Hasanudin Hasanudin <hasanudin@mipa.unsri.ac.id>

[Catalysts] Manuscript ID: catalysts-2029380 - Review Application for Manuscript

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Kepada: Hasanudin Hasanudin <hasanudin@mipa.unsri.ac.id>
Cc: Cathy Yang <cathy.yang@mdpi.com>, catalysts@mdpi.com

Dear Dr. Hasanudin,

Thanks for your application as a reviewer for the following manuscript:

Manuscript ID: catalysts-2029380
Type of manuscript: Article
Title: Hydrodeoxygenation-isomerization of methyl palmitate over SAPO-11 supported Ni-phosphide catalysts
Authors: Ivan V. Shamanaev *, Irina A. Shamanaeva, Ekaterina V. Parkhomchuk, Galina A. Bukhtiyarova *

We will contact and send you a reviewing link once your application is approved.

Do not hesitate to contact us if you have any questions.

Kind regards,
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Kepada: Hasanudin Hasanudin <hasanudin@mipa.unsri.ac.id>
Cc: Catalysts Editorial Office <catalysts@mdpi.com>, Cathy Yang <cathy.yang@mdpi.com>

Dear Dr. Hasanudin,

Thank you very much for applying to review the following manuscript:

Type of manuscript: Article
Title: Hydrodeoxygenation-isomerization of methyl palmitate over SAPO-11 supported Ni-phosphide catalysts

We are pleased to accept your review application. The manuscript and review report form can be found at the following link:
https://susy.mdpi.com/user/review/review/32365096/NVJq175X

The due date for the review report is 9 November 2022.

We look forward to receiving your valuable comments.

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Manuscript details:
Journal: Catalysts
Manuscript ID: catalysts-2029380
Type of manuscript: Article
Title: Hydrodeoxygenation-isomerization of methyl palmitate over SAPO-11 supported Ni-phosphide catalysts
Authors: Ivan V. Shamanaev *, Irina A. Shamanaeva, Ekaterina V. Parkhomchuk, Galina A. Bukhtiyarova *
Submitted to section: Biomass Catalysis,
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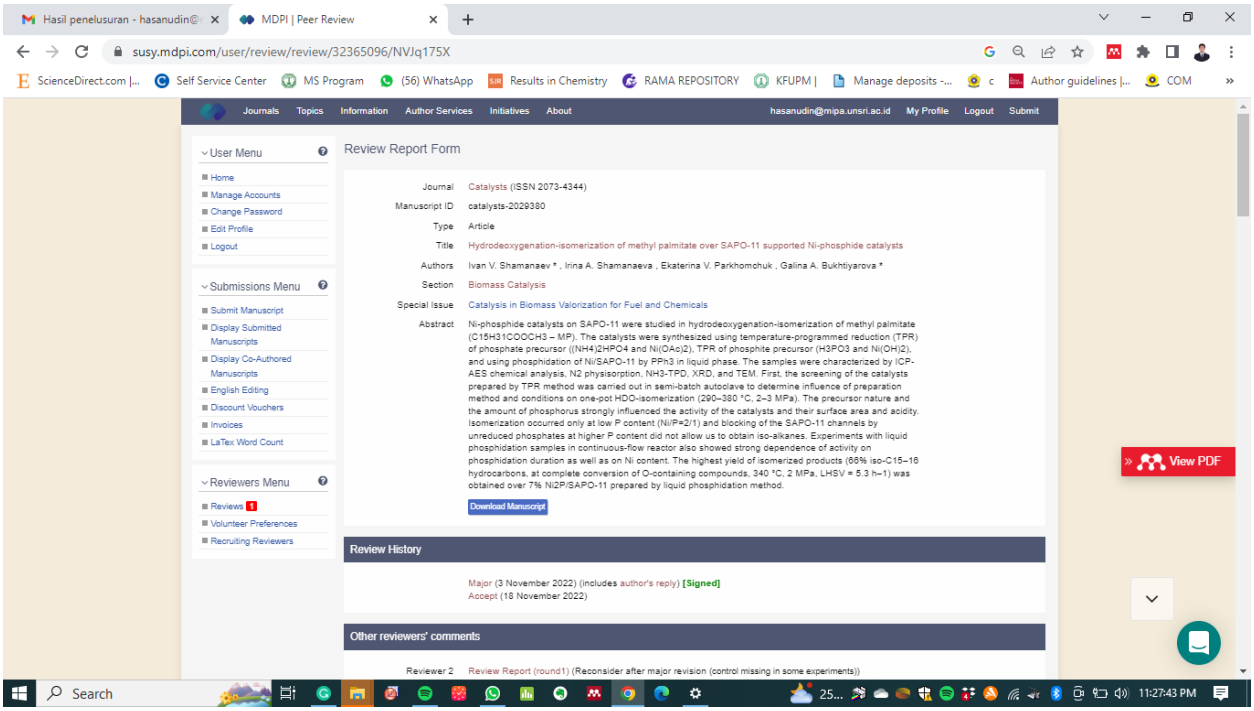
Catalysis in Biomass Valorization for Fuel and Chemicals
https://www.mdpi.com/journal/catalysts/special_issues/1060243_biomasscatalysis

