



# Preparation of V<sub>2</sub>O<sub>5</sub> and SnO<sub>2</sub> Nanoparticles and Their Application as Pollutant Removal

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## Article information

### Article history:

Received: June, 17, 2021

Accepted: October, 26, 2021

Available online: December, 04, 2021

### Keywords:

Vanadium pentoxide nanoparticle,  
Tin dioxide nanoparticles,  
Pollution removal

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## Abstract

Vanadium pentoxide and Tin dioxide nanoparticles were prepared by hydrothermal methods to remove methylene blue (MB) and bromothymol blue (BTB) dyes). First the prepared nanoparticles were heated at different temperature (90 °C and 500 °C). Then these nanoparticles were characterized by different methods such as UV/Visible, FT-IR Spectroscopy, X-Ray Diffraction (XRD), Atomic Force Microscopy (AFM), and Scanning Electron Microscopy (SEM). Our results explain that vanadium pentoxide nanoparticles appeared high remove activity against both types of organic dyes (MB and BTB) compared to tin dioxide nanoparticles. Furthermore, vanadium pentoxide heating at 90 °C has grain size (86.02 nm) more than that of Tin dioxide (48.02 nm) at same temperature. The activity of vanadium pentoxide is more than that of tin dioxide. may be related to high ionic potential (0.073), compared with Tin dioxide (0.048).

DOI: [10.53293/jasn.2021.3869.1049](https://doi.org/10.53293/jasn.2021.3869.1049), Department of Applied Science, University of Technology  
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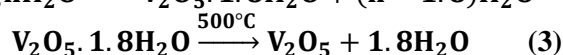
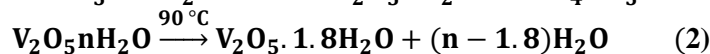
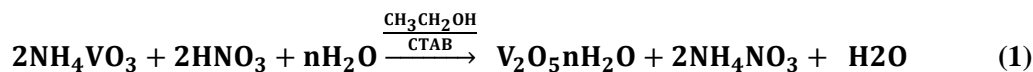
## 1. Introduction

Water is the essential element of life and it is a precious resource for human civilization. Clean water is a major global challenge in the twenty-first century. Thus, global climate change highlights the already unequal distribution of freshwater [1]. Clean water is water that is free from toxic substances, chemicals, and pathogens. Dyes are considered one of the most critical organic pollutants that cause water pollution and cause many diseases. The adsorption technique, is chemical adsorption, biological adsorption and ion exchange [2-4]. Nanomaterials are considered to be the absorbing materials for water pollutants by adsorption [2]. Nanotechnology has proven its presence in many fields such as sensing pollutants and water pollution, Nanomaterials such as metallic, metal oxides, carbon compounds, nanocomposites and filtering membranes have been used for filtering membranes in wastewater treating [5]. metal oxides such as (Al<sub>2</sub>O<sub>3</sub>, V<sub>2</sub>O<sub>5</sub>, Mn<sub>2</sub>O<sub>3</sub>, SnO<sub>2</sub>, La<sub>2</sub>O<sub>3</sub> are used in water purification technology due to their ability to separate the chemical and biological properties of water [6]. The dye absorption is done on the surface of nano absorbent materials. Carbon nanotubes, graphene, zeolites and nanocomposites have higher efficiency and larger surface areas. First the contaminants are absorbed on the surface of the nanoparticles, then the solution is accompanied by a change in the pH, a change in temperature, etc.[7].

## 2. Experimental Procedure

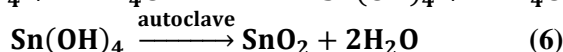
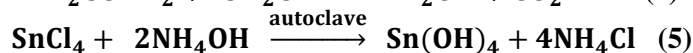
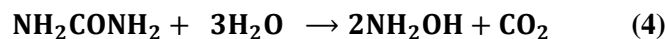
### 2.1 Preparation of Vanadium Pentoxide Nanoparticles ( $V_2O_5$ NPs)

Vanadium pentoxide was prepared by the hydrothermal (reflex) method by dissolving 2 grams of ammonium metavanadate ( $NH_4VO_3$ ) in a mixed solution of distilled water and ethanol in a ratio of a ratio (7:3) with add 0.5 of cetyltrimethylammonium bromide (CTAB) and drops of nitric acid( $HNO_3$ ) until it reaches the pH of 2.5, heat the solution for 6 hours, then washed several washes after the reaction ends, dried at 90 °C and annealed at 500°C for 2 hours [8, 9].



### 2.2 Preparation of Tin Dioxide Nanoparticles ( $SnO_2$ NPs)

Tin dioxide was prepared using the hydrothermal method (autoclave) by dissolving two grams of Tin tetrachloride ( $SnCl_4$ ) in distilled water, adding to it 0.92 g of urea and 0.5 g of cetyltrimethylammonium bromide (CTAB) placing it with an autoclave for 12 hours at 200 and washed several washes and dried at 90 and annealed at 500 for two hours [10].



### 2.3 The adsorption of methylene blue (MB) / or bromothymol blue (BTB) dyes.

We performed two experiments to verify the adsorption of two organic dyes (methylene blue (MB) and bromothymol blue (BTB)). 0.1 g of each nanoparticle ( $V_2O_5$  and  $SnO_2$ ) was added to 25 ml (20 mg/l) of the dye solutions (MB or BTB) in concentration ( $4 \times 10^{-5}$  M,  $64 \times 10^{-5}$  M) respectively. The first experiment was to adsorption of methylene blue (MB) dye onto nanoparticles. The second experiment was to adsorption of bromothymol blue (BTB) dye onto nanoparticles. In these two experiments, we used our nanoparticles after heating at 90 °C and 500 °C. The removal percentages (R%) of MB / or BTB dyes onto nanoparticles were calculated through the equation (7) [8].

$$(R\%) = [(C_o - C_t) / C_o] \times 100 \% \quad (7)$$

Where;  $C_o$  indicates the initial concentration of the dye mg/L, and  $C_t$  indicates the final concentration of the dye mg/ L.

