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Article

Preparation and Diagnosis of Nano-Titanium Dioxide and Its Use for Industrial Water Treatment

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Abstract: Titanium dioxide (TiO_2) nanoparticles were successfully synthesized, and their properties were accurately characterized using a range of advanced analytical techniques. These characterization methods included scanning electron microscopy (SEM), transmission electron microscopy (TEM), energy-dispersive X-ray spectroscopy (EDX), and X-ray diffraction (XRD). The synthesized TiO_2 nanoparticles were then effectively used as adsorbents to remove Eriochrome Black-T (EBT), a common contaminant, from industrial effluent. The study of the adsorption process revealed the optimal conditions for efficient removal of the dye. It was found that maximum adsorption efficiency could be achieved within a relatively short contact time of 20 minutes, using a precise dose of 0.05 grams of TiO_2 nanoparticles. Thermodynamic analysis of dye adsorption revealed a clear relationship between temperature and adsorption performance. Specifically, an increase in temperature led to a decrease in overall adsorption efficiency. This observation is of great significance as it strongly suggests that the adsorption process is exothermic in nature. Furthermore, as adsorption is a spontaneous process, the decrease in adsorption with rising temperature also implies a reduction in randomness or disorder within the system during the adsorption process. The study has unequivocally demonstrated the great potential of these TiO_2 nanomaterials for use in wastewater treatment. Their effectiveness stems from several inherent key properties, notably their high surface activity, which provides numerous sites for interaction with pollutants, and their well-documented photocatalytic properties. Taken together, these properties enable TiO_2 nanoparticles to effectively remove hazardous substances, even when these pollutants are present in wastewater at very low concentrations. This highlights their promising potential as a sustainable and effective solution for environmental remediation.

Keywords: adsorption, nano titanium dioxide, azo dyes, industrial water.

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1. Introduction

Titanium dioxide nanoparticles are considered among the most important nanostructured materials because of their high stability, low toxicity, strong photocatalytic activity, and relatively low cost. These characteristics have encouraged their application in environmental remediation, antimicrobial systems, and water treatment technologies. As antimicrobial materials, due to their low cost, reactivity, stability, durability, and bioactivity, due to the characteristics of these particles such as high surface area, corrosion resistance, high brightness and refraction, and resistance to color change, and others [1, 2]. Nano titanium dioxide is widely applied in different industrial and environmental fields, such as medical products, paints, and cosmetics, as a disinfectant on surfaces, and as a dye in paints, printing inks, crayons, plastics, paper, medicines, toothpaste, foods, and others. It is approved for use in food contact materials by the US Food and Drug Administration

(FDA) because it is considered a non-toxic substance [2, 3]. One of the most significant characteristics of TiO_2 nanoparticles is their photocatalytic activity, which contributes effectively to microbial elimination and environmental purification that these particles use to disinfect surfaces from microorganisms, as the process begins with the absorption of light by titanium dioxide nanoparticles, which excites an electron from the valence band leaving behind a positive charge, Both the negatively charged electron and the positive charge work in the redox reactions that take place with the species that are adsorbed on the surface of titanium dioxide nanoparticles such as water or organic hydroxyl ions (OH-), which leads to the formation of reactive oxygen species (ROS), which is the force The driving force behind antimicrobial activity [4, 5].

Water contamination can occur through chemical, biological, and physical pollutants that negatively affect both humans and the environment [6]. Throwing solid wastes into water, in general, detergents, and organic compounds are among the main causes of water pollution [7] and dyes are considered the most polluting organic compounds of water sources, because of their wide uses in the textile industries and in color photography, it is used in the food and medical industries [8] and in many other fields, where it is produced in various forms. Many industrial dyes may produce toxic or carcinogenic compounds after decomposition, which increases their environmental risk. When they decompose, such as benzene and amine groups, which led to interest in studying the environmental behavior of these substances effectively after confirming the toxicity and danger of these substances [9].

