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# Synthesis of TiO2 Thin Films Nanoparticles with Different Layers using Simple Sol-Gel Method

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**Abstract:**
In this study, titanium dioxide ($TiO_2$) are synthesized by sol– gel simple method. Thin films of sol, gel, and sol- gel on relatively flat glass substrates are applied with Spin coating technique with multilayers. The optical and morphological properties (studied using AFM) of $TiO_2$ layers show good properties, with particles diameters less than 4 nm for all prepared samples and have maximum length 62 nm for $TiO_2$ gel thin films of three layers. The results show low roughness values for all films especially for 4 layers sol (8.37nm), which improve the application in dye sensitive solar cell (DSSc) .

**Keywords:** Sol-Gel, TiO2, AFM, Morphology, Roughness.

# تحضير اغشية رقيقة متعددة الطبقات لمادة ثاني اوكسيد التيتانيوم النانوية بطريقة السول جل البسيطة

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**الخلاصة**
باستخدام طريقة السول–جل البسيطة تم تحضير ثاني اوكسيد التيتانيوم.الافلام المحضرة بطريقة السول والجل والسول–جل تم ترسيبها على الزجاج بطريقة الطلاء المغزلي ولطبقات متعددة. اظهرت الخصائص البصرية وخصائص التشكيل(المورفولوجي) جودة عالية حيث بينت التقارير والصور الناتجة ان قطر الجسيمات من التيتانيوم ولجميع النماذج المحضرة اقل من 4 نانومتر و اعلى قيمة لارتفاع الفلم 62 نانومتر, كما وان اقل خشونة سجلت لجميع النماذج وبالاخص لنموذج اربع طبقات سول ( 37, 8 نانومتر) .وهذه الميزات ذات فائدة كبيرة لاستخدامها في الخلايا الشمسية لمتحسسات الصبغة.

## 1.Introduction

Titanium dioxide($TiO_2$) is the most used material in the manufacturing of solar cells due to its photocatalytic and optical absorption properties. Titanium dioxide ($TiO_2$) has exceptional solidness and high band gap[1]. Temam et al. (2021) prepared $TiO_2$ thin films via a sol-gel dip-coating method. The effect of the photocatalysis process on the properties of $TiO_2$ thin films based on XRD results showed that un-doped $TiO_2$ thin films were grown with anatase phase, whereas, the Ni and Ni/Al-doped $TiO_2$ films show $Ti_4O_7$ single phase [2]. Chenaina et al. (2021) synthesized $SnO_2$- $TiO_2$ sol-gel-derived thin films deposited on a silicon wafer

