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15-Apr-2022

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TITLE: Hydrocracking optimization of palm oil to bio-gasoline and bio-aviation fuels using molybdenum nitride-bentonite catalyst

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10-May-2022

Dear Dr Hasanudin:

Manuscript ID: RA-ART-04-2022-002438
TITLE: Hydrocracking optimization of palm oil to bio-gasoline and bio-aviation fuels using molybdenum nitride-bentonite catalyst

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I look forward to receiving your revised manuscript.

Yours sincerely,
Dr Ji-Jun Zou
Associate Editor, RSC Advances

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REVIEWER REPORT(S):
Referee: 1

Recommendation: Major revisions

Comments:
This research emphasizes on the production of bio-gasoline and bio-aviation fuels from hydrocracking of palm oil over molybdenum nitride-bentonite catalyst. This research completely reports the optimization of reaction parameters for biofuels production using statistical analysis. However, some part of catalyst characterization and product analysis should be more explained as shown below:
1. In the introduction part, please provide the advantage of molybdenum nitride-based catalysts when they are compared to others for hydrotreating process.
2. For preparation of catalyst, the authors reduced the catalyst at 350 ^oC. Why did you select this temperature? The hydrogen-temperature programmed reduction (H₂-TPR) technique should be given. Moreover, the authors reported that the catalyst obtained after reduction was then sieved by 200-mesh. I think some part of reduced catalyst would be oxidized before activating the hydrocracking process.
3. For the hydrocracking of palm oil, the palm oil flow rate and how to control it should be stated.
4. The analysis for distinguishing the bio-gasoline and bio-aviation has to be declared.
5. For Eq. 1, I think the degree of conversion should be evaluated from the reduction of the triglyceride content in the liquid product. The Eq. 1 uses the volumes of feed and unreacted feed to calculate the conversion. This means the loss of liquid product to yield the gaseous or solid products at each given condition.
6. In the part of hydrocracking of palm oil (Page 7 of 29), the authors reported that “…in which the catalyst's active site was molybdenum nitride,…”. Did the nitride form still exist after reduction in the presence of hydrogen? Please provide the evidence.
7. In the same paragraph of the comment No. 6, the authors explained by related the Bronsted acid sites. The contents of Bronsted and Lewis acid sites of the molybdenum nitride-bentonite catalyst should be shown.

Additional Questions:
Does the work significantly advance the understanding or development in this field?: Yes

Is this work of relevance to the chemistry community?: Yes

Are the conclusions of the work convincing and sufficiently supported by experimental evidence?: Yes

Does the data provided fulfil the journal’s data requirements?

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Is the experimental section sufficiently detailed to allow others to reproduce the work?: Yes

Are the reported claims adequately discussed in the context of the literature?: Yes

Are the number of tables and figures in the manuscript appropriate and clear?: Yes

Referee: 2

Recommendation: Minor revisions

Comments:
List of comments:
Here they are, some more questions and comments for supporting my decision;
1. In the section of introduction, please give more details about the hydro-cracking reaction as well as the

downstream process to recovery product.
2. For the catalyst used, it was necessary to activate whether or not before starting reaction.
3. The authors should give more details for the catalyst packing and also how much it was used in batch reaction. In addition, how many times the catalyst can be used?
4. The raw material of palm oil used was missing. What kinds of palm oil used? It was needed to get pretreatment before reaction whether or not.
5. Test equipment in the section of materials and methods must be listed.
6. The procedure of fractional distillation to recovery product was missing. The details must be added.
7. The author should give more details why a conversion rate of 78.33% and equivalent to the oil yield of 50.32%, gas yield of 44%, and coke yield of 5.73%.

Additional Questions:
Does the work significantly advance the understanding or development in this field?: Yes

Is this work of relevance to the chemistry community?: Yes

Are the conclusions of the work convincing and sufficiently supported by experimental evidence?: Yes

Does the data provided fulfil the journal's data requirements?

