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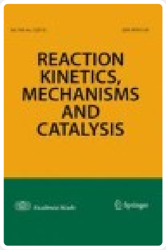
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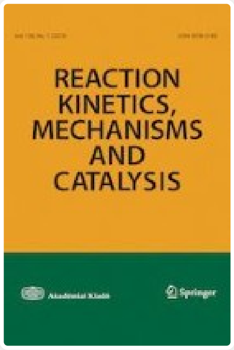
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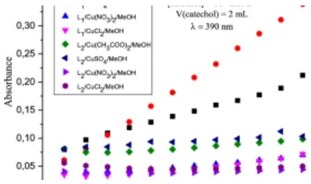

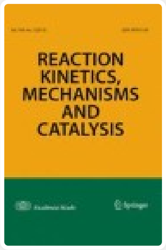

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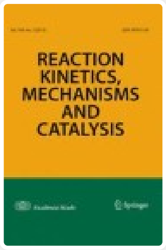

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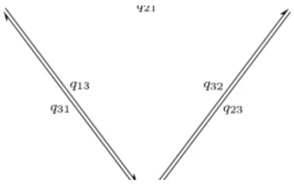

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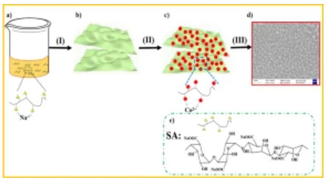

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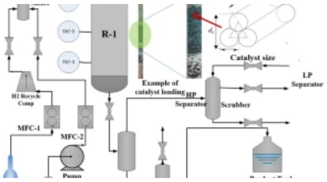

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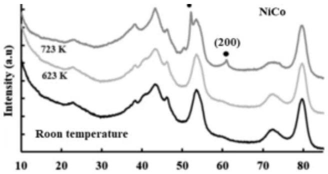

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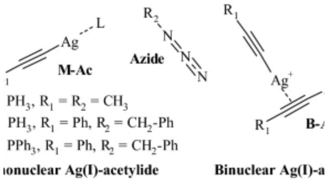

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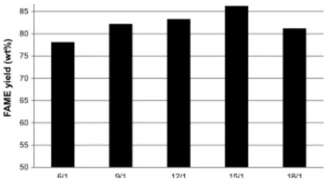

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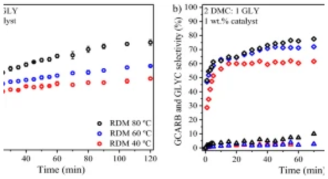

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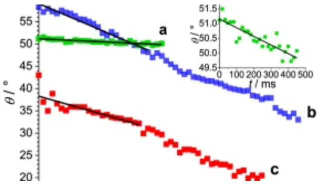

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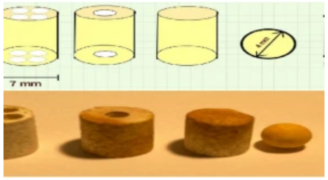

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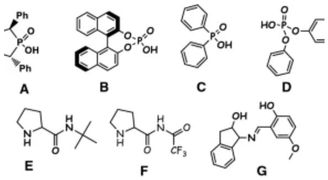

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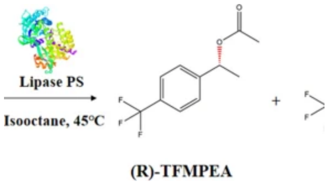

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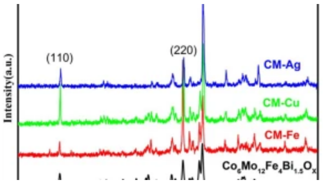

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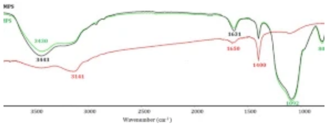

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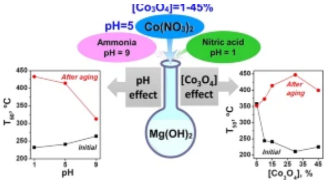

