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Manuscript Title: OAEN - (Efficient degradation of amoxicillin using Bi₂O₃/Fe synthesized by microwave-assisted precipitation method)

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Efficient degradation of amoxicillin using Bi₂O₃/Fe synthesized by microwave-assisted precipitation method --Manuscript Draft--

Full Title:	Efficient degradation of amoxicillin using Bi ₂ O ₃ /Fe synthesized by microwave-assisted precipitation method
Manuscript Number:	COGENTENG-2021-0443
Article Type:	Research Article
Keywords:	Bi ₂ O ₃ /Fe; Microwave Irradiation; Photodegradation; Amoxicillin
Manuscript Classifications:	10.7.1.13 Physical Chemistry; 10.7.1.6 Environmental Chemistry; 10.7.3.4 Composites
Abstract:	<p>Bi₂O₃/Fe material has been successfully synthesized using the precipitation method assisted by microwave radiation. This study aimed to analyze the effect of Fe on characteristics of Bi₂O₃ and to analyze its ability to photodegrade amoxicillin. The addition of Fe was carried out with various concentrations, namely 0%, 1%, 3%, 5%, 7% and 9%. The results of XRD characterization showed that the diffraction peaks were at $2\theta = 24.54^\circ, 25.75^\circ, 26.91^\circ, 27.38^\circ, 27.99^\circ, 32.30^\circ, 33.08^\circ, 35.04^\circ, 37.60^\circ, 39.75^\circ, 41.79^\circ, 43.77^\circ, 45.62^\circ, 46.31^\circ, 48.54^\circ, 52.62^\circ, 54.26^\circ, 55.88^\circ, \text{ and } 62.05^\circ$ with Miller indices (102), (002), (112), (121), (012), (211), (202), (212), (113), (222), (213), (040), (231), (223), (104), (004), (241), (323) and (330). The resulting crystal sizes were 25,702 nm, 27,161 nm, 29,111 nm, 23,751 nm, 24,046 nm and 21,767 nm. The photocatalytic testing of amoxicillin showed that the absorbance value of the sample decreased as the duration of degradation increased. Bi₂O₃/Fe 3% showed the most significant degradation efficiency of amoxicillin at 76.34% at a rate of 0.0079 ppm/min.</p>

Efficient degradation of amoxicillin using Bi₂O₃/Fe synthesized by microwave-assisted precipitation method

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¹Department of Physics, Faculty of Science and Mathematics, Diponegoro University, Semarang-Indonesia

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Abstract

Bi₂O₃/Fe material has been successfully synthesized using the precipitation method assisted by microwave radiation. This study aimed to analyze the effect of Fe on characteristics of Bi₂O₃ and to analyze its ability to photodegrade amoxicillin. The addition of Fe was carried out with various concentrations, namely 0%, 1%, 3%, 5%, 7% and 9%. The results of XRD characterization showed that the diffraction peaks were at $2\theta = 24.54^\circ, 25.75^\circ, 26.91^\circ, 27.38^\circ, 27.99^\circ, 32.30^\circ, 33.08^\circ, 35.04^\circ, 37.60^\circ, 39.75^\circ, 41.79^\circ, 43.77^\circ, 45.62^\circ, 46.31^\circ, 48.54^\circ, 52.62^\circ, 54.26^\circ, 55.88^\circ, \text{ and } 62.05^\circ$ with Miller indices (102), (002), (112), (121), (012), (211), (202), (212), (113), (222), (213), (040), (231), (223), (104), (004), (241), (323) and (330). The resulting crystal sizes were 25,702 nm, 27,161 nm, 29,111 nm, 23,751 nm, 24,046 nm and 21,767 nm. The photocatalytic testing of amoxicillin showed that the absorbance value of the sample decreased as the duration of degradation increased. Bi₂O₃/Fe 3% showed the most significant degradation efficiency of amoxicillin at 76.34% at a rate of 0.0079 ppm/min.

Keywords: Bi₂O₃/Fe, Microwave Irradiation, Photodegradation, Amoxicillin

Introduction

In recent years, environmental pollution from liquid medical waste has become increasingly widespread. It is a challenge for researchers and the pharmaceutical industry because drugs produce medical waste that can threaten ecosystems and human health (Wang et al., 2018) (Wang X et al., 2019). Amoxicillin (AMX) is the most widely used type of antibiotic because of its antibacterial properties [2] (Jung et al., 2012). AMX has low biodegradability and remarkable stability with many complex aromatics in its molecule, making it difficult to destroy by conventional methods [3] (Zhang, F et al., 2019). Therefore we need the proper technique to degrade AMX efficiently.

