

REAC-D-23-00011 - Submission Confirmation for Catalytic dehydration of 2-propanol over nickel phosphide immobilized on natural bentonite - [EMID:602735fa9821011a]

1 pesan

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Dear Dr Hasanudin,

Your submission entitled "Catalytic dehydration of 2-propanol over nickel phosphide immobilized on natural bentonite" has been received by journal Reaction Kinetics, Mechanisms and Catalysis

The submission id is: REAC-D-23-00011

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Thank you for submitting your work to Reaction Kinetics, Mechanisms and Catalysis.

Kind regards,

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REAC-D-23-00011 - Manuscript entitled Catalytic dehydration of 2-propanol over nickel phosphide immobilized on natural bentonite returned to author - [EMID:be25c0794d06a8cf]

1 pesan

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Dear Dr Hasanudin,

The above mentioned manuscript cannot start the review process until the following corrections are made to meet the journal's requirements:

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Please edit your submission and make the necessary changes by logging into the Editorial Manager at: https://www.editorialmanager.com/reac/

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You must then click on "Edit Submission", make the necessary changes, upload your revised manuscript, remove your old manuscript, and approve your submission.

If you have any questions, please do not hesitate to contact me.

Kind regards,

Gábor Lente, PhD. Editor in Chief Reaction Kinetics, Mechanisms and Catalysis

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REAC-D-23-00011 - Author Approve Changes or submits updated ms by author - [EMID:94d8159ced0e3ea0]

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The submission id is: REAC-D-23-00011

Dear Dr Hasanudin,

Re: Catalytic dehydration of 2-propanol over nickel phosphide immobilized on natural bentonite

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REAC: major revision requested for manuscript Catalytic dehydration of 2-propanol over nickel phosphide immobilized on natural bentonite - [EMID:3e3141a2d52a6cea]

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CC: reac@gamma.ttk.pte.hu, lenteg1206@gmail.com

Ref.: Ms. No. REAC-D-23-00011 Catalytic dehydration of 2-propanol over nickel phosphide immobilized on natural bentonite Reaction Kinetics, Mechanisms and Catalysis

Dear Dr Hasanudin,

You are kindly requested to submit a major revision of the above-mentioned manuscript, which was evaluated by two reviewers. Their opinions are appended below.

You will see that both reviewers propose a revision of this submission. The advice seems reasonably clear.

Please revise the manuscript according to these suggestions. In an accompanying cover letter, summarize what changes have been made. If you choose not to modify the manuscript in response to a particular comment, state the reasons why you think it is unnecessary. The revision will probably be sent back to the reviewers for re-review.

Your revision is due by 13-02-2023.

To submit a revision, go to https://www.editorialmanager.com/reac/ and log in as an Author. You will see a menu item call Submission Needing Revision. You will find your submission record there.

Yours sincerely

Gábor Lente, PhD. Editor in Chief Reaction Kinetics, Mechanisms and Catalysis

Reviewers' comments:

Reviewer #1: Overall Recommendation: Major Revision

The work deals with the synthesis of nickel phosphide bentonite to form a catalyst composite for the catalytic dehydration of 2-propanol by a simple wet impregnation method. The authors have used a variety of characterization techniques (XRD, FT-IR, SEM-EDS, NH3 gravimetric method and surface/textural analysis), and some of the work is perhaps interesting, especially the high reusability potential of the NiP/bentonite system, but the manuscript in its present form requires major revision.

1. What explains the large reduction in Si content (Table 1) during impregnation of bentonite with NiP, while no significant reduction is seen for the other elements?

2. For the EDS measurements, the values measured for O and C atoms should be disregarded, as the instrument has a large uncertainty for these light elements.

3. For N2 adsorption-desorption measurements, it is necessary to present measured data (surface area, pore diameter, pore volume) for all catalysts, not just one, in order to accurately assess the catalytic results, as was the case with the acidity measurements!

4. Figure 5 and Table 4 should be supplemented with standard deviation/error values, as there were relatively small

4/6/23, 3:13 PM Email Sriwijaya University - REAC: major revision requested for manuscript Catalytic dehydration of 2-propanol over nickel phosphi...

differences between catalyst performances, and in addition with DIPE selectivity or 2-propanol conversion values.

5. The authors mention that the very small decrease in catalytic activity during reuse is probably related to the leaching of active sites, which is worthwhile to confirm by analytical measurements.