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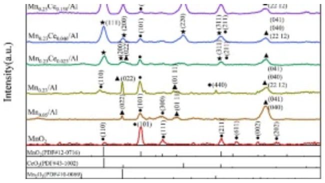

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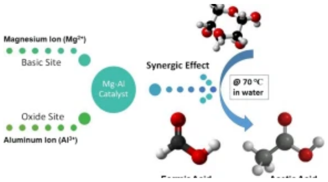

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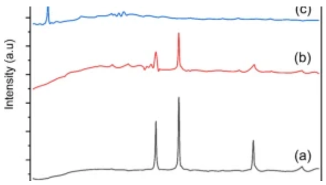

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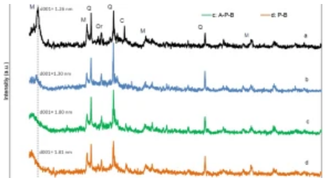

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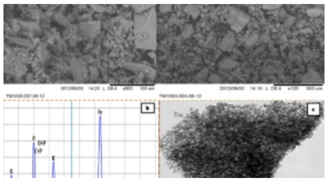

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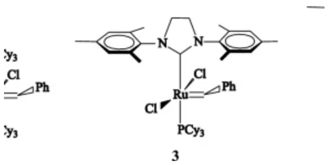

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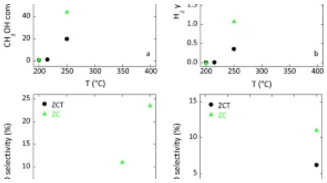

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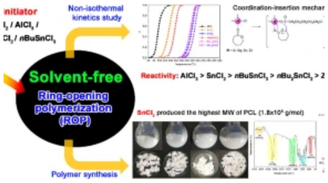

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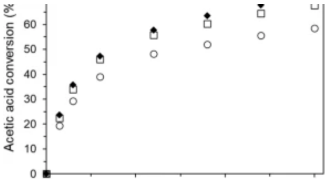

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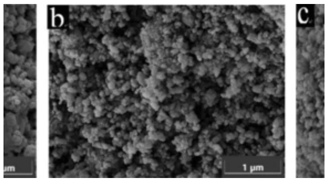

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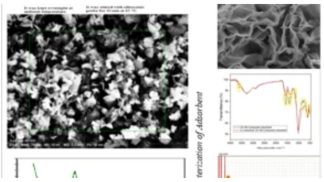

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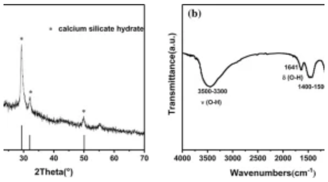

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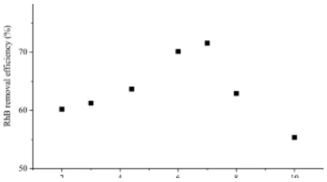

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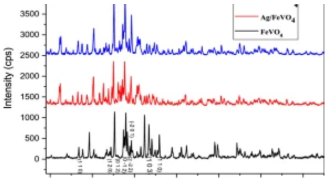

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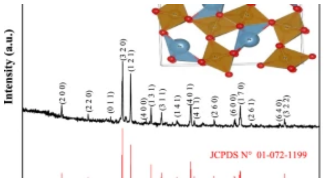

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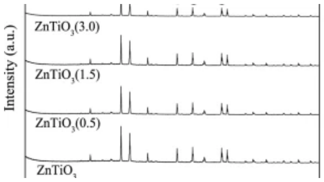

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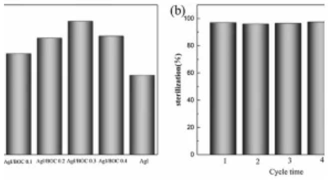

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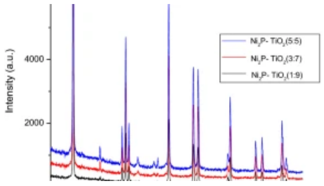

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Production of levulinic acid from glucose using nickel phosphate-silica catalyst