Several alternatives have been carried out to remove these compounds, including reverse osmosis, adsorption, and advanced oxidation technologies such as Fenton reaction, ozonation, and photocatalytic technology (Benitez et al., 2011). Photocatalytic shows were promising prospects among these methods due to their sustainability and environmental friendliness (Wang et al., 2018). Bismuth oxide (Bi₂O₃) is a photocatalyst material that is of interest to researchers because of its high redox inversion, environmental friendliness, thermal stability, and energy bandgap between 2.58-2.85 eV, which shows a response to visible light [6] (Chen, 2003). T et al., 2018). However, the photon-induced electron-hole pair has poor light utilization and a fast recombination rate, limiting photocatalytic activity [7] (Zhou et al., 2019).

In this research, Bi₂O₃ semiconductors will be inserted with Fe material using the microwave-assisted precipitation method. This experiment is believed to increase the electron-hole pair separation rate by acting as a shallow trap and reducing the electron-hole pair recombination rate in Bi₂O₃ materials [8] (Zhu et al., 2011). Finally, the authors hope to be able to produce innovative materials that are effective in degrading AMX.

Method

Synthesis of Bi₂O₃/Fe

The research materials used were Bi(NO₃)₃·5H₂O, Fe(NO₃)₃·9H₂O, HNO₃, NaOH, Aquades and Amoxicillin. The equipment used is a hot plate, magnetic stirrer, microwave, beaker, measuring cup, cup,

spatula, digital scale, glass bottle, and dropper. Bi₂O₃/Fe was synthesized using the precipitation method. The synthesis process of Bi₂O₃/Fe is shown in Figure 1. Bi(NO₃)₃·5H₂O 0.5 g and Fe are dissolved in 50 ml of 5% HNO₃ solution, stirred for 10 minutes. A total of 250 ml of NaOH was added to the solution and stirred for 2 hours, then allowed to stand to produce a precipitate. The precipitate was separated and heated on a hotplate for 2 hours at 120°C to produce Bi₂O₃/Fe powder. The resulting Bi₂O₃/Fe powder was then exposed to 100 Watt microwave power for 3 hours.

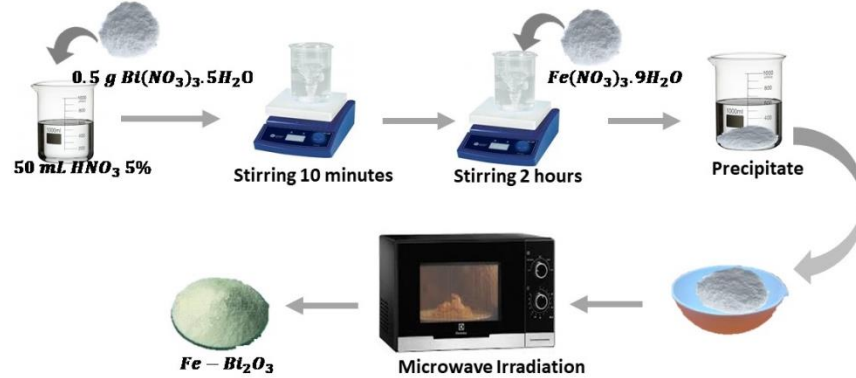


Figure 1. The synthesis process of Bi₂O₃/Fe

Characterization

Structural analysis of Bi₂O₃/Fe powder was carried out using Shimadzu XRD 6100/7000 with a wavelength used is 1.54016. Data retrieval is carried out every 0.02° angle range. The particle size of Bi₂O₃/Fe was calculated using the Debbye-Scherrer equation (Xie et al., 2019).

$$Ds = \frac{k \lambda}{\beta \cos \theta} \quad (1)$$

Where Ds is the crystal size (nm), =1.5406 is the wavelength of X-rays, =0.9 is the Scherrer constant, is the Full Width and Half Maximum or FWHM (in radians) and is the Bragg angle of diffraction or peak position (in radians)

Amoxicillin degradation

The degradation was carried out by adding 0.02 g of Bi₂O₃/Fe powder into 50 ml of 10 ppm Amoxicillin solution under visible light for 180 minutes. Photocatalyst efficiency (Ef) is calculated using the following equation (Sudrajat, H. et. al., 2018).

$$Ef (\%) = \left(1 - \frac{C_t}{C_0} \right) \times 100\% \quad (2)$$

where C₀ and C_t are the initial and final concentrations of the dye solution.

