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[Catalysts] Manuscript ID: catalysts-2008830 - Submission Received

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Complexed-Impregnation and Templated Methods for Crude Palm Oil to Biofuels Conversion via Catalytic Hydrocracking

Authors: Hasanudin Hasanudin *, Wan Ryan Asri, Zainal Fanani, Selvi Julpani Adisti, Fitri Hadiyah, Roni Maryana, Muhammad Al Muttaqii, Zongyuan Zhu, Nelio Teixeira Machado

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Conversion via Catalytic Hydrocracking

Authors: Hasanudin Hasanudin *, Wan Ryan Asri, Zainal Fanani, Selvi Julpani Adisti, Fitri Hadiah, Roni Maryana, Muhammad Al Muttaqii, Zongyuan Zhu, Nelio Teixeira Machado

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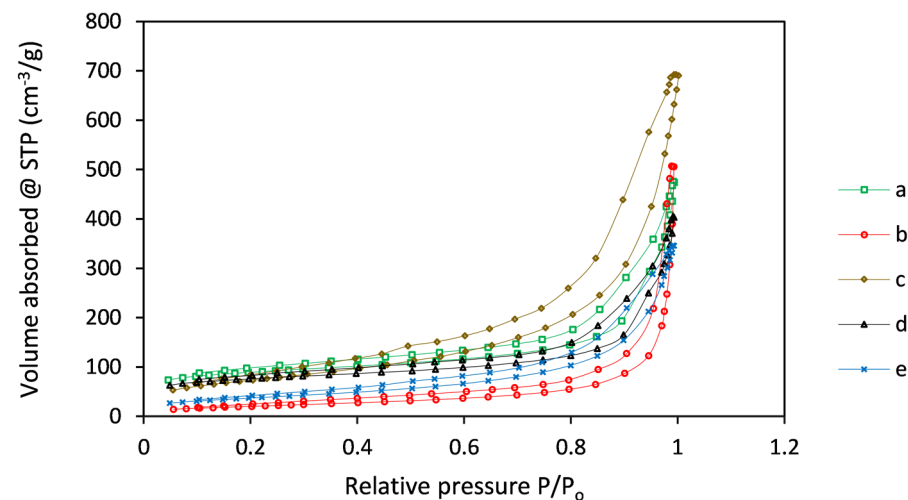
<p>Title : Facile Fabrication of SiO₂/Zr Assisted with EDTA Complexed-Impregnation and Templated Methods for Crude Palm Oil to Biofuels Conversion via Catalytic Hydrocracking</p> <p>Manuscript ID : catalysts-2008830</p> <p>Authors : Hasanudin Hasanudin * , Wan Ryan Asri , Zainal Fanani , Selvi Julpani Adisti , Fitri Hadiah , Roni Maryana , Muhammad Al Muttaqii , Zongyuan Zhu , Nelio Teixeira Machado</p>		
<p>Thank you for giving us the opportunity to submit a manuscript titled “Facile Fabrication of SiO₂/Zr Assisted with EDTA Complexed-Impregnation and Templated Methods for Crude Palm Oil to Biofuels Conversion via Catalytic Hydrocracking” for publication in the Catalysts. 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.</p>		
<p>Reviewer 1</p> <p>In this manuscript, the authors tried to report a new type of crude palm oil to biofuels through catalytic hydrocracking processes was an attractive technological route to produce biofuels. In this work, high biofuel yields were obtained from crude palm oil over SiO₂/Zr-CEDTA catalysts. Therefore, this manuscript is proposed to be accepted, after revising the following question.</p>		
No	Comment from Reviewer	Responses
1	Please clearly illustrate the novelty of present work in introduction section	Thank you for pointing this out. According to the literature review, the modification of SiO ₂ /Zr using several techniques, such as templating and chelating methods, and comparing their respective effects on the catalytic activity of hydrocracking, have not been studied yet. In this study, particularly, our novelty is employing the EDTA species as a chelating and templating agent for synthesizing SiO ₂ /Zr. The use of EDTA has the potential as a SiO ₂ template or chelating agent, considering that these species are capable of complex binding to zirconium, and calcination at high temperatures will cause EDTA to be