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Reaction Kinetics, Mechanisms and Catalysis **136**, 287–309 (2023)

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Abstract

A combination of nickel-silica and nickel phosphate is hypothesized to provide a synergistic effect on catalytic processes of LA production from glucose. This research aims to produce LA from glucose by employing nickel phosphate-silica catalysts. The effect of different phosphate catalyst precursors on the yield of LA was investigated. Catalysts were assessed by using FT-IR, SEM–EDX, XRD, and N_2 adsorption–desorption isotherms. Acidity analysis for the catalysts was performed by the gravimetric method. The product of levulinic acid was examined by HPLC. The study exhibited that the incorporation of phosphate ions into nickel-silica increased the acidity of the catalyst and inherently provided an increase in LA yield. It was found that the hydrogen atoms number from different phosphate precursors showed a positive correlation to the increase in LA yield as well. $Ni(H_2PO_4)–_2SiO_2$ catalyst had the highest activity compared to the others catalysts on LA formation, whereas $Ni_3(PO_4)_2–SiO_2$ dominantly promoted formic acid formation. Further optimization by RSM-CCD showed that the optimum condition was achieved at a temperature of 113.20 °C, a reaction time of 120 min, and a catalyst weight of 0.4573 g with an LA yield of 40.89%. The quadratic model as derived from RSM-CCD demonstrated good accuracy in estimating the output parameter. This catalyst was still active at 5 consecutive runs with a slight decrease toward LA formation.

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Ethics declarations

Conflict of interest

The authors have no competing interests to declare that are relevant to the content of this article.

Additional information

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Supplementary Information

Below is the link to the electronic supplementary material.

Supplementary file1 (DOCX 366 KB)

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

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Abstract

A combination of nickel-silica and nickel phosphate is hypothesized to provide a synergistic effect on catalytic processes of LA production from glucose. This research aims to produce LA from glucose by employing nickel phosphate-silica catalysts. The effect of different phosphate catalyst precursors on the yield of LA was investigated. Catalysts were assessed by using FT-IR, SEM–EDX, XRD, and N_2 adsorption–desorption isotherms. Acidity analysis for the catalysts was performed by the gravimetric method. The product of levulinic acid was examined by HPLC. The study exhibited that the incorporation of phosphate ions into nickel-silica increased the acidity of the catalyst and inherently provided an increase in LA yield. It was found that the hydrogen atoms number from different phosphate precursors showed a positive correlation to the increase in LA yield as well. $Ni(H_2PO_4)$–$_2SiO_2$ catalyst had the highest activity compared to the others catalysts on LA formation, whereas $Ni_3(PO_4)_2$–SiO_2 dominantly promoted formic acid formation. Further optimization by RSM-CCD showed that the optimum condition was achieved at a temperature of 113.20 °C, a reaction time of 120 min, and a catalyst weight of 0.4573 g with an LA yield of 40.89%. The quadratic model as derived from RSM-CCD demonstrated good accuracy in estimating the output parameter. This catalyst was still active at 5 consecutive runs with a slight decrease toward LA formation.

Keywords Nickel-silica · Nickel phosphate-silica · Levulinic acid production · RSM-CCD

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Introduction

The petrochemical industry is an industry that produces organic compounds derived from oil and natural gas. With the development of the petrochemical industry, the use of fossil fuels increases whilst the availability of fossil fuels decreases over time. Thus, finding other alternative precursors to fossil fuels is necessary [1–3]. To remedy such a situation, platform chemicals can be employed as a potential replacement for fossil fuels in the industries that process them, especially the petrochemical industry [4]. Levulinic acid (LA) is a platform chemical containing five carbon atoms of carboxylic acid, and ketone groups may be used as building blocks that has good reactivity [5, 6]. LA has now become a major platform chemical in biorefinery used for various applications [7, 8], and industries such as pharmaceuticals, textiles, solvents, food flavoring agents, and dyes [9].

