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Functionalized multi-walled carbon nanotubes for enhanced photocurrent in dye-sensitized solar cells

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Abstract

The influence of the incorporation of nitric acid-treated (functionalized) multi-walled carbon nanotubes (f-MWCNTs) and unmodified MWCNTs in TiO₂ films are investigated by observing the photocurrent-voltage characteristics of dye-sensitized solar cells (DSSCs) with and without MWCNTs. The short-circuit photocurrent (J_{sc}) of the modified DSSC increased by 46% compared with that of a cell with plain TiO₂ film. The open-circuit voltage remained the same for all cases. The enhanced J_{sc} is explained by the increased surface area of the film, enhanced cluster formation of TiO₂ particles around f-MWCNTs, and improved interconnectivity of TiO₂ particles in the presence of f-MWCNTs. The efficiency of the cell increased by 45% due to J_{sc} enhancement.

Keywords: Multi-walled carbon nanotubes, Dye-sensitized solar cell, Short-circuit photocurrent, Open-circuit voltage, Cluster formation

Background

Dye-sensitized solar cells (DSSCs) have attracted much attention in the last two decades since reported for the first time by Gratzel [1]. Attempts are ongoing to increase the adsorption of dyes across the solar spectrum, smoothen the function of the transport of photoexcited electrons, and encourage the diffusion of electrolyte ions. The process involves the injection of electrons from the photoexcited dye into the conduction band of the semiconductor oxide (TiO₂), from where they pass through the transparent conducting fluorine-doped tin oxide (FTO) current collector and finally into the external circuit. Furthermore, the sensitizer is regenerated by electron transfer from a donor, typically iodide ions, which are dissolved in the electrolyte present in the porous semiconductor. The triiodide ions formed during the reaction diffuse to the counter electrode, where they are reduced back to iodide by the conduction band electrons that have passed through the external circuit that performs the electrical work.

At present, poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate)(PEDOT:PSS)-coated FTO glass is used as a counter electrode instead of a costly platinum-sputtered FTO glass to catalyze the reduction of I³⁻ to I⁻ in the redox electrolyte [2-4]. Recently, it was reported that the conductivity of PEDOT:PSS film is enhanced if a liquid organic compound such as dimethyl sulfoxide (DMSO), dimethyl formamide (DMF), or tetrahydrofuran is added to the PEDOT:PSS aqueous solution [5,6]. The choice of preparation method for depositing conducting polymers on the conducting glass is also significant in determining the efficiency of DSSCs. Several methods, such as spin coating [7] and chemical polymerization [3], have been employed to deposit conductive polymers on the counter electrode, both of which provide good electrochemical stability. Lee et al. [8] further modified the counter electrode. Using multi-walled carbon nanotubes, they were able to replace the costly platinum electrode, with little change in efficiency of the cell.

Modification of the working electrode is also important. Vincent et al. [9] reported the inclusion of carbon nanotubes in an inorganic TiO₂ sol-gel matrix. Huang and Gao studied the growth behavior of multi-walled

