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Study about TiO_2/Nb_2O_5 Composite Electrode Thin Films for Photovoltaic Applications

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ABSTRACT

Nanopowders can be defined as powdered materials with individual particles in nanometer scale or materials with crystalline in nanometer scale. Nanoparticles (NPs) are made up of a large amount of atoms or molecules bonded with each other with a total size varying from 1 nm to around 100 nm. Due to their very small sizes, NPs possess an extraordinarily high surface area-to-volume ratio, which changes their physical-chemical properties compared to their macroscale counterparts. Nano sized powders of TiO_2 (titanium dioxide) and Nb_2O_5 (Niobium (V) oxide) were used to fabricate TiO_2/Nb_2O_5 composites thin films by EPD (electrophoretic deposition) technique. In an EPD cell, pellets of magnesium nitrate hexahydrate and metal oxide powders were suspended in propan-2-ol. 1.2 cm apart, the electrodes were submerged in the suspension with a DC potential supplied across them. Through visual examination and analysis of UV-Vis-NIR spectrophotometer spectra, key EPD process parameters, including as the applied DC electric field, deposition duration, and solid concentration in suspension, were improved. The films with deposition times of 90 s, powder concentrations of 0.01 g/40 mL, and direct current (DC) voltage of 35 V had the maximum transmittance (55%) of all the samples. XRD micrographs confirmed that TiO_2 and Nb_2O_5 particles were presented in the composite film. SEM (scanning electron microscope) micrographs of the composite electrode thin films showed that porous films of high quality with well controlled morphology were deposited by using the EPD technique.

Keywords: Electrophoretic deposition; TiO_2/Nb_2O_5 composite electrode thin films.

1. INTRODUCTION

Renewable energy technologies, particularly the integration of photovoltaic (PV) systems in the building envelope, are playing a major role in improving the

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building energy performance and reducing environmental effects [1]. Renewable energy technologies generate electricity and heat from renewable sources such as wind and sunlight. But manufacturing such technologies requires nonrenewable raw materials, which should ideally be used in sustainable ways [1a]. The development and application of renewable energy technologies (RET) is the focus of increased scientific research to help mitigate against challenges of climatic change and the possible depletion of the fossil fuels. Solar energy represents an important renewable energy resource which is carbon free and ubiquitous, with terrestrial potential about 600 TW. It is also useful for off-grid utilizations. The commonly used photovoltaic systems are crystalline silicon-based with positive attribute of high power conversion efficiencies (24.7%) [1b]. However, these modules require vacuum-based techniques for fabrication resulting in high production costs. A lot of research is focused on developing solar cells that require low production costs and have adequate conversion efficiencies. Consequently, among the excitonic solar cells, the DSSC (dye-sensitized solar cells) have attracted widespread interest owing to their easy fabrication processes, low production costs, less sensitivity to impurities, wide range of temperature for optimal operation, compatibility with glass and flexible substrates and fair solar-to-electrical conversion efficiencies [2].

Although DSSCs have reached conversion efficiencies of 11.6% [3], there are still challenges to be resolved before efficiency can be increased and the cells can be replicated on a big scale. A porous semiconductor material (photoelectrode) is used in dye-sensitized solar cells to conduct injected photoelectrons while absorbing dye molecules. By broadening the optical adsorption spectrum of the wide energy gap (Eg > 3 eV) semiconductor, the dye molecules perform the role of photon capture. Titanium dioxide (TiO2) is the semiconductor that is most frequently utilized to create DSSC photoelectrodes because it has a high energy conversion efficiency [2]. The high efficiency is the result of titanium dioxide films providing ease of electron transmission through them, thus enhancingelectron conduction to the conducting glass before recombination. However, to further reduce recombination and increase efficiency, research efforts are geared towards modification of the photoelectrode through use of other large band gap semiconductors and through use of composites [4,5]. According to Lin et al. [6], the importance of thin film technology in fabricating solar cells is that thin films shortens the diffusion length of photo generated carriers and decreases the recombination. Use of thin films saves on material and simplifies the synthesis of solar cells. Modification of photoelectrode can also be achieved by use of composites [7,8].

