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RESEARCH ARTICLE

Comparative Study of Green Synthesis to Prepare TiO₂ Nanoparticles from Plant Shard Leaves Extract by Using Microwave Irradiation and Microjet Plasma

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ABSTRACT

Plant extracts have been widely investigated as a safer alternative for the green synthesis of metal oxide nanoparticles. This study presents a green synthesis approach using shard leaf extract assisted by microwave irradiation and microjet plasma for the preparation of titanium dioxide nanoparticles (TiO₂ NPs). The synthesized nanoparticles were characterized using Field Emission Scanning Electron Microscopy (FESEM), X-ray diffraction (XRD), ultraviolet-visible spectroscopy (UV-Vis), Fourier Transform Infrared Spectroscopy (FTIR), and zeta potential (ZP) analysis. XRD results confirmed the formation of the anatase phase of TiO₂ without detectable brookite or rutile peaks, indicating high phase purity. The crystal structure was tetragonal with average crystallite sizes of 11.05 nm and 11.04 nm for microwave and microjet plasma methods, respectively. UV-Vis analysis revealed band gap energies of 3.55 eV and 3.43 eV for the respective synthesis methods. FESEM images showed irregular spherical nanoparticles with average diameters ranging from 43.48 to 46.52 nm. Zeta potential measurements indicated moderate stability of the nanoparticles with values of 76.8 mV and –68.5 mV. The antibacterial activity of TiO₂ NPs was evaluated against Gram-positive and Gram-negative bacteria, showing higher effectiveness against Gram-positive bacteria such as *Staphylococcus aureus* compared to *Escherichia coli*. Additionally, the nanoparticles demonstrated excellent adsorption efficiency for heavy metals, including lead, chromium, and cadmium, achieving up to 100% removal from aqueous solutions. These results highlight the potential of green-synthesized TiO₂ nanoparticles for environmental remediation and antibacterial applications.

Keywords: Biosynthesis, Chard leaf, Jet plasma, Microwave, TEM

Introduction

Environmental pollution, particularly water contamination by toxic heavy metals and pathogenic microorganisms, has become a serious global concern. Photocatalysis has emerged as an effective and sustainable approach for environmental remediation due to its ability to degrade pollutants and inactivate microorganisms.¹ The development of

recyclable and environmentally friendly photocatalysts is therefore essential to minimize secondary pollution and ensure long-term environmental sustainability.² Nanoparticles (NPs) are extremely small materials that exhibit unique size-dependent properties, including a high surface-area-to-volume ratio, which makes them attractive for various environmental and catalytic applications.³ To overcome these limitations, green synthesis nanotechnology has

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emerged, utilizing extracts from yeast, fungi, bacteria, algae, and medicinal plants.⁴ Researchers from a variety of disciplines have taken an interest in metal oxide nanoparticles because of their numerous technological uses.⁵ Titanium dioxide (TiO₂), in particular, is an essential industrial component used in paints, pigments, cosmetics, and other goods.⁶ It is also utilized in image splitting devices, anti-reflection coatings, and optical coatings due to its high dielectric constant and refractive index.^{7,8} At high temperatures, it has been employed as an oxygen and humidity sensor.^{9,10} TiO₂NPs may exist in three different phases at the nanoscale—anatase, rutile, and brookite—depending on the synthesis temperature. Despite its numerous advantages, the practical application of TiO₂ NPs is often limited by factors such as particle agglomeration, high energy consumption during synthesis, and the use of hazardous chemicals in conventional preparation methods.¹⁰ These limitations necessitate the development of alternative synthesis approaches that are both energy-efficient and environmentally benign.^{11,12} In addition to being biocompatible and commercially feasible, TiO₂NPs has demonstrated antibacterial action.¹³ Moreover, anatase-phase TiO₂NPs has proven effective as a heavy metal adsorbent.¹⁴ The potential of nano titanium dioxide as an effective adsorbent in treating contaminated water is therefore significant, enhanced by a study showing it was effective in removing heavy metals Hg²⁺, Cd²⁺, and Pb²⁺ from water.¹⁵ The use of microwave radiation was first introduced in synthetic chemistry in the 1980s; since then, it has offered a significant and eco-friendly method for creating metal oxide nanoparticles in a variety of sizes and shapes.¹⁶ High reaction rate, uniform volumetric heating, selectivity, and enhanced product yield are some of this radiation's unique characteristics.¹⁷ The microwave-assisted oxide synthesis approach is becoming more popular due to its quick reaction rate, effective heat transmission, and eco-friendly characteristics. In this technique, the material is heated directly with radiation instead of indirectly through heat sources, resulting in a more consistent temperature distribution within the reaction mixture.^{18,19} One of the several uses of cold plasma in material surface treatment is the production of supported metal nanoparticles.^{20,21} Cold plasma can be used to reduce noble metal nanoparticles in a number of ways, such as: (1) being environmentally friendly because it doesn't require chemical reducing agents harmful to the environment or biology; (2) operating at room temperature; (3) operating without impurities; (4) having a uniform distribution and small diameter of metal nanoparticles; (5) being economical; and (6) being easy to immobilize between metal nanoparticles and supporting

material surfaces.^{22,23} In general, there are several methods for creating cold plasma for material treatment, including discharge plasma, glow plasma, and microwave radiation plasma.²⁴ Plant extracts have been extensively studied as a safer substitute for conventional approaches in the production of metal oxide nanoparticles.²⁵ Although various green synthesis strategies for TiO₂ NPs using plant extracts have been reported, systematic comparative studies focusing on different energy-assisted synthesis techniques remain limited. In particular, a direct comparison between microwave irradiation and microjet plasma methods using the same plant extract has not been sufficiently explored. This lack of comparative evaluation represents a clear research gap in the current literature. In this study, titanium dioxide nanoparticles were synthesized in an efficient and environmentally friendly manner using chard leaf extract via microwave irradiation and microjet plasma techniques. The novelty of this work lies in the direct comparison of these two green synthesis approaches under similar conditions and in evaluating their influence on the structural, optical, antibacterial, and environmental performance of the produced nanoparticles. A comprehensive characterization was conducted to investigate the structural, optical, and morphological properties of the synthesized TiO₂ NPs. Furthermore, their antibacterial activity and heavy metal adsorption capability were assessed to demonstrate their potential applications in environmental remediation.

Materials and methods

The materials utilized for this investigation, titanium tetraisopropoxide (Ti[OCH(CH₃)₂]₄, TTIP) (DIREVO Industrial Biotech, Germany), ethanol (Eth) pure 99%, deionized water, Fresh chard leaves from the local market.

