

# PREPARATION OF S-DOPED

*by* Yusnaidar Unja

---

**Submission date:** 29-May-2023 09:02PM (UTC+0700)

**Submission ID:** 2104604798

**File name:** 929\_pdf.pdf (1.4M)

**Word count:** 1855

**Character count:** 9667

## PREPARATION OF S-DOPED TiO<sub>2</sub> VIA SOL-GEL/ HYDROTHERMAL METHOD AND ITS ACTIVITY AS PHOTODEGRADATION OF RB05

Adiansyah<sup>1,\*</sup>, I. Elisabeth Purba<sup>2</sup>, M. Tarigan<sup>1</sup>, Lisnadiyanti<sup>3</sup>, Yusnaidar<sup>4</sup>  
 and I.P. Mahendra<sup>5</sup>

<sup>1</sup>Department of Chemistry, Universitas Sari Mutiara, Indonesia

<sup>2</sup>Department of Public Health, Universitas Sari Mutiara, Indonesia

<sup>3</sup>Faculty of Nursing and Midwifery, Universitas Binawan, Indonesia

<sup>4</sup>Department of Chemistry Education, Faculty of Teacher Training and Education,  
 Universitas Jambi, Indonesia

<sup>5</sup>Department of Chemistry, Faculty of Mathematics and Sciences, Universitas Sumatera Utara

\*E-mail: adiansyah\_skd@yahoo.co.id

### ABSTRACT

S-doped TiO<sub>2</sub> photocatalyst has been prepared by the hydrothermal method at 180°C that assisted by the sol-gel method and its photocatalytic activity was evaluated on reactive black 05. The obtained photocatalyst was characterized by XRD and SEM. The phase composition of S-doped TiO<sub>2</sub> was influenced by the amount of sulfur.

**Keywords:** TiO<sub>2</sub>, Doping process, Sulfur, Photocatalyst, Reactive Black 05.

© RASĀYAN. All rights reserved

### INTRODUCTION

TiO<sub>2</sub> is classified as semiconductor materials that have excellent property as a catalyst, especially in the photodegradation of organic pollutants, i.e. dyes, phenolic compounds, etc. The previous researches that focused on the photoactivity of TiO<sub>2</sub>, mostly work under UV light due to it shows high activity and stable under UV light.<sup>1-3</sup>

The issues that were found during its applications, photoactivity of TiO<sub>2</sub> is not fully effective when it utilized under solar light. Several methods have been developed to improve its performance under solar light.<sup>4-6</sup> One of these efforts is through the doping technique. The doping technique also influences the bandgap of the doped materials with a range in the visible light area. As the dopant, either metal or non-metal elements can be incorporated with the TiO<sub>2</sub>, it depends to the atomic radius between dopant and oxygen atom of TiO<sub>2</sub>.<sup>7-10</sup> The previous work about the doping process of TiO<sub>2</sub> has reported results on the substitution of several elements, i.e. nitrogen, oxygen, and sulfur.<sup>10,11</sup> The incorporating of those elements into TiO<sub>2</sub> showed a significant change in the photoactivity of materials, especially sulfur.

S-doped TiO<sub>2</sub> in the previous work has been prepared using calcination technique, sol-gel, hydrothermal and in the supercritical condition in CS<sub>2</sub>/ethanol.<sup>12,13</sup> From those several methods, the combination method between sol-gel and hydrothermal provides a simple and best method to obtain nanosized of doped TiO<sub>2</sub> photocatalyst. Also, the photocatalyst of TiO<sub>2</sub> that prepared using the hydrothermal method can have a different ratio between retail and amateurs depend on the preparation of raw material and temperature during hydrothermal. Based on the literature review, there have been few reports on the preparation of S-doped TiO<sub>2</sub> using a hydrothermal method that assisted by the sol-gel method. In the present work, the S-doped TiO<sub>2</sub> was prepared using a hydrothermal method that assisted by the sol-gel method. The photoactivity of the obtained S-doped TiO<sub>2</sub> was evaluated for degradation of reactive black 05.