Most of the dyes are widely used to color cotton textiles; however, the large number of unstable dyes causes color in the sewage water, which is not acceptable to the environment [10] as it negatively affects most of the ecosystems because the water from the textile industries contains the residues of azo dye [11]. Thus, it requires the treatment and removal of toxic dyes from colored wastewater [12], so various associations or organizations for the protection of the environment have worked to address these problems [13] for the purpose of raising the water level to the desired level [14]. A surface phenomenon known as adsorption occurs when molecules or ions from liquid or gaseous phases build up on a solid material's surface as a result of chemical or physical interactions. Because of its great effectiveness in eliminating impurities, particularly at low concentrations, this method is frequently employed in wastewater treatment. involves the physical or chemical interactions of a gaseous or liquid material on the surface of a solid substance [6, 15]. The substance that is adsorbed is referred to as an adsorbate, and the substance that is on the surface of the adsorption process is referred to as an adsorbent. Depending on the kind of contact, adsorption processes are often divided into two categories: chemical adsorption and physical adsorption [16]. Due to its practical significance and wide range of environmental applications, adsorption has been thoroughly studied for many years. Since the solid and liquid states of matter naturally have specific surfaces in space, the surface contact areas that result in adsorption are (solid: solid), (solid: liquid), (solid: gas), (liquid: liquid), and (liquid: gas) [14]. This process has drawn the attention of many researchers in this field due to its necessity and wide range of applications [12]. One of the most effective strategies for eliminating low-concentration contaminants from wastewater that conventional biological procedures are unable to eliminate is adsorption. Because the molecules that undergo adsorption are constrained by their association with the adsorbing surface atoms and thus lose some of their degrees of freedom, adsorption results in the saturation of these fields of forces on the surface, which lowers the free energy of the surface on which the adsorption takes place. This is accompanied by a decrease in entropy. Remaining unsaturated force fields that prevent enough particles from coordinating or coming into contact with the surface, as is the case in the solid or liquid phase, are the source of this phenomenon. Numerous investigations are cited in the literature and research [17-19].

In this study, we used nano-titanium dioxide as an adsorbent for industrial water purification via adsorption.

Experimental part

titanium dioxide (Industrial grade), Sodium hydroxide (NaOH, ≥ 98%) was obtained from Merck (Germany), Hydrochloric acid (HCl, 37% analytical grade) was supplied by Sigma-Aldrich (USA), Eriochrome black T was purchased from Sigma-Aldrich.

Preparation of titanium dioxide nanoparticles [20]

10 g of industrial titanium dioxide was added to 50 ml of 10 M sodium hydroxide solution in a 250 ml flask. The prepared mixture was heated at 383.15 K for 35 h under intermittent stirring conditions and at intermittent intervals. Titanium dioxide nanoparticles were then obtained by filtering the precipitate and putting it in a drying oven set at 393.15 K for two hours after adding 10 ml of concentrated HCl gradually while stirring until the mixture achieved a pH of 7.

The structural, morphological, and optical properties of the prepared sample were investigated using various advanced techniques. X-ray diffraction (XRD) analysis was performed to determine the crystalline structure using a Bruker D8 Advance diffractometer (Bruker AXS, Karlsruhe, Germany) equipped with Cu-Kα radiation. The surface morphology and microstructure were examined via Scanning Electron Microscopy (SEM) using a JEOL JSM-IT200 microscope (JEOL, Tokyo, Japan). For detailed internal structure and particle-size determination, Transmission Electron Microscopy (TEM) imaging was performed on a JEOL JEM-2100 Plus system (JEOL, Tokyo, Japan) at an accelerating voltage of 200 kV. Additionally, the optical absorption and spectroscopic properties were recorded using a UV-Visible Spectrophotometer (Shimadzu UV-1900i, Kyoto, Japan) within the appropriate wavelength. **Standard curve of dye**

The maximum absorption wavelength of the dye solution was determined using a UV-Visible Spectrophotometer. To create the calibration curve, six diluted aqueous solutions ranging from 10 to 50 ppm were prepared in a volumetric flask containing 10 mL. The absorbance values of each of these diluted solutions were calculated using a spectrophotometer.

