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

REAC-D-22-00429 - Submission Confirmation for Production of levulinic acid from glucose using nickel phosphate-silica catalyst - [EMID:7e8f2b9ada6b3918]

1 pesan

Editorial Office(REAC) <em@editorialmanager.com> 15 September 2022 pukul 16.02
Balas Ke: "Editorial Office(REAC)" <reac@gamma.ttk.pte.hu>
Kepada: Hasanudin Hasanudin <hasanudin@mipa.unsri.ac.id>

Dear Dr Hasanudin,

Your submission entitled "Production of levulinic acid from glucose using nickel phosphate-silica catalyst" has been received by journal Reaction Kinetics, Mechanisms and Catalysis

The submission id is: REAC-D-22-00429

Please refer to this number in any future correspondence.

You will be able to check on the progress of your paper by logging on to Editorial Manager as an author.
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We have received this manuscript with the understanding that it or its parts have not been published or submitted for publication in another journal. If otherwise, please contact us promptly. After editorial checking, your manuscript will be sent out to independent experts for for review and we will contact you in an e-mail when a decision has been made about publication.

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

Kind regards,

Editorial Office
Reaction Kinetics, Mechanisms and Catalysis

Now that your article will undergo the editorial and peer review process, it is the right time to think about publishing your article as open access. With open access your article will become freely available to anyone worldwide and you will easily comply with open access mandates. Springer's open access offering for this journal is called Open Choice (find more information on www.springer.com/openchoice). Once your article is accepted, you will be offered the option to publish through open access. So you might want to talk to your institution and funder now to see how payment could be organized; for an overview of available open access funding please go to www.springer.com/oafunding.
Although for now you don't have to do anything, we would like to let you know about your upcoming options.

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

REAC-D-22-00429 - Manuscript entitled Production of levulinic acid from glucose using nickel phosphate-silica catalyst returned to author - [EMID:04834e1cf7b10b8b]

1 pesan

Editorial Office(REAC) <em@editorialmanager.com> 17 September 2022 pukul 17.14
Balas Ke: "Editorial Office(REAC)" <reac@gamma.ttk.pte.hu>
Kepada: Hasanudin Hasanudin <hasanudin@mipa.unsri.ac.id>

Dear Dr Hasanudin,

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

- Some of the figures in this submission are not in agreement with the scientific standards we would like to maintain in this journal. These figures contain experimental points and lines. In the high-quality chemical literature, this usually implies that the lines reflect fits to some sort of theoretical equation. However, this is clearly not the case here, the lines merely connect measured points or follow their trend. Do not use both lines and markers simultaneously, markers only are preferred in these cases.

- The number of figures is unreasonably large in this manuscript. Figure numbering is very misleading, the actual overall number of graphs and images is 26. Please make sure you only include figures which are necessary for understanding. I am asking you to reduce the overall number of individual figures to a maximum of 10. The rest should be moved to the Supplementary Information.

- Figure captions are not detailed enough. A figure caption should make it possible for the reader to understand the contents of the figure without consulting the text. The figure caption should also give all experimental conditions that are necessary to reproduce the measurements.

Please edit your submission and make the necessary changes by logging into the Editorial Manager at:
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and clicking on "Submissions Sent Back to Author".

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

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

REAC-D-22-00429 - Submission Confirmation for Production of levulinic acid from glucose using nickel phosphate-silica catalyst - [EMID:7e8f2b9ada6b3918]

1 pesan

Editorial Office(REAC) <em@editorialmanager.com> 15 September 2022 pukul 16.02
Balas Ke: "Editorial Office(REAC)" <reac@gamma.ttk.pte.hu>
Kepada: Hasanudin Hasanudin <hasanudin@mipa.unsri.ac.id>

Dear Dr Hasanudin,

Your submission entitled "Production of levulinic acid from glucose using nickel phosphate-silica catalyst" has been received by journal Reaction Kinetics, Mechanisms and Catalysis

The submission id is: REAC-D-22-00429

Please refer to this number in any future correspondence.

You will be able to check on the progress of your paper by logging on to Editorial Manager as an author.
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We have received this manuscript with the understanding that it or its parts have not been published or submitted for publication in another journal. If otherwise, please contact us promptly. After editorial checking, your manuscript will be sent out to independent experts for for review and we will contact you in an e-mail when a decision has been made about publication.

