IOP Conference Series: Earth and
Environmental Science

PAPER • **OPEN ACCESS**

Hydrothermal synthesis and characterization of TiO_2-silica composites for dye-sensitized solar cell electrode

To cite this article: R. Sule *et al* 2023 *IOP Conf. Ser.: Earth Environ. Sci.* **1197** 012005

Hydrothermal synthesis and characterization of TiO_2-silica composites for dye-sensitized solar cell electrode

R. Sule[1], O.C Makinde[1], M. R. Usikalu[1], O.C Olawole[1]

[1] Department of Physics, Covenant University, Ota, Ogun State, Nigeria

Corresponding email: rasidi.sule@covenantuniversity.edu.ng; rasidi.sule@gmail.com

Abstract. The current world energy crisis and increase in global temperature has further necessitated the need to conduct more research on solar cells. Dye sensitized solar cells (DSSCs) remain one of the cheapest solar cell technologies, but it has inherent challenges. Titanium dioxide (TiO_2) remains one of the suitable electrode materials, but it requires modification to withstand the current solar cells technology problems. In this paper, silica was produced from rice husk and incorporated into TiO_2 to tailor its wide band gap. Hydrothermal method was used to synthesize

TiO_2-silica composites at 150ºC. The energy band gap of virgin TiO_2, TiO_2-4.8 wt% silica and 20 wt% silica was found to be 3.73 eV, 2.91 eV and 2.85 eV respectively. The addition of 4.8 wt% silica resulted in good stability of anatase TiO_2 compared to the 20 wt% silica judging from the absorbance plots.

Keywords: Solar cell; TiO_2; Silica; Synthesis; Optical property

1. Introduction

The demand for sustainable and eco-friendly energy sources have driven the world towards solar energy. Dye sensitized solar cells (DSSC) are photovoltaic devices belonging to thin-film solar cells which consist of electrolyte dye that is in contact with inorganic material [1, 2]. It has a similar power conversion efficiency (PCE) relatively to the traditional silicon solar cells with lower material and production costs [1]. Moreover, Bose et al. [3] reported that the dye sensitized solar cells module offered good performance compared to the Silicon module. Several materials have been investigated for DSSC electrodes [4-7]. Zinc oxide (ZnO) would have been a preferred material for DSSC electrodes due to its high carrier mobility optical band gap and transparent properties. However, its susceptibility to corrosion as well as dye aggregates limit its usage [8]. The use of titanium dioxide (TiO_2) particles for the development of dye-sensitized solar cells (DSSC) has attracted much attention as a viable alternative for solar energy conversion [9]. Titanium dioxide unique properties such as low cost, good chemical stability, non-toxicity, optical band gap and resistance to corrosion made it suitable material for DSSC electrode [5]. However, bandgap of TiO_2 is too large to absorb visible light and about

4% of sunlight under UV range gets absorbed. [1,7]. The photocurrent produced could be enhanced if the bandgap is lowered [10].

On the other hand, the crystalline phase of TiO_2 (anatase, rutile and brookite) has been found to play a major role in photocatalytic activity [11]. However, the potential of a greater conduction band energy and a lower electron-hole pair recombination rate, has made the anatase electrode prominent phase for better conversion efficiency in DSSCs [12]. Because the anatase phase is metastable, its transformation to rutile phase readily occurs during grinding and heating [13]. The thermal stability limitation of anatase phase of TiO_2 could be improved with the successful incorporation of oxides materials [13-14]. Various materials have been incorporated into TiO_2 electrode to improve the performance of DSSCs [15, 5]. Amongst these materials silicon dioxide SiO_2 has been widely used and synthesized from different natural materials such as rice husk, fly ash, bamboo leaves etc (7,5]. The choice of SiO2 could be attributed to its low cost and availability of about 60 %wt SiO_2 content in natural sources [7]. Several synthesis methods have been used to produce both TiO_2 and TiO_2-SiO_2 [13,11, 5,16]. However, little information is available on the hydrothermal synthesis of TiO_2-silica composites that involve the use of TiO_2 powder and silica powder. The available report on TiO_2-silica composites shows that solgel-hydrothermal method is widely used.
In this paper, we report a simple and efficient method of synthesizing TiO_2-silica composites with modified band gap using the hydrothermal method.

