

Modification of Photoanode Surface Structure via Image Analysis on Organic Polymer Material based for Dye-Sensitized Solar Cell (DSSC) Applications

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ABSTRACT

In this study, the experiment on the modification of the photoanode with organic polymer material as copolymer template for dye-sensitized solar cell (DSSC) applications has been conducted. The two organic copolymer templates are polystyrene sphere (PS) and poly[2-methoxy-5(2-ethylhexyloxy)-1,4-phenylenevinylene] (MEH-PPV). The modification photoanodes were made using Dr. Blade's method. These organic copolymer templates were added to improve the surface of the mesoporous titanium dioxide (TiO₂) layer, which is used as the main component in DSSC photoanode. The unmodified TiO₂ photoanode has poor aggregation and porosity of TiO₂. The addition of either MEH-PPV or PS sphere to the photoanode layer was found to affect the surface of mesoporous TiO₂ in terms of porosity, particle size distribution and shape. The analysis of the TiO₂ modification was conducted using an image analysis processing method via a 2D scanning electron microscope (SEM) image. The image analysis processing method used was the ImageJ program. The DSSC of modified photoanode is fabricated using metal complex dye, Ruthenium (N719) dye. The data collected from the ImageJ program showed that by adding organic copolymer templates into TiO₂, the porosity of TiO₂ decreased from 45 % to 42 %. From the photovoltaic analysis obtained, the J-V characteristic is recorded with the photoanode of TiO₂ mixed with 1.00 wt% MEH-PPV gave the highest efficiency, which is 0.01 % with the following parameters – Voc = 0.43 V, Jsc = 0.17 mA/cm² and FF = 0.20. Meanwhile, the photoanode of TiO₂ mixed with 0.50 wt% PS sphere gave the highest efficiency which is 0.08 % with the following parameters – Voc = 0.39 V, Jsc = 0.86 mA/cm² and FF = 0.25.

Keywords: Dye-Sensitized solar cell, MEH-PPV, PS sphere, photoanode

1. INTRODUCTION

Renewable energy is the natural source of energy that has always been refilled up. There are many types of renewable energy, such as solar energy, biomass energy and hydroelectric power. In this paper, we will focus on solar energy, which the modification of the part in this type or energy can be used for the application of dye-sensitized solar cell (DSSC). Nowadays, the application of DSSC had been widely used by the researcher as it has many advantages such as low fabrication cost [1] and simple fabrication process [2] has lead researchers to focus more on this type of application. However, DSSC still has one main problem: the agglomeration of TiO₂ which leads to lower conversion efficiency [3]. In DSSC, several parts were used to ensure that the device can operate well, such as photoanode [3], dye sensitizer [4], electrolyte [5], and photoelectrode [6]. In this paper, the main focus is the photoanode of DSSC as highlighted in

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Figure 1 (a). Photoanode or working electrode is a part where the absorption process of photon [7] from sunlight to a layer that is called mesoporous Titanium Dioxide (TiO_2). The improvement on the photoanode material has been made to increase the performance of DSSC by introducing organic copolymers template in the surface of the photoanode [8-9], as shown in Figure 1 (b).

