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TiO_2 Nanoparticles Green Synthesis Utilising *Cynara scolymus* for Efficient removal of Pb (II) ions

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Abstract

Titanium dioxide nanoparticles were synthesized using Cynara scolymus seed extract as a reducing and stabilizing agent. TiO_2 NPs were fully characterized by FT-IR, SEM, and TEM techniques to confirm the formation of stable nanoparticles with a spherical morphology and sizes ranging from 10 to 30 nm. TiO_2 NPs demonstrated considerable and instantaneous efficacy to precipitate lead ions (Pb^{2+}) from aqueous solutions without pH or contact time adjustments. The green synthesis approach offers a sustainable and cost-effective alternative to traditional methods. Also, suggesting a potential application of synthesized nanoparticles for environmental remediation.

Keywords: Titanium dioxide nanoparticles, green synthesis, lead removal, and ultrasound-assisted extraction.

1. Introduction

Titanium dioxide nanoparticles (TiO_2 NPs) received a considerable attention recently for their excellent properties, and wide applications [1]. Titanium dioxide plays a significant role in water purification through various mechanisms, primarily photocatalysis, and membrane technology [2]. Besides, effective degradation of organic pollutants and enhancement of filtration systems to remove water contaminations [3]. Titanium dioxide NPs other application fields involved photocatalyst [4],

pigment [5] and biomedical applications [6]. Indeed, TiO_2, especially in the form of nanoscale (nm) (particle size <100 nm), with superior optical and electrical properties and photocatalytic activity enabling degradation of harmful pollutants [7] is certainly one of them. In addition, TiO_2 NPs have high oxidizing power [8], nontoxic [9], soluble and chemical stability [10], which permit their excellent potential for environmental remediation and other industrial processes [11]. Several methods are available to synthesize titanium dioxide nanoparticles, but most of the conventional chemical and physical methods involve the use of toxic chemicals, high energy consumption, and the production of toxic by-products [12]. However, the green synthesis of TiO_2 nanoparticles is more sustainable. The green synthesis methods involve the use of plant extracts or microorganisms as reducing agents, which results in environmentally friendly, cost-effective and biocompatible production processes [13].

A few plant extracts have been used for the reduction of metal ions and the stabilization of nanoparticles, especially due to their high content of bioactive compounds like terpenoids, flavonoids and polyphenols [14]. These biomolecules have been found to be very important in the biosynthetic process as they not only reduce the metal salts into metal nanoparticles but also prevent aggregation of the nanoparticles [15]. In the case of plant sources, artichoke (Cynara caruncuIous) is a source of bioactive components that are responsible for its reducing and stabilizing properties. These include polyphenols, flavonoids [16] and inulin [17].

The antioxidant and reducing property of the artichoke seed extract is very strong and especially the extract of artichoke seeds is a promising candidate for the green synthesis of TiO_2 nanoparticles [18]. The artichoke seed extract mediated synthesis of TiO_2 nanoparticles is a cost-effective, eco-friendly, and sustainable method. The simplicity, scalability and environmental friendliness of this strategy have made it an ever more popular approach for the synthesis of nanomaterials because it is quickly growing in the field [19].

One of the most promising applications of green synthesized TiO_2 nanoparticles is the removal of heavy metals from contaminated water among these applications. TiO2 nanoparticles have proven to have good photocatalytic activity, allowing for the degradation of a large variety of pollutants by adsorption and oxidation processes [20]. The potential of TiO_2 nanoparticles for water purification is further bolstered by their strong antimicrobial properties, making them an effective solution for water pollution and heavy metal toxicity issues. Overall, the applications of TiO_2 nanoparticles in water purification systems are promising and hold significant potential for improving water quality and ensuring sustainable development.

The lead (Pb) is an environmental persistent and highly toxic contaminant, especially in aquatic systems. It can dissolve in water bodies and then be uptaken by living organisms through the food chain which causes bioaccumulation and biomagnification [23]. The main industrial sources of lead pollution are mining, electroplating and smelting operations, all of which are significant sources of lead contamination and associated health hazards [24]. Lead exposure has been associated with serious health consequences, such as damage to the nervous system, and delayed physical growth and neuronal development in children [25]. Chronic exposure has also been associated with renal dysfunction and hypertension [26].

