SEMARAK ILMU PUBLISHING

Journal of Advanced Research in Micro and Nano Engineering

Journal homepage:
https://semarakilmu.com.my/journals/index.php/micro_nano_engineering/index
ISSN: 2756-8210

The Effect of Plasma Parameters on Titanium Dioxide (TiO_2) Thin Film for Hybrid Solar Cells Application

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ARTICLE INFO

Article history:
Received 8 September 2024
Received in revised form 14 October 2024
Accepted 20 November 2024
Available online 31 December 2024

Keywords:
Plasma treatment; natural dye photosensitizer; hybrid solar cell

ABSTRACT

Hybrid solar cell (HSC) one of the third-generation solar cells, was developed as one of the alternative energy generations which was extensively studied previously. The use of synthetic dye combined with TiO_2 as HSC achieved a power conversion efficiency (PCE) of 18%, but synthetic dye residues might harm aquatic life. In previous studies, a photosensitizer (natural dye from the plant) was used to replace synthetic dye. Unfortunately, the HSC PCE is low due to the roughness of the TiO_2 surface, which makes natural dye difficult to absorb. The problem was solved by applying plasma to the TiO_2 surface to increase surface area and natural dye absorption. The distance of plasma exposure was taken at 2 mm, 4 mm, 8 mm, and 12 mm toward surface TiO_2. Natural dye photosensitizer (NDP) was extracted from the tropical plant's mangosteen pericarp. The combination of TiO_2 with NDP was made in bulk heterojunction thin film, that was deposited on indium tin oxide (ITO) subtract. The morphology of TiO_2 before and after treatment was observed by scanning electron microscope (SEM). The optical properties were determined by UV-Vis spectrophotometry. The power conversion efficiency of HSC is determined by IV measurement. The results of SEM analysis showed that the particle sizes after exposure to plasma were smaller compared to those without Plasma, and the sizes of TiO_2 decreased with increasing the plasma distance. The PCE result of the ITO/TiO_2+NDP (without plasma) was a 0.13 % increase by 50% compared to the PCE of ITO/TiO_2+NDP (with plasma) was 0.25 % at an optimal distance of plasma exposure is 2 mm.

1. Introduction

Renewable energy is the energy source derived from natural resources, such as sun, wind, geothermal, or hydropower while photovoltaic energy is one of the generations of electricity from sunlight sources and solar cells manufactured because of cheap cost and high conversion efficiency

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https://doi.org/10.37934/armne.28.1.118129

that uses a variety of materials and processes. Silicone was the first material used to make solar cells [1,2]. However, solar energy generation today depends on silicon solar cells which are still expensive because they involve the processing of silicon production at a high temperature of 1500 ^{o}C and the problem of disposing of expired silicon solar panels. Therefore, until now research has continued by looking for organic materials as a substitute for silicon [3]. Thin film technology contributed to the second generation of solar cells. The photoactive material in thin-film solar cells contains more toxic materials than those used in silicon PV technology, including indium, gallium, arsenic, selenium, cadmium, and telluride [4,5]. First and second-generation solar cells are now being used as energy generators to meet global demand [6].

The third generation of solar cells was grown with new materials synthesis among inorganic materials and organic materials. The idea of progressive research in the third generation of solar cells is limited to raw materials source and side impark on the environment [7]. The third generation of solar cells are polymer solar cells, hybrid solar cells, perovskite solar cells and dye-sensitized solar cells were derived from the synthesized materials used in the fabrication of the solar cell, as shown in Table 1 [8,9]. The imbalance in the world today due to environmental damage has activated research to find natural materials that can be used as active materials or additives in the manufacture of third-generation solar cells.

Table 1
The synthesized materials are related to the type of third generation of solar cell

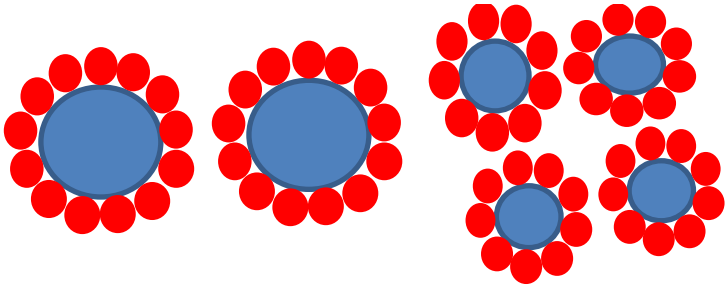
Type of solar cell	Synthesized materials	References
Polymer solar cell	Polymer (Poly (3-hexylthiophene)	[10]
Hybrid solar cell	Metal oxide + organic materials (Zinc oxide + polythiophene)	[4]
Perovskite solar cell	perovskite	[11]
Dye-sensitized solar cell	Dye-sensitized (Synthetic dye, phthalocyanines)	[12]