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substrate, which were processed by spin coating technique. Heat treatments of the precipitated films were performed at four different temperatures (200, 400, 600 and 800 °C). First characterization via XRD determined that the nanocrystalline phase begins to form at 400 °C of the long-range order and becomes well crystallized to a larger size with increasing annealing temperature.[3]. Bansal et al. (2021) synthesized titanium dioxide nanoparticles ($TiO_2$ NPs) by sol-gel hydrothermal colloid in two different devices: One is simple stainless steel ?????? (named $TiO_2$-B) and the other is an autoclave of constant temperature and pressure (named $TiO_2$-W). It was observed that the quality of the as-synthesized $TiO_2$-W NPs was comparable with that of the commercial $TiO_2$ NPs (TiO2-C),. The as-synthesized $TiO_2$ NPs are used as a photoanode in dyestuff solar cell (DSSC). DSSC with $TiO_2$-W showed better energy conversion efficiency compared to DSSC with TiO2-B.[4] The photovoltaic (PV)gadget execution is predominantly characterized by its power conversion efficiency (PCE), which depends on few boundaries, for example, light harvesting properties of the dynamic layer, age of excitons, and exciton dispersion and separation[5]. Developing and combining distinctive fluorescein derivatives are as yet intriguing to meet a few necessities in various applications [6]. Novel prepared titanium dioxide ($TiO_2$) thin films were utilized as electron transport layer in P3HT:PCBM-based OSCs as a component of different temperature treatment [7]. Three types of titanium dioxide ($TiO_2$) can be prepared as: brookite, anatase and rutile. Rutile is the most stable type .Mostly, the arrangement-dependent preparation techniques of TiO2 show the anatase-type arrangement. Moreover, anatase particles are hard to synthesize, due to the change from anatase to rutile by increasing preparation times and/or temperature [8]. Sol- gel technique has many benefits like effortlessness, cost-effectiveness, incredible compositional control the arrangement of the atoms, and its homogeneity in low crystallization temperature and high yield [9]. The properties of the nanoparticle rely upon many factors such as primary morphology, surface area, porosity, etc. The nanoparticles can be synthesized by different strategies like sol- gel, flame pyrolysis, hydrothermal, and chemical reduction[10]. The recent advancement in material science enhances the orchestration of $TiO_2$ nanocrystals with required shape, which brings about possible materials with shape dependent properties. The surface energy of the nanocrystals can be enhanced or decreased by surfactant bond which can lead to anisotropic nanocrystal growth [11]. The sol–gel method is particularly compatible for the preparation of thin films that require superb homogeneity, virtue, and consistency on any substrates types [12]. It is the most suitable procedure to develop thin films. It has enormous number of advantages over other techniques [13]. Several examinations have been done on composites made with polymers and nano-metal oxide such as titanium dioxide (TiO2) [14]. Electrospinning enjoys a few benefits, for example, huge surface area to volume proportion, high explicit surface region and little pore size, unrivaled mechanical properties and flexibility in surface functionalities[15].

## 2. Experimental

### 2.1 Materials:

TriTitanium chloride $TiCl_3$ (99.0%BDH), hydrochloric acid HCl (33%), Hydrogen peroxide $H_2O_2$ (35 w% aqueous solution)and Acetic acid($CH_3COOH$ 99.8%) were purchased from Sigma Aldrich and used without any further purification.

### 2.2 Preparation Method:

Deionized (DI) water (50 ml) was placed on a hotplate stirrer and 5ml of HCl was added dropwise with stirring. After that, 25ml of $TiCl_3$ was added very slowly to the solution until it turned white in color (Figure 1a). Then, 50 ml of Acetic acid was added to the white solution with constant stirring until the solution became violet (Figure 1b). After 6 hours stirring at ($80^oC$), the solution turned transparent white, as shown in Figure 1c. In order to obtain the collided nanoparticle of $TiO_2$ (Gel): a solution was prepared from 50 ml of 28% concentration $NH_4OH$ with stirring until the pH changes to 8. Gel can be obtained from 4 ml hydrogen

peroxide ($H_2O_2$) of 35% concentration which was added slowly to the solution to increase the oxidation process and to increase the pH to 9. It was noticed that after adding the $NH_4OH$, the precipitant of $TiO_2$ was obtained.

