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# Improved production of titanate nanotubes by hydrothermal method for adsorption of organic dyes

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## Abstract

**Background:** Increasing the yield of nanomaterials using the same reactor size and fixing most of the reactants and conditions will greatly improve the production process by saving time, energy and efforts. Titanate nanotubes are mainly prepared by hydrothermal process, in which  $\text{TiO}_2$  powder reacts with NaOH at certain conditions to form the desired nanotubes. It was reported that it is a must to use high concentrations of NaOH (10 N) to enable the tubular form formation, and the amount of NaOH from the stoichiometry point of view is much higher than that of  $\text{TiO}_2$ ; this means excess amounts of NaOH are not used and washed off. This work was designed to improve the production yield by making use of this excess amount of NaOH.

**Results:** More than 60 g of sodium titanate nanotubes was prepared using simple hydrothermal method. The prepared nanotubes were characterized by X-ray powder diffraction, high-resolution transmission electron microscopy, Fourier-transform infrared spectroscopy and BET surface area analysis. The adsorption capacity of these nanotubes was tested against three commonly used dyes: methyl orange, crystal violet and thymol blue. The samples showed great affinity toward crystal violet and lower activity toward methyl orange and thymol blue, where they achieved more than 90% removal efficiency under different experimental conditions.

**Conclusions:** Sodium titanate nanotubes were prepared in large amounts using modified hydrothermal method. The obtained nanotubes efficiently removed crystal violet from water. This improved synthesis of titanate nanotubes will reduce the total cost of nanomaterials production, and subsequently the treatment process, since titanate nanotubes are used in adsorption and photocatalysis processes.

**Keywords:** Titanate, Nanotubes, Water treatment, Organic dyes, Adsorbent, Large-scale production, Photocatalysis

## 1 Background

National security is closely related to the economic and social development, which is expected to increase the demand for freshwater for municipal and agriculture uses and electricity generation beyond existing levels [1]. One of the important solutions to this problem is to reuse and recycle raw water and wastewater [2]. Water is polluted

with organic dyes that generated from industrialization activities, although many dyes are non-toxic, but their presence in water can hinder the sunlight from reaching the aquatic life plants and animals [3]. Dyes can be removed from ground and wastewater by various methods such as chemical oxidation, membrane separation, electrochemical process, photocatalytic degradation and adsorption technique [4]

Adsorption still attracts researchers' attention because of its relatively low cost to other methods and its simplicity [5–13]. Many materials are used as adsorbents for organic dyes removal, such as activated carbon, zeolites,  $\text{ZnO}$ ,  $\text{TiO}_2$  and titanate nanotubes. Using adsorbents at

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the nanoscale level improves the process efficiency, since nanomaterials are smaller in size and larger in surface area when compared to conventional bulk materials [14].

Among these adsorbents, recently titanate nanostructures, especially nanotubes, have attracted more attention due to their high surface area, non-toxicity and their high adsorption and exchange capacity [14]. Titanate nanotubes are mainly prepared using the hydrothermal method [14–25]. But, unfortunately researchers did not pay attention to increase the yield of titanates in their studies; they tend to change the starting materials, time and temperature of reaction, the post-treatment solvents and acids [21, 26].

Herein, in this work, the hydrothermal method modified to increase the produced nanotubes by fixing all conditions except increasing the weight of the starting TiO<sub>2</sub> particles five times to get more than 60 gm of titanate nanotubes in one run instead of repeating the same experience using the same reactor size for five consecutive runs. To the best of our knowledge, this is the first report of its kind and will contribute significantly to the improvement in the wastewater treatment techniques by lowering the production cost of titanate nanotubes and by saving time and efforts.

## 2 Methods

### 2.1 Materials

Methyl orange (M.O), crystal violet (C.V) and thymol blue (T.B) stock solutions were prepared using distilled water. Nanosized titanium dioxide powder (anatase phase) was purchased from Loba Chemie Laboratory Reagents (India). Sodium hydroxide was purchased from El Nasr Company (Egypt).