WHO and the EU have recommended guidelines for lead in drinking water of 5 micrograms per litre (µg/L) or 0.005 parts per million (ppm) [27]. Advanced treatment methods include photocatalysis with TiO_2 nanoparticles, which is a promising and sustainable process for treating water contaminated with lead [28]. Dithizone (1,5-diphenylthiocarbazone) plays a crucial role in the detection of lead in biological and environmental samples using various analytical techniques. Dithizone is a colorimetric reagent that forms a stable, intensely coloured complex with the lead ion, Pb^{2+} (Scheme 1) which gives the organic solvent phase a characteristic colour. This color change allows for qualitative and quantitative measurement of a lead concentration.

The ability of dithizone to chelate increases the sensitivity and selectivity of the method and it is a useful reagent for spectrophotometric and electrochemical methods used to detect trace levels of lead [29]. The aim of this study is to assess the capability of the green synthesized TiO_2 nanoparticles, obtained from the extract of the Cynara scholium's seed, to remove the Pb^{2+} ions from aqueous solution. It also explains the synthesis route, considers the adsorption and photocatalytic mechanisms, and the possibility extended to the environment and large-scale industrial uses.

2. Experimental

2. 1 Chemicals and Materials

All chemicals and reagents used in this study were of analytical grade and were used as received without further purification. Titanium butoxide was purchased from Sigma–Aldrich (Germany). Dithizone, ethanol, and lead (II) nitrate was obtained from HDL Company. Double-distilled water was used throughout the experiments.

2. 2 Instruments

UV–Vis spectra and all absorbance measurements were carried out using a double-beam UV–Vis spectrophotometer (PG80+, UK), operating in the wavelength range of 190–1000 nm. Fourier-transform infrared (FT–IR) spectra were recorded using a PerkinElmer Spectrum Two FT-IR Spectrometer (USA). Field emission scanning electron microscopy (FESEM) analysis was performed using a TESCAN MIRA3 (Czech Republic), while transmission electron microscopy (TEM) images were obtained using a Philips CM120 (Netherlands). An ultrasonic homogenizer (SCIENTZ, China) was employed for ultrasonic-assisted extraction of the plant material and for sample dispersion.

2. 3 Preparation of *Cynara scholium's* seeds extract

Artichoke seeds (Cynara scolymus) were purchased from local markets in Kut City, Iraq. The seeds were thoroughly washed with deionized water to remove surface dust and impurities and then air-dried at ambient temperature for approximately seven days under dust-free conditions. Once completely dried, the seeds were ground into a fine powder. For the extraction process, 5 g of the seed powder was mixed with 100 mL of a 50:50 (v/v) ethanol–water solution.

Ultrasound-assisted extraction (UAE) was employed to extract polyphenolic compounds from the seed matrix. The extraction was performed using a probe-type ultrasonic processor operating at 20 kHz and 400 W for 5 min. In order to prevent thermal degradation of bioactive compounds the sample was kept in ice bath during the sonication process and the temperature was maintained below 30 °C. This gave rise to the extract which was separated by centrifugation and solid particles were removed using Whatman No. 1 filter paper. The filtrate was kept at 4 °C for subsequent use or it was directly used for the synthesis of TiO2 nanoparticles.

2. 4 Synthesis of TiO_2 NPs

8.5 mL of titanium butoxide was dissolved in methanol and then diluted to 25 mL to make a 1 M stock solution. The artichoke seed extract was dropwise added to the 1 M solution of titanium butoxide in Erlenmeyer flask with continuous stirring to synthesize TiO_2 nanoparticles. After adding about 2.5 mL of the extract, the solution slowly began to turn white and then a white precipitate started to appear, which revealed the creation of nanoparticles of TiO_2.

The reaction mixture was stirred continuously for 30 min with a magnetic stirrer followed by overnight reaction to complete the reduction and formation of nanoparticles. The precipitated nanostructured material was centrifuged for 24 hours at 4000 rpm. The centrifugation and washing were repeated three times with deionized water and were then repeated with methanol to get rid of any unreacted extract. The material obtained was then dried to produce the TiO_2 nano powder that was then analyzed further by UV–Vis, FT–IR, FE-SEM, and TEM.