Preparation of chard leaf extract

The chard leaves were subjected to a thorough washing process which included three rinses with tap water to remove dust particles, followed by a final rinse with distilled water to ensure optimal cleanliness. After removing excess moisture from the leaves, they were chopped and dried for five days in a room free from direct sunlight. The resulting dried leaves were then crushed into a fine powder using a home-made grinder. The selection of chard leaf extract was based on its rich phytochemical content, including phenolic compounds, flavonoids, tannins, and sugars, which act as natural reducing and stabilizing agents. These bioactive compounds



Fig. 1. The steps for preparing the extract from chard leaf.

facilitate controlled nucleation and prevent agglomeration of the TiO_2 nanoparticles, and provide advantages over other plant extracts previously used, such as easy availability, non-toxicity, and the production of nanoparticles with uniform size and enhanced functional properties.²⁶ The samples of chard leaf extract were prepared by combining 5 g of chard leaf powder with 100 ml of deionized water, then stirring at 70 °C for 30 min. Following that, centrifuge for half an hour. The extract was filtered using filter paper as shown in Fig. 1. Retaining the extract to create titanium dioxide nanoparticles was the last step.

Preparation of TiO_2 NPs from chard leaf extract using microwave irradiation

To synthesize titanium dioxide nanoparticles, we first take 8 ml of titanium tetraisopropoxide ($\text{Ti}[\text{OCH}(\text{CH}_3)_2]_4$, TTIP) and 15 ml of chard leaf extract in an Erlenmeyer flask and put it on a mag-

netic stirrer for half an hour. We put the flask in a microwave at 120 °C for 10 minutes. The color of the resulting solution formed after mixing titanium tetraisopropoxide ($\text{Ti}[\text{OCH}(\text{CH}_3)_2]_4$, TTIP) with the extract changes from transparent to white, brown as shown in Fig. 2. This color change mainly occurs due to the reduction of metal ions which indicates the formation of TiO_2 NPs. The synthesized nanoparticles are separated by centrifuging the mixture at 4000 rpm for 20 minutes. It was washed three times with distilled water and ethanol and dried using an oven. For three hours, the powder was calcined at 400 °C to obtain the TiO_2 NPs.

Preparation of TiO_2 NPs from chard leaf extract by low-temperature microjet plasma

To synthesize titanium dioxide nanoparticles, we first take 8 ml of titanium tetraisopropoxide and 15 ml of chard leaf extract in an Erlenmeyer flask

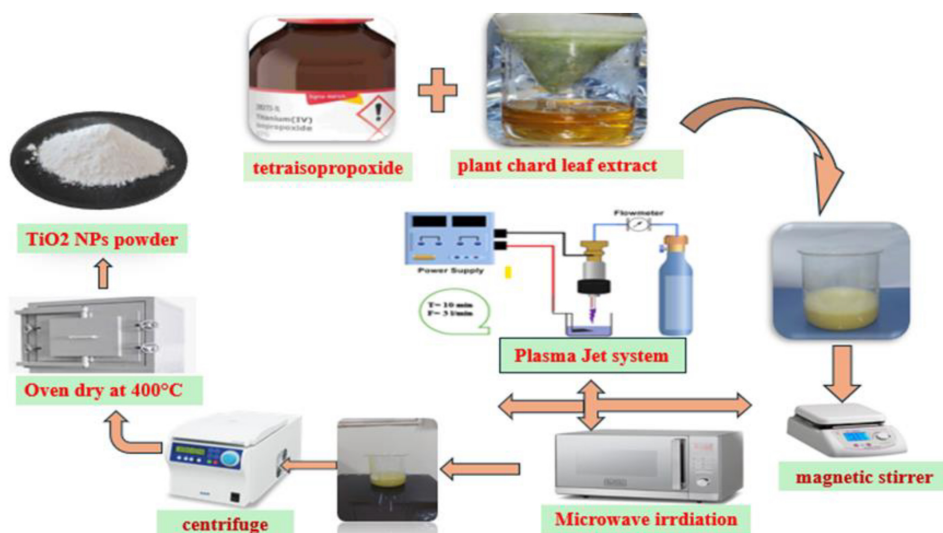


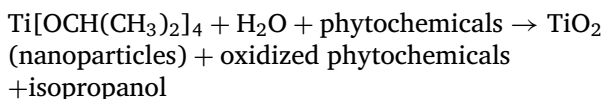
Fig. 2. Schematic green synthesis of TiO_2 NPs by microwave irradiation and microjet plasma.

and keep the flask on a rotating shaker for about half an hour. After establishing the distance between the electrodes, a milliliter of the solution is poured in a glass beaker beneath the plasma system's electrodes. Within the glass beaker, argon gas is shown to flow at a rate of 5 L/min for two minutes of exposure, as shown in Fig. 2. Following the application of a high electric potential difference, the gas molecules are ionized, a miniature plasma jet is created inside the solution, and reactive oxygen and nitrogen species, including ozone and hydroxyl radicals, react. Titanium oxide nanoparticles are produced as a result of this reaction, and the microjet plasma help keep the particles stable by supplying a charged environment that inhibits agglomeration and keeps them tiny.⁷ Centrifuging the mixture for 20 minutes at 4000 rpm separates the produced nanoparticles. It was washed three times with distilled water and ethanol and then dried using an oven. For three hours, the powder was calcined at 400 °C to obtain the TiO₂NPs.

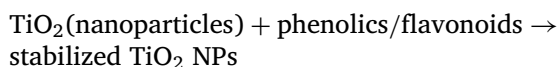
Chemical reaction pathway

The formation of TiO₂ NPs using titanium tetraisopropoxide (TTIP) and chard leaf extract can be represented by the following simplified pathway. Phytochemical compounds in the extract act as both reducing and stabilizing agents:

1. Hydrolysis and reduction of TTIP:



2. Stabilization of nanoparticles:



Note: This is a simplified representation; the chard leaf extract contains a mixture of bioactive compounds (such as phenolics and flavonoids) that contribute to both reduction and stabilization.

Results and discussion

Fourier transform infrared spectroscopy (FTIR)

Fourier transform infrared spectroscopy was used to evaluate the presence of phytochemicals on the surface of titanium dioxide nanoparticles. FTIR analysis of green TiO₂ nanoparticles (NPs) was carried out in the frequency range of 400–4000 cm⁻¹. The functional groups identified in the synthesized TiO₂ NPs prepared via microwave irradiation and microjet plasma-assisted methods are summarized in Table 1. Fig. 3 show peaks at 3429.81 cm⁻¹, 3430.07 cm⁻¹ using the microwave irradiation and jet plasma respectively, due to the vibration of water molecules on the surface of nanoparticles.^{26,27} The C–H and N–H groups were found at 2956.31 cm⁻¹–2074.95 cm⁻¹ using microwave irradiation, while at 2956.82 cm⁻¹, 2170.38 cm⁻¹ using microjet plasma, these groups show the presence of protein, carbohydrates, and fatty acids from the extract of chard leaves.⁸ Vibrational bands in the range 1735–1631.39 cm⁻¹ were recorded using microwave radiation, and in the range 1741.46–1632.19 cm⁻¹ using microjet plasma technology, attributed to carbonyl bond vibrations (C=O).⁹ These bands indicate the presence of organic compounds of plant origin, such as carboxylic acids or phenols, which potentially contribute to the stabilization or encapsulation of titanium dioxide nanoparticles. Bands also appeared in the range 1466.07–1112.93 cm⁻¹ using microwave radiation, and 1465.70–1113.31 cm⁻¹ using microjet plasma technology, attributed to C–C and C–O bond

Table 1. Functional groups found in TiO₂NPs using microwave irradiation and microjet plasma.