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

Kind regards,

Editorial Office
Reaction Kinetics, Mechanisms and Catalysis

Now that your article will undergo the editorial and peer review process, it is the right time to think about publishing your article as open access. With open access your article will become freely available to anyone worldwide and you will easily comply with open access mandates. Springer's open access offering for this journal is called Open Choice (find more information on www.springer.com/openchoice). Once your article is accepted, you will be offered the option to publish through open access. So you might want to talk to your institution and funder now to see how payment could be organized; for an overview of available open access funding please go to www.springer.com/oafunding.
Although for now you don't have to do anything, we would like to let you know about your upcoming options.

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

REAC: major revision requested for manuscript Production of levulinic acid from glucose using nickel phosphate-silica catalyst - [EMID:23ebc267a68e2e45]

1 pesan

Editorial Office(REAC) <em@editorialmanager.com> 29 Oktober 2022 pukul 08.33
Balas Ke: "Editorial Office(REAC)" <reac@gamma.ttk.pte.hu>
Kepada: Hasanudin Hasanudin <hasanudin@mipa.unsri.ac.id>

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

Ref.: Ms. No. REAC-D-22-00429
Production of levulinic acid from glucose using nickel phosphate-silica catalyst
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 Reviewer 1 proposes rejection, whereas Reviewer 2 recommends a major revision. The comments see rather detailed and helpful. Overall, I think a suitable revision of this submission may b publihable.

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 28-11-2022.

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: Rejection - Substandard Work

The paper in my view contains several shortcomings preventing from recommending it.
1. the whole idea of experimental design does not really help in understanding the mechanistic aspects of the reaction.
2. Why should the yield of levulinic acid be multiplied by a factor of 2 in eq. (1)?
3. The precision of surface area measurements is not that high, no need to report 67.68 m2/g.
4. It was unclear how acidity was measured, can one discriminate between Bronsted and Lewis acidity?
5. The yields are not that high and they are not that precise. What are the values of TOF/yields reported in the literature?
6. What was mass balance closure? What are the other products?Was formic acid observed? Was CO or H2 detected? Their ratio?
7. In my view eq. (3) does not provide any understanding of the process. each experiment should be used to extract some mechanistic explanations.
8. The authors are interested in statistics, rather than chemistry, while the readers of RKMC in my view are expecting something else.
9. Information of deactivation is absent

10. The productivity of a catalytic reaction is proportional to the mass of catalyst in general. The explanations of the authors regarding the influence of the mass are rather strange.

Reviewer #2:
Overall Recommendation: Major Revision

The paper deals with the production of levulinic acid from glucose using a supported heterogeneous catalyst. The catalyst was synthesized and characterized. The results about this part are convincing and the paper is well written. The second part of the paper deals with the investigation of the reaction kinetics, using a surface methodology. I have several concerns on this part, thus I suggest major revisions of the paper:

- All the results of the optimization investigation are reported in supplementary materials, but they are a big part of the paper and must be included in the paper and deepened.
- The authors are using a DOE approach. This must be stated and discussed, introducing all the approximation and assumptions of the model.
- The scope of the work must be better stated in the introduction.
- Statistical information and definitions must be introduced and discussed in the paper.

—

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

REAC - Your revision is due soon

1 pesan

Gábor Lente <em@editorialmanager.com> 21 November 2022 pukul 09.30
Balas Ke: Gábor Lente <reac@gamma.ttk.pte.hu>
Kepada: Hasanudin Hasanudin <hasanudin@mipa.unsri.ac.id>

CC: "Gábor Lente" reac@gamma.ttk.pte.hu;lenteg1206@gmail.com

Ref.: Ms. No. REAC-D-22-00429
Production of levulinic acid from glucose using nickel phosphate-silica catalyst
Reaction Kinetics, Mechanisms and Catalysis

Dear Dr Hasanudin,

This message has been sent to remind you that we are expecting the revision of REAC-D-22-00429 by 28-11-2022.

If you require more time, please contact the journal office. If you are ready to submit your revision, then please go to the Editorial Manager Website and submit the revision.

Your username is: hasanudin@mipa.unsri.ac.id
If you forgot your password, you can click the 'Send Login Details' link on the EM Login page at https://www.editorialmanager.com/REAC/

Thank you very much.