The synthesis of new chemicals also leads to the accumulation of toxic waste. So, researchers continue to study natural materials that can be developed as photosensitizers. Recently, natural dye photosensitizers from plants have been in the spotlight. The natural dye pigments that have been studied are chlorophyll, anthocyanins and flavonoids which have various molecular structures, where the special feature of these natural dyes is that they can absorb sunlight in a wide range of visible and ultraviolet light. This natural property is very compatible with the function of active ingredients in the manufacture of solar cells [13,14].

The combination of metal oxide with natural dye faces the low absorption of natural dye to metal oxide. Because of the surface structure of particles metal oxide is bigger than molecule dye size. So, one of the solutions for this problem is to increase the surface area with smaller particle sizes of metal oxide, as illustrated in Figure 1. Combination of molecules that makes them resistant to degradation. The plasma method was used in this study to reduce the size of titanium oxide particles. A plasma is an ionized gas consisting of approximately equal numbers of positively charged ions and negatively charged electrons. This ionized gas will hit the material's thin film and transfer the energy. The particles of the material's thin film can be broken by the collisions. Some very small particles projectized and some large particles will break after the collisions depending on the energy transferred.

The distance between the plasma outlet and the sample was varied to determine the effectiveness of plasma radiation on the changing of TiO_2 particle sizes. The attachment of natural dye on $TiO_{2(untreated)}$ and $TiO_{2(Plasma\ treated)}$ affected their optical properties that were observed by UV-Visible spectrophotometer. The power conversion efficiency of the hybrid solar cell of ITO/$TiO_{2Plasma}$

$_{treated}$/dye/P3HT/Au was determined. Poly(3-hexylthiophene), P3HT was the hole transport layer and gold, Au as the electrode.



(a) (b)

Fig. 1. The graphical analogy of the effect of metal oxide particle size with the attachment of dye molecules on its. The blue circle is a metal oxide, and the red circle is a dye molecule (a) 26 molecules of dye attached to 2 particles of metal oxide (b) 36 molecules of dye can be attached to 4 smaller particle sizes of metal oxide

2. Methodology

2.1 Materials

Titanium dioxide, TiO_2 nanoparticles and poly(3-hexylthiophene)-2.5-dial, P3HT were purchased from SIGMA company. The chlorobenzene (99.99%), methanol (99.95%), ethanol (99.95%), acetone (99.95%), and 2-propanol (99.95%) were used without any purification. The molecular weight of TiO_2 is 79.866 g/mol.

2.2 Preparation of Indium Tin Oxide (ITO)

The Indium tin oxide (ITO) substrate purchased from SIGMA had thicknesses of sheet and resistances of 80 nm and 12 Ωm^{-2}, respectively. ITO substrate was cut in different square sizes that were 1 cm x 1 cm, and 1 cm x 2 cm using the glass cutter. The ITO substrates were cleaned using an ultrasonic bath (JEIOTECH, United Kingdom). They were successively submerged for approximately ten minutes at 30^oC in ethanol, then in acetone with vibrating ultrasonic baths to get rid of any contaminants. Lastly final cleansing with the distilled water for another 10 minutes. After that, ITO substrates were dried using the hair dryer before it was kept in a Petri dish [15].

2.3 Preparation of P3HT Solution

P3HT solution will be prepared at 0.01 Mol concentration by weighing 0.0017 g P3HT and dissolving in chlorobenzene solvent. Then, the mixture was stirred for 24 hours at 350 rpm with a magnetic stirrer. Finally, P3HT solutions produced an orange colour [11].

2.4 Preparation of Natural Dye Solution

The natural dye source was mangosteen pericarp, collected in Kuala Terengganu. The mangosteen pericarp was washed with deionized water and vacuum-dried at a temperature of 60^o C. After that, the pericarp was crushed into fine powder by using a mortar. The powders of natural dye are put in absolute ethanol at room temperature in the dark for a week to extract the pigment

of natural dye. After a week, the solution was filtered. This extraction method was carried out as done by Ghazali *et al.,* [16].