Result and Discussion

Bi₂O₃/Fe has been synthesized using a microwave-assisted precipitation method. The powder shows a yellow physical colour that shifts brown with increasing Fe concentration, as shown in Figure 2. To confirm the success of Bi₂O₃/Fe synthesis, the first thing to do is test the crystallinity. Based on the XRD Bi₂O₃/Fe pattern as shown in Figure 3, the resulting diffraction peaks correspond to JCPDS No. 27-0053 (Devi K R S et al 2019).The main diffraction peaks were at 2θ = 24.54°, 25.75°, 26.91°, 27.38°, 27.99°, 32.30°, 33.08°, 35.04°, 37.60°, 39.75°, 41.79°, 43.77°, 45.62°, 46.31°, 48.54 °, 52.62°, 54.26°, 55.88°, and 62.05° with miller index (102), (002), (112), (121), (012), (211), (202), (212), (113), (222), (213), (040), (231), (223), (104), (004), (241), (323) and (330).

The synthesized Bi₂O₃ material is in the α-phase. These results indicate that the Bi₂O₃ material has been completely formed and has an excellent level of stability at low temperatures (Chen, R. et al., 2011). The increase in Fe concentration caused a difference in the intensity of the XRD results but did not result in a significant change in the lattice structure. Based on the Debbye-Scherrer equation, the crystal

size of Bi_2O_3 with the addition of Fe from 0-9% respectively was 25.702 nm, 27.161 nm, 29.111 nm, 23.751 nm, 24.046 nm, and 21.767 nm. The addition of Fe resulted in differences in the size of the resulting crystals. The addition of 3% Fe produces the largest crystal size.

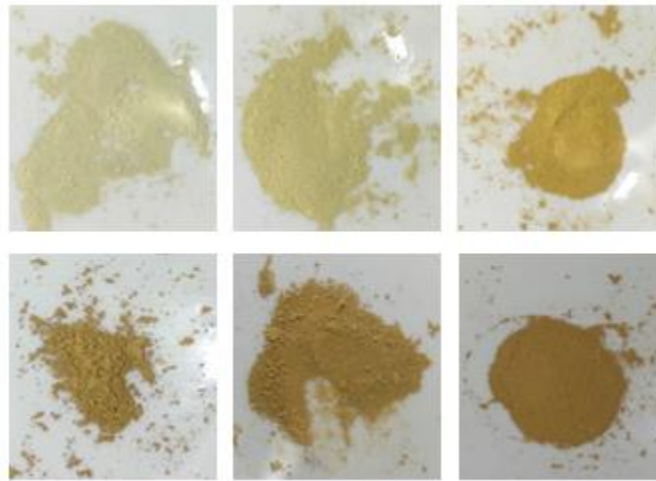


Figure 2. Physical appearance of the sample $\text{Bi}_2\text{O}_3/\text{Fe}$ (a) 0% (b) 1% (c) 3% (d) 5% (e) 7% (f) 9%