Kind regards,

Editorial Office
Reaction Kinetics, Mechanisms and Catalysis

Title : Production of levulinic acid from glucose using nickel phosphate-silica catalyst

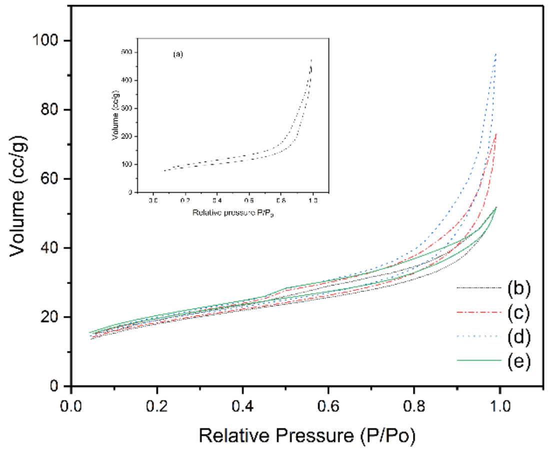
Manuscript ID : REAC-D-22-00429

	Thank you for giving us the opportunity to submit a manuscript titled "Production of levulinic acid from glucose using nickel phosphate-silica catalyst" 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 Overall Recommendation: Rejection - Substandard Work The paper in my view contains several shortcomings preventing from recommending it.	
No	Comment from Reviewer	Responses
1	the whole idea of experimental design does not really help in understanding the mechanistic aspects of the reaction.	Thank you for pointing this out. In this study, prior to optimization using RSM-CCD, we tested a series of catalysts, i.e., Nickel phosphate silica with different phosphate precursors, to convert glucose to LA. This study suggested that different phosphate precursors would generate different catalytic activity towards LA production, in which the acidity features influenced the LA formation. We also have added the parameters of glucose conversion, LA selectivity, as well as formic acid yield and selectivity, respectively, to strengthen the manuscript. Our RSM-CCD provides an understanding of the effect of catalyst loading, temperature, and reaction time on the

		LA yield with each interaction simultaneously and finding the optimum condition. The preliminary study of nickel phosphate-silica and their application for LA production lead us to further potential studies, especially large-scale production studies, and so on
2	Why should the yield of levulinic acid be multiplied by a factor of 2 in eq. (1)?	Thank you for pointing this out. Sorry for this omission, it is not necesarry to multiplied it by a factor 2 when calculating the levulinic acid. We have deleted it accordingly. The revised text as follows: "The glucose conversion (C_x), LA and formic acid yield (Y_x) as well as selectivity (S_x) were calculated according to the Eq. (1-3) as follows: $$C_X=\left(\frac{\text{reacted glocuse mole}}{\text{intial glocuse mole}}\right)\times 100\% \quad (1)$$ $$Y_X=\left(\frac{\text{LA or FA product mole}}{\text{Glucose initial mole}}\right)\times 100\% \quad (2)$$ $$S_X=\left(\frac{\text{LA or FA product mole}}{\text{reacted glucose mole}}\right)\times 100\% \quad (3)$$ "
3	The precision of surface area measurements is not that high, no need to report 67.68 m2/g.	Thank you for pointing this out. We calculated the surface area of all catalysts using multi-plot BET. There were no appreciable changes in the surface area between the prepared catalysts, which suggested that the phosphate precursors did not significantly change the catalyst's textural features. Comparably, we also have added the surface area of the parent SiO2. After being loaded by the nickel and nickel phosphate species, the surface area of the parent SiO2 decreased, which was presumably due to the pore blocking. After we analyzed the acidity features of the catalyst,

		it seemed that the phosphate precursor more influenced the acidity features rather than the surface area of the catalyst. The revised text as follows: "The surface area of the SiO_2 catalyst drastically decreased after being loaded by nickel and nickel phosphate species, which was probably caused by the pore's obstruction." " **Table 2** Catalysts textural properties

Catalysts	BET surface area (m^2/g)	Pore volume (cm^3/g)	Pore diameter (Å)
SiO_2	280.23	0.68	97.06
Ni-SiO_2	62.77	0.08	51.23
Ni_3PO_4-SiO_2	63.76	0.11	70.89
$NiHPO_2$-SiO_2	65.74	0.16	91.27
$Ni(H_2PO_4)_2$-SiO_2	67.68	0.15	88.06

