

Submission article paper RJC-2018

Heri Sutanto herisutanto@fisika.undip.ac.id Kepada: "RASĀYAN J. Chem." rasayanjournal@gmail.com 12 September 2018 pukul 13.17

Dear Rasayan Journal of Chemistry Editor,

We would like to submit a new manuscript entitled "A Simple Route Synthesis of Polycrystalline TiO₂-Ag by the Sonochemical Method for Photocatalytic Direct Blue71 Degradation" to be considered by the Rasayan Journal of Chemistry for publication. In this paper, we focused on the synthesis of TiO₂ doped with Silver (Ag) using the sonochemical method to be applied as a material capable of degrading the Direct Blue 71 (DB71) liquid waste. Direct Blue 71 itself is a very dangerous waste in the textile industry in Indonesia. The purpose of this research is to synthesis new photo catalyst material as a new potential material for direct blue 71 waste treatment. For the novelty in this research, we have successfully synthesized TiO₂ doped by Ag in the form of powder as an alternative material for photo catalyst. Profitability, the sample in powder form can be directly used to degrade waste from the textile industry, this is because the powder has a very small size and able to provide a chemical process that the photo catalyst process for the better. This has been shown with our results show that the use of TiO₂-Ag in powder form has reduced the Direct Blue 71 solution concentration and has a degradation efficiency of nearly 60%.

We believe that this manuscript is appropriate for publication in the Rasayan Journal of Chemistry. We have no conflicts of interest to disclose. In addition we have checked the plagiarism using Turnitin online software and Enago proofreading, which results we have submitted with this file. If you feel that the manuscript is appropriate for your journal, I am looking forward to hearing from you soon.

Please address all correspondence concerning this manuscript to my email address: herisutanto@fisika.undip.ac.id.

Thank you for considering this manuscript.

Regards,
Dr. Heri Sutanto
Material Physics Laboratory
University of Diponegoro, Indonesia
E-mail: herisutanto@fisika.undip.ac.id

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Revision-1

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RJC- 5034/2018 (Please use this	Sonochemical Method for Photocatalytic Direct Blue71 Degradation		
number in any future correspondence.)	H. Sutanto ^{a,b,*} , I. Marhaendrajaya ^a , G. W. Jaya ^a , E. Hidayanto ^a , M. Mukholit ^a , S. Wibowo ^a , Y. Wahyono ^a		
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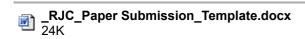
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Acceptance Letter: RJC-5034/2018

Dear Author,

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Your manuscript has been accepted for the publication in the latest issue of RASĀYAN Journal of Chemistry, i.e. RJC, Vol.11, No.4, 2018.

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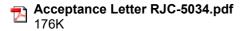
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Thank for the information you have provided. Please let me know in advanced.

Thank you

Regards,

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Dear Dr. Pratima Sharma Managing Editor of Rasayan Journal of Chemistry

Here I send the final revision of our manuscript RJC-5034 as your suggestion. Please let me know if our paper have already published.

With my best regards,

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A SIMPLEROUTE SYNTHESIS OF POLYCRYSTALLINE TiO₂-Ag BY THE SONOCHEMICAL METHOD FOR PHOTOCATALYTIC DIRECT BLUE71 DEGRADATION

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ABSTRACT

In this paper, silver-doped titania (TiO_2 -Ag) powders have been synthesized with Ag variants using a simple sonochemical method. The prepared samples were identified using X-ray diffractometry (XRD) and scanning electron microscopy (SEM) to investigate the crystal structure and morphology, respectively. All samples showed the polycrystalline anatase phase and the plane exhibited the (101) dominant orientation. The smallest crystallite sample size of about 14.9 nm was obtained with a 3% Ag doping (TAg3). At higher Ag concentrations, the metal peaks could be detected at 44.3° and 64.4°. SEM images showed that all samples were agglomerated except the TAg3 sample with the lowest surface area of the active sites. Photocatalytic activity was carried out using Direct Blue 71 (DB71) as an organic pollutant. TAg4 exhibited the highest activity under ultraviolet (UV) irradiation with a degradation rate of 0.01 ppm/min. Results suggest that our TiO_2 -Ag photocatalyst performed well when prepared by a simple sonochemical method.