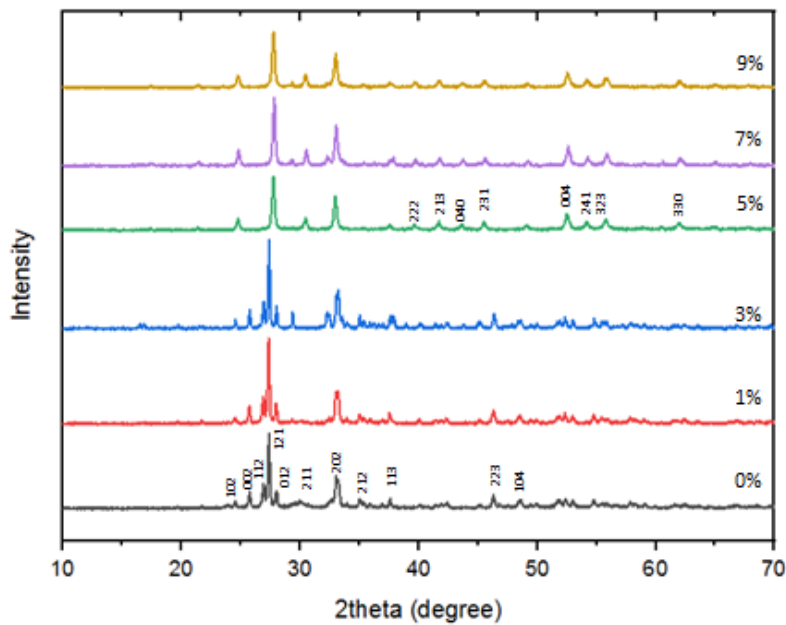


Figure 3. Results of XRD $\text{Bi}_2\text{O}_3/\text{Fe}$ 0-9% characterization

Figure 4 shows that the absorbance value of AMX decreases with the length of time of degradation. The decrease in AMX absorbance is inversely proportional to the degradation efficiency, where the lower the final absorbance, the more effective the degradation. Based on Figure 4, the degradation efficiency of $\text{Bi}_2\text{O}_3/\text{Fe}$ 0-9% material is 72.04%, 65.59%, 59.14%, 59.14%, and 58.06%, respectively. $\text{Bi}_2\text{O}_3/\text{Fe}$ 3% showed the most significant degradation efficiency, but more than 3% Fe doping showed a decrease in efficiency. These results indicate that adding Fe up to a concentration limit of 3% in Bi_2O_3 will increase the photocatalytic activity, while the addition of more than 3% will decrease photocatalytic activity. These results are consistent with previous research by Liang, J. et al. (2014). Photocatalytic activity and degradation efficiency can be related to the crystal size of $\text{Bi}_2\text{O}_3/\text{Fe}$.

(Nandiyanto, Zaen and Oktiani, 2020), where the larger the crystal size of a material, the greater the photocatalytic ability (Sharma, Vashishtha and Shah, 2014; Armaković et al., 2019).

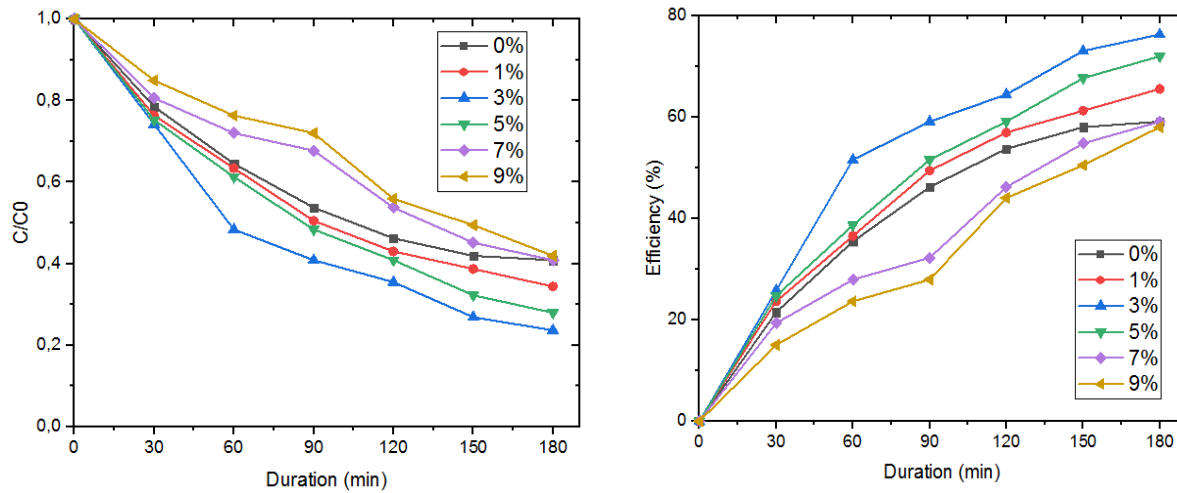


Figure 4. (a) Graph of the decrease in AMX absorbance (b) Degradation efficiency of Bi₂O₃/Fe

