

Research Article

Removal of Bromothymol Blue from Industrial Wastewater using Manganese Oxide-Titanium Dioxide/ Multi-Walled Carbon Nanotubes Nanocomposite

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Article Info	Abstract
Article History	Industrial wastewater treatment techniques for pollutant removal have restrictions due to their high cost and low efficiency. The objective of this recent work is to develop high-performance nanocomposites to remove the bromothymol blue dye (BTB) from industrial wastewater. The nanocomposites developed and tested are composed of manganese oxide-titanium dioxide/Multi-walled carbon nanotubes (Mn/Ti/MWCNTs) nanoparticles of various concentrations. The synthesized nanocomposites have been characterized using X-ray diffraction (XRD), Energy-dispersive X-ray spectroscopy (EDX), and Field Emission Scanning Electron Microscopy (FESEM) techniques. Results show that the addition of metal oxide nanoparticles to the MWCNTs matrix significantly enhanced removal efficiency. The developed Mn/Ti/MWCNTs nanocomposite achieved 78% removal of BTB from industrial wastewater. The Mn-Ti/CNTs nanocomposite exhibits a removal enhancement of MWCNTs alone was 45% and 32% over TiO ₂ :MnO ₂ nanocomposite. The results of the current study demonstrate that the hybrid nanocomposite offers efficient removal of BTB from dye industrial wastewater compared to nano elements alone.
Received Mar 12, 2025	
Revised Jul 09, 2025	
Accepted Jul 13, 2025	
Keywords	
Bromothymol Blue Dye (BTB)	
MnO ₂ nanoparticles	
Walled Carbon Nanotubes (MWCNTs)	
TiO ₂ nanoparticles	
Wastewater treatment	



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1. Introduction

Industrial wastewater resulting from various activities has significantly increased with the expansion of the textile and paper industries. Such industries are considered major producers of industrial wastewater [1-3]. This wastewater contaminated with dyes represents a threat to both human health and aquatic ecosystems. The removal of dyes is considered an essential factor in achieving ecological stability and water safety [4-6]. Bromothymol blue (BTB) is one of the most common water pollutants due to its stable

nature and the difficulty in treating water contaminated by BTB dyes. Filtration by membrane, coagulation, biological treatment and adsorption techniques are the traditional treatment techniques used. These techniques have many disadvantages, such as low efficiency, incomplete removal of impurities and high cost [7, 8].

Nanomaterials, mainly metal oxide nanoparticles such as TiO_2 , MnO_2 , and Carbon nanotubes offer a promising alternative water treatment technique due to their large surface area, ability to enhance reactivity, and specialized physiochemical characteristics [9, 10]. TiO_2 nanoparticles are non-toxic and highly stable; in fact, a fast charge carrier recombinant is the only constraint of this technology [11, 12]. TiO_2 and MnO_2 nanoparticles effectively complement each other by enhancing pollutant degradation and facilitating oxidation-reduction reactions [13, 14].

The composite of TiO_2 and MnO_2 nanoparticles enhances the efficiency of dye removal with a significant adsorption effect [15, 16]. Multi-walled carbon nanotubes (MWCNTs) [17] possess unique properties, including good stability, a non-toxic nature, and highly thermoelectric and strength characteristics, which enhance their efficiency in removing organic pollutants [7, 18]. The integration of various nanoparticles to form MWCNTs-like clusters within TiO_2 and MnO_2 structures enhances their absorption capacity.

Many researchers have investigated and analyzed the methods used to synthesize MnO_2 nanoparticles, including hydrothermal, chemical precipitation, and sol-gel process techniques, to optimize particle size and shape to improve the electrochemical properties that work to remove contaminants from wastewater [19-21].

A highly effective composite catalyst for visible light prepared by B- TiO_2 and activated carbon (AC) was synthesized in this study using the sol-gel method in conjunction with argon heat treatment. The synthetic material consists of a single anatase phase. There was an improvement in light absorption, an efficient suppression of electron-hole complexation, a decrease in electron-transport resistance, and a narrowing of the bandwidth of the outlawed band (2.38 eV) after adding AC to the catalyst. Activated carbon with black TiO_2 provided strong evidence of the development of a chemical link (Ti-C-) in the B- TiO_2 /AC. The AC composite improved the catalysts' adsorption and degradation efficiency. Mokaba, et al. [22] showed that a 25 weight % of B- TiO_2 /AC in degrading rhodamine B (RhB) demonstrated impressive and consistent performance. When exposed to visible light for 60 minutes, 82% of RhB was broken down, superior to B- TiO_2 by 16% and W- TiO_2 by 48%.

Mhalsekar, et al. [23] reported a successful preparation and characterization of MnTiO_2 /MWCNT sol-gel nanocomposites aiming to increase electron-hole pair separation and move the band edge towards longer wavelengths. The investigation intended to eliminate organic contaminants, to evaluate photocatalytic performance. Six percentage weight concentration of Mn/Ti/CNT nanocomposites showed

the most effectiveness in organic removal, with a degradation rate higher than 90%. Based on these findings, it appears that this chemical may be utilized for other industrial applications [23].