The adsorption capacity ( $q_t$ ) of the MB/ or BTB dyes onto nanoparticles was calculated through equation (8).

$$q_t = (C_o - C_t) V / W \quad (8)$$

Where; W indicates weight in grams of nanoparticles, V volume of dye solution in millilitres.

## 3. Results and Discussion

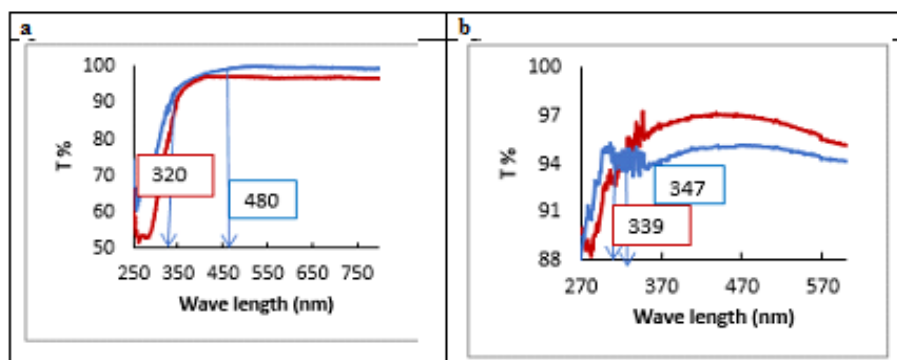
### 3.1 Optical properties of the nanoparticles solutions.

The optical properties of the prepared nanoparticle solutions ( $10^{-5}$  M) were done by dissolved the nanoparticle in Dimethylsulfoxide (DMSO) solvent. The plot of optical transmission curves against the wavelength for nanoparticles at different temperatures (90 °C and 500 °C,) from the region between 200 nm to 800 nm is explained in figure (1). The spectrum of vanadium pentoxide (figure (1-a)) shows the transmittance edge is shifted to a lower wavelength (blue shift) from 480 nm to 320 nm with an increase of annealing temperatures. We can produce the value of the energy gap (the space between HOMO and LUMO) according to the following experiment equation:

$$\text{Energy gap (eV)} = 1240/\lambda_{\text{max}} \quad (9)$$

Where 1240 is a factor that converts nm to eV,  $\lambda_{\text{max}}$  is the first maximum transmittance in nm. Therefore, the energy gap of  $\text{V}_2\text{O}_5 \cdot n\text{H}_2\text{O}$  equal to 2.583 eV and that annealing at 500 °C ( $\text{V}_2\text{O}_5$ ) equal to 3.875 eV respectively. These results are agreed with reference [11] which found equal to 2.63 eV and 3.48 eV at temperature 90 °C and 500 °C respectively..

The tin dioxide nanoparticles show a decrease in the transmittance edge (blue shift), from 347 nm to 339 nm, annealing temperatures, this may be relating to converting the as-prepared (tin hydroxide) nanoparticles to tin dioxide throw autoclave reaction. Therefore, the energy gap of as-prepared equal to 3.573 eV and that of annealing at 500 °C equal to 3.657 eV respectively, as in figure (1 –b). These results are agreed with reference [12] which is due to the fundamental absorption of light.



**Figure (1):** The optical transmittance for nanoparticles, a: vanadium pentoxide; b: tin dioxide; annealing at different temperatures (90 °C and 500 °C).

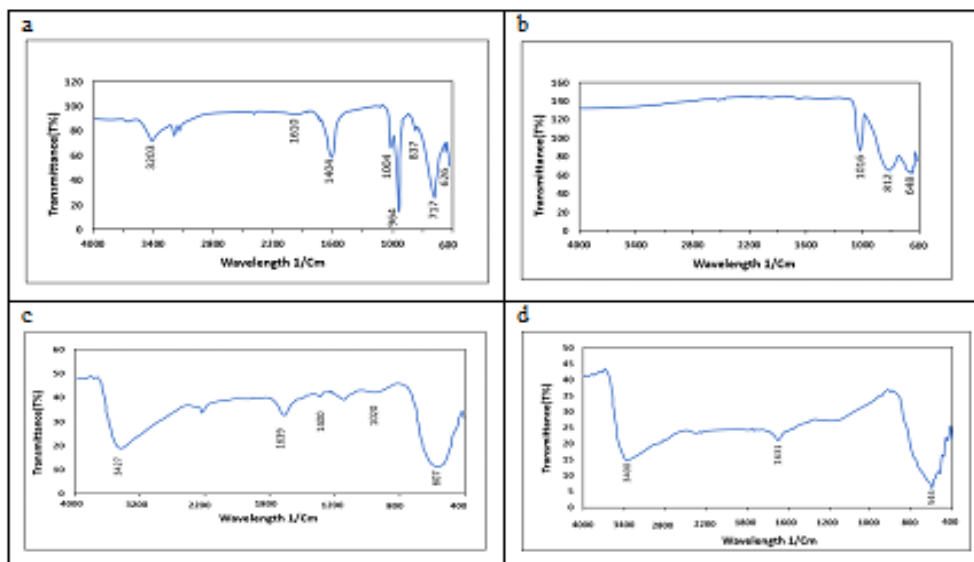
### 3.2 Fourier Transform Infrared Spectroscopy (FTIR)

#### 3.2.1 FTIR Spectra for $\text{V}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ and $\text{V}_2\text{O}_5$ nanoparticles