### 2.2 Synthesis of sodium titanate nanotubes

The nanotubes were prepared according to our previously published work [17–26], but with modification to prepare more than 60 gm in one step. In detail, 50 gm of TiO<sub>2</sub> powder was added into 500 ml of 10 N NaOH, and then this mixture was subjected to vigorous magnetic stirring till milky white suspension is formed. The formed suspension was transferred into a 1000-ml-capacity Teflon-lined autoclave, and then this autoclave was placed in an oven at 160 °C for 23 h. After reaching the room temperature, the white powder was collected and washed specific times with distilled water to remove the unreacted amounts of NaOH. Finally, the powder was dried at 100 °C for 12 h.

### 2.3 Characterization and spectroscopy

The microstructure of the prepared nanotubes was studied using a high-resolution transmission electron microscope (HRTEM) (JEOL-JEM 2100, Japan) with

an acceleration voltage of 200 kV. XRD patterns were recorded on a PANalytical (Empyrean) X-ray diffraction using Cu K $\alpha$  radiation (wavelength 0.154 nm) at an accelerating voltage of 40 kV, current of 35 mA, scan angle 5°–80° range and scan step 0.02°. Fourier-transform infrared (FTIR) spectra were obtained using a spectrometer (Vertex 70 FT-IR) in the range of 4000 to 400 cm<sup>-1</sup>. Brunauer–Emmett–Teller (BET) surface area was measured by N<sub>2</sub> adsorption using Micromeritics TriStar II.

### 2.4 Adsorption study

The dyes removal was studied using batch adsorption experiments under different conditions. The study was carried out in the pH range of 3–10 at fixed dyes concentration, and the solution pH was adjusted using diluted NaOH and HNO<sub>3</sub>. The effect of dyes' initial concentration was studied using different concentrations: 12.5, 25, 50, 75 and 100 ppm. The effect of contact time was evaluated by collecting samples at different time intervals, from 15 to 120 min, to determine the optimum adsorbent dose to achieve the best removal efficiency using the minimum dose. The adsorbent dose in this study was varied from 0.025 to 0.2 mg/50 ml.

The change of dyes concentration was followed up using UV–visible spectrophotometer (UV-2600, SHIMADZU).

The removal efficiency (%) was also calculated by the following equation:

$$(\%)\text{Removal} = \{(C_i - C_e)/C_i\} \times 100 \quad (1)$$

The adsorbed amounts of dyes were calculated using the following equation:

$$Q_e = (C_i - C_e)V/M \quad (2)$$

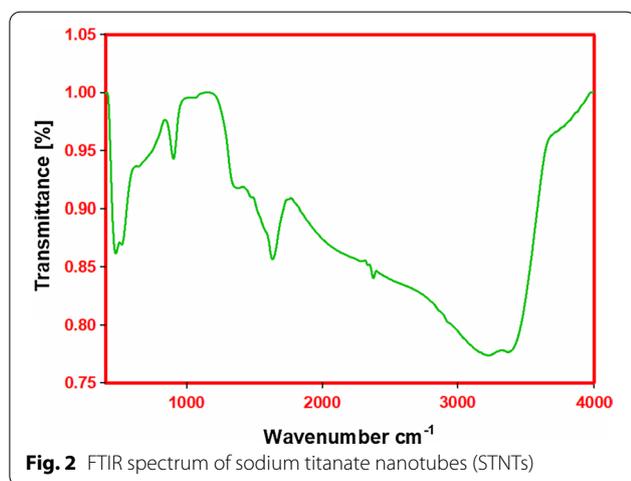
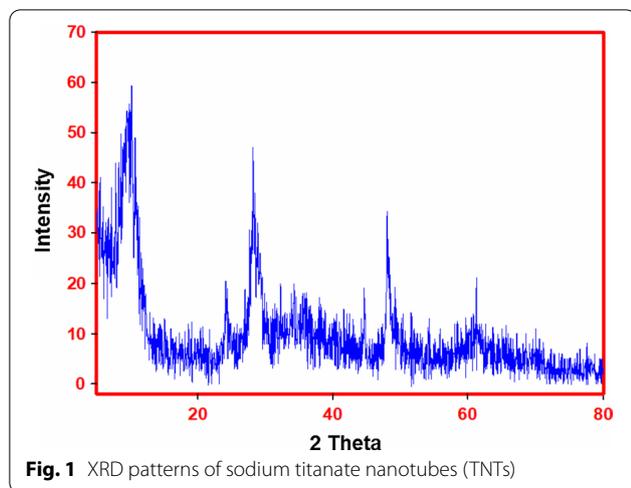
where  $Q_e$  represents the amount of adsorbed dyes,  $V$  is the volume of solution in liters,  $C_i$  is the initial dyes concentration, while  $C_e$  is the final dyes concentrations in mg/l, and the adsorbent weight in grams is expressed as  $M$ .