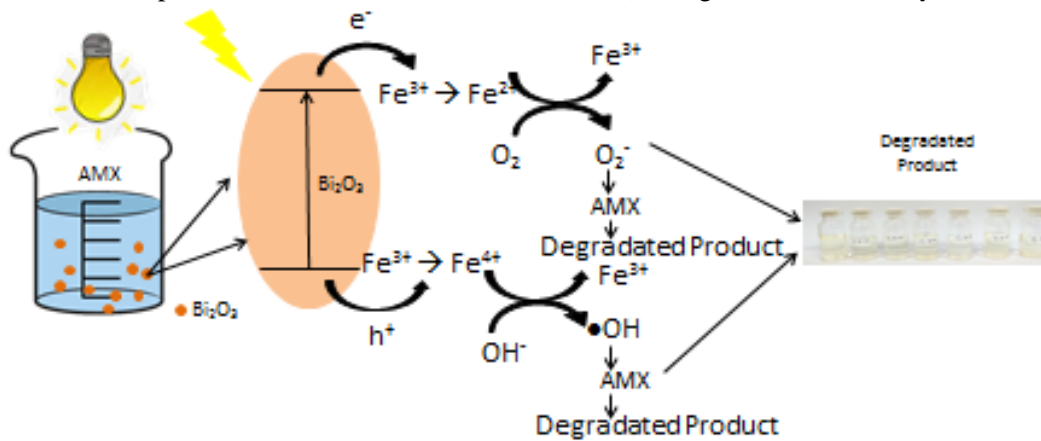


Figure 5. AMX degradation mechanism by Bi₂O₃/Fe

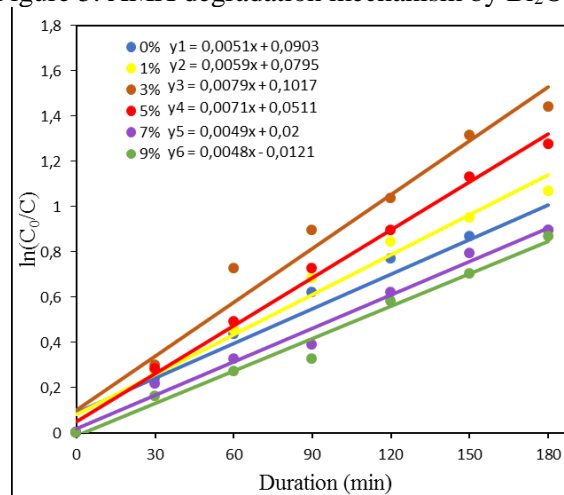
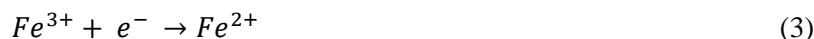


Figure 6. The degradation rate of Bi₂O₃/Fe

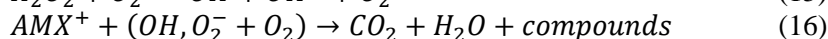
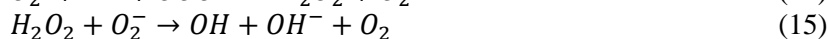
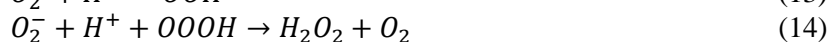
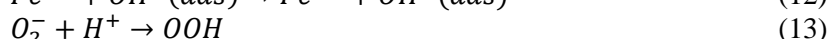
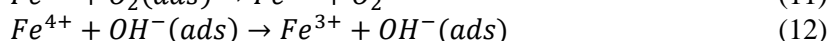
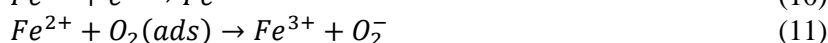
Chemically, the degradation of AMX by Bi₂O₃/Fe can be explained as shown in figure 5. Fe³⁺ ions act as photo-generated between holes and electron transfer, which can increase the lifetime of electrons and holes and reduce the rate of recombination of e⁻/h⁺ pairs based on the reaction as following:



Fe^{2+} and Fe^{4+} ions are relatively unstable ions when compared to Fe^{3+} ions. Therefore, the trapped charge can be easily released from the Fe^{2+} ion or Fe^{4+} ion and then migrate to the surface to start the photocatalytic reaction with the following reaction



Fe^{2+} ions can be oxidized to Fe^{3+} ions by transferring electrons to O_2 adsorbed on the surface of Bi_2O_3 . Meanwhile, the adsorbed O_2 is reduced to O_2^{-} (Eq.5), further degrading AMX. Likewise, Fe^{4+} ions are reduced to Fe^{3+} ions by losing electrons, while the surface hydroxyl groups become hydroxyl radicals (Eq. 6). Consequently, the corresponding Fe^{3+} ion is responsible for reducing the electron-hole recombination rate and favours the enhancement of photocatalytic activity. The photocatalytic mechanism can be explained through the following equation:



The excess of Fe^{3+} ions is entering the cluster formation. This cluster can resist AMX photodegradation by masking the active site from the Bi_2O_3 surface. These results also explain that the Bi_2O_3/Fe 3% has the fastest degradation rate of 0.0079 ppm/minute, while Bi_2O_3/Fe 9% has the slowest degradation rate of 0.0048 ppm/minute. The photocatalytic activity of Bi_2O_3/Fe is higher than that of pure Bi_2O_3 due to the synergistic effect of Fe^{3+} and Bi_2O_3 ions. The rate of degradation describes the speed of the material in degrading waste. The higher the value of the rate of degradation, the faster the material in degrading waste, so that there is less waste contained in the material.

Conclusion

The synthesis of Bi_2O_3/Fe has been successfully carried out using the microwave-assisted precipitation method. Bi_2O_3/Fe 3% has the most apparent crystal size, the fastest degradation rate, and optimal degradation efficiency. The addition of Fe to Bi_2O_3 material does not change the crystal structure, but a concentration of more than 3% will decrease its catalytic activity.

Acknowledgment

We would like to thank the Ministry of Research, Technology, and Higher Education – Indonesian for providing research funding support in 2021 with contract no: 187-24/UN7.6.1/PP/2021.

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Cogent Engineering

Comments from the Editors and Reviewers:

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Reviewer 1: Yes

Title, Abstract and Introduction - overall evaluation
Reviewer 1: Sound

Methodology / Materials and Methods – overall evaluation
Reviewer 1: Unsound or fundamentally flawed

Objective / Hypothesis – overall evaluation
Reviewer 1: Not applicable

Figures and Tables – overall evaluation
Reviewer 1: Sound

Results / Data Analysis – overall evaluation
Reviewer 1: Unsound or fundamentally flawed

Interpretation / Discussion – overall evaluation
Reviewer 1: Unsound or fundamentally flawed

Conclusions – overall evaluation
Reviewer 1: Unsound or fundamentally flawed

References – overall evaluation
Reviewer 1: Sound

Compliance with Ethical Standards – overall evaluation
Reviewer 1: Sound

Writing – overall evaluation
Reviewer 1: Unsound or fundamentally flawed

Supplemental Information and Data – overall evaluation
Reviewer 1: Not applicable

Comments to the author

Reviewer 1: This work presents the synthesis and characterization of Fe doped Bi₂O₃ materials, prepared by the microwave assisted precipitation method. The characterization of the materials is poor, and the photocatalytic performance is not especially high. Due to the lack of novelty of this report, along with the numerous writing issues, I consider it is not suitable for publication in this journal. Some suggestions are provided below.

The abstract is not clear, presenting results very specific of the material characterization, as the Miller indexes, and the crystallite size. Also, some parts are hard to understand. Please revise the units, the reaction constants are expressed in units of min⁻¹.

In the Introduction section. Bi₂O₃ is not thermally stable, actually it reduces from Bi⁺³ to Bi⁰ at temperatures higher than 300°C.

In the Introduction section. Please explain the term “shallow trap”.

In the “Synthesis of the Materials” section. It is not necessary to describe all the materials used in the synthesis, like spatula, hot plate and so on.

Please be more specific at describing the synthesis method. For example, provide information on the concentration of NaOH solution, the amount of Fe precursor used in the synthesis of the doped materials.

The characterization described in section 2 is scarce. Authors should include the determination of the band gap energy, the actual concentration of Fe for the different materials, microscopy images, among others.

Please provide more information about the degradation tests. What kind of light source did authors use? what method was used for the determination of the target molecule? how were the water samples taken and treated?

In the results and discussion section. Please develop a deeper analysis of the XRD results. Did iron doping resulted in the displacement of the diffraction patterns of Bi₂O₃? This would be a prove of the inclusion of Fe atoms within the crystalline structure of the oxide.

Please explain the changes in the XRD patterns observed for the materials doped with Fe loading higher than 5%. Is the loading of Fe presented as a percentage in weight (% wt.) or atomic (% at.)?