2.5 Determination of Lead Removal Using Dithizone as a Colorimetric Reagent

The dithizone method was used as a colorimetric reagent to measure the efficiency of removing the Pb^{2+} ions onto the TiO_2 nanoparticles. Dithizone solution (1 × 10-4 M) was prepared by dissolving dithizone in ethanol (organic solvent). Samples were collected prior to and following treatment and the volume of each sample was the same amount that was added to the dithizone solution to determine the absorbance peak in the UV–Vis spectrum of the sample after treatment, which would be the greatest for the sample containing the lowest concentration of lead. When the ions of lead (Pb2+) were present, the complex of dithizone was formed, which characteristically is red violet, and thus the presence of lead in the samples was confirmed.

Measurements of absorbance were performed on a UV-Vis spectrophotometer at the characteristic wavelength of 505 nm. The removal efficiency has been determined by the absorbance reduction before and after treatment using TiO2 nanoparticles.

3. Results and discussion

3.1 Fourier Transform Infrared Spectroscopy (FT-IR)

Fourier transform infrared (FT–IR) spectroscopy is an extremely powerful analytical tool utilized to elucidate the functional groups of a sample, by virtue of the characteristic vibrational modes of molecular bonds absorbing infrared radiation at their characteristic wavenumbers. FT–IR analysis was performed in this study to characterize both the artichoke (Cynara scolymus) seed extract and the synthesized nanoparticles of TiO_2, aiming to identify the main functional groups and elucidate the chemical structure of the two samples. FT–IR spectrum of the Cynara scolymus seed extract (Figure 1) showed a broad and intense band in the range of 3300–3400 cm^{-1}, which is associated with the hydroxyl O–H stretching vibrations.

This is a sign of the presence of hydroxyl-containing compounds like phenols, alcohols and carbohydrates, which are usually found in extracts derived from plants. The other peaks at ~2920 and ~2850 cm^{-1} are assigned to the asymmetric and symmetric stretching modes of $–CH_2$ and –

CH_3, respectively, and can be associated with the presence of aliphatic chains as commonly found in lipids, waxes and organic acids. Aromatic and carbonyl containing compounds can be detected from a moderate band in the region 1600–1650 cm^{-1}, which can be assigned to C=C stretching vibrations of aromatic rings or to C=O stretching of conjugated carbonyl groups. Moreover, the fingerprint region (400–1500 cm−1) showed a complex fingerprint pattern that included multiple peaks corresponding to bending vibration and C–O stretching modes, which is indicative of the structural diversity of the sugars, fibers and polyphenolic compounds that make up the extract.

The FT–IR was used to examine the nanoparticles of TiO_2 (Figure 2), and the resulting spectrum showed a strong and broad absorption band within the lower wavenumber region (400–800 cm^{-1}) which is representative of Ti–O–Ti stretching vibrations of the crystalline TiO_2 lattice, thus confirming successful formation of the nanoparticles. The presence of a wide band around 3400 cm-1, which is attributed to stretching vibrations of surface hydroxyl groups (Ti–OH) and adsorbed water molecules, suggests that the nanoparticles have a high surface area and are hydrophilic in nature.

A distinct band around 1630 cm^{-1} is attributed to the bending mode of molecular water (H–O–H) adsorbed on the surface. The lack of or weak bands corresponding to organic functional groups implies that the inorganic nanoparticles are highly pure, indicating that the organic species originating from the synthesis process were removed, probably by thermal treatment. The spectroscopic results unambiguously indicate that the plant extract is different from the metal oxide nanoparticles, thus giving some insight into the compositional differences between the two, and their respective chemical functionalities and surface characteristics.

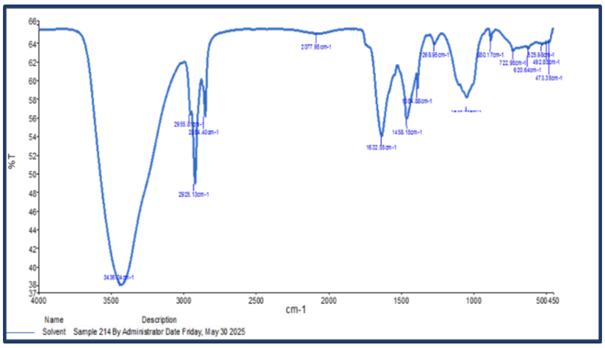


Figure 1: FT-IR of *Cynara scolymus* seeds extract.