		<p>removed, allowing changes in the physicochemical properties of SiO₂/Zr. This condition inherently affects the catalytic activity toward CPO hydrocracking.</p> <p>The revised text as follows:</p> <p>“The modification of SiO₂/Zr using several techniques, such as templating and chelating methods, and comparing their respective effects on the catalytic activity of hydrocracking, have not been studied yet. Furthermore, neither exploration nor report has been made for fabricating the SiO₂/Zr using an EDTA species as chelating and templating agents.”</p>
2	<p>Figure 1, Could you please summarize the meaning of the peak intensity and peak position in the XRD pattern?</p>	<p>Thank you for pointing this out. In brief, based on the XRD analysis, we have shown that the chelating and templating methods using EDTA affected the crystal structural properties of the SiO₂/Zr catalyst, in which the chelating method caused the catalyst to be amorphous, whereas the templating method promoting the amorphous structure of the catalyst to be crystalline, as justified by their two theta peaks.</p> <p>Moreover, the Zr phases were observed along with SiO₂ phases, both for SiO₂/Zr as well as for SiO₂/Zr-TEDTA (prepared by the templating method), which indicated that the SiO₂/Zr were successfully synthesized. Meanwhile, the SiO₂/Zr-CEDTA (prepared by the chelating method) revealed that none of the Zr phases were observed, which suggested that the Zr species was finely dispersed towards SiO₂ support. Other studies also reported similar results, as written in the manuscript. We hope this explanation could satisfy the reviewer's comments.</p>
3	<p>More details of how to use the Scherrer Equation to obtain the average metal-crystal size should be presented, for example, which peaks were</p>	<p>Thank you for pointing this out. We want to clarify that we could not calculate the average metal-crystal size using the Scherrer Equation. In this manuscript, we calculate the average particle size distribution of the catalyst using the Horiba Partica LA-960 equipped with a static light scattering method rather than exploring the crystalline size of the catalyst. This aspect noted that the modification of SiO₂/Zr affects the average particle size distribution of the catalyst. In the context of hydrocracking, the particle size</p>

	chosen to calculate the crystal size of which crystal phases.	distribution effect was dominant compared with their crystalline size. However, it would have been interesting to explore this aspect in our further study.
4	Figure 2, Could you please summarize the meaning of the wavelength peak in the FTIR spectra?	Thank you for pointing this out. Briefly, our FTIR analysis showed the existence of the functional groups of SiO ₂ , which indicated that the SiO ₂ had been successfully prepared. The functional groups of Zr were relatively undistinguishable, presumably because of the intensive band of silanol groups, thus overlapping the zirconia groups bands. Since catalysts involved the same functional groups, there was no appreciable peak change of SiO ₂ /Zr prepared by the chelate method compared with the parent SiO ₂ /Zr catalysts, whereas a slightly different absorption band at a low wavelength was observed when the template method was employed. This condition suggested that the template and chelate methods affected how the functional groups of silanol, as well as zirconia groups, bonded in the SiO ₂ /Zr catalyst.
5	How to measure the acidity concentration of the catalyst?	<p>Thank you for pointing this out. The acidity value of the catalyst was determined by the gravimetric method, as reported by several studies [1,2]. Prior to catalyst saturation by probes, the desiccator was vacuumed.</p> <p>The total, surface, and pore acidity were determined according to the following equations:</p> $\text{Total acidity (mmol NH}_3\text{/g)} = \frac{W_2 - W_1}{(W_1 - W_0) \times \text{MW NH}_3} \quad (1)$ $\text{Surface acidity (mmol pyridine/g)} = \frac{W_2 - W_1}{(W_1 - W_0) \times \text{MW pyridine}} \quad (2)$ $\text{Pore acidity (mmol/g)} = \text{Total acidity} - \text{Surface acidity} \quad (3)$ <p>Where W₀ and W₁ are the mass of the porcelain container and porcelain container+sampel, whereas the W₂ is the mass of porcelain container+sampel after the absorption of probe, the MW is denoted as the molecular weight of the corresponding probes.</p>