*Rasayan J. Chem.*, 13(1), 757-760(2020)

<http://dx.doi.org/10.31788/RJC.2020.1315461>



## EXPERIMENTAL

### Materials

Sulfur and TTIP (97%) were obtained from Merck and Sigma Aldrich, respectively. Absolute ethanol was provided by Guangdong Guanghua Sci-Tech Co. Ltd.

### Preparation of S-doped TiO<sub>2</sub>

The doping process was performed using the method that has been described previously.<sup>14</sup> The as-prepared S-doped TiO<sub>2</sub> then was characterized using several instruments, i.e Bruker D8 Advance X-ray diffractometer for crystallography analysis, scanning electron microscopy TM4000 Hitachi for morphological analysis and UV-Vis Diffuse Reflectance Spectrophotometry Shimadzu UV-2600 Series for determine the band gap value.

### Photoactivity Measurements

The photoactivity of S-doped TiO<sub>2</sub> was performed in a cylindrical glass that equipped with a condenser to control the temperature. The treatment was performed using a method that has been described as previously<sup>15</sup>. The obtained aliquots were characterized using UV-Vis spectrophotometer to determine the final concentration of imitated wastewater after treatment.

## RESULTS AND DISCUSSION

The crystallographic of doped TiO<sub>2</sub> is shown in Fig-1. The obtained S-doped TiO<sub>2</sub> with the incorporation of 0.5% S only consisted of anatase phase and rutile, no other phase can be found. However, when the amount of incorporated S increases the phase of TiO<sub>2</sub> was dominated by anatase. This result indicated the ratio of anatase, or rutile phase depends on the amount of S that doped to TiO<sub>2</sub>. The phase composition of TiO<sub>2</sub> was obtained using Match! software version 3.8.0.137 and the 0.5% S-doped TiO<sub>2</sub> showed a combination signal of JCPDS 96-900-1681 and JCPDS 96-720-6076, while 1.0% S-doped TiO<sub>2</sub> has similar signal to JCPDS 96-720-6076.

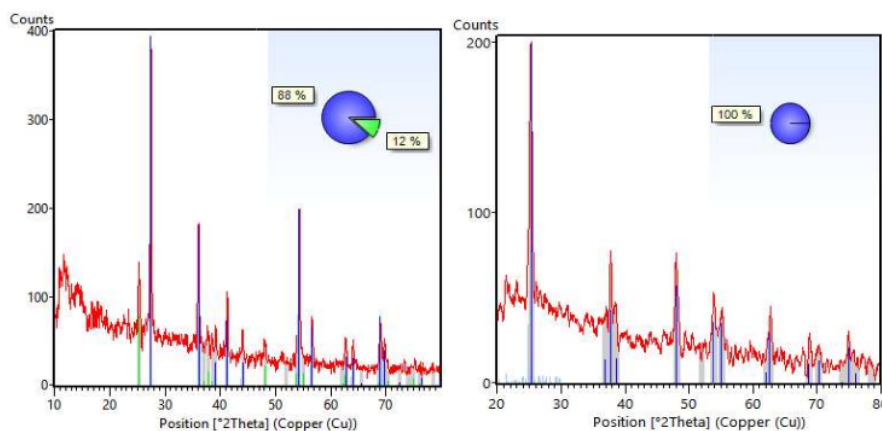


Fig.-1 XRD Spectra of (a) 0.5% S-doped TiO<sub>2</sub> and (b) 1.0% S-doped TiO<sub>2</sub>

Figure-2 shows the SEM image of doped TiO<sub>2</sub> obtained by hydrothermal assisted by sol-gel method. The effect of the amount of S that doped to TiO<sub>2</sub> can be seen in the particle size of the photocatalyst. The particle size of TiO<sub>2</sub> was reduced by the increase of the amount of S, the particle size of the TiO<sub>2</sub>. The nanoparticle of TiO<sub>2</sub> with particle size in the range > 1 μm was changed to 40-60 nm when the concentration of S increases.