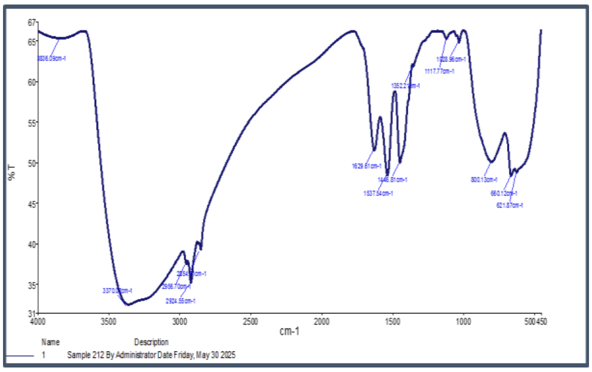


Figure 2: FT-IR spectrum of titanium dioxide nanoparticles.

3. 2 FE-SEM

The morphology of particles is irregular or quasi-spherical shape and rough with porous surface. The particle size is estimated to be in nanoscale range (average size: 31.46 nm) based on applied scale (500 nm) leading to high reactivity and application in heavy metal removal, photocatalysis, etc. The roughened surface observed is believed to facilitate the adsorption process by increasing the available active sites for both chemical and physical reactions and thus provide better performance in environmental applications. Figure 3 and 4 are FE-SEM images taken at an accelerating voltage of 15.0 kV which allowed the fine structural features and surface morphology to be observed in detail.

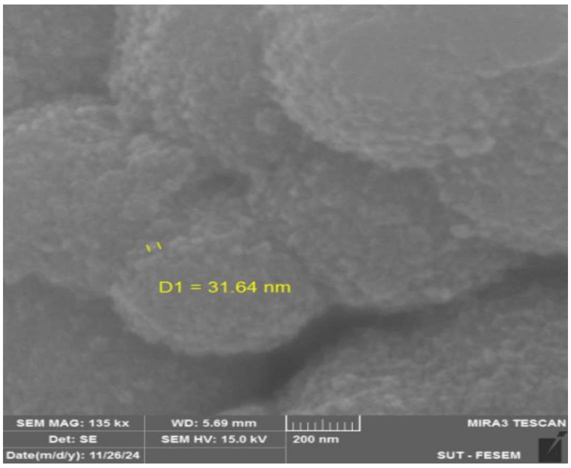


Figure 3: FE-SEM micrographs of TiO_2 nanoparticles at 200 nm.

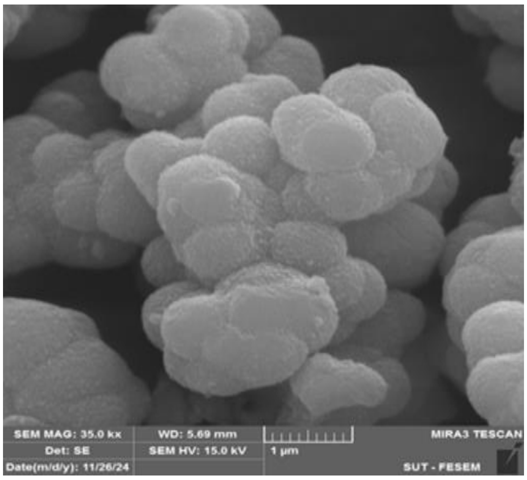


Figure 4: FE-SEM micrographs of TiO_2 nanoparticles at 1 μm.

3. 3 Transmission Electron Microscopy (TEM)

The TEM image (Figure 5) shows that the morphology of the biosynthesized titanium dioxide nanoparticles is mostly spherical with a size of 10-30 nm. The TEM analysis further indicated that most of the TiO_2 nanoparticles tend to cluster together in spherical shapes as a result of the forces of natural aggregation including van der Waals attractions and surface chemistry related interactions (figure 5). This behaviour is often seen in nanoscale materials as they have a high surface area to volume ratio and are small.

