

# Synthesis and characterization of titanium oxide nanomaterials using sol-gel method

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**Abstract:** This paper reports the effect of temperature on the properties of TiO<sub>2</sub> nanomaterials, synthesis and characterization. TiO<sub>2</sub> powders were synthesized by sol-gel method using TiCl<sub>4</sub> solution added in deionized water in ice bath under fume hood followed by the addition of ethanol with vigorous stirring for 30 min at room temperature. The gel solution was obtained and then got dried using oven at 200°C for 4 hours. Then, the dried gel was calcinated at 250°C, 400°C and 600°C using furnace for 4 hours each. The synthesized TiO<sub>2</sub> nanomaterials were characterized by XRD, UV-Vis spectrophotometer, Transmission electron microscope (TEM), Scanning electron microscope (SEM), Energy dispersive spectroscopy (EDS). XRD shows the particles size with high crystallinity and purity which is in good agreement with the TEM result. The particles size of the synthesized TiO<sub>2</sub> nanomaterial at calcination temperatures of 250°C, 400°C and 600°C were 9.22 nm, 14.33 nm and 36.72 nm respectively calculated from XRD result. The absorption edge for TiO<sub>2</sub> nanomaterials synthesized by sol gel synthesis method was found to be 350 nm and the corresponding calculated band gap energy was 3.54 eV. The average particles size of the synthesized TiO<sub>2</sub> nanopowder investigated from TEM using histograms at calcination temperatures of 250°C, 400°C and 600°C were found (8.55 ± 0.25) nm, (13.82 ± 0.41) nm and (36.06 ± 2.03) nm respectively. The polygonal structure of TiO<sub>2</sub> nanomaterials were studied by scanning electron microscope. The EDS result showed that at calcination temperatures of 250°C, 400°C and 600°C, the concentrations of titanium were 33.34%, 32.6% and 31.89%, and the concentrations of chlorine were 2.64%, 0% and 0%, and the concentrations of oxygen were 64.02%, 67.4% and 68.11% in the synthesized TiO<sub>2</sub> nanopowder respectively which is qualitatively confirmed by energy dispersive x-ray spectroscopy results.

**Keywords:** Spectroscopy, Sol Gel Synthesis, TiO<sub>2</sub> Nanomaterial

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## 1. Introduction

Nanocrystalline semiconductors have achieved a great importance in our industrial world today. They are cornerstones of nano-scale science and nanotechnology. Nanostructure science and technology is a broad and interdisciplinary area of research and development activity that has been growing explosively worldwide in the past decade [2]. It has the potential for revolutionizing the ways in which materials and products are created and the range and nature of functionalities that can be accessed. It is already having a significant commercial impact, which will assuredly increase in the future. Nanomaterials are defined as a set of substances where at least one dimension is less than approximately 100 nanometers.

Nanomaterials have a broad spectrum of applications.

Today they are contained in many products and used in various technologies. Most nanoproducts produced on an industrial scale are nanomaterials, although they also arise as by products in the manufacture of medicine [9]. Nanomaterials are emerging family of novel materials that could be designed for specific properties. The various properties: - optical, physical, chemical and electrical - could be tailored for specific applications. Nanoparticle reinforced polymers may be used to replace metallic components in automobile industry for reduced fuel consumption and CO<sub>2</sub> emission.

Semiconductors such as: TiO<sub>2</sub>, ZnO, Fe<sub>2</sub>O<sub>3</sub>, CdS, and ZnS can act as sensitizers for light reduced redox processes due to their electronic structure, which is characterized by a filled

valence band and an empty conduction band.  $\text{TiO}_2$  nanomaterials have one of great attractive significant interesting materials for scientists and physicists due to their properties and widely used in many technological applications. It has the advantage of being cheap, nontoxic and stable. It has proven to be one of the promising n-type semiconductors which have many attractive properties [7]. These properties include high refractive index, high dielectric constant, chemical stability and wide band gap [1].

Titanium dioxide ( $\text{TiO}_2$ ) nanomaterials have been widely studied in the last two decades [2]. Due to their versatile properties,  $\text{TiO}_2$  nanomaterials have possessed themselves vast applications, including paint, toothpaste, UV protection, photocatalysis, photovoltaics, electrochromics, as well as photochromics.