		 "Fig. 4 N2 adsorption-desorption isotherm curves of (a) SiO2, (b) Ni-SiO2, (c) Ni3(PO4)2-SiO2, (d) NiHPO4-SiO2 and (e) Ni(H2PO4)-SiO2 catalysts"
4	It was unclear how acidity was measured, can one discriminate between Bronsted and Lewis acidity?	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. The surface acidity was determined according to the following equation: $\text{Surface acidity (mmol pyridine/g)} = \frac{W_2 - W_1}{(W_1 - W_0) \times \text{MW pyridine}}$ (1) Where W_0 and W_1 are the mass of the porcelain container and porcelain container+sampel, whereas the W_2 is the mass of porcelain container+sampel after the absorption of probe, the MW

		is denoted as the molecular weight of the corresponding probes, we already added citations [34] regarding the measurement of the acidity value of the catalyst in the manuscript. The revised text as follows: "Acidity analysis followed the gravimetric method with pyridine gas, as reported by the previous study [34]." References: 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. Period. Polytech. Chem. Eng. 2022, 66, 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 a-Cellulose Pyrolysis Oil. Orient. J. Chem. 2018, 34, 955–962, doi:10.13005/ojc/340245. Regarding the Bronsted and Lewis acidity, we can discriminate using the FTIR-absorbed pyridine. This analysis would provide some absorption bands corresponding to the catalysts' Bronsted and Lewis acid sites. In this manuscript, those absorption bands' intensities were not that high, presumably due to the pyridine being vaporized out of the catalyst's surface. After we did the experiment, we did not analyze directly through FTIR because it took time and queued. Nevertheless, according to our FTIR-pyridine results, the absorption bands of the Bronsted and Lewis acid sites of our catalyst still existed.

5	The yields are not that high and they are not that precise. What are the values of TOF/yields reported in the literature?	Thank you for pointing this out. We have added the LA yield reported by the other literature. The revised text as follows: "Wei and Wu [12] reported that the catalyst 8%Cr/HZSM-5 promoted 53.7 % LA yield under temperature of 180 ˚C for 180 min with 0.75 g catalyst. Qu et al. [14] employed the Lys-PM2 catalyst and reported that this catalyst promoted the LA yield up to 57.90% under 9 h reaction with temperature of 150˚C with glucose and catalyst ratio of 1:0.25, whereas Kumar et al. [69] reported that the 98% glucose conversion with LA yield of 63% was achieved by ionic liquid catalyst of [C4SO3HPhim][Cl] under 3 h reaction at 180 ˚C. This result suggests that the Ni(H2PO4)2-SiO2 catalyst had comparable catalytic activity towards LA production compared with other catalysts."
6	What was mass balance closure? What are the other products? Was formic acid observed? Was CO or H2 detected? Their ratio?	Thank you for pointing this out. According to other reports, other products such as humin as a byproduct, fructose from isomerization reaction, HMF as an intermediate product from dehydration of glucose or fructose, as well as formic acid from rehydration of HMF, could exist and can be analyzed by HPLC. We have added the yield and selectivity of FA, whereas the fructose and HMF are undetected on the chromatogram. We also have added the glucose conversion results. Regarding the CO or H2, we cannot collect the gas and not analyze it directly since the instrument analysis was unavailable. It was possible to produce the H2 and COX when conducting the dehydration and rehydration due to the FA decomposition at the extension of reaction time and high temperature. Regarding the byproduct, the humins were formed when conducted the experiment, as indicated by the presence of black slurry on the product. Unfortunately, we did not analyze it gravimetrically. It is necessary to explore this aspect in our further study. The revised text as follows: "The conversion of glucose, the yield, and selectivity of LA, as well as FA, are presented in Fig. 6. It was revealed that all catalysts exhibited high conversion glucose ranging from 96.32-

