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Terdapat perubahan judul yang disesuaikan dengan saran/komentar dari reviewer selama proses submisi dan revisi berlangsung. Adapun histori perubahan judul yang dilakukan adalah sebagai berikut:

Submisi awal	Removal of methylene blue by TiO2-modified guar gum hydrogel through	
	adsorption and radiation-mediated degradation	
Judul setelah	TiO2/Guar gum hydrogel composite for adsorption and photodegradation of	
revisi	methylene blue	

Submission to International Journal of Biological Macromolecules - manuscript number

From: International Journal of Biological Macromolecules (em@editorialmanager.com)

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Manuscript Number: IJBIOMAC-D-21-04856

Removal of methylene blue by TiO2-modified guar gum hydrogel through adsorption and radiation-mediated degradation

Dear Dr. Santoso,

Your above referenced submission has been assigned a manuscript number: IJBIOMAC-D-21-04856.

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Date: Wednesday, June 30, 2021 at 03:26 AM GMT+7

Manuscript Number: IJBIOMAC-D-21-04856

Removal of methylene blue by TiO2-modified guar gum hydrogel through adsorption and radiation-mediated degradation

Dear Dr. Santoso,

Thank you for submitting your manuscript to International Journal of Biological Macromolecules.

I have completed my evaluation of your manuscript. The reviewers recommend reconsideration of your manuscript following major revision. I invite you to resubmit your manuscript after addressing the comments below. Please resubmit your revised manuscript by Jul 29, 2021.

When revising your manuscript, please consider all issues mentioned in the reviewers' comments carefully: please outline every change made in response to their comments and provide suitable rebuttals for any comments not addressed. Please note that your revised submission may need to be re-reviewed.

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International Journal of Biological Macromolecules values your contribution and I look forward to receiving your revised manuscript.

Kind regards, Ian Sims Editor

International Journal of Biological Macromolecules

Editor and Reviewer comments:

Reviewer #1: In this manuscript, guar gum (galactomannan polysaccharide) was used to prepare hydrogeladsorbent to treat azo dye-contaminated water. Modification of guar gum hydrogel (GGH) by TiO2 was performed to provide photodegradation ability to the adsorbent. And it showed some promising results. However, there are still many doubts about the paper. Thus, the reviewer thinks the paper might need a major revision.

- 1. Why choose Guar gum as the raw material? Does it have any advantages compared with other renewable materials (cellulose, chitosan, etc.)?
- 2. There have been a lot of researches on the removal of dyes by TiO2-modified renewable material, where is the innovation of this work?
- 3. The description of the effect of epichlorohydrin is incorrect. Please use a schematic diagram to show the formation process of the gel, and describe in detail the cross-linking effect of epichlorohydrin in it.
- 4. The paper lacks important characteristics of composite materials, such as XPS analysis to determine the composition. The distribution of TiO2 on the composite material was analyzed by EDS.
- 5. The basic data of the material is also very important, such as the thermal stability of the material. Thus, it is recommended to add some related references, for example, DOI: 10.1016/j.carbpol.2020.116463; DOI: 10.1016/j.carbpol.2020.116971.
- 6. There is a significant difference between the pores shown in the SEM image of the GGH sample and the pore diameter data of the BET test. The pore diameter of the SEM image reaches 100 µm, and the average pore diameter of

the BET test is only 74.5 nm. This is an obvious error, and the analysis in the paper is also incorrect. Please use the mercury intrusion method to retest the pore structure of the sample and analyze it.

- 7. The SEM picture of the <u>TiO2@GGH</u> sample is very important. Please change to a more ideal SEM picture. In addition, TiO2 can be well attached to the structure of the material in other studies, why TiO2 particles in this study will cause the collapse of the pore structure.
- 8. XRD test cannot show the relationship between TiO2 and GGH.
- 9. The text in Figure 2 is too small to read clearly. Figure 2e lacks a title. The serial number of the picture in Line 236 is not accurate.
- 10. The work needs to supplement the cyclic experiment of removing the dye by the sample.

Reviewer #2: This work entitled Removal of methylene blue by TiO2-modified guar gum hydrogel through adsorption and radiation-mediated degradation describes the performance of TiO2-modified guar gum hydrogel on the dye-containing water treatment. The topic is interesting and the work is well organized. However, some issues still need to be addressed.

Substantial results should be included in the Abstract.

It is recommended to add the gelation method in the Graphical Abstract.

Introduction should provide readers with some background information on this topic. Thus, some recently reported literature on guar gum hydrogel should be mentioned. Some examples might help, such as Journal of Power Sources, 2021, 493: 229711; Carbon, 2020, 157: 153-163.

As claimed by the authors, the porous structure was collapsed in <u>TiO2@GGH</u>. Then, is it appropriate to name it as hydrogel? Moreover, the authors mentioned "TiO2 particles probably induce the gelation of the GG particles and cause the collapse of the pore structure." Why? Any evidences or literature support?

The gelation mechanism should be described in the manuscript.

The salinity sometimes affects the hydrogel properties. It is recommended to add the influence of salinity on the hydrogel properties.

Figure 3d: the sample treated with radiation shows higher removal ratio than that without radiation. What is the reason?

Does the photodegradation process affect the hydrogel properties?

Some typos should be corrected, such as 3.2. Characterization (Line 196), and Figure 2. (a, b) N2 sorption isotherm curves, (c, d) XRD (FTIR is missing, Line 257).

Data in Brief (optional):

We invite you to convert your supplementary data (or a part of it) into an additional journal publication in Data in Brief, a multi-disciplinary open access journal. Data in Brief articles are a fantastic way to describe supplementary data and associated metadata, or full raw datasets deposited in an external repository, which are otherwise unnoticed. A Data in Brief article (which will be reviewed, formatted, indexed, and given a DOI) will make your data easier to find, reproduce, and cite.