Materials in the nanometer scale, such as colloidal dispersions and thin films, have been studied over many years and many physical properties related to the nanometer size, such as coloration of gold nanoparticles, have been known for centuries. One of the critical challenges faced currently by researchers in the nanotechnology and nanoscience fields is the inability and the lack of instruments to observe and measure, and manipulate the materials at the nanometer level by manifesting at the macroscopic level. In the past, the studies have been focused mainly on the collective behaviors and properties of nanostructures of  $\text{TiO}_2$  materials. A better fundamental understanding and various potential applications increasingly demand the ability and instrumentation to observe and measure, and manipulate the individual nanomaterial of  $\text{TiO}_2$  and nanostructures of  $\text{TiO}_2$ .

In the process of synthesis of  $\text{TiO}_2$ , the shape and crystal structure of the product significantly depends on raw material used. Characterization and manipulation of individual nanostructures of  $\text{TiO}_2$  require not only extreme sensitivity and accuracy, but also atomic-level resolution. Therefore, this leads to various microscopes that would play a central role in characterization and measurements of nanostructures of  $\text{TiO}_2$  materials.

In the present study, the most convenient ways of synthesizing various nanomaterials have preferable due to low cost, ease fabrication and low temperature. This method has various advantages over other method, such as coprecipitation or allowing impregnation, which can be achieved by introducing dopant, molecular scale mixing, high purity of the precursors and homogeneity of sol-gel products with high purity physical, morphological and chemical properties of nanomaterial. Therefore, that was the reason why sol gel synthesis method is preferred as the method of titanium dioxide nanomaterials synthesis.

## 2. Methods

### 2.1. Chemicals

In this study the following chemicals were used:  $\text{TiCl}_4$  (MW 189.87  $\text{g mol}^{-1}$ , 99.9%), Ethanol,  $\text{CH}_3\text{CH}_2\text{OH}$  (MW 58.03  $\text{g mol}^{-1}$ , Abcon Chemicals, 99.8%),  $\text{AgNO}_3$  (MW

169.87  $\text{g mol}^{-1}$ , 98.4%), Ammonium hydroxide,  $\text{NH}_4\text{OH}$  (MW, 17.03  $\text{g mol}^{-1}$ , 25%).

### 2.2. Method of Preparation of $\text{TiO}_2$ Nanomaterials

Titanium tetra chloride ( $\text{TiCl}_4$ ) of 3.5 ml was added to 50 ml deionized water in ice bath and the process was done under fume hood followed by the addition of 35 ml of ethanol with vigorous stirring for 30 min at room temperature. Drops of ammonium hydroxide were added wisely into solution of the titanium tetra chloride ( $\text{TiCl}_4$ ), ethanol and deionized water to neutralize it and precipitate was obtained. After stirring vigorously, the solution was made to settle for twelve hours. Then, precipitate was centrifuged. The obtained precipitate was washed with deionized water until the removals of chloride ion and was centrifugally separated. Then, using oven, the precipitate was dried at 200°C to remove part of the absorbed water for 4 hours and finally amorphous  $\text{TiO}_2$  was obtained. The obtained amorphous  $\text{TiO}_2$  was calcinated at the temperatures of 250°C, 400°C and 600°C for four hours step by step. Finally, the powder  $\text{TiO}_2$  nanomaterial was obtained [11, 8, 10].

### 2.3. Methods of Characterization of $\text{TiO}_2$ Nanomaterials

There are a large number of methods to characterize  $\text{TiO}_2$  nanomaterials. The most common methods used in this work were: powder x-ray diffraction (XRD), Ultraviolet-visible spectrometer, Transmission electron microscope (TEM), Scanning electron microscope (SEM) and Energy dispersive x-ray spectroscopy to study percentage yield of element, the crystal structure, defect structure, chemical analysis, and phase identification, crystal or grain size and morphological properties.