Abstract: Ni-phosphide catalysts on SAPO-11 were studied in hydrodeoxygenation-isomerization of methyl palmitate (C15H31COOCH3 – MP). The catalysts were synthesized using temperature-programmed reduction (TPR) of phosphate precursor ((NH4)2HPO4 and Ni(OAc)2), TPR of phosphite precursor (H3PO3 and Ni(OH)2), and using phosphidation of Ni/SAPO-11 by PPh3 in liquid phase. The samples were characterized by ICP-AES chemical analysis, N2 physisorption, NH3-TPD, XRD, and TEM. First, the screening of the catalysts prepared by TPR method was carried out in semi-batch autoclave to determine influence of preparation method and conditions on one-pot HDO-isomerization (290–380 °C, 2–3 MPa). The precursor nature and the amount of phosphorus strongly influenced the activity of the catalysts and their surface area and acidity. Isomerization occurred only at low P content (Ni/P=2/1) and blocking of the SAPO-11 channels by unreduced phosphates at higher P content did not allow us to obtain iso-alkanes. Experiments with liquid phosphidation samples in continuous-flow reactor also showed strong dependence of activity on phosphidation duration as well as on Ni content. The highest yield of isomerized products (66% iso-C15–16 hydrocarbons, at complete conversion of O-containing compounds, 340 °C, 2 MPa, LHSV = 5.3 h–1) was obtained over 7% Ni2P/SAPO-11 prepared by liquid phosphidation method.