The difficulties in removing BTB from industrial wastewater shown in the literature review, highlight the need for advanced materials and methods to treat dye-polluted water effectively. The researchers in this experimental work developed TiO₂ nanoparticles and MnO₂ nanoparticles added to MWCNTs. The Mn/Ti/MWCNTs nanocomposite was synthesized, characterized, and then tested for removing BTB from industrial wastewater. Standardized characterization procedures were used, and measurements of the removal efficiency are presented in the work.

2. Materials and Methods

2.1. Materials

All the chemicals used in this study were of analytical grade and did not require further purification. Ethanol (EtOH) had 97% purity; hydrochloric acid (HCl) had 99.9% purity; potassium permanganate (KMnO₄) had 99.9% purity; titanium tetrachloride (TiCl₄) had 99.9% purity; sulfuric acid (H₂SO₄), with 95% purity, was procured from BDH company. Analytical-grade hydrogen peroxide (H₂O₂) with 50% purity and bromothymol blue dye were purchased from Merck Company.

2.2. Adsorbent Synthesis

2.2.1. Functionalization of Multi-Walled Carbon Nanotubes

Acid-mediated functionalization was carried out by adding a 3:1(v/v) mixture of concentrated sulfuric acid (H₂SO₄) and nitric acid (HNO₃) and 0.3 g of synthesized MWCNTs to introduce carboxylate groups onto the MWCNT surface. The suspension was ultrasonically stirred for 1 hour to ensure uniform dispersion and functionalization. Subsequently, the mixture was transferred to a round-bottom flask and refluxed for 4 hours to complete the carboxylation process [17].

2.2.2. Synthesis of Manganese Dioxide and Titanium Dioxide Nanoparticles

The hydrothermal method was used to synthesize manganese dioxide (MnO₂) nano-powder. 4.1 g of Potassium permanganate (KMnO₄), was dissolved in 70 mL of deionized water under mechanical stirring for 10 minutes. The solution was filtered, and then 3.4 mL concentrated hydrochloric acid (HCl) was added dropwise while stirring continuously. The resulting precursor solution was transferred to a Teflon-lined stainless steel autoclave and heated at 200 °C for six hours in a muffle furnace. After cooling at room temperature, the produced Mn (OH)₄ intermediate was aged for 24 hours, washed five times with distilled water in a centrifuged unit, and then twice with ethanol. Finally, it was calcined and purified at 250 °C for 6 hours to produce the crystalline MnO₂ nanoparticles [14].

The TiO₂ nanoparticles were prepared by the hydrothermal method. Five ml of titanium tetrachloride (TiCl₄) was slowly dissolved in 75 ml of absolute ethanol in a cold environment to reduce the effect of the exothermal reaction. Afterwards, 25 ml of distilled water was added to the solution with stirring. Then, the TiO₂ nanoparticles were dried for 9 hours at 200 °C to sterilize the solution. Finally, at 250 °C, the TiO₂ nanoparticles were annealed and precipitated for 6 hours [11].

2.2.3. Synthesis of MnO₂ -TiO₂ Nanocomposite

The MnO₂-TiO₂ nanocomposite was prepared by mixing MnO₂ and TiO₂ nanoparticles in a 1:1 molar ratio. Next, absolute ethanol was added to the mixture while stirring to homogenize for six hours. To enhance the formation of composite and crystalline products, the suspension was annealed at 250°C for two hours [14].

2.2.4. Final Sorbent Synthesis of MnO₂:TiO₂/MWCNTs Nanocomposite (Mn/Ti/MWCNTs)

The final Mn/Ti/MWCNTs nanocomposite was prepared by mixing 10 wt% of the Mn/Ti/MWCNTs nanocomposite and 0.2 g of carbon nanotube in 250 ml of ethanol. The mixture was autoclaved in Teflon-coated stainless steel and dried at 250 °C for two hours. Subsequently, the final Mn/Ti/MWCNTs nanocomposite was separated and dried again in an oven at 100 °C.

2.3. BTB Dye Sorption Experiment

The BTB removal experiment was conducted in a batch reactor. 25 ml of BTB with a concentration of 20 ppm was mixed with 0.01 g of Mn/Ti/MWCNTs at a stirring speed of 250 rpm for two hours with a pH of seven. The dye-adsorbing nanocomposite was centrifuged for 20 minutes at 300 rpm. The concentration of dye pollutants was analyzed using a UV–visible spectrometer. The equilibrium adsorption of the BTB was determined by measuring the absorbance at 615 nm. To distinguish between the two concentrations and perform the calibration using distilled water as the solvent, all measurements were repeated three times [24]. The percentage of BTB removal efficiency (RE %) was calculated using Eq. (4) [25, 26].

$$RE\% = [(C_o - C_t) / C_o] \times 100 \quad (1)$$

where RE% is the percentage removal efficiency of the sorbent, C_o is the initial concentration of the pollutant in mg/L, and C_t is the final pollutant concentration in mg/L.

