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Study the Effect of TiO₂ Nanoparticles in Multilayers of Photoelectrode Prepared by Ball Milling Technique on The Performance of Dye Sensitized Solar Cells (DSSCs)

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Abstract. In this paper, dye sensitized solar cells (DSSCs) were prepared using nanoparticles of TiO₂ downscaled using ball milling technique. 8 hours milling time was used at a constant speed of up to 70% of the critical speed. The obtained average particle size of (2.8 nm) was examined by means of (Fe-SEM) electron microscopy images. Thin films of these particles were prepared, and dye-sensitized solar cells were made using (N719) dye. The effect of particle size on the performance of the conversion efficiency of DSSCs was studied by balancing light scattering and surface area. Large particles have a strong light-scattering lead to a significantly decreased surface area and small particles have a large surface area and weak light scattering. Therefore, it has been proposed to utilize multiple (mono, bi and trilayers) photoanode for dye-sensitized solar cells. The results showed that cell with trilayer photoanode has better efficiency than that of monolayers and bilayers. the conversion efficiency (0.9%) was obtained for the single layer and (1.01%) for the bilayer and (1.18%) for the trilayer.

1. Introduction

In recent years, the development of renewable energy sources is receiving much attention due to being emission-free, renewable, clean and environmentally friendly. In particular, sensitive dye solar cells (DSSCs) have attracted a lot of interest in the solar cell research community. Because of its low cost, ease of manufacturing process, good stability and high conversion efficiency [1-3]. DSSCs are mainly composed of the nanocrystalline photoanode that is deposited on the conductive glass substrate FTO, the sensitive-dye, the electrolyte solution (iodide / trioxide) and the counter electrode that is usually composed of (Pt or carbon). the photoanode plays an important role in determining the performance of solar cells by virtue of its role in capturing light and the process of charge transfer. Various semiconductors such as TiO₂, ZnO and SnO₂ etc. are used as photoanode in solar cells and have shown great photovoltaic performance [4,5]. by using (TiO₂) nanoparticles and multiple cell layers, high energy conversion efficiencies and various methods to continuously improve the performance of DSSCs can be achieved by researchers, such as developing dyes with a wide absorption range [6]. Increasing of the surface area of the nanoparticle [7], increasing the light-harvesting properties and controlling the particle size of the semiconducting (TiO₂) material, etc., leads to a significant improvement in the performance of solar cells [8].



The semiconductor material (TiO_2) is used as an electrode due to its unique properties, attracting a great deal of attention due to its low cost, non-toxicity, chemical and physical stability, and superior effectiveness [9].

In principle, the fast electron transfer and slow recombination would be better to obtain high conversion efficiency into DSSCs. For regular DSSCs, the (mesoporous) optical electrode is usually produced from containing TiO_2 nanocrystalline particles that provide a surface area for the purpose of absorbing light by the dye and facilitating electrolyte diffusion within these layers. Thus, improving the nanostructure of the (photoanode) is the target in most research for the purpose of its improvement [10]. Recently, the effect of light scattering on a (TiO_2) electrode has been proposed by (J. S. Im) and his group using particles of different sizes and multiple layers of (TiO_2), which can greatly improve the light-harvest efficiency [11]. Ferber, Luther [12], Rothenberger et al [13]. They confirmed that the effect of light scattering has a major role in the performance of solar cells' efficiency. Recently, there have been many efforts and focus to develop the nanostructure of TiO_2 particles and their use in the application of (DSSC) [14-17]. However, it lacks an examination of the effect of particle size. As it is known, whenever the size of the nanoparticles is small for the (TiO_2) layers, it provides a large surface area for the absorption of a large number of dye molecules and a low contact resistance, which leads to the expectation that the cell has a high (V_{oc}). This method is easy to achieve by adjusting the size of particles and components of the (TiO_2) paste [18]. The microstructure of TiO_2 thin films, such as the surface area, film porosity and particle size, all these factors greatly affect the conversion efficiency in solar cells, as nanoparticles (TiO_2) play an important role in the manufacture of high-efficiency solar cells. As the nanoparticles are necessary to increase the surface area, and thus good absorption of the dye particles, while the large size nanoparticles are required to enhance the absorption of red light through light scattering. Therefore, there must be a balance between the surface area and the light scattering. Therefore, a sophisticated multilayer structure was proposed with a gradual increase in particle size from the deepest layer to the outer layer to be tested [19].

