

# Synthesis, Characterization of Cu, S doped TiO<sub>2</sub> and Its Photocatalytic Activity for Degradation of Remazol Black B

*by* Didik Setiyo Widodo

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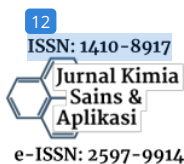
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## Synthesis, Characterization of Cu, S doped TiO<sub>2</sub> and Its Photocatalytic Activity for Degradation of Remazol Black B

Abdul Haris <sup>a,\*</sup>, Gunawan <sup>a</sup>, Didik Setiyo Widodo <sup>a</sup>, Rahmad Nuryanto <sup>a</sup>, Retno Ariadi Lusiana <sup>a</sup>, Mei Viantikasari <sup>a</sup>

<sup>a</sup> Chemistry Department, Faculty of Sciences and Mathematics, Diponegoro University, Tembalang, Semarang, Indonesia

\* Corresponding author: [a.haris@live.undip.ac.id](mailto:a.haris@live.undip.ac.id)

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### Abstract

Copper and sulfur modified TiO<sub>2</sub>(Cu-S-TiO<sub>2</sub>) photocatalyst was successfully synthesized using TiCl<sub>4</sub>, Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O and H<sub>2</sub>SO<sub>4</sub> as precursors by the sol-gel method and calcination at 450°C for 4 hours. The synthesized photocatalyst was characterized by X-Ray Diffraction (XRD), Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS), Diffuse Reflectance Spectroscopy (DRS), Brunauer Emmett Teller (BET) method. The XRD results showed that the Cu-S-TiO<sub>2</sub> photocatalyst had an anatase phase with a crystal grain size of 17.54 nm. However, the SEM image of the modified TiO<sub>2</sub> showed inhomogeneous phase due to the crystal clustering of imperfect homogenization during the synthesis and sintering processes. The patterns of EDS of Cu-S-TiO<sub>2</sub> depicted the elements of Ti, O, Cu and S with doping of Cu and S c.a. 7 and 1%, respectively. Analysis using DRS UV-Vis showed Cu-S-TiO<sub>2</sub> was able to shift the absorption of the TiO<sub>2</sub> photocatalyst wavelength to the visible region with a band energy gap of 1.9 eV. The BET analysis results showed that the specific surface area (S<sub>BET</sub>), pore volume (V<sub>p</sub>) and average pore volume radius (D<sub>p</sub>) were measured from large Cu-S-TiO<sub>2</sub>, the Cu-S-TiO<sub>2</sub> had good physicochemical and photocatalytic properties. The photocatalytic activity of 0.1 g Cu-S-TiO<sub>2</sub> with 15 Watt tungsten light irradiation for 4 h was able to degrade 50 mL remazol black B 10 mg/L c.a. 92.60 %.

### 1. Introduction

One potential method for treating organic waste is by photocatalysis. Photocatalyst material when subjected to the appropriate photon energy will produce electrons in the conduction band and holes in the valence band. The electrons can reduce heavy metals, while the holes can oxidize organic compounds [1]. Semiconductor of titanium dioxide is widely used as a photocatalyst because it is nontoxic, inexpensive, and has a high catalytic activity [2]. TiO<sub>2</sub> appears in 3 different polymorphic forms, namely rutile, anatase, and brookite. Two TiO<sub>2</sub> crystal structures, rutile and anatase, are most commonly used in photocatalysis. The structure of the two crystals is distinguished by octahedron distortion and the octahedron chain arrangement pattern. This difference in lattice structure causes differences in density and electronic band structure between the two forms of TiO<sub>2</sub> [3]. TiO<sub>2</sub> has a large band energy of 3.2 eV which is the absolute difference between the conduction band energy

and the position of the valence band energy level. This indicates that h<sup>+</sup> on the surface of TiO<sub>2</sub> is a strong oxidizing species so that it can oxidize other chemical species that have smaller redox potentials including H<sub>2</sub>O molecules which will produce hydroxyl radicals. The hydroxyl radical has a potential of 2.8 V at pH 1 so that it can oxidize organic substances which mostly have a smaller redox potential [4]. However TiO<sub>2</sub> has a band gap of 3.2 eV (anatase) which can only be applied under UV light (λ < 387 nm). Research on the modification of TiO<sub>2</sub> which aims to expand the absorption of wavelengths so that it can be applied to visible light is being developed, among others, by the combination of sensitized dye [5, 6], doping of transition metals [7] and insertion of precious metals [8]. Doping transition metals and non-metals in TiO<sub>2</sub> can modify its electronic structure effectively and be able to shift its absorption at lower energy levels [9]. In this study, synthesis of Cu and S-modified TiO<sub>2</sub>(Cu-S-TiO<sub>2</sub>) for degradation of remazol black B was applied to the visible light region [10, 11]. The synergy effect of S and

Cu doped on TiO<sub>2</sub> has a higher activity compared to bare TiO<sub>2</sub> in degrading organic compounds [12, 13].