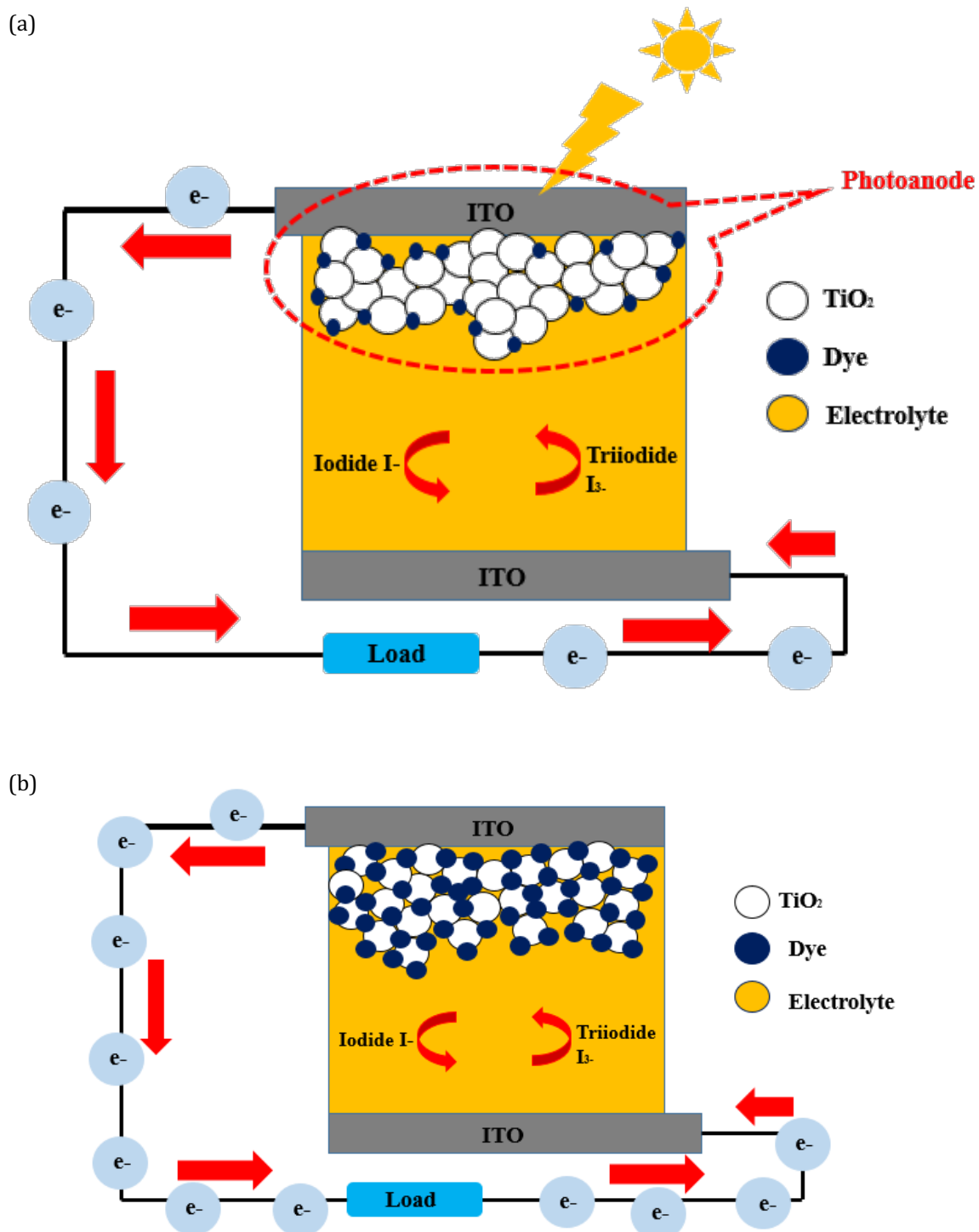


Figure 1. (a) TiO_2 mesoporous structure without organic polymer template (b) TiO_2 structure with organic template after annealing process in photoanode

For the past few years, studies on the polymer material for the DSSC photoanode had been investigated [3,10]. One type of polymer material used is the copolymer template type [10], as it

is added into TiO₂ layer. So, copolymer template strategies are used to produce an organized TiO₂ [10]. In this study, there are two copolymer templates that used to modified the TiO₂ layer of the photoanode, which are the polystyrene (PS) sphere and poly[2-methoxy-5-(2-ethyhexyloxy)-1,4-phenylenevinylene] (MEH-PPV).

Polystyrene (PS) sphere is a synthetic polymer obtained by thermal polymerization [11]. The melting point of PS sphere is 240 °C, and it decomposes above 330 °C. Wang *et al.* reported that when added PS in photoanode it is a general technique to prepare the TiO₂ thin film hollow sphere [12]. Then, Chen *et al.* proved that TiO₂ are more ordered using the PS sphere. It can filter the incidental light to improve the efficiency of DSSC [12].

Poly[2-methoxy-5-(2-ethyhexyloxy)-1,4-phenylenevinylene] (MEH-PPV) is an organic polymer material that has good electrical conductivity [13], high stability [13], and it is usually found in the fabrication of organic light-emitting diode (OLED) [14]. The melting point of MEH-PPV is 250 °C – 300 °C [15]. Previous work stated that MEH-PPV is a potential polymer as it is a high absorption coefficient in the visible range [16]. Moreover, the MEH-PPV act as a copolymer template agent in TiO₂ thin film in DSSC [17]. Therefore, based on the MEH-PPV characteristic, it is suitable to be used as a copolymer template in TiO₂ photoanode of DSSC. By adding MEH-PPV in the photoanode, the structure of TiO₂ is well organized with porous structure [14] where it can trap more dye sensitizer and help in the electron transfer for DSSC application [10].