2. Method and Procedure

2.1 Synthesis of silica from rice husk

The as-received rice husk was carefully rinsed with distilled water to rid it of dirt and dust before treatment with acid. A 200 ml solution containing 10g of rice husk and 2M of HCl in a capped beaker was stirred with a magnetic stirrer at 80°C for 2 hours and was left overnight to remove metal ions in the rice husk. Then the acid was filtered and washed with distilled water until the solution pH was 7. The filtrate was dried for 24 hours at 110°C in a muffled furnace to ensure all the water molecules evaporated. The black ash was crushed and screened using a standard sieve of 325 mesh size. It was further calcined at 700°C for 2 hours in a muffled furnace to produce whitish silica. The synthesized silica was characterized with X-ray fluorescence analysis.

2.2 Synthesis of TiO_2-silica composite

Titanium dioxide powder was produced by modifying the method described in Rajender et al. [17]. Two compositions of TiO_2-silica composites were produced by adding 4.8 wt% and 20 wt% silica synthesized powder into TiO_2. The powders were dissolved in 15 ml of ethanol and stirred vigorously for one hour using a magnetic stirrer. Then 5 ml of distilled water was added to the solution and stirred for 45 minutes. The slurry was transferred inside the autoclave and heated at 150 °C for 15 h. The material was washed and dried to produce TiO_2-silica composites.

3. Results and Discussion

The chemical composition of synthesized silica from rice husk was determined by X-ray Fluorescence analysis. Table 1 shows that 51.86 wt% SiO_2, 20.29 wt% CaO and 10.44 wt% MgO were the dominant contents with minor contents of 1.27 wt% TiO_2 and 1.28 wt% Fe_2O_3. Previous studies have revealed that addition of CaO and MgO into TiO_2 could improve the performance of DSSCs electrodes [18-19]. The result shows that high purity silica was obtained from the rice husk.

Table 1: Chemical composition of synthesized silica from rice husk

SiO_2	V_2O_5	Cr_2O_3	MnO	Fe_2O_3	Co_3O_4	NiO	CuO	Nb_2O_3	MoO_3
51.855	0.033	0.029	0.039	1.275	0.004	0.002	0.033	0.003	0.001
WO_3	P_2O_5	SO_3	CaO	MgO	K_2O	BaO	Al_2O_3	Ta_2O_5	TiO_2
0.002	0.036	0.113	20.288	10.444	2.329	0.036	5.805	0.006	1.274
ZnO	Ag_2O	LOI	ZrO_2	SnO_2					
0.005	0.006	1.169	0.198	0.014					

The quantitative XRD analysis of TiO_2 revealed that the anatase phase constitutes the larger phase with 65%. Figure 1 shows the UV-Vis diffuse reflectance spectrum of synthesized TiO_2 powder with two absorbance peaks. These peaks are located at the ultraviolent range. The most prominent peak was found at wavelength range (220 nm-268 nm) which corresponds to the anatase of TiO_2 while the other phase at around 350 nm [20-21]. The XRD result corroborated the UV-Vis absorbance spectrum which further justified that the anatase was the major phase of the TiO_2 produced. Figure 2 shows the effect of silica on synthesized TiO_2. It was observed that the stability of TiO_2 was improved with silica addition as only one absorbance peak with seen in contrast with that of pure TiO_2.