	Wave number of TiO ₂ (cm ⁻¹) Microwave irradiation	Wave number of TiO ₂ (cm ⁻¹) Microjet plasma	No. of Reference
O-H	3429.81	3430.07	9
C-H	2956.31	2956.82	8
N-H	2924.19	2924.61	
	2853.95	2853.55	
	2074.95	2170.38	
C=O	1735 1631.39	1741.46 1632.19	10
C-C	1466.07	1465.70	5
C-O	1384.98	1384.79	
	1270.01	1384.79	
	1112.93	1113.31	
T-O-T	719.96	722.31	5

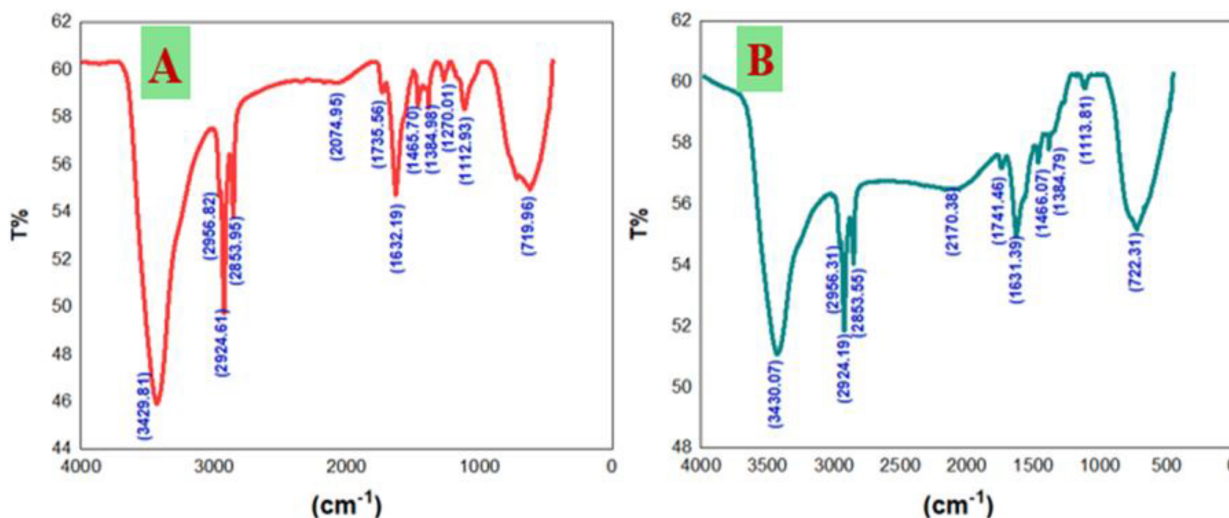


Fig. 3. The FTIR spectrum of TiO₂NPs prepared using green approach A. microwave irradiation, b. microjet plasma.

vibrations, indicating the presence of cellulose groups and aromatic compounds associated with plant biocomponents.⁴ The peaks at 719.96 cm⁻¹ using microwave and 722.31 cm⁻¹ using microjet plasma are attributed to T–O–T vibrations, which are clear evidence of the formation of TiO₂ NPs⁴. FTIR results for both microwave irradiation and microjet plasma prepared TiO₂NPs showed similar peaks, especially in the O–H, C–H, and Ti–O bands, indicating a similar structural composition with only minor differences in peak intensity or position. This convergence confirms studies.^{10,11} On the effectiveness of both methods in preparing TiO₂ NPs in a green method. Minor differences in FTIR band positions and intensities between microwave-assisted and microjet plasma samples can be attributed to slight variations in surface functional groups and interactions with residual phytochemicals. These shifts are small and do not affect the overall TiO₂ NPs structure or functional properties.

X-ray diffraction analysis

XRD analysis was performed to determine the crystalline phase and crystallite size of TiO₂ NPs synthesized using microwave irradiation and microjet plasma. As shown in Fig. 4, within the range of 20° to 80° (2θ), seven peaks were observed, corresponding to the anatase phase and in good agreement with JCPDS card number 98-009-2363. The observed diffraction peaks correspond to the anatase phase, with no peaks indicating the presence of rutile or brookite, confirming that all samples are pure anatase.⁸ The crystallite size of the nanoparticles was

calculated using the Debye–Scherrer Eq. (1):

$$D = \frac{0.9\lambda}{\beta \cos \Theta} \quad (1)$$

where D is the crystallite size, 0.9 is the Debye–Scherrer constant, β is the full width at half maximum (FWHM), and λ is the CuKα radiation wavelength (0.154 nm). Using this equation, the crystallite sizes were determined to be 11.050 nm and 11.043 nm for the TiO₂ NPs synthesized by microwave irradiation and microjet plasma, respectively. The similarity in crystallite size and diffraction patterns between the two methods is attributed to the rapid and homogeneous heating achieved at the molecular level, which promotes uniform nanoparticle formation.^{12,13} This negligible difference demonstrates that both synthesis methods produce TiO₂ NPs with comparable structural and size properties, as summarized in Table 2.

Zeta potential

A particle size analyzer was used to examine the electrostatic characteristics of TiO₂NPs submerged in distilled water, or the aqueous phase. The green-synthesized TiO₂NPs zeta potential is displayed in Fig. 5. Recent research indicates that particles tend to be more stable when their zeta potential is greater than +30 mV or less than -30 mV.¹⁴ By employing microwave irradiation Fig. 5 and the microjet plasma Fig. 5, the nanoparticles' zeta potential was determined to be 76.8 mV and -68.5 mV, respectively. This result for both cases shows that the particles have a high surface charge, which hinders their ability to aggregate by creating electrical repulsion between them. As a result, particles in this zeta potential

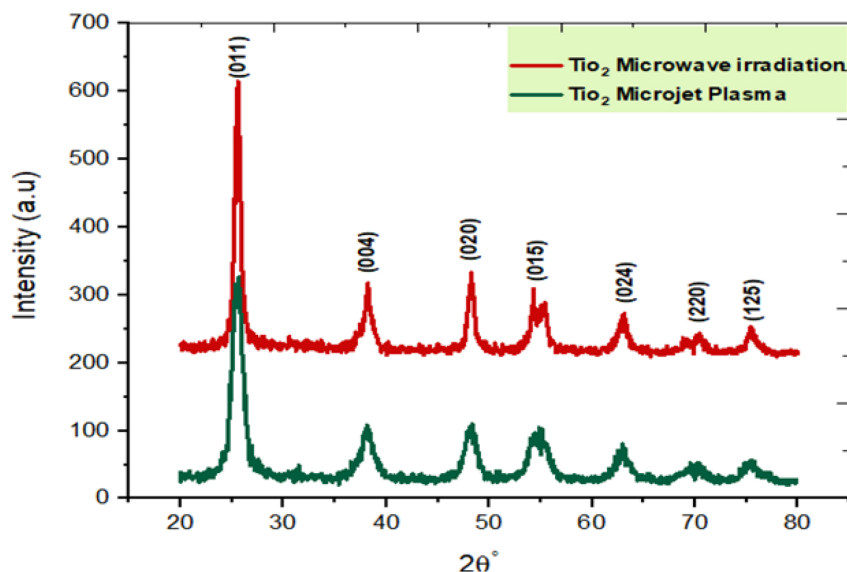


Fig. 4. XRD pattern of green synthesis TiO_2 NPs prepared using microwave irradiation and microjet plasma.