Generally, LA can be synthesized by dehydrating and rehydrating lignocellulosic biomass (cellulose, hemicellulose, and starch) [10] or hexose sugars (glucose, fructose, and galactose) [11]. The glucose dehydration produces 5-Hydroxymethylfurfural (5-HMF), and prolonged rehydration produces levulinic acid and formic acid as by-products [12]. The dehydration and rehydration processes usually use homogeneous catalysts, such as orthophosphoric, hydrochloric, and sulfuric acid as well. However, these strong acid catalysts are non-environmental friendly, difficult to be recycled and separated, and may cause corrosion [13, 14]. Therefore, heterogeneous catalysts are more preferred and widely used to tackle the shortcomings of homogeneous catalysts [15].

In the production of LA, heterogeneous catalysts of many types have previously been used, such as $SO_4^{2-}/TiO_2–Al_2O_3–SnO_2$ [16], metal–organic framework (MOF) [14], MFI-type zeolites [17], acid clay, and ion exchange resin [18], Al–Zr mixed oxides [19], and metal(IV) phosphate [20]. Likewise, nickel-silica is a catalyst with good stability that is often used in catalytic processes [21]. The catalyst has sufficient strong acid and active metal sites [22], in addition to large surface areas and strong nickel-silica interactions [23]. Meanwhile, nickel phosphate is a metal-phosphate catalyst of strong interest for its unique physical and chemical properties [24]. This catalyst has large surface areas, as well as good acidity and stability [25]. The presence of phosphate groups in the metals provides advantageous properties in catalytic processes as it provides medium-strong Lewis acid (metal site) and Brønsted acid (protonated phosphate group) sites in charge of the high catalytic activity of the catalyst [26, 27].

Combining nickel-silica and nickel phosphate is theorized to provide a synergistic effect on catalytic processes due to the similarity in acidity and stability of the two catalysts. Many studies have reported the use of nickel phosphate catalysts use in various applications [28–31]. However, reports nor studies have yet been made on the production of LA from glucose catalyzed by nickel phosphate-silica catalyst. The strength and acidity of a catalyst are influenced by the type of phosphate ion and the number of hydrogen atoms present in the precursor. These two conditions directly affect the performance of a catalyst during catalysis [32].

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). In this regard, the quadratic polynomial equation was utilized as a model and subsequently examined using ANOVA (95% confidence level). The 3D surface and 2D contour model graphs were plotted, and the interaction of variables on the response was thoroughly studied. The catalysts from the different precursors were assessed using FT-IR, SEM–EDX, XRD powder, and N_2 adsorption–desorption isotherms. Acidity analysis for the catalysts was performed by using the gravimetric method.

Experimental

Materials

The materials employed in this study were silver nitrate, ammonium dihydrogen phosphate, diammonium hydrogen phosphate, tributyl phosphate, absolute ethanol, 25% ammonia solution, glucose, and nickel(II) nitrate hexahydrate. All chemicals were analytical grade obtained from Merck.

Preparation of silica

The SiO_2 powder was prepared by firstly mixing 50 mL of absolute ethanol with 5 mL distilled water and subsequently stirred for 15 min. Into the mixture, 5.6 mL tetraethyl orthosilicate (TEOS) and 1 mL 28% ammonia solution was added and stirred using a magnetic stirrer for 3 h at ambient temperature. The gel formed was heated for 24 h at 85 °C. The dried powder was then sieved using a 200-mesh sieve.

Synthesis of nickel-silica

4.5 g of silica powder was dispersed in 100 mL of 0.02 M of nickel(II) nitrate hexahydrate solution and stirred with magnetic stirrer for 3 h at room temperature. The solution was subsequently dried in the oven for 24 h at 105 °C. The dried powder was calcined at 700 °C for 4 h. The Ni-SiO_2 catalyst was then sieved using a 200-mesh sieve [21].