From the previous research, the unmodified photoanode gave less than 0.05 % for the performance efficiency by using spin coating technique [18]. The research showed that, by adding MEH-PPV into TiO₂, the increment in efficiency value is 65 % [18]. The increment in efficiency value is due to the MEH-PPV that was fully evaporated when it is baked in 450 °C and leave mesoporous TiO₂ structure [18]. Besides, by adding PS sphere in TiO₂, the efficiency value also increased for 51 % [11]. The result of using PS sphere leaves larger pore structure in TiO₂ that enhances the light scattering and increased the optical path length for dye absorption process [19].

The parameters of TiO₂ fabrication process utilizing Dr. Blade's technique were based on the previous experiments [8,20]. In this study, the different weight concentrations (0.25 wt%, 0.50 wt%, and 1.00 wt%) between TiO₂ and organic polymer materials (PS sphere and MEH-PPV) were used as copolymer template. The analysis of TiO₂ added with copolymers template were based on scanning electron microscope (SEM) image. The surface morphology of SEM image were analyzed using ImageJ processing method to evaluate the porosity and particle size of TiO₂. The performance efficiency of complete device was measured by using solar simulator.

2. EXPERIMENTAL METHOD

Indium tin oxide (ITO) glasses were used and cleaned to obtain a clean surface for the next process. The ITO glasses were cut into six-part with the size of 2 cm x 2 cm. It was cleaned by using acetone in the sonicated bath for 10 minutes. Then, the ITO glass was dried on the hot plate for 5 minutes, and the same steps were repeated using ethanol and distilled water. The resistance of each ITO glass was measured and recorded using a multimeter. The average range of the ITO glass resistance was between 16 Ω to 18 Ω. In this study, three different weight concentrations of MEH-PPV and PS sphere mixed with TiO₂ were used: 0.25 wt%, 0.50 wt%, and 1.0 wt%, respectively.

The MEH-PPV was prepared by diluting it with chloroform to get three different weight concentrations that were 0.25 wt%, 0.50 wt%, and 1.0 wt%. Meanwhile, for the PS sphere, it was

diluted with distilled water to get the weight concentrations of 0.25 wt%, 0.50 wt%, and 1.0 wt%, respectively.

Next, 1 g of Degussa P25 powder was weighed and added with a stabilizer. Then, MEH-PPV was added to the TiO₂ mixture, and the mixture was ground until a white fine paste was formed, which is the TiO₂ paste. The same step was repeated by using the other concentration of organic polymers. For the TiO₂:PS sphere, the same step was repeated like TiO₂:MEH-PPV with the same concentrations. The ITO glass was then covered with Scotch tape with an active area of 1.5 cm x 1.5 cm. The TiO₂ was spread on the surface of ITO glass by using Dr. Blade's technique. All 7 photoanodes were annealed in the furnace for 1 hour at 450°C. The characteristic of the photoanode surface structure was analyzed using SEM and image analysis program, ImageJ. ImageJ is a public domain Java image processing program where it can calculate the size of particle and porosity of the materials. A further experiment was conducted by completing the DSSC into a complete device to calculate the performance efficiency.

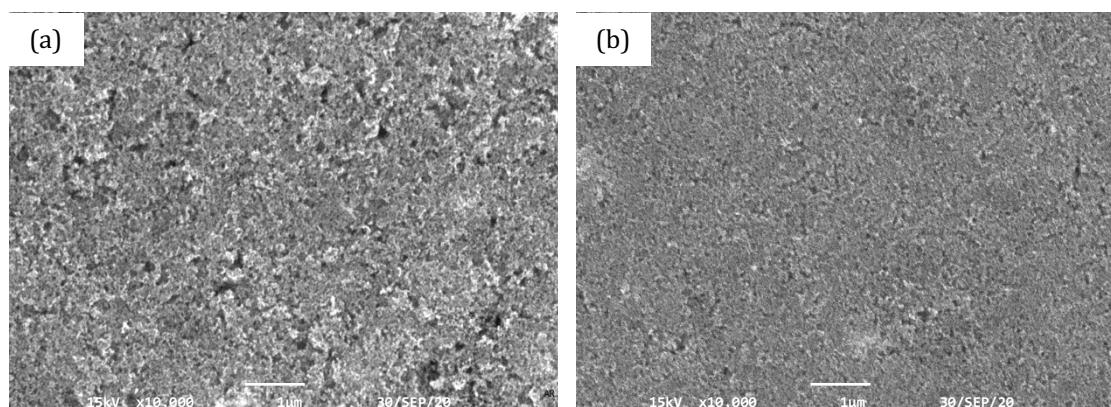
The photoanode was soaked into Ruthenium (N719) dye sensitizer for 24 hours. The photocathode of the DSSC, which was Platinum thin film on the ITO glass surface, was annealed for 30 minutes. The photoanodes were dried before it was assembled with photocathode. The photoanode was clipped together with the photocathode by using a clip at the end of the substrate. Few drops of iodine that act as an electrolyte were dropped between the substrates. The complete device was sent to a solar simulator machine for performance efficiency, and the data obtained were calculated.