Keywords: hydrodeoxygenation; isomerization; Ni-phosphide; SAPO-11; methyl palmitate; biofuel; green diesel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Journal Catalysts (ISSN 2073-4344)
Manuscript ID catalysts-2029380
Type Article
Title Hydrodeoxygenation-isomerization of methyl palmitate over SAPO-11 supported Ni-phosphide catalysts
Authors Ivan V. Shamanaev * , Irina A. Shamanaeva , Ekaterina V. Parkhomchuk , Galina A. Bukhtiyarova *
Section Biomass Catalysis
Special Issue Catalysis in Biomass Valorization for Fuel and Chemicals
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Major (3 November 2022) (includes author's reply) [Signed]
Accept (18 November 2022)
Other reviewers' comments
Reviewer 2 Review Report (round1) (Reconsider after major revision (control missing in some experiments))
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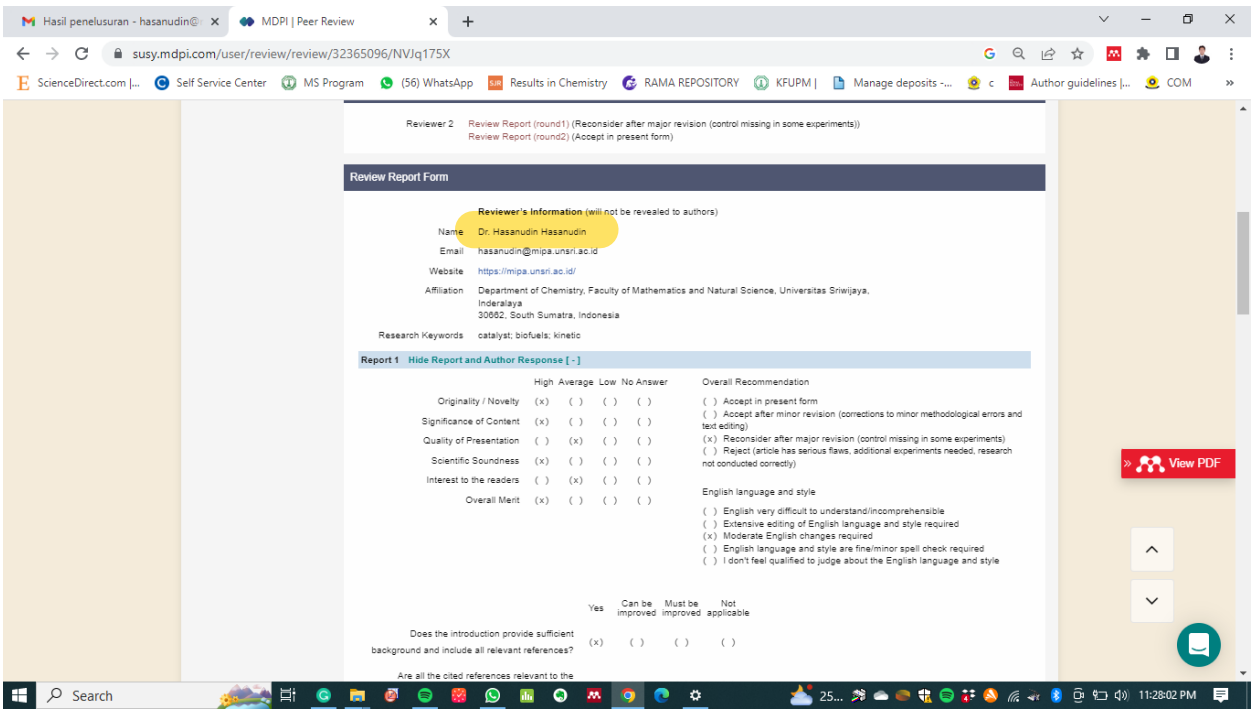
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Reviewer 2 Review Report (round1) (Reconsider after major revision (control missing in some experiments))
Review Report (round2) (Accept in present form)
Review Report Form
Reviewer's Information (will not be revealed to authors)
Name Dr. Hasanudin Hasanudin
Email hasanudin@mipa.unsri.ac.id
Website https://mipa.unsri.ac.id/
Affiliation Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Sriwijaya, Inderalaya
