



Publisher homepage: www.universepg.com, ISSN: 2707-4625 (Online) & 2707-4617 (Print)

<https://doi.org/10.34104/ijmms.022.075082>

International Journal of Material and Mathematical Sciences

Journal homepage: www.universepg.com/journal/ijmms

International Journal of
**Material and
Mathematical Sciences**



Synthesis of Titanium Dioxide by Sol-Gel Method and Comparison with Titanium Dioxide Pure

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ABSTRACT

This paper study the synthesis of the TiO₂ nanoparticles using the sol-gel method and TiCl₄ as raw material (TiO₂ sol-gel) and characterize it and TiO₂ pure by Powder X-Ray Diffraction (XRD), both samples show they are anatase phase with high crystallinity and purity. Fourier transform infrared spectrum (FTIR) both samples it showed in the graph that TiO₂ pure, TiO₂ sol-gel various frequency vibrations which are shown by different peaks formed, The specific surface area and porosity of the TiO₂ pure (46.962 m²/g) and TiO₂ sol-gel (38.264 m²/g) were evaluated by using the nitrogen adsorption and desorption isotherms by using Brunauer-Emmett-Teller (BET) method. Using Ocean Optio USB- 2000 spectrometer for optical properties which are related to the absorption spectrum but using diffuse reflectance spectrum in state of the Kubelka-Munk remission function replaces the Lambert-Beer law.

Keywords: Sol-gel method, Ocean Optio USB-2000 spectrometer, and Diffuse reflectance spectrum.

INTRODUCTION:

Nanotechnology is a broad and intense area of research and development that has been growing worldwide in the past decade. Nano-materials are substances whose dimension is less than approximately 100 nanometers (Hayle, 2014). Nanocrystalline semiconductors have achieved great importance in the industrial world today (Wang, 2010; Qu, 2013) which include metal oxides (ZnO, V₂O₅, Fe₂O₃, SnO₂, CdO, and TiO₂) and metal sulphide (CdS and ZnS) (Karunakaran, 2004). Titanium dioxide is an important semiconductor material due to its properties such as cheap, nontoxic, photo-chemical and chemical stability, high refractive index, wide band gap (Cassano, 2000; Wang, 2010). Soit was applied in environmental protection, paint, toothpaste, UV protection, photo-catalysis, photo-voltaic's, electrochromics, gas sensing, dye-sensitized solar cells, photo-induced hydrophilicity, and soon (Liu, 2007;

Chen, 2006; 2012; Ohenoja, 2013). Titanium dioxide crystallized in four natural phases: brookite (orthorhombic), anatase (tetragonal), rutile (tetragonal) and TiO₂ (B) (monoclinic). The three crystal structures of TiO₂ are shown in **Fig. 1**. Besides these polymorphs two additional high- pressure forms have been synthesized from rutile; the first form is TiO₂ (II) with a PdO₂ structure (Simons, 1967) and the second form is TiO₂ (H) with a hollandite structure (Latroche, 1989; Yousif and Ali, 2021).

Among the titanium dioxide forms rutile phase is the most thermodynamically stable whereas brookite are transformed to rutile on heating (Ohenoja, 2013). Various investigations have established that TiO₂ is much more effective as a photo-catalyst in the form of nanoparticles than in bulk powder. During the past two decades, several synthetic methods have been used to prepare TiO₂ nanoparticles (Su, 2006).

