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Effect of Alkaline Nitrates and Operating Temperature on the Performance of Dye Sensitized Solar Cells

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Abstract

This work sought to investigate experimentally the applicability of an alkaline nitrate (NaNO3 and KNO3)-coated TiO2 working electrode in a dye-sensitize solar cell (DSSC) in enhancing the cell's performance. A simple dipping method was used to modify the surfaces of TiO2 films with nitrate aqueous solutions with concentrations of 0.01M, 0.05M and 0.1M. Using standard SEM, XRD and UV-Vis apparatus, the modified cell performance was compared with an equivalent bare-TiO2 surface in terms of performance at two temperature levels. It was found that with higher alkaline concentrations, the electrode surface was covered with smaller, denser and nicely oriented nitrate nanoparticles, as well as superior optical performance and a quite successful incorporation of the alkaline nitrates with TiO2 paste. The modified DSSC consistently showed higher power conversion efficiency. Specifically, findings indicate that the power conversion efficiency using NaNO3–TiO2/ITO layer at all concentrations and temperature levels considered. The highest power conversion efficiency was recorded in the DSSC with NaNO3 (0.1)–TiO2/ITO layer at 1.029% and 0.84% at 13 oC and 40 oC, respectively. Moreover, the findings reaffirmed the positive effect of lower temperature on cell performance.

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Keywords: Dye-sensitized solar cells, Cell performance, Cell temperature, Electrode modification, Alkaline nitrates, TiO2 nanoparticles;

1. Introduction

It is almost universally accepted that fossil fuels are not a sustainable energy source as they were formed by the decomposition of dead plants and animals under extreme heat and pressure over millions of years. In contrast, renewable energy sources provide a promising energy source since they are naturally replenished. As per the World Energy Council for 2013, renewable sources made up about 11% of the world's total primary energy supply in 2011, and are projected to reach about 16% in 2020 [1].

Among all renewable sources, solar energy offers the most potential and promise [2-4]. In the long term (up to 2050) and due to its distinct features, solar energy is expected to play a prominent role as an energy source and in climate change mitigation. In addition, conversion of solar energy to electricity is one of the most desired and convenient conversion technologies and may be achieved in two ways: PV solar cells and concentrating solar power [5]. PV cells enjoy the advantage of directly converting solar radiation to electricity.

PV solar cells are classified into three generations based on their performance and cost effectiveness. Crystalline silicon cells (1st Generation), also called traditional, conventional o r wafer-based cells, are made of crystalline silicon [6]. Thin-film cells (2nd Generation) are made of thin layers of photosensitive materials (thickness < micrometer) on a lowcost substrate such as stainless steel, glass or a polymer foil [7]. Dye-sensitized cells (3rd Generation), widely known as DSSCs, are one of a third generation PV and are based on semiconductor structures.

Dye-sensitized solar cells represent one of the best alternatives and strongest substitutes for silicon or thin-film solar cells due to the low material cost and simplicity of fabrication [8]. Although they are still in the precommercial stage, commercial efficiencies have reached 4% to 5% and laboratory efficiencies of 12% [9]. Since their invention and development by Michael Grätzel and Brian O' Regan in 1991, DSSCs have attracted serious interest and gained widespread acceptance as a promising technology of renewable energy since they have an apparent edge over their counterparts [10, 11]. However, although the scientific community has succeeded for the past 20 years in improving DSSCs, the gains in efficiencies have not been staggering [12, 13, 14]. Recently, research efforts focus on enhancing power conversion efficiencies and reduce the gap between today's benchmark and Shockley-Queisser limit [15, 16]. DSSCs based on TiO2 as photo anodes and doped photo anodes have been also studied [17]. Studies discussed how TiO₂ comparatively stands as the best with alternative

