CHEESA: Chemical Engineering Research Articles
ISSN **2614-8757 (Print)**, ISSN **2615-2347 (Online)**
Available online at: http://e-journal.unipma.ac.id/index.php/cheesa

Research Article

Synthesis and Characterization of SCDs/TiO_2 Composite

Sintesis dan Karakterisasi Komposit SCDs/TiO_2

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Received: 18 September 2022;
Revised: 09 December 2022;
Accepted: 09 December 2022;

doi: *10.25273/cheesa.v5i2.13915.92-100*

Abstract

Synthesis of sulphur-doped carbon nanodots immobilized on the TiO_2 surface (SCDs/TiO_2) composite was carried out using the sol-gel method with SCDs and titanium tetraisopropoxide (TTIP) as precursors. SCDs were prepared from citric acid monohydrate, urea, and sodium disulphite using the microwave technique. SCDs/TiO_2 was then visually observed under UV 365 nm and characterized by UV-Vis diffuse reflectance spectrophotometry (UV-Vis/DRS), Photoluminescence (PL) spectroscopy, Fourier transform infrared (FT-IR), and X-ray diffraction (XRD). The SCDs/TiO_2 composite product had a brown solid with a green luminescent under UV light. Furthermore, UV-Vis/DRS for variations in SCDs concentrations of 0.5%; 1.25%, and 2.5% showed Eg values of 2.33 eV, 2.14 eV, and 1.61 eV, respectively. The results showed that SCDs caused the maximum emission peak (λ_{Em}) to redshift and also affected the intensity of PL TiO_2. There was also a shift in the absorption peak towards the visible light region. Based on the results, the 0.5% SCDs/TiO_2 was the optimum concentration with the lowest intensity as an indication of separation of the (e^-) and (h^+) charge pairs, which greatly enhanced the photocatalytic efficiency.

Keywords: microwave; photoluminescence; SCDs; sol-gel; TiO_2

Abstrak

Telah disintesis komposit sulfur karbon nanodots terimobilisasi pada permukaan TiO_2 (SCDs/TiO_2) menggunakan SCDs dan titanium tetraisopropoksida (TTIP) sebagai prekursor dengan metode sol-gel. SCDs dipreparasi dari asam sitrat monohidrat, urea dan natrium disulfit dengan metode microwave. SCDs/TiO_2diamati secara visual di bawah sinar UV 365 nm dan dikarakterisasi dengan metode UV-Vis diffuse reflectance spectrophotometry (UV-Vis/DRS), spektroskopi photoluminescence (PL), Fourier transform infrared (FT-IR) dan X-ray diffraction (XRD). SCDs/TiO_2 berupa padatan cokelat dan memiliki pendaran hijau di bawah sinar UV. Hasil pengukuran dengan UV-Vis/DRS, SCDs pada rasio konsentrasi SCDs/TiO_2 0,5% 1,25% dan 2,5% memberikan nilai Eg berturut-turut 2,33 eV; 2,14 eV dan 1,61 eV. Penambahan SCDs mengakibatkan puncak serapan bergeser ke arah sinar tampak. Karakteristik photoluminescence diketahui 0,5% SCDs/TiO_2 merupakan konsentrasi optimum yang memberikan intensitas paling rendah sebagai indikasi pemisahan pasangan muatan (e^-) dan (h^+) paling baik dengan meningkatkan efisiensi fotokatalisis.

Kata kunci: *microwave; photoluminescence; SCDs; sol-gel, TiO_2*

1. Introduction

Titanium dioxide (TiO_2) is a semiconductor material, commonly used in photocatalytic processes due to its high thermal stability and environmental friendliness [1]. Photocatalytic activity of TiO_2 is also often applied to destroy microorganisms and degrade organic compounds in air and water [2]. Furthermore, this material has 3 forms of crystal structure, namely rutile, anatase, and brookite [3]. The anatase form is commonly used, and it has a bandgap energy of 3.0-3.2 eV, which is equivalent to the wavelength of UV light < 380 nm [4].