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Date: Oct 06, 2021

To: "Shella Permatasari Santoso" shella_p5@yahoo.com;shella@ukwms.ac.id

From: "International Journal of Biological Macromolecules" support@elsevier.com

Subject: Decision on submission to International Journal of Biological Macromolecules

Manuscript Number: IJBIOMAC-D-21-04856R2

TiO2/Guar gum hydrogel composite for adsorption and photodegradation of methylene blue

Dear Dr. Santoso,

Thank you for submitting your manuscript to International Journal of Biological Macromolecules.

I am pleased to inform you that your manuscript has been accepted for publication.

My comments, and any reviewer comments, are below.

Your accepted manuscript will now be transferred to our production department. We will create a proof which you will be asked to check, and you will also be asked to complete a number of online forms required for publication. If we need additional information from you during the production process, we will contact you directly.

We appreciate you submitting your manuscript to International Journal of Biological Macromolecules and hope you will consider us again for future submissions.

Kind regards, Ian Sims Editor

International Journal of Biological Macromolecules

Editor and Reviewer comments:

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International Journal of Biological Macromolecules

TiO2/Guar gum hydrogel composite for adsorption and photodegradation of methylene blue --Manuscript Draft--

Manuscript Number:	IJBIOMAC-D-21-04856R2
Article Type:	Research Paper
Section/Category:	Carbohydrates, Natural Polyacids and Lignins
Keywords:	Photodegradation; Guar gum; Titanium dioxide.
Corresponding Author:	Shella Permatasari Santoso Widya Mandala Catholic University: Universitas Katolik Widya Mandala Surabaya Surabaya, INDONESIA
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	Hsien-Yi Hsu
	Phuong Lan Tran-Nguyen
	Felycia Edi Soetaredjo
	Suryadi Ismadji
Abstract:	The development of porous adsorbent materials from renewable resources for water and wastewater treatment has received considerable interest from academia and industry. This work aims to synthesize composite hydrogel from the combination of guar gum (a neutral galactomannan polysaccharide) and TiO2. The TiO2-embedded guar gum hydrogel (TiO2@GGH) was utilized to remove methylene blue through adsorption and photodegradation. The presence of TiO2 particles in the hydrogel matrix (TiO2@GGH) was confirmed by scanning electron microscopy-energy dispersive X-ray and X-ray photoelectron spectroscopy analysis. The mercury intrusion and N2 sorption isotherm indicate the macroporous structure of the TiO2@GGH composite, showing the presence of pore sizes ~420 µm. The dye removal efficiency of the GGH and TiO2@GGH was evaluated in batch mode at ambient temperature under varying pH. The effect of UV radiation on the dye removal efficiency was also assessed. The results demonstrated that the highest dye removal was recorded at pH 10, with the equilibrium condition achieved within 5h. UV radiation was shown to enhance dye removal. The maximum adsorption capacity of TiO2@GGH is 198.61 mg g-1, while GGH sorbent is 188.53 mg g-1. The results imply that UV radiation gives rise to the photodegradation effect.
Suggested Reviewers:	Chun-Hui Zhou Zhejiang University of Technology clay@zjut.edu.cn Ianatul Sunarso Nottingham University Press: University of Nottingham Ianatul.Khoiroh@nottingham.edu.my Ahmed Fazary King Khalid University aefazary@gmail.com; aefazary@kku.edu.sa
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[Answer] The title was shortened to <150 characters, i.e., "TiO2/Guar gum hydrogel composite for adsorption and photodegradation of methylene blue" The correspondence has been marked with "*", the postal address, telephone, fax, and

email address of the corresponding author has been provided.

2. Self contained abstract of approximately 200 words, outlining in a single paragraph the aims, scope and conclusions of the paper.

[Answer] The abstract has been modified as suggested.

- 3. Three keywords, for indexing purposes; [Answer] Three keywords has been provided.
- 4. The text suitably divided under headings.
 [Answer] The change has been made as suggested.
- 5. Acknowledgments (if any).
 [Answer] The acknowledgment has been included.
- 6. References (double spaced, and following the journal style). [Answer] The reference format has been revised according to the journal style.
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 [Answer] The article does not have appendix.
- 8. Tables (each on a separate sheet). [Answer] The tables have been placed in separate sheets under the references.
- 9. Captions to illustrations (grouped on a separate sheet or sheets). [Answer] The captions to illustration have been grouped in separate sheets.
- 10. Illustrations, each on a separate sheet containing no text, and clearly labelled with the journal title, author's name and illustration number.

 [Answer] The illustrations have been provided in separated sheets with no text.



Widya Mandala Catholic University Surabaya FACULTY OF ENGINEERING – CHEMICAL ENGINEERING DEPARTMENT Jl. Kalijudan 37 Surabaya 60114, East Java, Indonesia

Tel: +62 31 3893933 ext.: 103, 111, Fax: +62 31 3891267

October 5, 2021

Dear Editor,

Attached, please find the revised article IJIBIOMAC-D-21-04856R2. We have revised the format according to the Guide for Author for the journal. We would like to thank the Reviewers and Editor for the given chance to revise our manuscript, we believe that the revised manuscript now is acceptable for publication in the *International Journal of Biological Macromolecules*.

Sincerely,

Shella Permatasari Santoso Associate Professor Department of Chemical Engineering Widya Mandala Surabaya Catholic University, Indonesia shella p5@yahoo.com

Tel: +62-31-389-1264