2.5 Preparation of Titanium Dioxide

The Titanium Dioxide (TiO_2) colloid was prepared by using TiO_2 powder and was pretreated at 450 °C for 30 min in an Electric Muffle Furnace and then cooled to room temperature. A mixture of TiO_2 (pretreated), distilled water, and absolute ethanol of molar ratio 1:1:5 was stirred at room temperature. After adequately stirring, paste mixtures were hydrothermally treated in an autoclave (packing volume < 80%) at 185°C for 1217 h. The TiO_2 colloid was produced [17].

2.6 Preparation of Atmospheric Pressure Plasma Jet (APPJ)

The principal operation of the atmospheric plasma is the high voltage supplied to the flowing gas in a glass tube in atmospheric pressure, this experiment is called an atmospheric pressure plasma jet (APPJ) as shown in Figure 2 and Figure 3. The setup is composed of three main parts. The first part is the gas control system including the flow meter system, valve, power system, control system and gases. The second part is two electrodes where the ionization occurs. These electrodes are placed 25 mm between each other on the glass tube (diameter tube 1.50 mm). The flow rate of Helium gas was 1 L/min [18]. The third part is the power supply is a device that supplies the electric power to the electric load. A pulse inverter converted 8 V DC to 16 KV AC. It converts the AC or DC power into the required voltage. Abdullah, et. al. introduced this APPJ concept [19].

2.7 Fabrication of Hybrid Solar Cell

The fabrication of a hybrid solar cell used layer-by-layers of TiO_2, natural dye, P3HT and Au as illustrated in Figure 4(a). The first step was the preparation of ITO substrate as explained in section 2.2. The second step was TiO_2 deposited on ITO substrate using Dr. Blade method as explained in section 2.5. Then, baked in a furnace at 400 ^oC for 3 hours to crystallize TiO_2 into a rutile structure. The third step was plasma treatment on TiO_2 by APPJ. The effect of plasma treatment was observed based on the different distances between the plasma jet and the surface of the TiO_2 thin film (2 mm, 4 mm, 8 mm, and 12 mm) as shown in Figure 3. The fourth step was to deposit natural dye on TiO_2 layer using the immersion time method. TiO_2 was immersed in a solution of natural dye for 24 hours. After that, the sample was dried using a hot air gun for 10 minutes. The fifth step is the deposition of P3HT thin film onto the natural dye layer by spin coating method at a speed of 200 rpm at room temperature. The sixth step was to deposit Au thin film as the top electrode using the physical evaporation method. [4]. Figure 4(b) depicts the hybrid solar cell's architectural layout in this work. The concept of fabrication HSC was recommended by Triyanto *et al.,* [20]. There were 3 sample replications at each exposed TiO_2 distance being conducted.

2.8 Characterizations of Samples
2.8.1 Surface morphology of TiO_2

Surface morphology of TiO_2 before and after plasma treatment (PT) was observed using a scanning electron microscope (SEM, JEOUL JSM-6360 LA) under magnification at 5000x. All the samples were coated with Au to protect surface samples from charging during the observation of images.

2.8.2 Optical absorption measurement

Ultraviolet-visible (UV-Vis) spectrophotometer was used to observe the ability of natural dyes, TiO_2 and P3HT thin films to absorb the light spectrum. In this work, a Perkin Elmer Lambda 35 UV-visible spectrophotometer was used to record the absorption spectrum in the wavelength range of 200 to 900 nm [16].