Using Eq. (2), the capacity of BTB adsorbed at equilibrium, q_e , was determined as:

$$q_e = [(C_o - C_t) / W] \times V \quad (2)$$

where q_e is the sorbent saturated with solute at equilibrium, W is the sorbent weight [gm], and V is the volume of pollutant sample in L. The quantity of BTB that can be adsorbed at equilibrium, q_e , was determined using Eq. (2) [1].

3. Results and Discussions

3.1. X-Ray Diffraction Results (XRD)

The XRD method was used to characterize MWCNTs and Mn/Ti/MWCNTs nanocomposites. The diffraction peaks were observed at 26° and 44° 2θ for MWCNTs corresponding to the 002 and 100 reflections of graphite, respectively [9]. The XRD pattern of the nanocomposite showed major diffraction peaks at 2θ values of 14.06° , 16.9° , and 28.8° , which are indexed to the (001), (110), and (310) planes, respectively, which is characteristic of the alpha phase of MnO_2 . Furthermore, the diffraction peaks at 25.5° and 37.9° correspond to the 101 and 004 planes associated with the tetragonal structure of the anatase phase of TiO_2 nanoparticles. The XRD phenomena related to oxidized MWCNTs and Mn/Ti/MWCNTs nanocomposites show that acid treatment and metal oxide deposition do not affect the graphite layer of graphene inside MWCNTs. The sharp diffraction peaks at 25.3° and 43.7° 2θ that appear for the metal oxide-modified MWCNTs correspond to the 002 and 100 planes reflections of graphite. After modification with Mn/Ti/MWCNTs nanocomposites, the diffraction peaks corresponding to MnO_2 and TiO_2 nanoparticles were not distinctly observable, likely due to their low concentration. Nonetheless, the crystalline structures of TiO_2 and MnO_2 nanoparticles were identified in the samples, as illustrated in Fig. 1. Notably, the prominent peaks at 25.5° and 37.9° corresponding to the planes 101 and 004 were observed for TiO_2 nanoparticles, while individual peaks attributable to MnO_2 nanoparticles and the related findings align with previous results in the literature [8].

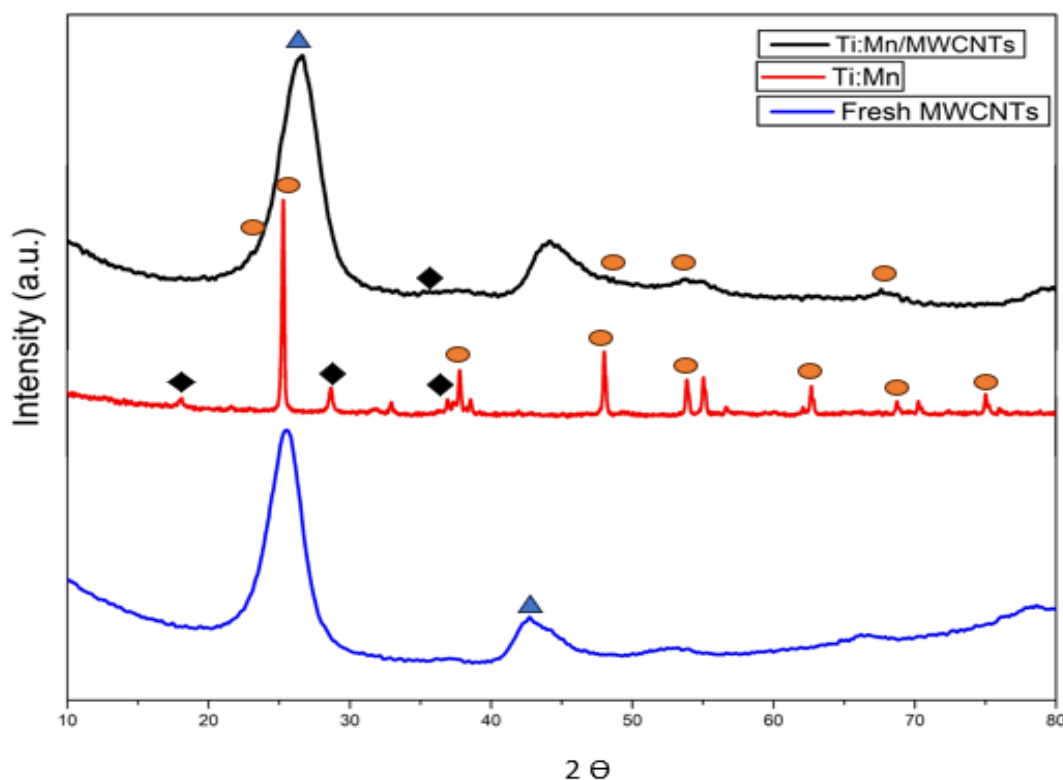


Figure 1. XRD results of oxidized MWCNTs, $\text{MnO}_2\text{:TiO}_2$, and Mn/Ti/MWCNTs nanocomposites.