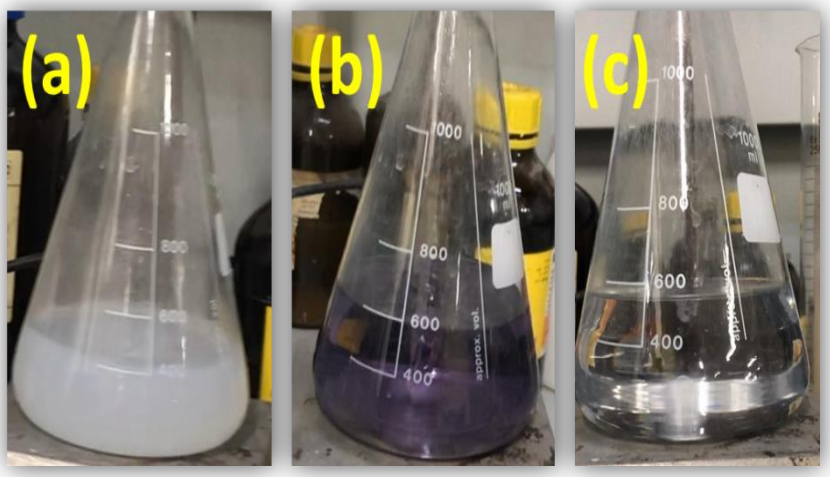


**Figure1**-Color change of the prepared solution (a) first process, (b) second reaction, (c) final approach.

The gel and sol $TiO_2$ thin films multilayers were prepared using spin coating method on glass substrates .Whereas, the single-layer TiO2 thin film was prepared by sol-gel technique. The transmittance curves were obtained with UV-Visible 1800 spectrometer, and AFM reports and images with scanning probe microscope AA3000.

**3. Results and discussions:**

**3.1Optical properties:**

Transmittance spectra for the prepared $TiO_2$ thin films with different layers of gel and sol , showed the same value of transmittance for all sol layers and wavelengths (Figure 2b) but they showed different transmittance values as function of wavelength for the gel layers (Figure 2a). Moreover, $TiO_2$ prepared from sol solution showed higher transmittance spectra compared with that of $TiO_2$ layers prepared from gel solution. One layer selection sol and one layer gel to companies to reach good features for prepared collides target ( Figure 2 ). The results demonstrated that increasing the $TiO_2$ thickness results in lower transmittance.

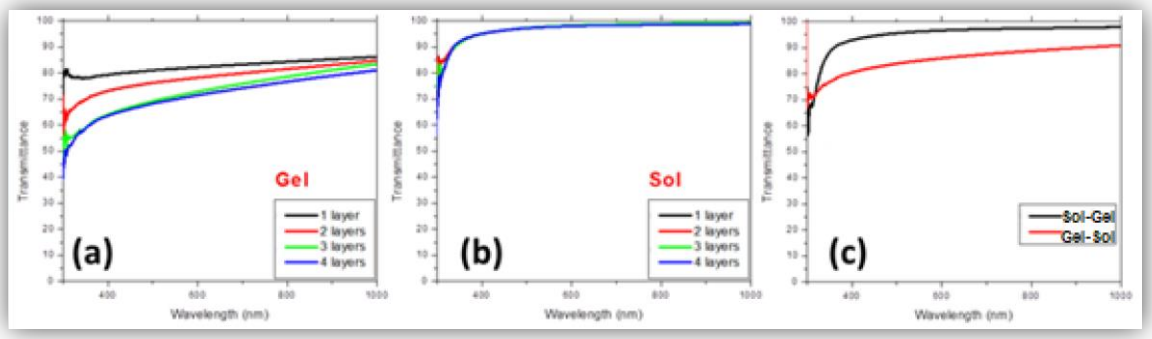


**Figure 2**-The transmitting spectrum (a) Gel(1-4layers), (b) Sol(1-4layers), and (c) Sol-Gel and Gel-Sol (1 layers)

3.2 **Morphological properties:**

The AFM images, shown in Figure 3, report the surface topography and morphology parameters ( average diameter of particles, average height, surface roughness and root mean square ) as listed in Table 1 and 2 for sol and for gel (1-4 layers), respectively. On the

other hand, Table 3 illustrates the AFM information for sol- gel one layer. AFM data demonstrates the surface roughness and morphology and the differences between the types and layers. Fig 3 shows the images of sol-gel one layer of TiO2 from which the morphology parameters can be deduced Figure 3a and 3b illustrates the two dimensions and three dimension AFM image of sol- gel one layer, respectively. Average particle diameters and average heights are shown in Figure 3c and 3d, respectively.

The tables show that all particles diameter are less than 4 nm, which is beneficial for the application as substrate for DSSC.