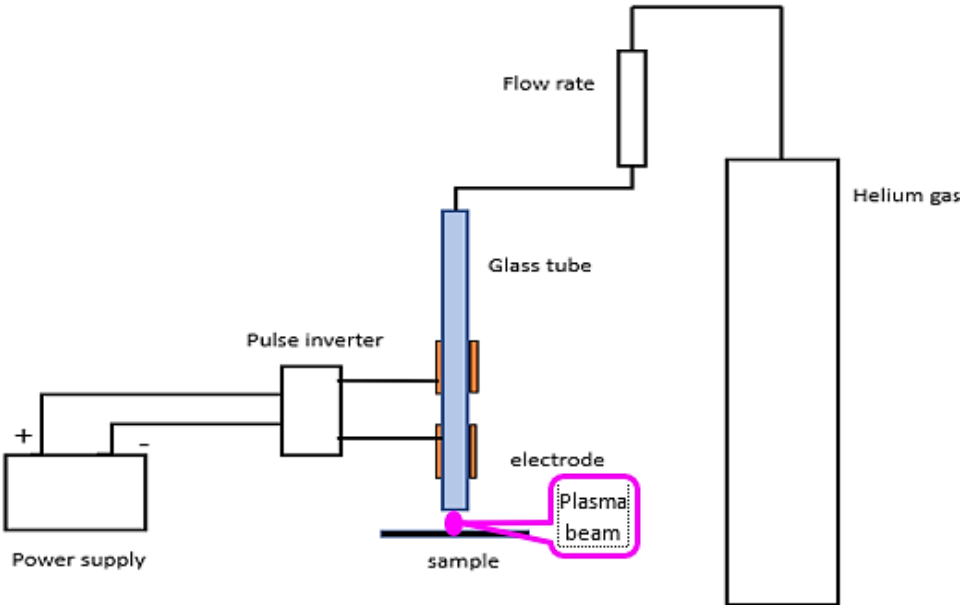


Fig. 2. The set-up diagram of atmospheric pressure plasma jet [18]

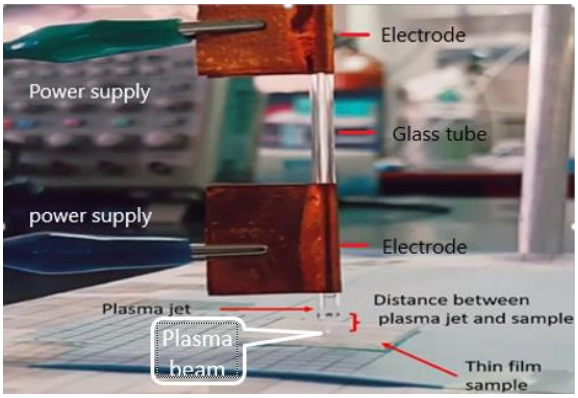
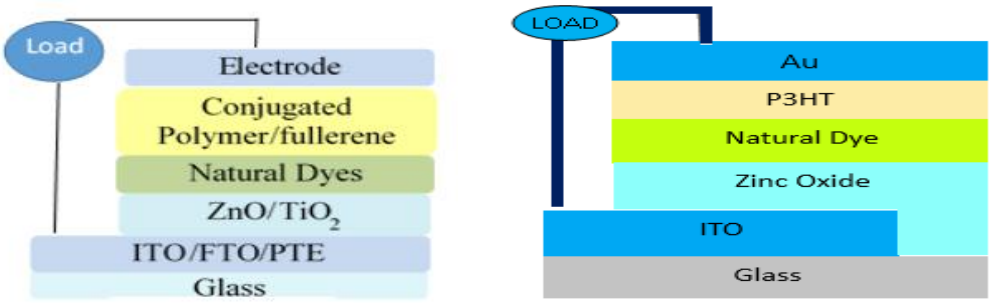


Fig. 3. The distance between the plasma jet and the sample of the plasma jet

At the same data, the calculation of the optical band gap energy of samples using the formula as shown by Eq. (1). E is the optical energy band gap, where h = Planck constant, c = speed of light, and λ= wavelength at maximum peak appeared in the UV-Visible absorption spectrum.

$$E = \frac{hc}{\lambda} \quad (1)$$



(a) (b)

Fig. 4. (a) General architecture of hybrid solar cells [3] (b) Architecture of hybrid solar cell in this work

2.8.3 Solar properties

Power conversion efficiency (PCE) can be determined using two-point Probes with the Keithley 4200-SCS Semiconductor Characterization System. The power conversion efficiency of a solar cell is defined as the ratio of optical output power (P_{out}) to input power (P_{in}). Maximum current *(I_{max}) and voltage (V_{max})* were found from the graph I-V measurement. The PCE of a solar cell can be calculated using Eq. (2)-(4) [21]. P_{in} involved intensity of light (I) which was 100 mW/m^2 and multiplied by the effective surface area (A) of the solar cell.

$$\eta = \frac{P_{out}}{P_{in}} \times 100\% \quad (2)$$

$$P_{in} = I \times A \quad (3)$$

$$P_{out} = I_{\max} \times V_{max} \quad (4)$$

3. Results and Discussion

3.1 The Morphology of TiO_2 Thin Film Before and After Plasma Treatment

Figure 5 shows an SEM micrograph of TiO_2 thin film (TiO_2 TF) that is without plasma treatment. The micrograph shows that the grain shape of TiO_2 is lumpy with rounded corners, like a potato, some like big and small circles, and some look like a combination of a few circles. The size of the grain shape was of various sizes and measured using SEM software, it was found that the average size was 3.13 ± 0.01 nm.