Composites are thought of as designed materials composed of two or more constituent materials with distinctly different physical or chemical characteristics that continue to exist at the microscopic level inside the completed product. A number of authors [9-11] have proposed that Nb_2O_5 (niobium (V) pentoxide), a wide band gap (Eg = 3.49 eV), and n-type semiconductor, can be combined with TiO_2 to fabricate composite thin films. Thin films of TiO_2 have been fabricated by varieties of methods like doctor blade [12], power screen printing [13], DC magnetron [10] and CVD (chemical vapor deposition) [14]. Some of these methods require high power consumption and vacuum chambers. In addition, EPD (electrophoretic deposition) technique has not been fully exploited in photoelectrode preparation for dye-sensitized solar cells despite being a relatively cheap technique and which could be scaled up for mass production if successfully developed.

Electrophoretic deposition (EPD) process is based on the movement and deposition of charged particles under electric field onto a conductive electrode to develop thin or thick films and coatings [14a].

By applying a DC electric field, charged particles are made to travel in the direction of and deposit on an electrode that is negatively charged in the EPD colloidal approach [15–17]. A few benefits of the EPD technology include its wide application to a variety of materials, low production temperature, quick deposition time, fast deposition speed, and inexpensive equipment for its construction.

Further, EPD does not require highly purified materials and vacuum chambers for film deposition [12,14,18]

Literature survey reveals that authors have used diverse values for EPD process parameters and therefore, there is need to optimize the same parameters for composite deposition [17,19-22]. As a result, EPD process parameters have been optimized and the technique used to deposit good quality-nanocrystalline and nanoporous TiO_2/Nb_2O_5 composite electrode thin films for application in the dye-sensitized solar cells.

2. MATERIALS AND METHODS

2.1 Materials

Commercial TiO_2 nanopowder (CAS No. 13463-67-7, Aldrich), Nb_2O_5 nanopowder (CAS No. 1313-96-8, Acros Organics BVBA, Belgium), 5×10^{-5}M-Magnesium nitrate hexahydrate ($Mg(NO_3)_2 \cdot 6H_2O$ 99.9%, Aldrich) and propan-2-ol (IPA: Isopropyl alcohol) (Scharlau chemie) were used in the study.

Glass substrates (16 mm × 25 mm × 1 mm) covered with a conducting layer of Fluorine doped TiO_2 (FTO (fluorine doped tin oxide)) (Pilkington, Hartford Glass Co. Inc., USA) having sheet resistances of 8 Ω/square were used as electrodes in EPD setup. These glass substrates were cleaned with soap detergent mixed with sodium hydroxide and then sonicated for 10 min in distilled water to remove any stains that could interfere with adhesion of glass surface deposited film.

2.2 Apparatus and Instruments

The equipment used included: Topedo weighing balance (Japan) used to weigh amount of metal oxide powder; Pyrex glass beaker as EPD cell, power sonic 405 used for cleaning FTO glass slides and to stir (10 min) the suspension to ensure homogeneity; DC power supply (Thurlby Thadar TS30225, 30 V, 2 A dual power supply) and UV-Vis-NIR spectrophotometer.

2.3 EPD Deposition of the Composite Film

Film preparation involved electrophoretic deposition of the TiO_2 and Nb_2O_5 nanoparticles on fluorine-doped TiO_2 (FTO) glass slide. To achieve this deposition, TiO_2 and Nb_2O_5 nanopowders were mixed with 40 mL propan-2-ol in a Pyrex glass to form the EPD suspension. Magnesium nitrate hexahydrate pellets were added to suspension to provide magnesium ions to attach to semiconductors and thereby control the zeta potential of suspension as reported by Refs. [18,19]. The pH of suspension was measured using digital pH meter. Experimental set up of electrophoretic deposition technique comprised of cathode and anode electrodes which were made of glass slides coated with conductive layer of FTO (fluorine doped tin oxide). The electrodes were arranged in a parallel configuration and partially immersed in suspension as shown in Fig. 1 [23]. A DC electric field was then applied across the electrodes.

