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Jun 26, 2022	Author Submits Revision Confirmation	Heri Sutanto	1
Jun 26, 2022	PDF Built and Requires Approval	Heri Sutanto	1
Jun 24, 2022	Author Revision Reminder - Before Due Date	Heri Sutanto	0
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Heri Sutanto <herisutanto@live.undip.ac.id>

Ref.: Ms. No. RINENG-D-22-00427

Optimization of The Bi2O3/Cu Synthesis Process Using Response Surface Methodology as a Tetracycline Photodegradation Agent Results in Engineering

Dear Prof. Dr. Sutanto,

Reviewers have now commented on your paper. You will see that they are advising that you revise your manuscript. If you are prepared to undertake the work required, I would be pleased to reconsider my decision.

For your guidance, reviewers' comments are appended below.

If you decide to revise the work, please submit a list of changes or a rebuttal against each point which is being raised when you submit the revised manuscript.

Please resubmit your revised manuscript by Jun 27, 2022.

To submit a revision, go to https://www.editorialmanager.com/rineng/ and log in as an Author. You will see a menu item call Submission Needing Revision. You will find your submission record there.

Yours sincerely

Daniel M Mulvihill, D.Phil. Associate Editor Results in Engineering

Comments from the Editors and Reviewers:

Your article would appear to be of interest to a wide engineering research community and in order to promote its visibility even more, may we recommend that you view the past published articles in Results in Engineering and if you find any relevant publications, CITE the article from this Journal.

Authors are invited to submit a revised manuscript and response to the reviewers

Reviewer 2: 1. Some equipments used need to be equipped with specifications: brand, power (for microwave)

2. Please write method in detail, especially on the material synthesis procedure. How much copper nitrate was added, what

concentration of nitric acid was used? the synthesis time applied and how much microwave power was used 3 Results and Discussion

3. Results and Discussion

a. Will all variations produce the same appearance? Author only provide 1 samples in Figure 3

b. Autor used the Debye-Scherrer equation to calculate the particle size. Some findings explain that this equation only determine the particle size if the particle size of the product/sample is in nanosize. In this study, based on SEM results, the particle size is micrometer

c. Why only 1 sample (R19) was characterized by several instruments such as xrd, ftir, drs-uv, sem-edx?

d. In Table 2, where did the degradation efficiency data for the actual data come from? Is it from lab experiment? If so, the author did not explain in the experimental method.

e. In table 2, the author claims that the data between the actual and predicted degradation efficiency values do not show a significant difference. This difference should have a deviation limit, for example ± 4 or ± 3 accompanied by a reference that the value of the difference is accepted.

f. The author chooses the quadratic model because the R square value is close to 1. Table 3 shows that the R square value is 0.8985. This value is far from 1. Normally, the R2 value being accepted is at least 0.98. Thus, the statistical data is less valid.

g. Table 4, definition of A squared, B squared, C squared, lack of fit and pure error must be explained

h. The XRD data in Figure 7 need to be clarified, which peaks are Cu and which ones are Bi2O3. It would be better if a standard Bi2O3 and garhaditte difactograms are provided so that it would be easier to compare with the diffractogram of the product
i. Figure 8. Determination of the band gap seems inaccurate. The band gap value seems to be around 2.2 eV. It is necessary to

show the R squared value from the selected curve range (eg 2.2-2.6) j. The author claims that the band gap value is getting lower after doping with Cu. In fact, the value of 2.63 eV is a value in the range of 2.58-2.85 eV. Please explain !

k. Figure 9. Can you provide SEM images at 500X magnification? Figure 9 shows very large Bi2O3/Cu particles and this image contrasts with the particle size calculations using the Debye-Schererr equation. How can you be sure that the spherical particles

are Cu particles and not Bi2O3/Cu?

I. The mechanism of degradation reactions using photocatalysts presented is a common degradation mechanism and has been discussed in many journals on the degradation of dye molecules by photocatalysts. The mechanism of tetracycline with Bi2O3/Cu should be well presented/proposed in this paper

Reviewer 3: The Bi2O3/Cu photocatalyst was synthesized for the degradation study of tetracycline (TC) wastewater. In general, the experiments were well designed by varying the copper concentration, power and synthesis time factors, and the synthesized materials was analyzed and characterized, which proved its application in the removal of refractory pollutants from wastewater. However, the main problem is the inadequate experimental characterization and analysis and the lack of analysis of the TC degradation mechanism, which is necessary for the photocatalytic performance by varying the copper concentration, power and synthesis time. In addition, the implications of the application of this synthetic material need to be clarified. The specific comments are as follows:

1. The introduction section is too lengthy in terms of antibiotics and contamination and unnecessarily overstated in relation to the present study. The basis of material selection is insufficient. The importance of the crystal phase structure of Bi2O3 is mentioned in the material selection, but the correlation with this study is not developed, and the review and comparison with other existing research progress of Bi2O3 photocatalysts are lacking.