The FTIR spectrum of vanadium pentoxide nanoparticles (as-prepared)  $\text{V}_2\text{O}_5 \cdot n\text{H}_2\text{O}$  that heated at 90 °C for 60 min explains in figure (2-a). The broadband at  $3203 \text{ cm}^{-1}$  relates to stretching vibration and the peak at  $1610 \text{ cm}^{-1}$  relate to bending of the O-H group in the water. The peaks at  $1404 \text{ cm}^{-1}$ ,  $1004 \text{ cm}^{-1}$ ,  $964 \text{ cm}^{-1}$  and  $837 \text{ cm}^{-1}$  corresponding to the bending vibration of C=C vibration of CTAB, vanadyl stretching of ( $\delta \text{ V-O}$ ), V=O vibration [13], and V-O-V vibration respectively. The bands at  $717 \text{ cm}^{-1}$  and  $626 \text{ cm}^{-1}$  corresponding to the asymmetric and symmetric stretch around V-O-V. Figure (2-b) shows  $\text{V}_2\text{O}_5$  annealing at 500°C for 120 min, there are no peaks that appeared to relate to water in this sample. The peaks at  $1016 \text{ cm}^{-1}$  correspond to stretching vibration of V=O and that at  $812 \text{ cm}^{-1}$  relate to stretching of V-O-V and that at  $648 \text{ cm}^{-1}$  correspond to V-O stretching [14].

#### 3.2.2 FTIR Spectra for $\text{SnO}_2$ nanoparticles

Figures (2-c and 2-d) show the FTIR spectra of  $\text{SnO}_2$  as prepared and annealed at 500 °C. There are no high differences between both spectra, which means both compounds are the same ( $\text{SnO}_2$ ). These may relate to converting tin hydroxide ( $\text{Sn(OH)}_4$ ) to tin dioxide ( $\text{SnO}_2$ ) during the reaction proses in the autoclave, due to the high reaction temperature (200 °C). The broad peaks around  $3400 \text{ cm}^{-1}$  and  $1631 \text{ cm}^{-1}$  correspond to O-H bond's stretching and bending in water in water respectively. The broad peak at  $544 \text{ cm}^{-1}$  assigned to the Sn–O vibration and this broad peak improves to  $544 \text{ cm}^{-1}$  [15].



**Figure (2):** FTIR spectra of  $V_2O_5$  a: as- prepared, b: annealing at 500° C and  $SnO_2$  c: as-prepared, d: annealing at 500 ° C.

### 3.3 X-Ray Diffraction (XRD)

#### 3.3.1 The X-Ray Diffraction for $V_2O_5 \cdot nH_2O$ and $V_2O_5$ nanoparticles

The results of the X-ray analysis of vanadium pentoxide prepared by the hydrothermal method (reflex), and heated at 90 °C, showed the main diffraction peaks at  $2\theta = (40.8^\circ, 44.81^\circ, 47.80^\circ, \text{ and } 49.60^\circ)$  corresponding to the characteristic diffraction peaks of (002), (411), (600), and (302) relate to  $V_2O_5 \cdot nH_2O$ , and that at  $2\theta = 27.7$  corresponding to VOOH according to the card number (JCPDS card No. 01-089-0612), as shown in figure (3-a) [16].

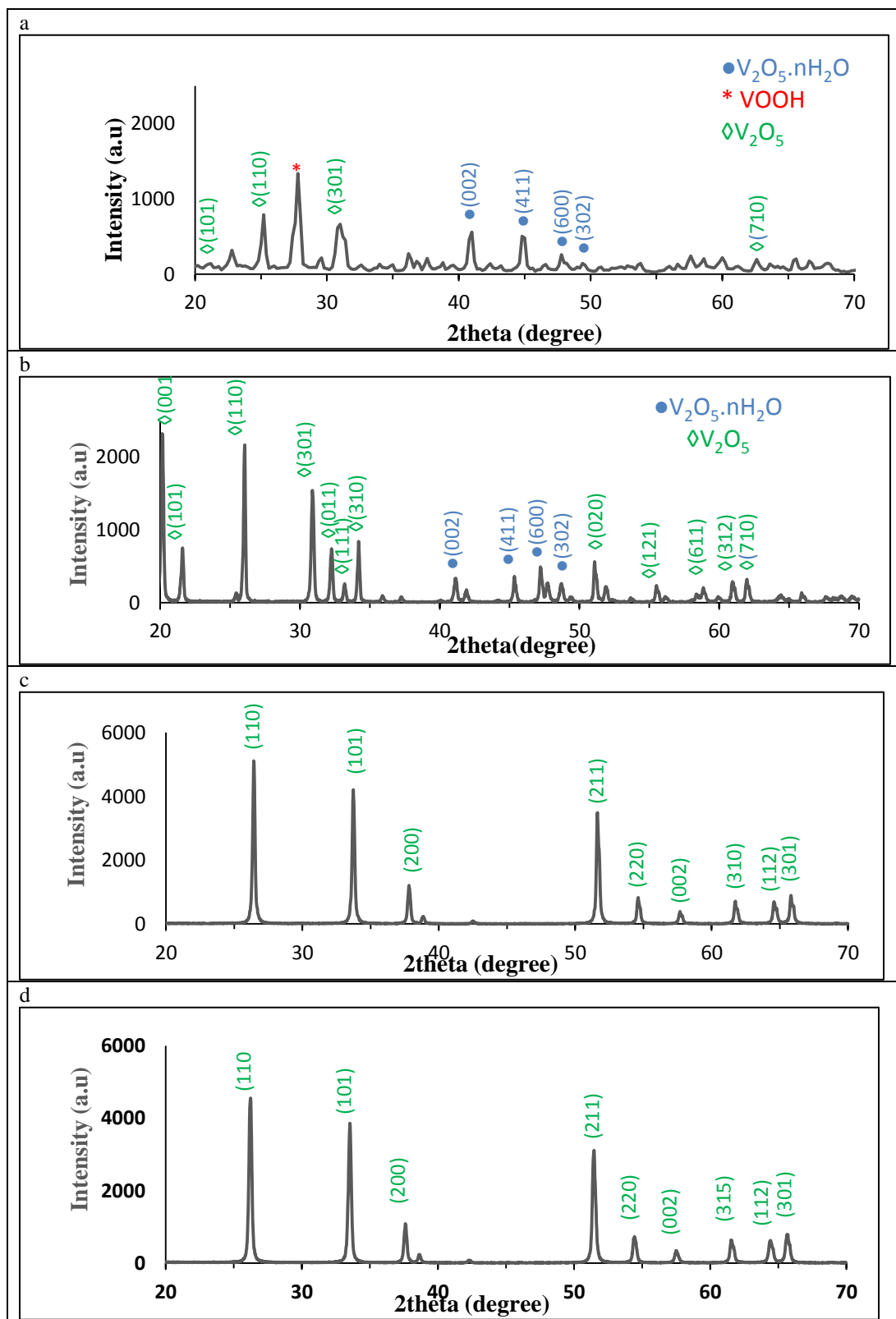
When annealing at 500 °C we notice the prominent diffraction peaks at  $2\theta = (20.15^\circ, 21.20^\circ, 25.20^\circ, 31.66^\circ, 32.03^\circ, 32.49^\circ, 34.07^\circ, 47.80^\circ, 49.60^\circ, 50.99^\circ, 54.61^\circ, 57.92^\circ, 60.93^\circ \text{ and } 62.80^\circ)$ , which corresponds to the peak's diffraction (001), (101), (110), (301), (011), (111), (310), (600), (302), (020), (121), (611), (312), and (710). Meanwhile, there are improve and increase in its density of many peaks such as those at  $2\theta = (20.15, 32.03, 34.07, 50.99, 60.93)$  which corresponds to the peak's diffraction (001), (011), (310), (020), and (312). These peaks are identical to the card number (JCPDS card no. 41-1426), as in figure (3-b) [17, 18]. The grain size of the nanoparticles has been estimated from the full width at half maximum (FWHM) of the diffraction peak using the Debye-Scherrer formula according to the equation:

$$D = K\lambda / \beta \cos\theta \quad (10)$$

Where, (D) is crystallite size, (k) is the shape factor ( $\approx 0.9$ ), ( $\lambda$ ) is the x-ray wavelength (1.540 Å), ( $\beta$ ) is the full width half maximum (FWHM) in radians, and ( $\theta$ ) is the Bragg's diffraction angle value [19]. Table (1) shows the average grain size for  $V_2O_5$  annealed at 500 °C according to the Scherrer equation.

#### 3.3.2 The X-Ray Diffraction for $SnO_2$ nanoparticles

The X-ray results of heated  $SnO_2$  at 90 °C, is very similar to that of annealing at 500 °C with a slight change in intensity, this indicates that the formation of  $SnO_2$  in both situations. The most prominent of the main peaks that appeared are  $2\theta = (26.45, 33.71, 51.63, 54.61, \text{ and } 65.85)$ , which corresponds to the diffraction values of (110), (101), (211), (220), and (301) as shown in the figures (3-c and 3-d). These peaks correspond to the card number (JCPDS part no. 041-1445) [20]. This indicates the sample prepared by autoclave completely converts to  $SnO_2$  without the formation of  $Sn(OH)_4$  as it happens when prepared by sol-gel. This may be related to the higher temperature (200 °C) used in the autoclave in preparation. Table (1) shows the average grain size for  $SnO_2$  annealed at 500 °C according to the Scherrer equation.



**Figure (3):** XRD of  $V_2O_5$  a: as-prepared, b: annealing at 500 °C and  $SnO_2$  c: as-prepared, d: annealing at 500 °C.

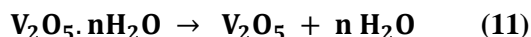
**Table (1):** The grain size of the  $V_2O_5$  and  $SnO_2$  nanoparticles calculate by the Scherer equation.

	$2\theta$ (deg)	hkl	FWHM (deg)	D(nm)
$V_2O_5$ annealing at 500 °C for 120 min	20.15	001	0.1732	45.136
	34.07	101	0.1968	38.575
	51.05	310	0.1440	49.757
$SnO_2$ annealing at 500 °C for 120 min	26.45	110	0.2125	36.373
	33.71	101	0.2362	32.171
	51.63	211	0.1968	36.319