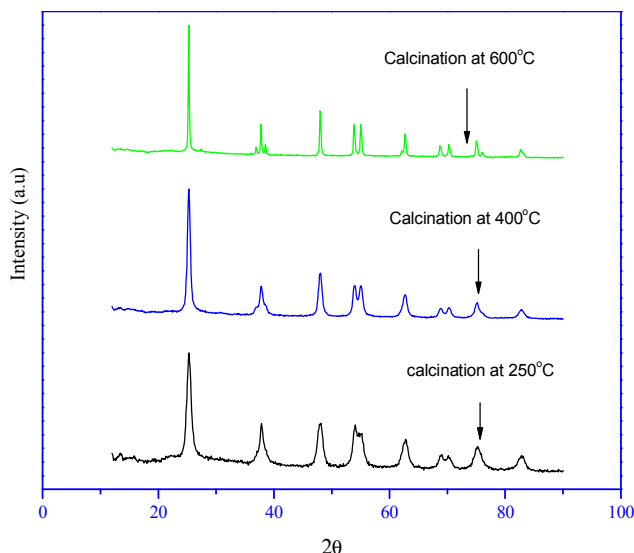
## 3. Results and Discussion

### 3.1. Powder Preparation of $\text{TiO}_2$ Nanomaterials

When 3.5 ml of  $\text{TiCl}_4$  solution was added to 50 ml of deionized water under fume hood, an exothermic reaction were observed. Then, a white precipitate was obtained after adding a drop of ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) wisely; and the yellow gel rose. The gel being yellow indicates the formation of  $\text{Ti}(\text{OH})_4$ . After stirring the solution using magnetic stirrer for 30 min and settling for 12 hours, the precipitate was centrifuged. Then, the obtained precipitate was placed in an oven at 200°C for 4 hours in order to dry, which leads to the formation of white amorphous  $\text{TiO}_2$ ; after cooling at room temperature, the amorphous white  $\text{TiO}_2$  was obtained. The obtained amorphous white  $\text{TiO}_2$  was placed in furnace at 250°C, 400°C and 600°C for 4 hours step by step to calcinate it. This calcination leads to the formation of white powder  $\text{TiO}_2$ . After cooling at room temperature, the product was ground using agate mortar. Finally, the ground  $\text{TiO}_2$  powder was subjected to calcination temperatures of 250°C, 400°C and 600°C respectively so that it could be labeled and kept for further study.

### 3.2. Powder X-Ray Diffraction (XRD) Characterization of TiO<sub>2</sub> Nanomaterials

Powder x-ray diffraction (XRD) was used for identification of the particles size, crystal structures, crystal orientation and lattice parameter. X-ray powder diffraction (XRD) was used to characterize the titanium dioxide powder at different temperatures and to determine particles structure and crystal size.



**Figure 1.** The XRD pattern of TiO<sub>2</sub> nanomaterials calcination temperatures at 250°C, 400°C and 600°C

As can be observed from figure 1, the XRD peaks in the angle range of  $12^\circ \leq 2\theta \leq 90.027^\circ$  determined that peaks for 250°C are  $13.26^\circ, 15.74^\circ, 25.31^\circ, 30.71^\circ, 37.85^\circ, 48.17^\circ, 54.05^\circ, 62.73^\circ, 69.00^\circ, 75.17^\circ, 82.79^\circ$ , the peaks for 400°C are  $25.31^\circ, 37.84^\circ, 48.07^\circ, 55.11^\circ, 62.62^\circ, 70.25^\circ, 75.17^\circ, 82.79^\circ$  and the peaks for 600°C are  $25.31^\circ, 37.75^\circ, 38.52^\circ, 47.98^\circ, 55.02^\circ, 62.64^\circ, 74.98^\circ$  and  $82.69^\circ$ . Among the XRD peaks, the width of  $25.31^\circ$  is useful peak since it has high intensity which in turn is used to determine crystals size. The peak value corresponds to the tetragonal anatase phase. The highest intensity was achieved for  $25.31^\circ$  at temperature of 600°C and lowest intensity of  $25.31^\circ$  was achieved at a temperature of 250°C. The intense sharp peak indicated that the crystalline phase of anatase TiO<sub>2</sub> was successfully formed. One can understand from figure 1 that, the intensity of the diffraction signal increases with increasing the calcination temperature. This indicates that the concentration of the particles increases.