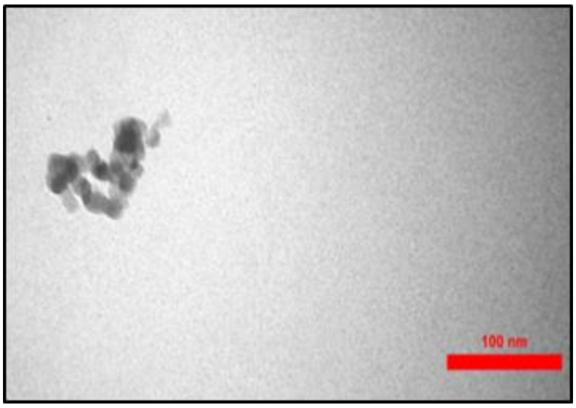


Figure 5: TEM image of the biosynthesized TiO_2 NPs.

3. 4 The Use of Titanium Dioxide Nanoparticles for the Removal of Lead Ions from Aqueous Solutions

Titanium dioxide nanoparticles (TiO_2 NPs) have large surface area and high catalytic activity which make them great candidates for water purification. They have been shown to be effective for the removal of both heavy metal pollutants, such as Pb^{2+} ions, from polluted water in several studies. These nanoparticles interact with the environment through several mechanisms such as surface adsorption, ion exchange and, for some, photocatalytic reactions when exposed to ultraviolet (UV) radiation.

Some parameters including contact time, initial ion concentration, pH, and particle size have effect on the efficiency of TiO_2 nanoparticles. TiO_2 nanoparticles are a cost-effective and environmentally friendly treatment alternative to conventional treatment methods, and they are promising to remediate lead-contaminated water, particularly in developing countries.

3. 5 Estimation of Lead Ion Removal Efficiency by TiO_2 Nanoparticles via Dithizone Spectrophotometry

Determination the final concentration of lead ions after treatment of an aqueous solution of known initial concentration using titanium dioxide nanoparticles, a calibration curve was first constructed using standard lead ion solutions with concentrations of (5, 10, 15, 20, 25, 30, 35, 40, 45, and 50) ppm. One of the concentrations within this range was then selected and treated with the TiO_2 nanoparticles under constant stirring for five minutes. After treatment, the mixture was filtered, and the filtrate was reacted with the organic reagent dithizone (1x 10^{-4} M.

The absorbance was measured spectrophotometrically scheme (1), and using the calibration curve, the residual lead concentration was calculated. This value was then compared with the initial concentration before treatment. The removal efficiency and adsorption capacity were calculated using the following mathematical equations [30].

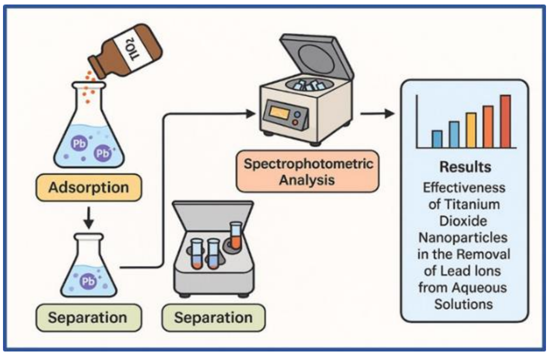


Figure 6: Estimation of Lead Ion Removal Efficiency by TiO_2 Nanoparticles via Dithizone Spectrophotometry.

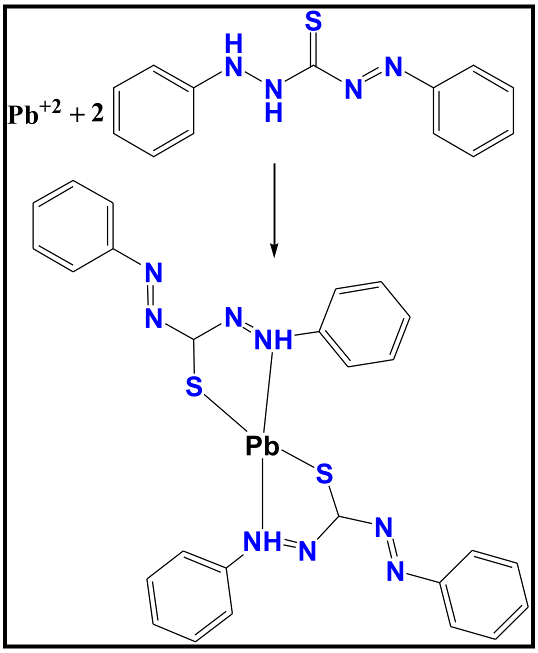
The removal efficiency of the Pb^{2+} ions by titanium dioxide (TiO_2) nanoparticles was estimated by measuring the initial (before treatment) and final (after treatment) concentrations of lead in the solution. The removal efficiency (R%) was calculated with the following equation.