### 3.4 Surface Morphology

#### 3.4.1 By atomic force microscopy (AFM)

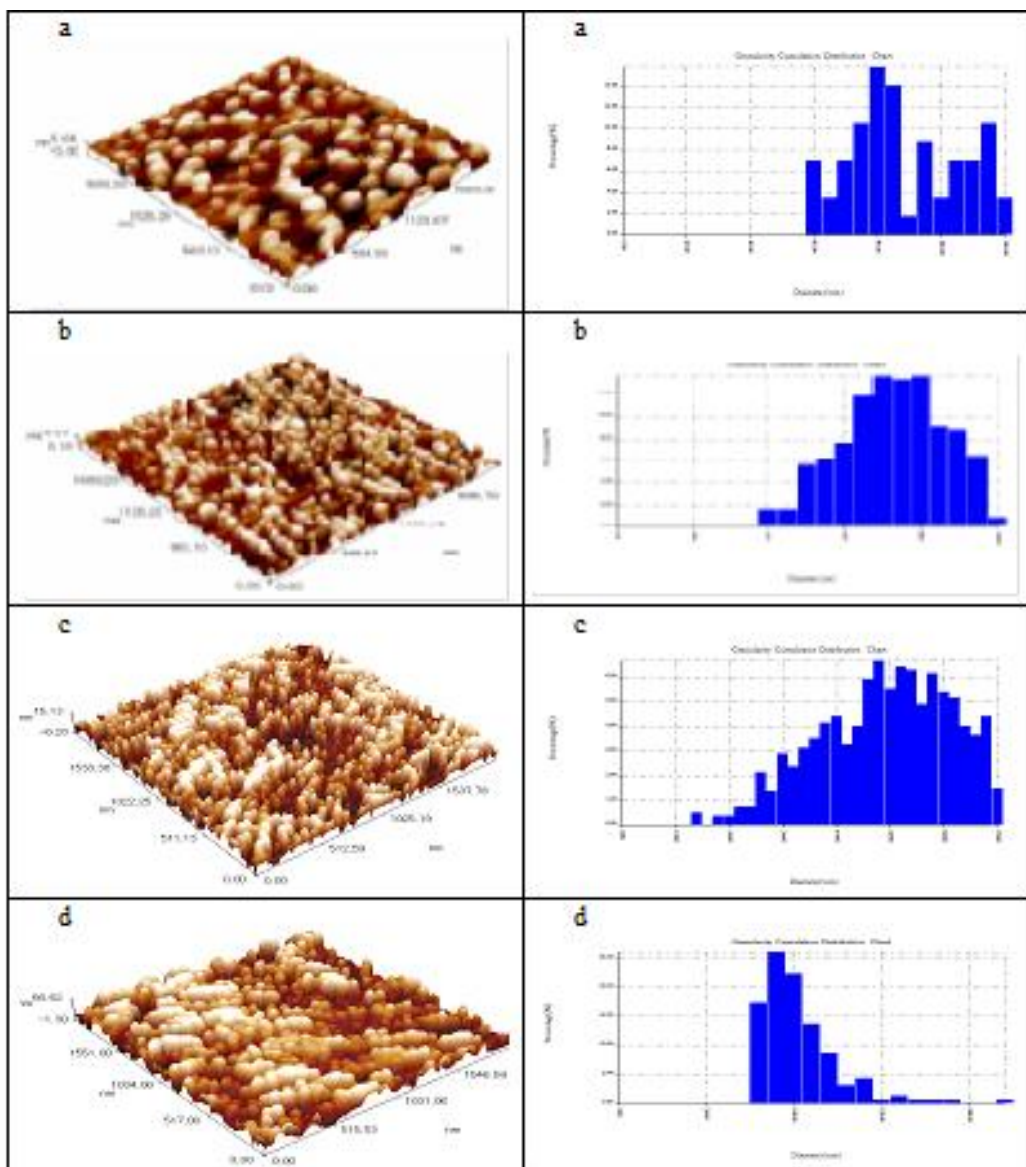
AFM results of vanadium pentoxide and tin dioxide in three (3D) dimension images with different temperatures as in figure (4). The distribution and accumulation of nanoparticles seem to large balls (figure 4-a) and convert to smaller when annealing at 500 °C (figure 4-b). This may relate to the loss of water molecules from the sample, as explained in equation [9].



These results agree with the average grain size obtained from AFM analysis, which converts the average grain size from 86.02 nm ( $V_2O_5 \cdot nH_2O$ ) to 70.34nm of  $V_2O_5$  when the heating increases from 90 °C to 500 °C respectively, as in table (2).

The distribution and accumulation of tin dioxide in nanoparticles like to smooth rods (figure 4-c) become large bubbles with an increase in size when annealing at 500 °C (figure 4-d). That is mean the product may be converted to tin dioxide nanoparticles during the preparation method (autoclave) and the grain size increases from 48.08 nm to 98.34 nm by increasing temperature as a result of improving the crystalline and the accumulation of the nanoparticles [21], as in table (2).





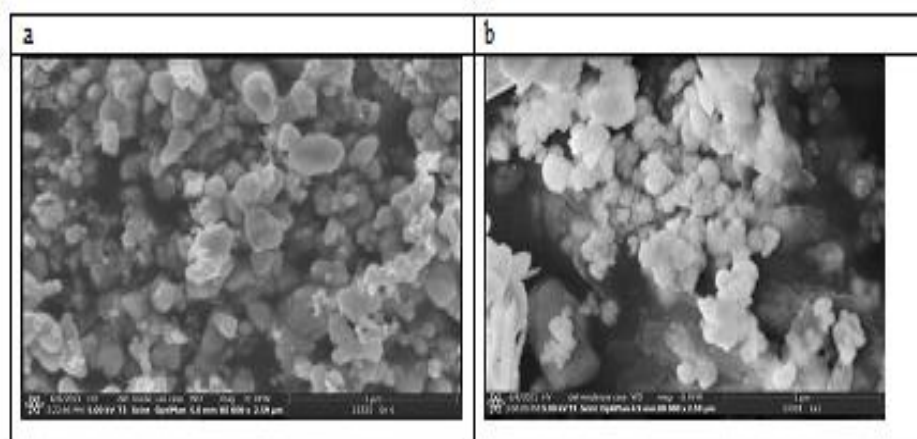
**Figure (4):** AFM images at 3D of vanadium pentoxide and tin dioxide at different temperatures

**Table (2):** Average grain size of nanoparticles of  $V_2O_5$  and  $SnO_2$  at (90 °C and 500 °C).

Nanoparticles	Average grain size(d) (nm) heating at	
	90 °C	500°C
Vanadium pentoxide	86.02	70.34
Tin dioxide	48.08	98.34

### 3.4.2 By Surface scanning Electron Microscopy (SEM)

The surface morphology magnification SEM images of the metal oxides ( $V_2O_5$  and  $SnO_2$ ) nanoparticles annealing at temperature 500 °C for 120 min, with a magnification of 100  $\mu m$ , are shown in figures (5). The surface morphology of  $V_2O_5$  (figure 5-a), has a high aggregate consisting of close rough leaves, and that for  $SnO_2$  (figure 5-b) seem to be nanoclusters.