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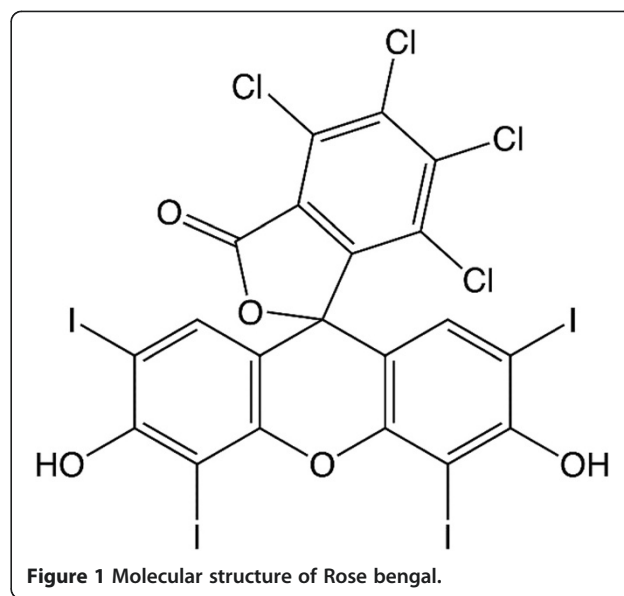
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carbon nanotubes (MWCNTs) by incorporating them on immobilized rutile TiO₂ (r-TiO₂) [10]. Photovoltaic devices were also fabricated by Ago and colleagues using composites of MWCNTs and conjugated polymers [11]. Ionic gel electrolytes were prepared by Usui et al. by dispersing MWCNTs into ionic liquid electrolytes. They achieved higher energy conversion efficiency with corresponding DSSCs, relative to those using untreated ionic liquid electrolytes [12]. It has been reported that the DSSCs fabricated with anatase and r-TiO₂ are comparable in their photovoltaic properties at one-sun intensity (100 mW cm⁻²) [13,14]. It has been shown that r-TiO₂ has superior light-scattering properties compared to anatase TiO₂ because of its higher refractive index. Other benefits of r-TiO₂ are that it is cheaper to produce and is chemically more stable [15]. However, the electron transport is slower in the rutile layer than in the anatase layer because of the interparticle connectivity associated with particle packing density and is evident from the data suggested by intensity-modulated photocurrent spectroscopy and scanning electron microscopy [13]. Sung et al. reported that the introduction of MWCNTs in poly(methyl methacrylate) polymers increased the conductivity of the composite [16]. On treating carbon nanotubes with a concentrated acid mixture of H₂SO₄ and HNO₃ [17-20] or of H₂SO₄ and KMnO₄ [21], carboxylic acid groups can be introduced into the nanotubes in addition to shortening them [22]. It is assumed that MWCNTs attached with carboxylic acid groups can adhere more strongly to TiO₂ particles than untreated MWCNTs due to the strong covalent bond formed between the dye molecule and the TiO₂, resulting in more efficient DSSCs.

In the present study, Rose bengal, an organic dye whose molecular structure is shown in Figure 1, is used as the photosensitizer, and a graphite-coated DMSO-treated PEDOT:PSS is used as the counter electrode for our dye-sensitized solar cell. In the present research, we have preferred MWCNTs and studied their beneficial influence, with and without modification, when incorporated into P25 TiO₂ films and their overall impact on the photovoltaic properties of our DSSCs. Three types of investigations are presented in this paper. First, MWCNTs are incorporated in P25 TiO₂ films and sensitized with Rose bengal dye. In the second investigation, P25 TiO₂ films are sensitized with a solution of Rose bengal dye and f-MWCNTs (MWCNTs functionalized with -COOH), and in the third one, f-MWCNTs are incorporated in the P25 TiO₂ films and then sensitized with Rose bengal dye.

Results and discussion

An FTIR spectrum of MWCNTs is shown in Figure 2a. It was then compared with the FTIR spectrum of