		99.82%, with the LA and FA observed as a major product. The maximum glucose conversion was attained by the Ni(H2PO4)2-SiO2 (99.82%), whereas other catalysts showed no significant difference in the glucose conversion." "Similarly, the Ni-SiO2 exhibited selectivity towards LA (0.10%), followed by the Ni3(PO4)2-SiO2, which only had 2.05% selectivity towards LA. The highest LA selectivity (26.49%) was achived by Ni(H2PO4)2-SiO2 catalyst. Weingarten et al. [20] reported that high catalyst acidity could exhibit high catalytic activity towards LA fornation. Interestingly, the Ni3(PO4¬)2-SiO2 catalyst promoted high FA yield (64.53%) and selectivity, whereas the other catalysts revealed low FA formation. This condition occurred probably due to the presence of micropores, limiting the diffusion of LA molecules so that their formation was not dominant. Because FA is a smaller molecule, consequently the formation of FA is higher than LA [17].
7	In my view eq. (3) does not provide any understanding of the process. Each experiment should be used to extract some mechanistic explanations	Thank you for pointing this out. Eq. (3) is a quadratic equation utilized as a model for RSM-CCD. This model provided a whole idea of the design of the experiment, generating the interaction between the variable input, i.e., catalyst loading, temperature, and time reaction as input variables to the response, i.e., levulinic acid yield, through the 3D surface plots and 2D contour plots. Several study the utilization of RSM-CCD have been published on Reaction Kinetics, Mechanisms and Catalysis, which indicates that this study is still worthy of consideration [1–6]. Our study explains what condition LA could exhibit either a maximum or minimum yield. Prior to optimization, we conducted the experiment by varying the catalyst type and studying their catalytic activity towards LA production, which is not part of the DoE. In this part, we explain how different phosphate precursors could generate different catalytic activity toward LA production, which is presumably due to different acidity features. Afterwards, the catalyst which provided the highest catalytic performance were optimized under DoE. References: 1. Asadi-Ghalhari M, Mostafaloo R, Ghafouri N, et al (2021) Removal of Cefixime from aqueous solutions via proxy electrocoagulation: modeling and optimization by response

		surface methodology. React Kinet Mech Catal 134:459–471. https://doi.org/10.1007/s11144-021-02055-z 2. Mayorga Betancourt MA, López Santamaria CA, López Gómez M, Gonzalez Caranton AR (2020) Experimental analysis of biodiesel synthesis from palm kernel oil: empirical model and surface response variables. React Kinet Mech Catal 131:297–317. https://doi.org/10.1007/s11144-020-01860-2 3. Yashni G, Al-Gheethi A, Mohamed R, et al (2020) Photodegradation of basic red 51 in hair dye greywater by zinc oxide nanoparticles using central composite design. React Kinet Mech Catal 130:567–588. https://doi.org/10.1007/s11144-020-01792-x 4. Elfghi FM, Amin NAS (2014) Applying response surface methodology to assess the combined effect of process variables on the composition and octane number of reformat in the process of reducing aromatization activity in catalytic naphtha reforming. React Kinet Mech Catal 111:89–106. https://doi.org/10.1007/s11144-013-0624-8 5. Zhang H, Li Y, Lu Z, et al (2017) Highly efficient synthesis of biodiesel catalyzed by CF3SO3H-functionalized ionic liquids: experimental design and study with response surface methodology. React Kinet Mech Catal 121:579–592. https://doi.org/10.1007/s11144-017-1171-5 6. Waghmare G V., Mudaliar C, Rathod VK (2020) Optimization of the enzyme catalyzed ultrasound assisted synthesis of cinnamyl butyrate using response surface methodology. React Kinet Mech Catal 129:421–441. https://doi.org/10.1007/s11144-019-01697-4 7. Qu H, Liu B, Gao G, et al (2019) Metal-organic framework containing BrØnsted acidity and Lewis acidity for efficient conversion glucose to levulinic acid. Fuel Process Technol 193:1–6. https://doi.org/10.1016/j.fuproc.2019.04.035
8	The authors are interested in statistics, rather than chemistry, while the readers	Thank you for pointing this out. Indeed, in this study, we utilized the RSM-CCD as the mathematical-statistics application for optimizing the LA yield with catalyst loading,