From the XRD pattern, the crystallite sizes of the synthesized TiO<sub>2</sub> nanomaterials were estimated using Scherere equation [4, 6, 12].

$$d = \frac{\lambda k}{\beta \cos \theta}, \quad (2)$$

where,

$d$  is crystallite size in nanometer

$k$  is shape factor constant, which is 0.89

$\beta$  is the full width at half maximum (FWHM) in radian

$\lambda$  is the wave length of the X-ray which is 0.15406 nm for Cu target K $\alpha$  radiation and

$\theta$  is the Bragg angle.

Using equation (2) the estimated crystalline size is given in the following table.

**Table 1.** Crystallite size ( $d$ ) of the synthesized TiO<sub>2</sub> nanomaterials calcination temperatures at 250°C, 400°C and 600°C

Calcinations temperature of samples(°C)	$2\theta$ (degrees)	$\beta$ (radians)	Crystalline size (nm)
250	25.31°	0.01491	9.22
400	25.31°	0.009643	14.33
600	25.31°	0.003748	36.72

As shown in table1 and figure 1, increasing particles size has direct relation to calcination temperature and inverse relation to FWHM. The sharp peaks by XRD pattern show the crystallinity and purity of titanium dioxide nanomaterial. This exactly matches with Scherere equation. The crystallite size (nm) increased as the synthesis temperatures increased from 250°C to 400°C and 400°C to 600°C.

### 3.3. Ultra Violet-Visible Spectrometer of TiO<sub>2</sub> Nanomaterials

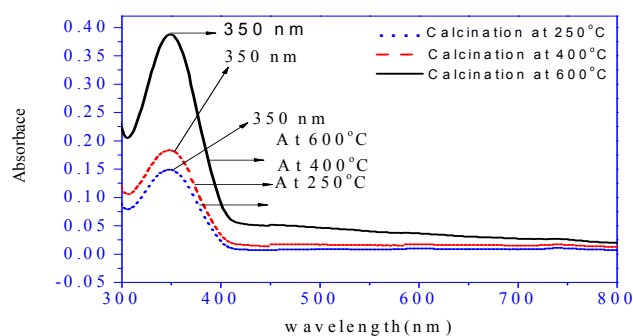
The bandgap energy ( $E_g$ ) of the synthesized TiO<sub>2</sub> nanomaterials was obtained using the equation [3].

$$E_g = \frac{1240}{\lambda} \text{ eV}, \quad (3)$$

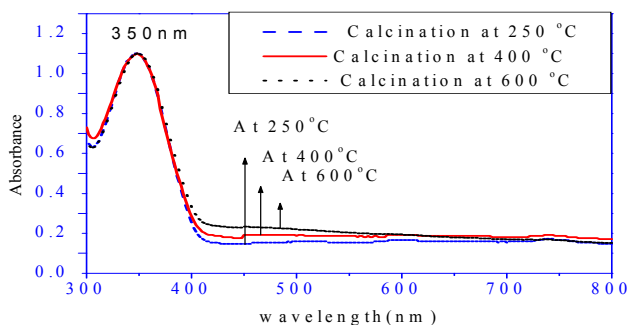
where  $E_g$  is the band gap in electron volt (eV) and  $\lambda$  is the wavelength of the absorption edges in the spectrum in nanometer (nm).

As observed in figure 2 and 3, the UV-Vis spectra from synthesized TiO<sub>2</sub> nanomaterials show the absorbance to be below 350 nm. Using equation (3) the calculated value for the band gap energy of the synthesized TiO<sub>2</sub> nanomaterials is 3.54 eV.

The graph which corresponds to the statements above is given below.