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oxides by virtue of its unique properties and the analysis of various factors affecting the performance of TiO2-based DSSCs. Moreover, the necessity and impact of doping in TiO₂ toward the improvement in the performance of DSSC was explored. In a systematical study, Chu et al. [18] investigated the nano-scale structural changes of the TiO2 DSSC electrode due to the alkaline and thermal treatments. Photovoltaic performance was measured and revealed the effect of microstructural changes. Such electrode coatings can be considered as potential candidates for the electrode material in a DSSC. Experimental results revealed that TiO2 layer with a 3D network 'nano-flaky' was prepared with sizes of around 100 to 200 nm across and thickness of less than 10 nm that cover the outermost surface, as well as the inner pores and voids. This scheme provides a larger surface area for dye absorption and thus increases the efficiency of assembled DSSC.

The basic concept of DSSCs is simple, and it, basically, imitates plant photosynthesis process. Figure 1 shows the structure and general mechanism of energy conversion in a typical DSSC [19, 20].

In this work, the main objective was to experimentally investigate the impact of introducing modifications to the photoelectrodes on the performance of a locally fabricated and assembled DSSC by attempting to reduce charge recombination and alleviate the negative effect of cell operating temperature [21, 22]. The modifications involved mainly applying different concentrations of dilute nanoscale aqueous solutions of two alkaline nitrates that were separately applied to the bare- TiO_2 photoelectrodes via a soaking process [23]. The cell performance was evaluated mainly by its voltage-current (V-J) characteristics at two temperature levels. Standard tests using UV-Vis, SEM, and XRD were conducted to evaluate relevant properties of the photoelectrodes and the size and morphology of the nanocrystals.

2. Materials and Methods

This work involved three basic parts, namely, modifying the cell's photoelectrodes which entailed preparing and using aqueous solutions with various concentrations, namely, 0.01 M, 0.05 M, and 0.10 M, of sodium nitrate (NaNO₃) and potassium nitrate (KNO₃) nanoparticles via a soaking process; fabricating and assembling a DSSC in the lab as per the modifications indicated above; and finally, evaluating the fabricated cell performance.

2.1. Preparation of solutions

2.1.1. NaNO3 and KNO3 aqueous solutions

Particles of NaNO₃ and KNO₃ were manually ground by mortar to get their respective nanoparticles which were later characterized by scanning electron microscope (SEM). Then, 50 mL of distilled water were added to the resulting NaNO₃ or KNO₃ nanoparticles to produce different concentrations of aqueous solutions. Table 1 shows the different amounts of materials used to produce desired concentrations of both aqueous solutions. The solutions of KNO₃ and NaNO₃ were stirred in a hot plate for 15 min at 60 °C, followed by placing them in an ultrasonic bath for 15 min.

2.1.2. TiCl₄ aqueous solution

To obtain 40 mM TiCl₄ solution, a volume of 0.45 ml of TiCl₄ was added to 99.55 ml of ice cold deionized water in a fume hood and then stirring the mixture for 30 min. A general procedure in the assembly of DSSCs is to deposit a thin film of TiO₂ by submerging the substrate in the 40 mM aqueous TiCl₄ solution at 70 °C for a period of time. The TiCl₄ is then used to produce the pigment titanium dioxide (TiO₂) according to the following equation:

$$TiCl_4 + 2 H_2O \rightarrow TiO_2 + 4 HCl$$
(1)

2.1.3. The dye solution

The N749 black dye was employed as the sensitizer. The dye solution (0.3 mM) was prepared by adding 0.0205 g of the dye in 50 ml of ethanol which was used as the solvent. The solution was stirred for 30 min and stored in a sealed container for 24 hours before use. The dye solution was stored away from light.



Figure 1: Structure and working principle of TiO₂- based DSSCs.