Synthesis of nickel phosphate-silica

A series of nickel phosphate-silica catalysts $Ni_3(PO_4)_2$–SiO_2, $NiHPO_4$–SiO_2, and $Ni(H_2PO_4)_2$–SiO_2 were prepared by using different phosphate precursors [33]. $Ni_3(PO_4)_2$-SiO_2 catalyst was synthesized from 0.002 mol of nickel(II) nitrate hexahydrate mixed with 0.0013 mol of tributyl phosphate and dissolved in distilled water in a 100 mL volumetric flask. After, 4.5 g of silica powder was then dispersed in the solution and stirred for 1 h. The mixture was then heated at 100 °C for 1 day followed by calcination at 700 °C for 4 h. The catalyst was then sieved using a 200-mesh sieve. Meanwhile, $NiHPO_4$-SiO_2 and $Ni(H_2PO_4)_2$–SiO_2 catalysts were synthesized from 0.002 mol of nickel(II) nitrate hexahydrate mixed with 0.002 mol of diammonium hydrogen phosphate and 0.004 mol of ammonium dihydrogen phosphate, respectively. The solutions were then dissolved in distilled water in a 100 mL volumetric flask. Subsequently, 4.5 g of silica powder was then dispersed in each of the solutions and stirred for 1 h. Subsequently, the mixtures were heated at 120 °C for 24 h. The dried powder was then sieved using a 200-mesh sieve.

Production of levulinic acid

Glucose was first dried at 105 °C for 3 h to achieve a constant weight. 2 g of the glucose was then added into 50 mL distilled water, followed by 0.5 g catalyst [12]. Next, the mixture was refluxed at 120 °C for 3 h with stirring. The refluxed solution was then centrifuged at 5000 rpm for 5 min at ambient temperature and the supernatant was analyzed using HPLC (Zorbax SB-C18 column, UV detector (268 nm) with a flow rate 1 mL/min using H_2SO_4 (0.0005 M) solution as the mobile phase at 30 °C). The glucose conversion (C_x), LA and formic acid yield (Y_x) as well as selectivity (S_x) were calculated according to the Eqs. 1–3 as follows:

$$C_X = \left(\frac{\text{reacted glocuse mole}}{\text{intial glocuse mole}}\right) \times 100\% \tag{1}$$

$$Y_X = \left(\frac{\text{LA or FA product mole}}{\text{Glucose initial mole}}\right) \times 100\% \tag{2}$$

$$S_X = \left(\frac{\text{LA or FA product mole}}{\text{reacted glucose mole}}\right) \times 100\% \tag{3}$$

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 calcined at 700 °C for 4 h to discharge the humin absorbed on the catalyst

Catalyst characterization

X-ray diffraction (XRD) powder was performed using Rigaku Mini Flex 600 with a Cu K_α radiation. The functional groups of catalysts were evaluated using Shimadzu FTIR 8201 (spectra were scanned from 400 to 4000 cm^{-1}). Acidity analysis followed the gravimetric method with pyridine gas, as reported by the previous study [34]. Scanning electron microscope-energy dispersive X-ray Spectroscopy (SEM–EDX) was performed using JEOL-JSM 6510 LA with 20 kV accelerating voltage. N_2 physisorption isotherms at 77.3 K measured using Quantachrome NOVA.

Experimental design

RSM-CCD were employed to provide the design of experiments (DoE) with the assistance of Design Expert Software 12. RSM requires a minimum number of experiments and is very useful in increasing the accuracy of the design process [35]. RSM-CCD is one of the essential tools for data modeling, collection, and optimization to determine how several input variables affect the responses [36, 37]. In general, CCD runs involved the sum of factorial (2^n), axial (2n), and central (n_c) points [38]. In this study, three input variables; ($n = 3$) temperature (A), reaction time (B), and catalyst weight (C), with LA yield (Y) as the output variable were investigated. Consequently, 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 [39]. Each variable was coded ± 1, 0, and $\pm\alpha$ (± 1.682) for the factorial, center, and axial points, respectively [40]. The ranges of variables studied are shown in Table 1.