Adsorption experiments

The effect of titanium oxide dosage on the adsorption of Eriochrome black T dye was investigated using a constant dye concentration (30 ppm) in a 100 mL volume, and different weights of prepared titanium oxide (0.01, 0.03, 0.05 g). The samples were placed in a water bath at 25 °C for 20 minutes at a stirring speed of 100 revolutions per minute; the samples were then filtered, and the absorbance of the filtrate was measured.

To determine the optimum time for equilibrium for the adsorption system. The standard dye solution was diluted to a 30-ppm solution in a 100 ml volumetric flask. The solution was supplemented with 0.05 g of the produced nanomaterial. The vibrator was set to run at 100 cycles per minute while the solution was heated to 25°C. At various intervals of 5, 10, 15, 20, 25, and 30 minutes, the solution was filtered. A visible-ultraviolet spectrophotometer was used to record the filtrate's absorbance values at each of these designated times. The adsorption efficiency of the prepared material was evaluated by calculating the percentage removal of the dye solution. That represents the adsorption efficiency of titanium oxide prepared as an adsorbent surface, as it works to remove the dye from its aqueous solution, by applying equation (1), and depending on the highest removal percentage [17].

$$\%Removal = \frac{C_e - C_o}{C_o} \times 100\% \quad (1)$$

While the adsorption capacity of the prepared titanium oxide expresses the amount of the adsorbed substance, which is expressed by equation (2) [18].

$$qe = \frac{(C_e - C_o)V}{m} \quad (2)$$

where C_o is the dye concentration in the liquid phase in units (ppm), C_e is the dye concentration in the equilibrium solution in units (mg/l), V is the volume of the solution in liters, and m is the weight of titanium oxide in grams. TiO_2.

In addition, the effect of temperature was assessed using a solution containing 30 ppm of dye (100 ml), and 0.05 of titanium dioxide (TiO_2); the solution was incubated in a water bath at a rotate of 100 revolutions per minute for 20 minutes at the following temperatures: (283.15, 293.15, 298.15, 303.15 °K), the absorbance of the filtrate was measured for the thermodynamic study.

2. Results and Discussion

Diagnostics of TiO2 nanoparticles

The surface of the prepared titanium dioxide was characterized using X-ray diffraction (XRD) to determine the crystal shape of the prepared nanoparticles through Miller's coefficients, where the purity of the prepared titanium dioxide is determined, and the size of the nanoparticles was calculated using the Debye Scherrer Equation [21]. Figure 1a shows the X-ray diffraction (XRD) spectrum of the prepared titanium oxide. It was found that the particle size of the prepared titanium oxide nanoparticles (35.4-37-37.4) nm, and the average overall size of the nanoparticles was 36.4 nm, which showed a great match with X-ray diffraction. For the prepared Nano-titanium oxide.

EDX measurements of TiO2 nanoparticles showed that titanium dioxide gave three peaks of large, medium, and small intensity, and they appeared in different places, as shown in Figure 1b.