In the current study, the effect of using different sizes of (TiO_2) particles obtained by using the ball milling method and making different layers (single, bi and tri) on the performance of DSSCs. The Ball Milling Method is a simple method for reducing particle size to different levels (nanoparticle). This method can be considered one of the effective mechanical methods and the grinding time has a very important role in this process. Wet milling was used in our study as the TiO_2 particles were ground in an oval mill in order to reduce their particle size (from micro to nanoscale).

2. The experimental part:

2.1 Synthesis of TiO_2 nanoparticles

The preparation process of titanium dioxide (TiO_2) nanoparticles is carried out by grinding with balls shown in "figure 1" by using (TiO_2) with high purity (99.8%). The grinding device that consists of a cylinder or an oval container that has a solid wall made of steel and is stainless and many small balls made of iron and the speed control device, which is an electric motor that is controlled using an external load resistance. TiO_2 with micro-particles was putting inside the container using the ball milling technique in order to obtain the nanoscale sizes as the milling process takes place from top to bottom (Top-Down process), and this process was done according to A milling time of 8 hours at a constant speed was determined according to 70% of the critical speed (V_c), as the grinding process operates on the principle of "Critical Speed" [20], based on the equation shown below.

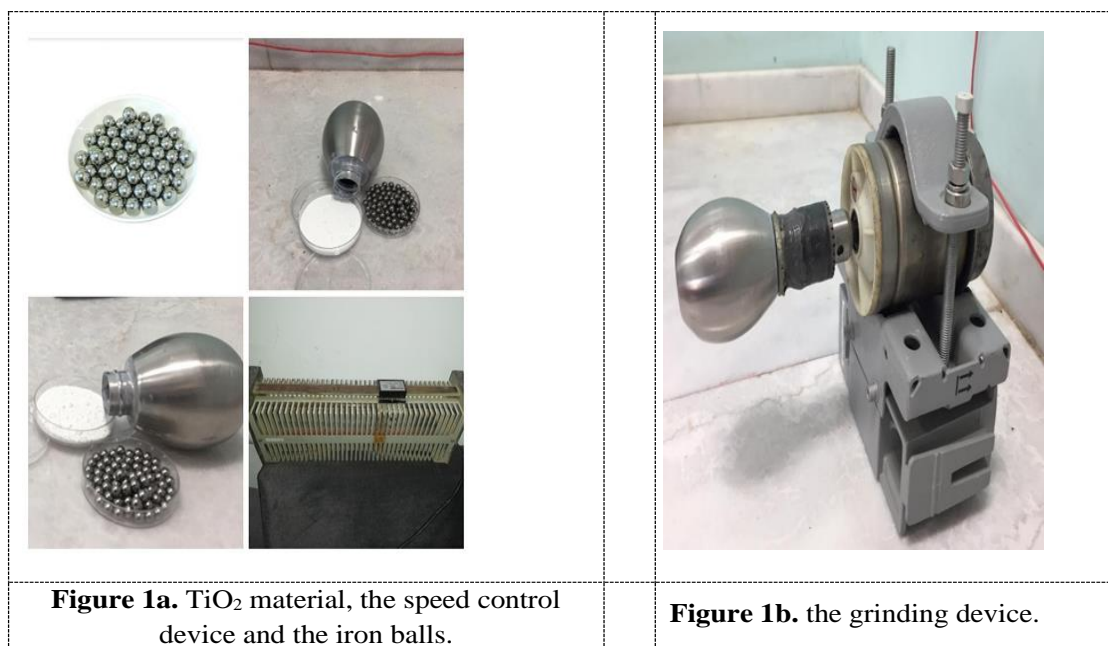


Figure 1a. TiO₂ material, the speed control device and the iron balls.