2.4 Methods

Various amounts (0.01 g to 0.3 g) of TiO_2 and Nb_2O_5 powder were added into 40 milliliters of propan-2-ol contained in a glass beaker to form EPD suspension. In each case, the two metal powders were mixed in ratio of 1:1. The suspension was then stirred using power sonic 405 vibrator for 10 min. The color of the suspension whose pH was 4.9 turned milky white after vibration. Various values of direct current voltage ranging from 25V to 60V were applied across electrodes. Electrophoretic deposition was then carried out using varied deposition times (60 s to 180 s). Film deposition occurred on the cathode showing that TiO2 and Nb2O5 particles acquired positive surface charges when suspended in propan-2-ol.

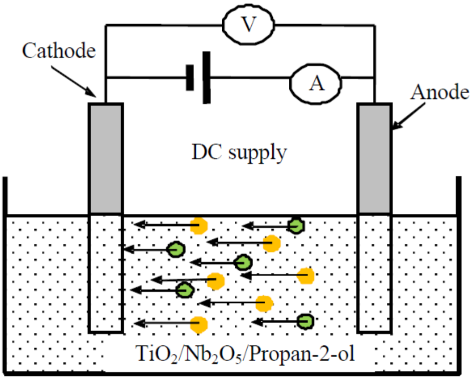


Fig. 1. A schematic drawing of the EPD setup showing the two FTO covered glass slides partially immersed into TiO_2/Nb_2O_5/propan-2-ol suspension [24,25]

The fabricated films were annealed at 400 °C for 30 min and then allowed to cool gradually to room temperature (25 °C).

The TiO_2/Nb_2O_5 composite electrode thin films were assessed from visual inspection and using UV-Vis-NIR spectrophotometer (transmission spectra) to judge the quality and morphology of the deposited films. Morphology of films was further determined using SEM (scanning electron microscope).

3. THEORY/CALCULATIONS

3.1 Theory of EPD Parameters

The mass of electrophoretically deposited film on conducting substrate is related to EPD parameter according to Hamaker's equation [15]:

$$m = \frac{2}{3} C \varepsilon_0 \varepsilon_r \xi \left(\frac{1}{\eta} \right) \left(\frac{V}{l} \right) t \tag{1}$$

where, *m* is deposit yield, *C* is particle mass concentration in the suspension, $_0$ is permittivity of free space, *r* is relative permittivity of the solvent, ξ is the zeta potential of the particles, is the viscosity of the solvent, *V* is the applied DC voltage, *l* as the distance between the electrodes, and *t* is the deposition time.

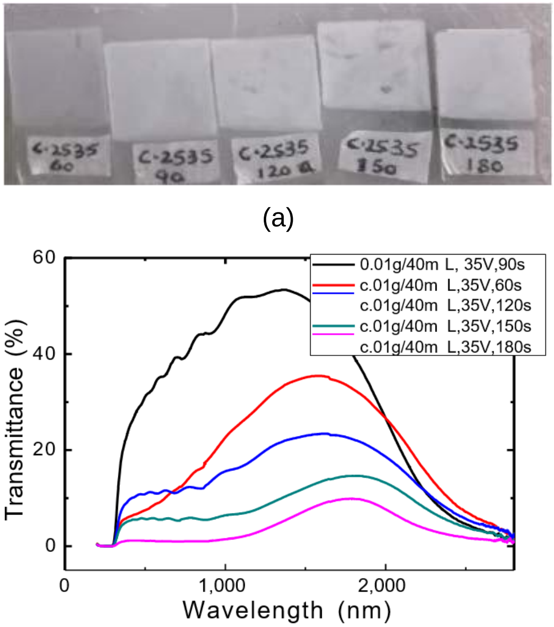
The parameters in Eq. (1) can be categorized into process parameters and materials-related parameters. Process related parameters include concentration (*C*), electric field (*E* = *V*/*L*) and deposition time (*t*). The Hamaker's equation reduces to:

$$m \propto c E t \tag{2}$$

Therefore, concentration, voltage and deposition time are the process related parameter we sought to optimize for fabrication of TiO_2/Nb_2O_5 composite electrode thin films.

3.2 Optimization of Deposition Time

Figs. 2 and 3 show results for optimization of deposition time. The photograph and corresponding transmittance spectra shown in Figs. 2a and 2b are for TiO_2/Nb_2O_5 composite electrode thin films electrophoretically deposited at 35 DC Voltage, from 0.01 g/40 mL suspension and for deposition times from 60 s to 180 s.



(b)

Fig. 2. (a) Photographs and (b) Transmittance spectra for TiO_2/Nb_2O_5 composite electrode thin films electrophoretically deposited from 0.01 g/40mL, at 35 DCV for varying deposition time

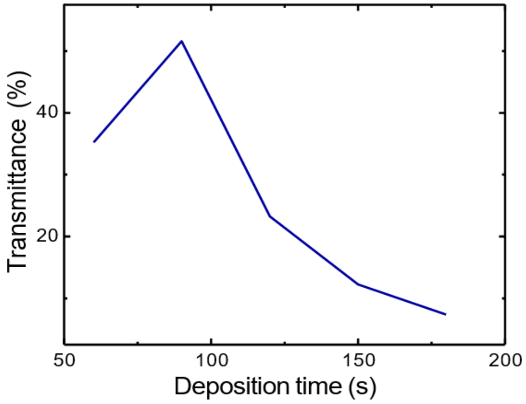
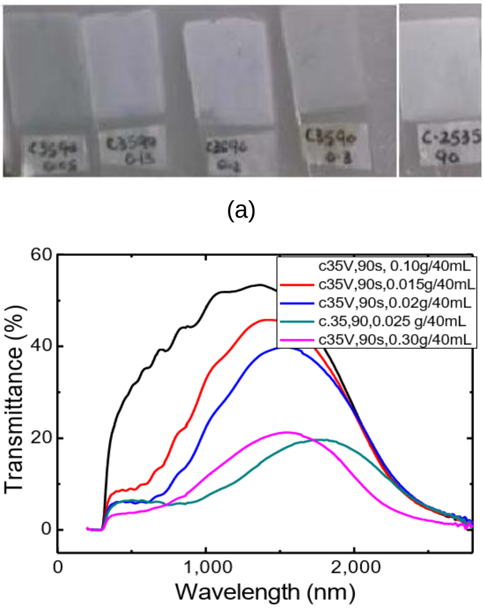


Fig. 3. Variation of transmittance (at 1,508 nm) with deposition time for films electrophoretically deposited at 35 V from 0.01 g/40 mL suspension

Transmittance increased from 0% to 55% in case of 90 s deposition time and then decreased with increased time (Fig. 3). The maximum value of transmittance was for deposition time of 90 s, which is therefore, the optimum deposition time. For short deposition times (< 90 s), the transmittance decreased since time was too short for formation of appreciable layer of film from the low level of concentration. Longer deposition times yielded thicker films, which led to reduction in the transmittance values. Thick films are generally non-porous and do not absorb much dye. They are undesirable in solar cells applications.

In addition, the best quality films from visual inspection correspond to deposition time of 90 s.