30662, South Sumatra, Indonesia
Research Keywords catalyst; biofuels; kinetic
Report 1 Hide Report and Author Response [-]
High Average Low No Answer
Originality / Novelty (x) ( ) ( ) ( )
Significance of Content (x) ( ) ( ) ( )
Quality of Presentation ( ) (x) ( ) ( )
Scientific Soundness (x) ( ) ( ) ( )
Interest to the readers ( ) (x) ( ) ( )
Overall Merit (x) ( ) ( ) ( )
Overall Recommendation
( ) Accept in present form
( ) Accept after minor revision (corrections to minor methodological errors and text editing)
(x) Reconsider after major revision (control missing in some experiments)
( ) Reject (article has serious flaws, additional experiments needed, research not conducted correctly)
English language and style
( ) English very difficult to understand/incomprehensible
( ) Extensive editing of English language and style required
(x) Moderate English changes required
( ) English language and style are fine/minor spell check required
( ) I don't feel qualified to judge about the English language and style
Yes Can be improved Must be improved Not applicable
Does the introduction provide sufficient background and include all relevant references? (x) ( ) ( ) ( )
Are all the cited references relevant to the
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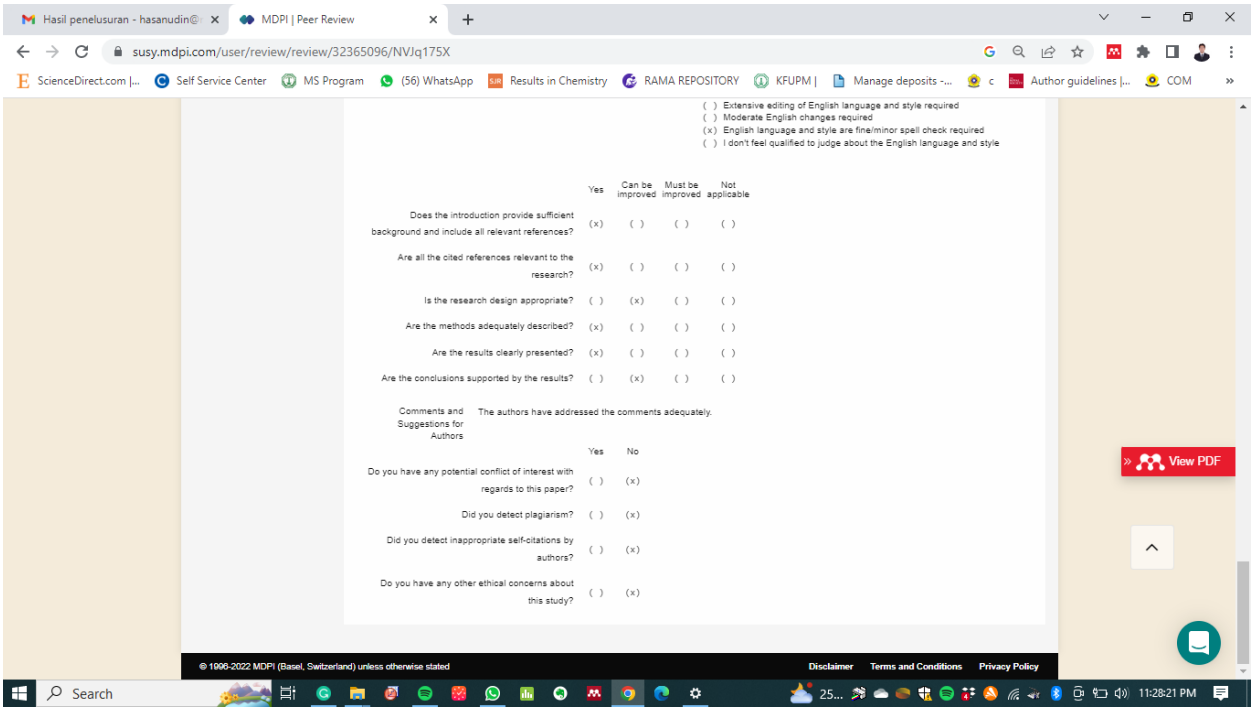