Figure 1b. the grinding device.

$$V_c = \frac{1}{2\pi\sqrt{\frac{g}{R-r}}} \dots\dots\dots (1)$$

Where R is the radius of the cylinder, R is the radius of the balls and g is the gravity constant. The grinding speed was determined based on the radius of the oval bowl and the iron balls. The diameter of the oval bowl is (22.5cm) while the diameter of the iron balls is (6.36 mm). Thus, the grinding speed can be determined by relying on a measuring device for the number of revolutions, which is about (167 revolutions per minute). In this process, wet grinding is used by placing distilled water, iron balls, and powdered material (TiO₂) inside the bowl according to specific weights that were relied upon in our preparation method by placing (3gm of TiO₂ with 100gm of distilled water inside the oval container) in order to obtain the product required. "Figure 1" shows the grinding device, TiO₂ material, the speed control device and the iron balls.

2.2 Synthesis TiO₂ paste

After cleaning the glass slides and determining the conductive part of them using the resistance measuring device, TiO₂ paste is prepared by using the Nano powder material (TiO₂), which was prepared by the (ball milling) method by taking (6gm) of the powder substance with a mortar and pestle for 10 minutes until it is done To obtain a homogeneous paste, then (10 mm) of an acid solution (HNO₃) with (PH) ranges between (3 and 4). Then drops of washing liquid are added to increase the adhesion of the paste to the glass plate. Then it is mixed well and then left for a quarter of an hour to get a homogeneous good dough ready to obtain the thin film, as space and good grinding helps to disperse the nanoparticles. Then they are heat treated in a convection oven (450°C) for half an hour and then these slides are allowed to cool down naturally at room temperature [21]. After that, it is dipped in the dye solution N719 dye (C₅₈H₈₆N₈O₈RuS₂) for (48 hr.).

2.3 Preparation of the counter electrode

The second glass electrode (counter electrode) was prepared by depositing a layer of carbon onto the FTO plate by using a candle until it was coated and a layer of black was formed.

2.4 Assembling of DSSCs

The solar cell is assembled after placing the top of the TiO₂ thin film immersed with dye (N719) face to face with the counter electrode (using a candle flame). Then drops of the prepared electrolyte solution are added by dissolving (0.127gm) of iodine (I₂) with (0.83gm) of potassium iodide (KI) in (10 ml) in an ethylene glycol solvent separately. Then they are mixed well using an electric mixer and the electrodes are pressed using Mask [22].

3 Results and Discussion:

3.1 Scanning Electron Microscopy (SEM)

The results of the nanoparticles that were prepared by the ball milling method using an electron microscope (FE-SEM) were illustrated, as the images were clarified after the milling process of the milling model with a time (8hrs.) and with depend on (70%) of a constant speed called (Critical Speed). Which was clarified in paragraph (2.1). The results of (FE-SEM) showed a clear reduction in the size of (downscaling) TiO₂ nanoparticles according to "Figure 2" and by using the image analysis program (Image-J) for the purpose of calculating the nanoscale sizes of (TiO₂) particles for each image of these grinding models, and the (Origin) software was used for the purpose of measurements and statistical work to know the mean value and standard deviation of these models. The information was taken and the graphical messenger for the particle size distribution was obtained for this model. Since the graphs (continuous blue line) for each volume distribution were executed by (Log-Normal) function, the mean value of D which represents the average particle size was taken from the distribution and the standard deviation (σ). Note that the statistic was studied for all grinding models using several functions that show the average particle size and the extent of its conformity with the particle size distribution, and Log-Normal function was the best. The results we obtained are shown in Table (1). the shapes of the particles start to change, also the rate of particle size begins to decrease slightly with the advancement of the milling time.