		<p>Since these equations are well reported, to simplify the manuscript, we have added citations regarding the measurement of the acidity value of the catalyst in the manuscript. The revised text as follows:</p> <p>“The total and surface acidity were determined using the gravimetric method with pyridine and ammonia as a probe [74],”</p> <p>References:</p> <ol style="list-style-type: none"> 1. Aziz, I.T.A.; Saputri, W.D.; Trisunaryanti, W.; Sudiono, S.; Syoufian, A.; Budiman, A.; Wijaya, K. Synthesis of Nickel-Loaded Sulfated Zirconia Catalyst and Its Application for Converting Used Palm Cooking Oil to Gasoline via Hydrocracking Process. <i>Period. Polytech. Chem. Eng.</i> 2022, <i>66</i>, 101–113, doi:10.3311/PPch.18209. 2. Marsuki, M.F.; Trisunaryanti, W.; Falah, I.I.; Wijaya, K. Synthesis of Co, Mo, Co-Mo and Mo-Co Catalysts, Supported on Mesoporous Silica-Alumina for Hydrocracking of α-Cellulose Pyrolysis Oil. <i>Orient. J. Chem.</i> 2018, <i>34</i>, 955–962, doi:10.13005/ojc/340245.
6	<p>In stability experiments, how to determine the amount of acidic sites for reused catalysts in order to evaluate the probable leaching of active acidic sites during catalytic experiment. Did the try to activate the catalyst?</p>	<p>Thank you for pointing this out. In this study, we have activated the spent catalyst using heat treatment through re-calcination (to oxidize the coke) and re-reduction (to produce the active metal site) methods, with the same temperature described in the experimental section. The catalyst probably occurred due to the fouling and coke formation. The acidic site of the reused catalysts might be gradually decreased at a three consecutive run. The acidity value of reused catalysts can be calculated by the gravimetric method. However, we did not determine the acidity value of the spent catalyst. It would be interesting to explore this aspect in our further study.</p>
7	<p>More characterization of catalyst such as BET surface, Sorption isotherm and XPS</p>	<p>Thank you for pointing this out. We have added the BET surface and the sorption isotherm. Regarding the XPS analysis, exploring this aspect would have been interesting. However, this instrumentation was</p>

<p>analysis are required. [BET surface and Sorption isotherm is used technique to obtain surface area and porous properties. X-ray photoelectron spectroscopy (XPS) is a widely used technique to obtain surface elemental composition].</p>	<p>not available in Indonesia, and it would take time to send the sample outside of the country. We have provided the EDX-mapping analysis to evaluate the elemental composition of the catalyst.</p> <p>The revised text as follows:</p> <p>“The N₂ physisorption of SiO₂, SiO₂/Zr, and their modification using chelate and template methods are presented in Figure 5. Based on the IUPAC categorization, it was readily apparent that all the catalysts had a type IV isotherm with a hysteresis loop of type H4, which suggested that the catalysts were mesoporous. Furthermore, the H4 hysteresis loop was related to the micropores with narrow slits feature [56].”</p>
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“**Figure 5.** N₂ physisorptions of (a) SiO₂ (b) SiO₂/Zr (c) SiO₂-TEDTA (d) SiO₂/Zr-TEDTA (e) SiO₂/Zr-CEDTA”

“The textural features of all catalysts are shown in Table 2. The surface area of SiO₂ significantly decreased after being loaded by the Zr species, presumably due to the pore blocking of Zr species [61]. It can be seen that the SiO₂/Zr-KEDTA and SiO₂/Zr-CEDTA had higher surface area compared with the parent SiO₂/Zr catalyst, whereas the SiO₂ prepared by the template method relatively decreased the surface area of the catalyst but increased the total pore volume as well as the average pore radius of the catalysts. This condition indicated that the template and chelate method affected the textural properties of catalysts. The porous structure generated by the EDTA template method enhanced the pore volume of catalysts, whereas the EDTA chelate method reduced the total pore volume of SiO₂/Zr-CEDTA.”