## 3 Results

### 3.1 Materials characterization

The XRD pattern of prepared sample is shown in Fig. 1, where the observed peaks at  $2\theta$  9.68°, 24.35°, 28.20°, 48.20° and 60.86° confirmed the tubular structure of the prepared titanate (ICDD card no. 04-009-1210).

The prepared nanotubes were studied by FTIR spectroscopy, and the obtained spectrum is shown in Fig. 2. Three bands were observed at 901 cm<sup>-1</sup>, 1633 cm<sup>-1</sup> and 3400–3200 cm<sup>-1</sup>, which are corresponding to the Ti–O stretching vibration, O–H stretching vibration, and H–O–H bending vibration, respectively; the presence of

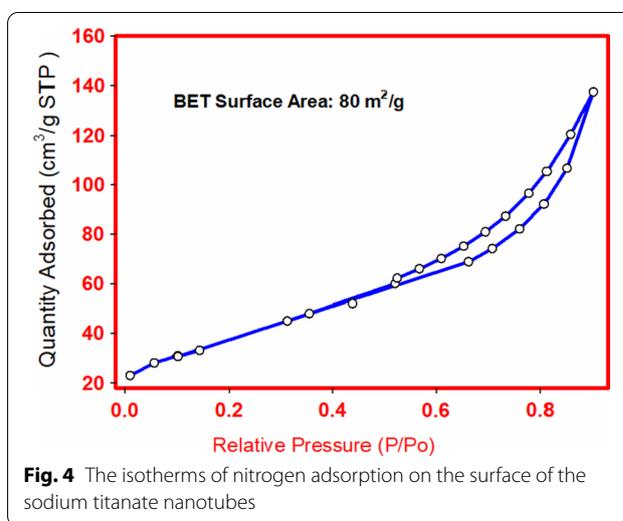
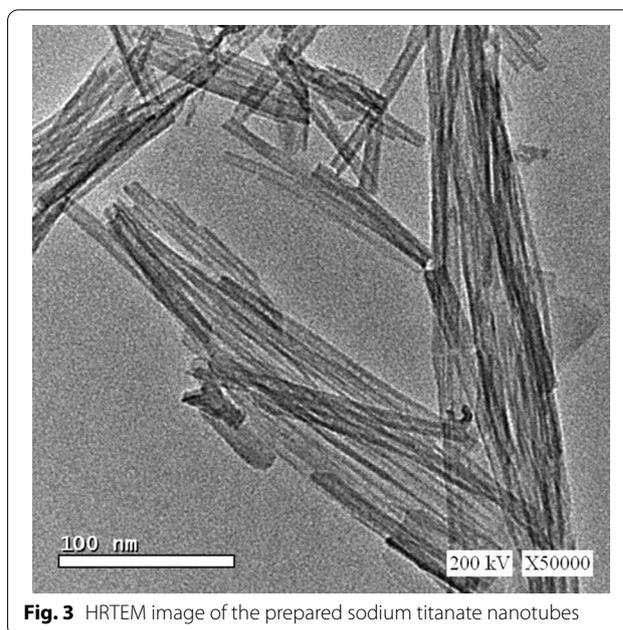


bands at  $1633\text{ cm}^{-1}$  and  $3400\text{--}3200\text{ cm}^{-1}$  indicates the presence of water molecules in the prepared titanate.

Figure 3 shows the HRTEM micrograph of the synthesized nanotubes. The figure confirmed the production of titanate with the desired tubular structure with an average diameter of less than 10 nm.

The surface area was calculated using Brunauer–Emmett–Teller (BET) method, which was found to be  $80\text{ m}^2/\text{g}$ . The observed hysteresis loop at high relative pressure, as shown in Fig. 4, indicates that the tubes are mesoporous, which may be attributed to the inner cavities of the tubes.