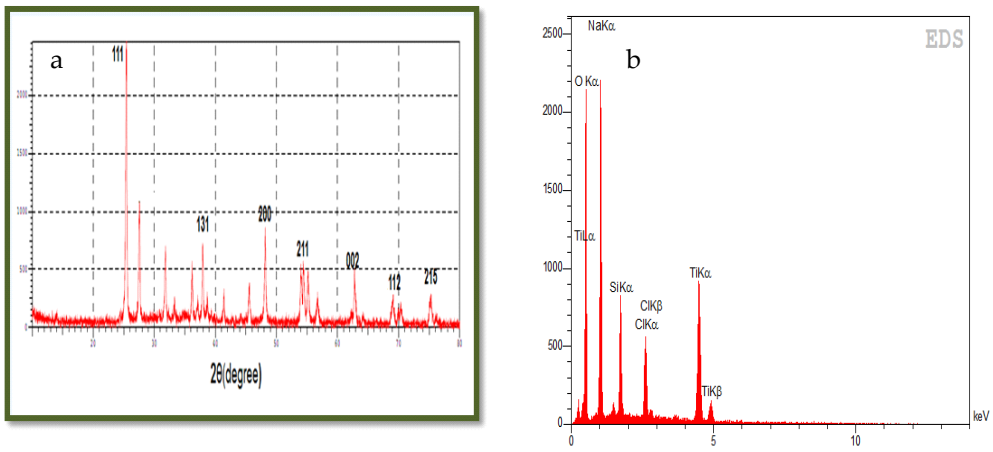


Figure 1. a-XRD analysis, b-EDX of nanoscale TiO2

As shown in Figure 2, it is clear that the scanning electron microscope (SEM) measurement of SEM of TiO2 nanoparticles at different magnification powers at micro and nano scales shows that the material is formed in the form of small particles and their approximate diameters are in the range (43-333 nm).

The results shown in Figure 3, from a transmission electron microscope at different magnifications, indicate that the prepared titanium dioxide particles are nanoscale and in the form of spherical aggregates.

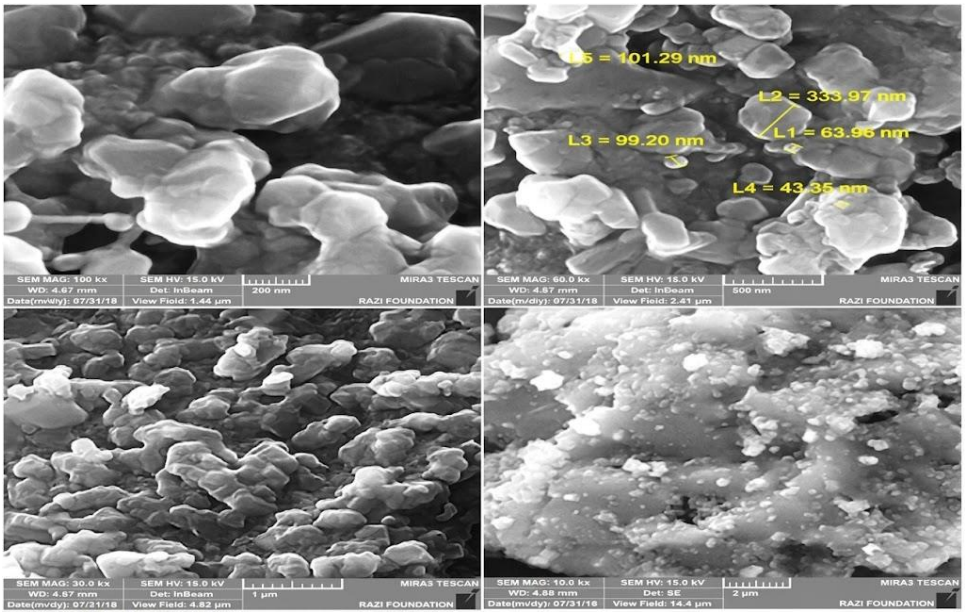


Figure 2. Images of the scanning electron microscope of nanoscale titanium oxide TiO2

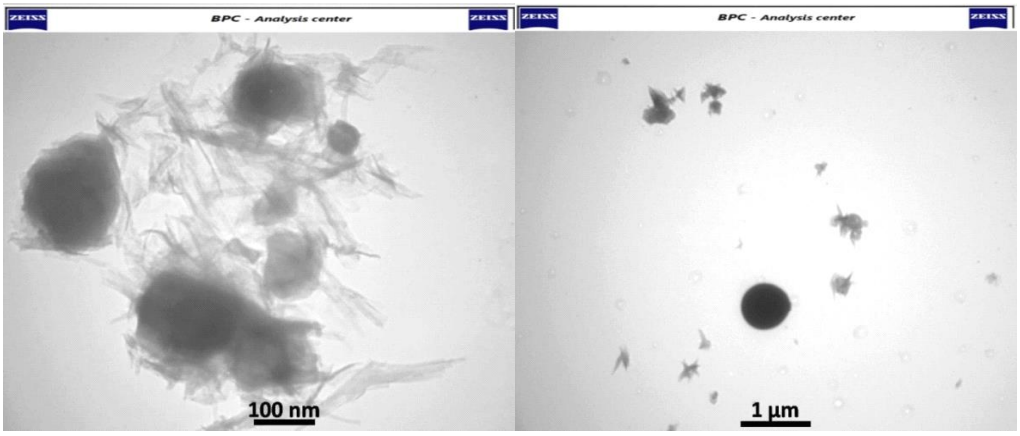


Figure 3. Transmission electron microscopy of titanium oxide nanoparticles, TiO2.