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( ) Moderate English changes required
(x) English language and style are fine/minor spell check required
( ) I don't feel qualified to judge about the English language and style

	Yes	Can be improved	Must be improved	Not applicable
Does the introduction provide sufficient background and include all relevant references?	(x)	( )	( )	( )
Are all the cited references relevant to the research?	(x)	( )	( )	( )
Is the research design appropriate?	( )	(x)	( )	( )
Are the methods adequately described?	(x)	( )	( )	( )
Are the results clearly presented?	(x)	( )	( )	( )
Are the conclusions supported by the results?	( )	(x)	( )	( )

Comments and Suggestions for Authors: The authors have addressed the comments adequately.

	Yes	No
Do you have any potential conflict of interest with regards to this paper?	( )	(x)
Did you detect plagiarism?	( )	(x)
Did you detect inappropriate self-citations by authors?	( )	(x)
Do you have any other ethical concerns about this study?	( )	(x)

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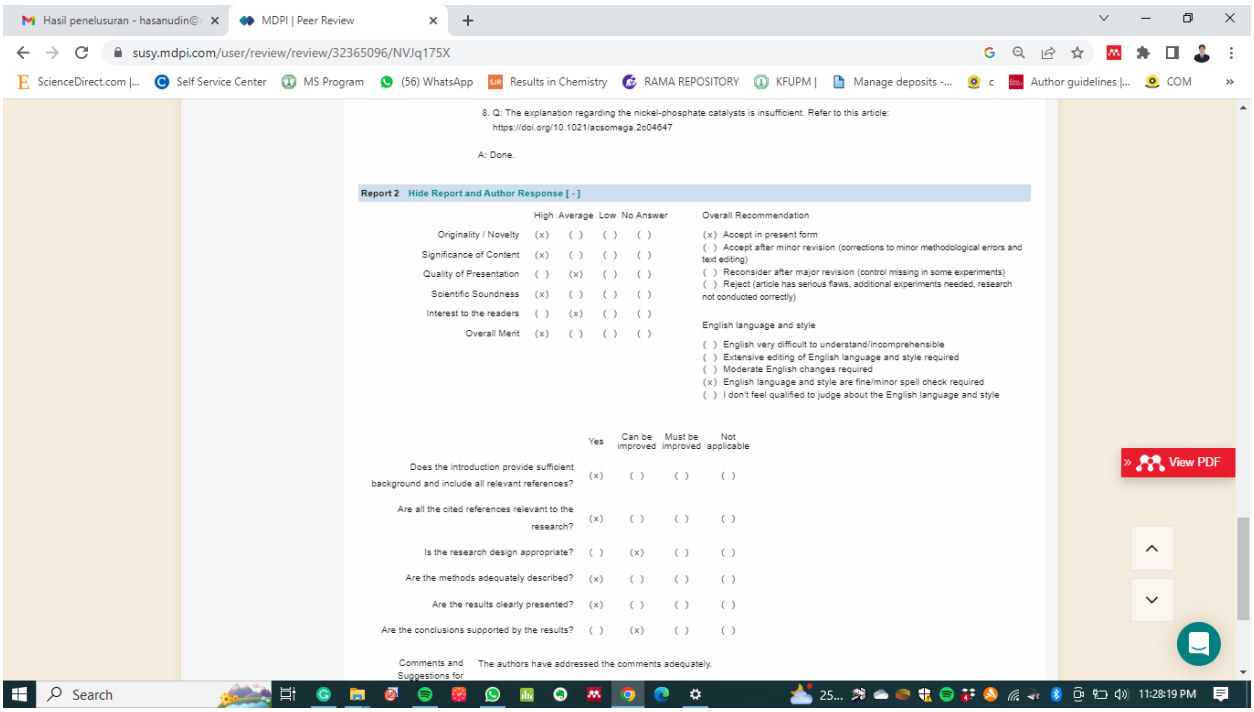

8. Q: The explanation regarding the nickel-phosphate catalysts is insufficient. Refer to this article: https://doi.org/10.1021/acsomega.2c04647

A: Done.

Report 2 Hide Report and Author Response [-]

	High	Average	Low	No Answer
Originality / Novelty	(x)	( )	( )	( )
Significance of Content	(x)	( )	( )	( )
Quality of Presentation	( )	(x)	( )	( )
Scientific Soundness	(x)	( )	( )	( )
Interest to the readers	( )	(x)	( )	( )
Overall Merit	(x)	( )	( )	( )

Overall Recommendation

(x) Accept in present form
( ) Accept after minor revision (corrections to minor methodological errors and text editing)
( ) Reconsider after major revision (control missing in some experiments)
( ) Reject (article has serious flaws, additional experiments needed, research not conducted correctly)

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( ) I don't feel qualified to judge about the English language and style

	Yes	Can be improved	Must be improved	Not applicable
Does the introduction provide sufficient background and include all relevant references?	(x)	( )	( )	( )
Are all the cited references relevant to the research?	(x)	( )	( )	( )
Is the research design appropriate?	( )	(x)	( )	( )
Are the methods adequately described?	(x)	( )	( )	( )
Are the results clearly presented?	(x)	( )	( )	( )
Are the conclusions supported by the results?	( )	(x)	( )	( )

Comments and Suggestions for: The authors have addressed the comments adequately.

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3. Q: It is necessary to report the yield of the hydrodeoxygenation-isomerization product.

A: Done for NiP_A catalysts with different Ni/P ratio tested at 290 °C. For the other experiments the temperatures were higher 340-380 °C, and X_{MP} was 100%. So the selectivities were equal to the yields. We added this to the manuscript.

4. Q: It is necessary to propose the mechanism reaction regarding the hydrodeoxygenation-isomerization of methyl palmitate

A: Done.

5. Q: The N_2 adsorption-desorption curve was missed. It is necessary to add those curves and elaborate it in order to understand the textural properties of the catalyst.

A: The isotherms are quite similar and it is difficult to understand differences between the support and catalysts. Thus, we added pore diameter distributions to the Supplementary material.

6. Q: Why does the author only report the selectivity of C-15 and C-16? How about the lower range C fraction? The chromatogram of the product needed to be shown.