3. RESULTS AND DISCUSSION

3.1 Characterization of organic polymer material: surface structure morphology of TiO₂:MEH-PPV and TiO₂:PS sphere using SEM Image

All the samples left a flat surface of TiO₂ with mesoporous structure after the annealing process. The structure was formed due to high annealing temperature (450 °C) which exceed the melting point of both organic copolymers. To differentiate the modified TiO₂, we called TiO₂:MEH-PPV (mh-TiO₂) and TiO₂:PS sphere (ps-TiO₂).

This section discussed the surface roughness for the 7 samples of TiO₂ and TiO₂ mixed with organic copolymer material with different weight concentrations. SEM images (magnification 10,000x) were used in order to analyze the surface structure of the samples. ImageJ is a public domain Java image processing program from the USA's National Institute of Mental Health [21]. This study used the ImageJ program to analyze the porosity [21] and the particle size of TiO₂ and modified TiO₂.



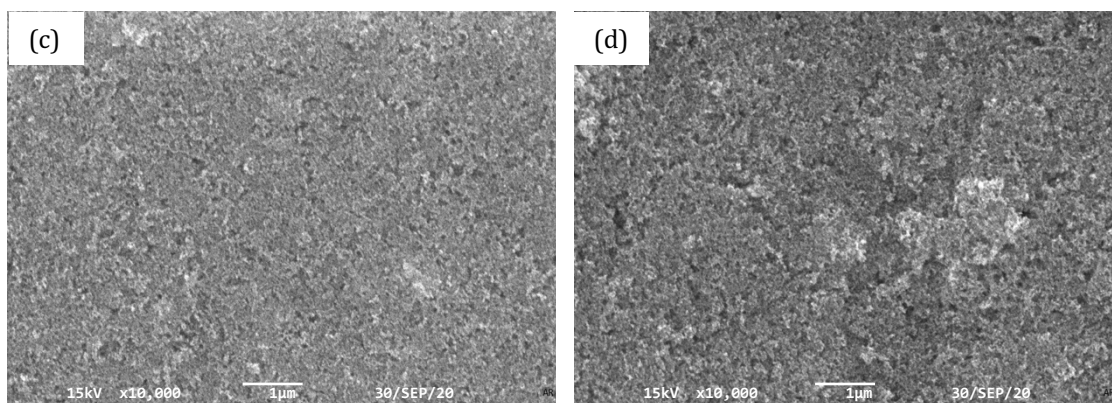


Figure 2. SEM images of TiO₂ and mh-TiO₂ with different weight concentrations. (a) TiO₂, (b) 0.25 wt%, (c) 0.50 wt%, (d) 1.0 wt%.

Figure 2 shows the surface morphology of mh-TiO₂ with three different weight concentrations that are 0.25 wt%, 0.50 wt%, and 1.0 wt% under SEM analysis. Figure 2 (a) shows the sample of TiO₂ where the TiO₂ particles are less compact and like crumpling to each other [22]. The crumpled part is not aggregated smoothly on the surface of the ITO glass. Meanwhile, Figure 2 (b) shows the TiO₂ particle aggregated smoothly on the ITO glass surface and mesoporous structure is formed. As for Figure 2 (c), the porous TiO₂ structure is increasing due to the increment of the weight concentration of MEH-PPV. Figure 2 (c) showed much better porous TiO₂ structure compared to Figure 2 (a). Figure 2 (d) showed the structure of TiO₂ is much better with the increment of weight concentration which is 1.0 wt%. More porous TiO₂ structure was formed due to the evaporated MEH-PPV during the annealing process which modified the agglomerate of unmodified TiO₂ structure. With this new surface structure, it can be applied for DSSC application and could be used for the absorption of photon process in the DSSC.