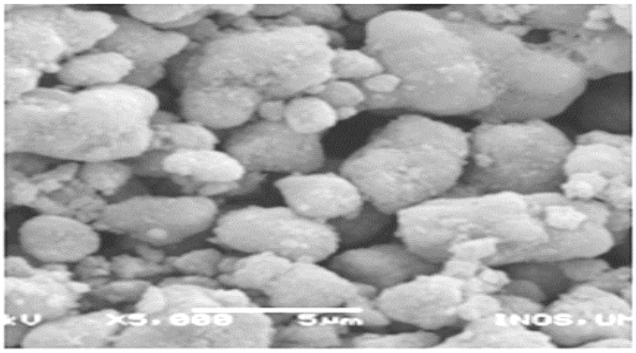
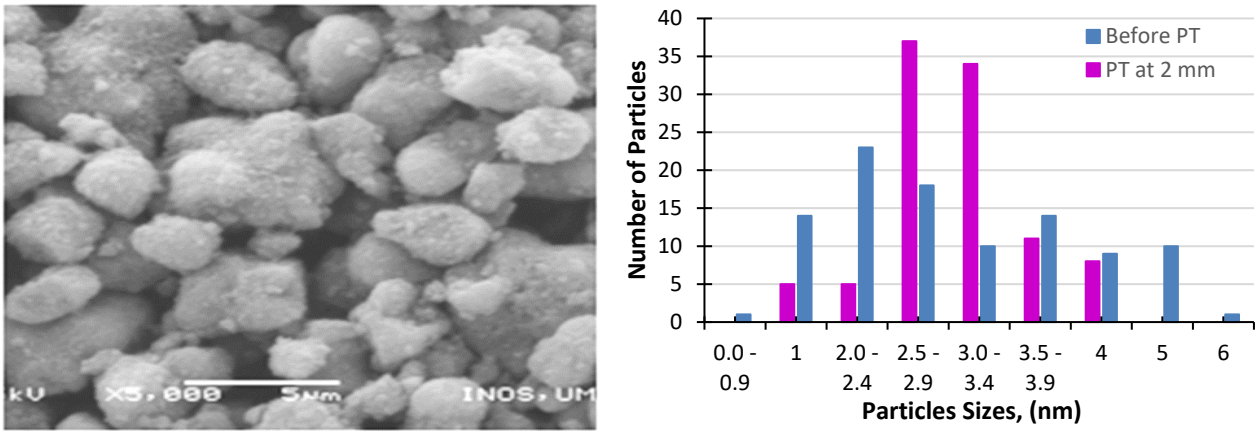


Fig. 5. Micrograph of TiO_2 nanoparticle before plasma treatment

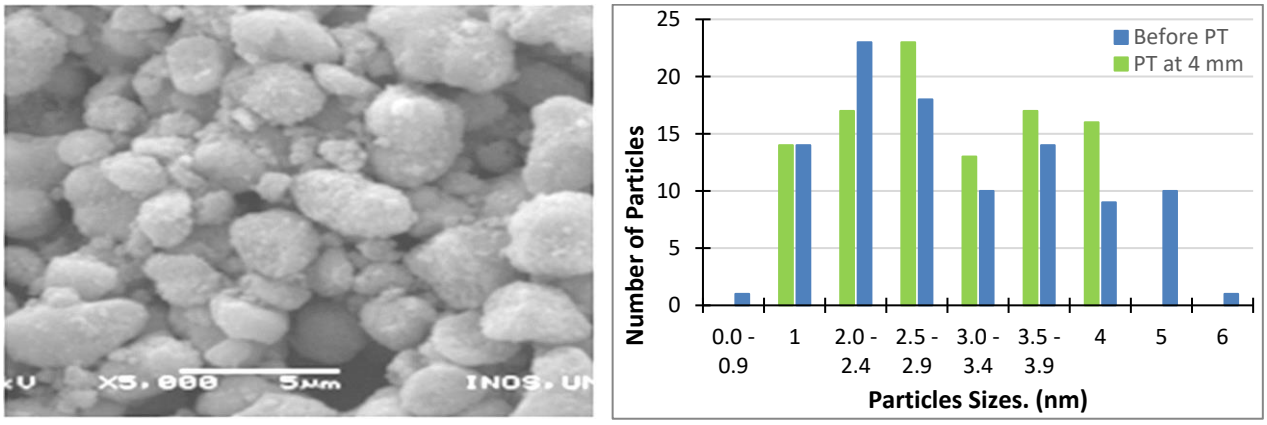
The SEM micrograph of the plasma-treated TiO_2 at a 2 mm distance was displayed in Figure 6(a) and Figure 6(b) the distribution of TiO_2 nanoparticle sizes in thin film [22]. The SEM micrograph cannot see the sizes of TiO_2, but the measurement of TiO_2 sizes showed a decrease at 2.5 – 3.4 nm. The TiO_2 particle sizes at 5 – 6 nm in Figure 6(a) were broken by plasma, so there was no in Figure 6(b). This means that plasma is useful for breaking the TiO_2 particles, in terms of small sizes is increases the surface area and increases the absorption of natural dye as photosensitizer into the TiO_2. The average particle size of TiO_2 (2 mm) was 3.06 nm. 2.24 % decrease in TiO_2 nanoparticle sizes after plasma treatment [23].