6. Some common typing mistakes: The NiP species was not altered (highlights), $2\theta = 26.43^{\circ}$ and 29.23° (page 6), absorption band at 3582 cm-1 (Fig. 1a) corresponds (page 7), NiP/bentonite (Fig.3b). (page 8)

Reviewer #2: Overall Recommendation: Minor Revision

The reviewed paper deals with the reaction of 2-propanol decomposition to diisopropyl ether. The development of catalysts working in this process is recommended due to the important application of DIPE as a gasoline additive.

I recommend this paper for publication in Reaction Kinetics, Mechanisms and Catalysis, but I have several remarks:

1. In the experimental section more details concerning the characterization techniques should be given, for example: XRD - 2theta range of measurements, scan step etc.

N2 ads/des isotherms - the conditions of thermal treatment of catalysts before measurements

FTIR - range of measurements, the sample preparation (sample with KBr, fresh sample, sample after thermal treatment before measurements.....)

2. I suggest to remove decimal numbers and leave whole numbers for BET surface area (Table 2).

3. The Authors concluded: The total acidity of Na-bentonite gradually increased as the NiP loading increased, presumably due to the availability of Lewis acid sites from Ni and Brønsted acid sites from the phosphor species. Do the Authors know what is the LAS/BAS ratio in the catalysts (e.g. on the basis of FTIR studies with pyridine adsorption)?

4. In is known that ether is formed from 2-propanol on the pairs of Lewis acid-base centers. Did the Authors consider the role of basic sites (from zeolite)?

5. The Table/Figure showing the conversion, selectivity and yield will be useful. I am interested if using the catalysts studied the other products except DIPE were also formed. This information must be added to the amended version of manuscript.

Moreover, the catalytic results for Ni (8 mEq/g Ni) -zeolite (without P) and P-zeolite (without Ni) should be shown for comparison.

6. Other mistakes:

Page 7 - The IR bands characteristic of functional groups of parent Na-bentonite and NiP/bentonite are shown in Fig.2 Page 7 - adsorbed OH groups

Table 1- Na-Bentonite

Fig. 4. N2 adsorption-desorption isotherms

Fig. 11 (...) which promoted more reactant to be adsorbed on the surface

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: Catalytic dehydration of 2-propanol over nickel phosphide immobilized on natural bentonite

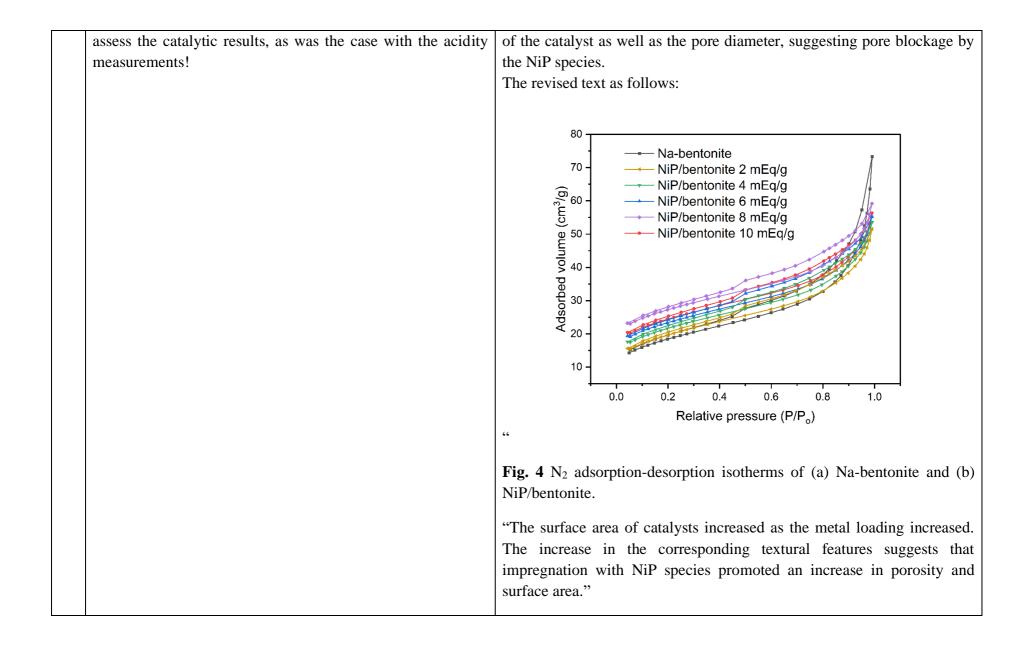
Manuscript ID: REAC-D-23-00011 Thank you for giving us the opportunity to submit a manuscript titled "Catalytic dehydration of 2-propanol over nickel phosphide immobilized on natural bentonite" for publication in the Reaction Kinetics, Mechanisms and Catalysis. We appreciate the time and effort that 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** The work deals with the synthesis of nickel phosphide bentonite to form a catalyst composite for the catalytic dehydration of 2-propanol by a simple wet impregnation method. The authors have used a variety of characterization techniques (XRD, FT-IR, SEM-EDS, NH3 gravimetric method and surface/textural analysis), and some of the work is perhaps interesting, especially the high reusability potential of the NiP/bentonite system, but the manuscript in its present form requires major revision Comment from Reviewer No Responses