The photodegradation of RB05 was evaluated by determining the concentration of dyes in the system at a specific time. In Fig-3, the photodegradation of RB05 by anatase, 0.5% S-doped TiO<sub>2</sub> and 1.0% S-doped

TiO<sub>2</sub> were 83.10; 20.73 and 98.31%, respectively. The as-prepared 1.0% S-doped TiO<sub>2</sub> showed to be more photoactive than undoped TiO<sub>2</sub> with a rate constant of  $4.03 \times 10^{-2}$ . The 0.5% S-doped exhibited the lowest photoactivity with a rate constant of  $2.24 \times 10^{-3}$ .

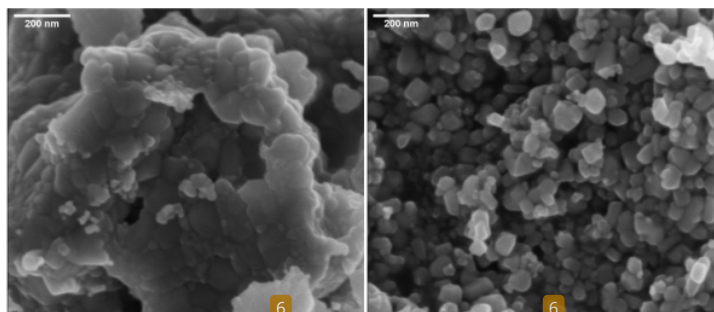


Fig.-2 The SEM Images of (a) 0.5% S-doped TiO<sub>2</sub> and (b) 1.0% S-doped TiO<sub>2</sub>

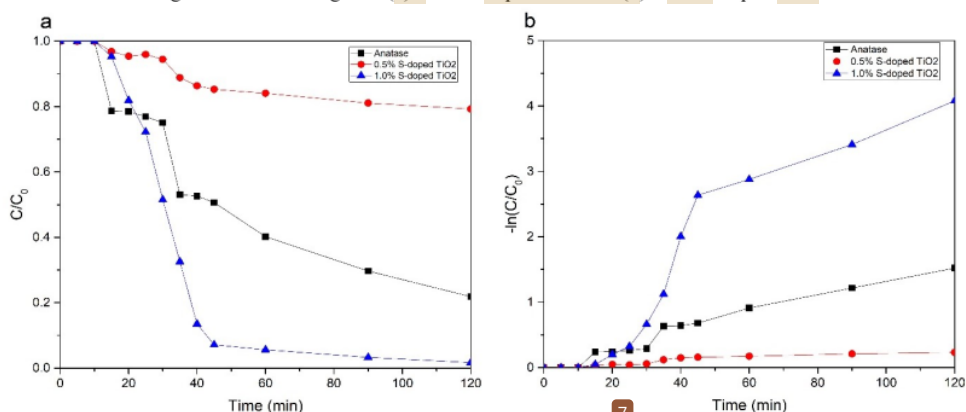


Fig.-3 (a) Photocatalytic Activity of doped TiO<sub>2</sub> and (b) Rate of Photocatalytic Activity of doped TiO<sub>2</sub>