The author’s claim “The decrease in AMX absorbance is inversely proportional to the degradation efficiency” is incorrect. Please correct.

Please use correct terms. The phrase “but more than 3% Fe doping showed a decrease inefficiency” is grammatically incorrect.

Indeed, the photocatalytic activity of the semiconductors can be related to the crystallite size, but also to other characteristics that authors did not assess, as the specific surface area.

Authors claim that the Fe⁺⁴ ions within the crystalline structure of the doped materials produce •OH radicals, therefore it is necessary to provide the oxidation potential of the F⁺⁴ – Fe⁺³ pair, and evaluate the possibility of oxidizing the OH⁻ species into •OH.

Conclusions are a summary of the results. Please provide significant conclusions.

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Reviewer 2: Yes

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Reviewer 2: Sound with minor or moderate revisions

Methodology / Materials and Methods – overall evaluation

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References – overall evaluation

Reviewer 2: Sound

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Reviewer 2: Sound

Writing – overall evaluation

Reviewer 2: Sound with minor or moderate revisions

Supplemental Information and Data – overall evaluation

Reviewer 2: Sound

Comments to the author

Reviewer 2: Dear Author

I read your manuscript and I found you resent a good idea

Besides, I write some commutes in the PDF text need to correct. I hope to see these comments

Many thanks,

Reviewer

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Reviewer 5: Sound

Writing – overall evaluation
Reviewer 5: Sound with minor or moderate revisions

Supplemental Information and Data – overall evaluation
Reviewer 5: Not applicable

Comments to the author
Reviewer 5: Dear Editor

The authors have studied “Efficient degradation of amoxicillin using Bi₂O₃/Fe synthesized by microwave-assisted precipitation method”. I have read the manuscript carefully, This study try to reach the efficient photocatalytic elimination of amoxicillin by synthesis of Bi₂O₃/Fe using the microwave-assisted precipitation method. The results shows nanoparticle well synthesized with most apparent crystal size, the fastest degradation rate, and optimal degradation efficiency. However, the manuscript has several flaws that need to be rectified before consideration for publication.

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Reviewer 6: Yes

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Reviewer 6: Sound with minor or moderate revisions

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Reviewer 6: Sound

Writing – overall evaluation
Reviewer 6: Sound with minor or moderate revisions

Supplemental Information and Data – overall evaluation
Reviewer 6: Sound

Comments to the author

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1. How to make sure the Fe(III) ion existing on the Bi₂O₃/Fe surface? XPS was suggested.
2. What is the purpose of microwave irradiation in the preparation procedure?
3. What is the morphology of the prepared composites? TEM/SEM was suggested.
4. How about the Fe(III) distribution on the Bi₂O₃ surface? EDS-mapping was suggested.
5. How about the effective surface area of the prepared composites? BET was suggested.
6. How about energy band gap for the prepared composites?

7. How about recycling used ability for the prepared composites?
8. The possible mechanism was proposed. Please prove the reactive species by using EPR or scavenger test.
9. How about degradation AMX in the environmental water sample under solar light irradiation?
10. How about TOC analysis of AMX by using Bi₂O₃/Fe 3% composites?

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Reviewer 7: Yes

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Reviewer 7: Sound

Methodology / Materials and Methods – overall evaluation

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Results / Data Analysis – overall evaluation

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Interpretation / Discussion – overall evaluation

Reviewer 7: Unsound or fundamentally flawed

Conclusions – overall evaluation

Reviewer 7: Unsound or fundamentally flawed

References – overall evaluation

Reviewer 7: Sound

Compliance with Ethical Standards – overall evaluation

Reviewer 7: Sound

Writing – overall evaluation

Reviewer 7: Unsound or fundamentally flawed

Supplemental Information and Data – overall evaluation

Reviewer 7: Not applicable


Comments to the author

Reviewer 7: The aim of the paper was the study the degradation of amoxicillin using Bi₂O₃/Fe. Although the paper is written in a good English, I donnot think it is suitable to be published in a scientific journal paper. To my opinion it would have been a very good report for a student work but the content is too light for a journal paper.