Analysis of variance 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) [41]. Analysis of regression was conducted to investigate the Y response function [42], as formulated in Eq. 4 as follows:

$$Y = \beta_o + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{j=2}^{k} \beta_{ij} X_i X_j + \varepsilon \tag{4}$$

Here i and j are linear and quadratic coefficients, respectively, βo was the coefficient of regression, and k was the number of independent variables studied. According to Behera et al. [43], CCD was utilized for the quadratic effect due to an individual second-order effect could not be separately estimated by 2^n factorial designs.

Table 1 Independent variables used for CCD in the production of LA

Variables	Unit	Levels				
		− 1.682	− 1	0	1	+ 1.682
Temperature (A)	°C	103.18	110	120	130	136.82
Reaction time (B)	min	79.09	120	180	240	280.91
Catalyst weight (C)	g	0.25	0.35	0.5	0.65	0.75

The RSM method was reasonable to fit a typical surface of quadratic and it helped optimizing the parameters of process.

Results and discussion

Catalyst characterizations

The characterization using XRD was employed to determine the structural properties of nickel phosphate-silica catalysts. The diffractogram in Fig. 1a shows that Ni-SiO_2 catalyst had a wide peak at 2θ of 22°, corresponding to amorphous silica [44]. Sharp crystalline peaks were observed in Ni-SiO_2 catalyst at 2θ of 37.2°, 43.2°, and 62.7°. According to Mallesham et al. [22], these peaks represent NiO compounds with a face-centered cubic Ni structure. The $Ni_3(PO_4)_2$–SiO_2 catalyst as shown in Fig. 1b, had sharp peaks at 2θ of 37.1°, 43.25°, and 75.5°, indicating crystalline phases that could be attributed to the calcination of nickel phosphate [25]. This finding was in line with JCPDS No. 00-035-0987. Meanwhile, the $NiHPO_4$-SiO_2 catalyst, as shown in Fig. 1c, revealed diffraction peaks at 2θ of 8.96° and 35.52°, which represented the $NiHPO_4$ compound [33], whereas the broad peak at 2θ of 22.43° corresponded to the silica compound [45]. Furthermore, $Ni(H_2PO_4)_2$-SiO_2 diffractogram in Fig. 1d showed sharp peaks at 2θ of 17.2°, 24.04°, and 29.30°, confirming the presence of amorphous nickel phosphate [28], and silica compound (JCPDS No. 76-0933).

The spectra of Ni–SiO_2 catalyst in Fig. 2a revealed an absorption peak band at ~ 1051 cm^{-1} and 822 cm^{-1} indicating stretching vibration of Ni–O bond [46], and Si–O-Si bonding vibration, respectively, confirming the interaction between

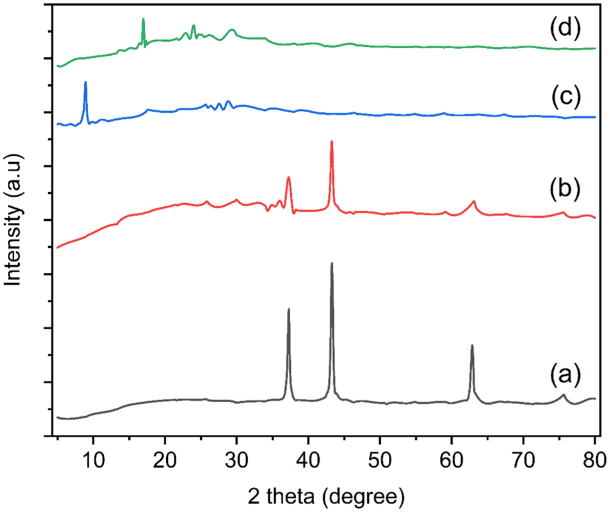

Fig. 1 XRD diffractograms of (a) Ni–SiO_2, (b) $Ni_3(PO_4)_2$–SiO_2, (c) $NiHPO_4$–SiO_2 and (d) $Ni(H_2PO_4)$–SiO_2 catalysts