	of RKMC in my view are expecting something else.	temperature, and time reaction as input variables. This method can explain one more variable and their interaction simultaneously, which is different from the one variable at a time (OVAT) which is rather beneficial in the context of optimization. We realized that the RSM-CCD is just a tool but not the main aspect. However, in this study, we have explained the interaction of those variables to the corresponding parameters thoroughly, not just the statistical stuff. Further, we also characterized the catalysts using various instruments, which we thought had something to do with chemistry. We hope this answer can satisfy the reviewer's consideration.
9	Information of deactivation is absent	Thank you for pointing this out. We have added the information regarding the reusability of the catalysts. The revised text as follows: "In order to evaluate the catalyst reusability, the spent catalyst was regenerated through the washing process using water followed by heat treatment. The spent catalysts were dried at 100 ˚C for 24 h and calcinated at 700 ˚C for 4 h to discharge the humin absorbed on the catalyst." "As presented in Table 5, the reusability performance of the $Ni(H_2PO_4)_2$-SiO_2 catalyst on the glucose conversion and LA yield was evaluated at 5 consecutive runs under optimized conditions. It can be seen that at 2 consecutive reactions, the catalytic activity of the catalyst only decreased by 1.15% compared with the first catalyst tests. Afterward, the catalytic activity tended to decrease up to 3.12% at 3 consecutive runs and gradually decreased at 5 consecutive runs. The decrease in LA yield was presumably due to the loss of active catalytic sites during the multiple cleaning steps [82]. However, this catalyst was still active at 5 consecutive runs with a slight decrease toward LA formation" **Table 6** Reusability performance of $Ni(H_2PO_4)_2$-SiO_2 catalyst for LA fornation

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<tr><th>Run</th><th>LA yield (%)</th></tr>
<tr><td>1st</td><td>40.70</td></tr>
<tr><td>2nd</td><td>40.25</td></tr>
<tr><td>3rd</td><td>39.43</td></tr>
<tr><td>4th</td><td>36.53</td></tr>
<tr><td>5th</td><td>34.46</td></tr>
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<td>The productivity of a catalytic reaction is proportional to the mass of catalyst in general. The explanations of the authors regarding the influence of the mass are rather strange</td>
<td>Thank you for pointing this out. The acidity value affected the catalytic activity of nickel phosphate-silica on the glucose conversion to LA production, in which the higher acidity led to a higher LA yield. This condition is also consistently reported by other studies [7, 8]. The textural features may affect catalytic activity, but in this study, the influence of catalyst acidity is likely dominant.

References:

7. Qu H, Liu B, Gao G, et al (2019) Metal-organic framework containing BrØnsted acidity and Lewis acidity for efficient conversion glucose to levulinic acid. Fuel Process Technol 193:1–6. https://doi.org/10.1016/j.fuproc.2019.04.035

8. 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.2013.03.023</td>
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<tr><td></td><td colspan="2">Reviewer 2

Overall Recommendation: Major Revision

The paper deals with the production of levulinic acid from glucose using a supported heterogeneous catalyst. The catalyst was synthesized and characterized. The results about this part are convincing and the paper is well written. The second part of the paper deals with the investigation of the reaction kinetics, using a surface methodology. I have several concerns on this part, thus I suggest major revisions of the paper:</td></tr>
<tr><td>1</td><td>All the results of the optimization investigation are reported in supplementary materials, but they are a big part of the paper and must be included in the paper and deepened</td><td>Thank you for pointing this out. We have moved the Figure of the 3D surface and the 2D contour plots (Fig. 7) from the supplementary materials to the main text, as suggested by the reviewer.</td></tr>
<tr><td>2</td><td>The authors are using a DOE approach. This must be stated and discussed, introducing all the approximation and assumptions of the model</td><td>Thank your for pointing this out. The approximation and assumptions of the model have been described on the Experimental design section. This study employed the RSM-CCD with 20 runs. The model was tested by the ANOVA and the data from the model was validated. The results of the RSM-CCD model are mentioned in the RSM-CCD analysis section on the manuscript. It shows that the P-value of the model was 0.0051 and the F-value was 5.98, indicating that the model was statistically significant and represented good experimental data.