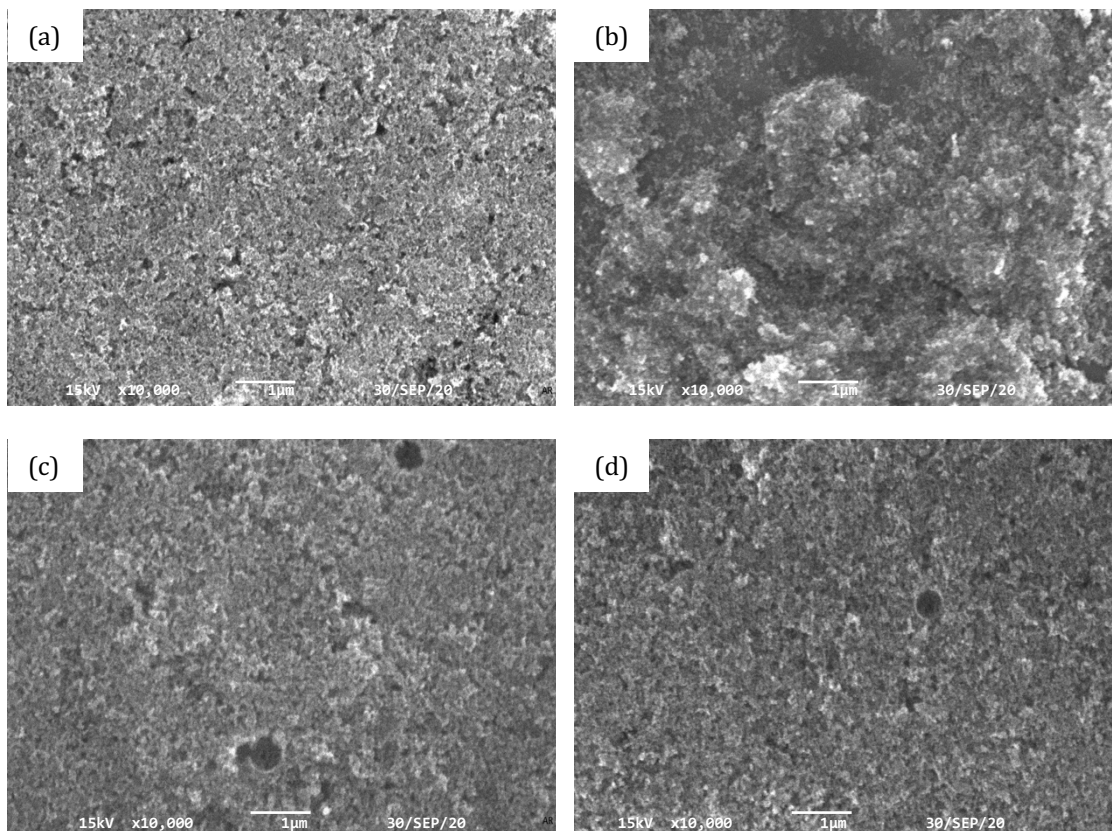


Figure 3. SEM images of TiO₂ and ps-TiO₂ with different weight concentrations. (a) TiO₂, (b) 0.25 wt%, (c) 0.50 wt%, (d) 1.0 wt%.

Figure 3 shows the surface morphology of the ps-TiO₂ with three different weight concentrations that are 0.25 wt%, 0.50 wt%, and 1.0 wt%. In the comparison between Figure 3 (a) and Figure 3 (b), the surface of Figure 3(b) is less crumpled with a few tiny hollow structures. Figure 3 (c) shows the PS sphere tiny hollow structure started to appear more on the surface than the surface on Figure 3 (b). Meanwhile, the surface in Figure 3 (d) shows the tiny hollow structure of the PS sphere increased and can be clearly seen on the surface of the photoanode. The presence of larger pore size in TiO₂ was believed to help in absorbing more dye sensitizer in DSSC.