Table 2. The structure properties for green synthesis TiO_2 NPs using microwave irradiation and microjet plasma.

Sample	2θ (Deg)	d_{hkl} ($\text{\AA}^0$)	FWHM (Deg)	Hkl	D (nm)
TiO_2 NPs (Microwave irradiation)	25.55	3.501	0.736	(011)	11.050
	38.15	2.357	1.156	(004)	7.267
	48.25	1.885	0.769	(020)	11.300
	54.25	1.686	1.752	(015)	5.105
	63.15	1.472	0.986	(024)	9.436
	70.1	1.333	0.836	(220)	11.616
	75.4	1.257	0.816	(125)	12.301
TiO_2 NPS (Microjet plasma)	25.55	3.501	0.737	(011)	11.043
	38.15	2.357	1.161	(004)	7.237
	48.25	1.885	0.771	(020)	11.276
	54.25	1.686	1.755	(015)	5.094
	63.15	1.472	0.991	(024)	9.392
	70.1	1.333	1.616	(220)	5.998
	75.4	1.257	0.822	(125)	12.218

region exhibit high stability.¹⁵ This suggests that TiO_2 NPs produced using environmentally friendly techniques have good stability. The stable zeta potential indicates the presence of functional groups attached to the nanoparticles. The stability of the nanoparticles and the reduction of metal ions can be attributed to the phenolic compounds and proteins present in the plant extract used in the synthesis process.¹⁶ The particles prepared by microwaves carried a positive surface charge (76.8 mV), due to the interaction of microwaves with organic compounds, which contributes to the formation of a positively

charged surface layer. Meanwhile, the particles prepared by microjet plasma exhibited a negative charge (-68.5 mV), resulting from the high ionization caused by the microjet plasma, which generates negative ions on the particle surface. Despite the difference in charge type, the electrical stability in both cases is attributed to electrostatic repulsion. Studies by¹⁷ showed that microwave heating promotes the formation of positive charges, while the results of¹⁸ confirmed that microjet plasma contributes to the formation of stable negative charges on the particle surface.

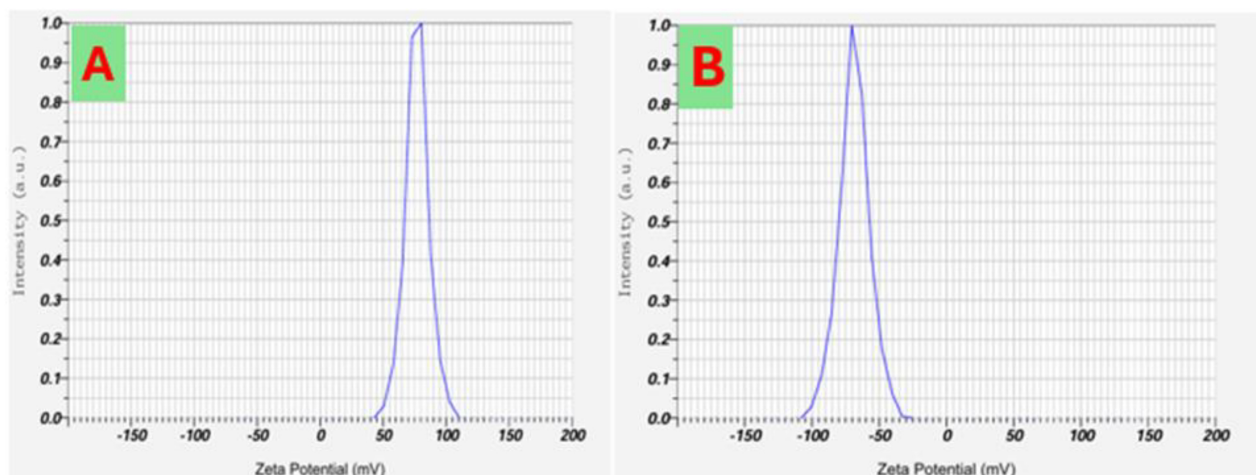


Fig. 5. The zeta potential of TiO_2 NPs, (A) microwave irradiation (B) microjet plasma.

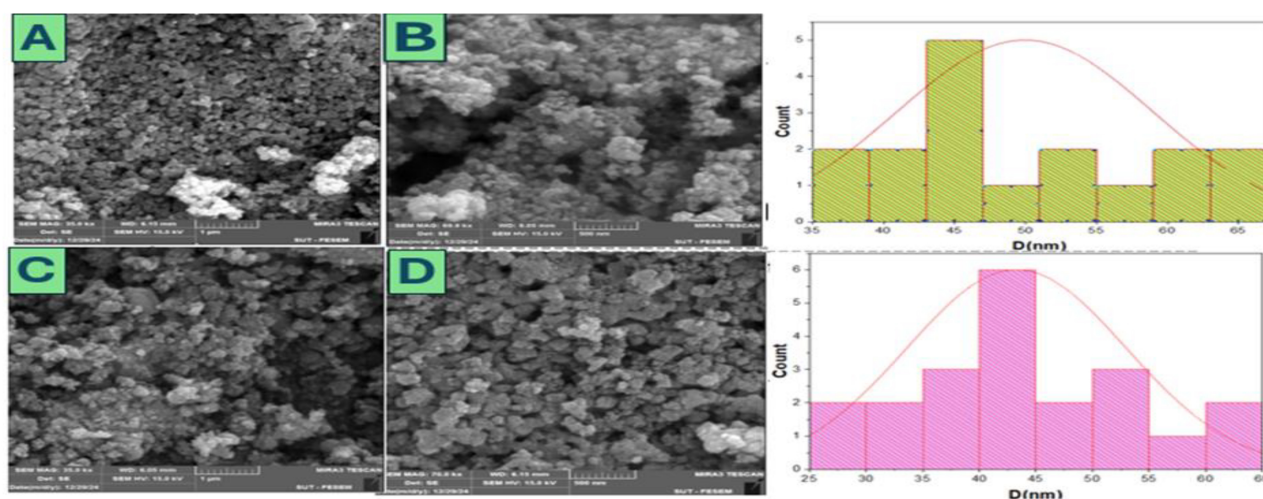


Fig. 6. Shown the FESEM of TiO_2 NPs prepared by green method (A), (B) microwave irradiation and (C), (D) microjet plasma.

