Facile method to synthesis of anatase TiO\(_2\) nanorods

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Abstract. A simple wet chemical method was used to synthesise TiO\(_2\) nanorods using Titanium isopropoxide (TTIP) as a precursor material. The chemical transformation of TTIP by ethylene glycol (EG) was demonstrated as a strategy for regulating and controlling the shape of formation TiO\(_2\) nanorods. The structure of the sample was studied by X-ray diffraction and it revealed that the prepared TiO\(_2\) exhibit a pure anatase phase. While the Fourier transfom infrared spectroscopy (FTIR) was showing the vibration pattern in the spectrum of the sample. The morphology of sample was studied by scanning electron microscopy (SEM) and it showed that the synthesised TiO\(_2\) made of nanorods with length about (698 nm) and a diameter (220 nm).

1. Introduction
Titanium dioxide (TiO\(_2\)) is a semiconductor material that has a wide energy gap (3.2 eV) [1, 2]. Many studies have been focused in the present time on TiO\(_2\), due to its large surface area and characteristic electrical properties such as physical and chemical stability, high refractive index, low cost, non-toxicity and high photo-catalytic activities…etc. TiO\(_2\) can be found in three crystalline phases: anatase, rutile both tetragonal structure and brookite with orthorhombic structure. Each of these phases have various chemical and physical properties depending on the composition of the atoms, thus leads to a varied performance in its application [3, 4]. Anatase and brookite are metastable, while rutile phase is stable under certain conditions [5]. The anatase and brookite phases are easily turned to rutile phase during a high temperature [6, 7]. Different morphologies of TiO\(_2\) have been synthesized such as nanorods, nanowires, nanotubes, nanoparticles and nanobelts…etc. One dimension (1D) structures of TiO\(_2\) especially nanorods, nanowires and nanotubes have been attracted a lot of attention due to the unique properties and great applications comparison nanoparticles. TiO\(_2\) with (1D) structures have exhibited the faster process of generating electron-hole and a lower recombination rate comparison TiO\(_2\) nanoparticle [8, 9]. Therefore, TiO\(_2\) nanorods have been used in many applications such as self-cleaning coating [10], dye-sensitized solar cells [11], sensors [12], wastewater purification [13], lithium-ion batteries [14], electrochromic devices [15, 16]…etc. On the other hand the most important uses of TiO\(_2\) are in photocatalytic applications, due to the cheap and friendly to the environment and human [17, 18].

There are several methods to prepare TiO\(_2\) nanorods such as sol-gel [19], hydrothermal [20], microwave [21, 22], solvothermal [23] …etc. In this paper, anatase TiO\(_2\) nanorods with high purity
were synthesized via a simple wet chemical method from Titanium Isopropoxide and Ethylene Glycol at temperature 100 °C.

2. Experimental

2.1. Raw materials

All the chemicals used in the experiment are used with high purity. Titanium (IV) Isopropoxide (TTIP) Ti [OCH (CH₃)₂]₄, Ethylene Glycol (EG), absolute ethanol C₂H₅OH and deionized (DI) water were used in the TiO₂ preparation process.

2.2. Synthesis of TiO₂ powder

TiO₂ powder has synthesized by using the wet chemical method from (TTIP) and (EG) as a precursor materials. A specific amount (60 ml) of EG was placed in a glass vessel and heated up to a temperature 100 °C under magnetic vigorous stirring for 30 min. Next, 2 ml of TTIP was added dropwise into a vessel with vigorous stirring at 700 rpm. The maxing solution was kept under this condition of stirring for 2 h. When the titration process achieved, the resulting solution was changed from transparent color to a milky white, which indicates the formation of the TiO₂ nanoparticle.

After that, the precipitate was washed with deionized (DI) water and absolute ethanol several times, to eliminate any residual organic species remaining in the final products. The precipitate was then dried at temperature 60 °C overnight. Finally, the white fine powder was calcined at (450°C) in the air for 1h to obtain TiO₂ nanoparticle.