Keywords: Titania, Ag doping, sonochemical, photocatalytic, direct blue 71.

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INTRODUCTION

Environmental pollution is a global problem today¹. Indonesia faces environmental problems due to water pollution from industries such as Batik wastewater². Water treatment technology is neededto overcome this problem. Recently, the use of photocatalyststreatment for the degradation of organic pollutants has been developed by many researchers³. The commonly used photocatalyst is titanium dioxide or titania (TiO₂). This material is widely used due to its high efficiency, affordability, non-toxicity, inert-properties and biological photo stability^{4,5}. There have been several attempts to improve the ability of TiO₂ materials, e.g., through synthesis of TiO₂-nanocarbon composites.

This method increases in photocatalytic ability, however some problems inhibit the efficiency of TiO₂-nanocarbon composites, such as weakening of light intensity on catalytic surfaces and lack of reproducibility in sample processing and preparation⁶. TiO₂ Nanoparticles coating on the transparent plastic granules is conducted by variedelectrostatic and heating technique for the photocatalytic degradation of organic pollutants in water^{7,8}. The main drawback of TiO₂ materials is their enormous energy band gap which is about 3.2 eV.It can be excited only under ultraviolet (UV) light, which has a wavelength of less than 387.5 nm and represents only 5% of the solar spectrum⁹. Therefore, there have been various attempts at obtaining TiO₂ materialsthat can work under visible and solar light and that can be utilized optimally¹⁰.



In addition, the modification of TiO₂ materials has been carried out to improve the physical properties of semiconductors in terms of electrical, optical and structural properties¹¹. Element-doping onto TiO₂ has drawn the attention of many researchers in photocatalysis field. Some metals such as silver (Ag), aluminum (Al), and cobalt (Co), and nonmetals such as nitrogen (N), sulfur (S), and flour (F) have been used to enhance the photocatalytic performance of TiO₂^{12,13}. Recently, modification of TiO₂ with precious metals has received more attention as the implanted precious metals can reduce recombination of electron-hole pairs, increasing photocatalytic activity. Ag metal is more attractive because of itis nontoxic and affordable ¹⁴⁻¹⁵. TiO₂-Ag can be prepared using several methods such as the sol-gel method ^{16,17}, chemical vapor deposition (CVD)¹⁸, solvothermal synthesis¹⁹, electrodeposition²⁰, hydrothermal synthesis²¹, microwave-assisted synthesis²², spray pyrolysis^{23,24}, and laser deposition²⁵. In this paper, TiO₂-Agwas synthesizedby a simple sonochemical method. This method utilized irradiation with ultrasound waves imposed on a liquid medium. The ultrasonic waves in the liquid medium compressed and stretched the liquid medium structure continuously to form bubbles. These bubbles grew rapidly to their critical size and then erupted. When the bubbles erupted, they acted as hotspots that generated extremes of temperature and pressure²⁶. The raised temperature reached 1000°C, with a pressure of 1000 atm, and a heating-cooling speed of 10 billion/s. The sonochemical method has not been widely used by some researchers to synthesize TiO₂-Ag.

The prepared samples were evaluated by XRD and SEM to check the crystal structure and morphology, respectively. Photocatalytic evaluation was carried out under UV light irradiation using DB71 as an organic pollutant, typically used in Batik industries.