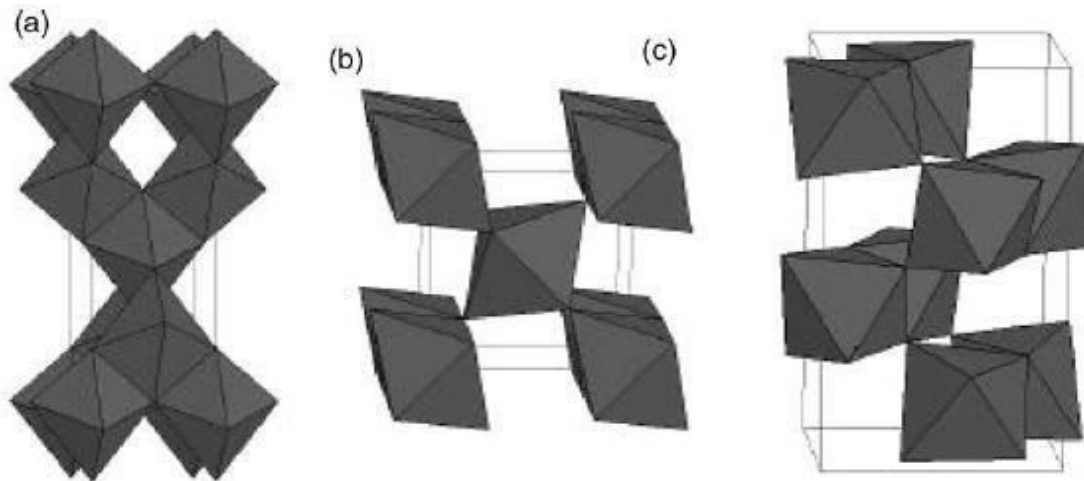


Fig. 1: Crystal structures of (a) TiO₂anatase, (b) brookite, and (c) rutile.

Generally, the methods of preparing nano-TiO₂ are gas methods (chemical vapor deposition CVD, physical vapor deposition PVD and spray pyrolysis) and liquid methods (co-precipitation, solvothermal, microemulsion, combustion synthesis, electrochemical synthesis, sol-gel methods) (Carp, 2004).

Liquid methods are often used because they do not need any special equipment in comparison with gas methods (Chen, 2006). Properties of nano-TiO₂ particles are dependent on synthesis method which is responsible for their chemical purity and its crystal form (anatase or rutile) also on various particle features (specific surface area and particle size), crystallinity and surface chemistry (Carp, 2004). Among these methods, sol-gel method is regarded as good method for synthesis ultra-fine metallic oxide and has been widely employed for preparing titanium dioxide nanoparticles (Su, 2006).

The sol-gel method has many advantages such as purity, stoichiometry control, ease of processing, and control over composition (Carp, 2004). The usually sol-gel procedure includes three steps; hydrolysis, drying, and calcination. There are two types of titanium sources for preparation of nano-TiO₂ particles which include organic titanium alkoxide (titanium iso-propoxide Ti(i-OP)₄, Titanium ethoxide Ti(OE)₄, etc.) and inorganic titanate (titanium tetrachloride TiCl₄, titanium trichloride TiCl₃ or Titanium sulphate TiO(SO₄)₂) (Carp, 2004). When using Titanium alkoxide expensive chemicals must be used and the hydrolysis process is difficult to control; otherwise, tetrachloride TiCl₄ is cheap, easy control of hydrolysis (Zhu, 2000; Chen, 2006). Titanium dioxide has been prepared in different

morphologies powder, thin film, nanotubes and nanorods (Burda, 2005; Banu *et al.*, 2021).

The sol-gel method was used for the synthesis titanium dioxide nano particle with using titanium tetrachloride as a precursor. And study its and titanium dioxide pure properties by using X-Ray Diffraction (XRD), micrometric gas adsorption analyzer by using Brunauer-Emmett-Teller (BET) method, Ocean Optic-USB-2000 spectrometer.

MATERIALS AND METHODS:

Pure Titanium dioxide from Aldrich, Titanium tetrachloride TiCl₄ (M_{wt} 189.87gmol⁻¹, 99.9%), absolute Ethanol, Ammonium hydroxide (M_{wt} 17.03gmol⁻¹, 25%), di-ionized water, silver nitrate (M_{wt} 169.87 gmol⁻¹, 98.4%).

Preparation catalyst

A sample of titanium dioxide nanoparticles was synthesized by using the Sol-gel method, 30cm³ of titanium tetra chloride was added to 500 cm³ di-ionized water in an ice bath under fume-hood followed by the addition 350 cm³ of ethanol with vigorous stirring for half of the hour at room temperature. Justified the pH to 8 by adding ammonia solution 25% drop by drop and precipitate was obtained.