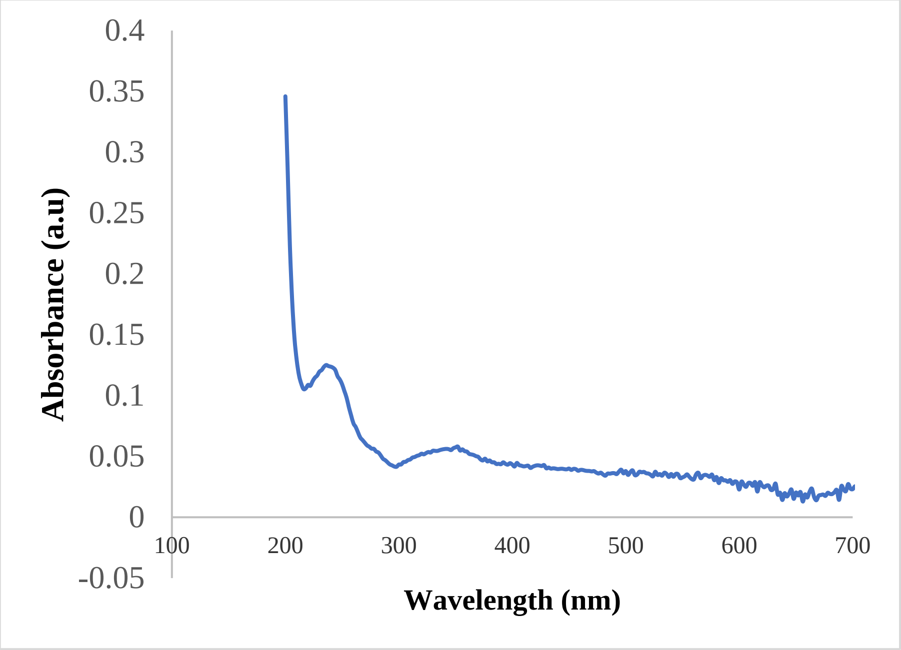

Figure 1: Absorbance curve of synthesized TiO_2

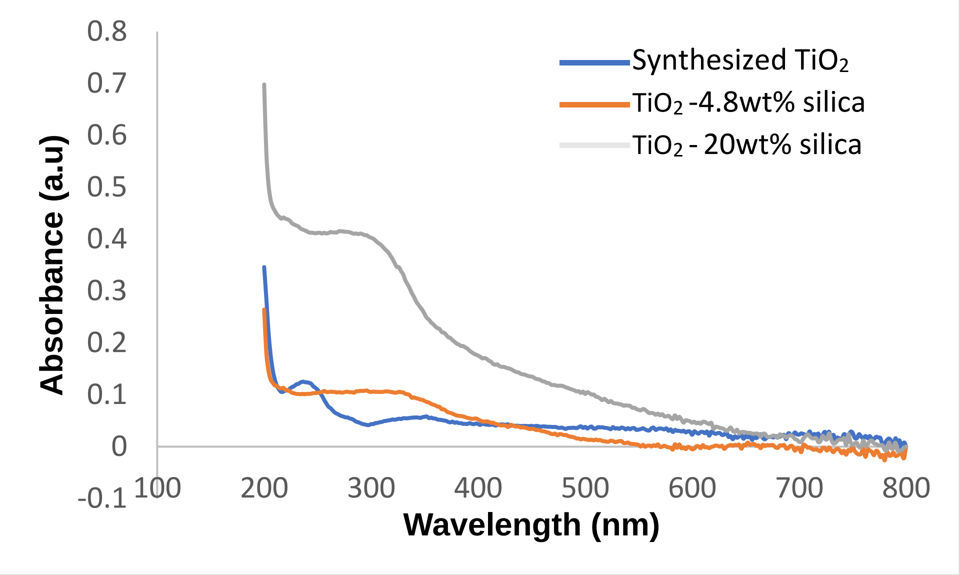

Figure 2: Stack absorbance curve of synthesized TiO_2, TiO_2-4.8 wt% silica composite and TiO_2-20 wt% silica composite.

Tauc's method expressed in Equation 1 [7] have been widely used to determine the band gap energy of semiconductor materials. The optical band gap energy of the synthesized TiO_2 was calculated using Tauc plot.

$$(\alpha h v)^{1/n} = B(hv - E_g) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

Where h is the Planck constant, ν is the frequency of photon photon energy, α is the absorption coefficient, B is a constant value, Eg is the band gap energy, and n is a parameter depends on the nature of the electronic transition in which ½ correspond to direct and 2 indirect transitions band gap.

The extrapolation of the linear fit of the $(\alpha h\nu)^{1/2}$ to energy (hν) axis gives the estimated energy band gap value of synthesized TiO_2 and TiO_2-silica composites. Figure 3 shows the Tauc plot of typical semiconductor characteristics with linear increase of absorbance with respect to photon energy [22]. An estimated optical band gap of 3.73 eV was obtained in synthesized TiO_2. This value is suitable for the solar-cell application since it is within the UV region.