3.2. Field Emission Scanning Electron Microscopy Results (FESEM)

Figure 2 depicts images of these metal oxide nanocomposites, the MWCNTs and modified MWCNTs obtained from FESEM. A representation of the surface morphology of the $\text{MnO}_2\text{:TiO}_2$ nanocomposite is shown in Fig. 2a, indicating the presence of nanocomposites, some of which exhibit a cubic morphology with an average size of approximately 50 nm. Figure 2b provides images of both the MWCNTs and modified MWCNTs on FESEM scale; the cylindrical structure of the MWCNTs is visible, allowing the dimensions of the carbon nanotubes to be determined. It is observed that these carbon nanotubes are in the nano-scale range. Moreover, the carbon nanotubes also appear structurally sound, as their morphology does not significantly change during the thermal treatment process in the presence of the nanoparticles, as shown in Figure. 2b. After adsorption, FESEM images showed changes in the shape of the nanocomposite surface, indicating that the adsorbent blocked some pores and a dye layer had formed. The initially rough and porous surface became smoother. BTB molecules are adsorbed onto the surface and may even be inside the pore structures of the composite [18].

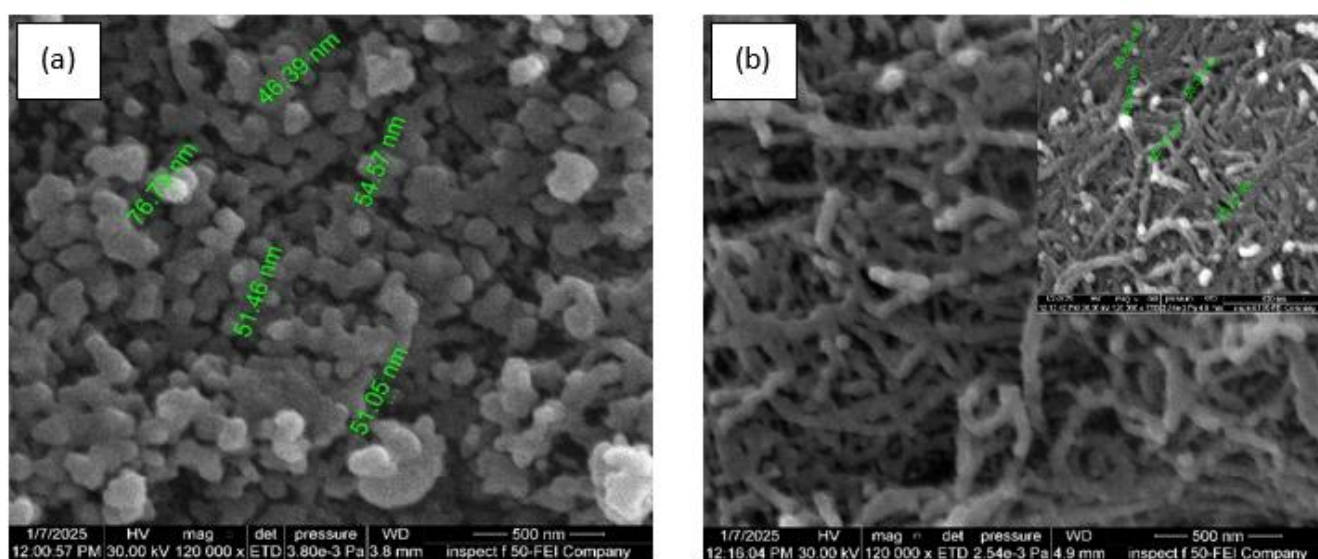


Figure 2. FESEM records for (a). $\text{MnO}_2\text{:TiO}_2$ nanocomposite, (b). Fresh MWCNTs and Mn/Ti/MWCNTs nanocomposite.

3.3. Electron Dispersion X-ray Results

The elemental composition of carbon, titanium, and manganese on sample surfaces was determined using energy-dispersive X-ray spectroscopy (EDX) analysis. Figures 3 and 4 show the average elemental compositions of the near-surface layers of the samples. The data in Table 1 and Figure 3 show the compositional data of the $\text{MnO}_2\text{:TiO}_2$ nanocomposites, while Table 1 presents the quantitative data. EDX analysis of the Mn/Ti/MWCNTs nanocomposites confirms the theoretical abundances used in their preparation. Therefore, the results support the claim that the composites of metal oxides were successfully coated onto the surface of every carbon nanotube, as shown in Fig. 4. After adsorption, EDX analysis

revealed a significant increase in carbon (C) and sulfur (S) signals, consistent with the BTB structure. The results prove that the dye molecules are physically sticking to the surface of the adsorbent. The titanium and manganese signals remained unchanged, which indicates the presence of a strong nanocomposite structure after adsorption .