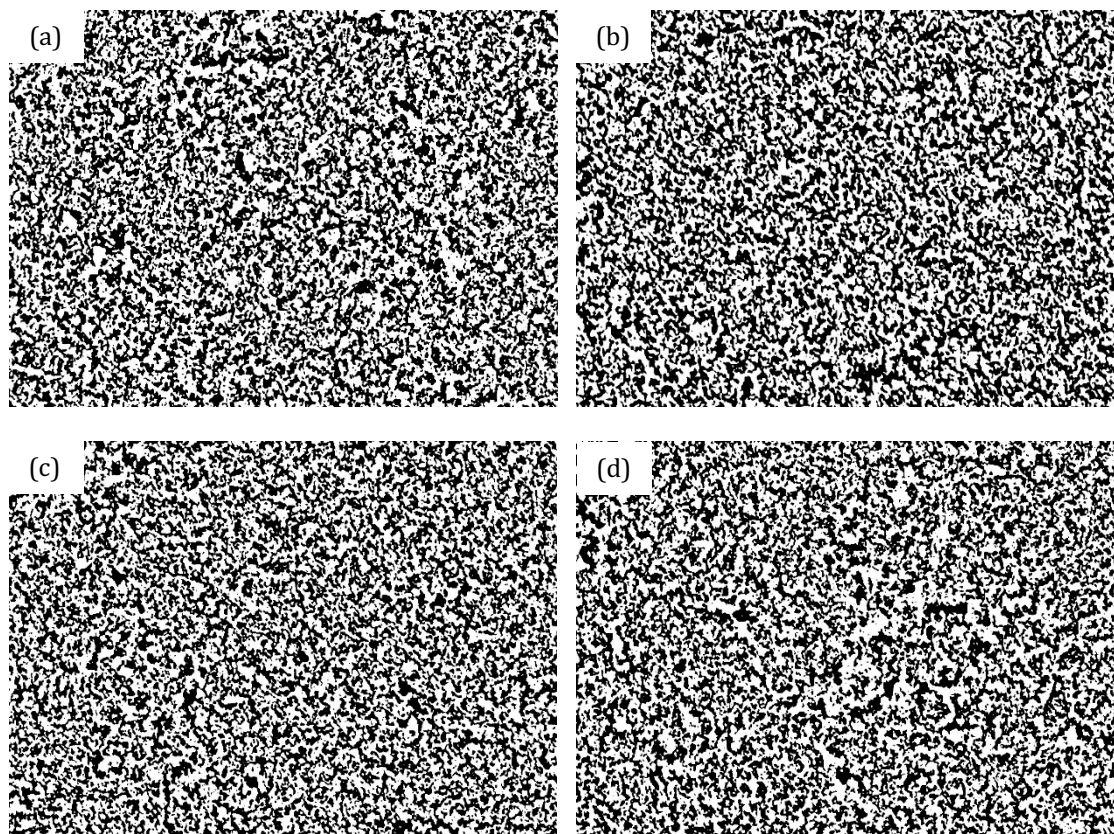


Figure 4. SEM images of mh-TiO₂ analyzed by the ImageJ program with different weight percentages. (a) TiO₂ only, (b) 0.25 wt%, (c) 0.50 wt%, (d) 1.0 wt%.

Figure 4 shows the SEM images from Figure 2 for all samples of photoanode, which are mh-TiO₂ with three different weight concentrations after being analyzed by the ImageJ program. The ImageJ program was used in order to identify the porosity of TiO₂ after being mixed with organic copolymer materials. The white area showed the mh-TiO₂, and the black area showed the unfilled space. From Figure 4, the black area decreased as the weight concentration of the organic copolymer mixed with TiO₂ increased. As the results, in Table 1, the porosity of the TiO₂ mixed with MEH-PPV increased from 44.02% for 0.25 wt% MEH-PPV to 44.29 % for 0.50 wt% MEH-PPV but decreased to 42.51 % with the MEH-PPV of 1.00 wt%. The decrement of 1.78% in the porosity for mh-TiO₂ of 1.00 wt% is probably due to the small inter-particle distance between TiO₂ and MEH-PPV [20]. The addition of MEH-PPV into TiO₂ led the particle size of TiO₂ increased from 2.21 pixel² to 5.69 pixel². Mh-TiO₂ with 0.50 wt% gave 5.84 pixel² and mh-TiO₂ with 1.00 wt% is 7.07 pixel². With the particle size of TiO₂ increased, the number of holes for the particles also increased, as tabulated in Table 1. The dispersion of the particles in the paste related with the degree of mixing TiO₂ with MEH-PPV that gave an important impact on the pore size distribution of TiO₂ and clear broad pore size distribution [21].