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Is the experimental section sufficiently detailed to allow others to reproduce the work?: Yes

Are the reported claims adequately discussed in the context of the literature?: Yes

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Title : Hydrocracking optimization of palm oil to bio-gasoline and bio-aviation fuels using molybdenum nitride-bentonite catalyst Manuscript ID : RA-ART-04-2022-002438 Author name : Hasanudin Hasanudin, Wan Ryan Asri, Muhammad Said, Putri Tamara Hidayati, Widia Purwaningrum, Novia Novia, and Karna Wijaya		
	Thank you for giving us the opportunity to submit a manuscript titled "Hydrocracking optimization of palm oil to bio-gasoline and bio-aviation fuels using molybdenum nitride-bentonite catalyst" for publication in the Journal of RSC Advances. We appreciate the time and effort that you dedicated to providing feedback on our manuscript and are grateful for the insightful comments and valuable improvements to our paper. We have incorporated the suggestions made by the reviewers. Those changes are written in yellow highlight text within the manuscript.	
	Referee 1 This research emphasizes on the production of bio-gasoline and bio-aviation fuels from hydrocracking of palm oil over molybdenum nitride-bentonite catalyst. This research completely reports the optimization of reaction parameters for biofuels production using statistical analysis. However, some part of catalyst characterization and product analysis should be more explained as shown below:	
No	Comment from Referee	Rebuttals
1	In the introduction part, please provide the advantage of molybdenum nitride-based catalysts when they are compared to others for hydrotreating process.	Thank you for pointing this out, we have provided the advantage of molybdenum nitride-based catalyst, as a reviewer suggested. As we mentioned earlier in the introduction section, molybdenum nitride has a high surface area which promotes high catalytic activity. Furthermore, they can function as active catalysts as well as catalytic supports, having unique features that encourage high catalytic activity. Compared to other catalysts, molybdenum is easily nitrided and has a more favorable interaction than other elements such as cobalt nitride and nickel. This catalyst is also less costly than the

		noble metal catalyst. Hence, this catalyst provides good potential properties with a relatively less expensive and more selective alternative than other catalysts. The revised text as follows: "Currently, metal catalysts with diverse phases,[36–38] including noble metal[39] have been used in hydrogen-involved reactions. However, those catalysts have drawbacks due to high cost and high coke deposition, which hinder industrial-scale processes[40]." "Molybdenum is easily nitrided and has a more favorable interaction than other compounds such as cobalt nitride and nickel nitride[43]. As a result, this catalyst provides good potential properties with a relatively less expensive and more selective alternative than other catalysts.[44]" For more additional clarification, we have changed the term "nitrite" to "nitride" due to a typo, and we have added a sentence of "potentially solving" in the same paragraph since it has made the sentence clear to read.
2	For preparation of catalyst, the authors reduced the catalyst at 350 ˚C. Why did you select this temperature? The hydrogen-temperature programmed reduction H_2-TPR) technique should be given. Moreover, the authors reported that the catalyst obtained after reduction was then sieved by 200-mesh. I think some part of reduced catalyst would be oxidized before activating the hydrocracking process.	Thank you for pointing this out. Sorry for this omission, we missed written some part of reduction process. The method we wrote is less detailed. Sorry for this omission. We missed writing some parts of the reduction process. In this study, the reduction temperature was gradually increased up to 600 ˚C, with a controlled heating rate. First, the temperature was raised at 276.65 ˚C/min to 623.15 K, afterward at 273.45 K/min to 773.15 K, then subsequent at 274.15 K/min to 873.15 K, and held for 2 hours. We have changed the

		Celsius unit to Kelvin to make the unit consistent. This method refers to the modified method of Hamdan et al. Reference: M. Abou Hamdan, A. Nassereddine, R. Checa, M. Jahjah, C. Pinel, L. Piccolo and N. Perret, *Front. Chem.*, 2020, **8**, 1–12. The revised text as follows: "In the reduction process, the temperature was raised at 276.65 ˚C/min to 623.15 K, afterward at 273.45 K/min to 773.15 K, then subsequent at 274.15 K/min to 873.15 K, and held for 2 hours.[48]" We sieved the catalyst by 200-mesh to prevent the catalyst's agglomeration post-reduction. The agglomeration of the catalyst could reduce the catalyst contact between the reactant, which lowers the catalytic activity. Furthermore, to ensure that the catalyst is not oxidized, we saturate the reactor with H_2 gas prior to the hydrocracking process to remove the oxygen gas. Hence, this would avoid the oxidation of the catalyst. The revised text as follows: "The reactor was saturated with H_2 gas under pre-determined conditions prior to the hydrocracking process to remove the oxygen gas.[54]"