2.Lack of supplementation of nitric acid and sodium hydroxide solution concentrations in the material system, please supplement the amount of catalyst injected during the degradation of tetracycline.

3.Please provide the effect of dark adsorption of different photocatalyst composites on tetracycline to reveal the role of adsorption capacity on photocatalysis.

4.Please give the design basis for 20 samples. R3、R4、R8、R9、R11、R12 samples with the same control of influence factors, please explain why six sets of parallel experiments were set up? And the photocatalytic efficiency of these six samples changed from 41.6 to 58.91%, please explain the reason for the change.

5. The impact factor C parameter of the R14 sample is wrong, please verify. Please represent the TC degradation efficiency data of Bi2O3/Cu samples by graphs for different factors in Table 2, and the optimization of the influencing factors is recommended to be studied by the control variable method.

6. The photodegradation efficiency of the best R19 sample is 61.09%, which is not significant compared to other photocatalytic studies. Please compare the photocatalytic activities of photocatalysts with similar catalyst systems and explain the research value of this manuscript.

7.Please indicate the diffraction peaks corresponding to Bi2O3 and Cu in the composite sample separately in the XRD analysis and supplement the XRD pattern of the single sample for comparison to prove the successful synthesis of the composite. 8.More solid evidence should be provided to demonstrate the presence of metallic Cu rather than Cu ions in Bi2O3/Cu photocatalysts, such as comparison of XPS spectra of monomer and composite samples.

9.Supplement the UV-Vis spectra of Bi2O3 and Bi2O3/Cu samples, indicating that adding Cu metal can reduce the energy band gap and increase visible light absorption.

10. The discussion of the photocatalytic mechanism is not robust and not supported by literature. Please add active species test experiments. In the mechanism discussion, the comparison between the energy level structure of the catalyst and the redox potential of active radicals should be proposed to explain the role of active species, which corresponds to the mechanism of photocatalytic degradation of tetracycline wastewater.

11. There are many colloquial expressions and grammatical errors in the manuscript, the grammar editing needs to be improved, and the reference format needs to be adjusted.

Reviewer 5: The manuscript proposed the synthesis of Bi2O3 and Cu-based materials with a microwave method. The powders obtained were used as photocatalysts to degrade tetracycline in an aqueous medium. The authors present some results of the characterization of the materials and the photocatalytic activity utilizing a design of experiments. In general, I don't recommend its publication in Results in Engineering Journal since the highlight of this work is not clear and the results provided are not new. Instead, I kindly suggest that the authors consider the following comments to improve the quality of the manuscript:

Abstract. Include the formula of Garhadite since it is unclear the meaning of the sentence.

Highlights. Revise the English grammar since there are some mistakes. Also, the third highlight is not clear. Introduction.

Ref. 30 does not include the study of different Bi-photocatalyst. Change the meaning of the sentence or change the reference. What is the objective to reduce the band gap of Bi2O3 after the Cu incorporation? since the Bi2O3 is a visible-light active. Most importantly, how are the energy bands changed after copper's incorporation in Bi2O3?

It is not why the authors selected the factors for the design of experiments.

4. Experimental methodology. It is not clear why the authors add 0.5 g of copper nitrate in moles, which are about three times higher than the Bi-salt. This quantity did not favor a doping structure, just a mixture of phases. Revise the experimental methodology carefully. Also, include why the authors chose 1g TC in 1 L of distilled water. 5. Results.

XRD. It is not clear the analysis of Figure 7 since the JCPDS cards are not shown in the Figure. Also, the formula of the copper formed is not clear. Also, the authors specified that they found 28.1% of "garhadite", which is not a doping amount. Figure 8. Include the reference spectrum of Bi2O3.

Figure 9. More images are required for at least all of the design of experiments.

The efficiencies reported are not promising. Please revise: 1) Ecotoxicology and Environmental Safety Volume 210, 1 March 2021, 111866. 2) Scientifc Reports | (2021) 11:85 3) Separation and Purification Technology Volume 237, 15 April 2020, 116365.

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2. We set table (5) on page 8 and table (7) is on page 13

We highlighted the manuscript revision using yellow color.

Regards,

Heri Sutanto

Mon, Jul 4, 2022 at 7:47 AM

Heri Sutanto Physics Department Diponegoro University Tembalang Semarang Indonesia E-mail: herisutanto@live.undip.ac.id Mobile Phone : +6285325202360

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