(a) (b)

Fig. 6. SEM Micrograph of TiO_2 thin film after being exposed to plasma at 2 mm distances and (b) the distribution of TiO_2 sizes from 0 – 6 nm

Figure 7(a) showed the micrograph of SEM of TiO_2 after the plasma treatment, the shape of TiO_2 was a very small change, but Figure 7(b) showed that the sizes of TiO_2 became smaller compared to before treatment [22]. The range particle sizes of TiO_2 at 2.5 – 4 nm showed the number of particles more than untreated. And no more particles at size 5 – 6 nm. The average size of TiO_2 particles after plasma treatment at a distance of 4 mm was 3.05 nm, it was about 2.56% decrement.

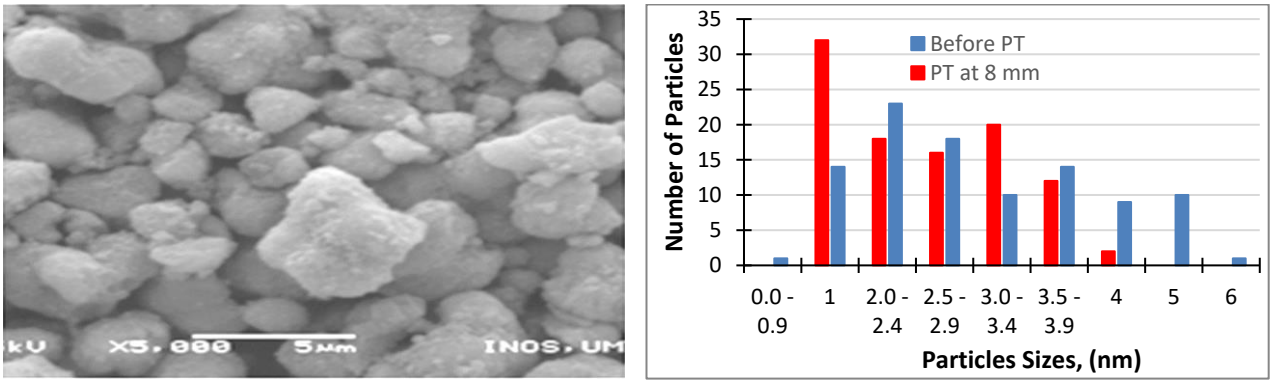


(a) (b)

Fig. 7. SEM Micrograph of TiO_2 thin film after being exposed to plasma at 4 mm distances and (b) the distribution of TiO_2 sizes from 0 – 6 nm

Figure 8(a) shows the shape of TiO_2 particles like lumps of stone of various sizes. After measuring the size of 100 TiO_2 particles and categorizing the size into 9 groups as shown in Figure 8(b), it was found that the TiO_2 particles are getting smaller and the largest number is at 1.0 nm in size. The

number of particle sizes at 4 nm is greatly reduced while at 5 – 6 nm it is completely absent. This situation illustrates the size of TiO_2 particles successfully reduced by plasma treatment with the average particle size after plasma treatment at a distance of 8 mm being 2.55 nm, which was about 18.53 % reduction [24].