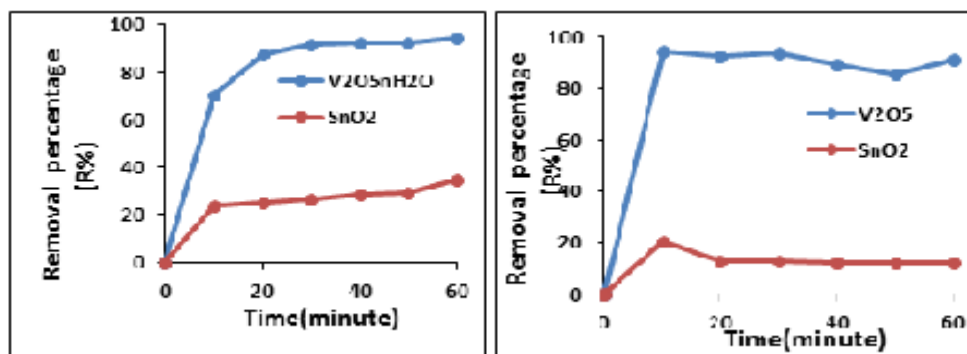


**Figure (5):** SEM analysis of the surface shape annealing at 500 °C for 120 min: (a)  $V_2O_5$  and (b)  $SnO_2$ .

#### 4. Applications

##### 4.1 The adsorptions of methylene blue (MB) dye onto nanoparticles.

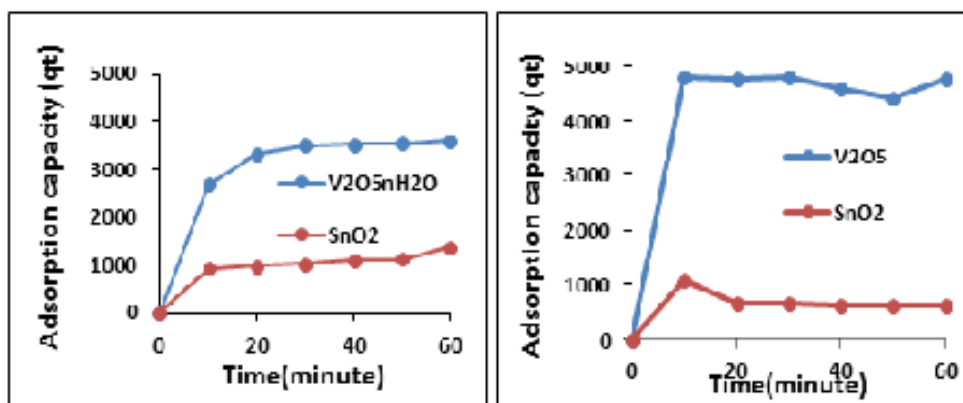
Vanadium pentoxide ( $V_2O_5 \cdot nH_2O$ ) nanoparticles have the highest value of removal percentage (R%) compare with tin dioxide ( $SnO_2$ ) nanoparticles, as in figure (6). Our results showed that methylene blue dye onto nanoparticles increased sharply during the first 10 min, it may be related to the availability of large bonding sites on the surface area of the nanoparticles. After that, very slow adsorption happens between 10 min to 60 min, this situation continues until the equilibrium happens. While the low increase in adsorption onto nanoparticles takes place (after 10 min), it relates to the aggregation or overlap of the adsorption sites [22]. The annealing at 500 °C is similar to the same sequences (to some extent) of the nanoparticles heating at 90 °C, except for the result at 50 min (for  $V_2O_5$ ) which decreases in adsorption compared with the measurements between 10 min to 60 min. This decrease may relate to the dissociation of clusters of dye on the surface of the nanoparticles, as in figure (6). While the removal percentage (R%) for kerosene by  $V_2O_5$  doping onto oxide multiwall carbon nanotube was 80 %. [5].



**Figure (6):** The removal percentage (R%) of MB onto  $V_2O_5 \cdot nH_2O$  and  $SnO_2$  nanoparticles heating at 90 °C and 500 °C for 60 min).

When the adsorption capacity ( $q_t$ ) of MB onto nanoparticles plot against contact time of nanoparticles (heating at 90 °C), figure (7), we found the adsorption capacities ( $q_t$ ) are precisely like the situations that happen in the removal percentage operation (at 90 °C), with the identical sequences. As well as the adsorption capacities ( $q_t$ ) of the samples heated at temperature 500 °C, show same the sequences of removal percentage. This similarity is attributed to using of the same volume of dye and the exact weight of nanoparticles in all experiments. Except for the result at 10 min of  $SnO_2$  nanoparticles which gave some increase in removal percentage ( $q_t$ ) compared with the measurements after that (20 min to 60 min), this increase may relate to providing large surface areas after that dissociation of clusters dyes on the surface of the nanoparticles. As in figure (7). While the adsorption capacity ( $q_t$ ) for kerosene by  $V_2O_5$  doping onto oxide multiwall carbon nanotube was 10.2 [5].