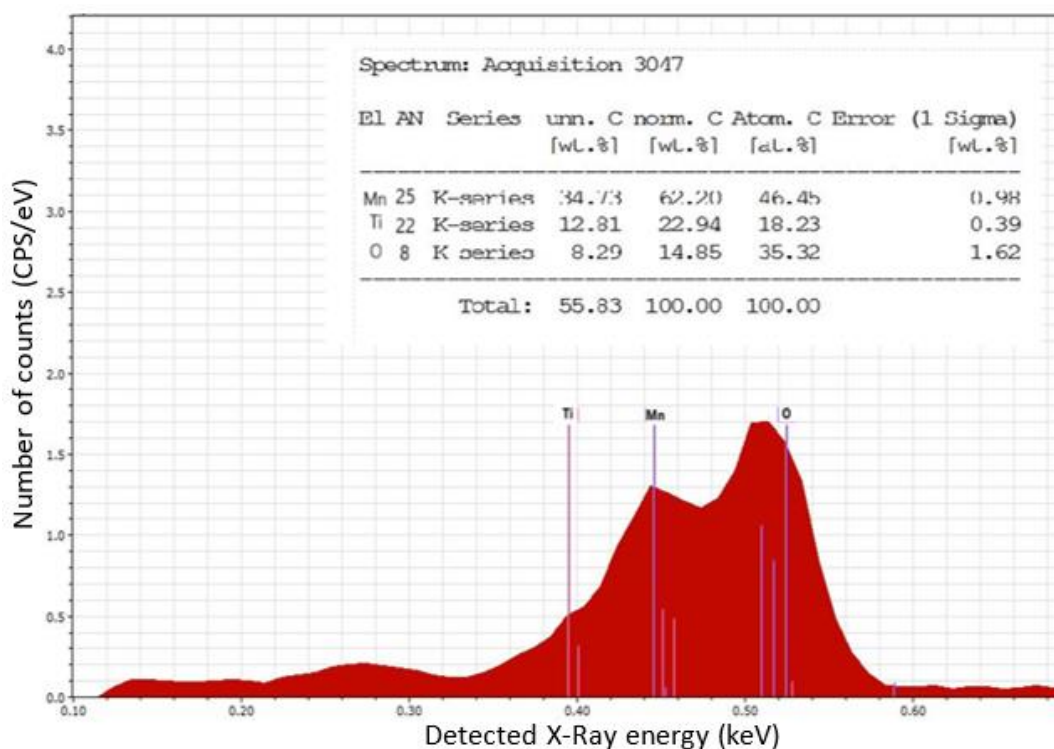


Figure 3. EDX results for TiO₂:MnO₂ nanocomposites.

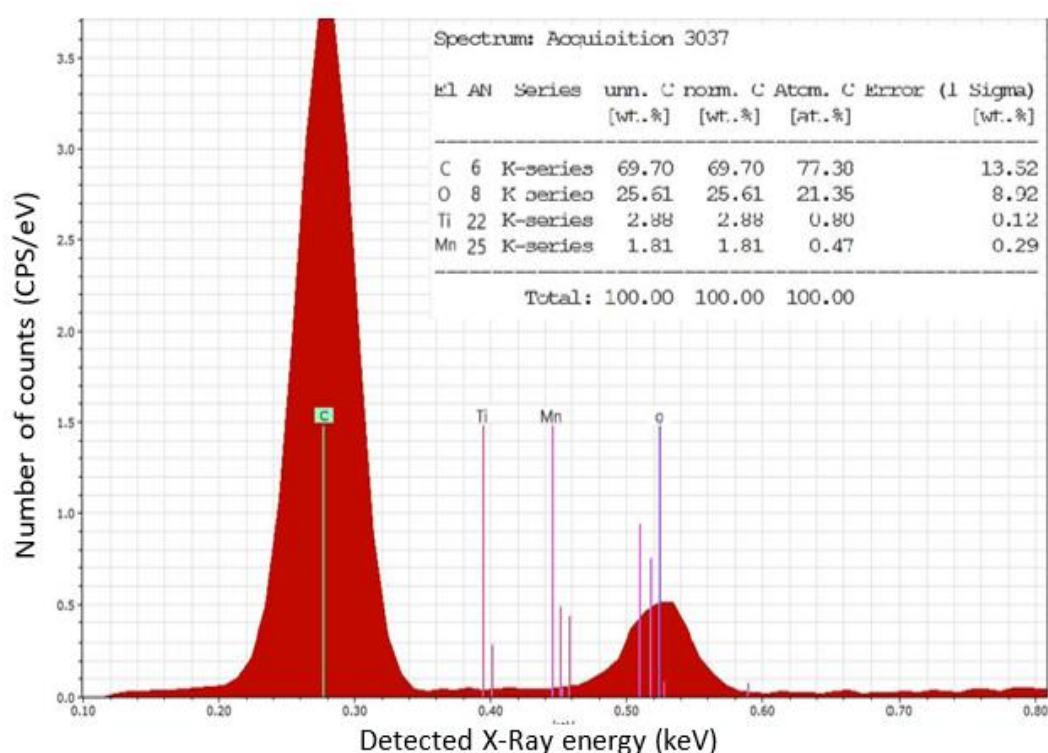


Figure 4. EDX results for Mn/Ti/MWCNTs nanocomposites.

