</td>
<td>Silver</td>
<td>Size range 44–64 nm, antioxidant, anticancer, antimicrobial agent</td>
<td>[40]</td>
</tr>
<tr>
<td>20</td>
<td>Pomegranate (P. granatum)</td>
<td>Fe2O3</td>
<td>Semi-spherical, nano-range, 95.08% dye degradation</td>
<td>Present study</td>
</tr>
</tbody>
</table>
2. Material and methods

2.1. Chemical, reagents and sample collection

The PSE fruits were collected Bahawalpur, Pakistan (local market). The seeds were separated from fruits and used for extract preparation. Iron chloride (99.99%) was obtained from chemical supplier (Sigma-Aldrich). Ultrapure water (18.2 MΩ cm) was used for extraction and solution preparation purposes.

2.2. Preparation of seed extracts of Punica granatum

The PSE were washed with ultrapure water and then, 20 g seeds were ground in electrical grinder and mixed with 250 mL of water and shaken for 2 h. The extract was filtered and filtrate was stored at 4 °C and used for the preparation of Fe₂O₃ NPs.

2.3. Iron oxide NPs synthesis procedure

Both the PSE and iron chloride solution (1 M) were mixed in 1:2 ratio and the mixture was heated at 70 °C for 15 min along with continuous stirring on magnetic stirrer till the pale yellow color changed to brownish black [41]. Then, it was centrifuged at 15,000 rpm for 10 min and precipitates were collected, which was washed with water and ethanol (3–4 times) and powder thus obtained was dried for 3 h in furnace at 60 °C and used for further analysis and PCA evaluation.

2.4. Characterization

The UV–vis absorbance spectroscopy was performed using double beam spectrophotometer (CE Cecil 7200, UK). X-ray diffraction (XRD) analysis were recorded from 20° to 65° with a diffractometer (Bruker, German) using Cu Kα radiation with an accelerating voltage of 40 KV at scanning rate of 1°/min. The element analysis of Fe₂O₃ NPs was carried out using EDX analysis. The SEM was performed with a Hitachi SX-650 (Tokyo, Japan) at 20 KV of accelerating voltage. Moreover, the confirmation of the particle size and morphology was also carried out by atomic force microscopy (AFM).

2.5. Photocatalytic activity

The PCA of fabricated NPs was evaluated by degrading reactive blue 4 dye. In a typical procedure, 15 mg of Fe₂O₃ NPs was added in 100 mL of 20 mg/L of reactive blue 4 dye (Fig. 1) and the suspension was placed in dark with slow stirring for 30 min to achieve the adsorption–desorption on the surface of cata-

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Fig. 2 – Seeds and fruits of P. granatum used for the synthesis of Fe₂O₃ NPs.

Fig. 3 – UV–vis spectrum of Fe₂O₃ NPs synthesized using P. granatum seed extract.

Fig. 4 – SEM image of Fe₂O₃ NPs synthesized using P. granatum seed extract.
Fig. 5 – Energy dispersive X-ray (EDX) spectrum of Fe$_2$O$_3$ NPs synthesized using _P. granatum_ seed extract.

Fig. 6 – X-ray diffraction (XRD) pattern of Fe$_2$O$_3$ NPs synthesized using _P. granatum_ seed extract.

lys. Then, mixture was irradiated to UV light (400 W high pressure mercury lamps). After specific time interval, sample was withdrawn (2 mL), absorbance was measured at 595 nm and percent degradation was measured as shown in Eq. (1).

Where, $C_0$ and $C_f$ are the absorbance values before and after UV irradiation.

\[
\text{Degradation (\%)} = \frac{C_0 - C_f}{C_0} \times 100
\]  

(1)

2.6. **HPLC analysis for degradation monitoring**

RP-HPLC analysis was carried out to investigate the transformation of reactive blue 4 dye into by-products. For this purpose, control and treated samples were vortexed for 2 min and 15 uL of the sample was injected into ZORBAX SB C-column after filtration through 0.5 um syringe filter and eluted using 40% hexane at 0.5 mL/min (flow rate). The response was recorded at 550 and 600 nm and 200–900 nm was scanned in a diode array detector, whereas LCMS/MS was performed precisely as reported elsewhere [42].