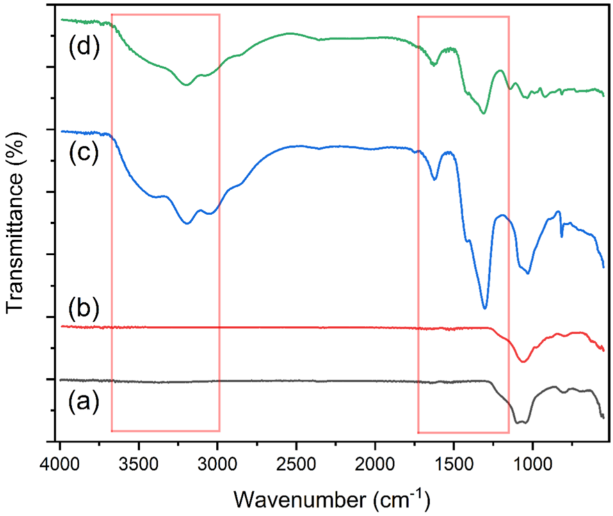

Fig. 2 FTIR spectra of (a) Ni–SiO_2, (b) $Ni_3(PO_4)_2$–SiO_2, (c) $NiHPO_4$–SiO_2 and (d) $Ni(H_2PO_4)$–SiO_2 catalyst

silica and Ni–O [47]. These peaks were also observed in all the other catalysts. Subsequently, a weak absorption at 721 cm^{-1} as in Fig. 2b of $Ni_3(PO_4)_2$–SiO_2 catalyst corresponded to the vibration of P–O bonding [48], and the absorptions at ~ 936 cm^{-1} and 994 cm^{-1} represent the stretching vibration of PO_4^{-3} tetrahedral structure [49]. These findings were consistent with the previous reports [50]. For the $NiHPO_4$-SiO_2 catalyst as shown in Fig. 2c, the spectra revealed a wide absorption peak at 3205 cm^{-1}, which is associated with HPO_4 vibration [51].

The absorption bands at 3052 and 1633 cm^{-1} indicated the stretching vibration of the hydroxyl group and the bending mode of water molecules that were physically adsorbed onto the catalyst surface [49, 50]. In the meantime, the weak absorption peak at 2358 cm^{-1} indicated the P–O–H bonding vibration [52]. This phosphate absorption was consistently reported by Liu et al. [30] previously. The same absorption bands were also observed in $Ni(H_2PO_4)$–SiO_2 (Fig. 2d). However, the P–O–H bonding vibration was not observed in Ni–SiO_2 and $Ni_3(PO_4)_2$–SiO_2 catalysts confirming that they did not have hydrogen bonding interaction from the phosphate ion.

Micrographs from the surface morphology analysis of the catalysts using SEM are shown in Fig. 3. Ni–SiO_2 catalyst in Fig. 3a shows a nearly uniform size and shape of pores, indicating good interaction between nickel and silica [53]. Meanwhile, $Ni_3(PO_4)_2$–SiO_2 catalyst in Fig. 3b showed nickel phosphate aggregations that were evenly distributed on the surface with a tight arrangement as a result of the calcination process that had changed the layered structures [29]. Fig. 3c shows $NiHPO_4$-SiO_2 surface with layered structures with relatively close distances between one pore to another. Subsequently, $Ni(H_2PO_4)_2$–SiO_2 catalyst in Fig. 3d also had layered surfaces with small aggregates dispersed on the surface, indicating the active sites of the nickel phosphate [52].