The authors prepared Bi₂O₃ containing iron from bismuth and iron nitrates in solution by precipitation after addition of soda. A precipitate was obtained and exposed to microwave. There is no indication in the manuscript on the effect of the microwave treatment, whether the powder composition was the same before and after. Depending on the iron content a change in the powder colour is to be noticed in Figure 2 but there is no indication on how iron is incorporated in the material. X-ray diffractograms are given in figure 3, a change in the diagrams is clearly observed for iron content higher than 5% but the authors just refer to a difference in the intensity. They claimed they obtained alpha-Bi₂O₃. I think this is true up to 3% of iron but may be more complicated for higher iron content. The report crystallite size with 3 digits after the decimal point: 25.702 nm ! I think from X-ray diffraction, the accuracy is in the order of the manometer. The kinetics study may be of interest but the structural characterisation of the catalyst needs major revision for this paper to be published.

Editor's comments: The paper needs a major revision considering the comments made by the reviewers. A detailed response to all the review comments must be submitted along with the revised paper. It is essential that the revised paper should be read carefully by all the authors to avoid technical/grammatical/presentation errors.

In compliance with data protection regulations, you may request that we remove your personal registration details at any time. (Use the following URL: <https://www.editorialmanager.com/cogenteng/login.asp?a=r>). Please contact the publication office if you have any questions.

 **COGENTENG-2021-0443_reviewer.pdf**
673K

215625984 (Cogent Engineering) A revise decision has been made on your submission

Cogent Engineering <em@editorialmanager.com>
Reply-To: Cogent Engineering <oaen-peerreview@journals.tandf.co.uk>
To: Heri Sutanto <herisutanto@live.undip.ac.id>

Thu, Nov 4, 2021 at 8:15 AM

Ref: COGENTENG-2021-0443
215625984
Efficient degradation of amoxicillin using Bi₂O₃/Fe synthesized by microwave-assisted precipitation method
Cogent Engineering

Dear Heri Sutanto,

Thank you for your patience following your submission to Cogent Engineering. Your manuscript entitled "Efficient degradation of amoxicillin using Bi₂O₃/Fe synthesized by microwave-assisted precipitation method", has now been reviewed. It has been decided that your manuscript will require a major/minor revision before publication. Reviewer comments are available to be viewed at the bottom of this email.

The deadline for this revised submission is Dec 03, 2021. If you do not wish to carry out this revision, and would prefer to submit elsewhere, please let us know via emailing OAEN-peerreview@journals.tandf.co.uk.

However, we encourage you to proceed with revising your manuscript. Please provide an editable word document.

To submit your revised manuscript please go to <https://rp.cogentoa.com/dashboard/> and log in. You will see an option to Revise alongside your submission record.

Please ensure you include the following elements in your revised submission/Please check the attachment for information on what you will need to include in your revised submission. If you are unsure how to submit your revision, please contact us on OAEN-peerreview@journals.tandf.co.uk where a member of our Editorial Team will be more than happy to assist you.

I look forward to receiving your revised manuscript.

Best wishes,
Sanjay Kumar Shukla, PhD
Senior Editor
Cogent Engineering

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Reviewer 2: Yes

Title, Abstract and Introduction - overall evaluation
Reviewer 2: Sound with minor or moderate revisions

Methodology / Materials and Methods – overall evaluation

Reviewer 2: Sound with minor or moderate revisions

Objective / Hypothesis – overall evaluation

Reviewer 2: Sound

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Reviewer 2: Sound with minor or moderate revisions

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Reviewer 2: Sound with minor or moderate revisions

Interpretation / Discussion – overall evaluation

Reviewer 2: Sound

Conclusions – overall evaluation

Reviewer 2: Sound

References – overall evaluation

Reviewer 2: Sound

Compliance with Ethical Standards – overall evaluation

Reviewer 2: Sound

Writing – overall evaluation

Reviewer 2: Sound with minor or moderate revisions

Supplemental Information and Data – overall evaluation

Reviewer 2: Sound

Comments to the author

Reviewer 2: Dear Author

I read your manuscript and I found you resent a good idea

Besides, I write some commutes in the PDF text need to correct. I hope to see these comments

Many thanks,

Reviewer

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Reviewer 5: Yes

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Supplemental Information and Data – overall evaluation
Reviewer 5: Not applicable

Comments to the author
Reviewer 5: Dear Editor

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In compliance with data protection regulations, you may request that we remove your personal registration details at any time. (Use the following URL: <https://www.editorialmanager.com/cogenteng/login.asp?a=r>). Please contact the publication office if you have any questions.

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