2.2. Fabrication and assembly of the DSSC

2.2.1. Materials used in preparing the solar cells

The following materials were used: Indium tin oxide (ITO, In₂O₃: Sn) coated glass electrodes (2.5 cm×1.8 cm, 2 slides per cell); Anatase TiO₂ nanoparticle colloid; Black dye N749; Platinum precursor solution; A redox (I⁻/I₃⁻) electrolyte solution composed of 0.5M lithium iodide and 0.05M iodine in acetonitrile with γ -Butyrolactone as a solvent to improve volatility; Binder clips (small, 2 pieces for 1 cell); Ethanol; Deionized water, Distilled water; and Detergent solution.

2.2.2. Fabrication and assembly of the cell

Indium-doped tin oxide (ITO) conductive glass substrate was cut into pieces of dimensions 1.8cm×2.5cm. ITO glasses were cleaned by a detergent solution for 10 min, water for 10 min and ethanol for 10 min using an ultrasonic bath, and then dried in a furnace at 70 °C for 20 min. The ITO glasses were immersed in a 40 mM TiCl₂ solution at 70 °C for 30 min.

A TiO₂ paste was prepared by adding 3.5g of TiO₂ nanopowder to 15 ml of ethanol, followed by adding a Polyvinylpyrrolidone binder (PVP) to the nano-powder in a weight ratio of 10:1. The TiO₂ mixture was stirred on a hot plate until the paste became uniform. The paste was finally subject to sonication for 15 min. An active film of TiO₂ paste was formed on the ITO glass via a doctor-blade method.

The TiO₂ paste films were gradually heated by air at 60 °C for 5min, 80 °C for 5 min and then 100 °C for 5 min using a hair dryer to drive off water and carbon dioxide. Again, the TiO₂ films were treated with 40 mM TiCl₄ solution for 1 min., annealed at 500 °C for 40 min and then slowly cooled in a furnace to develop equilibrium structure.

The NaNO₃–TiO₂ /ITO and KNO₃–TiO₂ /ITO electrodes were fabricated via a dipping process in which TiO₂/ITO electrodes were separately soaked into the aqueous solutions of NaNO₃ and KNO₃ at concentrations of 0.01 M, 0.05 M and 0.1 M for 30 sec, followed by rinsing with deionized water. Then, seven electrodes were dried at 120 °C for 30 min in a furnace. Upon cooling them down to 80 °C, the bare-TiO₂ /ITO electrode, the NaNO₃–TiO₂/ITO electrodes and the KNO3–TiO₂/ITO electrodes were separately immersed into a 0.3 mM of N749 black dye solution for 24 h.

The counter electrodes were prepared by placing a drop of 3 mM H₂PtCl₆ solution on ITO glass, then drying it at 150 °C for 30 min. The working electrodes and platinumcounter electrodes were assembled into a sandwich type cell and sealed with binder clips. Then, a drop of electrolyte was placed between the working electrode and the platinumcounter electrode. To improve conductivity, copper paste was added to the edges of the cell.

2.3. Evaluation of cell performance

Various techniques were used to examine the cell performance after introducing the modifications indicated in this study. Such techniques included scanning electron microscopy (SEM), X-ray diffraction (XRD), ultraviolet–visible spectroscopy (UV–Vis). The cell's current-voltage data was gathered by a Keithley 2425 source meter along with the Lab-View software as shown in Figure 2.

PV tests of surface-modified (NaNO₃–TiO₂/ITO and KNO₃–TiO₂/ITO) and non-modified (bare-TiO₂/ITO) photoelectrodes were performed by experimentally producing the electric current density vs. voltage, J vs. V, curves using a LED array of 10 mW/m² at two different temperatures, namely, 40 °C and 13 °C. The area of the modified surface was 0.0001 (1×10⁻⁴) m². The cell performance was evaluated by measuring the short circuit current density (J_{sc}), open circuit voltage (V_{oc}), fill factor (FF) and energy conversion efficiency (η). The values of J_{sc} and V_{oc} can be obtained directly using the current-voltage, I-V, data. The FF and η were calculated, respectively, from the following two equations [11] (Okoli, 2010; Lellig, 2012):

$$FF = \frac{Im \times Vm}{Isc \times Voc} = \frac{Pm}{Isc \times Voc}$$
(2)

$$\eta = \frac{Po}{Pi} = \frac{FF \times Voc \times Isc}{Pi}$$
(3)

where, Po= the electrical power output

Pi= the energy input from the sun

 $m=\mbox{the subscript}$ "m" stands for the maximum power point



Figure 2. Keithley 2425 instrument, Lab-View program and light simulator used for the measurement of the photocurrent-photovoltage characteristics.