3.3 Optimization of Concentration



(a)

(b)

Fig. 4. (a) Photographs and (b) transmittance spectra for TiO_2/Nb_2O_5 composite electrode thin films electrophoretically deposited at 35 DCV for 90 s and from varying powder concentration

Figs. 4 and 5 show the results for optimization of concentration at voltage of 35 V ad deposition time of 90 s. Fig. 4a show the photographs from digital camera of the TiO_2/Nb_2O_5 composite electrode thin films electrophoretically deposited from powder concentration from 0.05 g/40 mL to 0.3 g/40 mL. The corresponding transmission spectra for varied concentrations are shown in Fig. 4b. The concentration of 0.01 g of metal oxide powders in 40 mL of propan-2-ol (equivalent to 0.25 g/L) produced electrophoretically deposited films with a 55% transmittance, which was the highest obtained in the study. Visual inspection showed that the films corresponding to 0.01 g/40 ml concentration had the best quality films.

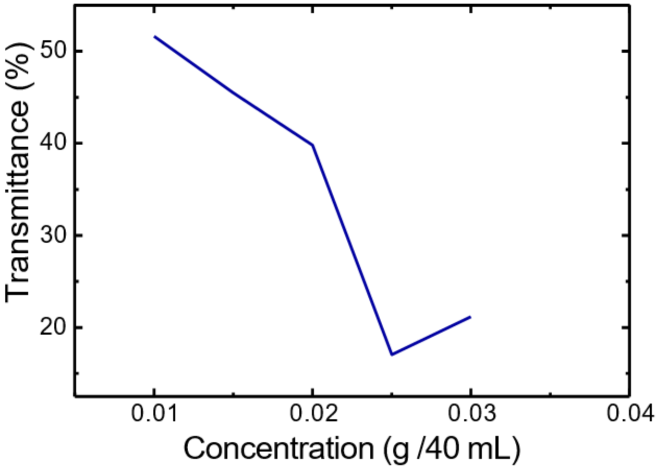


Fig. 5. Variation of transmittance (at 1,508 nm) with concentration for films electrophoretically deposited at 35 V from 0.01 g/40 mL suspension

Fig. 5 shows that for fixed applied voltage and deposition time, transmittance decreased with increased concentration. This decrease in transmittance resulted from development of thick films which hinder transmittance of light rays. Further, films deposited at high concentration would be non-porous and unsuitable for dye absorption for solar cell application. The uncoated glass gave the highest transmittance value. It was difficult, therefore, to determine the minimum concentration from transmittance values as shown in Fig. 5. Powder concentration of 0.01 g/40 mL was, therefore, chosen as optimum EPD concentration value for fabrication of TiO_2/Nb_2O_5 composite electrode thin films.

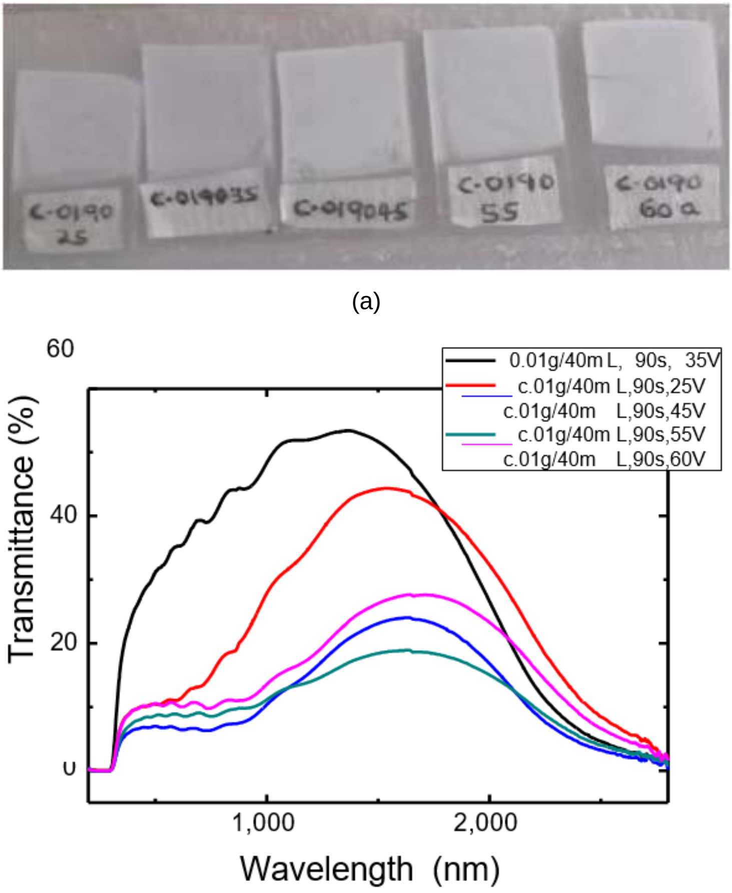
Deposition time of 90 s became the optimum EPD deposition time for fabrication of TiO_2/Nb_2O_5 composite electrode thin films