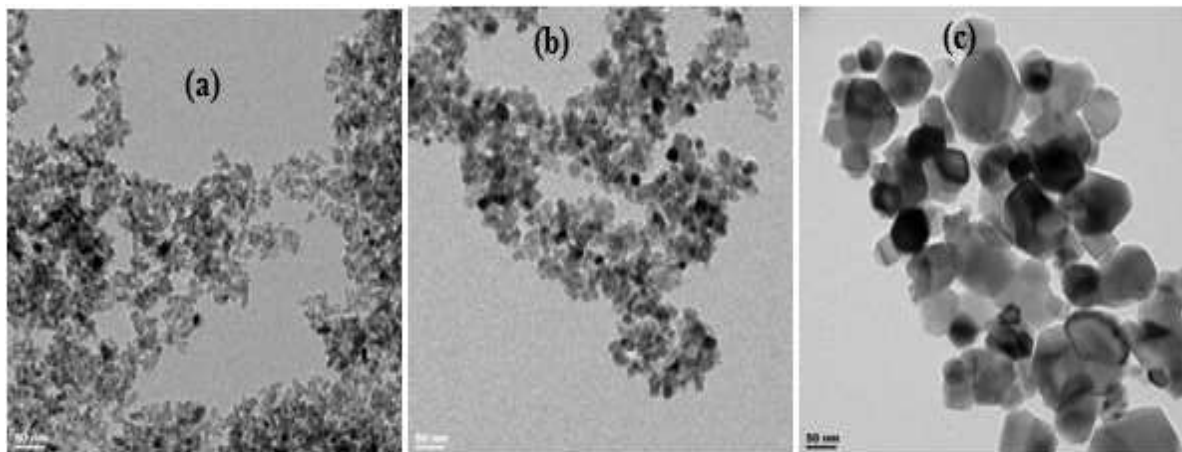


**Figure 2.** UV-Visible spectrum of synthesized TiO<sub>2</sub> nanomaterials dispersed in hot deionized water at calcinations of 250°C, 400°C and 600°C



**Figure 3.** UV-Visible spectrum of synthesized  $\text{TiO}_2$  nanomaterials dispersed in hot deionized water normalized graph calcination temperatures at  $250^\circ\text{C}$ ,  $400^\circ\text{C}$  and  $600^\circ\text{C}$

The synthesized  $\text{TiO}_2$  nanomaterials band gap energy (3.54 eV) are larger than the value of 3.2 eV for the bulk  $\text{TiO}_2$  nanomaterials. This is true due to the fact that the band gap of the semiconductors has been found to be particle size dependent [5, 10]. The band gap increases with decreasing particle size and the absorption edge is shifted to a higher energy (blue shift) with decreasing particle size.



**Figure 4.** The TEM images of the synthesized  $\text{TiO}_2$  nanomaterials (a) calcination of  $250^\circ\text{C}$ , (b) calcination of  $400^\circ\text{C}$  and (c) calcination of  $600^\circ\text{C}$

As observed from figure 4 (a) of TEM  $\text{TiO}_2$  nanomaterials calcination at temperature of  $250^\circ\text{C}$ , consists of particles with unfortunate fusion and aggregation. Again, observed from figure 4 (a) and 4 (b) when the calcination temperature of  $250^\circ\text{C}$  the size of the nanoparticles uniform compare with calcination temperatures of  $400^\circ\text{C}$ . As the calcination temperature increased to  $600^\circ\text{C}$ , the sizes of particle are no quite uniform compared with calcination temperatures of  $250^\circ\text{C}$  and  $400^\circ\text{C}$  as observed from figure 4 (c). It was learnt from figure 4 that, an increase in calcination temperatures leads to increase particle size, particles fusion and aggregation.

The size distribution was obtained by measuring each particle diameter. The random orientation of particles allows for a statistical measure of the size distribution to be generated. As it is clearly shown in figures 5, 6 and 7, the synthesized  $\text{TiO}_2$  nanomaterial average particles size at a calcination temperatures of  $250^\circ\text{C}$ ,  $400^\circ\text{C}$  and  $600^\circ\text{C}$  are 8.55 nm, 13.82 nm and 36.06 nm and the estimated errors

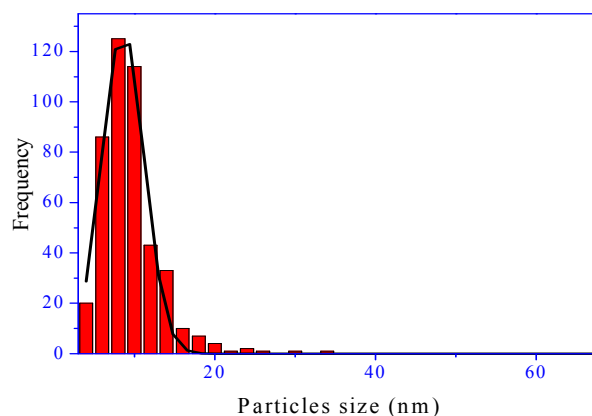
The band gap values validates our crystallite size results according to which smaller crystallite size should have larger band gap and large crystallite size should have smaller band gap.