-	What explains the large reduction in Si content (Table 1) during impregnation of bentonite with NiP, while no	Thank you for pointing this out. The reduction in Si content happened presumably due to interaction between (NH4) ₂ HPO ₄ acidic solution with bentonite during the impregnation. Since the EDX is a semiqualitative
	significant reduction is seen for the other elements?	analysis, we could not make further precise interpretations regarding this
		phenomenon. However, the same results were also reported by another
		study, which showed that the Si content on SiO_2 decreased from 24.09%

Title

		 to 14.84% after being impregnation by NiMo [1]. Similarly, Siregar et al. [2] reported that there was a decrease in the Si content (25.52 % mass) of CTAB-Montmorilonite to 11.56 % mass after being modified by Fe3O4. Meanwhile, Wijaya et al. [3] reported a decrease in Si content on modified SiO₂ due to increased C and Na content from the addition of NaHCO₃. References: Wijaya K, Nadia A, Dinana A, et al (2021) Catalytic hydrocracking of fresh and waste frying oil over ni-and mo-based catalysts supported on sulfated silica for biogasoline production. Catalysts 11 https://doi.org/10.3390/catal11101150 Siregar SH, Wijaya K, Kunarti ES, Syoufian A (2018) Synthesis and characteristics of the magnetic properties of Fe3O4-(CTAB-montmorillonite) composites, based on variation in Fe3+/Fe2+ concentrations. Orient J Chem 34:716–722. https://doi.org/10.13005/ojc/340213 Wijaya K, Saputri WD, Aziz ITA, et al (2021) Mesoporous Silica Preparation Using Sodium Bicarbonate as Template and Application of the Silica for Hydrocracking of Used Cooking Oil into Biofuel. Silicon. https://doi.org/10.1007/s12633-021-00946-3
2	For the EDS measurements, the values measured for O and C atoms should be disregarded, as the instrument has a large uncertainty for these light elements.	Thank you for pointing this out. We have agreed with the reviewer that the EDS measurements have a large uncertainty for O and C atoms. However, the total number of elements is not 100%, which might result in a bias for the reader. The total EDS measurements should be 100%. Since our parent aluminosilicate (bentonite) was taken from nature, elements such as O and

		C possibly existed. Some studies also displayed the values measured for O
		and C atoms for aluminosilicate-based materials [1–5].
		1. Agustina TE, Anakotta AR, Widhaningtyas H, Gayatri R (2022)
		The effect of weight ratio of ZnO and natural zeolite on the performance
		of ZnO-natural zeolite nanocomposites for synthetic dyes degradation.
		Mater Today Proc 63:S66–S72.
		https://doi.org/10.1016/j.matpr.2022.01.284
2. Kurniawan T, Nuryoto, Rahmayetty (2020) Charao		
		application of bayah natural zeolites for ammonium capture: Isotherm and kinetic. Mater Sci Forum 988 MSF:51–64.
		kinetic. Mater Sci Forum 988 MSF:51–64. https://doi.org/10.4028/www.scientific.net/msf.988.51
		3. Cheng J, Zhang Z, Zhang X, et al (2019) Sulfonated mesoporous
		Y zeolite with nickel to catalyze hydrocracking of microalgae biodiesel
		into jet fuel range hydrocarbons. Int J Hydrogen Energy 44:1650–1658.
		https://doi.org/10.1016/j.ijhydene.2018.11.110
		4. Wibowo E, Rokhmat M, Sutisna, et al (2017) Identification of
		natural zeolite from Sukabumi, West Java, Indonesia: Structure, chemical
		composition, morphology and molecular vibration. Mater Res Express
		4:064002. https://doi.org/10.1088/2053-1591/aa731d
		5. Gea S, Haryono A, Andriayani A, et al (2020) The stabilization of
		liquid smoke through hydrodeoxygenation over nickel catalyst loaded on
		sarulla natural zeolite. Appl Sci 10:.
		https://doi.org/10.3390/APP10124126
	For N2 adsorption-desorption measurements, it is necessary to present measured data (surface area, pore diameter, pore volume) for all catalysts, not just one, in order to accurately	Thank you for pointing this out. We have added the BET measurements of
3		other catalysts, as suggested by the reviewer. We found that as the metal
		loading increased, the surface area, as well as the pore diameter increased.
		However, prolonged metal loading (10 mEq/g) decreased the surface area



"However, prolonged metal loading (10 mEq/g) decreased the surface area of the catalyst as well as the pore diameter, suggesting the pore blockage by the NiP species. According to Table 4, catalyst with NiP metal loading of 8 mEq/g exhibited the most optimum textural features that provided the catalytic reaction of 2-propanol conversion towards DIPE product."