Through this study and according to the results obtained through calculations in the programs (Image-J and Origin), the milling time caused a clear and effective decrease in the average particle size according to the statistic shown in " Figure 2a " show the size distribution of bulk (un milled)

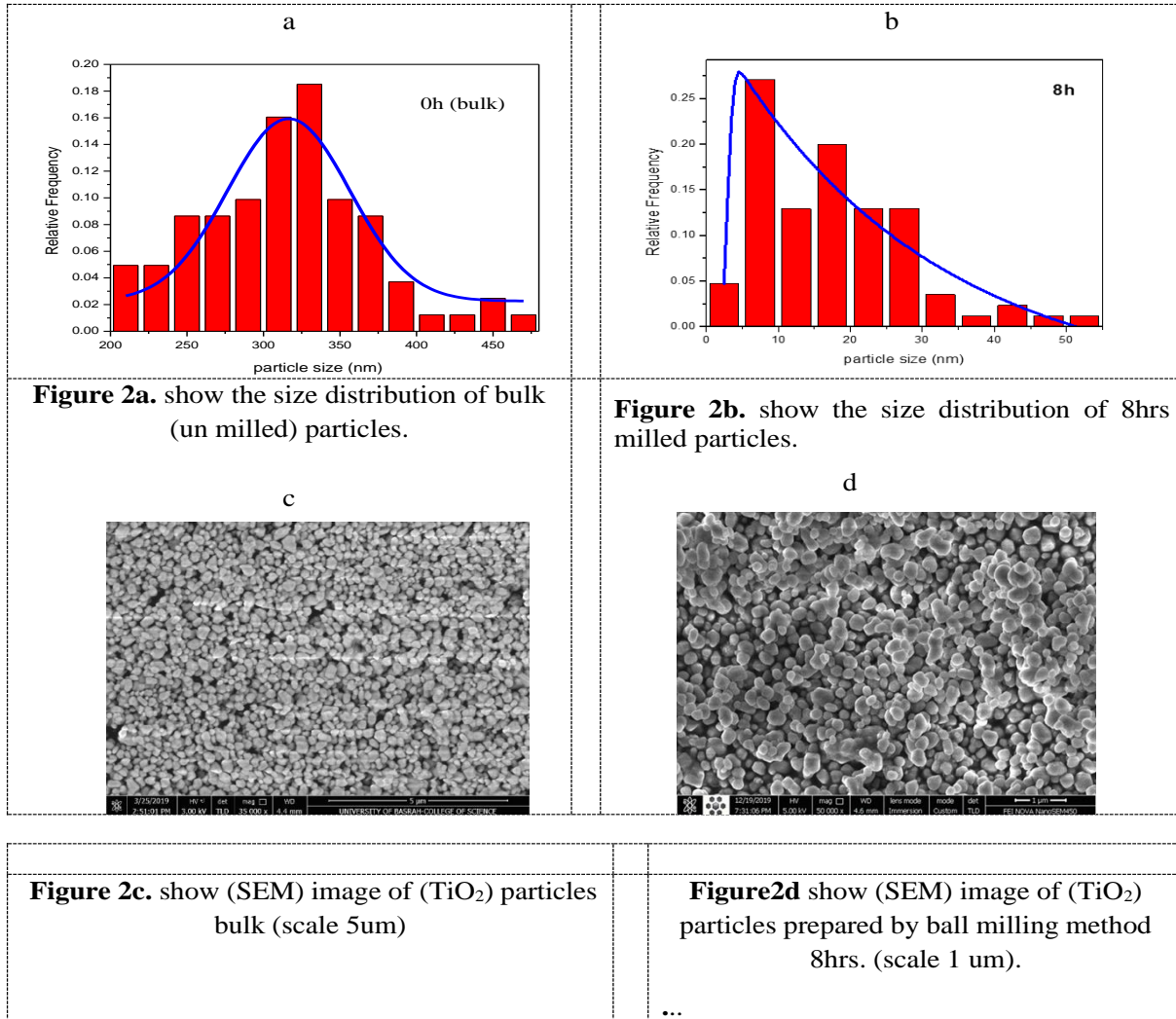


Table 1. shows mean particle size and standard deviation of bulk and milled particles using Log-Normal fit.

Standard deviation σ	Average (nm) $\langle D \rangle$	Milling Time
39.9	316	0 hrs.
0.73	2.84	8 hrs.