Field emission scanning electron microscopy (FESEM)

Field emission scanning electron microscopy (FESEM) is a sophisticated scanning electron microscope (SEM) that can image objects at extremely high resolutions, all the way down to the nanoscale range. The FESEM images of two TiO_2 NPs powders generated by green synthesis using microwave irradiation and microjet plasma are shown in Fig. 6, at various magnifications, it shows spherical and irregularly shaped nanoparticles for TiO_2 NPs, and the agglomeration of nanoparticles is found on the surface due to several biomolecules on the surface of the nanoparticles, the electrostatic force on the surface of the nanoparticles brings in other molecules.²⁸ The average size of TiO_2 nanoparticles was 46.52 nm when microwave irradiation was used,

while it was 43.48 nm when microjet plasma was used. Similar results were observed when microwave irradiation and microjet plasma were used, with the smaller size in microjet plasma being due to the influence of the preparation method on the size and distribution of TiO_2 nanoparticles, reaction conditions, and material flow. This comparison is supported by studies.^{12,19} EDS was used to determine the nanoparticles' elemental composition, as seen in Fig. 7(A, B) and Table 3. The distinctive peaks in the resultant spectrum verified the existence of [Ti, O]. According to the specified preparation target, the sharp peaks of oxygen (O) and titanium (Ti) suggest that the sample's primary composition was TiO_2 NPs. By demonstrating that the elemental composition of the sample matches the predicted one, the results validate the purity of the generated TiO_2 NPs and the efficiency of the production method in both ways.

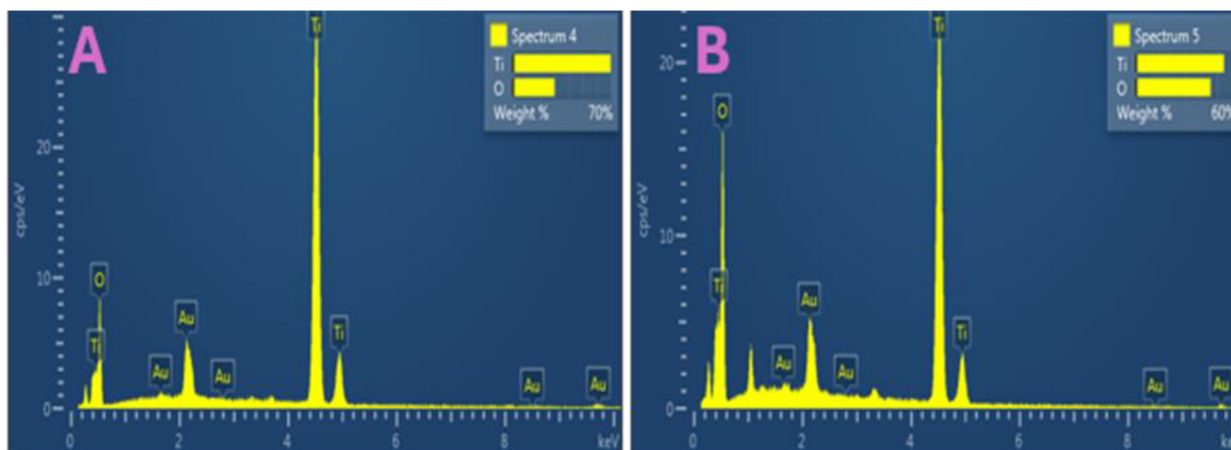


Fig. 7. EDX spectra and composition of elements for TiO_2 NPs (A) microwave irradiation (B) microjet plasma.

Table 3. Quantitative EDS elemental composition of TiO_2 NPs synthesized by microwave irradiation and microjet plasma.

Element	Wt%	Wt% Sigma	Atomic %	
O	45.92	0.58	71.77	Microwave irradiation
Ti	54.08	0.58	28.23	
Total:	100.00		100.00	
O	30.04	0.72	56.24	Jet Plasma
Ti	69.96	0.72	43.76	
Total:	100.00		100.00	

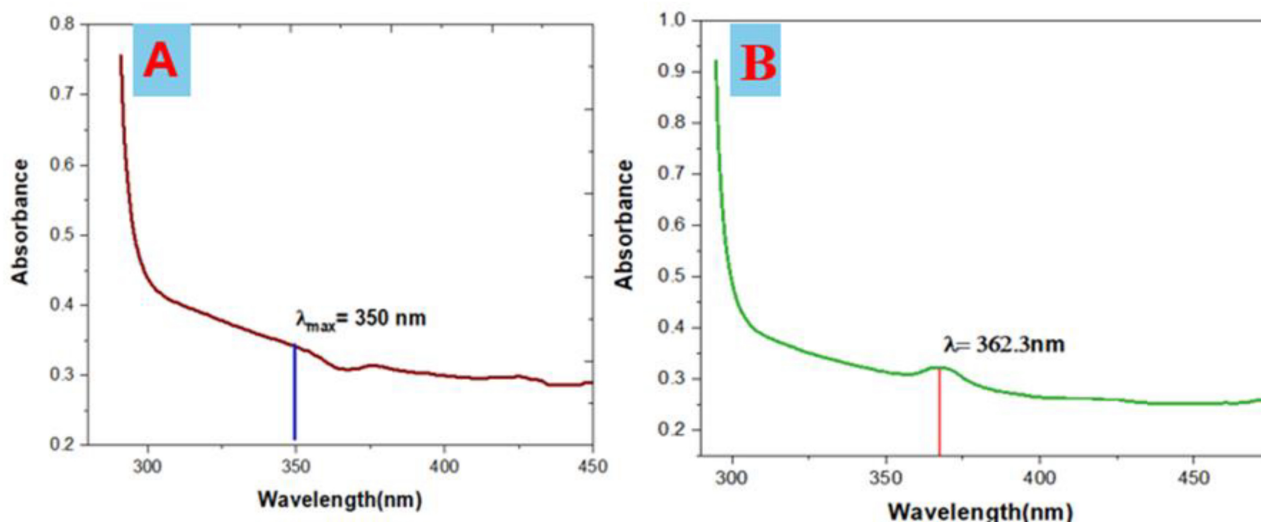


Fig. 8. UV-Vis absorption spectra of TiO_2 NPs (A) microwave irradiation (B) microjet plasma.

Titanium (Ti) and oxygen (O) elements were clearly present in the samples prepared using microwave irradiation and microjet plasma, confirming the formation of TiO_2 in both cases. These results reflect the similarity of the chemical composition of the resulting particles regardless of the preparation method, which is supported by studies.^{2,11}

Optical properties of TiO_2 NPs

Titanium dioxide (TiO_2) nanoparticles were synthesized using the green technique, employing chard leaf extract as a catalyst. The optical properties of the nanoparticles were examined using UV-Vis spectroscopy. The maximum absorption wavelengths (λ_{max}) observed in Fig. 8 (A and B) were 350 nm

Table 4. Systematic comparison of TiO₂ NPs synthesized via microwave irradiation and microjet plasma, showing key structural, morphological, and optical properties.