3	For the hydrocracking of palm oil, the palm oil flow rate and how to control it should be stated.	Thank you for pointing this out. The palm oil flow rate was 11.94 g/min and pumped using a peristaltic pump. This feedstock flow rate was proportional to 100 mL of palm for a 7.8 min reaction, as we convert the volume of palm oil into weight using its density. The revised text as follows: "The palm oil flow rate was 11.94 g/min and pumped using a peristaltic pump"
4	The analysis for distinguishing the bio-gasoline and bio-aviation has to be declared.	Thank you for pointing this out. We have analyzed the bio-gasoline and bio-aviation using a GC-MS. According to the GC-MS analysis, the lower molecular weight would show a shortened retention time rather than the higher molecular weight. In this context, the carbon atom ranging from C_5 to C_{12} is considered a bio-gasoline, whereas the carbon atom ranging from C_{13}-C_{16} is considered a bio-aviation fuel. The revised text as follows: "The biogasoline and bio-aviation fuel fractions were evaluated based on the hydrocarbons group with C_5-C_{12} and C_{13}-C_{16}, respectively."
5	For Eq. 1, I think the degree of conversion should be evaluated from the reduction of the triglyceride content in the liquid product. The Eq. 1 uses the volumes of feed and unreacted feed to calculate the conversion. This means the loss of liquid product to yield the gaseous or solid products at each given condition.	In Eq. 1, we have realized that the use of volume feed is not appropriate. We have changed the equation regarding the conversion determination. The volume of feed was changed to the weight of the feed. In this condition, we assumed that the weight of palm oil feed is a triglyceride that further undergoes the hydrocracking process.

		The revised text as follows: $$X\ (\text{w/w}\ \%) = \frac{\text{Weight}_{\text{feed}} - \text{Weight}_{\text{unreacted feed}}}{\text{Weight}_{\text{feed}}} \quad (1)$$
6	In the part of hydrocracking of palm oil (Page 7 of 29), the authors reported that "…in which the catalyst's active site was molybdenum nitride,…". Did the nitride form still exist after reduction in the presence of hydrogen? Please provide the evidence.	Thank you for pointing this out. In this study, we used ammonium heptamolybdate tetrahydrate as molybdenum and reacted with ammonium nitrate to form a molybdenum nitrate ($Mo(NO_3)_6$). Precisely, the hydrogen gas in this study was used to reduce the molybdenum nitrate to molybdenum nitride. In this condition, nitride exists, and we have provided the evidence by XRD characterization. Furthermore, form SEM-EDX analysis showed an increase in bentonite's nitrogen content from 0 to 0.49 % after modification, which strengthened the evidence.
7	In the same paragraph of the comment No. 6, the authors explained by related the Bronsted acid sites. The contents of Bronsted and Lewis acid sites of the molybdenum nitride-bentonite catalyst should be shown.	Thank you for pointing this out. In this manuscript, unfortunately, we did not evaluate the specific content of Bronsted and Lewis acid quantitatively. Hence, we choose to delete the sentences. Nevertheless, we have calculated the total acidity of the catalyst, which clearly showed that the modification of bentonite using molybdenum nitride increased the acidity of the bentonite catalyst.

	Referee 2	
1	In the section of introduction, please give more details about the hydro-cracking reaction as well as the downstream process to recovery product.	Thank you for pointing this out. As we mentioned earlier in the introduction section, hydrocracking is a two-stage process combining catalytic cracking and hydrogenation, in which feedstocks with a longer number of carbon atoms are cracked in the presence of hydrogen and a bifunctional catalyst into shorter carbon atoms. Furthermore, we have added the details about the hydrocracking reaction as the reviewer suggested. The revised text as follows: "A further vacuum distillation process is conducted to separate hydrocracking fraction product. The hydrocracking process involves the removal of double bonds and carboxyl groups in fatty acids to obtain compounds with a lower number of carbon atoms. In this process, with the presence of hydrogen gas and the bifunctional catalyst, the hydrodeoxygenation reaction is entailed as the primary reaction, whereas the decarboxylation is the side reaction. Furthermore, the liquid hydrocarbon product generally contains a rich n-alkanes compound obtained through three complex reactions, namely decarbonylation, decarboxylation, and hydrodeoxygenation.[12]" As additional clarification, we have changed the phrase "decomposition" to "catalytic cracking" since it has made the sentence more appropriate. Also, we have added a new sentence of