Table1. EDX results for TiO₂:MnO₂ nanocomposites

Samples	wt% (TiO ₂ :MnO ₂) 1:1	
	Theoretical	Experimental
Mn	16.666	46.185
Ti	16.666	19.356
O	66.666	34.485
Total %	99.998%	99.999%

3.4. BET Surface Area of Adsorbents

The results for surface area from the BET analysis are presented in Table 2 for MWCNTs, acid-treated MWCNTs, and Mn/Ti/MWCNTs nanocomposites. The weight loss occurred through the outgassing process before the BET analysis, and the prominent reduction in weight for Mn/Ti/MWCNTs nanocomposites was observed at 7.2 wt%. The weight loss suggests that some moisture and acid condensate remained within the samples after acid treatment and metal oxide deposition despite drying at 100 °C. The addition of strong acids to MWCNTs led to the appearance of defect sites on nanocomposite surfaces, splitting the tube and forming a new opening that increased the total volume of material [1].

As indicated in Table 2, the acid treatment significantly reduced the micropore volume. The introduction of functional groups, such as carboxyl during acid treatment, may have blocked smaller pore openings, resulting in a slight decrease in the specific surface area (SBET) from 148 to 139 m²/g. Conversely, the MWCNTs doped with Mn₂-TiO₂ nanocomposites exhibited a lower surface area of 123 m²/g.

Table 2. BET surface area analysis, weight loss during outgassing, total and micropore surface area of fresh, oxidized MWCNTs and Mn/Ti/MWCNTs

Samples	Weight loss (wt.%)	S _{BET} (m ² /g)	S _{micro} (m ² /g)	V _{1.7-300} (cm ³ /g)	V _{micro} (cm ³ /g)	D _{av} (nm)
MWCNTs	0.11	148	24.4	0.7178	0.0109	15.8
ox/MWCNTs	6.9	139	12.3	1.0173	0.0048	27.1
Mn/Ti/MWCNTs	7.2	123	9.1	0.7004	0	22.6

3.5. Fourier Transform Infrared (FTIR) Before and After Adsorption

The FTIR method conducted before and after BTB adsorption shows that the functional groups on the surface of Mn/Ti/MWCNTs nanocomposites are actively involved in binding to dyes. Spectra changes and the appearance of new bands indicate that physicochemical interactions occur during adsorption, which are critical to the composite's effectiveness in cleaning. FTIR spectroscopy was used to investigate how BTB and the Mn/Ti/MWCNTs nanocomposite interact with each other [8]. The spectra wavelengths range between 4000 to 400 cm⁻¹, as shown in Figure 5.

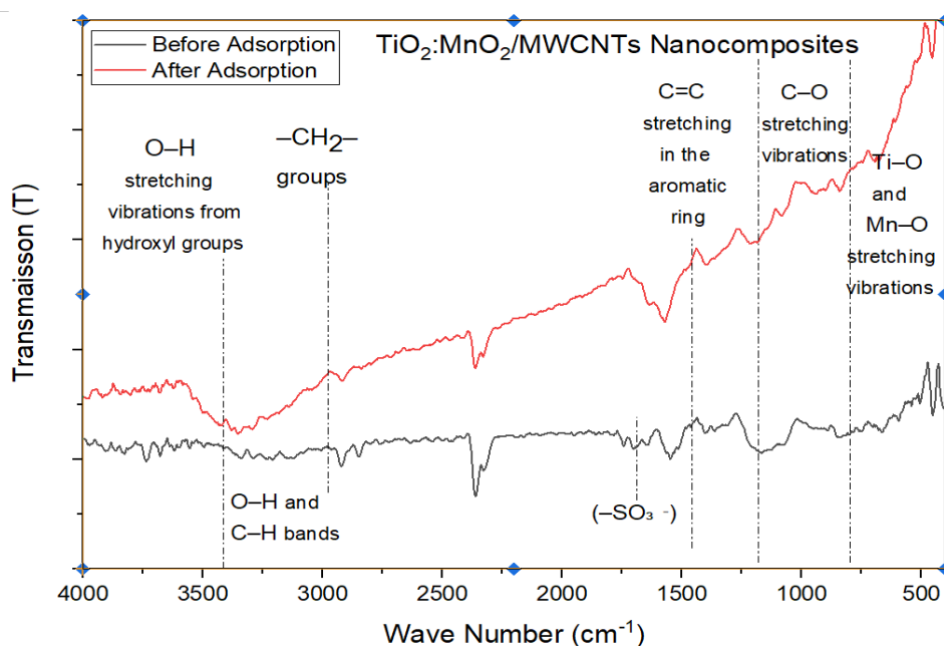


Figure 5. FTIR spectra of the new Mn/Ti/MWCNTs nanocomposite before and after adsorption.