The above results of XRD, TEM, and FTIR reveal that titanate nanotubes were successfully prepared in large amounts compared to the previously published work; the comparison is listed in Table 1. To confirm the effectiveness of this modified method compared to other reported results, the amount of the reacting  $\text{TiO}_2$  and the obtained  $\text{Na}_2\text{Ti}_3\text{O}_7$  were normalized



to each 100 ml of the starting 10 N NaOH. Theoretically, each 2 mol of NaOH (80 gm) reacts with 3 mol of  $\text{TiO}_2$  (about 240 gm) to produce 1 mol of  $\text{Na}_2\text{Ti}_3\text{O}_7$ . Practically, researchers tended to use few grams of  $\text{TiO}_2$  and huge amounts of NaOH, since the formation of the desired nanotubes needs higher concentration of NaOH (10 N is preferred), and they neglected using the excess amounts of NaOH. It is clear from Table 1 that the yield of  $\text{Na}_2\text{Ti}_3\text{O}_7$  in previously published papers is ranging from 0.95 to 4.12 gm. For each 100 ml of 10 N NaOH, the amount increased to 12.5 in the current work.

**Table 1** Comparison between the yields of titanate with the other published work using the same preparation technique

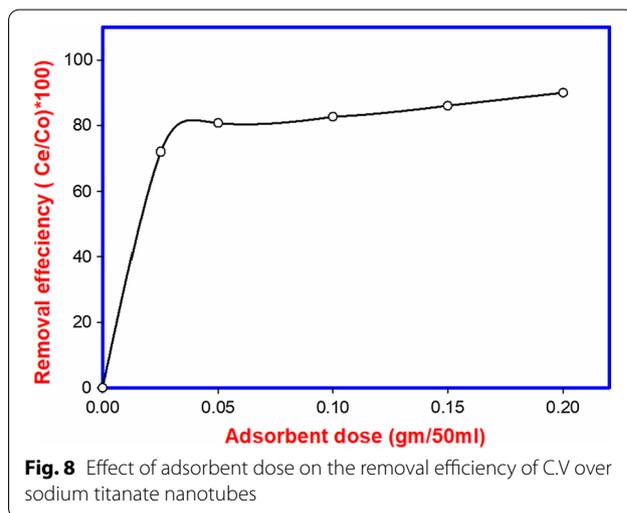
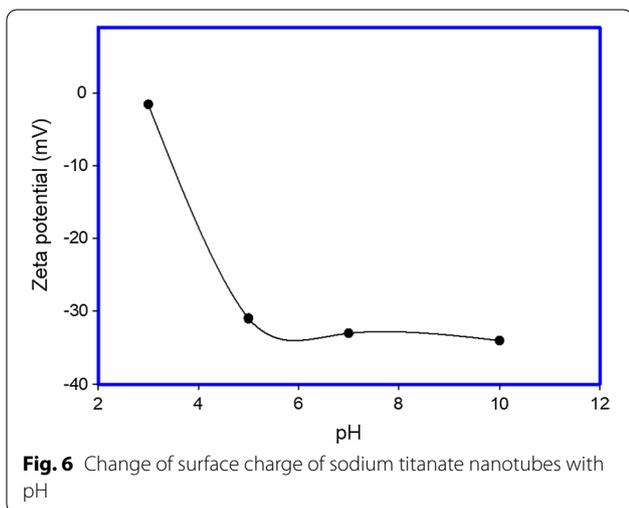
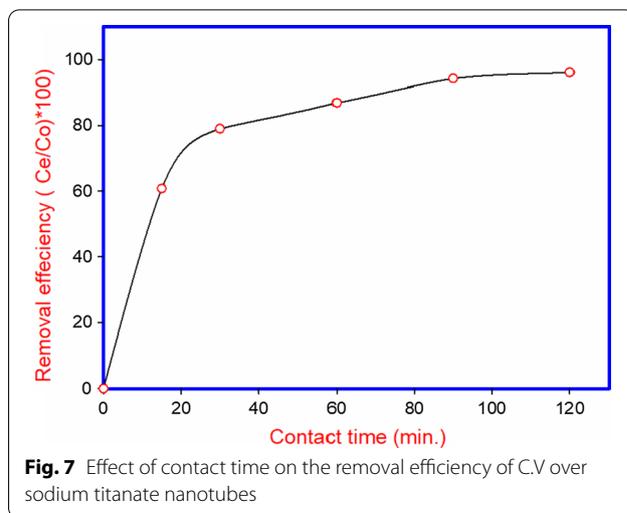
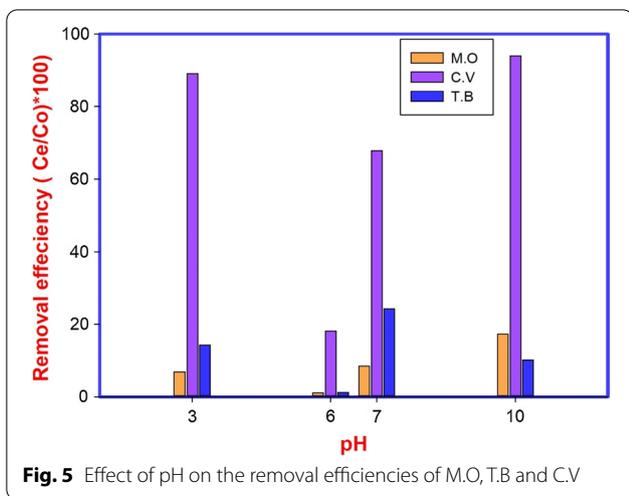
Starting materials	Weight of TiO <sub>2</sub> for each 100 ml NaOH	Yield of Na <sub>2</sub> Ti <sub>3</sub> O <sub>7</sub> for each 100 ml NaOH	Reaction time	Reaction temperature	Reactor capacity	References
2.5 g TiO <sub>2</sub> (Anatase) + 200 mL of 10 M NaOH	1.25	1.56	20 h	130 °C	Capacity 250 mL	[27]