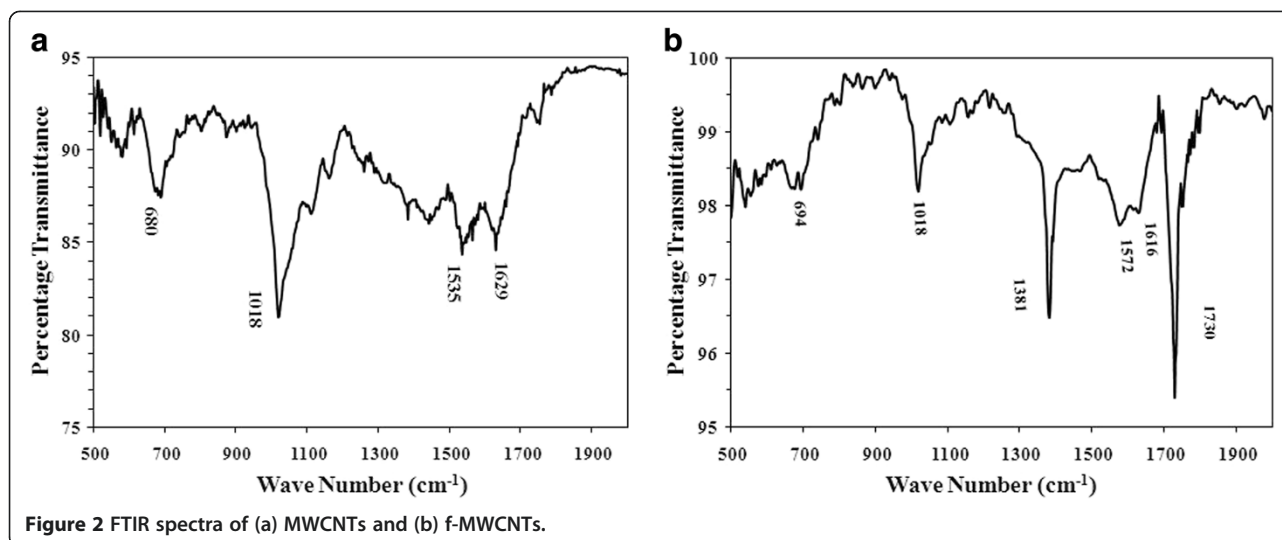


f-MWCNTs shown in Figure 2b. The attachment of carboxylic acid groups to acid-treated MWCNTs (f-MWCNTs) is identified. FTIR spectra of the f-MWCNTs showed a C=O peak of the COOH groups at 1,730 cm⁻¹ and a broad OH peak around 3,400 cm⁻¹ (not shown); an additional C=C peak of MWCNTs occurred near 1,600 cm⁻¹. On the basis of these results, the presence of COOH groups in the f-MWCNTs was confirmed.

As a result of the shortening and simultaneous functionalization with COOH groups by nitric acid, f-MWCNTs dispersed well in water [23] and DMF as shown in Figure 3. Their good miscibility with water is beneficial for the fabrication of TiO₂ film electrodes.

The AFM images of the prepared films, unmodified TiO₂ film (Figure 4a), MWCNT-incorporated TiO₂ film (Figure 4b), and f-MWCNT-incorporated TiO₂ film (Figure 4c), were taken and analyzed. Interestingly, the calculated root-mean-square (RMS) roughness values were 117, 153, and 103 nm, respectively. By the introduction of MWCNTs, the roughness value was found to have increased due to the MWCNTs not fitting well in the gaps between the TiO₂ nanoparticles. However, when the MWCNTs were functionalized, the RMS roughness value was found to have decreased. This reduction in roughness was attributed to the smoothing effect induced by the f-MWCNTs as they better fit in the gaps between the TiO₂ nanoparticles. This indicates that there is more favorable cluster formation in P25 TiO₂ films in the presence of f-MWCNTs. Also, there is an increased surface area of the films fabricated in the presence of f-MWCNTs.

Figure 5 shows the Raman spectra of the TiO₂ film which is compared with the TiO₂ film in the

