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Manuscript Needs Revision (Major Revision) (#IJC-2206-1942 (R1))

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I can now inform you that the reviewers have evaluated your manuscript. Major revision has been requested.
Attached you can find comments from the reviewers. Please, submit the revised manuscript with blue marking on the revised parts. The revised manuscript should include Figures, Tables, and other files that you mentioned in the manuscript.
With the resubmission, please enclose a detailed description of your revision in response to the reviewers' comments.

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Reviewers Recommendation:

Reviewer 1:

Reviewer Comment For Author:

Dear Author(s)
The paper contains a good idea about modification of sodium montmorillonite using zirconium phosphate. In the same time, the style of writing the manuscript is good. The paper need to add the FTIR test of the prepared catalysts and explain the relation of this test with the conversion process in detail.

Reviewer 2:

Reviewer Comment For Author:

Dear Authors,
The catalytic activity of clay-catalysts exchanged and activated by different zirconium phosphate solutions in the conversion of methanol dimethyl ether is discussed in this paper.
The authors obtained very good results in conversion and selectivity, and they proved that their catalysts are catalytically active and more selective towards conversion reactions. The authors show a very close relationship between the catalytic activity and acidity of M-ZrP2 and M-ZrP2. The preparation, characterization, and activity of the conversion catalyst I suggest accepting this paper with the addition of some explication about catalyst characterization and acidity. In my opinion, the catalytic activity of the described materials should be compared with other clay catalysts and tested in the same reaction conditions. Below are pointed out several aspects of the ms to be improved for a possible new examination.

The values on the line is not clear from the text. Moreover, in Fig. 1, 2θ values are in the range of 0-100, so how can the peaks be observed.
Specify the type of reactor in the text and in the catalytic test part, and give the different dimensions of the reactor (diameter, ...)
According to the NH3-TPD method, the authors showed that there were strong and weak acid sites, and in

particular on the Lewis acid sites. It is preferable to know the distributions of these acids by methods (example: IFTR adsorption of pyridine or others) to better compare the active catalysts like M-ZrP2 and M-ZrP2).
The authors must explain clearly in the text, the participation of the ziconium-phosphate solution in the effect of the acidity of the catalysts.
What do you mean by (cc/g) in table 2
In the synthesis part, the catalysts were calcined at 450 °C, and the catalytic application was carried out at temperatures between 150 and 350 °C. I would like to know an explanation of what stability the authors are talking about and at what temperature the synthesized catalysts resist.
The authors must add an explanation of the variations of the percentage of the elements Al, Si, and Fe during the exchange with the solutions compared to the effect of the acidity of the catalysts.
The BET method shows that the catalysts obtained are of type II according to the classification and that the catalysts are non-porous. I find that the pores are very important (79 A) (table2), contrary to the results obtained.
The results of the NH3-TPD showed that the strong acid sites were detected after 350C°, whereas the catalytic test was carried out at 150-350C°
I find that the specific surface area obtained is low compared to the results obtained on the specific surface by other research (add references).

Title : Montmorillonite-Zirconium Phosphate Catalysts for Methanol Dehydration Manuscript ID : IJC-2206-1942		
	Thank you for giving us the opportunity to submit a manuscript titled “Montmorillonite-Zirconium Phosphate Catalysts for Methanol Dehydration” for publication in the Iranian Journal of Catalysis. 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.	
	Reviewer 1	
No	Comment from Reviewer	Responses
1	The paper contains a good idea about modification of sodium montmorillonite using zirconium phosphate. In the same time, the style of writing the manuscript is good. The paper need to add the FTIR test of the prepared catalysts and explain the relation of this test with the conversion process in detail.	Thank you for pointing this out. It would have been interesting to explore this aspect. Indeed, we did not discuss regarding the presence of a functional group that we know can be confirmed by FTIR as well as their relation to the conversion process. However, in our study, this would not be possible due to the covid pandemic, which limits working hours, it takes a relatively long time to be analyzed with a long queue in our region. Furthermore, the sample must be sent to the outer region, which takes a relatively long time. Nevertheless, in our study, the presence of the bentonite phase and the zirconium phosphate was clearly confirmed by the characterization of XRD (according to standard reference) and EDX (according to the addition of Zr elemental composition, etc). It is very interesting and necessary to explore this aspect (FTIR) in the other study.