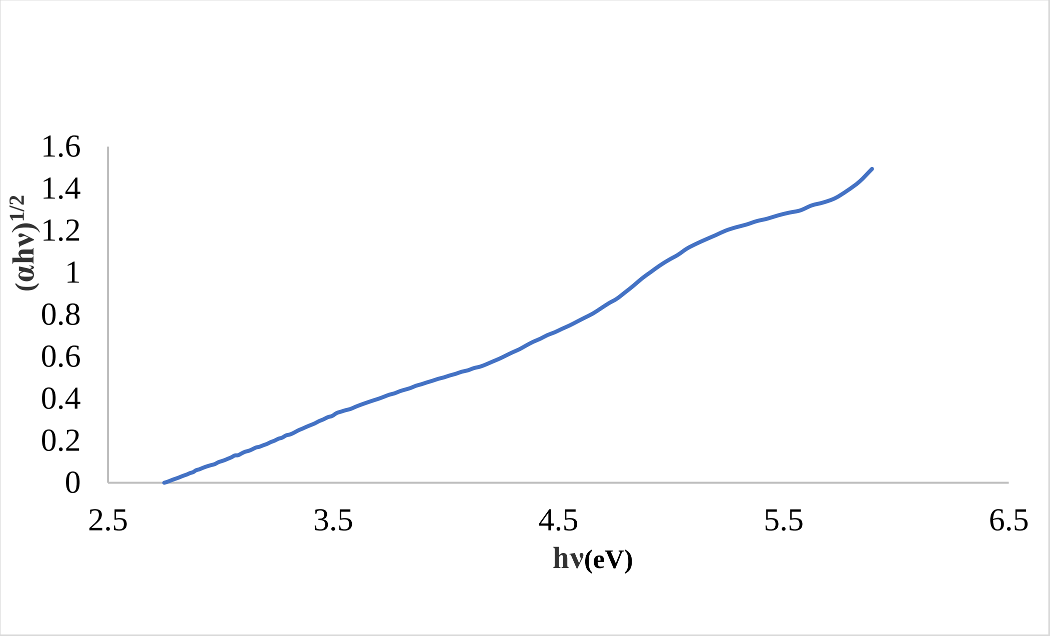

Figure 3: Tauc plot showing the band gap energy of synthesized TiO_2

Furthermore, the effect of silica was examined on the band gap energy and stability of the TiO_2. The calculated band gap energy of virgin TiO_2, TiO_2-4.8 wt% silica and 20 wt% silica was found to be 3.73 eV, 2.91 eV and 2.85 eV respectively. It can be observed that the addition of silica reduced the energy band gap of parent TiO_2 significantly and this can be attributed to the method used to synthesize TiO_2-silica composites. Figure 4 shows the comparison of the Tauc plot of synthesized TiO_2 and TiO_2-silica composites. It can be observed that the addition of 4.8 wt% silica resulted in good thermal stability of anatase TiO_2 compared to the 20 wt% silica although a slight reduction in band gap was noticed in that sample. It is worth mention that the processing method adopted in this study yields a homogenous mixture of TiO_2 and silica that resulted in strong interfacial bonding of Ti-O-Si which prevents the transformation of anatase to rutile phase. Another reason for the result obtained could be the quality of the silica powder produced from the rich husk. This result clearly shows that low content of silica could improve the stability of TiO_2 and as well lower its band gap. The TiO_2-silica composites reported in the research could be used as electrodes in DSSCs with potential to absorb not just sunlight from ultraviolet range but also light from visible region due to the reduction in its band gap.