In this study, the sol gel method was used which is one method that is often used to synthesize TiO<sub>2</sub> doped with metals and non-metals. This method allows to control several parameters with a relatively slow reaction process, including homogeneity of composition, grain size, particle morphology and porosity [14, 15, 16]. The process of sol gel generally includes the hydrolysis stage of a metal alkoxide or precursor alkoxide, usually in alcohol, so that the hydroxide is obtained, then proceed with the condensation process, so that the sol is formed. Removal of solvent by heating gives a gel and then xerogel. Cu-S-TiO<sub>2</sub> powder was obtained as a result of calcination [17, 18, 21]. Synthesized Cu-S-TiO<sub>2</sub> powder was carried out by characterization and photocatalytic activity test for degradation of remazol black B.

## 2. Experimental Details

### 2.1. Synthesis of Cu-S-TiO<sub>2</sub>

Synthesis of Cu-S-TiO<sub>2</sub> was done by addition of 2-propanol dropwise into 10 mL TiCl<sub>4</sub> until total volume of 100 mL (d=1.73 g/cm<sup>3</sup>) to form a clear yellow solution. The solution was stirred for 30 min at room temperature. Then, gradually adding with NH<sub>4</sub>OH solution until pH 3–4, then stirred for 1 h until white solution was obtained. After that, solution containing 7.7 g Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O and 4.9 mL H<sub>2</sub>SO<sub>4</sub> (98%, density=1.84g/cm<sup>3</sup>) as dopants was added dropwise with stirring for 6 h until a green solution was formed, and finally a green Cu-S-TiO<sub>2</sub> gel was formed. The gel was left for 3 days and dried in oven at 80–100 °C for 24 h so that xerogel of Cu-S-TiO<sub>2</sub> was obtained, then calcined at 450 °C for 4 hours. The product was greenish Cu-S-TiO<sub>2</sub> powder. All chemicals were purchased from Merck.

### 2.2. Characterization of Cu-S-TiO<sub>2</sub>

Synthesized Cu-S-TiO<sub>2</sub> was characterized using X-Ray Diffraction Instrument (XRD) (Shimadzu 7000), Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS), UV-Visible Diffuse Reflectance Spectrophotometer, Instrument Quantachrome Novawin version 11.0 for BET test.

### 2.3. Photocatalytic process of sample solution

The photocatalyst process was carried out at a room temperature in a static cylindric flask reactor by soaking 0.1 g Cu-S-TiO<sub>2</sub> into a sample solution of 50 mL remazol black B 10 mg/L, then the reactor was irradiated with a 15 Watt tungsten lamp (Philips) at a distance of 20 cm for varied times of 1, 2, 3, 4 h. The remaining remazol black B, further, was measured using UV-Vis spectrophotometer (PG Instruments Limited Model T60U). The percentage of degradation was calculated using the following equation:

$$\text{Degradation (\%)} = \frac{C_0 - C_t}{C_0} \times 100 \%$$

Where C<sub>0</sub> and C<sub>t</sub> are initial sample concentration and sample concentration after photocatalysis process, respectively.

## 3. Results and Discussion

### 3.1. XRD characterization of Cu-S-TiO<sub>2</sub>

X-ray diffractogram results were analyzed using the Match! Application, and compared it with the data in JCPDS to know the shape of the crystal and the constituent compounds formed from the synthesized Cu-S-TiO<sub>2</sub>. Fig. 1 shows the XRD diffractogram of Cu-S-TiO<sub>2</sub>.

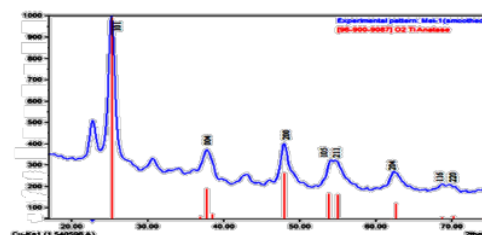


Figure 1. XRD patterns of Synthesized Cu-S-TiO<sub>2</sub>.