750 mg of P25 Degussa was mixed with three different NaOH 100 mL	0.75	0.94	24 h	120 °C	100 mL	[28]
2.0 g of P25 was mixed with 60 mL of 10 M NaOH	3.3	4.12	48 h	150 °C	100 mL	[29]
P25 (1.0 g) powder was added to 13 mol L <sup>-1</sup> NaOH solution (50 mL)	1.5	2.5	24 h	150 °C	100 mL	[30]
500 ml of 10 N NaOH (purity 99.1%) aqueous solution and 10 g of pure TiO <sub>2</sub>	2	2.5	23 h	160 °C	1000 ml capacity	[31]
6 g of TiO <sub>2</sub> (Degussa P25) powder was mixed in 180 ml of 10 N sodium hydroxide	3.3	4.12	24 h	135 °C	200 ml	[32]
1.0 g TiO <sub>2</sub> powder was added to 80 ml of 10 M NaOH solution	1.25	1.56	5 days	180 °C	100 ml	[33]
5 g TiO <sub>2</sub> (Anatase) + 250 mL of 10 M NaOH	2	2.5	16 h	160 °C	500 ml	[17]
5 g TiO <sub>2</sub> (Anatase) + 250 mL of 10 M NaOH	2	2.5	16 h	160 °C	500 ml	[24]
5 g TiO <sub>2</sub> (Anatase) + 250 mL of 10 M NaOH	2	2.5	16 h	160 °C	500 ml	[23]
5 g TiO <sub>2</sub> (Anatase) + 250 mL of 10 M NaOH	2	2.5	16 h	160 °C	500 ml	[22]
10 g TiO <sub>2</sub> (Anatase) + 500 mL of 10 M NaOH	2	2.5	23 h	160 °C	1000 ml	[34]
10 g TiO <sub>2</sub> (Anatase) + 500 mL of 10 M NaOH	2	2.5	23 h	160 °C	1000 ml	[35]
10 g TiO <sub>2</sub> (Anatase) + 500 mL of 10 M NaOH	2	2.5	20 h	160 °C	1000 ml	[25]
10 g TiO <sub>2</sub> (Anatase) + 500 mL of 10 M NaOH	2	2.5	23 h	160 °C	1000 ml	[21]
10 g TiO <sub>2</sub> (Anatase) + 500 mL of 10 M NaOH	2	2.5	23 h	160 °C	1000 ml	[19]
10 g TiO <sub>2</sub> (Anatase) + 500 mL of 10 M NaOH	2	2.5	23 h	160 °C	1000 ml	[36]
10 g TiO <sub>2</sub> (Anatase) + 500 mL of 10 M NaOH	2	2.5	23 h	160 °C	1000 ml	[37]
50 g TiO <sub>2</sub> (Anatase) + 500 mL of 10 M NaOH	10	12.5	23 h	160 °C	1000 ml	Current work