	Reviewer 2 The catalytic activity of clay-catalysts exchanged and activated by different zirconium phosphate solutions in the conversion of methanol dimethyl ether is discussed in this paper. The authors obtained very good results in conversion and selectivity, and they proved that their catalysts are catalytically active and more selective towards conversion reactions. The authors show a very close relationship between the catalytic activity and acidity of M-ZrP2 and M-ZrP2. The preparation, characterization, and activity of the conversion catalyst I suggest accepting this paper with the addition of some explication about catalyst characterization and acidity. In my opinion, the catalytic activity of the described materials should be compared with other clay catalysts and tested in the same reaction conditions. Below are pointed out several aspects of the ms to be improved for a possible new examination.	
No	Comment from Reviewer	Responses
1	The values on the line is not clear from the text. Moreover, in Fig. 1, 2θ values are in the range of 0-100, so how can the peaks be observed.	Thank you for pointing this out. We have revised Fig. 1 by marked the peaks on XRD diffractograms to make the reading easier. In this study, the diffraction angle (2θ) was scanned from 5˚ to 80˚ intervals. As can be seen in Fig. 1, the peaks at 6.15˚ and 19.69˚ corresponded to the montmorillonite mineral, whereas the peaks at 15.53˚, 20.68˚, and 26.59˚ attributed to the typical peaks of zirconium phosphate.
2	Specify the type of reactor in the text and in the catalytic test part, and give the different dimensions of the reactor (diameter, ...)	Thank you for pointing this out. In this study, we used the continuous fixed bed reactor, with an inner diameter of 0.025 m, a length of 0.4 m, and a volume of 196.40 L. The revised text as follows: “The continuous fixed bed reactor was used to investigate the catalytic activity of NaM, M-ZrP1, M-ZrP2, and M-ZrP3 catalysts

		on dimethyl ether production via dehydration of methanol. The volume capacity of the reactor was 196.40 L, with an inner diameter and length of 0.025 m and 0.4 m, respectively.”
3	According to the NH3-TPD method, the authors showed that there were strong and weak acid sites, and in particular on the Lewis acid sites. It is preferable to know the distributions of these acids by methods (example: IFTR adsorption of pyridine or others) to better compare the active catalysts like M-ZrP2 and M-ZrP2).	Thank you for pointing this out. We have agreed with the reviewer. It has been reported that we can see the Lewis and Bronsted acid sites can be distinguished at a certain range of wavenumber by using FTIR-pyridine adsorbed or other probes. However, in our study, this would not be possible to conduct additional experiments for FTIR-adsorbed pyridine due to the covid pandemic, which limits working hours, it takes a relatively long time to be analyzed with a long queue in our region. Furthermore, the sample must be sent to the outer region, which takes a relatively long time. However, the FTIR-adsorbed pyridine only showed the presence of Lewis or Bronsted acid sites, not the strength (i.e., weak, strong, or moderate acids). NH_3-TPD was more favorable to reveal those properties, regardless, the FTIR-adsorbed pyridine is still necessary, as suggested by the reviewer.
4	The authors must explain clearly in the text, the participation of the ziconium-phosphate solution in the effect of the acidity of the catalysts.	Thank you for pointing this out. The new Brønsted acid site was introduced through the presence of a phosphate species as well as Lewis acid sites through the presence of zirconium. Some studies reported that the attainability of geminal P(OH) groups that interacted with the zirconium considerably affected the acidity of the catalyst [1, 2].

		Reference: 1. Weingarten R, Kim YT, Tompsett GA, et al (2013) Conversion of glucose into levulinic acid with solid metal(IV) phosphate catalysts. J Catal 304:123–134. https://doi.org/10.1016/j.jcat.2013.03.0230 2. Sinhamahapatra A, Sutradhar N, Roy B, et al (2010) Mesoporous zirconium phosphate catalyzed reactions: Synthesis of industrially important chemicals in solvent- The revised text as follows: "As can be seen in Fig. 4, the acidity properties of bentonite were remarkably enhanced, presumably due to the synergetic effect of the new Brønsted site acid presented by the P(OH) germinal groups as well as the Lewis acid promoted by the zirconium groups [1, 2]. These acidic sites potentially increase the catalytic activity of catalyst towards dehydration reaction."
5	What do you mean by (cc/g) in table 2	Thank you for pointing this out. cc/g is the unit of the pore volume. Some study used cc/g when represent the pore volume [3]. However, cc/g is the non-SI unit of cm^3/g. We have revised it to the SI units. References: 3. Marini AT, Wijaya K, Sasongko NA (2018) Synthesis of H/Bentonite and Ni/Al2O3-bentonite and its application to produce biogasoline from nyamplung seed (Calophyllum inophillum Linn) oil by catalytic