A previous study reported that only 5% of UV light produced by the sun reaches the earth, while 45% is visible light [5]. This indicates that modifications are needed, such as the use of the photocatalytic activity of TiO_2 in the visible light absorption region. This can be performed by doping metal cations in its structure to reduce the band gap energy (Eg), thereby absorbing visible light energy. Metal cations as a dopant in the TiO_2 structure can accelerate the recombination of charge pairs (e^-) and (h^+) because they act as recombination centers [6].

Combining TiO_2 with other semiconductor materials with lower Eg can suppress the charge pair recombination process. One of the materials that have the potential to inhibit the recombination process is carbon nanodots (CDs) due to their high electronic conductivity, electron storage capacity, visible light absorption, and chemical stability [7]. CDs can also be used as strong energy transfer components in photocatalyst designs [8][9]. CDs that are composited on TiO_2 act as an electron transfer facility on the surface of titanium oxide, hence, the charge pairs (e^-) and (h^+) formed can be separated [10]. Kumar et al [8] reported that CDs/TiO_2 nanocomposites synthesized by the hydrothermal method have a more ability to degrade methylene blue comparedTiO_2-P_{25}. Titirici et al [11] succeeded in synthesizing N-doped CDs/TiO_2 using the solvothermal method. Furthermore, the composite obtained was used for the photoelectrochemical oxidation of water. Martins et al [12] also synthesized a similar product, which had high photocatalytic activity against methylene blue degradation and N conversion.

Sulphur (S) quantum dots (SQD) has unique optical properties, can absorb visible light, is non-toxic, and has the potential to be used as an alloy for photocatalysts [13]. The development of CDs materials is often carried out using S doping, which forms SCDs. Loukanov et al [14] reported that there was an improvement in the fluorescence properties of the product as metal cation sensors.

This study used the sol-gel method to synthesize SCDs/TiO_2 composite because it is easy to carry out and uses simple equipment [15]. Furthermore, it focused on the composition of SCDs in TiO_2 to obtain the optimum combination with good photocatalytic activity under visible light illumination.

2. Research Methods

2.1 Tools and Materials

The tools were used in this study included a 750 Watt Panasonic NNSM32HMTTE microwave, oven, furnace, Fourier transform infrared (FT-IR) Shimadzu IR Prestige 21, Photoluminescence (PL) Spectrophotometer Horiba FluoroMax 4, UVGL-55 Handheld UV Lamp 365 nm,

Ultra Violet-Visible Spectrophotometer (UV-Vis) Shimadzu UV-1280, Ultra Violet-Visible Diffuse Reflectance Spectrophotometer (DR/UV-Vis) Agilent Cary 60, and X-ray diffraction (XRD) X'Pert PRO PANalytical.

Meanwhile, the materials used were distilled water (H_2O), acetic acid (CH_3COOH; Merck), citric acid monohydrate ($C_6H_8O_7.H_2O$; Merck), acetylacetone ($C_5H_8O_2$; Merck), ethanol (C_2H_5OH; 97%, Merck), sodium disulfite ($Na_2S_2O_5$; Merck), urea (CH_4N_2O; Sigma Aldrich), and titanium tetraisopropoxide (TTIP; 97%, Sigma Aldrich).

2.2 Procedure

SCDs were synthesized using the microwave method [13]. A total of 2 g citric acid monohydrate, 4 g urea, 1 g sodium disulphite, as well as sodium disulphite: citric acid monohydrate with a ratio of 0.5 (w/w) were dissolved in 60 mL of distilled water, and stirred using a magnetic stirrer for 5 minutes. Subsequently, the solution was heated in a 450 W microwave on high mode for 12 minutes, and dried in an oven at 100 °C for 10 minutes to produce 1.3 g SCDs powder.