Catalant	Surface area	Pore diameter	Pore volum
Catalyst	(m^2/g)	(Å)	(cm ³ /g)
Na-bentonite	51	52.31	0.063
NiP/bentonite 2 mEq/g	55	54.12	0.075
NiP/bentonite 4 mEq/g	56	54.64	0.094
NiP/bentonite 6 mEq/g	61	60.24	0.079
NiP/bentonite 8 mEq/g	67	66.68	0.113
NiP/bentonite 10 mEq/g	65	64.21	0.102

 Table 2 Textural features of Na-bentonite and NiP/bentonite

		Thank you for pointing this out. We have added the error bar for Fig. 5 and Table 4, respectively.
4	Figure 5 and Table 4 should be supplemented with standard deviation/error values, as there were relatively small differences between catalyst performances, and in addition with DIPE selectivity or 2-propanol conversion values.	
		Fresh 68.50 ± 0.16
		1 68.05 ± 0.11
		2 67.51 ± 0.17

		3 66.21 ± 0.10
5	The authors mention that the very small decrease in catalytic activity during reuse is probably related to the leaching of active sites, which is worthwhile to confirm by analytical measurements.	Thank you for pointing this out. We could not confirm exactly which species are leached during the regeneration due to lack of instrumentation. In this context, we reanalyzed the acidity value of reused catalyst using the same procedure as we mentioned in the methods section. We found that the acidity value of the first reused catalyst was relatively decreased compared with the fresh catalyst. Moreover, the prolonged use of catalysts also exhibited the same trend. This condition justifies our assumption regarding the loss of the catalyst's active site during the regeneration process. Furthermore, the change of textural features, such as pore clogged, may also be involved in the reduction of catalyst performance. However, we cannot confirm this phenomenon since the SEM analysis takes a lot of time and queue in our region. The revised text as follows: "The acidity value of the reused catalyst was further evaluated to confirm the relation between the acidity value of the catalyst with the decreased catalyst activity after three consecutive runs. The first reused catalyst decreased catalyst acidity from 4.63 mmol/g (fresh catalyst) to 4.59 mmol/g and tended to decrease up to 4.56 mmol/g after two consecutive runs. Moreover, the catalyst at three times reused showed a decrease in the acidity value to 4.49 mmol/g. The regeneration process could decrease catalyst acidity, implying less catalyst active site. Consequently, the catalyst performance also decreased."
6	Some common typing mistakes: The NiP species was not altered (highlights), $2\theta = 26.43^{\circ}$ and 29.23° (page 6),	Thank you for pointing this out. We have revised the text accordingly.

	absorption band at 3582 cm-1 (Fig. 1a) corresponds (page 7),	The revised text as follows:
	NiP/bentonite (Fig.3b). (page 8)	"The NiP species were not altered"
		"26.43° and 29.23°"
		"The absorption band at 3582 cm-1 (Fig. 2a)"
		"was related to the stretching vibration of the metal-OH"
		"SEM micrograph of NiP/bentonite (Fig. 3b)."
Reviewer 2		1
	The reviewed paper deals with the reaction of 2-propanol decomposition to diisopropyl ether. The development of catalysts working this process is recommended due to the important application of DIPE as a gasoline additive. I recommend this paper for publication in Reaction Kinetics, Mechanisms and Catalysis, but I have several remarks:	
	recommend and paper for pronoution in reduction remoted, is	AECHAILINHIN AHO V ALAIVNIN DHI I HAVE NEVELAL LEHIALKN
No	Commont from Deviewor	
No	Comment from Reviewer	Responses
No	In the experimental section more details concerning the	
No		Responses Thank you for pointing this out. We have revised the text as suggested by