As figure 2 and 3 clearly shows that the wavelength corresponding to the absorbance edge of  $\text{TiO}_2$  nanomaterial with calcination temperatures at  $250^\circ\text{C}$ ,  $400^\circ\text{C}$  and  $600^\circ\text{C}$  is the same (350 nm) and the concentration measurement of the absorbance increased as the calcination temperature is increases.

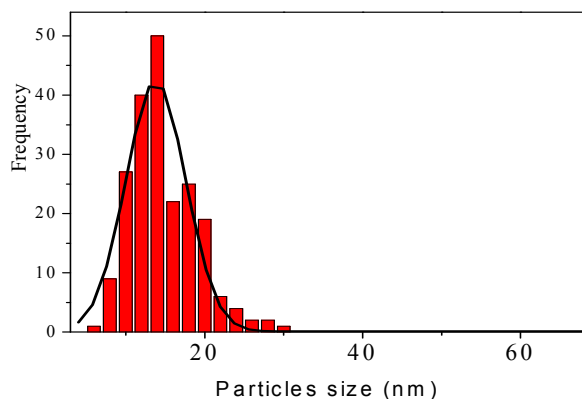
### 3.4. Transmission Electron Microscope (TEM)

Transmission electron microscope (TEM) was used to study the crystal structure, morphology, shapes and particle size. Figure 4 clearly showed that polygonal structure, shapes and particle size of  $\text{TiO}_2$  nanomaterial were dependent on the calcination temperatures. When the calcination temperatures increased the shapes of the  $\text{TiO}_2$  nanomaterials leads to spherical.

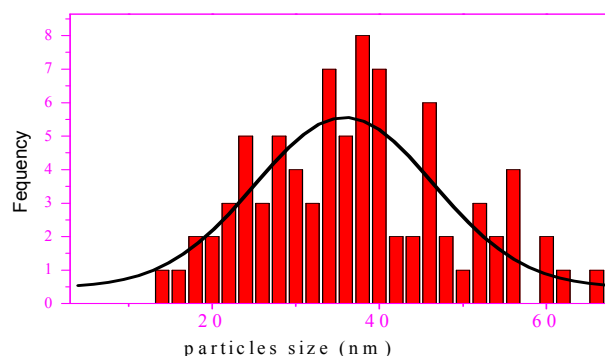
at a calcination temperatures of  $250^\circ\text{C}$ ,  $400^\circ\text{C}$  and  $600^\circ\text{C}$  are  $\pm 0.25$  nm,  $\pm 0.41$  nm and  $\pm 2.03$  nm respectively. Again, from figures 5, 6 and 7, we observe that the particles size increase with increasing calcination temperature.



**Figure 5.** Particles size distribution of synthesized  $\text{TiO}_2$  nanomaterials at calcination temperature of  $250^\circ\text{C}$



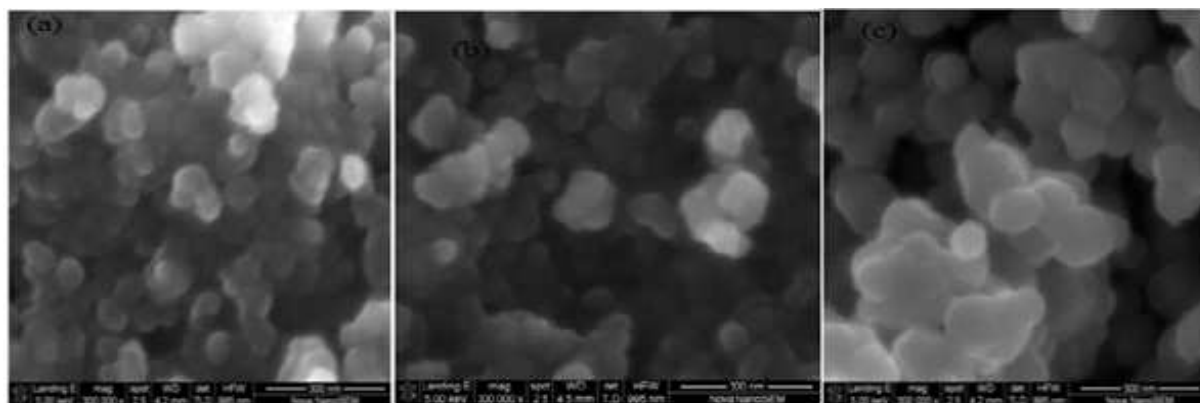
**Figure 6.** Particles size distribution of synthesized  $\text{TiO}_2$  nanomaterials at calcination temperature of  $400^\circ\text{C}$