The text that relating to the reviewer suggestion as follows:</td></tr>
</table>

		"The statistical diagnostic by RSM-CCD to optimize LA yield are shown in the Supplementary Information. Internal studentized of the residues with normal probability showed that all points were close to the line, indicating no major problems with the design normality [9] According to Tan et al. [10], normally distributed data presented in a straight line represents insignificant errors in the range of operating parameters. Random scattering data in the studentized residues versus predited LA yield plot showed that the proposed model was adequate. These results demonstrated that the response had original variance observation, and there was no issue with the output variable [11]. Behera et al. [12] stated that if data are randomly scattered, the variation of the original observation should be constant and there is no need for a transformation of the response variable. The actual and predicted value for LA yield showed that the predicted values were consistently distributed close to the actual response. According to Garg and Jain [13], the suggested minimum value of R^2 should be 0.80 for a good model fit. In this study, the R^2 value was 0.8424, indicating that the model could evaluate up to 84.24% LA yield validated by the equation. Hence, there was a reasonable in accordance between the experimental LA yield results and the predictions from the quadratic model [14]. The outlier t plot represented the eminence residual of each run to specify which run was the individually dominant residual. According to Helmi et al. [9], the preponderance of the residuals should fall within the range +3.879 and -3.879 to represent operational errors in the model and actual data. In this study, the outliers from the experimental run obviously showed that all focuses in the outlier range had a good dissemination for CCD design and no data was outside this interval."
3	The scope of the work must be better stated in the introduction	Thank you for pointing this out. In this study, we studied two major aspects. First, the facile fabrication of nickel phosphate-silica with various phosphate precursors and evaluate their catalytic activity towards LA production. Second, the catalysts that provided the highest catalytic activity would be further optimized based on the design of the experiment (DoE) using response surface methodology with a rotatable central composite design.

		The revised text as follows: "This research explores two primary aspects, starting with the fabrication of nickel phosphate silica using different phosphate precursors (ammonium dihydrogen phosphate, diammonium hydrogen phosphate, and tributyl phosphate) and evaluating their catalytic activity in the production of LA from glucose. The catalyst with the best properties and highest yield would be further investigated for reaction temperature, reaction time, and catalyst weight effects on LA yield through the design of experiment (DoE) using response surface methodology (RSM) with rotatable central composite design (RCCD)"
4	Statistical information and definitions must be introduced and discussed in the paper.	Thank you for pointing this out. In this study we employed the Design of Experiment (DoE) using response surface methdology with central composite design. As we described in the experimental design section from the manuscript, analysis of variance (ANOVA) was employed to examine the experimental result as well as the model. The statistical significance was assessed using F-test and P-value (95% confidence level). Analysis of regression was conducted to investigate the Y response function, as formulated in Eq. (2) as follows: $Y=\beta_o+\sum_{i=1}^{k}\beta_i X_i+\sum_{i=1}^{k}\beta_{ii} X_i^2+\sum_{i=1}^{k-1}\sum_{j=2}^{k}\beta_{ij} X_i X_j+\varepsilon$ (2) where i and j are linear and quadratic coefficients, respectively, βo was the coefficient of regression, and k was the number of independent variables studied. Regarding to obtain the experimental data, 8 factorial, 6 axial, and 6 central runs (20 total experiments) were selected in this study. The center point was repeated six times to calculate the error and standard deviation. Each variable was coded ±1, 0, and ±α (±1.682) for the factorial, center, and axial points, respectively.

		The statistical information has been comprehensively discussed in the RSM-CCD analysis section. We reported that the the quadratic polynomial equation (in the form of coded values) from the multiple linear regression analysis is presented in the Eq. (3) as follows: $$Y = 35.72 - 1.55A - 5.94B + 1.89C - 0.3143AB + 2.13AC + 3.38BC - 1.35A^2 - 1.39B^2 - 5.17C^2 \quad (3)$$ According to Eq. (2), AB, AC, AD, BC, BD, as well as CD are denoted as the interaction terms, whereas A^2, B^2, C^2, as well as D^2 are the square terms of the input variables. Based on Table 5 (ANOVA) on the manuscript, the P-value of the model was 0.0051 and the F-value was 5.98, indicating that the model was statistically significant and represented good experimental data, whereas the statistical diagnostic by RSM-CCD to optimize LA yield also well descirbed and cited a proper reference. We have added the text regarding the stastisticall information in the introduction section as follows: "The catalyst with the best properties and highest yield would be further investigated for reaction temperature, reaction time, and catalyst weight effects on LA yield through the design of experiment (DoE) using response surface methodology (RSM) with rotatable central composite design (RCCD). In this regard, the quadratic polynomial equation was utilized as a model and subsequently examined using ANOVA (95% confidence level). The model was statistically diagnostics using various parameters and validated. The 3D surface and 2D contour model graphs were plotted, and the interaction of variables on the response was thoroughly studied."

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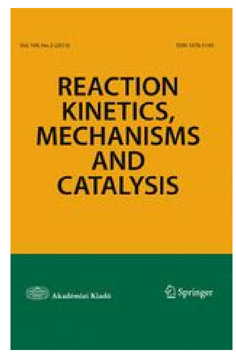

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