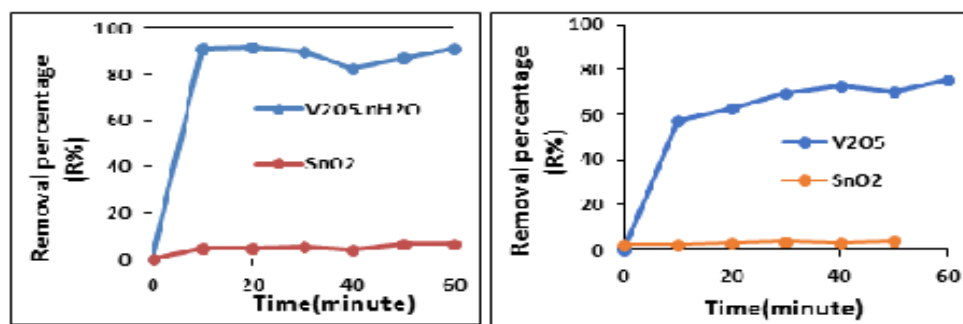


**Figure (7):** Adsorption capacity ( $q_t$ ) of MB onto  $V_2O_5.nH_2O$  and  $SnO_2$  nanoparticles heating at 90 °C and 500 °C for 60 min)

#### 4.2 The adsorption of bromothymol blue (BTB) dye onto nanoparticles.

Vanadium pentoxide ( $V_2O_5.nH_2O$ ) nanoparticles have the highest value of removal percentage compare with tin dioxide ( $SnO_2$ ) nanoparticles, as in figure (8). Our results show that the bromothymol blue dye's removal percentage (R%) onto nanoparticles increased sharply during the first 10 min of  $V_2O_5.nH_2O$ . After that, a prolonged decrease happens between 10 min to 40 min. Then there is a slow increase between 40 min to 60 min. The high increase in adsorption onto nanoparticles in the first 10 min may be related to the availability of large bonding sites on the surface area of the nanoparticles. While the low increase in adsorption onto nanoparticles takes place (between 10 min to 40 min), it may be related to the aggregation or overlap of the adsorption sites, which are the dissociation of clusters of dyes on the surface of the nanoparticles. Then the surface of nanoparticles will get some empty spaces to continue adsorption.

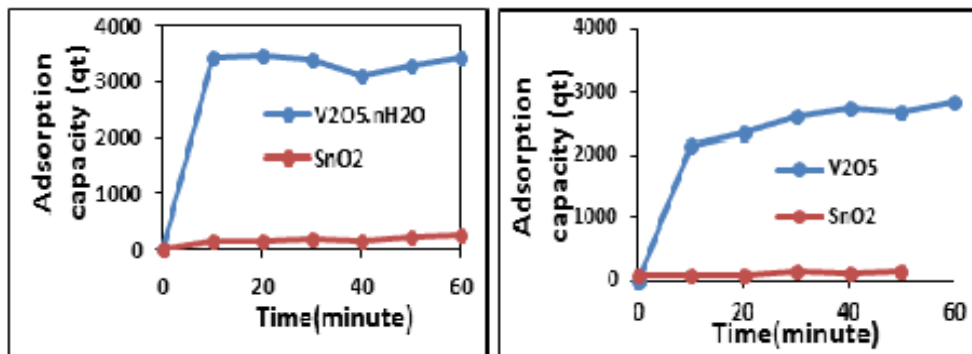
The removal percentage (R%) of the  $V_2O_5$  sample (heating at 500 °C) is the highest among other nanoparticles and different that at 90 °C. The activity to removal (R%) continues to increase after 10 min and becomes maximum at 40 min. After that, there is a decrease (at 50 min) and a return to increasing (at 60 min), the increase in removal relates to providing large surface areas to bond with dyes molecules. In contrast the decreases in removal relate to the dissociation of clusters of dyes on the surface of the nanoparticles, as in figure (8).



**Figure (8):** The removal percentage (R%) of BTB onto  $V_2O_5.nH_2O$  and  $SnO_2$  nanoparticles heating at 90 °C and 500 °C for 60 min).

When the adsorption capacity ( $q_t$ ) of BTB onto nanoparticles plot against contact time of nanoparticles (heating at 90 °C), figure (9). The adsorption capacity ( $q_t$ ) of nanoparticles is a similarity in shape compared to removal percentage (R %) at the same temperature (heating at 90 °C). The increase continues (in the  $V_2O_5$  nanoparticles) to get the maximum adsorption capacities ( $q_t$ ) at 10 min, then began to decrease at 40 min after that the increase continues. The increase in adsorption capacities ( $q_t$ ), related to nanoparticles' surface will get some empty spaces to bond with dyes molecules. While the decrease relating to the dissociation of clusters dyes on the surface of the nanoparticles.

When the adsorption capacity ( $q_t$ ) of BTB onto nanoparticles plot against contact time of nanoparticles (heating at 500 °C), figure (9), we found the adsorption capacities ( $q_t$ ) are precisely like the situations that happen in the removal percentage operation (at 500 °C), with the identical sequences. This similarity is attributed to the use of the same volume of the dye and the exact weight of nanoparticles in all experiments. The highest adsorption capacities ( $q_t$ ) were to  $V_2O_5$  nanoparticles. The maximum value of  $q_t$  it was at 40 min, may be relate to providing large surface areas after that dissociation of clusters of the dyes on the surface of the nanoparticles.