3.5.1. Before Adsorption (Black Spectrum)

The spectrum displays distinct peaks for the Mn/Ti/MWCNTs nanocomposite, which means that the following functional groups and bonds are present:

~3420 cm^{-1} : A wide band caused by O–H stretching vibrations from hydroxyl groups (water or hydroxyl groups that are on the surface) .[7]

~3420 cm^{-1} : A wide band caused by O–H stretching vibrations from hydroxyl groups (water or hydroxyl groups that are on the surface) .

~2920 cm^{-1} : C–H stretching vibrations from aliphatic –CH₂– groups, which could come from leftover organic molecules or the carbon framework of MWCNTs.

~1620 cm^{-1} : The vibrations of water molecules' O–H bonds bending.

~1380–1450 cm^{-1} : C=C stretching in the aromatic ring, which shows the presence of the graphitic MWCNTs structure.

~1100–1000 cm^{-1} : C–O stretching vibrations show that oxygen-containing groups were added during oxidation or synthesis.

~500–700 cm^{-1} : stretching vibrations of Ti–O and Mn–O, which are typical of metal oxides (TiO₂ and MnO₂ nanoparticles).

3.5.2. After Adsorption (Red Spectrum)

The FTIR spectrum shows several changes after BTB adsorption, which means that the dye molecules are interacting with the nanocomposite surface. The O–H and C–H bands (~3420 and ~2920 cm^{-1}) become less intense or wider, which could indicate that hydrogen bonding or interactions with the BTB functional

groups are occurring. New peaks or stronger signals in the $1500\text{--}1600\text{ cm}^{-1}$ range could be caused by vibrations of aromatic rings or stretching of sulfonate groups ($-\text{SO}_3^-$) from BTB. This confirms that the dye has been absorbed. Small changes in the peak positions indicate that the dye is interacting with the surface or altering it. The fact that transmittance is higher in lower wave frequency regions suggests that organic dye molecules may be covering the surface of metal oxides, hiding the metal–oxygen peaks [7].

3.6. Adsorption Results

The present study investigates the adsorption characteristics of MWCNTs alone, Mn_2TiO_2 nanocomposites, and Mn/Ti/MWCNTs nanocomposites for 120 minutes using a UV-V Spectrophotometer. Figures 6 and Figure 7 show the removal efficiency of BTB dye from industrial wastewater by Mn/Ti/MWCNTs nanocomposites, reaching up to 78%. Specifically, the BTB dye dosage was at its minimum following the adsorption experiments utilizing the Mn/Ti/MWCNTs nanocomposite. The results of BTB dye removal from wastewater showed significant efficiency after one hour. Also, the results indicated a high adsorption removal capacity of 46 mg/g for Mn/Ti/MWCNTs nanocomposite, which was higher than the MWCNTs alone and MnTiO_3 nanocomposites. Integration of $\text{MnO}_2\text{:TiO}_2$ nanocomposite with MWCNTs was proven by EDX and SEM analysis, which improved the bonding with BTB dyes in industrial wastewater. The development of Mn/Ti/MWCNTs nanocomposite could be economically suitable for removing the dye from industrial wastewater. The synergistic effects of MnO_2 and TiO_2 nanoparticles and fictionalization of MWCNTs led to active binding sites and increased surface area [7].