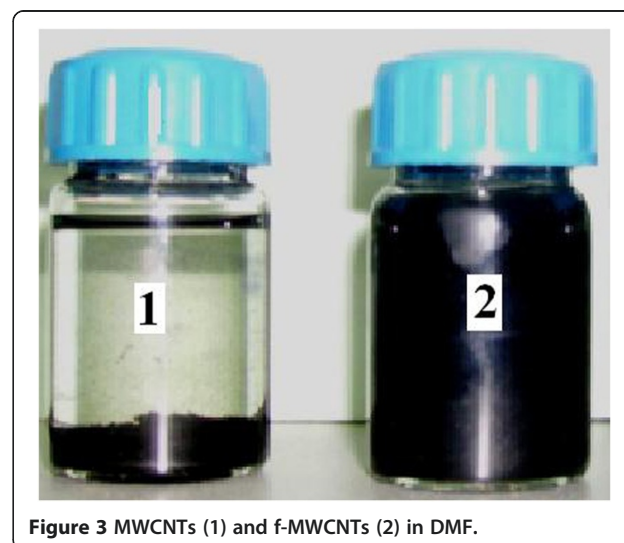


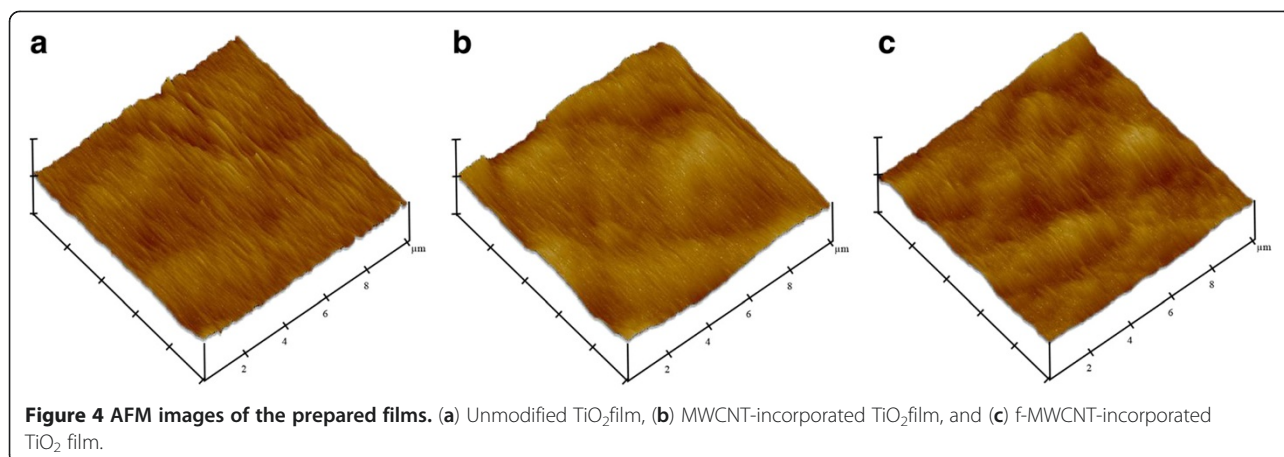
presence of MWCNTs and f-MWCNTs. It is found that the MWCNT-incorporated film shows blueshifts by 4 cm^{-1} and f-MWCNTs shifts by 7 cm^{-1} . These blueshifts can be attributed to the strain effects [24] at the f-MWCNT/TiO₂ interfaces, which may influence the vibration frequencies. The differences in the Raman spectra between f-MWCNTs versus MWCNTs are due to the increased adherence of TiO₂ particles to f-MWCNTs.

The optical absorption spectra of the P25 (the standard electrode), PE1, PE2, and PE3 electrodes are shown in Figure 6. It is seen in the figure that for all the photoelectrodes, the absorption band extends from 320 to 600 nm, but the intensity of absorption is different for different electrodes. The graphs show that the dye-enhanced absorption in PE3 is more than that of other electrodes. This is due to the increased surface area of the films fabricated in the presence of f-MWCNTs.

Figure 7 shows the current density-voltage (J - V) curves of DSSCs made by incorporating MWCNTs, with and without functionalization, in the TiO₂ films, compared with that of a cell fabricated with P25 film only. The results are summarized in Table 1. The illumination intensity used was 100 mW cm^{-2} . Compared to a cell fabricated with P25 film only, the cell with PE1 shows a higher J_{sc} by about 5%. Similarly, the cells with PE2 and PE3 show J_{sc} improvements of about 17% and 46%, respectively. However, there are hardly any changes in the open-circuit voltage (V_{oc}) of the four cells. The essential reason for the enhanced J_{sc} is attributed to the increased surface area of the TiO₂ films in the presence of f-MWCNTs. This can be verified by measuring absorbance of desorbed dye from the TiO₂ film. Enhanced quantity of dye adsorption is indicative of the enhanced surface area of TiO₂ film for a monolayer of dye on the TiO₂ particles.