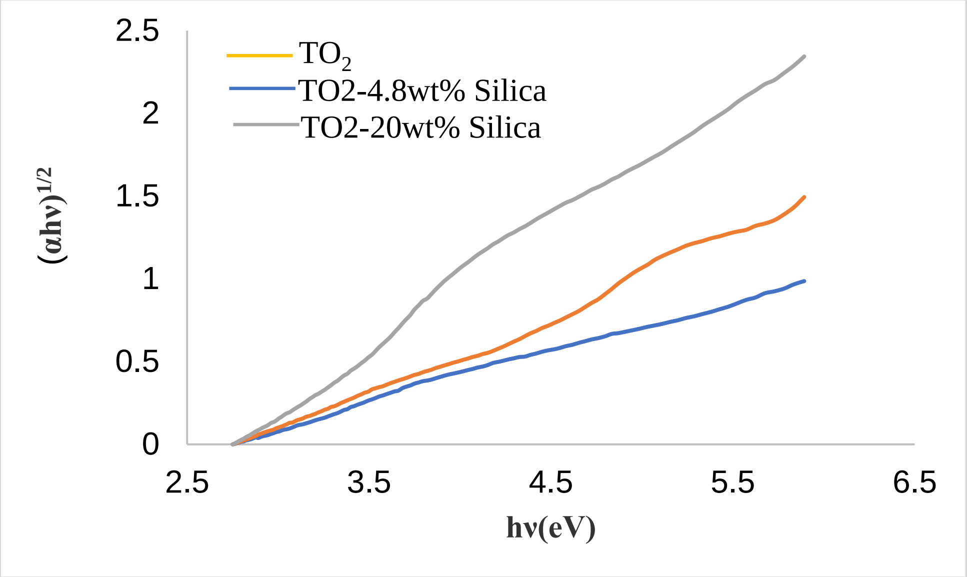

Figure 4: Tauc plot showing the band gap energy of synthesized TiO_2 and TiO_2-silica composites

Conclusion and Recommendation

This article provides insight on hydrothermal synthesis of TiO_2-silica composites with good interfacial bonding without calcined powder after drying. The minor content of silica was found to reduce the band gap of parent TiO_2 from 3.73 eV to 2.85 eV visible range of sunlight for efficient energy conversion. The result revealed that phase transformation of TiO_2 could be inhibited with minor silica content using this approach. However, further research is needed to study the robustness of these materials for DSSCs.

Acknowledgement

The authors would like to thank the Covenant University and Covenant University Centre for Research, Innovation and Discovery (CUCRID) for conference attendance and publication support. The assistance of the Chemical Engineering Department, Covenant University is highly appreciated.

Reference

[1] Müller-Buschbaum, P., Thelakkat, M., Fässler, T. F., & Stutzmann, M. (2017). Hybrid photovoltaics–from Fundamentals towards application. *Advanced energy materials*, *7*(16), 1700248.

[2] Sharma, K., Sharma, V., & Sharma, S. S. (2018). Dye-sensitized solar cells: fundamentals and current status. *Nanoscale research letters*, *13*(1), 1-46.

[3] Bose, S., Soni, V., & Genwa, K. R. (2015). Recent advances and future prospects for dye sensitized solar cells: A review. *International Journal of Scientific and Research Publications*, *5*(4), 1-8.

[4] Suhaimi, S., Shahimin, M. M., Alahmed, Z. A., Chyský, J., & Reshak, A. H. (2015). Materials for enhanced dye-sensitized solar cell performance: Electrochemical application. *Int. J. Electrochem. Sci*, *10*(4), 2859-2871.

[5] Snaith, H. J., & Ducati, C. (2010). SnO2-based dye-sensitized hybrid solar cells exhibiting near unity absorbed photon-to-electron conversion efficiency. *Nano letters*, *10*(4), 1259-1265.

[6] Fatimah, I., Prakoso, N. I., Sahroni, I., Musawwa, M. M., Sim, Y. L., Kooli, F., & Muraza, O. (2019). Physicochemical characteristics and photocatalytic performance of TiO2/SiO2 catalyst synthesized using biogenic silica from bamboo leaves. *Heliyon*, *5*(11), e02766.

[7] Ardani, M., Imani, M., & Tadjarodi, A. (2022). Core shell Fe3O4@ TiO2/silica aerogel nanocomposite; synthesis and study of structural, magnetic and photocatalytic properties. *Microporous and Mesoporous Materials*, *338*, 111757.

[8] Abodunrin, T., Boyo, A., Usikalu, M., Kotsedi, L., Yenus, Z., & Maaza, M. (2017, March). Comparative study of N719 dye on two different photo-anodes. In *Journal of Physics: Conference Series* (Vol. 817, No. 1, p. 012027). IOP Publishing.