3.2 Results of the optical properties of (TiO₂) particles

This study presents an explanation of the properties of the prepared 8-hour ball milling model and the effect of the grinding process on the (paste) TiO₂films by measuring the absorbance spectrum of these films. And calculate the direct energy gap (E_g) by drawing the relationship between (hν & (α·hν)²), then extending the straight part of the curve to cross the axis of the photon energy, so we get the gap energy value for the direct transmission and by using the tauc relationship shown in equation (2).

As note that the absorption spectrum for this model ranges between (290-800 nm) as shown in Figure 4 and the absorption edge was calculated for this model and was explained in Table 2. As it is noticed that the energy gap increases with the grinding time (8 hours) compared to the energy gap of (TiO₂) particles, which has a value of (3.3 eV) with respect to the (anatase) phase. As the smaller the nanoparticle size, the energy gap value increases.

$$(\alpha h\nu)^2 = B^2 (h\nu - E_g) \dots\dots\dots (2)$$

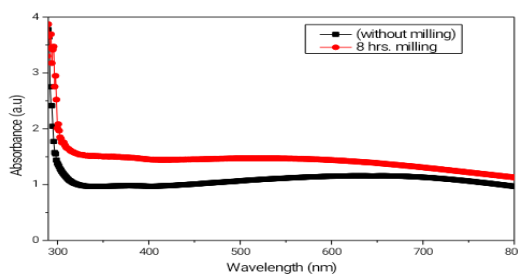
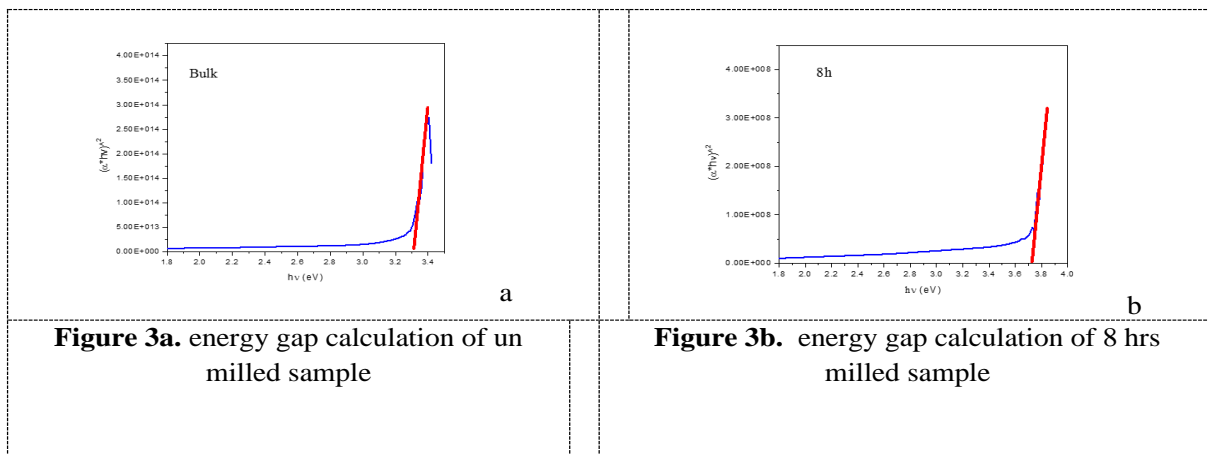


Figure 3c. The absorbance spectrum of the (TiO₂) bulk and 8hrs.

Table 2. shows Edge of Absorption and E_g values.

Absorption edge	E _g (eV)	Samples
310	3.30	Without milling (bulk)
332	3.71	milling (8 hrs.)

3.3 The XRD analysis

The XRD analysis of the prepared sample of TiO₂ (bulk) and TiO₂ nanoparticles was done using ball milling method with time milling (8 hrs.), The XRD pattern of the synthesized TiO₂ (bulk) and TiO₂ nanoparticles is shown in " Figure 4 " and the peak details are in Table 3. where average particle size has been evaluated by using Debye-Scherer formula (equ.3) and Inter planar spacing between atoms (d-spacing) is calculated using Bragg's Law (equ.4).