Absorbance measurements of the desorbed dye from TiO₂ films with and without f-MWCNTs in Figure 8 indicate increased dye adsorption on the surface. The J_{sc} increases are consistent with this order of increase of surface area. The increased surface area of TiO₂ films with the incorporation of f-MWCNTs may be due to additional adsorption sites gained from the adherence of TiO₂ particles to f-MWCNTs that are otherwise not available when TiO₂ particles aggregate themselves in the P25 film. As a result of the J_{sc} enhancements, the overall energy conversion efficiency (η) increases by 7%, 32%, and 60% for the cells prepared with films PE1, PE2, and PE3, respectively, when compared to that of the cell made with P25 film only. The surface area of the TiO₂ films is increased in the presence of f-MWCNTs which

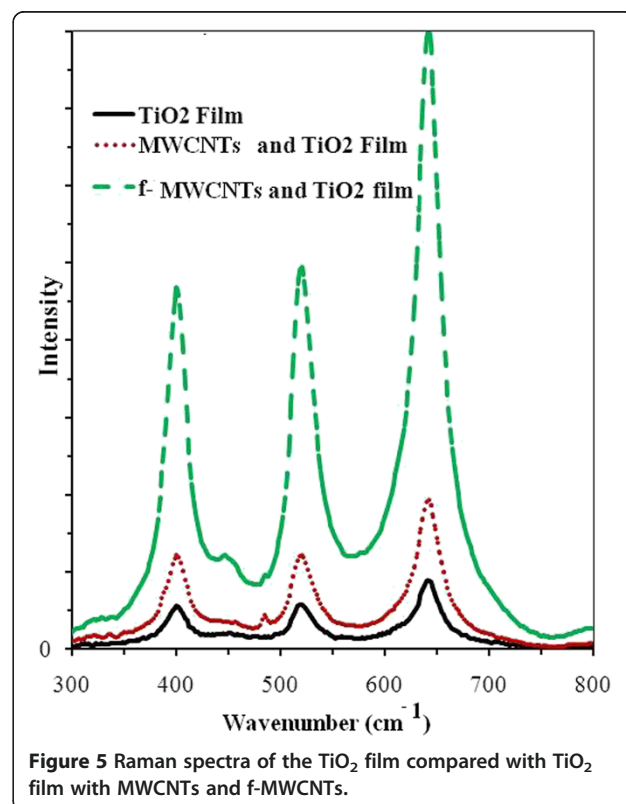




is the reason for the enhanced J_{sc} . This is evident from the absorption spectra of the photoelectrodes (Figure 6).

Enhanced quantity of dye adsorption in the presence of f-MWCNTs is indicative of the enhanced surface area of the TiO₂ film. This suggests that the surface area of TiO₂ films for dye adsorption has increased in the order of surface area of P25 < PE1 < PE2 < PE3. It may be due to additional adsorption sites gained from the adherence of TiO₂ particles to f-MWCNTs. The J_{sc} increases are consistent with this order of increase of surface area. The increased surface area of TiO₂ films and the incorporation of f-MWCNTs provide for better dispersion of TiO₂ particles compared to TiO₂ particle aggregation in unmodified P25 film. Because of the fine distribution of f-MWCNTs in the TiO₂ films, as can be understood from the AFM images (Figure 5), a better dispersion of TiO₂ particles in the PE3 can be visualized. The more even dispersion of TiO₂ particles provides additional adsorption sites for the dye. The favorable TiO₂ cluster formation induced by f-MWCNTs in the films on the order of that of P25 < PE1 < PE2 < PE3 is in agreement with the order of the J_{sc} increase. AFM studies of the photoelectrodes also verify the same. The J_{sc} enhancement can also be explained by the increase in interconnection among the TiO₂ particles in the TiO₂ films prepared with f-MWCNTs. Formation of f-MWCNT-induced clusters, as mentioned already, enables an increase in interconnection among the TiO₂ particles in the film, relative to that among particles in films without f-MWCNTs. The increased interconnectivity in turn increases the electrical conductivity of the film in the presence of f-MWCNTs. In addition to improved interconnectivity among TiO₂ particles in the films in the presence of MWCNTs, the anchoring of TiO₂ particles to nanotubes can promote charge separation, owing to the fact that carboxylic acid groups of f-MWCNTs are also able to attach themselves to TiO₂ particles. Since absorbance is proportional to the free

electron density in the conduction band of TiO₂, the implication is a higher electron transport in films PE1, PE2, and PE3. The absorbance pattern of films P25, PE1, PE2, and PE3 are consistent with their respective J_{sc} enhancements. Electrons trapped in the surface states appear to be responsible for the slower photocurrent response in the case of the DSSC with bare P25 film [25,26]. It is to be emphasized here that the performance of the cell has not been optimized with regard to film thickness, dye purification, and device architecture. This study focused on the influence of MWCNTs and TiO₂/f-MWCNTs, when incorporated