Table 1 Characteristics of surface structure for TiO₂ with and without organic polymer materials

Device	Holes	Particle size of TiO ₂ (pixel ²)	Porosity (%)
TiO ₂	4	2.21	45.02
mh-TiO ₂ (0.25 wt%)	5	5.69	44.02
mh-TiO ₂ (0.50 wt%)	15	5.84	44.29
mh-TiO ₂ (1.00 wt%)	18	7.07	42.51

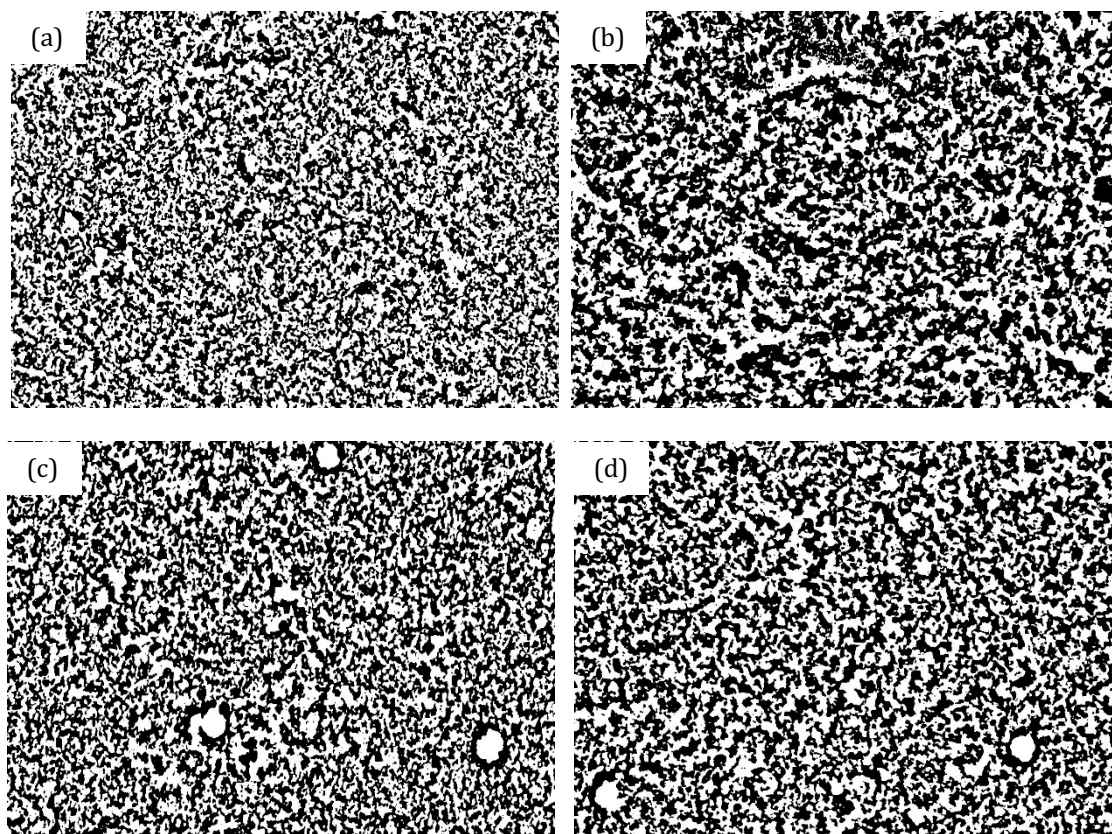


Figure 5. SEM images of ps-TiO₂ analyzed by the ImageJ program with different weight percentages. (a) TiO₂ only, (b) 0.25 wt%, (c) 0.50 wt%, (d) 1.0 wt%.

Figure 5 shows the SEM image from Figure 3 for ps-TiO₂ with different weight concentrations being analyzed using the ImageJ program. The porosity of the photoanode increased as the weight concentration of PS sphere mixed in TiO₂ increased but decreased at the highest concentration, which is 1.00 wt% with a porosity value of 42 %. Meanwhile, the ps-TiO₂ of 0.25 wt% and 0.50 wt% had the porosity value of 44.03 % and 46.40%, respectively. Larger pores were formed as the PS sphere's weight percentage increased and help to increase the porosity of the surface structure [22]. With the addition of PS sphere into TiO₂, the particle size of TiO₂ increased. For example, ps-TiO₂ with 0.25 wt% gave 5.79 pixel² compared to TiO₂ device only that gave 2.21 pixel². For ps-TiO₂ with 0.50 wt%, the particle size is 6.14 pixel² and ps-TiO₂ 1.00 wt%; is 4.71 pixel². The increasing of particle size for TiO₂ also led to the increment of the number of holes which can help in the absorption process. As the weight concentration of PS sphere increased, we can see that porosity of the device increased but decreased as the highest weight concentration (1.00 wt%). The decreasing of porosity when the weight concentration of PS sphere is 1.00 wt% might be due to the formation of large pore size and the agglomeration of PS sphere in TiO₂ paste [23]. The simplified data was tabulated in Table 2.