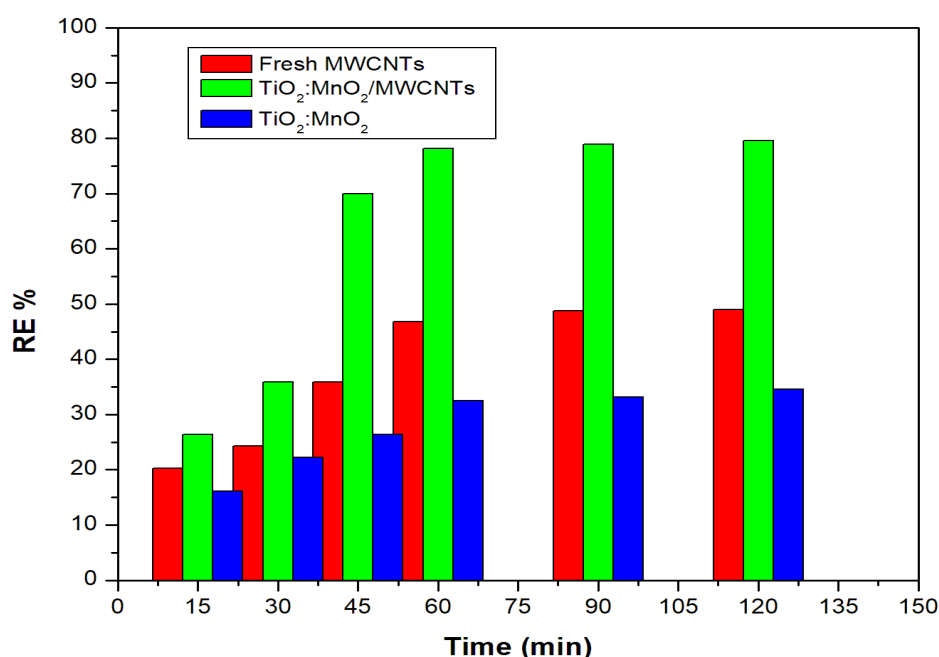


Figure 6. The removal percentage of BTB dye using MWCNTs, $\text{TiO}_2\text{:MnO}_2$, and Mn/Ti/MWCNTs nano absorbent with time.

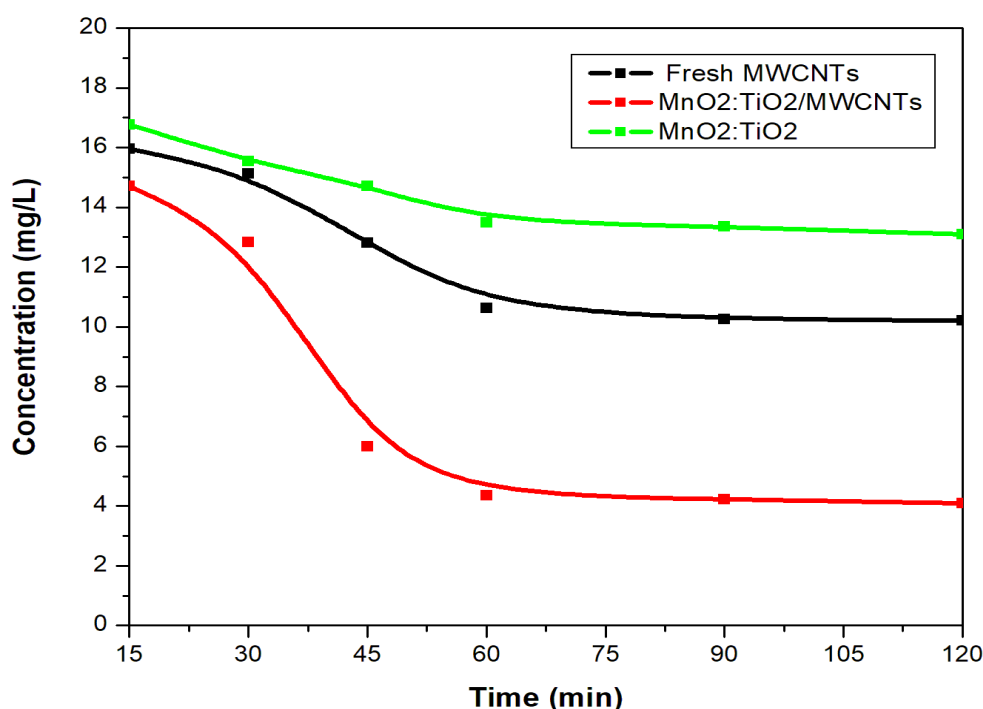


Figure 7. Present the effect of nanoparticles concentration on BTB removal.

4. Conclusions

This study examined the performance of the MWCNTs, Mn₂TiO₂ nanocomposites, and Mn/Ti/MWCNTs nanocomposites for removing BTB dye from industrial wastewater. The results show the significant and high removal efficiency of BTB dye by the Mn/Ti/MWCNTs nanocomposite. The Mn/Ti/MWCNTs nanocomposite exhibited an adsorption capacity of 46 mg/g with a 78% removal rate, higher than with MWCNTs and Mn₂TiO₂ nanocomposite. The FTIR analysis conducted before and after BTB adsorption confirmed that functional groups of the Mn/Ti/ MWCNTs nanocomposite surface are actively involved in binding with the dye. The spectral shifts observed new bands, which indicate physicochemical interactions occurred in the adsorption process. These new bands enhanced the activity of the nanocomposite in removing the dye from industrial wastewater. The BTB dye was adsorbed onto Mn/Ti/MWCNTs nanocomposites, as confirmed by the data of FTIR. These results highlight the importance of Mn/Ti/MWCNTs nanocomposites in treating industrial wastewater contaminated by dye.

Acknowledgement: The authors express their sincere gratitude to Al-Mustaqbal University, Iraq, the University of Technology, Baghdad, Iraq, and Dr. Thamer A. Abdullah for their valuable support and insightful contributions..

Data Availability Statement: Data are available from the corresponding author upon reasonable request.

Funding: The authors declare that no funding was received for this research.

Declaration of Competing Interest: The authors declare they have no known competing interests.

Research Involving Human/Animal Participants: This article does not involve any studies conducted by authors on animals or human participants.

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