[9] Xie, Y., Huang, N., You, S., Liu, Y., Sebo, B., Liang, L., ... & Zhao, X. Z. (2013). Improved performance of dye-sensitized solar cells by trace amount Cr-doped TiO2 photoelectrodes. *Journal of Power Sources*, *224*, 168-173.

[10] Lim, S. P., Pandikumar, A., Lim, H. N., Ramaraj, R., & Huang, N. M. (2015). Boosting photovoltaic performance of dye-sensitized solar cells using silver nanoparticle-decorated N, S-Co-doped-TiO2 photoanode. *Scientific reports*, *5*(1), 1-14.

[11] Muniz, E. C., Góes, M. S., Silva, J. J., Varela, J. A., Joanni, E., Parra, R., & Bueno, P. R. (2011). Synthesis and characterization of mesoporous TiO2 nanostructured films prepared by a modified sol–gel method for application in dye solar cells. *Ceramics International*, *37*(3), 1017-1024.

[12] Cheng, P., Deng, C., Dai, X., Li, B., Liu, D., & Xu, J. (2008). Enhanced energy conversion efficiency of TiO2 electrode modified with WO3 in dye-sensitized solar cells. *Journal of Photochemistry and Photobiology A: Chemistry*, *195*(1), 144-150.

[13] Li, Z., Hou, B., Xu, Y., Wu, D., Sun, Y., Hu, W., & Deng, F. (2005). Comparative study of sol–gel-hydrothermal and sol–gel synthesis of titania–silica composite nanoparticles. *Journal of Solid State Chemistry*, *178*(5), 1395-1405.

[14] Ferreira-Neto, E. P., Worsley, M. A., & Rodrigues-Filho, U. P. (2019). Towards thermally stable aerogel photocatalysts: TiCl4-based sol-gel routes for the design of nanostructured silica-titania aerogel with high photocatalytic activity and outstanding thermal stability. *Journal of Environmental Chemical Engineering*, *7*(5), 103425.

[15] Xie, Y., Huang, N., You, S., Liu, Y., Sebo, B., Liang, L., ... & Zhao, X. Z. (2013). Improved performance of dye-sensitized solar cells by trace amount Cr-doped TiO2 photoelectrodes. *Journal of Power Sources*, *224*, 168-173.

[16] Araoyinbo, A. O., Oyedepo, S. O., & Fajobi, M. A. (2021, April). Titanium Dioxide Nanotube Fabricated at Ambient Temperature via pH5 Acidic Solution. In *IOP Conference Series: Materials Science and Engineering* (Vol. 1107, No. 1, p. 012155). IOP Publishing.

[17] Rajender, G., & Giri, P. K. (2016). Strain induced phase formation, microstructural evolution and bandgap narrowing in strained TiO2 nanocrystals grown by ball milling. *Journal of Alloys and Compounds*, *676*, 591-600.

[18] Kumari, N., Singh, S. K., & Kumar, S. (2022). A comparative study of different materials used for solar photovoltaics technology. *Materials Today: Proceedings*.

[19] Jung, H. S., Lee, J. K., Nastasi, M., Lee, S. W., Kim, J. Y., Park, J. S., ... & Shin, H. (2005). Preparation of nanoporous MgO-coated TiO2 nanoparticles and their

application to the electrode of dye-sensitized solar cells. *Langmuir*, *21*(23), 10332-10335.

[20] Hong, S. M., Lee, S., Jung, H. J., Yu, Y., Shin, J. H., Kwon, K. Y., & Choi, M. Y. (2013). Simple preparation of anatase TiO 2 nanoparticles via pulsed laser ablation in liquid. *Bulletin of the Korean Chemical Society*, *34*(1), 279-282.

[21] Panda, J., Singh, U. P., & Sahu, R. (2018, September). Synthesis, characterization of TiO_2 nano particles for enhancement of electron transport application in DSSC with Cu-BPCA Dye. In *IOP conference series: materials science and engineering* (Vol. 410, No. 1, p. 012008). IOP Publishing.

[22] Makuła, P., Pacia, M., & Macyk, W. (2018). How to correctly determine the band gap energy of modified semiconductor photocatalysts based on UV–Vis spectra. *The journal of physical chemistry letters*, *9*(23), 6814-6817.