3.4 Optimization of Applied Voltage

The results for optimization of applied DC voltage correspond to Figs. 6 and 7.

The photographs of electrophoretically deposited TiO_2/Nb_2O_5 composite thin films show that the films were deposited on the glass slides (Fig. 6). The corresponding transmission spectra, for films electrophoretically deposited from 0.01 g/40 mL suspension and 90 s, yielded the 55% transmittance value corresponding to applied DC voltage of 35 V (Fig. 6).

Fig. 7 shows that transmittance decreased with increased applied voltage. Maximum transmittance (55%) was obtained for voltage of 35 V. Higher voltages (< 35 V) produced relatively thick films which yielded low transmittance values. Typically, thick films hinder transmission of light rays through them resulting to low transmittance values. The thick films resulting from deposition at high voltages were non-porous and unsuitable for dye absorption. In addition, thick films decrease the electron diffusion which results in low conversion efficiency. It follows, that optimum applied DC voltage for deposition of TiO_2/Nb_2O_5 composite thin films is 35 V.



(a)

(b)

Fig. 6. (a) Photographs and (b) transmittance spectra for TiO_2/Nb_2O_5 composite electrode thin films electrophoretically deposited for 90 s from 0.01 g/40 mL, and for varying voltages

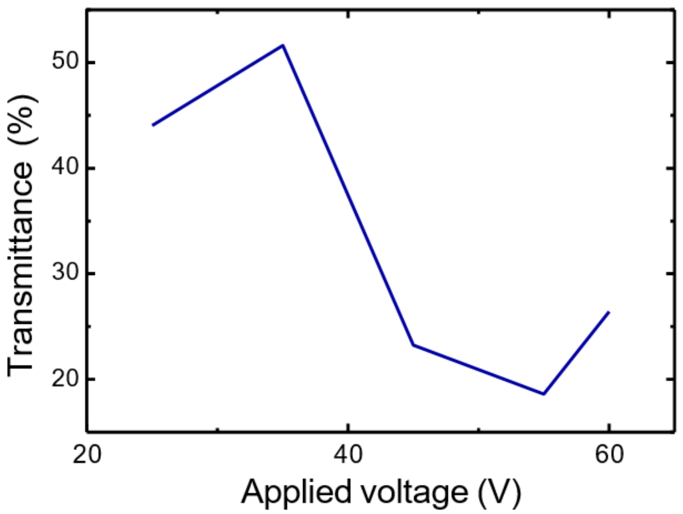


Fig. 7. Variation of Transmittance (at 1,508 nm) with applied voltage for films electrophoretically deposited at 35 V from 0.01 g/40 mL suspension

The optimum EPD parameters are presented in summary in Table 1. Table 2 gives comparison of different EPD process parameter, namely DC applied voltage, deposition times and concentrations. Even for electrophoretic deposition of titanium dioxide alone, a great variation in the EPD process parameters was observed. These results underscore the importance of EPD process optimization especially when coating a composite film of TiO_2/Nb_2O_5 as used in this study