Property / Characteristic	Microwave Irradiation	Microjet Plasma	Comment / Observation
FTIR – Ti–O–Ti (cm ⁻¹)	719.96	722.31	Evidence of TiO ₂ formation; minor shift in peak position
XRD Crystallite Size (nm)	11.050 – 12.301	5.094 – 12.218	Comparable, minor variations due to synthesis conditions
FESEM Particle Size (nm)	46.52	43.48	Microjet plasma produces slightly smaller particles
Zeta Potential (mV)	+76.8	-68.5	Different surface charge type, both highly stable due to electrostatic repulsion
EDS Composition (Wt%)	Ti 54.08, O 45.92	Ti 69.96, O 30.04	Confirms primary TiO ₂ composition and sample purity
UV-Vis Band Gap (eV)	3.55	3.43	Slightly higher E _g in microwave due to smaller particle size; microjet plasma shows better crystallinity

for the microwave-assisted sample and 362.3 nm for the microjet plasma sample, corresponding to energy bandgaps (E_g) of approximately 3.55 eV and 3.43 eV, respectively, calculated using the Kubelka–Munk (K–M) method following the procedure reported in ¹⁶:

$$E_g = \frac{hc}{\lambda} = \frac{1240}{\lambda} \quad (2)$$

where c is the speed of light (3×10^8 m/s), h is Planck's constant (6.626×10^{-34} J.s), and $\lambda_{\max}$ is the wavelength at the absorption maximum. ¹⁹ Typically, TiO₂ NPs have a bandgap ranging from 3.3 to 3.5 eV, depending on crystal phase, particle size, and synthesis method. ²⁰ The slightly higher E_g value in the microwave-assisted sample (3.55 eV) is attributed to its smaller particle size and the quantum confinement effect, while the slightly lower E_g in the microjet plasma sample (3.43 eV) indicates better particle crystallinity. These minor differences reflect the distinct synthesis conditions, while the nanoscale properties remain similar and effective in both methods, making them suitable for medical and environmental applications. ^{21,22}

Comparison between microwave and microjet plasma syntheses

A systematic comparison between TiO₂ NPs synthesized via microwave irradiation and microjet plasma was carried out. Key differences and similarities in structural, morphological, and optical properties are summarized in Table 4.

As summarized in Table 4, TiO₂ NPs synthesized using microwave irradiation and microjet plasma exhibit comparable structural, morphological, and optical properties, with minor differences attributable to the synthesis method. FTIR analysis confirms the presence of Ti–O–Ti bonds in both samples, indicating successful TiO₂ formation. The slight peak shift in the microjet plasma sample (722.31 cm⁻¹ vs. 719.96 cm⁻¹) suggests minor variations in bond environment or crystallinity. XRD results show that the crystallite sizes of both samples are similar,

with microwave-assisted nanoparticles slightly larger (11.05–12.30 nm) than microjet plasma nanoparticles (5.09–12.22 nm), likely due to differences in thermal energy input and particle growth dynamics during synthesis. FESEM images reveal that particle sizes are slightly smaller in microjet plasma samples (43.48 nm) compared to microwave-assisted nanoparticles (46.52 nm), reflecting the influence of synthesis conditions on particle growth and distribution. Zeta potential measurements indicate that microwave-assisted nanoparticles carry a positive surface charge (+76.8 mV), due to interactions with organic compounds from the green extract, while microjet plasma nanoparticles are negatively charged (-68.5 mV) because of high plasma ionization. In both cases, the high magnitude of zeta potential ensures strong electrostatic stability and prevents particle aggregation. EDS analysis confirms that both samples are composed primarily of Ti and O, verifying the purity of TiO₂. Minor differences in the Ti/O ratio reflect small variations in surface chemistry or oxygen adsorption. UV-Vis spectroscopy shows a slightly higher band gap in microwave-assisted nanoparticles (3.55 eV) compared to microjet plasma nanoparticles (3.43 eV), which can be attributed to smaller particle size and quantum confinement effects. The slightly lower band gap in microjet plasma nanoparticles indicates slightly improved crystallinity. Overall, both synthesis methods effectively produce high-purity TiO₂ nanoparticles with similar structural and functional properties, while microwave-assisted synthesis offers faster preparation and energy efficiency, and microjet plasma provides slightly improved crystallinity. This systematic comparison highlights minor variations in crystallite size, particle size, surface charge, and optical properties, providing a clear and structured overview of the two synthesis methods.

Antibacterial activity of TiO₂NPs

Antibiotic-resistant pathogenic bacterial species have been demonstrated to be significantly eradicated by biogenic metallic nanoparticles. ²⁹ Because of their nanoscale size, which makes it easier for

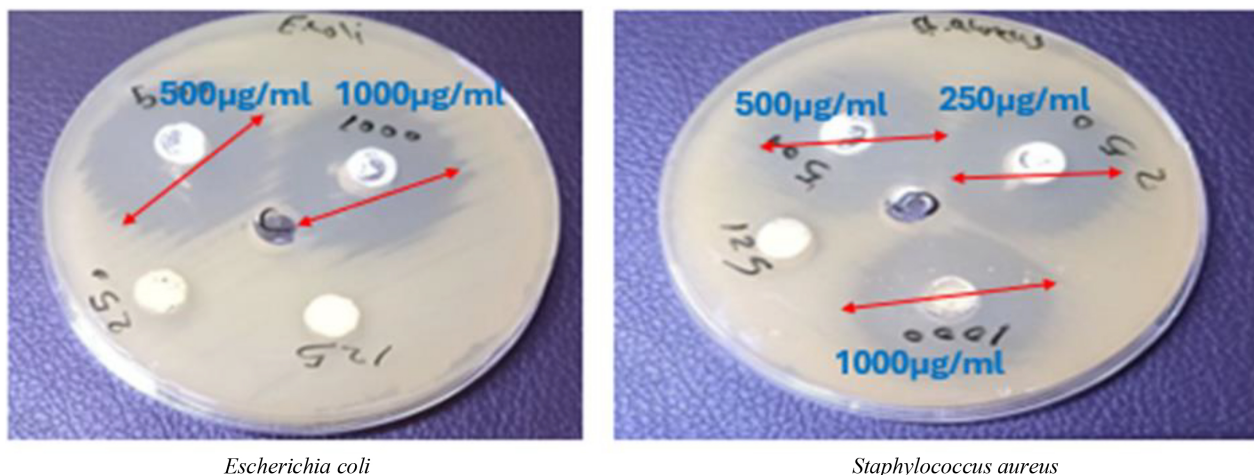


Fig. 9. Antibacterial activity of TiO₂ NPs using microwave irradiation by disk diffusion method.

Table 5. Antibacterial activity for TiO₂ NPs using microwave irradiation by agar disk-diffusion method.