Table 2 Characteristics of surface structure for TiO₂ with and without organic polymer materials

Device	Holes	Particle size of TiO ₂ (pixel ²)	Porosity (%)
TiO ₂	4	2.21	45.02
ps-TiO ₂ (0.25 wt%)	4	5.79	44.03
ps-TiO ₂ (0.50 wt%)	9	6.14	46.40
ps-TiO ₂ (1.00 wt%)	12	4.71	42.00

3.2 Characterization of organic polymer material: Efficiency of Dye-Sensitized Solar Cell (DSSC)

Table 3 shows the photovoltaic test of DSSC by using mh-TiO₂ and ps-TiO₂ with different concentrations in the N719 dye sensitizer. In addition, there were 7 samples that were analyzed which are TiO₂ and TiO₂ with organic copolymer materials using a solar simulator machine with the help of IV Tracer Software by IVT Solar. The performance analysis was calculated from the open-circuit voltage (Voc), short-circuit current (Jsc), fill factor (FF), and the conversion efficiency of DSSC, η (%).

Table 3 Characteristics of DSSCs using mesoporous TiO₂ and mh-TiO₂ with different weight concentrations

Device	Porosity (%)	Voc (V)	Jsc (mA/cm ²)	Fill Factor (FF)	Efficiency, η (%)
TiO ₂	45.02	0.37	0.005	0.28	0.00053
mh-TiO ₂ (0.25 wt%)	44.02	0.08	0.27	0.23	0.004
mh-TiO ₂ (0.50 wt%)	44.29	0.43	0.41	0.16	0.007
mh-TiO ₂ (1.00 wt%)	42.51	0.11	0.17	0.20	0.01

Based on Table 3, the device of mh-TiO₂ with the concentration of 1.00 wt% obtained the highest efficiency, which is 0.01%, compared TiO₂ only device that gave the efficiency of 0.00053%. As the mh-TiO₂ porosity is 44.02%, high performance efficiency was obtained. This is due to the small inter-particle distance between TiO₂ and MEH-PPV as the porosity lower. Meanwhile, as the porosity increased, the lower performance efficiency was obtained. This is because the high porosity influence the electron injection into TiO₂ film become slower and resulting in higher recombination process of electron.

Table 4 Characteristics of DSSCs using mesoporous TiO₂ and ps-TiO₂ with different weight concentrations

Device	Voc (V)	Jsc (mA/cm ²)	Fill Factor (FF)	Efficiency, η (%)
TiO ₂	0.37	0.005	0.28	0.00053
ps-TiO ₂ (0.25 wt%)	0.44	0.14	0.25	0.01
ps-TiO ₂ (0.50 wt%)	0.39	0.86	0.25	0.08
ps-TiO ₂ (1.00 wt%)	0.46	0.0008	0.36	0.0001

Table 4 tabulates the device's sample for the ps-TiO₂, with the highest efficiency of 0.08% with the weight concentration of 0.50 wt% of the PS sphere. There is a significant increment of efficiency for a device of TiO₂ without organic copolymer with TiO₂ with an organic copolymer which is PS sphere. The addition of the PS sphere into TiO₂ helps in the surface structure of the sample. It helps enlarge the size of holes for TiO₂ particles with larger pore size appearing in the device's surface structure. The efficiency of ps-TiO₂ (1.00 wt%) decreased as the negative contact

and more pores of TiO₂ increased the resistance. This improvement can lead to the boosting of performance for DSSC for the adsorption process of dye sensitizer by allowing the smaller particles from the sensitizer were filling the gap.

4. CONCLUSION

In this paper, the samples of TiO₂ with and without organic polymer materials with different weight concentrations (MEH-PPV and PS sphere) are demonstrated. In addition of organic copolymer (MEH-PPV and PS sphere) into TiO₂, it gives the high efficiency. The increment of ~0.08% efficiency between TiO₂ and modified TiO₂ are observed. The SEM analysis was performed to analyse the surface structure morphology of the devices. As a result, it proves that the addition of organic copolymer into TiO₂ helps improve the surface structure of TiO₂, where it reduces the agglomeration of TiO₂ particles and improves the porosity of the surface structure. Furthermore, the analysis of SEM images by using ImageJ also proved that by adding MEH-PPV and PS sphere with different weight concentrations, the particle size of mesoporous TiO₂ also increased and helped in the performance of efficiency for DSSC.

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