The structure and morphological characterization of the DSSC were studied by scanning electron microscope (SEM: Quanta FEI 450 SEM machine). The intention was to get a high-resolution surface image and thus have the shape and size of clusters from which the diameter of the TiO2 nanoparticles can be determined. As for the crystalline structure of the photoelectrode, an X-ray diffraction technique (XRD-6000, Shimadzu) using Ni-filtered Cu Ka line ($\lambda = 0.15418$ nm) at a scanning rate of 2° min⁻¹ in 20 ranging from 0° to 80° was utilized. Bare-TiO₂/ITO films were dipped into KNO₃ or NaNO₃ aqueous solution and during the soaking process, the nitrate ions were adsorbed onto the surface of TiO₂. NaNO₃ or KNO₃ phase was verified by the XRD.

2.4. Effect of temperature on cell performance

Experiments were conducted at two temperature levels, namely, 40 $^{\circ}$ C and 13 $^{\circ}$ C. The intention was to examine and validate the effect of temperature on cell performance.

3. Results and Discussion

3.1. The fabricated/assembled cell

Figure 3 depicts the cell that was fabricated and assembled in this study. The same cell was also used in all measurements made in this work. DSSC consists of a transparent conducting glass electrode with a porous TiO₂ layer coated with an organic dye (in our case) that serves as light sensitizer, an electrolyte layer, and a counter electrode, typically coated with graphite. The electrodes were fixed using two clips and sealed firmly in order to prevent leakage of the dye and electrolyte. In DSSCs, dyes play an important role in solar energy harvesting. Therefore, cell performance is mainly dependent on the type of dyes used as a sensitizer.

The following notes should be considered during DSSC fabrication:

• Temperature stability given that at low temperatures the electrolyte can freeze, preventing power production and potentially leading to fluctuation in cell conversion efficiency due to mechanical defects in the microstructure.

• Porous microstructures of the TiO₂ layers with high surface area are essential for achieving high efficiency in the DSSC system because a large amount of dye can be adsorbed on the surface of the nc-TiO₂ particles resulting in an increased solar light absorption and an increased reacting interface per unit area.

3.2. Photoelectrode morphology (SEM results)

The SEM image of the bare-TiO₂/ITO in Figure 4 shows that the particles are of irregular shape and of non-uniform size. Figure 4 also shows that the particles have highly mesoporous foam-like structure and agglomerated to nearly spherical morphology which may be due to annealing of TiO₂ powder. Also, the same figure shows that there are some pores and voids between the grains, and the grain boundaries are randomly oriented. Similarly, SEM images were obtained for the 0.01 and 0.05 M concentrations for both nitrates and demonstrated particles with better features.



Figure 4. Scanning electron microscopy image of bare-TiO₂/ITO surface.

In contrast, Figure 5 presents the SEM image for samples with the highest concentration considered in this study, i.e., 0.10 M. As seen from the microstructure, the surface of the electrode is covered with NaNO₃ or KNO₃ nanoparticles with an average size of approximately 1.0 nm, which is smaller than that for the samples modified with lower concentrations. Figure 5 further exhibits a significantly denser microstructure with nicely oriented grain boundaries.



Liquid Electrolyte Solution

Figure 3: The fabricated/assembled cells used in this study.