The SCDs/TiO_2 composite was synthesized using the sol-gel method based on the method proposed by Sugiarti et al [16]. The Ti(OH)n sol was prepared by mixing 2 solutions, namely A and B. Solution A was a mixture of 26.5 mL ethanol, 2 mL acetic acid, and 2 mL of distilled water, while B was prepared from 7.5 mL TTIP, 26.5 mL ethanol, and I mL acetylacetone in a reflux flask. Furthermore, solution A was dropped slowly into B while stirring using a magnetic stirrer. The reflux process was then carried out at 55 °C for 2 hours to produce a light yellow Ti(OH)n sol, which was aged for several days to form a Ti(OH)n gel. The product was dried in an oven at 80 °C, followed by calcination at 450 °C for 3 hours to produce TiO_2 in the form of white powder.

A total of 0.4 g TiO_2 powder was mixed with SCDs at ratios of 0.5, 1.25, and 2.5% (w/w). Subsequently, 5 mL of distilled water was added to the mixture while stirring for 20 minutes, followed by drying in an oven at 80 °C for 12 hours to produce a powder-shaped SCDs/TiO_2 composite. The resulting composite was then characterized using UV-Vis/DRS, PL, FT-IR, and XRD.

3. Results and Discussion

SCDs composite synthesis was carried out using the microwave heating method [13] with citric acid monohydrate as carbon chain precursor and urea as CDs surface passivation agent. Furthermore, sodium disulphite was the source of sulphur (S), which served as a dopant in the CDs structure.

Citric acid reacts with urea to form a citric acid amide. Heating with microwave energy triggers a carbonization process, which causes dehydration and deammoniation of the hydroxyl and amino groups in intermolecular compounds [16]. Furthermore, the carbon chains undergo rearrangement to form nanosheet structures and grow into quantum dots/nanodots SCDs material [17]. SCDs solids still contain water, which was used as a solvent, and then dried in an oven at 100 °C [14]. Based on observations of the luminescence properties of SCDs using UV light illumination (λ = 365 nm), a yellowish-green color was produced during the process. The emergence of this color was due to competition between various emission centers (bright edge states) that

dominate the optical properties of SCDs [18].

The synthesis of TiO_2 was carried out based on the method proposed by Aritonang et al. [4], starting with the preparation of $Ti(OH)_4$ sol using solution B containing titanium tetraisopropoxide (TTIP) precursor in ethanol solvent. Acetylacetone was then added to the mixture [16], and it was refluxed at 55 ^oC, followed by calcination to form sol [17-18]. This process produced white TiO_2 crystals, as previously reported by Aritonang et al [19]. The composite was synthesized by mixing TiO_2 with SCDs at various concentration ratios of 0.5-2.5, which led to the production of a light brown solid SCDs/TiO_2 composite, as illustrated by Li et al [20].

3.1 UV-Vis/DRS Characterization

UV-Vis/DRS characterization as a relationship (%) of reflectance to wavelength is shown in Figure 1. Furthermore, TiO_2 gave an absorption peak in the region below 380 nm. The results also showed that the addition of SCDs on TiO_2 widened the peak to the visible light region [18].

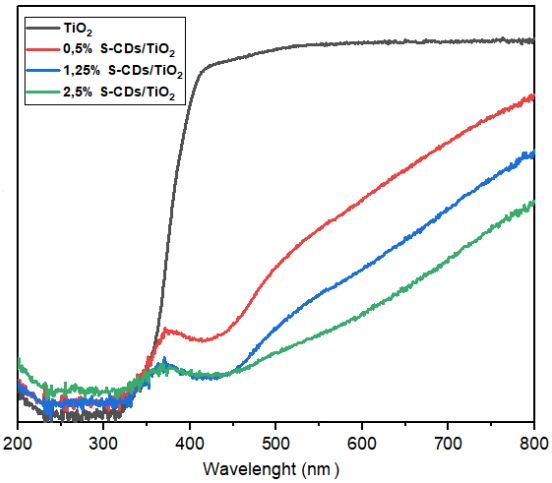

Figure 1. Spectra %R versus Wavelength (nm): TiO_2 and SCDs/TiO_2

The band gap energy of the SCDs/TiO_2 composite was determined using the Tauc equation (1):

$$(\alpha h\upsilon)^2 = B\,(h\upsilon - Eg) \quad \text{.....(1)}$$

Where, α is the absorbance coefficient, $h\upsilon$ is photon energy, B is constant, and Eg is bandgap energy. The value of α can be substituted with the Kubelka-Munk coefficient into equation (2).

$$(Kh\upsilon)^{1/n} = B(h\upsilon - Eg) \quad \text{.....(2)}$$

The graph of the relationship between $(Kh\upsilon)^{1/2}$ to hυ produced a band gap energy (Eg), which is presented in Table 1.