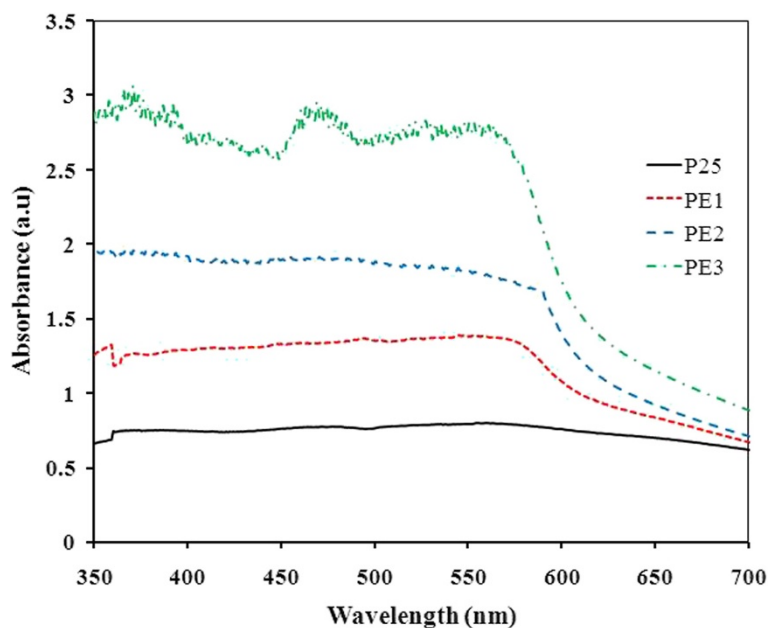


Figure 6 Optical absorption spectra of P25, PE1, PE2, and PE3 electrodes.

in P25 TiO₂ film, on the photovoltaic properties of the corresponding DSSCs. Comparative measurements are made in this regard with reference to a cell without f-MWCNTs.

Conclusions

Incorporation of acid-treated f-MWCNTs in P25 TiO₂ films yields a considerable increase in J_{sc} of about 46%

over unmodified P25 TiO₂ films with the same open-circuit voltage for the pertinent dye-sensitized solar cells, with respect to the unmodified cell. The enhanced essential J_{sc} is attributed to the increased surface area of the films fabricated in the presence of f-MWCNTs. The J_{sc} enhancement also arises from more favorable cluster formation in P25 TiO₂ films in the presence of f-MWCNTs than that in the absence of them. Improved

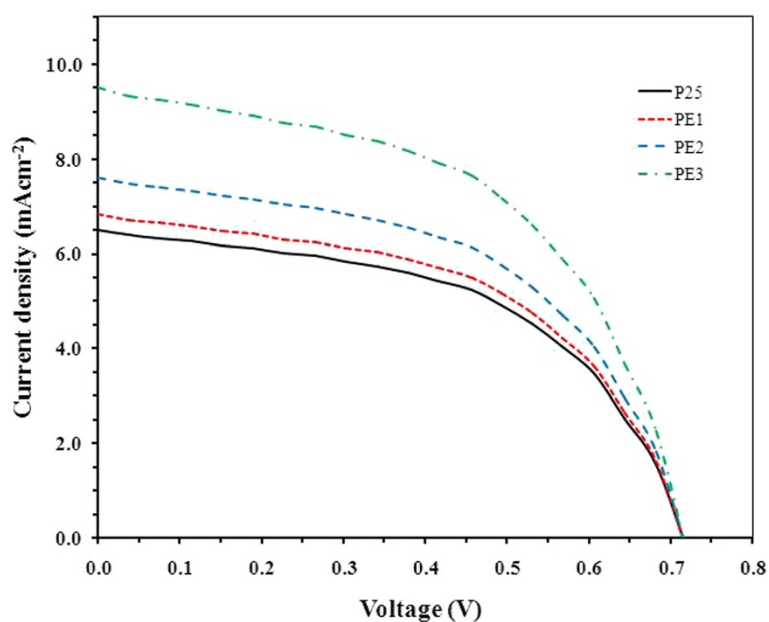


Figure 7 J-V curves of DSSCs made by incorporating MWCNTs in the TiO₂ films.