The process of dyes photodegradation could be said as a complicated process that cannot be only viewed from one perspective, but it should be observed from all perspectives, i.e. particle size, morphology, and phase/ chemical composition of photocatalysts<sup>16-20</sup>. In this study, the photoactivity of doped TiO<sub>2</sub> was influenced by two factors, i.e. the particle size of doped TiO<sub>2</sub> and phase composition of doped TiO<sub>2</sub>. Based on kinetic theory, particle size has a big influence on the reaction rate. Smaller particle size will have a larger surface area and it can facilitate the dyes photodegradation be more effective as the impact of the increase of photogenerated electrons. The other perspectives, the phase composition of doped TiO<sub>2</sub> has an important role on the dye photodegradation process. The ratio of rutile phase in the 0.5% S-doped TiO<sub>2</sub> was 88%, the high rutile phase content in S-doped TiO<sub>2</sub> influenced to the lower photocatalytic activity. When the amount of S was changed, the doped TiO<sub>2</sub> phase was dominated by anatase and improved the photocatalytic activity. This result also supported by the previous work, the ratio of rutile and anatase phase has an important role to the photocatalytic activity, when the rutile ratio is high the photocatalyst will show lower activity and vice versa.<sup>4,16,21,22</sup>

### CONCLUSION

S-doped TiO<sub>2</sub> was successfully synthesized using hydrothermal assisted sol-gel method. The morphological and photocatalytic activity of doped TiO<sub>2</sub> was totally dependent on the S amount that incorporated into TiO<sub>2</sub>.

## ACKNOWLEDGEMENT

<sup>2</sup> This research was supported by Direktorat Riset dan Pengabdian Masyarakat Direktorat Jenderal Riset dan Pengembangan Kementerian Riset, Teknologi, dan Pendidikan Tinggi Penelitian through Hibah Dosen Pemula.

## REFERENCES

1. E. Carpio, P. Zúñiga, S. Ponce, J. Solis, J. Rodriguez, W. Estrada, *Journal of Molecular Catalysis A: Chemical*, **228**(2005), DOI: 10.1016/j.molcata.2004.09.066
2. R.J. Tayade, P.K. Surolia, R.G. Kulkarni, R. V. Jasra, *Science and Technology of Advanced Materials*, **8**(2007), DOI: 10.1016/j.stam.2007.05.006
3. B. Liu, X. Wang, G. Cai, L. Wen, Y. Song, X. Zhao, *Journal of Hazardous Materials*, **169**(2009), DOI: 10.1016/j.jhazmat.2009.04.068
4. H. Xu, L. Zhang, *The Journal of Physical Chemistry C*, **113**(2009), DOI: 10.1021/jp8089903
5. A. Ramchiary, S. K. Samdarshi, *Solar Energy Materials and Solar Cells*, **134**(2015), DOI: 10.1016/j.solmat.2014.12.031
6. N.T. Nolan, D.W. Synnott, M.K. Seery, S.J. Hinder, A. Van Wassenhoven, S.C. Pillai, *Journal of Hazardous Materials*, **211,212**(2012), DOI: 10.1016/j.jhazmat.2011.08.074
7. N.R. Mathews, M.A. Cortes Jacome, C. Angeles-Chavez, J.A. Toledo Antonio, *Journal of Materials Science: Materials in Electronics*, **26**(2015), DOI: 10.1007/s10854-014-2294-3
8. M. Bettinelli, V. Dallacasa, D. Falcomer, P. Fornasiero, V. Gombac, T. Montini, *Journal of Hazardous Materials*, **146**(2007), DOI: 10.1016/j.jhazmat.2007.04.053
9. J. Wang, D.N. Tafen, J.P. Lewis, Z. Hong, A. Manivannan, M. Zhi, *Journal of the American Chemical Society*, **131** (2009), DOI: 10.1021/ja903781h
10. T. Ohno, M. Akiyoshi, T. Umebayashi, K. Asai, T. Mitsui, M. Matsumura, *Applied Catalysis A: General*, **265**(2004), DOI: 10.1016/j.apcata.2004.01.007
11. Y. Cong, J. Zhang, F. Chen, M. Anpo, *The Journal of Physical Chemistry C*, **111**(2007), DOI: 10.1021/jp0685030
12. T. Umebayashi, T. Yamaki, H. Itoh, K. Asai, *Applied Physics Letters*, **81**(2002), DOI: 10.1063/1.1493647
13. H. Li, X. Zhang, Y. Huo, J. Zhu, *Environmental Science & Technology*, **41**(2007), DOI: 10.1021/ES062680X
14. I.P. Mahendra, A. Huda, H.M. Ngoc, P.T. Nghia, Tamrin, B. Wirjosentono, *Arab Journal of Basic and Applied Sciences*, **26**(2019), DOI:10.1080/25765299.2019.1610209
15. A. Huda, I.P. Mahendra, R. Ichwani, C.T. Handoko, H.M. Ngoc, B. Yudono, *Rasayan Journal of Chemistry*, **12**(2019), DOI: 10.31788/RJC.2019.1215084
16. H. Tian, J. Ma, K. Li, J. Li, *Ceramics International*, **35**(2009), DOI: 10.1016/j.ceramint.2008.05.003
17. M. Anpo, T. Shima, S. Kodama, Y. Kubokawa, *The Journal of Physical Chemistry*, **91**(1987), DOI: 10.1021/j100300a021
18. T. Kawahara, Y. Konishi, H. Tada, N. Tohge, J. Nishii, S. Ito, *Angewandte Chemie*, **41**(2002), DOI: 10.1002/1521-3773(20020802)41:15<2811::AID-ANIE2811>3.0.CO;2
19. T. Jeyapaul, K. Prakash, S. Harikengaram, A. Chellamani, V. Selvam, *Rasayan Journal of Chemistry*, **11**(2018), DOI: 10.31788/RJC.2018.1143076
20. H. Sutanto, S. Wibowo, Z. Arifin, K.S. Firdausi, E. Hidayanto, Hadiyanto, *Rasayan Journal of Chemistry*, **10**(2017), DOI: 10.7324/rjc.2017.1041793
21. S. Mahshid, M. Askari, M. Sasani Ghamsari, N. Afshar, S. Lahuti, *Journal of Alloys and Compounds*, **478**(2009), DOI: 10.1016/j.jallcom.2008.11.094
22. R.G. Nair, S. Paul, S.K. Samdarshi, *Solar Energy Materials and Solar Cells*, **95**(2011), DOI: 10.1016/j.solmat.2011.02.017.