Table 1. Bandgap Energy Value

Material	Eg (eV)
TiO_2	3,27
0,5% SCDs/TiO_2	2,33
1,25% SCDs/TiO_2	2,14
2,5% SCDs/TiO_2	1,61

Based on Table 1, the band gap energy of TiO_2 was 3.27 eV, and this is in line with the results of Linsebigler et al [6]. The Eg values of SCDs/TiO_2 composite at a CDs ratio of 0.5 %; 1.25 % and 2.5% were 2.33 eV, 2.14 eV, and 1.61 eV, respectively. A low Eg value indicates the formation of SCDs/TiO_2 composite. Furthermore, the lower the band gap energy generated, the lower the energy required to excite electrons from the valence to the conduction band. Based on the Eg value, the SCDs/TiO_2 composite at the three concentration ratios was estimated to be active when illuminated with visible light.

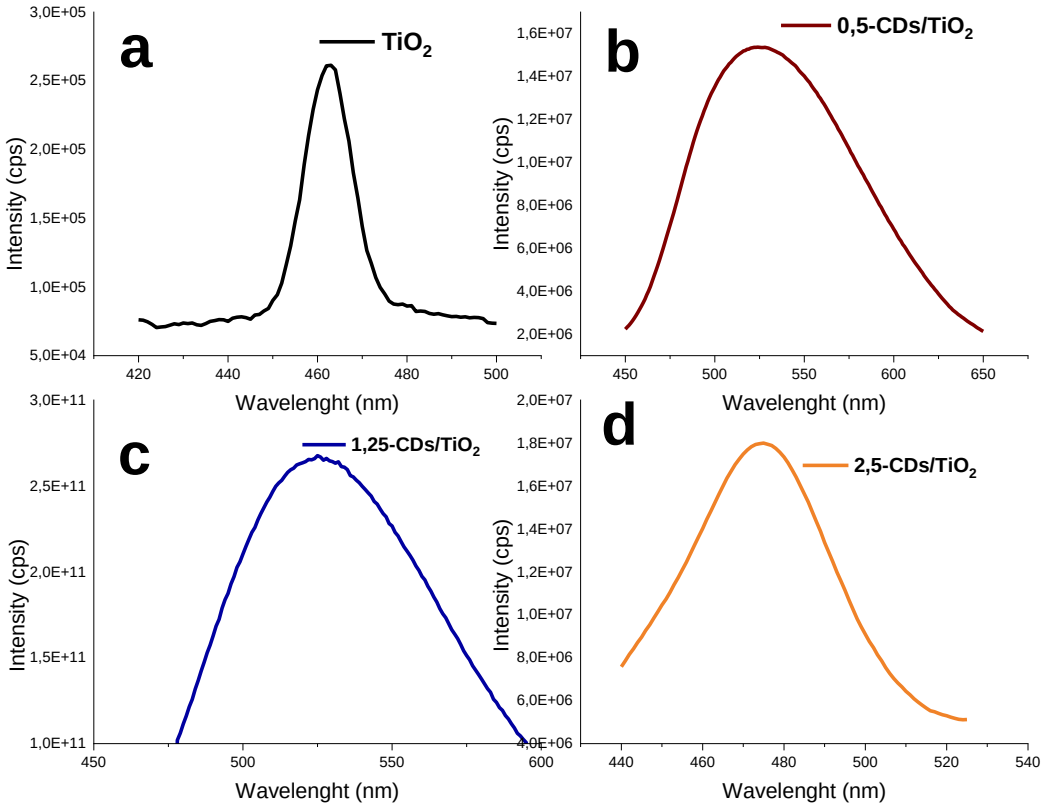

Figure 2. Photoluminescence spectra: (a) TiO_2; SCDs-TiO_2 with a CDs ratio of (b) 0.5%, (c) 1.25% and (d) 2.5%.