The intensive peaks centered at 25.3°, 37.4° and 48.02° of TiO₂ (bulk) and 25.9°, 37.8° and 48.8° for TiO₂ nanoparticles indicating the formation of TiO₂ in the anatase phase. From the figure all the peaks are of the anatase phase, no other phases were specific. It can be observed that there are sharp diffraction peaks which indicate the sample has crystalline nature and no amorphous phase has been formed through present milling conditions.

$$D = \frac{0.9 \lambda}{\beta \cos \theta} \dots \dots \dots (3)$$

$$n\lambda = 2d \sin \theta \dots \dots \dots (4)$$

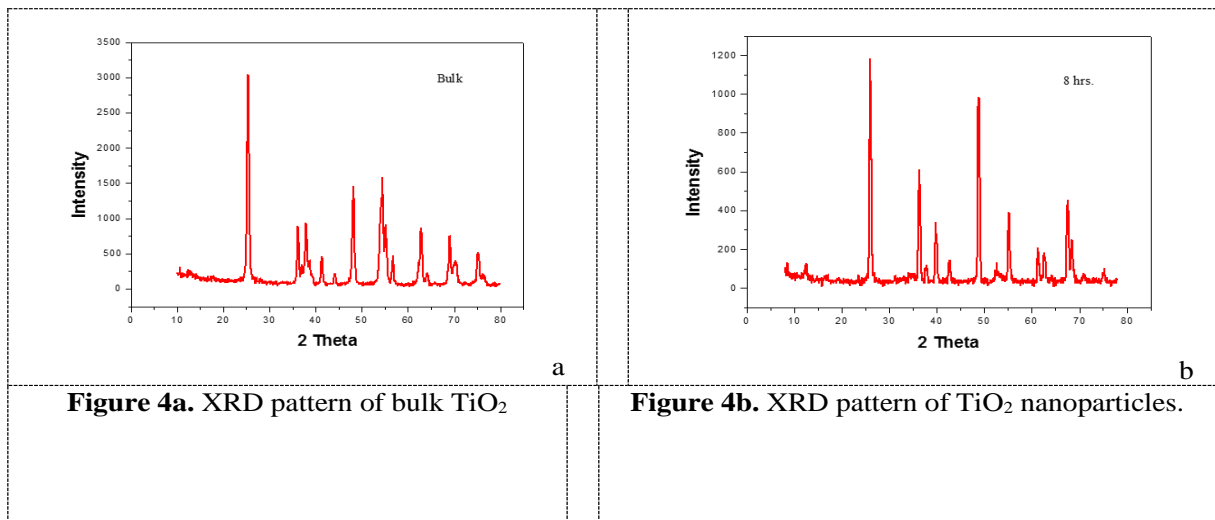


Table 3. XRD Data of TiO₂ (bulk and Nanoparticles).

Bulk						8 hrs.					
2θ	(θ)	Cos (θ)	FWHM M [°2Th.]	d-spacing g [Å]	[D-nm]	2θ	(θ)	Cos (θ)	FWHM M [°2Th.]	d-spacing g [Å]	[D-nm]
25.3	12.6	0.9	0.2460	3.52	33.2	25.	12.9	0.9	0.3444	3.81	23.7
	5	7			8	9	5	7			7
37.4	18.8	0.9	0.1476	2.37	57.2	37.	18.9	0.9	0.4920	2.27	17.1
		4			4	8		4			7
44.1	22.0	0.9	0.3444	2.05	25.0	42.	21.3	0.9	0.2460	2.16	34.7
	5	2			6	6		3			1
48.0	24.0	0.9	0.2460	1.89	35.4	48.	24.4	0.9	0.2952	1.67	29.5
	2	1			8	8		1			7

3.4 Results of solar cell measurement

The results of the electrical properties of the prepared solar cells were studied with a light intensity of ((416 W/m²)). From characteristic (I-V), filling factor and efficiency were calculated using (equ.5) and (equ.6) respectively. Also calculate the maximum power, maximum voltages, maximum current, and the resistance of the series and shunt.