3.5 Morphological and XRD Characterization of Fabricated TiO2/Nb2O5 Composite Thin Films

3.5.1 Morphology of Deposited Films

Figs. 8a and 8b show the SEM (scanning electron microscope) image of the surface morphology of TiO_2/Nb_2O_5 composite thin films electrophoretically deposited using combination 0.01 g/40 mL, 90 s and 35 DCV and at magnification of 100 K and 50 K, respectively. SEM used had *EHT* = 10.00 KV.

The SEM images of deposited TiO_2/Nb_2O_5 composite thin films, reveal that TiO_2 and Nb_2O_5 nanoparticles in the composite films were of near uniform distribution with a small fraction of uncoated glass surface. The more connected the composite particles are to each other, the more the electron transport to the conductive substrate. The surface coating appears to adhere well to the glass surface. This can be attributed to magnesium ions that attached to the surface of metal oxide particles. In addition, these films have the desired porosity for dye absorption and are therefore suitable for application in the DSSC.

Table 1. Optimized electrophoretic deposition parameters

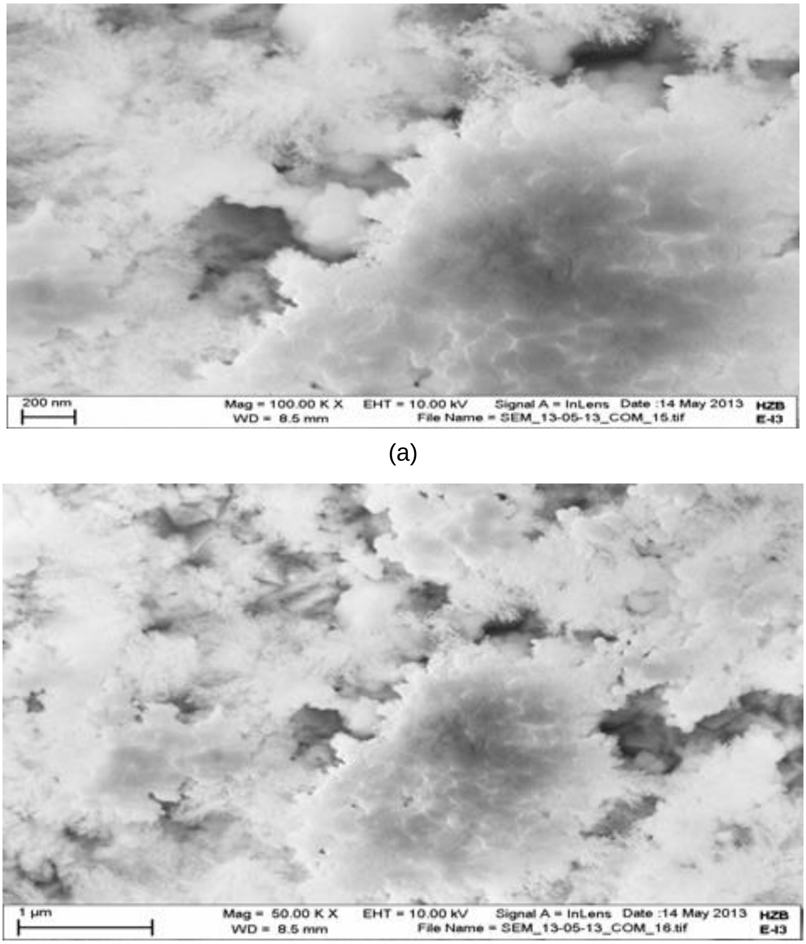
Concentration of TiO2/Nb2O5 particles	Applied DC voltage across electrodes	Distance between electrodes	Deposition time
0.01 g/40 mL (= 0.25 g/L)	35 V	12 mm	90 sec