		"In the matter of the hydrocracking feedstocks" to continue the paragraph regarding the explanation of hydrocracking feedstocks.
2	For the catalyst used, it was necessary to activate whether or not before starting reaction.	Thank you for pointing this out. In order to ensure that the molybdenum nitride-bentonite catalyst is ready to start the hydrocracking reaction, first of all, the reactor is saturated by hydrogen gas. This treatment was done to expel the oxygen gas and make sure the catalyst is thoroughly reduced in the form of molybdenum nitride. This molybdenum nitride acts as an active catalyst site.
3	The authors should give more details for the catalyst packing and also how much it was used in batch reaction. In addition, how many times the catalyst can be used?	Thank you for bringing it to our attention. We used a catalyst weight of 12 g catalyst and prepared with 200-mesh, as previously stated in the materials and method section. Subsequently, as the reviewer is concerned, the study of regeneration of catalyst as well as the deactivation of catalyst is critical to understanding the catalyst performance. As this topic is a huge aspect, we currently explore this aspect in the other manuscript. In this paper, we focused on the optimization of hydrocracking process using RSM-CCD.
4	The raw material of palm oil used was missing. What kinds of palm oil used? It was needed to get pretreatment before reaction whether or not.	We thank the reviewer for pointing this out. We have incorporated the revision throughout the manuscript. The palm oil used was RBD (Refined, Bleached, and Deodorized) palm oil which obtained from PT. Agro Indralaya Mandiri. This RDB palm oil was taken without further pretreatment.

		The revised text as follows: "RBD (Refined, Bleached, and Deodorized) palm oil was obtained from PT. Agro Indralaya Mandiri without further pretreatment"
5	Test equipment in the section of materials and methods must be listed.	Thank you for pointing this out. We already listed the test equipment regarding the catalyst characterization in the materials and method, whereas the hydrocracking of palm oil was conducted using a fixed-bed reactor's feed column. In the context of optimization, The RSM-CCD and statistical analysis (ANOVA) were conducted using Design-expert 12 software, as previously mentioned in the manuscript.
6	The procedure of fractional distillation to recovery product was missing. The details must be added.	Thank you for pointing this out. This observation is correct. We have provided the procedure of distillation of recovery product fraction. The revised text as follows: "The liquid of the hydrocracking product was later heated in the vacuum distillation at 200 °C to acquire the respective biofuel fractions. After distillation, the leftover liquid in the flask was comprised of unreacted triglycerides from the palm oil feedstock"
7	The author should give more details why a conversion rate of 78.33% and equivalent to the oil yield of 50.32%, gas yield of 44%, and coke yield of 5.73%.	Thank you for pointing this out. As we know that the term conversion is used to describe ratios of how much of a reactant has reacted, i.e., the palm oil itself, which contained triglyceride, regardless of what product it goes to, whereas the term yield is used to describe how much of a product was formed, i.e., the oil, gas, and coke. In our study, based on the optimum condition, we got the

		conversion of 78.33%, which indicated that 78.33% of the reactant (triglyceride) reacted to form a product, whereas 21.67% was the unreacted triglyceride. In this case, the hydrocracking reaction produces an oil yield of 50.32%, a gas yield of 44%, and a coke yield of 5.73%. The method of calculating those parameters was described in the materials and section method. We hope that it is now clearer.
Additional clarifications		
1	We have changed the Table 2 caption from “The range of levels variables studied using RSM-CCD” to “Analysis of catalyst acidity” due to an inaccurate caption.	
2	In the acknowledgment section, we have changed the sentences from “This work was supported by Universitas Sriwijaya through grant competitive 2021 Contract No. 0010/UN9/SK.LP2M.PT/2021, April 28, 2021.” to “This work was supported by Universitas Sriwijaya through Hibah Kompetitif 2022 No. 0109/UN9.3.1/SK/2022.” Lastly, thank you for the valuable reviewer comments and corrections.	

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13-May-2022

Dear Dr Hasanudin:

TITLE: Hydrocracking optimization of palm oil to bio-gasoline and bio-aviation fuels using molybdenum nitride-bentonite catalyst
AUTHORS: Hasanudin, Hasanudin; Asri, Wan; Said, Muhammad; Hidayati, Putri; Purwaningrum, Widia; Novia, Novia; Wijaya, Karna

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Decision on submission to RSC Advances - RA-ART-04-2022-002438.R1

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25-May-2022

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