Table 1 Photovoltaic parameters of the DSSCs with different photoelectrodes

Photoelectrode	J_{sc} (mAcm ⁻²)	V_{oc} (V)	FF	Efficiency(%)
P25	6.51	0.68	0.54	2.41
PE1	6.83	0.68	0.54	2.53
PE2	7.62	0.68	0.54	2.82
PE3	9.50	0.68	0.54	3.52

interconnectivity among TiO₂ particles in the presence of the f-MWCNTs is seen as another reason for the J_{sc} enhancements. The J_{sc} enhancement is also consistent with the increase in dye adsorption of the respective photoelectrodes in the cells.

Methods

Materials

MWCNTs with a length of 1 to 2 μ m and a diameter of 10 to 30 nm were prepared by CVD and purchased from Intelligent Materials Pvt. Ltd., Chandigarh, India. Fluoride-doped tin oxide-coated glass substrate with a resistance of 10 Ω /cm² was purchased from Solaronix, Aubonne, Switzerland. Titanium dioxide (Degussa P25) and Rose bengal (RB) dye were procured from Orion Chem. Pvt. Ltd., Navi Mumbai, India, and HiMedia Laboratories Pvt. Ltd., Mumbai, India, respectively. For electrolyte preparation, polyethylene glycol (PEG;MW 400), potassium iodide, and iodine were procured from Aldrich, St. Louis, MO, USA. PEDOT:PSS is also from Aldrich. Other chemicals such as DMSO, nitric acid,

DMF, 2-propanol, acetylacetone, Triton X-100, and acetonitrile are from Merck, Mumbai, India.

Preparation of photoelectrodes

The conducting glass substrates (FTO) are cleaned and rinsed with deionized water and 2-propanol and then soaked in propanol for 12 h. The FTO substrates are air-dried prior to film preparation. The TiO₂ colloidal solution is prepared by grinding the TiO₂ (Degussa P25) powder purchased from Aldrich with 2 ml of distilled water and acetylacetone for 30 min. Finally, 8.0 ml of distilled water and 0.1 ml of Triton X-100 are added with continuous mixing for 10 min, and the FTO is coated using the doctor blade technique. Functionalization of MWCNTs is done as follows: 10 mg of MWCNTs is mixed with 70% concentrated HNO₃ and is refluxed for 8 h at 140°C. The resulting product is cooled and centrifuged at 4,500 rpm for 30 min, followed by washing with distilled water at 3,000 rpm for 45 min. MWCNTs and f-MWCNTs in DMF are shown in Figure 3. It can be seen that f-MWCNTs disperse well in organic liquids. The final product is dried at 80°C, designated as f-MWCNTs, and characterized by Fourier transform infrared (FTIR) spectrophotometry and Raman spectroscopy.

The standard photoelectrode, P25, was prepared as follows: TiO₂ was coated on FTO conducting glass and fired at 450°C for 30 min, and Rose bengal dye was adsorbed through 12 h of sensitization. The other electrodes, PE1, PE2, and PE3, were prepared as follows: PE1: MWCNTs (0.5mg) were incorporated in P25 TiO₂ (1.2 g) by direct mixing to form a colloidal paste, coated