Microorganisms	Type Bacteria	Zone of inhibition (mm)			
		(1000) µg/ml	(500) µg/ml	(250) µg/ml	(125) µg/ml
<i>Escherichia coli</i>	Gram-negative	38	36	0	0
<i>Staphylococcus aureus</i>	Gram-positive	32	28	27	0

them to adhere to and damage bacterial cell walls or penetrate the interior, where they disrupt vital biological components, metal nanoparticles (NPs) have antibacterial action.³⁰ TiO₂NPs are known to be safe, stable, and effective antibacterial agents that don't hurt either people or animals.³¹ TiO₂NPs change the microenvironment of the contact area when they come into touch with bacterial cells because they build up inside the microorganisms.²⁶ Bacterial cells get damaged and disorganized as a result of this contact, which releases antimicrobial ions inside the cells, causing toxicity.⁴ These ions could enter the intracellular space and harm the cell membrane.²⁶ Due to the similarity of structural and optical results of titanium dioxide prepared using microwave and microjet plasma, microwave irradiation method has been adopted in applications bacterial inhibition and adsorption of heavy metals from water. This finding also demonstrates the novelty of our study, showing that microwave-assisted synthesis can provide nanoparticles with comparable functional performance to microjet plasma while being faster and more energy-efficient.

Antibacterial activity of TiO₂ NPs prepared by microwave irradiation using the agar disc method

The agar disk-diffusion method was employed to inhibit *Escherichia coli* and *Staphylococcus aureus* in this work. To prepare the nanoparticle solution for

bacterial inhibition, 0.01 g of TiO₂NPs was added to 10 ml of distilled water, bringing the concentration to 1000 µg/ml. From this concentration, diluted concentrations of 500 µg/ml, 250 µg/ml, and 125 µg/ml were prepared, respectively. Fig. 9 and Table 5 demonstrate that the TiO₂NPs exhibited the highest zone of inhibition against *A. Staphylococcus aureus* and *Escherichia coli* bacteria at 1000 µg /mL.

Adsorption of heavy metals using TiO₂NPs prepared by microwave irradiation

Research has proven the effectiveness of using nanoparticles in treating polluted water. Titanium dioxide nanoparticles have better heavy metal adsorption capabilities than traditional materials like activated carbon because of their distinct physical and chemical characteristics.³² Titanium dioxide's nanostructure offers a high surface-to-volume ratio, which creates a large number of surface interaction sites and adsorption sites for heavy metals.²⁸ To investigate the effectiveness of the TiO₂NPs for heavy metal adsorption, 10 ppm (standard solution) of each of lead(Pb), chromium(Cr) and cadmium(Cd) was prepared in the laboratory to be used at crude concentrations to study the efficiency of the nano-material in absorbing heavy metals. and compare the results Depending on standards Central Organization for Standardization and Quality Control Iraqi (COSQC.IQ). TiO₂NPs are effective at adsorbing

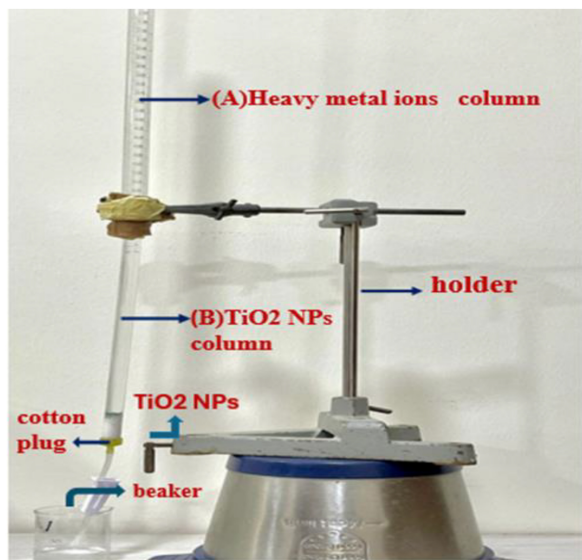


Fig. 10. A vertical adsorption system to remove heavy metals.

heavy metals in columns, according to the results. To carry out the adsorption procedure, the contaminated water was run through two glass columns. The diameter of column (A) is 2.5 cm and that of column (B) is 1 cm, as shown in Fig. 10. Column A is filled with water contaminated with heavy metals, while the glass column (B) is packed with TiO_2NPs and secured with a cotton plug at the end to prevent loss during the heavy metal adsorption process. The pH value was

5. The contaminated water was divided into 50 mL portions, and the adsorption time for each portion was determined based on the time required for complete adsorption of heavy metals from that portion. Specifically, the first portion required 3 hours, the second 4 hours, the third 4.5 hours, and the final portion 5 hours. This stepwise approach ensures that each portion is fully treated and allows systematic investigation of the effect of adsorption time on removal efficiency and the progression toward equilibrium. Although the adsorption experiment was conducted using a single prepared batch of TiO_2NPs , the contaminated water was divided into five 50 mL portions, each treated separately for different adsorption durations. This stepwise approach allowed assessment of adsorption efficiency at multiple contact times and provides a practical measure of reproducibility within the experiment. Reported removal efficiencies and adsorption capacities therefore reflect the average performance across these portions. Possible sources of experimental variability include minor fluctuations in pH, slight losses during solution transfer, and instrumental measurement uncertainty of the atomic absorption spectrometer (AAS). An atomic absorption spectrometer (AAS) was used to measure the heavy metal concentration after treatment, and Table 6 displays the concentration of heavy metal ions for the sample both before and after treatment, the removal efficiency (R) and adsorption capacity (q) and maximum allowed

Table 6. Heavy metal adsorption using TiO_2NPs and adsorption capacity (q), removal efficiency (r) after treatment.

Heavy metal ions	Time (hour)	Standard Concentration Prepare at the laboratory mg/l	Concentration after treatment by TiO_2NPs	Adsorption Capacity (q) After Treatment by TiO_2NPs Mg/g	Removal efficiency (R) After Treatment by TiO_2NPs %	Maximum Allowed (mg/L) (COSQC.IQ)
Pb	2	10	7.0	3	30%	0.01 Based on guide No.703
	3	10	5.0	5	50%	
	4	10	4.0	6	60%	
	4.5	10	0	10	100%	
	5	10	0	10	100%	
Cr	2	10	8.0	2	%20	0.05 Based on guide No.820
	3	10	6.0	4	%40	
	4	10	3.0	7	%70	
	4.5	10	0	10	%100	
	5	10	0	10	%100	
Cd	2	10	7.5	2.5	25%	0.003 Based on guide No.703
	3	10	4.5	5.5	55%	
	4	10	2.5	7.5	75%	
	4.5	10	0	10	100%	
	5	10	0	10	100%	

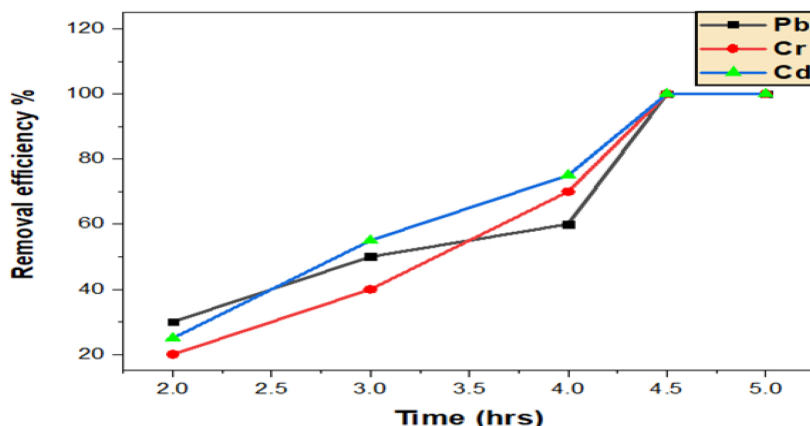


Fig. 11. Relationship between adsorption efficiency and the adsorption time using the TiO_2 NPs.