After stirring vigorously then the solution was made to settle for 24 hours. Then the precipitate was separated by using a centrifuge and washed to remove chloride ions using silver nitrate to detect that then the precipitate was dried at 200°C for two hours and an amorphous TiO₂ was obtained. At last obtained the powder of titanium di-oxide nanoparticles (Uddin *et al.*, 2021; Hayle, 2014)

Methods of Characterization of TiO₂ Nanomaterials

There are many methods to characterize TiO₂ nano-material. In this study used X-ray powder diffraction (XRD) was performed using PAN-alytical’s X-ray diffract meter (Cu X-ray λ = 1.540598Å) over a range (10-60)° 2θ the powdered sample was packed on the sample holder by back pressure technique and mounted on the sample stage of the machine then determination the particles structure crystal size from the XRD pattern.

The crystallite sizes of the TiO₂ nano-materials were estimated using Scherer equation (1) (Han, 2012; Hayle, 2014).

$$d = \frac{\lambda K}{\beta \cos \theta} \quad (1)$$

Where d is the crystallite size in nanometer, K is the shape factor constant, which is 0.89, β is the full width at half maximum (FWHM) in radian, λ is the wave length of the X-ray which is 0.1540598nm for Cutarge tKα radiation and is the Bragg angle, Micrometric gas adsorption analyzer.

The specific surface area and porosity of the two samples were evaluated by using the nitrogen pressure sata constant temperature (typically liquid N₂, 77K) the sample loaded (0.1036g of sample 1, 0.60

$$slope = \frac{C - 1}{W_m C}, Intercept = \frac{1}{W_m C} \quad (2)$$

$$\frac{1}{W((P_0 / P) - 1)} = \frac{1}{W_m C} + \frac{C-1}{W_m C} \left(\frac{P}{P_0} \right) \quad (3)$$

Where W = weight of gas adsorbed, P/P₀ =relative pressure, W_m = weight of adsorbate as monolayer, C = BET constant. Using FTIR-8400S Fourier transform infrared spectroscopy to analyzed the both samples.

The samples were grind down with KBr and press to make disk and insert into the sample holder then take the FTIR spectra in range (400 – 3000cm⁻¹).

98g of sample 2) (Analysis Time is 281.6 min for sample 1,571.2 min for sample 2) then nitrogen adsorption isotherm of both samples were analyzed for the specific surface area using the BET equation (3) which required a linear plot of 1/[W(P/P₀)-1] against P/P₀.

Ocean optio USB2000 Spectrometer stat up by connect it to desktop PC via USB port or serial port, Ocean Optics OOI Base32 software application installed and configured for use with the USB 2000 OOIBase32 can use to get spectroscopic measurements (such as absorbance, reflectance, and emission) .

At first stored reference and dark measurements then the light transmits through an optical fiber to the sample. There is another optical fiber collects and transmits the result of the interaction to the spectrometer, then OOIBase32 compares the sample to the reference measurement and displays the spectral information. In reflectance spectroscopy, the Kubelka-Munk remission function F(R) replaces the Lambert-Beer law (Sikorska, 2003).

$$F(R) = \frac{(1-R)^2}{2R} = \frac{K}{S} \quad (4)$$

RESULTS AND DISCUSSION:

Sol-gel TiO₂ catalyst

When TiCl₄ solution was added to two of di-ionized water under fume hood, an exothermic reaction was observed. Then, a white precipitate was obtained after adding a drop of ammonium hydroxide (NH₄OH) wisely; and the yellow gel rose this indicates the formation of Ti(OH)₄.

After stirring the solution settling and centrifuging and drying the precipitate which leads to the formation of white amorphous TiO₂; then cooling, grinding and calcination lead to the form-ation of white powder TiO₂ nanoparticles. The Weight of precipitate before calcimined is 19.2005g. The weight of precipitate after calcinated is 14.4458g.

Characterization of the catalysts

As can be observed from Fig. (1 and 2), the XRD peaks in the angle range of 20° < 2θ < 60° for TiO₂ (pure) which are 25.4039°, 36.9877°, 37.8628°, 38.6280°, 48.0839°, 53.9365° and 55.1030°an for TiO₂(sol-gel) which are 25.3275°, 37.8174°, 48.

0607°, 53.8852°, and 55.0714° among the XRD peaks. The width of 25.4039° for TiO₂ (pure) and 25.3275° for TiO₂ (sol-gel) are useful peak since they have a high intensity which in turn are used to

determine crystals size. According to (Hayle, 2014) the peaks values correspond to the tetragonal anatase phase. Using equation (1) the estimated crystalline size is explored in the following table.