		hydrocracking. IOP Conf Ser Earth Environ Sci 124:0–6. https://doi.org/10.1088/1755-1315/124/1/012009
6	In the synthesis part, the catalysts were calcined at 450 °C, and the catalytic application was carried out at temperatures between 150 and 350 °C. I would like to know an explanation of what stability the authors are talking about and at what temperature the synthesized catalysts resist.	Thank you for pointing this out. As the temperature catalyst calcination (450 ˚C) was higher than the temperature of catalytic application, it would not resist the catalyst activity. In this study, we demonstrate the stability of catalyst in time on stream towards methanol conversion. As we state on the manuscript, a prolonged time on steam gradually decreased the catalytic activity towards methanol conversion. The deactivation catalysts were primarily associated with either acid site coverage or clogged pores due to coke formation. Coverage site closure, i.e., the deficiency of the active site by coked deposits, generated the active sites either pores or cavities, was inaccessible for catalyzing the methanol conversion. Reactants were adsorbed to the catalytic site within the pore, but when pores are clogged by deposits of carbon compounds on cavities or channel junctions, the pores cannot be accessed by the reactants. It appears to be most of the strong acid sites in the M-ZrP1 catalyst are deactivated by pore-clogging, and the site of the medium and strong acids are responsible for catalyst stability. The medium/strong acid site of M-ZrP1 was more than M-ZrP2. Hence, the deactivation was more dominant in the M-ZrP1 catalyst.
7	The authors must add an explanation of the variations of the percentage of the elements Al, Si, and Fe during the exchange with the solutions compared to the effect of the acidity of the catalysts	Thank you for pointing this out. As EDX is a semi-quantitative method, we cannot comprehensively discuss regarding the effect of the phosphate precursor on particular elements analyzed by EDX. More accurate method (ICP-MS or XRF) should be done in order to conclude the effect of acidity on the elemental results optimally. However, in this analysis, we have revealed the presence co-existed

		of phosphor and zirconium, which strongly indicated the successful bentonite modification. This presumption also has been explained by other studies when modifying the bentonite [4–6]. Nevertheless, we have added more explanation regarding the Si/Al ratio, and the decrease of exchangeable cation after modification. The revised text as follows: "Moreover, bentonite's Si/Al ratios decreased after modification using zirconium phosphate, which corroborated the success of bentonite's modification [37]" "Tomul [39] stated that the decrease of exchangeable cation, i.e., calcium, iron, and magnesium, suggested the successive bentonite's modification." References: 4. Soliemanzadeh A, Fekri M (2017) The application of green tea extract to prepare bentonite-supported nanoscale zero-valent iron and its performance on removal of Cr(VI): Effect of relative parameters and soil experiments. Microporous Mesoporous Mater 239:60–69. https://doi.org/10.1016/j.micromeso.2016.09.050 5. Ain QU, Rasheed U, Yaseen M, et al (2020) Superior dye degradation and adsorption capability of polydopamine modified Fe3O4-pillared bentonite composite. J Hazard Mater 397:122758.

		https://doi.org/10.1016/j.jhazmat.2020.122758 6. Ayari F, Manai G, Khelifi S, Trabelsi-Ayadi M (2019) Treatment of anionic dye aqueous solution using Ti, HDTMA and Al/Fe pillared bentonite. Essay to regenerate the adsorbent. J Saudi Chem Soc 23:294–306. https://doi.org/10.1016/j.jscs.2018.08.001
8	The BET method shows that the catalysts obtained are of type II according to the classification and that the catalysts are non-porous. I find that the pores are very important (79 A) (table2), contrary to the results obtained	Thank you for pointing this out. We have made mistake and agreed to the review. We have deleted the "non-porous" term.
9	The results of the NH3-TPD showed that the strong acid sites were detected after 350C°, whereas the catalytic test was carried out at 150-350C°	Thank you for pointing this out. The experimental of NH_3-TPD was independently not related to the catalytic text. The NH_3-TPD was used to know the strength and the presence of the Lewis and Bronsted acid sites.
10	I find that the specific surface area obtained is low compared to the results obtained on the specific surface by other research (add references).	Thank you for pointing this out. We want to clarify that we made a typo regarding the units of surface area. It should be m^2/g instead of cm^2/g. The literature shows that the bentonite's surface widely variates, which depends on the source of bentonite (natural or synthetic), the pre-treatment, as well as the modification species. Furthermore, we have compared these results with previously reported. We suggested that our proposed catalyst had adequate comparable increased in the surface area when modifying bentonite using zirconium phosphate.

		The revised text as follows: “The previous report showed that molybdenum phosphide could increase the surface are of Na-bentonite from 52.84 to 63.69 m^2/g [28]. Marini et al. [45] reported that the H/bentonite and Ni/Al_2O_3/bentonite had a surface area of 79.08 and 37.63 m^2/g, respectively, whereas the Cr-ZrO_2-bentonite prepared by Wijaya et al. [47] had 105.80 m^2/g which higher than as-prepared HF-bentonite (96.64 m^2/g).”

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

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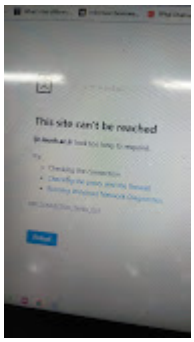

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