If we assumed that the price per gram of TiO<sub>2</sub> and NaOH and other parameters are fixed, then the yield of this method will be much lower in price and also will save the time needed for repeating the experiment many times using the same reactor size to obtain greater amounts of titanate nanotubes. This will lower the cost of titanate nanotubes that are used in many vital and commercial applications, such as adsorption and photocatalysis.

### 3.2 Effect of pH value on the efficiency of dyes removal

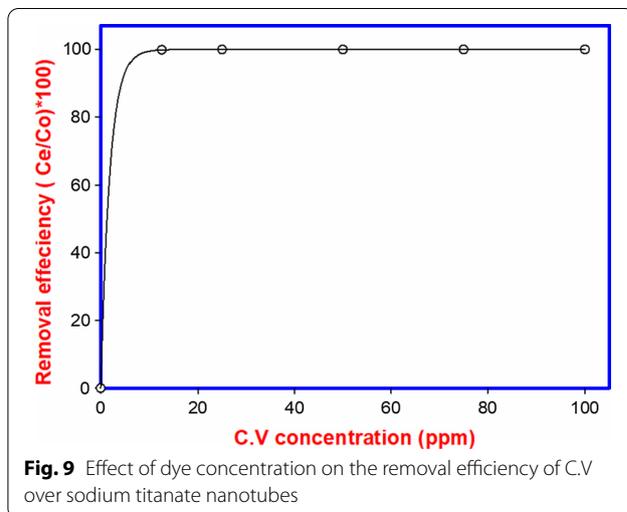
The effect of solution pH on the removal efficiency of dyes using sodium titanates is shown in Fig. 5. The

removal efficiency was evaluated at controlled pH values ranging from 3 to 10 using 100 ppm of adsorbate and 0.1 g of adsorbent for 2 h at room temperature. It is clear from Fig. 5 that removal % of M.O and T.B did not reach 30% at all pH values, while C.V removal reached 90% at pH 3, 94% at pH 10, 68% at pH 7 and 18% at pH 6. The removal percentages of M.O and T.B are attributed to the similarity in charges of the dye molecules and titanate surface (Fig. 6), since both of them are negatively charged, and hence weak electrostatic attraction or repulsion. In the case of C.V, the removal percentage is high.



**3.3 Effect of contact time, adsorbent dose and dye concentration on the efficiency of dyes removal**

Figures 7 illustrates the impact of contact time on the removal efficiency of C.V. There is a sharp increase in the first 30 min, where the removal efficiency reached about 80%. An equilibrium state was achieved after 120 min, where the removal efficiency reached about 96%. This reveals that the majority of dyes' molecules can be removed in a short time. While Fig. 8 shows the change of removal % with adsorbent dose, the results revealed that increasing the adsorbent dose increases the removal % as a result of increasing the amount of adsorbent particles available for adsorption; it is worth mentioning that the adsorbent dose of 0.001 g achieved about 75% removal % and the dose of 0.2 g achieved more than 90%; this means that small doses can be used to achieve relative high removal %. The effect of dye concentration on removal % is illustrated in Fig. 9, it is clear from this