EXPERIMENITAL

TiO₂:Ag was prepared by the sonochemical method as described previously⁴. Titanium (IV) isopropoxide (TTiP) and silver nitrate (AgNO₃) were used as Ti and Ag sources, respectively. Acetone and methanol were used as solvents. 5 ml of TTiP was mixed with 1 ml of acetone and 1 ml of methanol using a magnetic stirrer. AgNO₃was then added to the solution at concentrations of 2%, 3%, 4%, 5%, and 6%, which were denoted as TAg2, TAg3, TAg4, TAg5, and TAg6, respectively. The solution was irradiated with a 300 watts ultrasonic source (Krisbow, Type: 10039597)at a frequency of about 35 kHz at 60°C for 3 h.The dried powders were collected followed by sintering at a temperature of 500°C for 2 hours. The preparation procedure is displayed in Fig.-1. The samples were identified by XRD (Shimadzu XRD-7000) to investigate the crystallinity. The crystallite sizes of TiO₂-Ag samples were calculated using the following Scherrer formula:

$$D = 0.9\lambda/(\beta\cos\theta) \tag{1}$$

Where D is crystallite size (grain size), λ is wavelength of X-rays, β is full width at half maximum (FWHM), and θ is diffraction angle.

Crystal strain wasidentified using the Williamson-Hall (W-H) equation as follows:

$$K = \left(\frac{2\sin\theta}{\lambda}\right) \tag{2}$$

$$K = \left(\frac{2\sin\theta}{\lambda}\right) \tag{2}$$

$$\Delta K = \left(\frac{\beta\cos\theta}{\lambda}\right) \tag{3}$$

The microstructure was investigated by SEM (JEOL 6510-LA). Photocatalytic activity was carried out under aUV-C Lamp at 10 watts ($\lambda = 250 \sim 280$ nm) for 1 hour²⁷. 10 ppm of DB71 was used as an organic pollutant. In order to observe the effect of Ag on TiO₂, bare TiO₂powder was applied for comparison. The absorbance data of DB71 before and after reaction was measured by UV-Vis spectrophotometry (Shimadzu 1240 SA). From these data, the photo-degradation activity was calculated showing the performance of the samples.

RESULTS AND DISCUSSION

XRD Analysis

The crystal structure of samples was analyzed by XRD and Fig.-2 shows the pattern of samples.All samples exhibited the anatase phase of the TiO₂ crystals (JCPDF 211272). The peaks of Ag metal were detected at an angle of 44.3° indicating the (200) plane (JCPDF 040783) and at64.4° indicating the (220) plane (JCPDF 040783). The Ag peaks could be observed only at TAg6, indicating that the metal was formed at higher Ag concentrations. At TAg5 and lower Ag concentration samples, Ag ion might have been incorporated with the TiO₂ crystalline structure. Therefore, the metal peak could not be detected at lower Ag concentrations. The Ag ion might have subtituted Ti or O ion in the crystalline structure, leading to a shift in the XRD peaks.

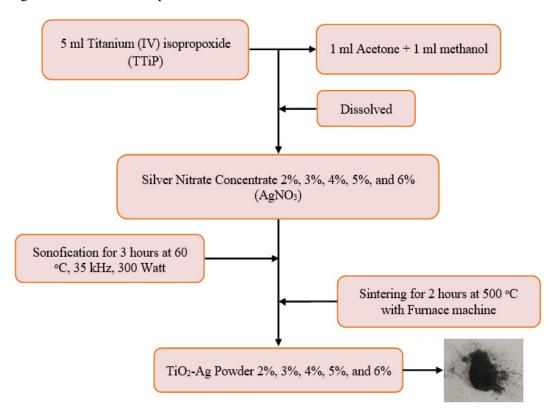


Fig.-1: Flowchart of Sample Preparation.

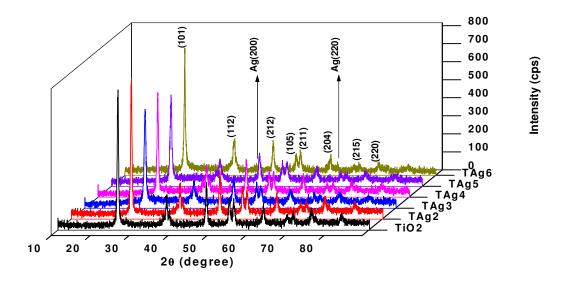


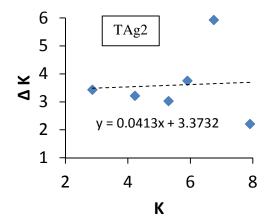
Fig.-2: XRD Characterization of Samples.