**Table 1-** AFM information of sol TiO2 thin film (1-4 layers)

| Sol layers | Avg. Diameter (nm) | Avg. Height (nm) | Roughness Average (nm) | Root Mean Square(nm) |
|---|---|---|---|---|
| 1 | 2.54 | 46.461 | 9.17 | 11.5 |
| 2 | 3.20 | 54.413 | 11 | 14.1 |
| 3 | 1.80 | 43.729 | 9.33 | 11.8 |
| 4 | 2.06 | 37.259 | 8.37 | 10.2 |
| 5 | 1.95 | 41.148 | 11.4 | 13.3 |

**Table 2-** AFM information of gel TiO2 thin film (1-4 layers)

| Gel layers | Avg. Diameter (nm) | Avg. Height (nm) | Roughness Average (nm) | Root Mean Square(nm) |
|---|---|---|---|---|
| 1 | 2.66 | 50.361 | 11.3 | 13.9 |
| 2 | 2.73 | 50.361 | 10.9 | 13.9 |
| 3 | 2.78 | 61.123 | 11.5 | 14.2 |
| 4 | 3.36 | 51.026 | 11.1 | 13.9 |
| 5 | 2.14 | 30.636 | 8.88 | 11.2 |

**Table 3-** AFM information of sol- gel TiO2 thin film (1:1)layers

| Sol-Gel layer | Avg. Diameter (nm) | Avg. Height (nm) | Roughness Average (nm) | (Root Mean Square(nm) |
|---|---|---|---|---|
| 1:1 | 2.36 | 34.273 | 9.04 | 10.6 |

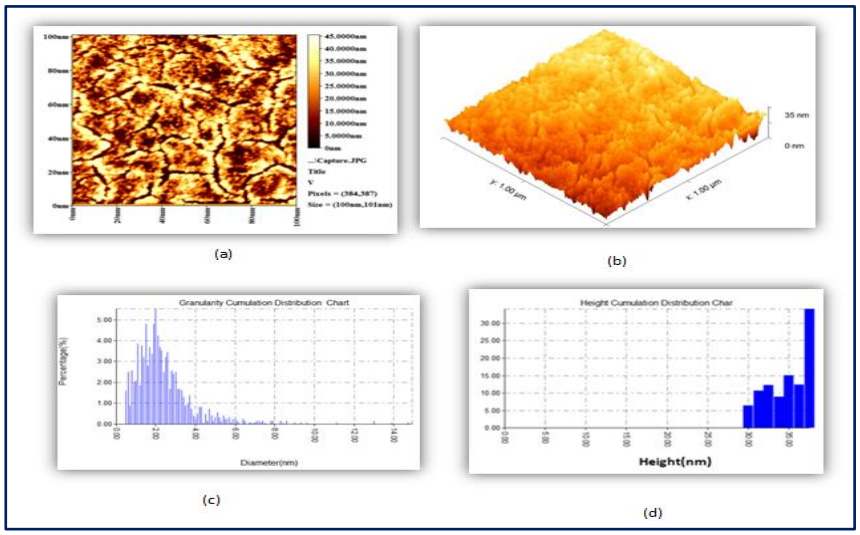


**Figure 3**-AFM images for the sol- gel preparations $TiO_2$ thin films

## Conclusions

Sol-gel is one of the widely used techniques for the preparation of nano-sized metallic oxide materials. The deposition was performed on glass substrates at room temperature. The

effect of the number of coatings and the prepared $TiO_2$ films with different layers from gel/ sol and sol/ .Periodically deposited thin films of two distinct materials have the ability to interact with light waves. The results showed that increasing the TiO2 thickness reduce transmittance. Furthermore, a higher transmittance spectrum was obtained from $TiO_2$ prepared from sol solution compared to that of TiO2 layers prepared from gel solution. All $TiO_2$ synthesized by sol- gel method have particle diameters less than 4 nm which is suitable for the dye-sensitized solar cell. .

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