Table 1: Crystallite size (d) of pure TiO₂ & sol-gel TiO₂ nano-materials.

Sample	2θ°	FWHM radians	Crystalline size Nm
TiO ₂ (pure)	25.4039	0.0030908	49.11070
TiO ₂ (sol-gel)	25.0819	0.0068696	22.08194

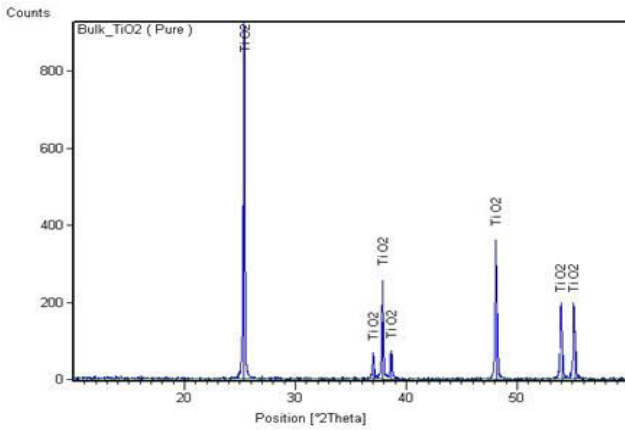


Fig. 1: The XRD pattern of TiO₂ (pure).

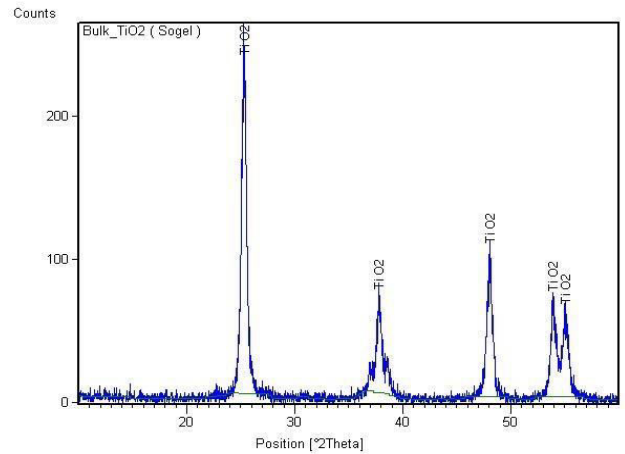


Fig. 2: The XRD pattern of TiO₂ (sol-gel).

A sharp increase in adsorption volume of N₂ was observed and located in the P/P₀ range of (0.067 - 0.88), (0.36 - 0.89) for TiO₂ pure and TiO₂ sol-gel respectively. The specific surface area value of the TiO₂ pure is 46.962 m²/g and the specific surface area value of the TiO₂ sol-gel is 38.264 m²/g (Govindaraj, 2015).

Micrometric gas adsorption analyzer

Fig. (3a, b) is shown the nitrogen adsorption and desorption isotherm which exhibits a type IV pattern with hysteresis loop characteristic of mesoporous material according to the classification of IUPAC.

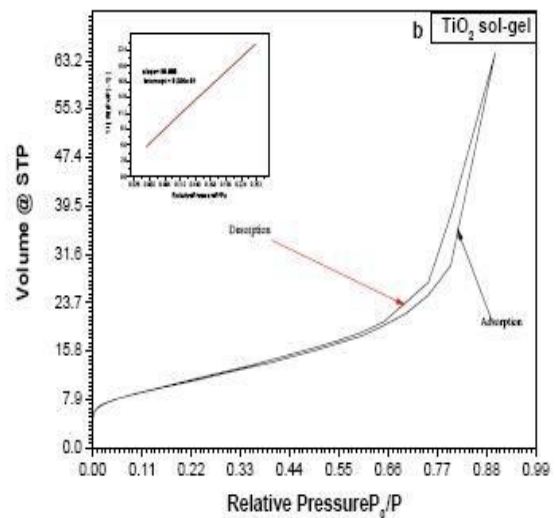
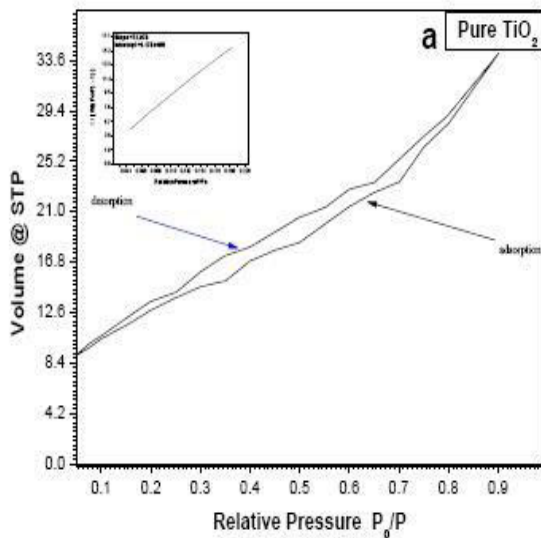