(mg/L) (COSQC.IQ). The removal efficiency (R) and adsorption capacity (q) were calculated using (3) and (4), following the method reported in reference³³

$$R = (C_0 - C) * 100 / C_0 \quad (3)$$

$$q = (C_0 - C) * V / M \quad (4)$$

where C_0 (mg/L) is the initial concentration (before adsorption), C (mg/L) is the final concentration (after adsorption). M is the mass of the magnetic nanoparticles, and V is the volume of the solution. Fig. 11 shows the relationship between adsorption time and adsorption efficiency. As the adsorption time increases, the adsorption efficiency of metal ions increases to 100%. Nanomaterials, thanks to their unique properties, have a large surface area and multiple active sites that improve adsorption efficiency are among the best materials for the adsorption of heavy metals from water. The amount of adsorbed metals can be gradually increased until saturation is reached by extending the adsorption time, allowing more time to reach equilibrium between the adsorbed ions and those still in solution.³⁰

Conclusion

In this work, it is concluded that green synthesis using microwave irradiation and microjet plasma is a rapid, environmentally friendly, and efficient synthesis method for successfully generating TiO_2 NPs. According to X-ray diffraction, samples created by microwave irradiation and microjet plasma displayed the tetragonal structure and anatase phase with extremely close crystal sizes. The presence of several bio functional materials functioning as capping and reducing agents to transform TiO_2 NPs was revealed by

FT-IR data. showed many bios functional molecules were found, operating as capping and reducing agents to change titanium dioxide nanoparticles. The energy band gap of TiO_2 NPs produced using green synthesis was determined to be 3.55 eV when using microwave irradiation and 3.43 eV when using microjet plasma. Using FESEM analysis, the spherical and irregular shapes of titanium dioxide nanoparticles produced using microwave irradiation and microjet plasma were verified. Zeta potential testing showed high stability of TiO_2 NPs. The result of adsorption efficiency of heavy metals (lead, cadmium, and chromium) was 100%. Titanium dioxide nanoparticles proved their effectiveness against Gram-positive bacteria *Staphylococcus aureus* and Gram-negative bacteria *Escherichia coli*. Through this research, it was proven that the results are similar in the preparation of titanium dioxide nanoparticles by the green method and via microwave radiation and microjet plasma.

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Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images that are not ours have been

included with the necessary permission for republication, which is attached to the manuscript.

- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at Al-Mustaqbal University.

Authors' contributions statement

Z.F.M. performed all of the experiments as part of his project and wrote the manuscript. Z.J.S. and T.S.G. were the principal investigators and supervised the whole work. All authors have read and agreed to the published version of the manuscript.

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دراسة مقارنة للتخليق الأخضر لتحضير جسيمات نانوية من ثاني أكسيد التيتانيوم من مستخلص نبات أوراق السلق باستخدام إشعاع الميكروويف وبلازما ميكروجيت

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الخلاصة

أجريت دراسات مكثفة على مستخلصات النباتات كبديل أكثر أماناً للتقنيات التقليدية في إنتاج جسيمات نانوية من أكاسيد المعادن. وبحثت الدراسة الحالية في نهج تخليق أخضر من مستخلص أوراق السلق باستخدام إشعاع الميكروويف وبلازما ميكروجيت لتخليق وتوصيف جسيمات نانوية من ثاني أكسيد التيتانيوم (TiO₂NPs). واستُخدمت تقنيات المجهر الإلكتروني الماسح للانبعاث الميداني (FESEM)، وحيود الأشعة السينية (XRD)، والتحليل الطيفي للأشعة فوق البنفسجية المرئية (UV-vis)، والتحليل الطيفي للأشعة تحت الحمراء بتحويل فورييه (FTIR)، وجهد زيتا (ZP) لتحليل هذه المادة. وقد تم تحديد البنية من خلال بيانات حيود الأشعة السينية، وأظهرت النتائج أن طور الأناتاز لجسيمات ثاني أكسيد التيتانيوم النانوية، وعدم وجود قمم حيود أخرى، مرتبطان بمرحلة البروكيت أو الروتيل. كان الهيكل رباعي الأضلاع بأحجام بلورية 11.05 نانومتر و11.04 نانومتر للعينات التي تم تعريضها للموجات الدقيقة والبلازما الميكروجيت، على التوالي. كانت فجوة النطاق لجسيمات TiO₂ النانوية عبر تعريض الموجات الدقيقة والبلازما الميكروجيت 3.55 إلكترون فولت و3.43 إلكترون فولت على التوالي، وفقاً لتوصيف الأشعة فوق البنفسجية المرئية. تم التحقيق في مورفولوجيا جسيمات TiO₂ النانوية الناتجة عن تعريض الموجات الدقيقة والبلازما الميكروجيت باستخدام مجهر مسح إلكتروني (FESEM). تراوحت أقطارها بين 43.48 و46.52 نانومتر، وكان لها شكل كروي غير منتظم. كشفت قياسات جهد زيتا أن الجسيمات كانت مستقرة إلى حد ما عند 76.8 و-68.5 مللي فولت، على التوالي. تم تأكيد فعالية جسيمات TiO₂ النانوية كعامل مضاد للبكتيريا لكل من البكتيريا موجبة الجرام وسالبة الجرام. أظهرت الجسيمات النانوية المُصنَّعة فعالية أكبر ضد البكتيريا موجبة الجرام مقارنةً بالبكتيريا سالبة الجرام، بقيم مُتقاربة (بالنسبة للمكورات العنقودية الذهبية والإشريكية القولونية). أجريت دراسة حول امتصاص المعادن (الرصاص والكروم والكاديوم) بواسطة جسيمات نانوية من ثاني أكسيد التيتانيوم لاستخدامها في إزالة الملوثات من الماء. وأظهرت النتائج أن جسيمات ثاني أكسيد التيتانيوم النانوية قادرة على إزالة الرصاص والكروم والكاديوم من المحاليل المائية بنسبة امتصاص 100%.

الكلمات المفتاحية: التخليق الحيوي، أوراق السلق، البلازما النفاثة، الميكروويف، المجهر الإلكتروني النافذ.