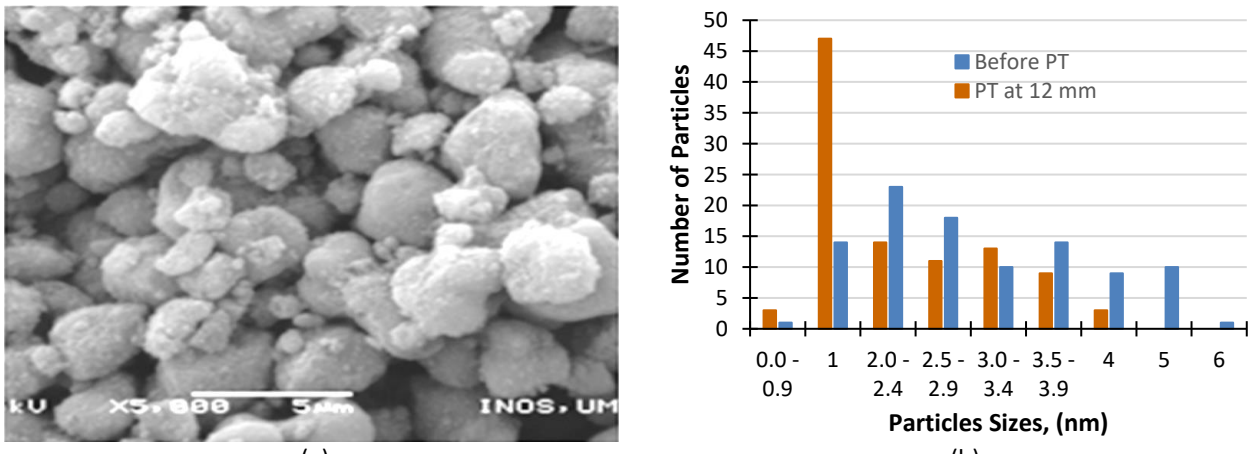
(a) (b)

Fig. 8. SEM Micrograph of TiO_2 thin film after being exposed to plasma at 8 mm distances and (b) the distribution of TiO_2 sizes from 0 – 6 nm

When TiO_2 is subjected to plasma treatment at a distance of 12 mm from the plasma source, the SEM micrograph displays particles that resemble round stone lumps, as seen in Figure 9(a) [22]. The largest number of TiO_2 particle sizes measured was 1.0 nm. However, there is no record of the number of particle sizes at 5–6 nm as shown in Figure 9(b). At a distance of 12 mm, the average particle size of the TiO_2 was 2.22 nm and the decrement was 29.07 % [23].

3.2. UV-Visible Absorption Analysis.

Figure 10 displays the absorption spectrum of the TiO_2 thin film on the ITO substrate before and after plasma treatment and dye immersion. A broad peak that emerged between 400 and 550 nm in wavelength belonged to the anthocyanin pigment that was present in the TiO_2.



(a) (b)

Fig. 9. SEM Micrograph of TiO_2 thin film after being exposed to plasma at 12 mm distances and (b) the distribution of TiO_2 sizes from 0 – 6 nm

The TiO_2 exhibited the maximum absorption with an average particle size of 2.55 nm, indicating a greater absorption of dye contained in the TiO_2 thin film. Because there was less dye in the TiO_2

without plasma treatment had average particle size was 3.13 nm, and the absorption spectrum was the lowest.

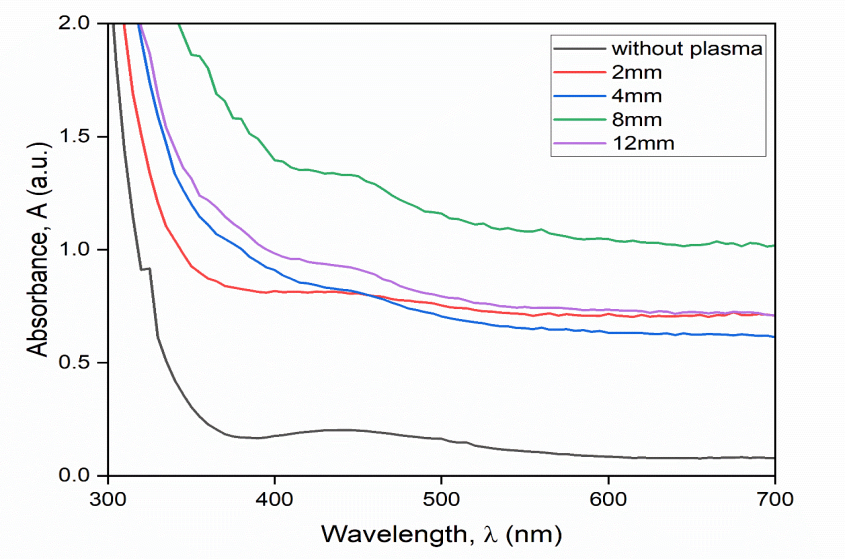


Fig. 10. UV-Visible absorption spectrum of TiO_2 before and after treatment with plasma at different distances