Fig. 3: a. Nitrogen Adsorption-Desorption isotherm of pure TiO₂ b. Nitrogen Adsorption-Desorption isotherm of TiO₂ sol-gel.

Fourier transforms infrared (FTIR) spectroscopy

In this graph different peaks formed at different wave numbers (400-3000cm⁻¹). It is noticed that TiO₂ pure, TiO₂ sol-gel various frequency vibrations which are shown by different peaks formed. The

peak in the range (400 – 800cm⁻¹) was characteristic Ti-O. The peak in range 1426-1696cm⁻¹ was characteristic of the O-Ti-O bond and Ti=O bending region. In Fig. 4b there is the peak in the range (2000-3000) is characteristic O-H (Grujić, 2006).

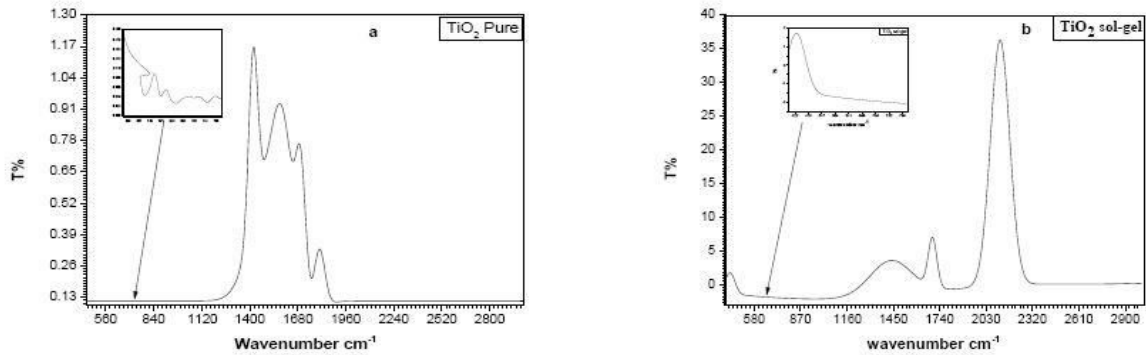


Fig. 4: (a). The FTIR spectra of TiO₂ pure; (b). The FTIR spectra of TiO₂ sol-gel.

Optical properties using USB 2000 spectrometer

Fig. 5 displays the reflectance spectra of TiO₂ pure which has a maximum value at region (371-408nm), (483- 506nm) and above 650nm. We noticed the reflectance spectrum is decreased in region (410-629). The reflectance spectra of TiO₂ sol-gel it. Has a maximum value at region (373-694nm) the curve s reach’s saturation above 669nm. We noticed the reflectance spectra are decreased in the region (520-

540nm),(576 - 584) and (657- 690nm).

The optical absorption coefficient, α , which is the relative rate of decrease in light intensity along its path of propagation, was calculated using equation (5) from the transmittance and reflectance data in the wavelength range 300-800nm. The nature of the optically induced transitions was determined from these data (Baydogan, 2013; and Rabeh, 2014).

$$\alpha(h\nu) = \frac{1}{d} \left[\frac{(1 - R)^2}{T} \right] \quad (5)$$

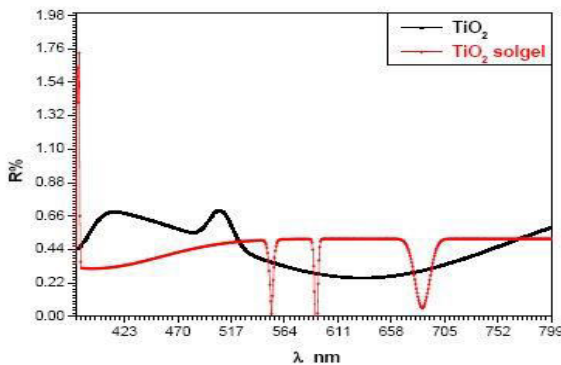


Fig. 5: The reflectance spectra of (TiO₂pure) and TiO₂sol-gel.