**Figure 7.** Particles size distribution of synthesized  $\text{TiO}_2$  nanomaterials at calcination temperature of  $600^\circ\text{C}$

### 3.5. Scanning Electron Microscope (SEM)

Scanning electron microscopy (SEM) studies were used to examine morphology and shape of nanomaterials. The EDS (Energy dispersive x-ray spectroscopy) was connected to the SEM machine and used to study the elemental composition of our sample.



**Figure 8.** The SEM images of the synthesized  $\text{TiO}_2$  nanomaterials (a) calcination of  $250^\circ\text{C}$ , (b) calcination of  $400^\circ\text{C}$  and (c) calcination of  $600^\circ\text{C}$

As shown from figure 8, as the calcination temperatures increases the size of the nanoparticles is also increases. This figure shows high homogeneity emerged in sample by increasing the calcination temperature. Whatever the calcination temperature was low, the samples were less agglomerated. With increasing the calcination temperature boundaries between nanoparticles were better and the morphology of particles changed to spherical shape and the nanopowder were more agglomerated.

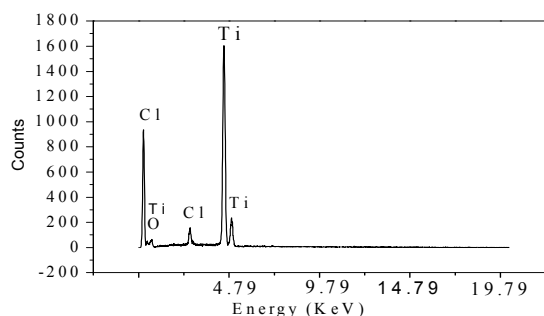
### 3.6. Energy Dispersive X-Ray Spectroscopy (EDS)

Energy dispersive x-ray spectroscopy (EDS) is an analytical technique used for the elemental analysis or chemical characterization of a sample. Energy Dispersive Spectroscopy (EDS) allows one to identify what those particular elements are and their relative proportions (Atomic percentage). EDS analysis usually involves the generation of an X-ray spectrum from the entire scan area of the SEM. The elemental analyses of the synthesized  $\text{TiO}_2$  nanomaterials are clearly shown below in figures 9, 10 and 11. The EDS results provided evidences that the

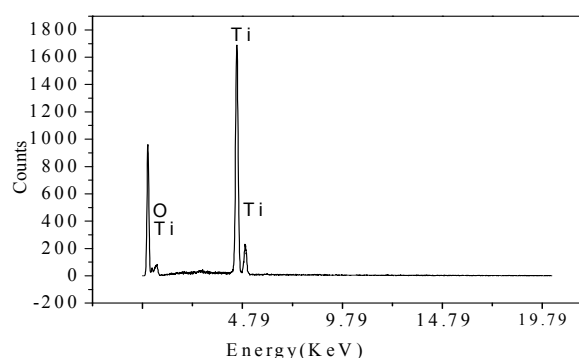
required phase of titanium (Ti), chlorine (Cl) and oxygen (O) were present in the sample at calcination temperature of  $250^\circ\text{C}$ . In addition to this, EDS results provided evidences that the required phase of titanium (Ti) and oxygen (O) were present in the sample at calcination temperatures of  $400^\circ\text{C}$  and  $600^\circ\text{C}$ . The figure 9 shows the presence of chlorine (Cl) phase in the sample at calcination temperature of  $250^\circ\text{C}$  as shown in table 2. This indicates that at low calcination temperature, the chlorine is not removed completely. The chlorine atom occurred in the sample comes from titanium tetra chloride ( $\text{TiCl}_4$ ) solution.

