



Ain Shams University
Faculty of Science
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Preparation of mixed oxides nanotube arrays and its applications

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B.Sc. in Chemistry, Faculty of Science
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List of Abbreviations

PEC	Photoelectrochemical cells
Eg	Band gap
UV	Ultraviolet
DSSCs	Dye-sensitized solar cells
TNTs	TiO₂ nanotube arrays
FE-SEM	Field emission scanning electron microscope
XRD	X-ray diffraction
EDX	Energy dispersive X- ray spectroscopy
DRS	Diffuse reflectance spectroscopy
SEM	Scanning electron microscope
WDXRF	Wavelength Dispersive X-ray Fluorescence
UV-Vis	Ultraviolet–visible spectroscopy
E.G.	Ethylene glycol

Summary

Due to the urgently need for energy and as result of the continuous increase of the population, the scientists turned to find a new constant source of energy. Many researches have been held to achieve this goal. Among these was the research of hydrogen production using titanium dioxide nanotubes. Therefore, in this work we tried to improve the efficiency of TiO_2 nanotube arrays by means of electrodeposition technique. We have studied the effect of the solvent through preparation of different samples of the TiO_2 nanotube arrays by execute the samples using the same conditions with varying the solvent type. The TNTs have been prepared using two solvents: glycerol and ethylene glycol. Then, a modification has been proceeded using electrodeposition process in organic medium. Iron oxide has been deposited on the surface of TNTs samples using different concentration baths at different times of deposition.

The samples were characterized by field emission scanning electron microscope (FE – SEM), energy dispersive X-ray spectroscopy (EDX), X-ray diffractometer (XRD) and UV–Vis diffuse reflection spectroscopy (DRS). Evaluation of all samples was done using a solar simulator.

FE-SEM images showed that iron oxide was successfully deposited on the surface and in-between the TiO_2 nanotube arrays. XRD analysis reported that the phase of TiO_2 nanotubes which was prepared either using ethylene glycol or glycerol was anatase phase.

The photocurrent was measured and the photoconversion efficiency was calculated. The measurements confirmed that iron oxide was successfully deposited on the surface of TiO_2 nanotubes and in-between the TiO_2 nanotube arrays. The photoelectrochemical measurements showed a significant enhancement of the photocurrent of the mixed oxide samples compared with TiO_2 nanotubes seldom in both solvents (glycerol and ethylene glycol) used in anodization of electrodes. The photocurrent showed a significant enhancement after 30 minutes of depositing iron oxide from 0.05 M $\text{Fe}(\text{NO}_3)_3$ bath in case of TNTs prepared by glycerol resulting a photoconversion efficiency of 0.55% compared with unmodified TNTs which was 0.28 %. Also, a significant enhancement after 30 minutes of depositing iron oxide from 0.07 M $\text{Fe}(\text{NO}_3)_3$ bath in case of TNTs prepared by ethylene glycol resulting a photoconversion efficiency of 0.43 % compared with unmodified TNTs which was 0.22 %.

The hydrogen production was measured and the maximum hydrogen production achieved was $15.87 \mu\text{mol}/(\text{cm}^2.\text{h})$ in case of TNTs prepared by glycerol and $12.52 \mu\text{mol}/(\text{cm}^2.\text{h})$ in case of TNTs prepared by ethylene glycol compared with the unmodified TiO_2 nanotube arrays which achieved hydrogen production of $4.17 \mu\text{mol}/(\text{cm}^2.\text{h})$ in case of TNTs prepared by glycerol and $5.40 \mu\text{mol}/(\text{cm}^2.\text{h})$ in case of TNTs prepared by ethylene glycol.

After evaluation of the samples which were prepared using ethylene glycol and which were prepared using glycerol and comparing between them, the TNTs electrode which was prepared by glycerol was chosen to make another modification.

The same work was performed by preparation of another modified TNTs using molybdenum. The modification was proceeded by electrodeposition of molybdenum oxide on the surface of TNTs prepared by glycerol by the same previous method with different bath concentrations. The samples were characterized by the same techniques used before in characterizing iron modified TNTs. Evaluation of the samples was measured using a solar simulator. The photoelectrochemical measurements showed a significant enhancement of the photocurrent of the mixed oxide samples

compared with TiO_2 nanotubes seldom. The photocurrent showed a significant enhancement after 30 minutes of depositing molybdenum oxide from 0.05 M sodium molybdate bath. The photoconversion efficiency estimated is 0.44%. The hydrogen production was measured and the maximum hydrogen production achieved is $11.67 \mu\text{mol}/(\text{cm}^2\cdot\text{h})$ compared with the unmodified TiO_2 nanotube arrays which was $4.17 \mu\text{mol}/(\text{cm}^2\cdot\text{h})$.

Aim of Work

Our goal will not only be to achieve excellence in the academic world but also to provide some insights towards the solutions to technological applications in the real world. We will extend the synthesis and optimization of uniquely advantageous one-dimensional (1D) nanoarchitectures of various metal oxides and employ them to various solar energy conversion devices. The proposed work will include materials synthesis optimization while monitoring the dynamics of charge carriers.

This aim will be achieved by preparation of TiO_2 nanotube arrays electrodes using different solvents. Then, electrodeposition of various metal oxide on the surface of TNTs will be proceeded. In order to optimize the optical properties and photoconversion efficiency of TNTs, evaluation of the prepared electrodes will be done by measuring the photocatalytic efficiencies of them.