[RJC-5461/2019]

# PREPARATION OF S-DOPED

## ORIGINALITY REPORT

19%

SIMILARITY INDEX

9%

INTERNET SOURCES

16%

PUBLICATIONS

8%

STUDENT PAPERS

## PRIMARY SOURCES

1

Submitted to Universitas Brawijaya

Student Paper

2%

2

Yuan Lukito, Antonius Rachmat Chrismanto. "Recurrent neural networks model for WiFi-based indoor positioning system", 2017 International Conference on Smart Cities, Automation & Intelligent Computing Systems (ICON-SONICS), 2017

Publication

2%

3

[pubs.rsc.org](https://pubs.rsc.org)

Internet Source

1%

4

Daorong Li, Xia Wang, Dongjin Wan, Suhua Duan, Chang Liu, Kunfeng Zhang, Baoling Fang. " Adsorption of Cu Cations from Aqueous Solution by S-doped TiO ", Separation Science and Technology, 2011

Publication

1%

5

Chairul Amni, Ismet, Sri Aprilia, Mariana, Said Ali Akbar. "MECHANICAL PROPERTIES OF BIOPLASTICS JANENG STARCH (Dioscorea hispida) FILM WITH GLYCEROL AND ZINC

1%



# OXIDE AS REINFORCEMENT", Rasayan Journal of chemistry, 2020

Publication

6

M. Hamadani, A. Reisi-Vanani, A. Majedi. "Preparation and characterization of S-doped TiO<sub>2</sub> nanoparticles, effect of calcination temperature and evaluation of photocatalytic activity", Materials Chemistry and Physics, 2009

Publication

1 %

7

Nur 'Aliaa Razali, Siti Amira Othman. "Sol-gel technique in study of titanium dioxide (TiO<sub>2</sub>) photocatalytic activity- a short review", Materials Today: Proceedings, 2022

Publication

1 %

8

[www.fortunejournals.com](http://www.fortunejournals.com)

Internet Source

1 %

9

Dinda Gusti Ayu, Saharman Gea, Andriyani, Dewi Junita Telaumbanua et al.