Adsorption study of the dye (EB-T) on TiO_2 nanoparticles

The dark violet-black Eriochrome black T dye exhibited maximum absorption at a wavelength of 530 nm, and a calibration curve was plotted over a range of diluted concentrations (10–50 ppm), showing a linear relationship, indicating that it obeys Beer–Lambert's law [3].

The time required for the adsorption system to reach equilibrium was determined by spectroscopically monitoring the change in the adsorbent concentration (C_e) with the contact time between the dye solution and the adsorbing surface until equilibrium was reached. The study was carried out using fixed dye (EBT) concentrations of 30 ppm, the optimum surface weight of 0.05 g, a temperature of 25º C, and a dye wavelength of 530 nm, as indicated in Table 1.

Table 1. Determination of equilibrium time and adsorption efficiency

Time (minute)	C_e (mg/L)	%Adsorption
5	15.69	52.30
10	17.97	59.91
15	20.25	67.50
20	24.00	80.10
25	24.00	80.10
30	23.12	77.08

A significant reduction in dye (EBT) absorbance was observed after the adsorption process compared with the initial solution. This is because the number of active sites makes the adsorbent surface highly efficient. As the concentration of the adsorbent material increases, the unoccupied active sites become saturated with the material, which is consistent with the kinetic theory of adsorption. As a result, the adsorption process gradually slows down over time [22]. According to the results, the adsorption process reaches the equilibrium state in 20 to 25 minutes, which results in the highest adsorption efficiency. At the equilibrium state, we observe that an additional increase in the equilibrium time did not improve the dye adsorption process because there are no available sites for adsorption, which causes the particles to return to the solution [18], due to the occurrence of the process of desorption, where the unstable particles gradually separate from the surface and an increase in absorbance values has been observed, which results in a decrease in the adsorption efficiency. The investigation of the impact of the adsorbent weight is presented in Table 2, which also provides an estimate of the adsorption effectiveness and the minimal weight needed to remove the greatest quantity of dye [23].

Table 2. Effect of the weight of the prepared activated charcoal on the adsorption efficiency.

Weight (g)	C_e (mg/L)	%Adsorption
0.01	**16.08**	53.60
0.02	**20.10**	67.01
0.05	**6.00**	80.10

The results showed that (0.05 g) represents the value of the weight of the adsorbent surface, which gave its capacity to absorb the largest amount of dye, at which most of the adsorption sites are saturated. It was observed that as the adsorbing weight increased, the percentage of dye removal increased, due to the increase in the adsorbent surface area of the dye, which gives a greater number of effective sites for dye adsorption [18].

The impact of dye adsorption on the surface of the generated titanium oxide was investigated using a temperature range of 283.15, 293.15, 298.15, and 303.15 °K. The weight of the adsorbent (0.05 g), constant concentration (30 ppm), and equilibrium time (20 min) for the dye in a water bath at a constant speed (100 rpm) are shown in Table 3.

Table 3. Effect of temperature on adsorption efficiency.

T(K)	Q_e(mg/g)	Adsorption%
283.15	**5.31**	82.30
293.15	**6.00**	80.10
298.15	**6.15**	79.53
303.15	**7.20**	76.09

The obtained results indicated that the adsorption behavior was exothermic in nature, as adsorption efficiency decreased with increasing temperature of the adsorbed dye on the surface of the prepared activated charcoal, and this corresponds to the thermodynamic requirements of the adsorption process, and as indicated by the physical nature of the adsorption system as the forces that bind Between the surface of the adsorbent and the dye are weak physical forces, As the temperature increases, the kinetic energy of the dye molecules adsorbed on the adsorbing surface increases, which leads to the destruction of the bonding forces (Van der Waals) between them, which helps their separation from the adsorbing surface and their return inside the solution, and thus no diffusion occurs within the surface pores [23, 24].