DSSCs solar cells were prepared using (TiO₂) nanoparticles prepared with a grinding time (8 hrs.) And in different layers represented by single, double and triple layers that were prepared by the method of paste for DSSCs, an organic dye (N719) was also used, and then the results of these layers were compared on the basis of the obtained solar cell efficiency with the interpretation of its results based on the results.

To clarify and examine the effect of particle size on the performance of sensitive dye solar cells (DSSCs), we created three different combinations of (single, bi and tri) TiO₂ layers with different particles and sizes depending on the milling time (8 hrs.) and the particle size was verified by studying the shown particle size ratio. In paragraph (3.1), since observe the response of the prepared solar cells as shown in the figures for the characteristic of the solar cells (I-V) for each layer of the milling layer, and for the calculation of the efficiency value, the (equ.5) was used.

To find out effect of size of TiO₂ on performance of DSSC, two solar cells of (DSSCs) of single layer were made one from bulk TiO₂ (cell 1) and the other made from the milling TiO₂ (cell 2), as showed in Table (4) and "Figure 5 a,b". The (cell 1) reached an open voltage (399 mV) while the (cell2) reach (576 mV) indicating increasing in open voltage. However, the current of density value obtained ranged (2-1.95 mA/cm²) while the filling factor ranged (0.32-0.33%) for (cell 1) and (cell 2) respectively, as it indicates the presence of large particles that lead to a small surface area that may impeded a good dye to impregnate and, therefore, the photon adsorption was poor., and thus solar cells with large particles give less efficiency than those with a small size of TiO₂ particles.

As for the bi layers, the solar cells (DSSCs) that were as shown in Table (4) and "Figure 5c", and these cells were made of two layers. The first layer included thin films of nanoparticles from the milling model

as the bottom layer while the second layer included thin film of large particles as a top layer. The cell 3 made of double layers reached an open circuit voltage range (669 mV). However, the current of current value obtained is (1.68 mA/cm²) while the filling factor is (0.37%), thus, the efficiency performance of solar cells was better than the single layer and as shown in Table (4) and "Figure 5d" due to strong light scattering that occurs with a large surface area and due to poor back- scattering of light . Thus, our obtained results indicate that choosing the particle size in the lower layer is more important than in the upper layer to achieve better efficiency.

As for the triple layers, solar cells were made with three layers, the first layer included nanoparticles thin films prepared from the milling model as first layer while the middle layer included a mixture of equal amounts (50%) from nanoparticles and 50% from bulk TiO₂) and the third layer made from bulk TiO₂. the open circuit voltage was (580 mV). However, the current of density value obtained about (2.06 mA/cm²) while the filling factor is (0.41%). although the Bilayer was better than the single layer in terms of efficiency, the triple layers was better as the solar cells gave a better result and higher solar efficiency, as shown in Table (4) and "Figure 5".

$$\eta = \frac{J_{sc} V_{oc}}{P_{in}} * F.F \quad \dots \dots \dots (5)$$

$$F.F = \frac{P_{max}}{I_{sc} * V_{oc}} \quad \dots \dots \dots (6)$$

Table 4. show result of DSSCs.

	Sample Code	η (%)	F. F	Pmax (mW)	Jsc(mA/cm ²)	Voc (mV)	R _{sh} (Ω)	R _s (Ω)	Time of milling
Single layer	Cell.1	0.61	0.32	5.10*10 ⁻³	2	399	42985.07	8166.81	0hrs. (bulk)
	Cell.2	0.9	0.33	7.57*10 ⁻³	1.95	576	39617.48	18763.79	8 hrs.
Bi layer	Cell. 3	1.01	0.37	8.41*10 ⁻³	1.68	669	30270.27	10107.81	8 hrs.
Tri layer	Cell.4	1.18	0.41	9.84*10 ⁻³	2.06	580	30909.09	8827.23	8 hrs.