3. **Results and discussion**

3.1. **Characterization**

The Fe$_2$O$_3$ NPs was prepared via green route using _P. granatum_ seeds (Fig. 2) extracts at room temperature. The absorption spectrum of Fe$_2$O$_3$ NPs is shown in Fig. 3 and the peak observed at 371.71 nm belong to Fe$_2$O$_3$ NPs, and is in line with already reported [43]. The morphology of Fe$_2$O$_3$ NPs was monitored by SEM analysis and the particle size was found in the range of 25–55 nm, the particles were of variable shapes and in agglomerated form (Fig. 4). The agglomeration of agglomerated might be due to presence of biological compounds on the surface of particles. Due to H-bonding present in bioactive molecules, the particles appeared to be in the form of aggregates [12–14]. The EDX spectrum showed the presence of iron and oxygen (Fig. 5). The EDX analysis revealed the presence of 58.5% of iron and 17% oxygen along small amount of carbon, which was due to phenolic compounds in the extract of _P. granatum_. X-ray diffraction (XRD) is an important tool to study the crystal formation and to estimate the crystalline size of the fabricated NPs. The peaks observed in XRD analysis revealed the spinal structured magnetite and exhibiting peaks at 2 theta value of 30.60, 35.21, 42.13, 53.53, 57.6 and 63.01, which corresponds to diffraction planes of 220, 311, 400, 422, 511 and 440, respec-

Fig. 7 – Atomic force microscopy of Fe$_2$O$_3$ NPs synthesized using _P. granatum_ seed extract.
Table 2 - Identification of phytochemicals in pomegranate (P. granatum) seed extract used for iron oxide NPs synthesis.

<table>
<thead>
<tr>
<th>S. no</th>
<th>Molar Mass</th>
<th>m/z M (+/-)</th>
<th>MS/MS ions m/z (relative intensity)</th>
<th>Rt (min)</th>
<th>Compounds</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>138</td>
<td>139</td>
<td>122, 121, 81, 69</td>
<td>0.75</td>
<td>p-Hydroxy benzoic acid</td>
</tr>
<tr>
<td>2</td>
<td>170</td>
<td>169(−)</td>
<td>165, 150, 140, 100, 57</td>
<td>2.17</td>
<td>Gallic acid</td>
</tr>
<tr>
<td>3</td>
<td>184</td>
<td>183(−)</td>
<td>168, 139, 124</td>
<td>0.67</td>
<td>Methyl gallate</td>
</tr>
<tr>
<td>4</td>
<td>289</td>
<td>290</td>
<td>272, 260, 242, 124, 93</td>
<td>6.92</td>
<td>Catechin</td>
</tr>
<tr>
<td>5</td>
<td>302</td>
<td>301(−)</td>
<td>301, 257, 229, 185</td>
<td>8.67</td>
<td>Ellagic acid</td>
</tr>
<tr>
<td>6</td>
<td>184</td>
<td>183(−)</td>
<td>168, 139, 124</td>
<td>0.67</td>
<td>Methyl gallate</td>
</tr>
<tr>
<td>7</td>
<td>610</td>
<td>609(−)</td>
<td>67, 593, 565, 551, 489, 476</td>
<td>2.72</td>
<td>Kaempferol-3-O-sophoroside</td>
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<tr>
<td>8</td>
<td>226</td>
<td>225(−)</td>
<td>207, 189, 171, 159, 141, 129</td>
<td>1.12</td>
<td>3-Deoxyflavonoids</td>
</tr>
<tr>
<td>9</td>
<td>198</td>
<td>199</td>
<td>190, 185, 171, 161, 157, 143, 124, 103, 81, 63</td>
<td>0.89</td>
<td>Syringic acid</td>
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<tr>
<td>10</td>
<td>265</td>
<td>266</td>
<td>251, 225, 171, 155</td>
<td>0.36</td>
<td>Magnolol</td>
</tr>
<tr>
<td>11</td>
<td>270</td>
<td>269(−)</td>
<td>246, 110, 93, 91</td>
<td>1.95</td>
<td>Apigenin</td>
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<tr>
<td>12</td>
<td>284</td>
<td>283(−)</td>
<td>242, 124</td>
<td>2.79</td>
<td>Retusin (isoavone)</td>
</tr>
<tr>
<td>13</td>
<td>454</td>
<td>455</td>
<td>437, 418, 381, 293, 275</td>
<td>2.13</td>
<td>Flavogallonic acid dilactone</td>
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<tr>
<td>14</td>
<td>193</td>
<td>192(−)</td>
<td>175, 164, 120, 108</td>
<td>0.58</td>
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<tr>
<td>15</td>
<td>391</td>
<td>390(−)</td>
<td>217, 191, 373</td>
<td>1</td>
<td>Citric acid derivative</td>
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<tr>
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<td>191</td>
<td>190(−)</td>
<td>111, 173</td>
<td>1.14</td>
<td>Citric acid</td>
</tr>
<tr>
<td>17</td>
<td>255</td>
<td>254(−)</td>
<td>213, 211, 151</td>
<td>11.98</td>
<td>Pinocembrin</td>
</tr>
<tr>
<td>18</td>
<td>168</td>
<td>169</td>
<td>151, 137, 111, 95, 82</td>
<td>8.77</td>
<td>Vanillic acid</td>
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<tr>
<td>19</td>
<td>273</td>
<td>272</td>
<td>167</td>
<td>10.55</td>
<td>Phloretin</td>
</tr>
<tr>
<td>20</td>
<td>433</td>
<td>432</td>
<td>301</td>
<td>7.93</td>
<td>Ellagic acid-pentoside</td>
</tr>
<tr>
<td>21</td>
<td>447</td>
<td>446</td>
<td>300</td>
<td>8.21</td>
<td>Ellagic acid deoxyhexoside</td>
</tr>
<tr>
<td>22</td>
<td>463</td>
<td>462</td>
<td>301</td>
<td>7.35</td>
<td>Ellagic acid-hexoside</td>
</tr>
</tbody>
</table>