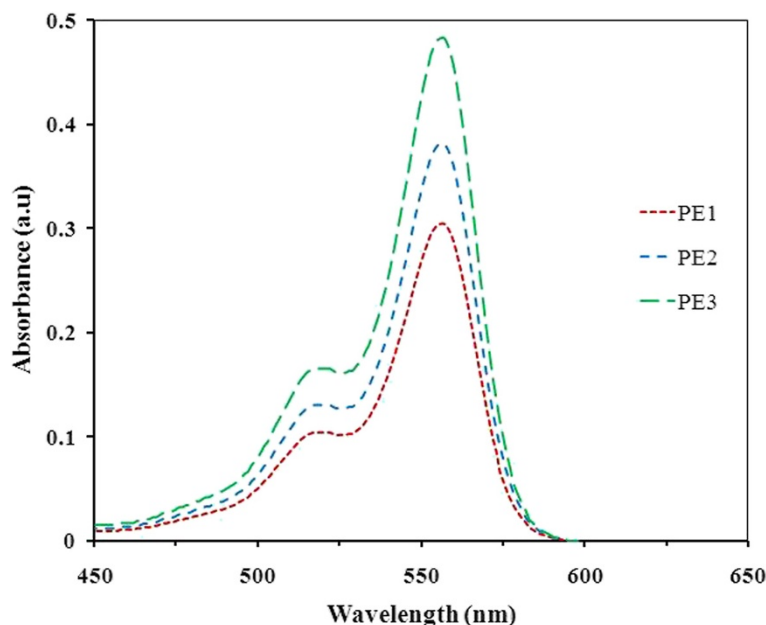


Figure 8 Absorbance measurements of the desorbed dye from TiO₂ films with and without f-MWCNTs.

on FTO conducting glass, and fired at 450°C and Rose bengal dye was adsorbed through 12 h of sensitization. PE2 was made by sensitizing fired P25 TiO₂-coated FTO glass with a mixture of Rose bengal dye and f-MWCNTs. PE3 was made by sensitizing the Rose bengal dye on fired FTO glass coated with a mixture of P25 TiO₂ and f-MWCNTs. Nearly the same TiO₂ film thickness is maintained in all four cases, this being 16 μm. The reason for these thick films is to prevent MWCNTs from protruding from the films, which could have caused short circuits in the DSSCs.

Preparation of counter electrode and assembling of DSSC

The PEDOT:PSS-coated counter electrode is prepared as explained in [27]. The dye-sensitized solar cell is assembled by clamping the dye-sensitized photoelectrode with a DMSO-treated PEDOT:PSS-coated FTO counter electrode to form a sandwich-type structure. Electrolyte solution was prepared as reported in [27], by taking a proportionate quantity of 0.5 mol of KI, 0.05 mol of iodine, and 0.14 mol of PEG (MW 400) in 50 ml of acetonitrile solvent. The conductivity of the electrolyte solution is recorded with a conductivity bridge meter, and it was found to be 13.6 mS cm⁻¹. One drop of this electrolyte solution was injected into the interspace between the photoelectrode and the counter electrode. The schematic diagram of the device is shown in Figure one of our previous paper [27].

The absorption spectrum of the fabricated device was recorded using an Analytik Jena photodiode array (New Delhi, India) or a PerkinElmer Lambda 35 spectrophotometer (Waltham, MA, USA). FTIR spectra were obtained using a Shimadzu IRAffinity-1 FTIR spectrophotometer (Kyoto, Japan) and were used to identify the formation of carboxylic acid groups on the f-MWCNTs. Raman spectra were obtained using an Avalon Instruments Raman Station R3 (Boston, MA, USA) in which a green laser of λ=532 nm was used. The scanning range was from 200 to 4,000 cm⁻¹ to identify the attachment of TiO₂ to f-MWCNTs. Atomic force microscopy (AFM) images were taken using a Veeco Digital Instruments AFM Nanoscope 111A, software version 5 (Plainview, NY, USA). The tips used were contact mode tips. Photocurrent-voltage curves were measured using a Keithley Electrometer 6517A (Cleveland, OH, USA). A 300-W Xe lamp with an AM 1.5 spectrum and an output power of 100 mW cm⁻² was used to illuminate the active area, 1 cm² of the TiO₂ electrode.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

Jl and KBSP fabricated and characterized the device, and COS, KSS, and MSR performed the technical analysis. COS, KSS, and MSR participated in the

design of the study and coordination. All authors read and approved the final manuscript.

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