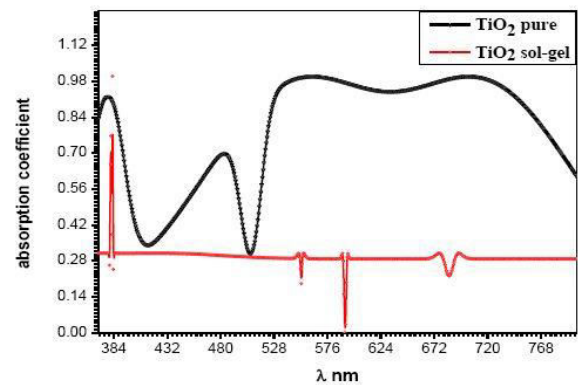


Fig. 7: The optical absorption coefficient of TiO₂ pure and TiO₂sol-gel.

Where T is the transmittance, R is the reflectance, and d is the sample thickness. The optical absorption coefficient was found to be exponentially dependent on the photon energy. However, when the particle size decreases the optical absorption coefficient will increase **Fig.7** show that TiO₂ sol-gel has a high value of 0.99 at 384nm and TiO₂ pure has 0.92 at 379nm.

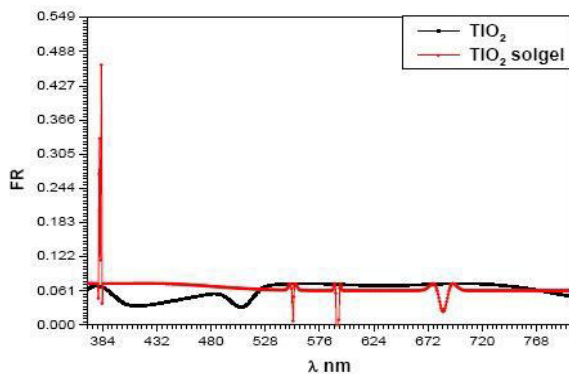


Fig. 6: Diffuse reflectance spectrum of TiO₂ pure and TiO₂ sol-gel with Kubelka-Monk conversion.

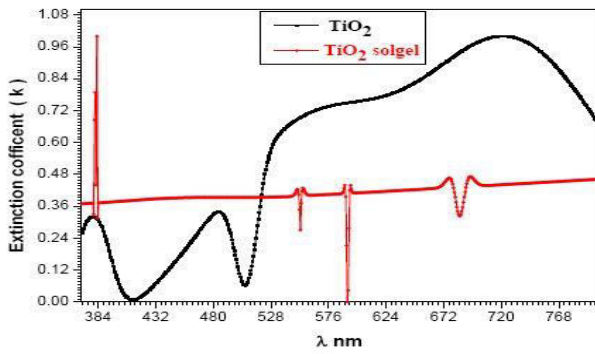


Fig. 8: The extinction coefficient TiO₂ pure and TiO₂sol-gel.

The extinction coefficient which is shown in Fig. 8 was calculated using equation (6)

$$k = \frac{\alpha\lambda}{4\pi} \quad (6)$$

Where k is the extinction coefficient, α is the absorption co-efficient and λ is the wavelength. The difference in values of extinction coefficient for two samples TiO₂ pure (0.32), and TiO₂ sol-gel due to that TiO₂ sol-gel has a small particle size. The refractive index determined using transmittance and reflectance measurements from Fig. 9 is noticed that there refractive index has a maximum value of 11 at wavelength 415 nm for TiO₂ pure and 16.3 at wavelength 384 nm for TiO₂ sol-gel which is due to interactions takes place between photons and electrons. The refractive index changes with the variety of the wavelength of the incident light beam due to these interactions, i.e. the optical loss caused by absorption and scattering (Baydogan, 2013).