As observed from table 2, when the calcination temperature increases from  $250^\circ\text{C}$  to  $600^\circ\text{C}$ , the titanium atomic percentage yield decreases while the oxygen atomic percentage yield increases. In relation to this, the theoretical yields of titanium and oxygen are given by 33.33% and 66.67% respectively. Again from the same table 2 it is observed that the theoretical percentage yields of titanium and oxygen almost matches with percentage yields of titanium and oxygen obtained at calcination temperature  $400^\circ\text{C}$ .

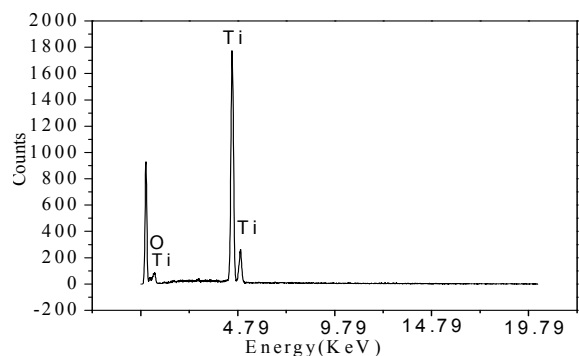




**Figure 9.** The energy dispersive x-ray spectroscopy (EDS) spectrum of  $\text{TiO}_2$  nanomaterials calcination temperature at  $250^\circ\text{C}$



**Figure 10.** The energy dispersive x-ray spectroscopy (EDS) spectrum of  $\text{TiO}_2$  nanomaterials calcination at temperature at  $400^\circ\text{C}$



**Figure 11.** The energy dispersive x-ray spectroscopy (EDS) spectrum of  $\text{TiO}_2$  nanomaterials calcination temperature at  $600^\circ\text{C}$

**Table 2.** The energy dispersive x-ray spectroscopy (EDS) spectrum of  $\text{TiO}_2$  nanomaterial results in atomic percent calcination temperatures at  $250^\circ\text{C}$ ,  $400^\circ\text{C}$  and  $600^\circ\text{C}$

Calcinations temperature of samples	Results in atomic percentage		
	Ti	O	Cl
Atom			
$250^\circ\text{C}$	33.34	64.02	2.64
$400^\circ\text{C}$	32.6	67.4	0
$600^\circ\text{C}$	31.89	68.11	0

## 4. Conclusions

$\text{TiO}_2$  nanomaterials were synthesized using the most convenient ways of synthesizing method known as sol gel synthesis. This method is an easy fabrication method due to

low cost and is done at low temperature. This method has various advantages over other methods, such as coprecipitation, introducing dopant, molecular scale mixing, and high purity of the precursors and homogeneity of sol-gel products with high purity physical, morphological and chemical properties.

XRD results showed that when the calcination temperatures increase, the FWHM decrease, the particles size increase and for the three selected temperatures the interesting peak value ( $2\theta$ ) are the same ( $25.31^\circ$ ). As observed from the result of TEM and XRD, in this project work, anatase  $\text{TiO}_2$  nanomaterial is obtained. UV-Vis spectrometers clearly show that the wavelength corresponding to the absorbance edge of  $\text{TiO}_2$  nanomaterials with calcination temperatures  $250^\circ\text{C}$ ,  $400^\circ\text{C}$  and  $600^\circ\text{C}$  is the same (350 nm) and the corresponding energy is 3.54 eV.

Transmission electron microscopy clearly showed that the produced micrographs of  $\text{TiO}_2$  nanomaterials size and shape are polydisperse. The synthesized  $\text{TiO}_2$  nanopowder average particle sizes obtained from TEM micrographs with calcination temperatures at  $250^\circ\text{C}$ ,  $400^\circ\text{C}$  and  $600^\circ\text{C}$  were found to be  $(8.55 \pm 0.25)$  nm,  $(13.82 \pm 0.41)$  nm and  $(36.06 \pm 2.03)$  nm respectively. SEM image clearly showed that the shape of synthesized  $\text{TiO}_2$  nanomaterials is polygonal structural morphology with small agglomeration that is when temperature increase. EDS results show that at low calcination temperature ( $250^\circ\text{C}$ ), the chlorine is not removed completely. The atomic percentage yield of oxygen increases while atomic percentage yield of titanium decreases with increasing calcination temperatures.

Generally, as has been observed so far from XRD and TEM, when the calcination temperature increases, the size of  $\text{TiO}_2$  nanomaterial increases.

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