In contrast, Figure 5 presents the SEM image for samples with the highest concentration considered in this study, i.e., 0.10 M. As seen from the microstructure, the surface of the electrode is covered with NaNO₃ or KNO₃ nanoparticles with an average size of approximately 1.0 nm, which is smaller than that for the samples modified with lower concentrations. Figure 5 further exhibits a significantly denser microstructure with nicely oriented grain boundaries.

3.3. Optical performance of the photoelectrodes

Figure 6 shows the UV-Vis transmission spectrum of absorption spectra of both non-modified (bare) and modified samples using both nitrates at the three concentrations. Figure 6 indicates that in the short wavelength region (<300 nm), both the bare-TiO₂/ITO substrate and the alkaline nitrates-TiO₂/ITO substrates show essentially the same behavior. Beyond that, the figure shows a substantial decrease in light absorption. Moreover, Figure 6 shows that the optical absorption is slightly reduced with increasing solution concentration and further that NaNO₃ samples are less absorbent than their KNO₃ counterparts at the same concentration. This observation indicates the excellent optical absorption of alkaline nitrates-TiO₂ /ITO film.

In a similar fashion, Figure 7 portrays the UV-Vis transmittance spectra of the tested samples and indicates that samples with higher concentration of an aqueous solution show lower transmittance in the long wavelength region (λ >400 nm) while for λ <400, samples show the same behavior, the penetrated light decreases seriously.



Figure 5: SEM images of NaNO₃-TiO₂/ITO surface (left), and KNO₃-TiO₂/ITO surface (right) at 0.10M concentration.



Figure 6. Absorption spectra of the photoelectrode samples.

3.4. XRD results

Figure 8 shows the XRD patterns of the photoelectrode samples. Crystallographic planes of TiO₂ anatase ((101), (004), (200), (105), (211), (204), (116), (220), (215)) could be observed at 20 of 25.2°, 37.7°, 48.1°, 53.9°, 55°, 62.5°, 68.5°, 70.4°, and 74.7°, respectively. In contrast, NaNO₃ phase and KNO₃ phase start to appear at 20 of 31.82 (006) peak and 27.33 (012) peak, respectively, indicating that the alkaline nitrates were successfully incorporated with TiO₂ paste.

3.5. The cell's J-V characteristics

Table 1 summarizes the cell's PV parameters at 40 °C and 13 °C including short-circuit current density (J_{sc}), opencircuit voltage (V_{oc}), fill factor (FF) and photoelectric conversion efficiency (η). In addition, Figures 9 and 10 display the J-V characteristics of the cell tested in this study for both the surface-modified and non-modified (bare- bare-TiO₂/ITO) photoelectrodes at 40 °C and 13 °C, respectively.



Figure 7. Transmittance spectra of the photoelectrode samples



2 Theta (degree) Figure 8. XRD patterns of TiO₂ - NaNO₃ and KNO₃ nanocomposites.

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Surface photoelectrode		J_{sc} (mA/cm ²)	$V_{oc}(V)$	$J_m (mA/cm^2)$	$V_m(V)$	Fill Factor(%)	η (%)
bare-TiO ₂ /ITO at 40 °C (13 °C)		0.709 (0.793)	0.422 (0.429)	0.388 (0.438)	0.211 (0.211)	27.4 (0.271)	0.082 (0.092)
Modified	TiO ₂ /ITO Concentration (M)	0.957	0.473	0.479	0.263	27.8	0.126
KNO ₃ at 40 °C (13 °C)	0.01	(0.996)	(0.560)	(0.505)	(0.316)	(0.286)	(0.159)
	0.05	1.297 (1.016)	0.515 (0.760)	0.725 (0.518)	0.263 (0.369)	28.6 (0.247)	0.191 (0.191)
	0.10	2.643 (2.942)	0.695 (0.784)	1.520 (1.674)	0.368 (0.421)	30.5 (0.306)	0.560 (0.705)
NaNO ₃ at 40 °C (13 °C)	0.01	1.154 (1.171)	0.505 (0.490)	0.635 (0.632)	0.263 (0.263)	28.7 (0.290)	0.167 (0.167)
	0.05	2.332 (2.420)	0.565 (0.718)	1.207 (1.256)	0.316 (0.421)	28.9 (0.304)	0.381 (0.529)
	0.10	3.507 (4.013)	0.777 (0.830)	1.994 (2.173)	0.421 (0.474)	30.8 (0.309)	0.840 (1.029)