Fig. 8 – LC–MS analysis of P. granatum seed extract used for Fe$_2$O$_3$ NPs synthesis.

Fig. 9 – Representative chromatogram of methyl gallate of P. granatum seed extract analyzed by ESI-MS/MS.

Moreover, AFM analysis was also performed to evaluate the nature of Fe$_2$O$_3$ NPs and response is shown in Fig. 7. The morphology of particles can be accessed from the grooves present in the dimensional views of NPs. From the three dimensional view, it is clearly observed that the size of the particle was variables and maximum distribution was in the range of 28.4–66.2 nm. This behavior of NPs was due to presence (coating) of different bioactive molecules [46–51], since

tively (Fig. 6). The formation of Fe$_2$O$_3$ NPs index with JCPDS card no: 82-1533 [44]. Analysis also revealed that the fabricated NPs were crystalline [45]. The average grain size was 48 nm, which was calculated using relation shown in Eq. (2). Where, $\lambda$ is the wavelength of the X-rays (0.1541 Å), 0.90 is a constant value known as shape factor, $\beta$ is the FWHM and $\theta$ is the angle.

$$D = 0.9\lambda /\beta \cos \theta$$  \hspace{1cm} (2)
bioactive molecules have different functional groups, which interact with each other by intermolecular forces, particle may hold together and particle size may vary. The existence of NPs in aggregates is an indication of strong intermolecular forces, especially H-bonding between hydroxyl groups and other moieties in the structure of phenolic compounds [4–6,12–14,50,52–54].

3.2. Identification of bioactive constituents

The LC/MS/MS is ideal for the identification of bioactive constituents in seed extracts [55,56]. LC-ESI-MS/MS analysis is widely used for identification of bioactive molecules in plant extracts [57]. The molecules containing free carboxyl groups, yields [M−H]− ion that corresponds to the carboxylate anion [58]. So far, LC-ESI-MS/MS analysis was used for the identification of phenolic compounds in extracts [59]. The constituents identified in the pomegranate (P. granatum) seeds extract are shown in Table 2, chromatograms are shown in Figs. 8 and 9 and the structures of representative compounds are shown in Fig. 10. The identified components were alkaloids, flavonoids and polyphenols in the seeds extract of P. granatum. The antioxidant activity of NPs was reported to be due the phenolic compounds that are used as capping and stabilizing agents. Phenolic compounds present in the extract contain hydroxyl and carboxylic groups which have very high tendency to bind heavy metals [53,60]. Metal ions in the salt solution interact with phenolic compound and due to π track conjugation (ester oxygen atom and ortho-phenolic hydroxyl group) and this esterification results in the loss of hydrogen from ortho-phenolic hydroxyl group [14] and a structure (semi-quinone) is produced due to H loss. \( H^+ \) radical is formed due to the electron loosening form bioactive molecule (i.e., ellagic acid). Metal ions are to reduce to nano size during this process [12]. Also, bioactive molecule have excellent antioxidant property, which also furnished NPs by scavenging reactions along with the formation of \( H^+ \) specie and resultantlly, the ions in to solution are converted into stable atom [43].