		"The textural properties of the catalysts were evaluated using Quantachrome NOVA. Prior to analysis, the catalyst was vacuum degassed up to 300 °C for 1 h with a heating rate of 10 C°/min. The surface areas of the catalysts were calculated using a multipoint BET procedure"
2	I suggest to remove decimal numbers and leave whole numbers for BET surface area (Table 2).	Thank you for pointing this out. We have removed decimal numbers from BET surface area accordingly.
3	The Authors concluded: The total acidity of Na-bentonite gradually increased as the NiP loading increased, presumably due to the availability of Lewis acid sites from Ni and Brønsted acid sites from the phosphor species. Do the Authors know what is the LAS/BAS ratio in the catalysts (e.g. on the basis of FTIR studies with pyridine adsorption)?	Thank you for pointing this out. It would have been interesting to explore this aspect. The acidity features of the catalyst can be provided by pyridine adsorption. This method could distinguish the Lewis and Bronsted acid sites of the catalyst. Theoretically, the Ni species can act as a lewis acid since they provide empty d orbitals, whereas the phosphors can interact with hydrogen and provide a proton that acts as a bronsted acid site. Unfortunately, we did not conduct the FTIR-absorbed experiment, hence we did not know the LAS/BAS ratio in the catalysts. Nevertheless, exploring this aspect in the other study is fascinating and necessary.
4	In is known that ether is formed from 2-propanol on the pairs of Lewis acid-base centers. Did the Authors consider the role of basic sites (from zeolite)?	 Thank you for pointing this out. The role of the acid center is more dominant than the basic sites since this reaction only produced the DIPE. If the basic sites are dominant, they will produce high propylene products. Similar results were also reported in the previous study [1]. 1. Armenta MA, Valdez R, Silva-Rodrigo R, Olivas A (2019) Diisopropyl ether production via 2-propanol dehydration using supported iron oxides catalysts. Fuel 236:934–941. https://doi.org/10.1016/j.fuel.2018.06.138

		Thank you for pointing this out. The GC chromatogram of the product is
		shown below:
5	The Table/Figure showing the conversion, selectivity and yield will be useful. I am interested if using the catalysts studied the other products except DIPE were also formed. This information must be added to the amended version of manuscript. Moreover, the catalytic results for Ni (8 mEq/g Ni) -zeolite (without P) and P-zeolite (without Ni) should be shown for comparison.	Rt 000-48

	ngle Ni catalyst is not characterized and reported their
	Likewise, Wijaya et al. [2] prepared a nickel catalyst 2/SO ⁴ pillared bentonite, and they did not characterize
	nd showed their catalytic activity as the same as other
	support and metal site catalysts [3, 4].
However, it would	have been interesting to explore this aspect, comparing
the other catalysts	
	5
References:	
1. Susi EP,	Wijaya K, Wangsa, et al (2020) Effect of nickel
	atural zeolite as catalyst in hydrocracking process of
used cooking https://doi.org/10.1	oil. Asian J Chem 32:2773–2777. 14233/ajchem.2020.22708
https://doi.org/10.1	14255/ajchem.2020.22706
	Kurniawan MA, Saputri WD, et al (2021) Synthesis of
	supported on ZrO2/SO4pillared bentonite and its aversion of coconut oil into gasoline via hydrocracking
process. J	Environ Chem Eng 9:105399.
1	1016/j.jece.2021.105399
3. Marini A	Г, Wijaya K, Sasongko NA (2018) Synthesis of
	Ni/Al2O3-bentonite and its application to produce
	yamplung seed (Calophyllum inophillum Linn) oil by
	cking. IOP Conf Ser Earth Environ Sci 124:0-6.
https://doi.org/10.1	1088/1755-1315/124/1/012009

		4. Mirzan M, Wijaya K, Falah II, Trisunaryanti W (2019) Synthesis and characterization of Ni/ZrO2-bentonite. Asian J Chem 31:229–234
6	Other mistakes: Page 7 - The IR bands characteristic of functional groups of parent Na-bentonite and NiP/bentonite are shown in Fig.2 Page 7 - adsorbed OH groups Table 1- Na-Bentonite Fig. 4. N2 adsorption-desorption isotherms Fig. 11 () which promoted more reactant to be adsorbed on the surface	Thank you for pointing this out. We have revised the manuscript as suggested by the reviewer. The revised text as follows: "Fig. 4 N ₂ adsorption-desorption isotherms of" "Table 1 Na-Bentonite" "The IR bands characteristic of functional groups of parent Na-bentonite and NiP/bentonite are shown in Fig. 2." "whereas the adsorbed OH groups or water molecules can be found at 1625 cm ⁻¹ " "which promoted more reactant to be adsorbed on the surface"



REAC: PDF Catalytic dehydration of 2-propanol over nickel phosphide immobilized on natural bentonite has been built and requires approval

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Ref.: Ms. No. REAC-D-23-00011R1 Catalytic dehydration of 2-propanol over nickel phosphide immobilized on natural bentonite

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