Analysis results of Match! application there is a red line which is a specific peak that refers to the anatase TiO<sub>2</sub> database (entry # 96-900-9087). Based on the picture shows that the crystal structure formed is anatase TiO<sub>2</sub> which is characterized by the presence of peaks that correspond to the anatase TiO<sub>2</sub> database in the Match! Application. The diffractogram results then were matched with the data contained in (JCPDS 21-1272) as showed X-ray diffraction patterns from samples of nanocrystalline anatase structures confirmed by (1 0 1), (0 0 4), (2 0 0), (1 0 5) and (2 1 1) with the main peak at 2θ = 25.28. The average nanoparticle size is estimated from the Scherrer equation on anatase peak diffraction (1 0 1). The size of the crystal sample is determined quantitatively using Scherrer method with the equation:

$$D = \frac{k \times \lambda}{\beta \times \cos \theta}$$

Where k is a constant of 0.89; λ is the wavelength of the X-ray source (in this case Cu Kα is 0.15418 nm), and β is half width of the diffraction peak (FWHM) in radians. The β value used in this case is the maximum peak value possessed by the anatase peak at 2θ of 25.28° which is equal to 0.48505° and obtained a crystal size of 17.54 nm. The small size of the crystal can expand the catalyst surface so that its performance becomes more effective. The diffractogram shows the peaks at 2θ of 22.835, 30.728, and 42.926° was also formed and estimated as S6, SO<sub>3</sub> and CuO from the Match! Database. The diffractogram are mapped to the data base in JCPDS 74-1654, 73-2169, and 78-0428. The emergence of these peaks indicates that some of the dopants were not completely dispersed in the crystal lattice structure of TiO<sub>2</sub> either at substitutional or interstitial position but the dopants were only embedded on the TiO<sub>2</sub> surface. The synthesis process which could not create a great homogeneity allows the added dopants not to be all dispersed. The calcination environment, rich in oxygen, formed some of the dopants were not in the atom forms but also in the form of their oxides, namely SO<sub>3</sub> and CuO.

### 3.2. SEM-EDS characterization of Cu-S-TiO<sub>2</sub>

SEM images of synthesized Cu-S-TiO<sub>2</sub> are shown in Fig. 3. Fig. 2. The surface SEM image at a 1000x magnification only shows the presence of many chunks in

same surface with different intensity and dimension indicating the presence of different matter that is supposed as the dopants on the  $\text{TiO}_2$  surface. At 5000x magnification the visible surface is bumpy and uneven with many cavities, making it possible to assist in trapping the target compounds on the surface of the photocatalyst so as to facilitate the photocatalyst to oxidize or reduce the compounds. Whereas, at 15,000x magnification can be seen the morphology of the material surface is viewed not homogeneous. The matter may form clusters due to imperfect homogenization during synthesis and sintering processes. Next characterization by EDS informs elemental composition of the surface that is as shown in fig. 3 as well.

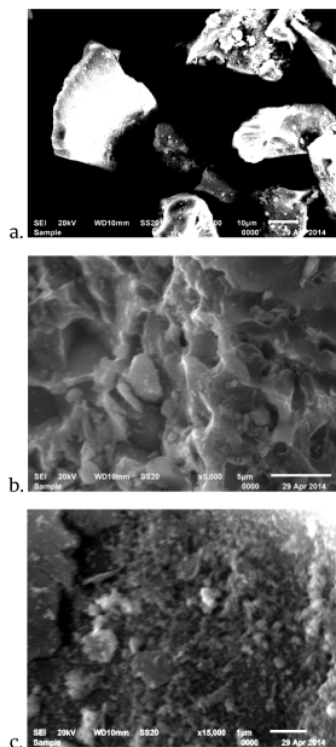


Figure 2. SEM images of Cu-S- $\text{TiO}_2$  at magnifications of a. 1000x; b. 5000x; c. 15000x.

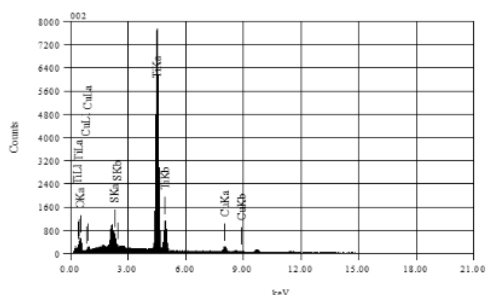


Figure 3. EDS profile of synthesized Cu-S- $\text{TiO}_2$ .

Table 1. Elemental composition of Cu-S- $\text{TiO}_2$  measured by EDS.