The hypothesis of small size had a large surface area is validated in this work. The TiO_2 at a small size absorbs more dye compared to the bigger size, when the dye is more, the absorption spectrum increases. But at certain particles, much smaller than the optimum size, the dye cannot absorb more at TiO_2, that's why at TiO_2 with 2.22 nm, the absorption spectrum was lower.

The optical gap of TiO_2-containing dye was determined using Eq. (1). From the UV-Vis spectrum data, the energy gap, E_g was extracted from the graph A^2 versus energy Photo (eV), where A is absorption. The value of E_g for TiO_2 thin film before and after plasma treatment is shown in Table 2. All the E_g for after plasma treatment was reduced. The lowest E_g was obtained at TiO_2 after plasma treatment at an 8 mm distance. This means that plasma treatment can change the E_g of TiO_2 and other metal oxide. This work result was agreed with the F modification of titanium dioxide through a plasma method achieved for the first time. The characterization results showed that the band gap of Ar/HF/DBD-TiO_2 was reduced to 2.78 eV [25].

Table 2
Energy band gap, E_g of TiO_2 before and after plasma treatment

Distance	Band gap(eV)
Without plasma	3.76
2 mm	3.72
4 mm	3.56
8 mm	3.38
12 mm	3.64

3.3 The Power Conversion Efficiency of Hybrid Solar Cells

The power conversion efficiency (PCE) of hybrid solar cells consists of ITO/TiO_2/Dye/P3HT/Au that is fabricated layer-by-layer. The layout of the solar cell is shown in Figure 4(b). The PCE of hybrid solar cells obtained with TiO_2 before and after plasma treatment at different distances is shown in Table 3. The highest PCE was 0.25 % containing TiO_2 with particle sizes of 3.06 nm. PCE of HSC containing

TiO_2 before plasma treatment was 0.13 %. Indicates that PCE of HSC increases when TiO_2 is treated by plasma. The best distance to expose TiO_2 with plasma was at 2 mm. This finding was supported by the Plasma treatment on the TiO_2 surface between dye molecules and photoelectrode. Plasma-treated TiO_2 at optimum conditions had increased efficiency by 40% relative to untreated TiO_2 [26].

Table 3
The power conversion efficiency (PCE) of HSC

ITO/……. /Dye/P3HT/Au	PCE, %	Average particle sizes (nm)	E_g, (eV)
TiO_2 (0 mm)	0.13	3.13	3.76
TiO_2 (2 mm)	0.25	3.06	3.72
TiO_2 (4 mm)	0.04	3.05	3.56
TiO_2 (8 mm)	0.01	2.55	3.38
TiO_2 (12 mm)	0.001	2.22	3.64

4. Conclusions

This study concluded that plasma treatment reduced the TiO_2 nanoparticles. The particle sizes were directly proportional to the distance of plasma treatment, increase in the distance produced smaller TiO_2 particles size, where from without plasma treatment to 2 mm, 4 mm, 8 mm and 12 mm distance, the average sizes of TiO_2 were 3.13 nm, 3.06 nm, 3.05 nm, 2.55 nm and 2.22 nm which the percentage reduction of particles sizes were 2.24%, 2.56%, 18.53% and 29.07% respectively. The UV-visible spectrum showed the highest absorption was at ITO/TiO_2 (8 mm)/Dye thin film with the lowest energy gap produced of 3.36 eV. The highest PCE of HSC ITO/TiO_2 (2 mm, 3.06 nm, 3.72 eV)/ Dye/P3HT/Au was 0.25% increased by about 92.31% compared with untreated. Plasma treatment successfully reduced TiO_2 nanoparticle sizes and increased the PCE of HSC.

Acknowledgement
The authors greatly thank to University Malaysia Terengganu for providing the facilities. This project was financially supported by the Malaysian Ministry of Higher Education, under the Fundamental Research Grant Scheme (FRGS), FRGS/1/2018/TK10/UMT/02/04 (VOT 59511)

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