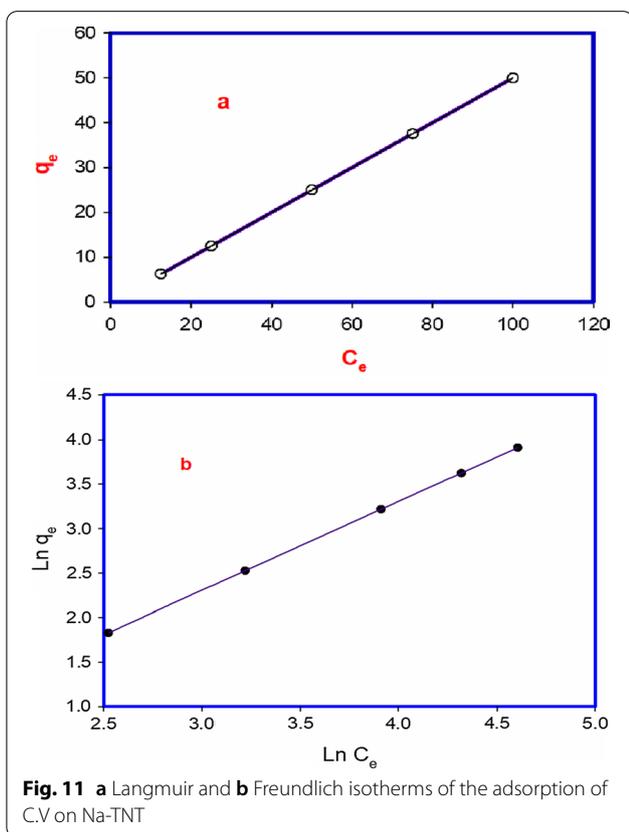
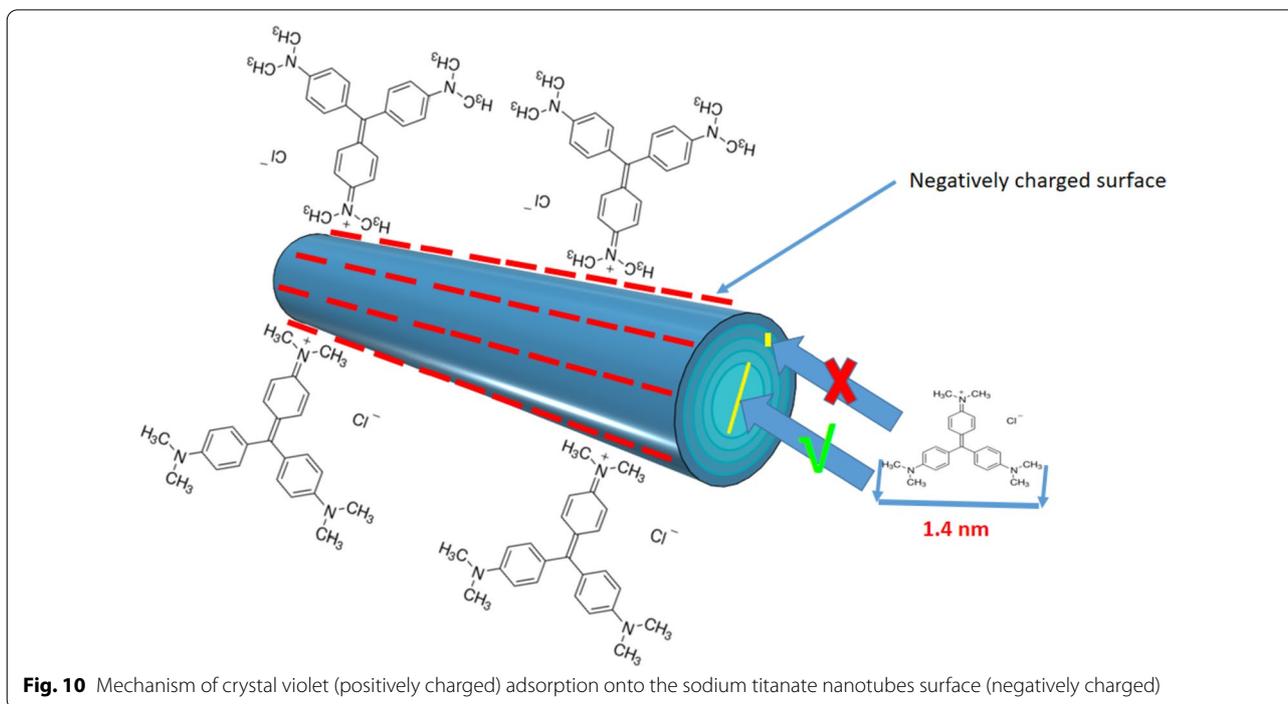


figure that the prepared nanotubes achieved the same efficiency at all concentrations in this study.