Element	Atom (%)
O	25.62
S	1.03
Ti	66.42
Cu	6.93

Based on the EDS pattern of Cu-S- $\text{TiO}_2$  photocatalyst as seen in Fig. 3 and Table 1, the synthesized photocatalyst composed of several elements, namely oxygen (O), sulfur (S), titanium (Ti) and copper (Cu). So that from these results proved that in the synthesis process,  $\text{Cl}^-$  which binds to  $\text{Ti}^{4+}$  was substituted all by the alkoxide group ( $-\text{OCH}(\text{CH}_3)_2$ ) from isopropanol to form titanium (IV) isopropoxide and HCl gas. Then the aging and calcination processes carried out was able to remove the Cl component so that the synthesized photocatalyst had no impurity. The percentages of each dopant were 1% and 7% for sulfur (S) and copper (Cu) elements, respectively from total mass.

### 3.3. Characterization of Cu-S- $\text{TiO}_2$ photocatalyst using Diffuse UV-Visible Reflectance Spectrophotometer.

Characterization using a UV-Vis diffuse reflectance spectrophotometer aims to determine the band gap energy. The analysis using diffuse UV-Vis reflectance spectrophotometer are shown in Fig. 4 as the following:

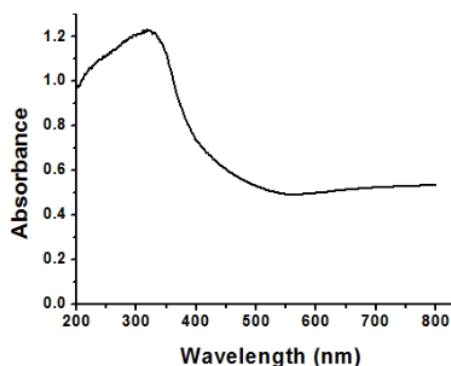


Figure 4. Variation of absorbance with wavelength of Cu-S- $\text{TiO}_2$  measured using UV-Vis diffuse reflectance spectrophotometer.

Based on the spectrum in Fig. 4 shows that the presence of Cu and S doping was able to shift the absorption of the  $\text{TiO}_2$  photocatalyst wavelength at the visible light as evidenced by the absorption (absorbance > 0.4) at wavelengths above 400 nm and an increase in absorption in the wavelength region of 700 nm. The value of band gap energy generated from the doping process can be estimated by Kubelka-Mulk equation by processing sample reflectance data from the analysis results. The band gap energy value is obtained by extension of the line from the maximum slope of the graph from relationship between  $[F(R)h\nu]^n$  and  $h\nu$  at  $[F(R)h\nu]^n=0$  in Fig. 5, then the band gap energy value found is 1.9 eV. Therefore, it is proven that the doping can reduce the band gap energy of  $\text{TiO}_2$  which generally has a band gap of 3.2 eV. The smaller the band gap energy value,



the smaller the energy required by the photocatalyst to excite electrons from the valence band to the conduction band. This is very beneficial in photocatalyst applications because it is able to work at wavelength 350 with a wider range of visible light region. Curve of  $[F(R)h\nu]^{1/2}$  vs  $h\nu$  for determination of band gap as shown in Fig. 5 below:

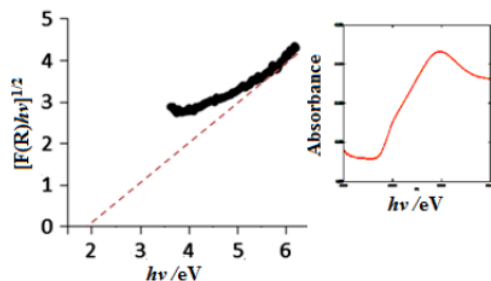


Figure 5. Curve of  $[F(R)h\nu]^{1/2}$  vs  $h\nu$  for determination of band gap.

Doping either metal or non metal occupying the constitutional and interstitial sides of the  $\text{TiO}_2$  crystal lattice provides changes to the electronic band structure [15]. The size of  $\text{Cu}^{2+}$  ion (0.87 Å) is not much different from  $\text{Ti}^{4+}$  (0.75 Å) so that it is possible to dope Cu into the crystal lattice which can then distort the crystal structure of  $\text{TiO}_2$  [16]. Copper doping which is on the constitutional side of the  $\text{TiO}_2$  crystal lattice will produce a band gap energy level under the  $\text{TiO}_2$  conduction band which as if it is in the interstitial position it will produce a band gap energy between the valence band and the conduction band [2]. The presence of dopant sulfur will replace O atoms in  $\text{TiO}_2$ . This substitution produces a mixture of p orbitals from S and O atoms which causes a rise in the valence band so that a smaller band gap value is obtained [10]. The change in band gap energy in the synthesized Cu-S  $\text{TiO}_2$  indicates that some of the dopants are dispersed into the  $\text{TiO}_2$  crystal lattice.