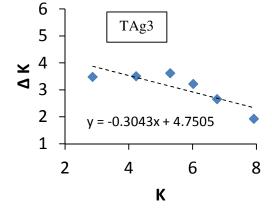
The crystallite size of samples was calculated using the Scherrer formula as presented in Table-1. TAg3 exhibited the smallest crystallite size among the samples, which might have affected their photocatalytic performance. The crystallite size of the sample was inconsistent with concentrations of Ag doping, which might have been caused by substitution and insertion of atoms. The substitution process reduced the crystallite size because Ti atoms (ionic radii of 147 pm) are replaced by Ag atom (ionic radii of 144), while the insertion decreased the crystallite size without any replacement of atoms. The same case in increasing Cu concentrations in the dimension of crystallite size for Cu doped TiO₂ nanoparticle²⁸. Table-1 shows that substitution in TAg3 and TAg5 samples was a dominant process, while insertion was dominant for TAg2, TAg4, and TAg6 samples.

Table-	1.	Grain	Size	of Sa	mnles
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Sample	D (nm)		
TiO_2	$21.4284^{\ 29}$		
TAg2	17.6991		
TAg3	14.9053		
TAg4	20.6694		
TAg5	15.3517		
TAg6	21.8918		

Figure-3 shows the crystal strain from XRD data calculatedusing the Williamson-Hall equation. The value of the crystal strain could be obtained from the line gradient of each sample graph. TAg2 and TAg5 samples exhibited positive crystal lattice strain values, while the others showed negative values. A positive value indicated that the sample had a lattice strength. This was due to the influence of the crystal size, where a smaller crystal size would produce a definite lattice strain, and the larger crystal size would result in a negative lattice strain. In addition, there were several other factors such as the influence of ultrasonic waves and the temperature of the furnace, which possibly affected the value of the crystal lattice strain. The strain values are various disorder, because of the increment in amount of unit cell per particle as a substitution of Ag1+, Ag2+ and Ag3+ ions in Ti site. This results is similar to photocatalytic performance of Cu-doped TiO2 nanoparticles30.





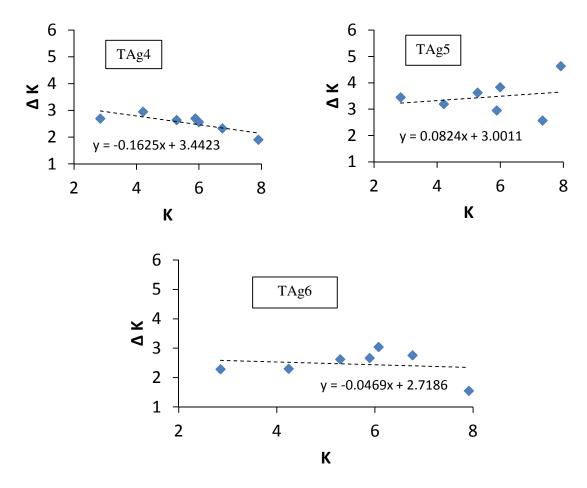


Fig.-3: The W-H plot of **K** versus **ΔK** for TiO₂-Ag.

The morphology of TiO₂-Agcan be seenby the SEM images as shown in Fig.-4. From these images, the morphology of the photocatalysts are shown to have been in accordance with crystallite size from XRD data. TAg4 and TAg6 samples had large lumps on the surface. This was due to agglomeration as a result of the Ag atom insertion. For the TAg5 sample, little lumps were found on the surface and resulted in a more substantial crystal size than in the TAg3. For the TAg2 and TAg3 samples, the lumps were found to be less dense as in the TAg4 and TAg6 samples.