Different techniques were used to characterize the synthesized sample such as X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscope (SEM). The schematic diagram for the whole synthesis procedure and characterization of TiO₂ nanorods is shown in Figure 1.

**Figure 1.** Schematic diagram of the steps of TiO₂ preparation.
2.3. Characterization of the sample

The crystal structure of TiO$_2$ powder was studied using X-ray diffractometer (Shimadzu- Japan) model 6000 (CuKα1 radiation, λ = 1.54056 Å). While the chemical bonding on the sample was investigated using Fourier transform infrared spectroscopy (Shimadzu-Japan, FTIR- 8400s) with a single beam in the range between (4000-400) cm$^{-1}$. The morphology of TiO$_2$ powder was characterized by scanning electron microscopy SEM (Inspect 550, FEI company made in the Netherlands).

3. Result and discussion

The XRD patterns of synthesized TiO$_2$ at temperature 100 °C were shown in figure 2. The XRD diffraction of the prepared sample detects a sharp domain (101) peak at about 2θ=25.3° ascribed to anatase phase of TiO$_2$. Comparing the XRD pattern with the standard (JCPDS file No. 21-1272), it can be observed that there are no peaks of brookite or rutile phase in the pattern, indicating the high purity of the synthesized sample. The crystal size was calculated by applying the Debye-Scherrer equation (1) on diffraction peaks of anatase phases [24].

$$D = \frac{K\lambda}{\beta\cos\theta}$$  \hspace{1cm} (1)

where D is the mean crystallite size of the sample. K is the shape factor which varies depending on the actual shape of the crystallite, but typically has a value of 0.9. $\lambda$ is the X-ray wavelength, in this case, it was CuKα radiation ($\lambda = 1.54056$ Å$^1$). $\beta$ is the full width of the peak at half maximum (FWHM) in radians and $\theta$ is defined as Bragg angle.

The average crystallite sizes of the sample of the anatase TiO$_2$ prepared at 100 °C and calcined at 450 °C for 1h was appeared to be 10.76 nm.

![Figure 2. XRD pattern of the anatase TiO$_2$ nanorods sample.](image)

Figure 3 was shown the FTIR spectrum of the prepared anatase TiO$_2$. The broad and strong absorption band of IR spectrum between (3000-3500) cm$^{-1}$, was attributed to the stretching vibration of O-H groups which is due to absorption of water molecules during the post-installation period because of the higher surface area of interaction with the organic additives and also small particle size [25]. The weak bending vibration at (1651.12 cm$^{-1}$) and (2802.46 cm$^{-1}$) indicated the existence of water and C-H aldehydic respectively, which may be derived from the absorption of the products when exposed in the atmosphere. Another peak at (1384.94 cm$^{-1}$) attributed to carboxylate stretch with
symmetric vibration. While the absorption band below (950 cm\(^{-1}\)) attributed to the characteristic vibration of TiO\(_2\). The peaks at (736.83 cm\(^{-1}\)) and (538.16 cm\(^{-1}\)) were observed the growth of Ti-O-Ti linkage in spectrum [26].

**Figure 3.** FTIR spectrum of TiO\(_2\) nanorods.

The morphology of synthesized TiO\(_2\) nanoparticles was characteristic using SEM as shown in Figure 4. According to SEM image, it was observed that the synthesised TiO\(_2\) have uniform rod-like shapes with the average length and diameter about 698 nm and 220 nm respectively.

**Figure 4.** Shows SEM image of TiO\(_2\) nanorods prepared at 100 ºC.
4. Conclusions
In this study, a simple wet chemical method was used for preparation TiO$_2$ nanorods using Titanium (IV) Isopropoxide as a precursor and Ethylene Glycol as a solvent. The X-ray diffractions results detected that TiO$_2$ nanoparticles are highly crystalline with anatase phase only. FTIR results have clarified that Ti-O bonds formed between 400-900 cm$^{-1}$ in the spectrum. SEM image was showed that the morphology of prepared TiO$_2$ in nanorods shape with an average length (698 nm) and diameter of (220 nm).

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