#### 3.4. BET Analysis

The results of BET analysis of Cu-S- $\text{TiO}_2$  photocatalyst had a specific surface area ( $S_{\text{BET}}$ ) of 85.737  $\text{m}^2/\text{g}$ , pore volume ( $V_p$ ) of 0.1137  $\text{cc}/\text{g}$  and radius of average pore volume ( $D_p$ ) of 1060,100 Å, thus the modified photocatalyst had physicochemical properties and can be used as a photocatalyst material.

#### 3.5. Test of photocatalytic activity of Cu-S- $\text{TiO}_2$ on degradation of Remazol Black B

Before the photocatalytic activity tests using Cu-S- $\text{TiO}_2$ , 50 mL remazol black B 10 mg/L solution was irradiated using 15 Watt tungsten lamp at a distance of 20 cm for 4 h and the concentration reduced c.a. 9.09 % due to the oxidation process by the irradiation. Then, solution of 50 mL remazol black B 10 mg/L was evaluated by addition with 0.1 g of Cu-S- $\text{TiO}_2$  photocatalyst for 4 h, which could reduce concentration of 16.36% due to adsorption process of the photocatalyst. Fig. 6 shows the decrease of the absorbance of remazol black B obtained by both irradiation and photocatalyst Cu-S- $\text{TiO}_2$  without irradiation.

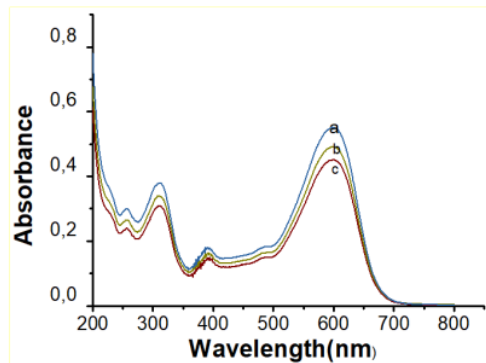


Figure 6. UV-Vis spectra of remazol black B (a) at initial, (b) after irradiation with tungsten lamp 15 Watt for 4 h without photocatalyst, (c) after addition with 0.1 g Cu-S- $\text{TiO}_2$  for 4 h without irradiation.

From the results of remazol black B analysed using UV-Vis spectrophotometer before and after contacted with modified photocatalys as depicted in Fig. 7 below.

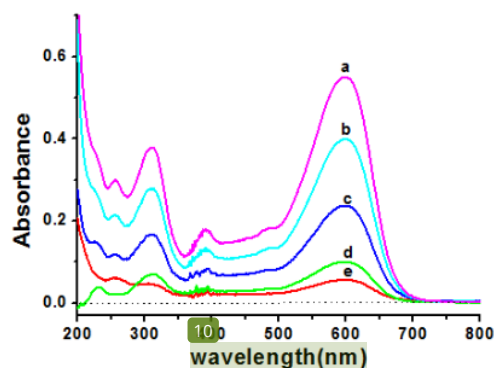


Figure 7. UV-Vis spectra of remazol black B before and after contacted with modified photocatalyst of Cu-S- $\text{TiO}_2$  for (a) 0, (b) 1, (c) 2, (d) 3, and (e) 4 h under tungsten lamp irradiation.

From UV-Vis spectra, it can be seen that the longer the photocatalytic process increases the degradation of remazol black B from 1; 2; 3 and 4 hours with degradation percentages of 24.54; 55.86; 84.32; and 92.60%, respectively. The results also shows that the photocatalyst activity in degrading remazol black B decreases with increasing time of photocatalysis.

#### 4. Conclusion

Modified Cu and S doped  $\text{TiO}_2$  had been synthesized by sol-gel and calcination methods using precursors  $\text{TiCl}_4$ ,  $(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  and  $\text{H}_2\text{SO}_4$ . The resulting Cu-S- $\text{TiO}_2$  had an anatase phase with a crystal size of 17.54 nm. The doped copper and sulfur of 7 and 1% respectively, was able to shift the wavelength absorption of the  $\text{TiO}_2$  photocatalyst towards the visible region with a band gap energy of 1.9 eV. BET analysis of Cu-S- $\text{TiO}_2$  gave large specific surface area, pore volume, and a measured radius of the average pore volume, consequently the synthesized Cu-S- $\text{TiO}_2$  had good physicochemical and photocatalytic

properties. The photocatalytic activity test results of Cu-S-TiO<sub>2</sub> were able to degrade remazol black B.

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GENERAL COMMENTS

Instructor

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