Table 2. Variety of EPD optimum process parameters

Applied voltage (V)	Electrode separation	Concentration	Deposition time	Photoelectrode	Reference
75	2 cm	0.25 g/L	180 s	TiO_2 on flexible DSSC	[19]
40	2 cm	0.25 g/L	15 min	TiO_2 on FTO glass	[7]
5-20	1 cm	0.5 g/50 mL	1-8 min	TiO_2 on FTO glass	[23]
6	0.3 mm	-	0.5-1 min	TiO_2 on conductive glass	[16]
40	18 mm	2.4 g/L	2 min	TiO_2 FTO glass compressed	[24]
55	1 cm	0.6/100 mL	4 min	CNT (carbon nanotube)/TiO_2 composites	[25]

3.5.2 X-RD (X-Ray Diffraction) Characterization

Fig. 9 show the X-RD graphs for composite TiO_2/Nb_2O_5 electrophoretically deposited at 35 DCV, 0.01 g/40 mL, and 90 s. Dominant peak of TiO_2 was at 2-theta of 25.5 deg while that of Nb2O5 was at 26.5 deg. However, the peak (counts = 1,600) of TiO2 was longer than that of Nb2O5 (900 counts) which translates to ratio 1.78:1. The length of peaks corresponds to degree of crystallinity [26]. It follows, therefore, that the crystallinity of TiO_2 is higher than that of Nb_2O_5.



(a)

(b)

Fig. 8. SEM image of TiO_2/Nb_2O_5 composite thin film on a F: SnO_2 (a) magnification = 100 K and (b) magnification = 50 K.

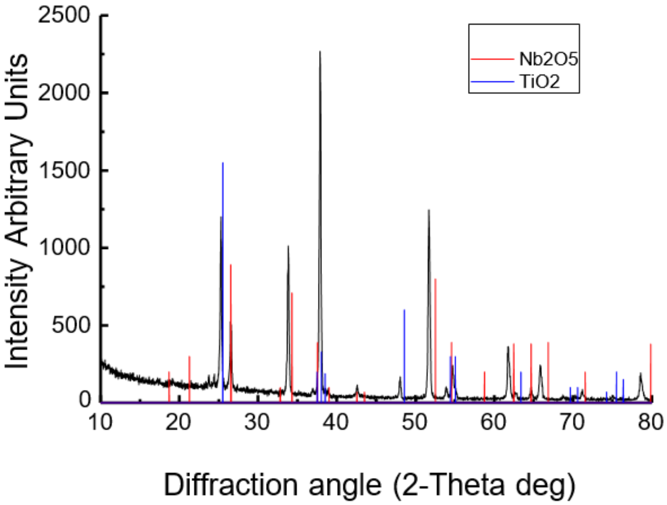


Fig. 9. XRD spectra of TiO_2/Nb_2O_5 composite thin films annealed at 400 °C for 30 min under atmospheric conditions

4. CONCLUSIONS

EPD process parameters which include concentration, applied voltage and deposition time, were successfully optimized in this study. Values of 0.01 g/40 mL, 35 DCV and 90 s, respectively were obtained as optimum values for fabrication of TiO_2/Nb_2O_5 composite electrode thin films. This combination of deposition time (90 s), DCV voltages (35 V) and solid concentration (0.01 g/40 mL) resulted in crack free and porous thin films. Moreover, a pH of ≈ 4.9 was found to be optimal for propan-2-ol suspension used in EPD method. Both TiO_2 and Nb_2O_5 nanoparticles were shown, by XRD graphs, to be presented in the composite films in the ratio 1.78:1 (or 1:0.56). The EPD technique after optimization, has been used in the study to fabricate good-quality TiO_2/Nb_2O_5 composite thin films, and of smooth morphology as shown by SEM images. Further work is needed to evaluate the potential of TiO_2/Nb_2O_5 composite films deposited by EPD for solar cell applications.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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