#### 4 Discussion

Results revealed that the removal efficiency was greatly affected by titanate surface charge, since the surface of the titanate was negatively charged at almost all pH values, the prepared nanotubes were capable of removing CV with high percentage, while the low removal % of M.O and T.B is attributed to the similarity in charges of the dyes (anionic dyes) and titanate surface; since both of them are negatively charged, there is weak electrostatic attraction or repulsion. Results also showed that increasing the adsorbent dose increased the removal % as a result of increasing the amount of adsorbent sites available for adsorption. As previously mentioned, about 75% removal was achieved using adsorbent dose of 0.001 g and the dose of 0.2 g achieved more than 90%; this means that small doses can be used to achieve high removal percentages. It was also found that the prepared nanotubes achieved the same efficiency at all concentrations in this study (Fig. 9).

##### 4.1 Mechanism of crystal violet adsorption on doped titanate nanotubes

It is well known and detailed that the adsorption of crystal violet particles on negative surfaces is basically

**Table 2** Freundlich and Langmuir isotherms parameters for crystal violet adsorption on sodium titanates at room temperature

Isotherm	Crystal violet	Parameters description
Langmuir	1.0000 1.0000 100 0.01	$R^2$ is the regression coefficient $R_L$ value lies between 0 and 1 for favorable adsorption $R_L$ higher than 1 means unfavorable adsorption, $R_L$ equal to 1 represents linear adsorption $R_L = 0$ adsorption process is irreversible $q_m$ is the maximum adsorption capacity of adsorbent, expressed in mg/g $K_L$ is the constant of Langmuir model
Freundlich	0.98852 0.99 1.2 0.9	$R^2$ is the regression coefficient $K_F$ and $n$ are the Freundlich constants and give indication about the maximum adsorption capacity If $1/n$ is less than 0.5, the adsorption would be easily carried out If $1/n$ is larger than 0.5, the adsorption is difficult

electrostatic, because the crystal violet molecules are positively charged, and it is reported that the titanate surfaces are negatively charged at most of the pH values.

As shown in Fig. 10, it is clear that the C.V molecules can be adsorbed in many sites on the prepared nanotubes, where it can be adsorbed on the outer surface of titanate due to the opposite charges, and also it can be adsorbed on the inner cavities of the tubes. The d-spacing value of the nanotubes is about 0.9 nm which hinders the adsorption of the dye molecules in between these layers.

#### 4.2 Adsorption isotherms

The adsorptive behavior of crystal violet onto Na-TNT was studied using two common models: Langmuir and Freundlich isotherm models.

The direct frame of Langmuir and Freundlich models is outlined in Eqs. 3 and 4, respectively.

$$1/q_e = 1/q_m + 1/(K_L q_m C_e) \quad (3)$$

$$\ln q_e = \ln K_F + 1/n(\ln C_e) \quad (4)$$

The values of adsorption parameters obtained from the straight fitting of Langmuir (Fig. 11a) and Freundlich (Fig. 11b) are recorded and clarified in Table 2. The results best fitted with Langmuir isotherm, where  $R^2$  values are 1 in the case of Langmuir and 0.98 in the case of Freundlich; this means that the dye molecules are adsorbed in single layer on the titanate surface.

#### 5 Conclusions

Sodium titanate nanotubes were prepared by using the hydrothermal method. The amount of the starting  $TiO_2$  particles was increased five times to improve the production yield. The obtained nanotubes retained the same features when compared to the previously prepared nanotubes, where their surfaces were negatively charged at different pH values, which enabled the nanotubes from removing cationic dyes (crystal violet) with high

efficiency. Using this modified method to prepare titanate nanostructures in large amounts with lower cost will reduce the total cost of the treatment process which is based on adsorption and photocatalysis.

#### Abbreviations

TNTs: Sodium titanate nanotubes; M.O: Methyl orange; C.V: Crystal violet; T.B: Thymol blue; XRD: X-ray powder diffraction; FTIR: Fourier-transform infrared; HRTEM: Using high-resolution transmission electron microscope; BET: Brunauer–Emmett–Teller.

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#### Authors' contributions

AH, AA, and MA conceived the research idea and designed the experiments. SA and AH performed the experiments and wrote the original manuscript; AA and MA revised and edited the manuscript to be in the final form. All authors have read and approved the final manuscript.

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#### Declarations

#### Ethics approval and consent to participate

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#### Consent for publication

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#### Competing interests

The authors declare no competing interest.

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