$$\%\text{Removal} = \frac{C_i - C_f}{C_i} x100 \quad \text{where}$$

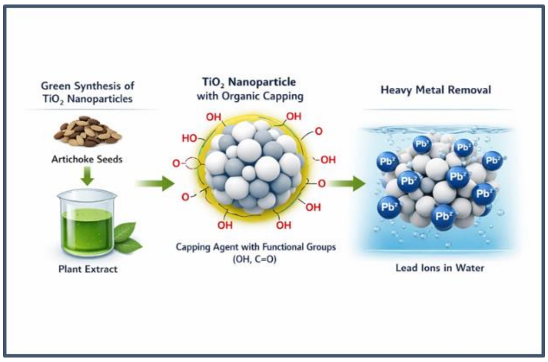
The initial concentration of the ions of Pb^{2+} (mg/L) is denoted as Ci and the concentration after treatment is denoted by Cf. This equation gives a clear indication on the efficiency of removal of lead by the nanoparticles via adsorption or removal mechanism. All the tests were repeated three times to ensure the reliability and reproducibility of the results and the average value was reported. Similarly, the adsorption capacity (q, mg/g) of TiO_2 nanoparticles was also determined by the following equation.

$$q = \frac{(C_i - C_f) \cdot V}{W}$$

Ci: initial concentration (ppm), C_f: final concentration (ppm), V: Volume of solution (L), W: Weight of the adsorbent (g).



Scheme 1: Complexation reaction of Pb^{2+} ion with dithizone forming a Pb–dithizone chelate.



Scheme 2: Schematic illustration of green synthesis, organic capping, and heavy metal adsorption by TiO_2 nanoparticles.

The optimization of green synthesis of TiO_2 nanoparticles using different plant extracts and reaction condition to increase the properties of nanoparticles and the efficiency of lead removal will be the main task for future work. Further research is needed on the long-term stability and reusability of the synthesized TiO_2 nanoparticles for water treatment applications.

Furthermore, it is necessary to take measures for protecting the environment by determining the toxicity of the nanoparticles and any related by-products. Finally, the applicability of this sustainable approach to environmental remediation would be expanded by adapting it to the removal of other heavy metals and emerging pollutants found in water would be extended.

Tabel 1: Efficiency of Lead Removal Using TiO_2 Nanoparticles Under Various Conditions.

TiO_2 wt (g)	Time(min)	pH Adjustment	Initial conc.(ppm)	Adsorption capacity q (mg/g)	% Removal	Ref.
0.1 g	90	9-10	20	-	97.727%	[31]
0.1 g	90	9-10	60	-	68.26%	[31]
0.08 g	120	5	100	32.89	92%	[32]
0.01 g	5 min	Without adjustment	15	13.392	89.28%	This work

4. Conclusion

The results of the present study confirmed the successful application of Cynara scolymus seed extract as a green reducing and stabilizing agent for the synthesis of TiO2 nanoparticles. The obtained nanoparticles showed spherical morphology, and the particle size range was found to be in the range 10-30 nm, which clearly proves the effectiveness of the proposed synthesis route. TiO2 nanoparticles were synthesized, which exhibited a very good capacity to uptake the Pb^{2+} ions from water rapidly without the requirement of pH adjustment or longer contact time, thus promising as an effective adsorbent for water treatment.

The use of ultrasound-assisted extraction in addition to green synthesis offers a simple, inexpensive, and eco-friendly method for the production of nanoparticles with fewer hazardous chemicals. The results indicated that the prepared TiO2 nanoparticles could be practically used in the environment because of its potential in remediating lead contaminated water. They should be further studied for regeneration, reusability and upscaling, for their application in sustainable wastewater treatment.

5. References

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