"Photocatalytic Degradation of Methylene Blue Using N-Doped ZnO/Carbon Dot (N-ZnO/CD) Nanocomposites Derived from Organic Soybean", ACS Omega, 2023

Publication

1 %

10

Submitted to Universiti Brunei Darussalam

Student Paper

1 %

11

[vdoc.pub](http://vdoc.pub)

Internet Source

1 %

12

[irgu.unigoa.ac.in](http://irgu.unigoa.ac.in)

Internet Source

1 %

13

[www.studystack.com](http://www.studystack.com)

Internet Source

1 %

14

A.R. Khataee, H. Aleboyeh, A. Aleboyeh.  
"Crystallite phase-controlled preparation,  
characterisation and photocatalytic properties  
of titanium dioxide nanoparticles", Journal of  
Experimental Nanoscience, 2009

Publication

1 %

15

Submitted to PEC University of Technology

Student Paper

1 %

16

Tia Okselni, Adlis Santoni, Abdi Dharma, Mai  
Efdi. "BIOLOGICAL ACTIVITY OF METHANOL  
EXTRACT OF *Elaeocarpus mastersii* KING:  
ANTIOXIDANT, ANTIBACTERIAL, AND  $\alpha$ -  
GLUCOSIDASE INHIBITOR", Rasayan Journal of  
Chemistry, 2019

Publication

1 %

17

Todorova, N.. "Structure tailoring of fluorine-  
doped TiO<sub>2</sub> nanostructured powders",  
Materials Science & Engineering B, 20080825

Publication

1 %



18

Changzhi Ai, Pengcheng Xie, Xidong Zhang, Xusheng Zheng, Jin Li, Andreas Kafizas, Shiwei Lin. " Explaining the Enhanced Photoelectrochemical Behavior of Highly Ordered TiO Nanotube Arrays: Anatase/Rutile Phase Junction ", ACS Sustainable Chemistry & Engineering, 2019

Publication

1 %

19

Guidong Yang, Zifeng Yan, Tiancun Xiao. "Low-temperature solvothermal synthesis of visible-light-responsive S-doped TiO<sub>2</sub> nanocrystal", Applied Surface Science, 2012

Publication

1 %

20

Fahad Irfan, Muhammad Usman Tanveer, Muhammad Abdul Moiz, Syed Wilayat Husain, Muhammad Ramzan. "TiO<sub>2</sub> as an effective photocatalyst mechanisms, applications, and dopants: a review", The European Physical Journal B, 2022

Publication

<1 %

21

Wee-Jun Ong, Lling-Lling Tan, Yun Hau Ng, Siek-Ting Yong, Siang-Piao Chai. " Graphitic Carbon Nitride (g-C<sub>3</sub>N<sub>4</sub>)-Based Photocatalysts for Artificial Photosynthesis and Environmental Remediation: Are We a Step Closer To Achieving Sustainability? ", Chemical Reviews, 2016

Publication

<1 %

Ye Cong, Jinlong Zhang, Feng Chen, Masakazu Anpo. " Synthesis and Characterization of Nitrogen-Doped TiO Nanophotocatalyst with High Visible Light Activity ", The Journal of Physical Chemistry C, 2007

Publication

---

<1 %

---

Exclude quotes      On

Exclude matches      Off

Exclude bibliography      On

# PREPARATION OF S-DOPED

---

GRADEMARK REPORT

---

FINAL GRADE

/1000

GENERAL COMMENTS

Instructor

---

PAGE 1

---

PAGE 2

---

PAGE 3

---

PAGE 4

---