Table 2. Textural features of catalysts

Catalyst	Surface area (m ² /g)	Total pore volume (cm ³ /g)	Average pore radius (Å)
SIO2	294	0.73	49.76
SIO2/Zr	76.43	0.78	20.48
SIO2-TEDTA	268	0.81	60.44
SIO2/Zr-TEDTA	220.5	0.83	75.37
SIO2/Zr-CEDTA	134	0.53	79.58

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<p>Reviewer 2</p> <p>Below are a few comments that can help the authors improve their manuscript:</p>		
No	Comment from Reviewer	Responses
1	Introduction is way too long. It can be made more concise.	Thank you for pointing this out. We have reduced the text in the introduction section.
2	Overall structure. Unless it is a journal requirement, the experimental methods usually come before the results. I had a hard time trying to understand the results without knowing first how things were done.	The results and discussion come before the methods, as appeared in the catalysts' manuscript template. We have also followed the catalysts guideline of the authors.
3	Hydrocracking experiments. The temperature used was way too high and this could have been the	Thank you for pointing this out. We have agreed with the reviewer high temperatures could generate a high gas yield, and we have incorporated it through the manuscript.

	<p>reason behind such high gas yields (Table 3). This could also explain why the catalyst lost so much activity after 3 runs (Table 5), since coke formation at 500C would be really high. This is really my biggest issue with this study, I would have expected temperatures of 400C or below for palm oil.</p>	<p>In this study, we did not vary the temperature since we focused on the preliminary study regarding the preparation and modification of SiO₂/Zr and their potential for hydrocracking reactions. However, in the next study, we would optimize the hydrocracking condition, such as temperature, catalyst weight, H₂ flow rate, and so forth, to get an optimum result with low gas yield.</p> <p>The revised text as follows: “Based on Table 3, all catalysts relatively generated high yields due to high-temperature hydrocracking”</p>
4	<p>Test results missing. There are some details of the results that are missing. For example, authors don't explain in Table 3 what the gas and residue products are constituted of. Likewise, hydrogen consumption is not reported. This is a key process parameter that impacts economics and energy intensity</p>	<p>Thank you for pointing this out. The gas product commonly consists of the uncondensable gas such as CO, CO₂, and other C₁-C₅ hydrocarbons, but we cannot provide the exact constituent of the gas product since we did not analyze the gas product through GCMS. The residue consisted of unreacted triglyceride. Regarding hydrogen consumption, we cannot calculate this parameter since we did not trap the gas and just released it out of the system. However, exploring this aspect in the other study is very interesting and necessary.</p> <p>The revised text as follows: “The gas product commonly consists of the uncondensable gas such as CO, CO₂, and other C₁-C₅ hydrocarbons”</p> <p>“Based on Table 4, the residue yield of the hydrocracking process for all catalysts ranges from 10.04-28.58%. The residue product consists of the unreacted triglyceride, and the lowest residue yield (10.04%) was achieved by the SiO₂/Zr-CEDTA, which suggested that the SiO₂/Zr prepared by the chelate method was the most effective for CPO hydrocracking compared with other prepared catalysts.”</p>
5	<p>Authors did not explained what happened with the oxygen in the feed. Did turn into gas or water? If so, how much of each.</p>	<p>Thank you for pointing this out. In this study, we used triglyceride as feedstock which consisted of oxygen-containing compounds. After the hydrocracking process, the oxygen-containing compounds would turn into CO₂ and CO gas, and we did not analyze it quantitatively since the product gas was not trapped. The oxygen-containing compounds such as 1-Octanol, 2-butyl-, Cyclohexanol, and so forth were also observed according to the GCMS analysis.</p>

6	<p>The discussion on the effect of catalyst composition and prep method on product distribution (Table 4) is not satisfactory. Authors briefly mention diffusivity, but they should have looked at reaction mechanisms of hydrocracking and from there explain the observed trends.</p>	<p>Thank you for pointing this out. We have revised the text as suggested by the reviewer.</p> <p>The revised text as follows: “During the CPO hydrocracking process, triglycerides and hydrogen gas diffused to the surface or pores of the catalyst and were followed by adsorption to the active site of the catalyst. This step would crack the triglycerides into carbon atoms with lower chains forming the biofuel fraction and some gases. After that, the product was desorbed from the surface of the catalyst and subjected to diffusion into the gas phase.”</p>
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