3.2 Photoluminescence (PL)

The measurements of the PL spectra of TiO_2 and SCDs/TiO_2 at various SCDs concentration ratios are presented in Figure 2. Based on Figure 2a, TiO2 gave a PL absorption peak at 460 nm. The calculation results of Eg showed that the combination of SCDs in a titanium oxide matrix with a concentration ratio of 0.5 and 1.25% (w/w) caused a shift in the PL absorption peak to the visible region (>400 nm). Huang et al [21] also stated that the interaction between SCDs and TiO_2 occurs physically (couple heterojunction) on the composite surface, and this led to a decrease in Eg. However, the addition of SCDs at a concentration of >1.5% caused an insignificant shift due to particle agglomeration.

The fluorescence emission absorption peak of the SCDs/TiO_2 composite was formed due to the recombination of photoelectron (e^-) and photo hole (h^+) charge pairs induced by photons [22]. Based on Table 2, SCDs/TiO_2 composite with an SCDs ratio of 0.5% had the lowest intensity of $1.53x10^7$ cps, and the lower the value obtained, the better the charge pair separation [8][11]. The PL emission analysis on the three SCDs/TiO_2 samples showed that the composite with a CDs ratio of 0.5% has photocatalytic activity under light illumination with the highest efficiency, which corresponds to the Eg value.

Table 2. λEm and TiO_2 and SCDs/TiO_2 emissions

Sample	λEm (nm)	Intensity (cps)
TiO_2	463	$2,61x10^5$
0,5% SCDs/TiO_2	524	$1,53x10^7$
1,25% SCDs/TiO_2	525	$2,6x10^{11}$
2,5% SCDs/TiO_2	475	$1,92x10^7$

3.3 FT-IR Spectrophotometry

The FT-IR spectrophotometry characterization of the SCDs/TiO_2 composite is shown in Figure 3.

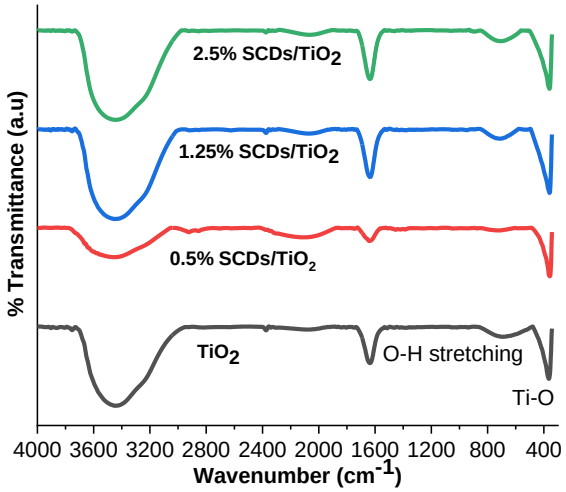

Figure 3. FT-IR spectra of TiO_2 and SCDs/TiO_2 composites

The absorption peaks of TiO_2 and the SCDs/TiO_2 composite observed at wave numbers 3446-3459 cm^{-1} were the stretching vibrations of the O-H groups [14]. Furthermore, the absorption at wave numbers 1635-1640 cm^{-1} was an O-H bending vibration due to the uptake of water vapor [23].

Absorption at wave numbers 696-718 cm^{-1} was a stretching vibration of Ti-O [29,30]. TiO_2 and SCDs/TiO_2 composites were observed for absorption peaks at wave number 412 cm^{-1} as a characteristic of Ti-O stretching vibration [24][6]. The results showed that the combination of SCDs with TiO_2 does not cause a shift in the titanium oxide absorption peak. This indicated that the interaction of these materials occurs physically on the surface, and does not distort the TiO_2 structure.

3.4 X-ray diffraction (XRD)

Characterization with the XRD method aimed to determine the phase structure and size of the crystallites. Furthermore, XRD analysis was carried out on TiO_2 as well as 0.5, 1.25, and 2.5 (w/w) SCDs/TiO_2. The results of XRD characterization of TiO_2 and SCDs/TiO_2 are presented in Figure 4.