Photocatalytic Activity

Figure-5 shows the degradation of DB71 concentration during the photocatalytic reaction of TiO₂-Ag samples.DB71 concentration decreased after being degraded by TiO₂-Ag powders for60 min. It was noted that, under dark conditions and UV light irradiation, the performance of bare TiO₂ was almost the same and quite low. This result indicated that Ag plays important role in enhancing the photo-degradation of DB71. Inserting Ag to TiO₂, Ag²⁺ ion should replace Ti⁴⁺ in TiO₂ lattice so that O-Ti-O turns to Ag-Ogenerate increasing of oxygen vacancies. The enhancement adsorbed oxygen on the surface will stabilize equilibrium oxygen vacancies in TiO₂ lattices³⁰. We also checked the reaction under dark conditions, which showed no activity. TAg4showed the highest activity in reducing concentrations of DB71,which decreased by up to 51.28% after 60 min. Figure-6 shows the degradation rate of DB71 by prepared samples. TAg4 also exhibited the fastest rate of about 0.01 ppm/min.IncreasingAg atom in TiO₂ will decrease crystallite size of TiO₂-Ag, so that surfacearea of TiO₂-Ag particle will increase leads to grow up the number of active sites of photocatalyst for adsorption. This result correspond to Fig.-5b.

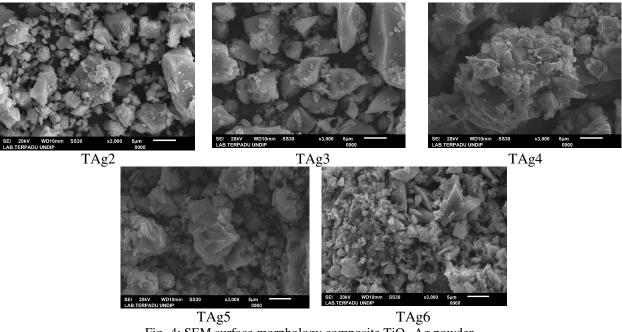


Fig.-4: SEM surface morphology composite TiO₂-Ag powder.

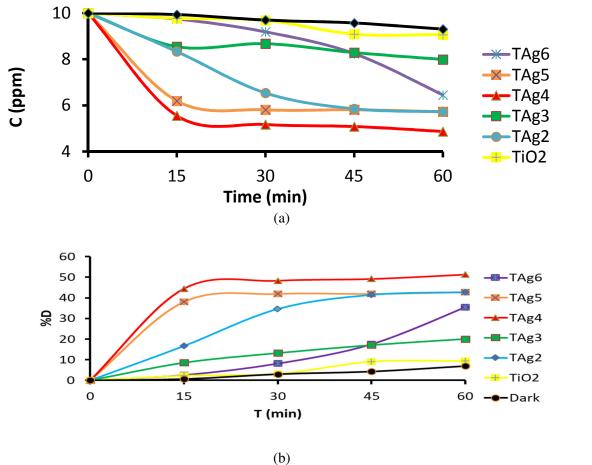


Fig.-5: (a) The decrease of DB71 concentration value every 15 minutes, and (b) Photo-degradation of DB71 by TiO₂-Ag samples under UV light irradiation.

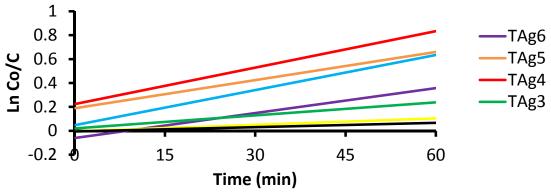


Fig.-6: The degradation rate of DB71.

CONCLUSION

The TiO₂:Ag powder with various concentration of Ag was successfully preparedby a simple sonochemical method. XRD results showed that all samples had an anatase polycrystalline structure phase with a dominant orientation plane of (101). The SEM images displayed a clumped morphologyof the samples, except for the TAg3 sample as a result of the agglomeration of Ag particles. The photocatalytic tests showed that Ag-doped samples exhibit superior performance compare with bare TiO₂ powder, and that TAg4 performed best, with DB71 a degradation percentage of 51.28% and a degradation rate of 0.01 ppm/min.

ACKNOWLEDGEMENT

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