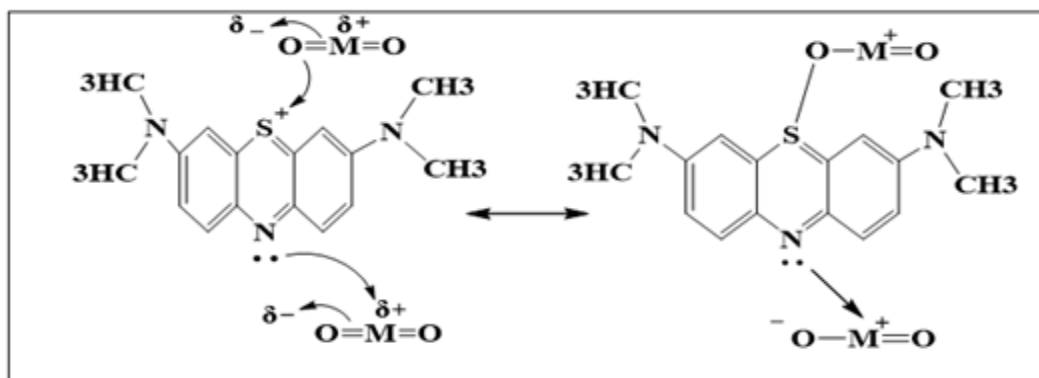
**Figure (9):** Adsorption capacity ( $q_t$ ) of BTB onto  $V_2O_5.nH_2O$  and  $SnO_2$  nanoparticles heating at 90 °C and 500 °C for 60 min.

The ability of vanadium pentoxide to adsorb MB or BTB dyes is the largest. This may be related to the ionic potential (charge/radius) of the nanoparticles. The highest ionic potential appears to be vanadium pentoxide (0.073), and the second appears to be tin dioxide (0.048). These results agree with the explanation of removal percentage (R%) sequence and adsorption capacities ( $q_t$ ) of MB or BTB dyes onto nanoparticles in both situations (90 °C and 500 °C). Therefore, we can consider that the ionic potential may be the critically affected agent in this operation.

## 5. Mechanisms to Remove MB and BTB Dyes

### 5.1 Mechanisms to Remove Methylene blue (MB)

The mechanism to remove Mb from aqueous solution by nanoparticles can be occurred by two methods: first method by the interaction between the positive charge on the sulphur atom of the MB and the partial negative charge on the oxygen atom of the nanoparticle to form an ionic bond. The second method is the interaction between alone pair electrons (as base) on the nitrogen atom of the MB and metal ion (as acid) in the nanoparticles to form a coordination bond [22]. Therefore there is two bonds (ionic and covalent) formation between MB and nanoparticles, as in figure (10) [23].

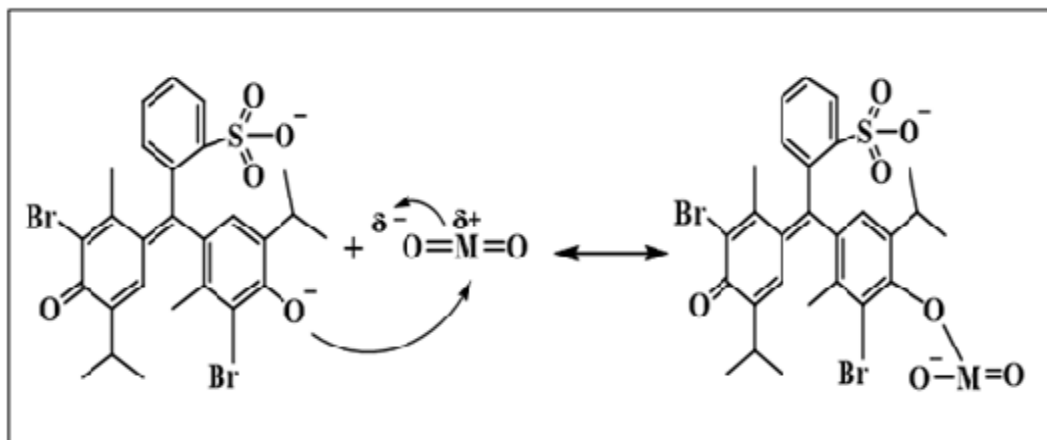


**Figure (10):** The suggested mechanism to remove methylene blue (MB) by nanoparticles.

### 5.2 Mechanisms to Remove Bromothymol blue (BTB).

The mechanism to remove BTB from aqueous solution by the nanoparticles can occur through the interaction between the partially positive charges on the metal in the nanoparticle, with a negative charge in the

BTB to form an ionic bond be explained in figure (11).



**Figure (11):** The suggested mechanisms to remove bromothymol blue (BTB) by nanoparticles.

According to these mechanisms, MB can bond with nanoparticles from two sides (sulphur and nitrogen), while BTB can bond with nanoparticles only by one side (phenoxide). Therefore, we can conclude why the removal of nanoparticles by MB is higher than that of BTB.

## 6. Conclusion

The conclusion can be summarized in the following points:

1. We successfully prepared some nanoparticles ( $V_2O_5 \cdot nH_2O$  and  $SnO_2$ ) and their oxides ( $V_2O_5$  and  $SnO_2$ ) by different methods.
2. After heating temperature at  $90^\circ C$   $SnO_2$  has the lowest average grain size (48.08) nm.
3. After heating temperature at  $500^\circ C$ ,  $V_2O_5$  has the lowest average grain size (70.34) nm,
4. Both  $V_2O_5 \cdot nH_2O$  and  $V_2O_5$  have the highest removal percentage (R%) and adsorption capacity ( $q_t$ ) against MB and BTB dyes (as a pollutant), after heating temperatures ( $90^\circ C$  and  $500^\circ C$ ), and the activity is more when heating at  $500^\circ C$  compared to other nanoparticles.
5. The highest values of R% and  $q_t$  for vanadium pentoxide nanoparticles may be relate to the highest ionic potential (0.073). Moreover, we conclude the adsorption may be depend on this factor and do not depend on the average grain size or surface area.

## Acknowledgement

We are grateful to Inorganic Lab. Applird Chemistry, App;ied Sciences, University of Technology, for their support of the work.

## Conflict of Interest

There is no conflict of interest.

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