3.3. Photocatalytic activity

The PCA of Fe\(_2\)O\(_3\) NPs was evaluated by degrading the reactive blue 4 dye under UV light irradiation since photocatalysts are efficient for the degradation of recalcitrant and toxic agents [61–67]. Moreover, the photocatalytic treatment convert the pollutants completely in to harmless end products like carbon dioxide and water [68]. The dye degradation response of Fe\(_2\)O\(_3\) NPs is shown in Fig. 11. It can be observed that the dye was degraded efficiently due to excellent activity of Fe\(_2\)O\(_3\) NPs. The dye absorption peak was decreased rapidly as a function of UV irradiation time, which was due to breakdown of the chromophoric group in the dye and dye was degraded up to 95.08\% in 56 min of reaction time. The dye degradation mechanism UV light irradiation in the presence of Fe\(_2\)O\(_3\) NPs is elaborated in Fig. 12. When Fe\(_2\)O\(_3\) NPs was irradiated, an electron (\( e^- \)) and hole (\( h^+ \)) pair is produced and electron is excited from valence band to the conduction band, leaving the \( h^+ \) in the VB. This hole (\( h^+ \)) is actually responsible for the conversion of water into hydroxyl radical, which is responsible for oxidative degradation of dye. On the other hand, free electron combine with molecular oxygen and converted into superoxide radical. The superoxide radical is also converted in to hydroxyl radical. The hydroxyl radical is a strong oxidizing agent and degrades the organic species non-selectively in to harmless end products [69]. Reversed-phase chromatog-
Fig. 11 – UV–vis absorption spectra of reactive blue 4 dye (0–56 min of UV irradiation in the presence of Fe$_2$O$_3$).

Fig. 12 – Proposed dye degradation mechanism by Fe$_2$O$_3$ NPs under UV irradiation.

Fig. 13 – HPLC chromatograms of treated (using Fe$_2$O$_3$ NPs as catalyst under UV irradiation) and untreated reactive blue 4 dye.

HPLC (RP-HPLC) separates molecules on the basis of variation in their hydrophobicity. In order to investigate the degradation end products, the RP-HPLC analysis was performed before and after irradiation of dye and response thus obtained is shown in Fig. 13. The untreated dye shows a single peak at retention time of 1.86 min. A dye solution treated for 3 min showed peak of less intensity and after 56 min of irradiation, the peak at 1.86 min disappeared and peaks were appeared at 1.06, 2.02, 2.46, 3.61, 4.29, 5.37, 6.92, 7.36 and 8.44 min, which indicates that reactive blue 4 dye was complete degraded in to low molecular weight components and these findings are in line with previous studies [68,70] that after degradation the organic species are converted in to harmless end products [71–74]. Also, under the current scenario of environmental pollution [63,64,75–78], there is a need to adopt efficient approaches for the remediation of toxic pollutants and photocatalytic treatment using Fe$_2$O$_3$ NPs is proved to be highly efficient, which could possibly be used for the treatment of textile wastewater contains dyes.
4. Conclusions

The Fe$_2$O$_3$ NPs were successfully fabricated via green route using pomegranate (P. granatum) seeds extract, which were confirmed by UV–vis, XRD, EDX SEM and AFM techniques. The adopted green route furnished Fe$_2$O$_3$ NPs in nano-range size, semi-spherical shape and in agglomerated form. The P. granatum seeds extract analysis revealed a variety of bioactive components that act as a capping and stabilizing agents. The main constituents were p-hydroxy benzoic acid, gallic acid, methyl gallate, catechin, kaempferol-3-O-sophoroside, 3-deoxyflavonoids, magnolol, ferulic acid, vanillic acid and pinocembrin. The Fe$_2$O$_3$ NPs showed promising photocatalytic activity for reactive blue 4 dye degradation under UV light irradiation and up to 95.08% dye degradation was achieved with 56 min of UV irradiation. Results revealed that P. granatum seeds extract is potential biomolecules that can be employed for Fe$_2$O$_3$ NPs synthesis since it is one of green, cost effective and eco-benign methods and Fe$_2$O$_3$ NPs could be used for dye degradation in wastewater. Future studies can be focused on the bioactivity profiling (antioxidant and antimicrobial activities) of Fe$_2$O$_3$ NPs prepared via green route.

Conflict of interest

The authors declare no conflicts of interest.

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