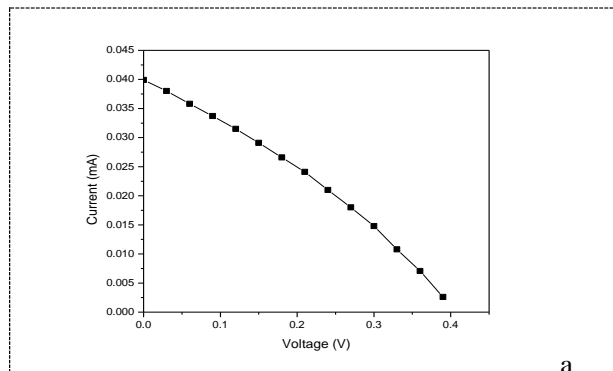


Figure 5a. Results of bulk monolayer solar cell

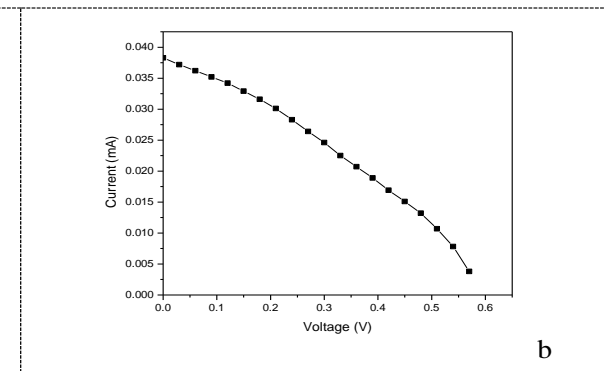
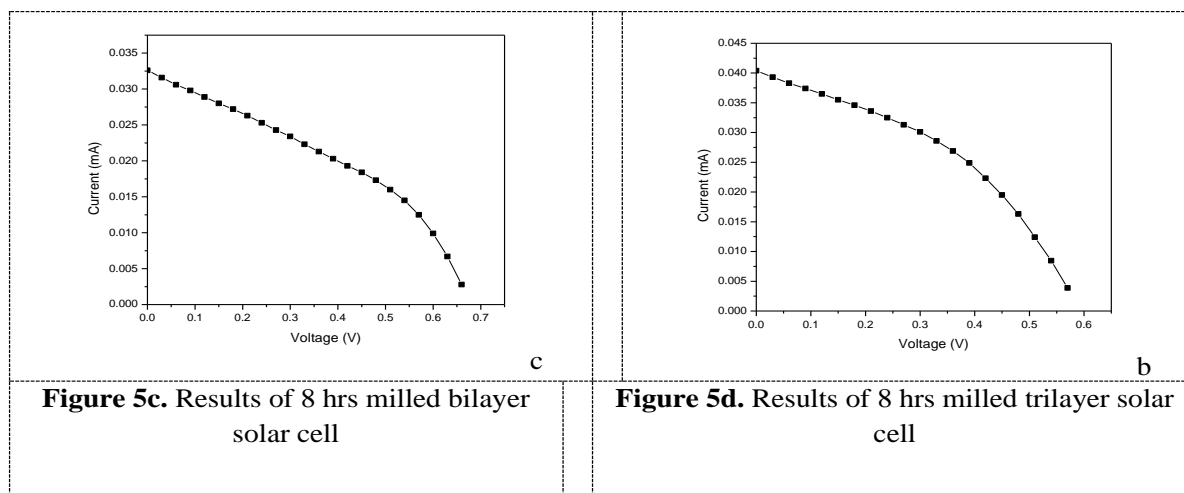


Figure 5b. Results of 8 hrs milled monolayer solar cell



4. Conclusion:

we can conclude that, it is impossible to increase the surface area and the scattering of light simultaneously, because they are opposed to each other, so there must be a balance between the surface area and the scattering of light through the use of a multilayer structure (with a gradual increase in the size of the particles) [23]. As it works with a balance between the surface area and the scattering of light, that is, the combination of large particles in the upper layer and smaller particles in the lower layer will lead to better performance of the efficiency of solar cells (DSSCs). Therefore, it was proposed to make layers represented by a layer (single, double and triple), and from noting the results that have been demonstrated it was observed that the bilayer is better than the single layer and that the tri layer is better than the bilayer, and this indicates a balance between the surface area and the scattering of light, The results also indicate that the ball milling method played an important and effective role in improving the performance of the efficiency of the solar cells.

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