A: Lower range C fraction (C6-14) was also detected over several catalysts The amount of cracked products are shown on Figures 5 and 7. The chromatograms are added to the Supplementary material. Unfortunately it is not possible to determine the amount of individual compounds like C6, C7 and so on, because solven (n-dodecane) also cracks and contributes to cracked products. We can only calculate the total amount of cracked products based on the mass-balance.

7. Q: Why does the author use transitional metal phosphide, whereas there also exist such as transitional metal nitride, and transitional metal sulfide. Refer to this article: http://dx.doi.org/10.1039/D2RA02438A and https://doi.org/10.1039/D2RA03941A

A: Done. Phosphide catalysts and especially Ni phosphides are among the most active in hydroprocessing and HDO.

8. Q: The explanation regarding the nickel-phosphate catalysts is insufficient. Refer to this article: https://doi.org/10.1021/acsomega.2c04647

A: Done.

Report 2 Hide Report and Author Response [-]

High Average Low No Answer Overall Recommendation

Do you have any other ethical concerns about this study? ( ) (x)

Author response to report 1:

Author's Notes

We are grateful to the reviewers for reading and evaluating our work.

Please find attached our answers.

Answers to the Reviewer 1

1. Q: It is necessary to evaluate the stability of catalysts towards the hydrodeoxygenation-isomerization of methyl palmitate through the reusability and the regeneration catalyst study

A: Ni-phosphide catalysts proved to be stable during 8-9 hours of time on stream without any loss of activity. Conversion and selectivity vs time on stream graphs are added to the Supplementary material.

2. Q: The author mentioned that the surface area of the NiP catalyst had a low surface area due to pore blocking. How does the author prove this presumption? More characterization regarding the spent catalyst must be conducted.

A: The S_{BET} after phosphide preparation was ~20 m^2/g. After reaction S_{BET} decreased to ~5-8 m^2/g. The data are provided in Supplementary material.

3. Q: It is necessary to report the yield of the hydrodeoxygenation-isomerization product.

A: Done for NiP_A catalysts with different Ni/P ratio tested at 290 °C. For the other experiments the temperatures were higher 340-380 °C, and X_{MP} was 100%. So the selectivities were equal to the yields. We added this to the manuscript.

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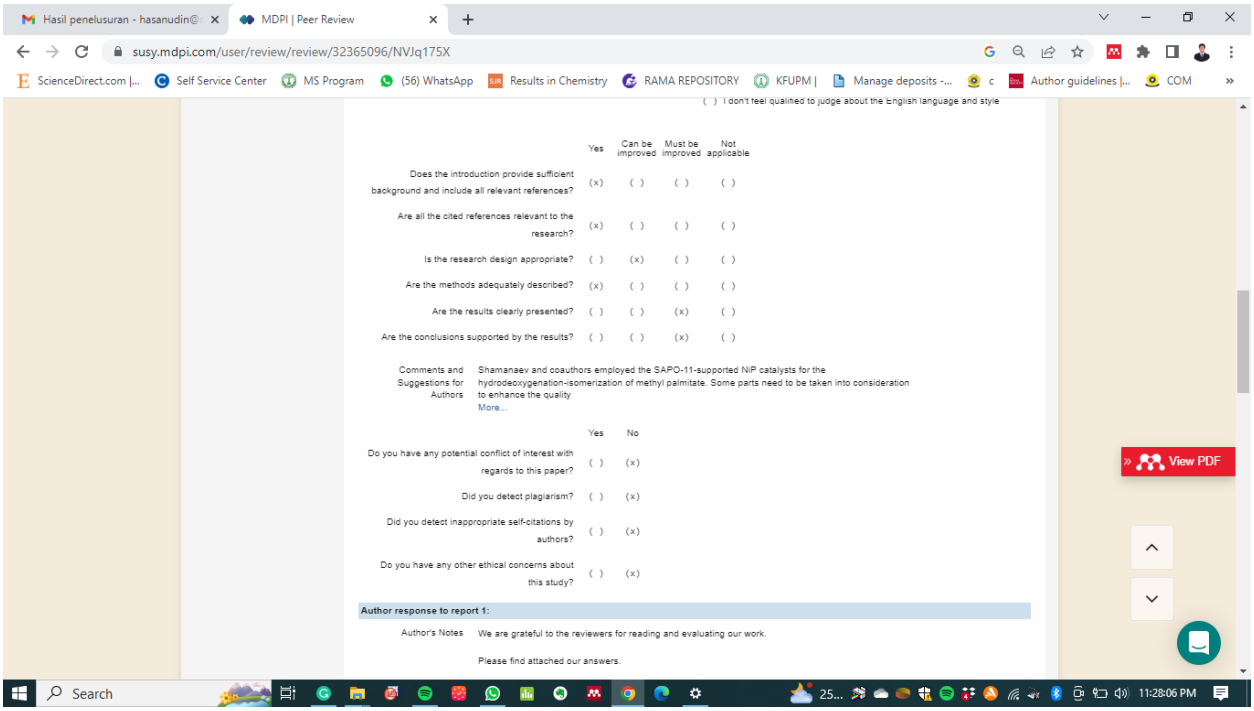
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Yes
Can be improved
Must be improved
Not applicable
Does the introduction provide sufficient background and include all relevant references? (x) ( ) ( ) ( )
Are all the cited references relevant to the research? (x) ( ) ( ) ( )
Is the research design appropriate? ( ) (x) ( ) ( )
Are the methods adequately described? (x) ( ) ( ) ( )
Are the results clearly presented? ( ) ( ) (x) ( )
Are the conclusions supported by the results? ( ) ( ) (x) ( )
Comments and Suggestions for Authors
Shamanaev and coauthors employed the SAPO-11-supported NiP catalysts for the hydrodeoxygenation-isomerization of methyl palmitate. Some parts need to be taken into consideration to enhance the quality
More...
Yes
No
Do you have any potential conflict of interest with regards to this paper? ( ) (x)
Did you detect plagiarism? ( ) (x)
Did you detect inappropriate self-citations by authors? ( ) (x)
Do you have any other ethical concerns about this study? ( ) (x)
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Dear Dr. Hasanudin,