Temperature-dependent experiments were conducted to evaluate the thermodynamic parameters governing the adsorption system, in knowing the nature of the adsorption system in terms of its direction of flow, and the nature of the forces controlling it at equilibrium. The values of the equilibrium constant for the adsorption process (Keq) were calculated at different temperatures, upon reaching the equilibrium state. Free energy for the adsorption process can be calculated using Gibbs' equation ($\Delta G^o = -RT \ln Keq$).

The adsorption process occurs spontaneously when the adsorbent material's degrees of freedom diminish, which is thermodynamically represented as a drop in entropy (ΔS^o). As a result, this procedure was carried out under isothermal conditions using the Gibbs equation ($\Delta G^o = \Delta H^o - T\Delta S^o$).

Standard enthalpy is ΔH^o calculated using the Van't Hoff Equation ($lnK_{eq} = -\frac{\Delta H}{RT}+C$) to determine whether the adsorption processes were endothermic or exothermic, as suggested by some studies [17, 19], as shown in Table 4.

Table 4. Temperature's impact on the adsorption system's equilibrium constant.

T (K)	1/T	C_e (mg/L)	%adsorption	K_{eq}	$\ln K_{eq}$
283.15	0.0035	5.31	82.30	4.64	1.53
293.15	0.0034	6.00	80.10	4.00	1.38
298.15	0.0032	6.15	79.53	3.87	1.35
203.15	0.0031	7.20	76.09	3.16	1.15

As shown in Table 5, the adsorption process happens spontaneously through the values of ΔG° with a negative charge. Because the forces driving the adsorption process are weak, the negative value of ΔH^o confirms that the adsorption process was exothermic, releasing heat and proceeding through physical interactions of pigments on the surface [25, 26].

The results show that the negative ΔS^o values reduce the randomness of the studied system due to the loss of molecules after adsorption. This is consistent with a previous study [17,18] that examined thermodynamic functions at the ideal conditions for adsorption.

Table 5. Thermodynamic functions for the adsorption of EBT dye on TiO_2 prepared

T(°K)	ΔH^o (J. mole^{-1})	ΔG^o (J. mole^{-1})	ΔS^o (J. mole^{-1}. K^{-1})
283.15		-3601.78	-10.47
293.15		-3363.40	-8.15
303.15	-6568.06	-3402.52	-10.44
313.15		-2994.05	-11.78

3. Conclusion

The present study confirmed the successful synthesis of TiO_2 nanoparticles with nanoscale dimensions using several characterization techniques. The prepared nanoparticles exhibited efficient adsorption performance toward the removal of Eriochrome Black-T dye from industrial wastewater. The adsorption process was influenced by contact time, adsorbent dosage, and temperature. Thermodynamic evaluation indicated that the adsorption process was spontaneous and exothermic in nature. Overall, nano-TiO_2 can be considered a promising material for industrial wastewater treatment applications because of its enhanced surface properties and adsorption capability. (TiO_2) and confirmed its nanoscale properties using XRD, SEM, TEM, and EDX techniques. The prepared nanoparticles showed high efficiency in

removing Eriochrome Black-T dye from industrial wastewater through the adsorption process. The results indicated that adsorption equilibrium was reached within 20 minutes using 0.05 g of TiO_2 nanoparticles, with high dye removal efficiency. Increasing the adsorbent weight enhanced the adsorption process, while increasing the temperature reduced the adsorption efficiency, indicating that the process was exothermic and mainly physical in nature. Thermodynamic parameters showed that the adsorption process was spontaneous and accompanied by a decrease in randomness. Overall, nano-TiO_2 proved to be an effective adsorbent material for industrial water treatment due to its high surface activity and unique nanoscale properties.

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