Table 1: Cell's photoelectric parameters for the surface-modified and non-modified (bare-TiO2/ITO) photoelectrodes at 40 °C and 13 °C.



 $\label{eq:Figure 9. Histograms of distribution of current density J(mA/cm^2) and photo-conversion efficiency $$\eta$ (%) (a) KNO3 at 40 °C (b) KNO3 at 13 °C (c) NaNO3 at 40 °C (d) NaNO3 at 13 °C.$

Figure 9 presents variations of current density, J, and efficiency, η , of DSSCs with various KNO₃ and NaNO₃ contents at different operating temperatures (40 °C and 13 °C). Results revealed that for all tested DSSCs samples, the current densities and the photo-conversion efficiency increases gradually with increasing the alkaline content. However, for DSSCs with 0.1 KNO₃ at 13 °C (Figure 9 – b), a drastic increase (about three times) in both current density and efficiency was observed. This may suggest that further increase in the alkaline content may give rise to the main DSSCs parameters and thus enhances the cell performance. Almost similar behavior was also observed at T = 40 °C for the same DSSC.

Figure 10 (a, b) shows that the modified DSSC exhibited higher power conversion efficiency (PCE) than that with bare-TiO₂/ITO. For example, the same figure indicates that the PCE of the DSSC with NaNO₃ (0.01)–TiO₂/ITO and with KNO₃ (0.01)–TiO₂/ITO increased by, respectively, 200% and 150% relative to a cell with a bare-TiO₂/ITO.

Moreover, Figure 10 (a, b) demonstrates consistent increase in PCE with concentration of aqueous solutions. It may be readily noted form the same figure that the highest PCE was for the DSSC with NaNO₃ (0.1M)–TiO₂/TTO and reached 0.840. As evident from Figure 11 (a, b), similar trends were obtained for the tests at 13 °C. This enhancement may be ascribed to the addition of NaNO₃ or KNO₃, which greatly reduced charge recombination between the dye and TiO₂ film, as well as between the electrolyte and TiO₂ film. Further, Figure 11 (c) reveals that the PCE of the surface modified with NaNO₃–TiO₂/ITO) was higher than that for the surface modified with KNO₃–TiO₂/ITO at any given concentration or operating temperature within the ranges considered here.

As far as the influence of operating temperature on cell performance, Figures 10 and 11, clearly and consistently demonstrate the positive influence of lower operating temperature on the cell performance.



Figure 10: The cell's J-V curves at 40 °C with different photoelectrodes (a) KNO₃-modified (b) NaNO₃-modified, and (c) Comparison between NaNO₃- and KNO₃-modified electrodes.







Figure 11: The cell's J-V curves at 13 °C with different photoelectrodes (a) KNO₃-modified (b) NaNO₃-modified, and (c) comparison between NaNO₃- and KNO₃-modified electrodes.

4. Conclusion

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Based on the findings obtained in this work, it may be concluded that DSSCs, unlike existing solar cells, enjoy the attractive feature of easy, low-cost manufacturing under simple low-tech setups and limited resources. In addition, the results of this study indicate that coating the working electrodes of a typical DSSC with alkaline nitrates nanoparticle solutions have the potential to substantially improve the cell performance in terms of its power conversion efficiency and other characteristics. Moreover, DSSCs perform better at lower temperatures as is the case for their traditional PV counterparts.

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