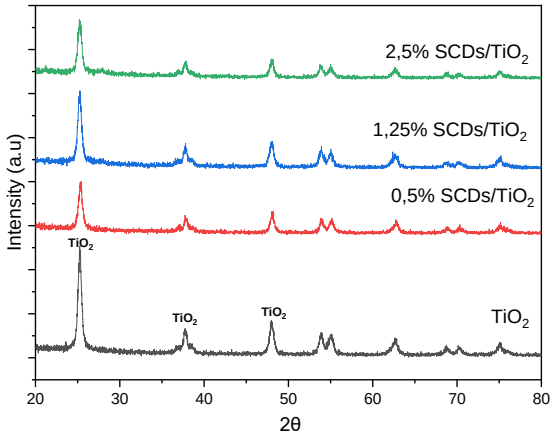

Figure 4. XRD diffractogram of TiO_2 and SCDs/TiO_2 composite

The TiO_2 and SCDs/TiO_2 diffractograms showed that there were similarities in the three highest peaks produced, namely in the range of 2θ 25°, 37°, and 48°. These peaks indicated that TiO_2 had an anatase crystal phase, which was similar to the JCPDS database catalog No. 01-075-8897, and this was in line with the results obtained from Aritonang et al [19]. The similarity of the diffraction angle peaks produced by SCDs/TiO_2 and TiO_2 showed that no new compounds were formed from the SCDs/TiO_2 composite. The size of the crystallites was then determined using the Deybe-Scherrer equation (3) [4].

$$D = k\lambda / B\cos\theta \quad \text{.....(3)}$$

Where, D is the crystal thickness (size) (nm), k is a material constant with value < 1 (common value is 0.9), λ is the X-ray wavelength during measurement (nm), B is the width of the half-peak on the diffractogram, and θ is derived from the graph data of 2θ on the diffractogram. The

calculation results of the crystallite size are presented in Table 3.

Table 3. Size of TiO_2 and SCDs/TiO_2 crystallites

Material	2 θ	d (nm)	D (nm)
TiO_2	25,2834 37,8352 48,1053	0,2597	4,045
0,5% SCDs/TiO_2	25,2772 37,6632 47,9844	0,2502	3,938
1,25% SCDs/TiO_2	25,3332 37,7934 48,0287	0,2596	3,942
2,5% SCDs/TiO_2	25,3648 37,7796 48,0902	0,2598	3,946

Table 3 shows that the addition of SCDs decreased the size of TiO_2 crystallites, but there was no 2θ shift in angular position. Therefore, the formation of the composite does not occur in the anatase TiO_2 crystal phase transformation. The results showed that 0.5% SCDs/TiO_2 had the smallest crystallinity size of 3.938 nm compared to TiO_2 and other composites. This indicated that 0.5% SCDs is the optimum concentration for the production of composites. These findings were supported by PL characteristic data and UV-Vis/DRS spectrum analysis results. The addition of SCDs at a concentration of 2.5% increased the crystallinity size of TiO_2. This was due to the inhomogeneous mixture of SCDs and TiO_2 from the physical mixing process.

4. Conclusion

The SCDs/TiO_2 composite produced is a brown solid, which has a green glow under 365 nm UV light. Furthermore, the UV-Vis/DRS characteristics include band gap energy values of 2.33, 2.14, and 1.61 eV for the 0.5%, 1.25%, and 2.5% composites, respectively. The results showed that SCDs caused a shift in the absorption peak of the visible region. PL characteristics also revealed that 0.5% SCDs/TiO_2 is the optimum concentration, which gave the lowest intensity. Based on XRD diffractogram analysis, this product has an anatase crystalline structure with a crystallite size of 3.938 nm.

Acknowledgment

The authors are grateful to the Dean and Head of the Chemical Research Laboratory, Faculty of Mathematics and Natural Sciences, Universitas Tanjungpura, for supporting this study.

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