Thank you for submitting your review of the following manuscript:

Manuscript ID: catalysts-2029380
Title: Hydrodeoxygenation-isomerization of methyl palmitate over SAPO-11 supported Ni-phosphide catalysts
Authors: Ivan V. Shamanaev *, Irina A. Shamanaeva, Ekaterina V. Parkhomchuk, Galina A. Bukhtiyarova *

Our Editorial Office and Academic Editors will contact you if they have any questions about your review report. We ask that you remain available, as far as possible, during the peer-review process in case of follow-up questions. To help us improve our services, we kindly ask you to fill in our online survey on the peer-review process at
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Dear Dr. Hasanudin,

You recently kindly reviewed the original version of the following manuscript, submitted to Catalysts:

Title: Hydrodeoxygenation-isomerization of methyl palmitate over SAPO-11 supported Ni-phosphide catalysts
Authors: Ivan V. Shamanaev *, Irina A. Shamanaeva, Ekaterina V. Parkhomchuk, Galina A. Bukhtiyarova *

The authors have now provided a revised version along with a cover letter in which they address the referees' comments.

Please let us know, within 3 days, if you believe the manuscript has been sufficiently improved to warrant publication in Catalysts. If you need more time to check the revised version, please let the Editorial Office know in advance. Please click on the link below to access the revised manuscript and the review report form:

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Kind regards,
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Dear Dr. Hasanudin,

We are writing to inform you that the following paper which you kindly reviewed has been published:
https://www.mdpi.com/2073-4344/12/11/1486

Thank you for your participation in the peer-review process. The paper was accepted by the Academic Editor following the peer review process, in which review reports were received from 2 reviewers. To see comments from the manuscript's other reviewers please create an account on our submission system at https://susy.mdpi.com with your review email, hasanudin@mipa.unsri.ac.id, and visit the "Reviews" Section.

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All review reports were made available for the Academic Editor to support their final decision. We acknowledge your efforts to review this article and support the author to improve their manuscript. We look forward to your future participation in the review process.

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REVIEW CONFIRMATION CERTIFICATE

We are pleased to confirm that

Hasanudin Hasanudin

has reviewed 2 papers for the following MDPI journals in 2022:

Energies, Catalysts

Dr. Shu-Kun Lin, Publisher and President
Basel, 22 December 2022

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