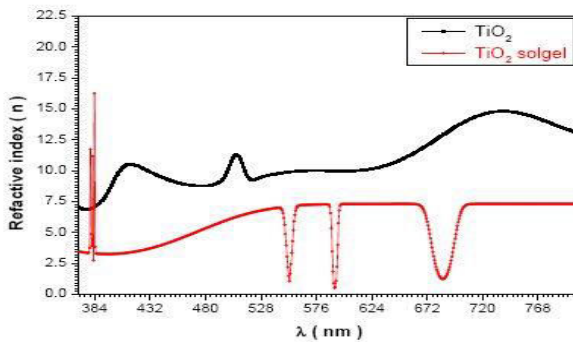


Fig. 9: The refractive index of TiO₂ pure and TiO₂sol-gel.

The energy band gap of these materials is determined using the reflection spectra. According to the Tauc relation, the absorption coefficient is given by Sharma equation (7) (Rabeh, 2014).

$$\alpha(h\nu) = (B - E_g)^n \quad (7)$$

Where E_g is the energy gap, B constant is different for different transitions, (hν) is the energy of photon and n is an index which expects the values 1/2, 3/2, 2, and 3 depending on the nature of the electronic transition responsible for the reflection. The exponent r = 1/2, 3/2 for indirect transition is allowed or forbidden in the quantum mechanical sense, and r=2, 3 for allowed and forbidden direct transition, respectively. From Fig. 10 the energy band gap is 3.07 eV for TiO₂ pure and 3.23 for TiO₂ sol-gel (Baydogan, 2013; Rabeh, 2014).

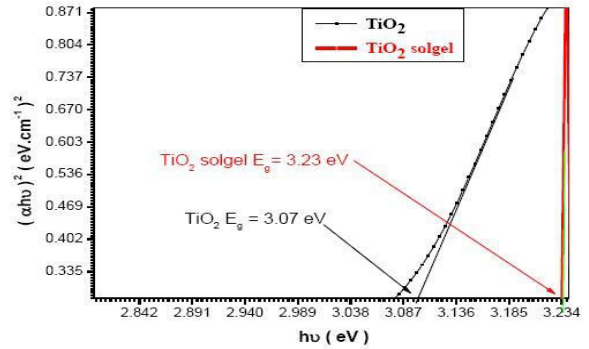


Fig. 10: The optical energy gap spectrum for TiO₂ pure and TiO₂sol-gel.

CONCLUSION:

TiO₂ nanomaterial was prepared by using the most convenient ways of synthesizing method known as sol gel synthesis. Sol-gel is and easy method due to low cost and is done at low temperature. XRD results exhibited the interesting peak value are (25.4039°, 25.3275°) which is anatase TiO₂ nano-material due to (49.11070, 22.08194nm) for TiO₂ pure and TiO₂ sol-gel respectively. The specific surface area and porosity of theTiO₂ pure and TiO₂ sol-gel were evaluated by using the nitrogen adsorption and desorption isotherms, the specific surface area of the TiO₂ pure are 46.962 m²/g and The specific surface area of the TiO₂ sol-gel are38.264 m²/g. The isotherm exhibits a type IV pattern with a hysteresis loop. From the FTIR spectrum both samples observed that TiO₂ pure, TiO₂ sol-gel various frequency vibrations which are shown by different peaks formed. Optical properties are studied by using the Diffuse reflectance spectrum in which the Kubelka - Munk remission function replaces the Lambert-Beer law. TiO₂sol-gel has an optical absorption coefficient (0.99), extinction coefficient (1), a refractive index 16.3 and the energy band gap of 3.23eV while TiO₂ pure has optical absorption coefficient of 0.92, extinction coefficient (0.32), there refractive index 11 and the energy band gap 3.03eV.

ACKNOWLEDGMENT:

Express gratitude to all who encouragement and helping. Thank Industrial Research and Consulting Center and especially thank Dr. Abd Elsakhi for his help.

CONFLICTS OF INTEREST:

The authors declare that the manuscript has no competing interests to the publication.

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Citation: Elrazeg YA, Badiruzzaman M, Islam M, Morshed MM, and Elmugdad AA. (2022). Synthesis of titanium dioxide by sol-gel method and comparison with titanium dioxide pure, *Int. J. Mat. Math. Sci.*, **4**(3), 75-82. <https://doi.org/10.34104/ijmms.022.075082> 