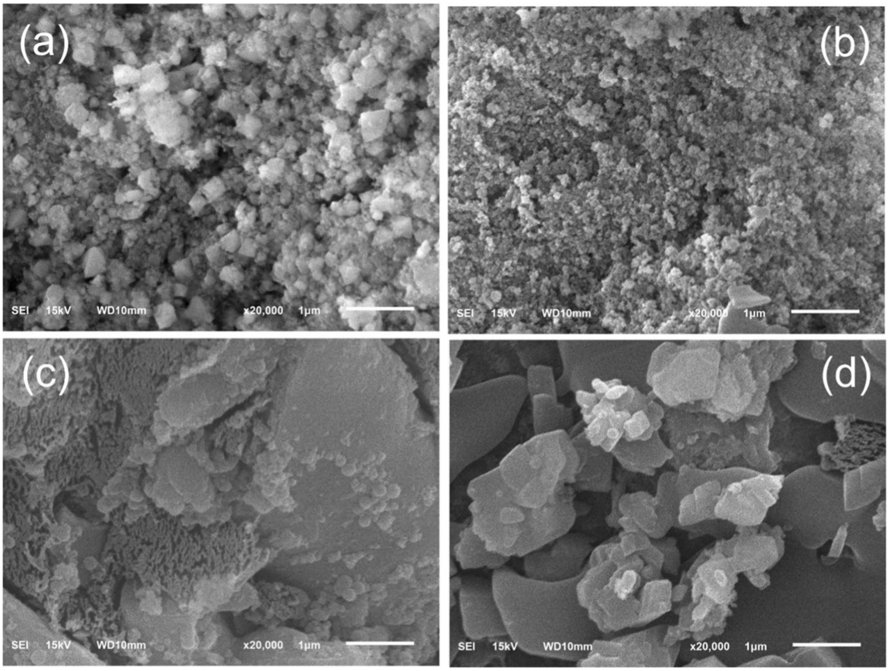

Fig. 3 SEM micrographs of **a** Ni–SiO_2, **b** $Ni_3(PO_4)_2$–SiO_2, **c** $NiHPO_4$–SiO_2 and **d** $Ni(H_2PO_4)$–SiO_2 catalysts

The EDX analysis (Supplementary Information) confirmed the existence of Si, P, O, and Ni, indicating that the nickel phosphate-silica catalysts were successfully synthesized. The P content in Ni–SiO_2 was 0% as phosphate was not incorporated onto the Ni–SiO_2 catalyst. These results also confirmed the coherency between FTIR and XRD analysis.

N_2 physisorption isotherm curves of the catalysts are presented in Fig. 4. According to the IUPAC classification, all catalysts exhibit type II adsorption isotherms [54]. Type II adsorption isotherm is associated with non-porous or macroporous solids [55]. For type II adsorption isotherm, at relatively low pressures, the volume of adsorbed gas increases sharply, indicating the incident of pore filling. Subsequently, due to completely developed macropores, the adsorption increases sharply as the P/P_o value approaches one [56]. Furthermore, at low pressures, the nearly linear point is usually associated with the monolayer adsorption and the gradual curvature, indicating conjoin of monolayer followed by the emergence of multilayer adsorption [57]. Fig. 4 also shows that all catalysts had precipitous desorption when compared to the adsorption curves. This condition is considered as hysteresis loop type B, which indicated that the catalyst had a wide distribution of pore types and diameters that usually take place in two aligned-plate cracks and slit-like pores as the major constituents [58].

The textural properties of nickel-silica phosphate derived from the N_2 physisorption isotherms are presented in Table 2. The surface area of the SiO_2 catalyst

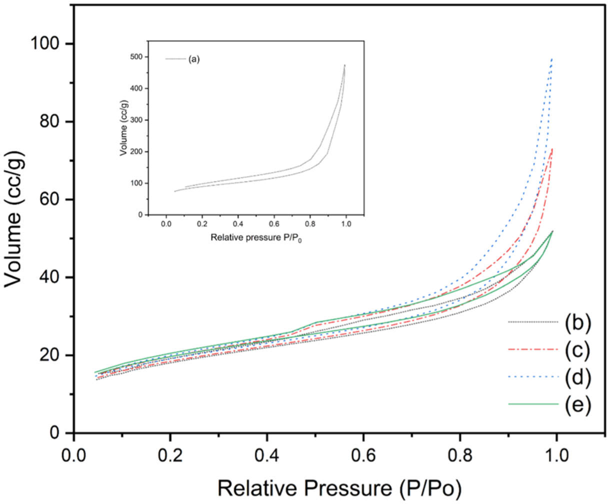

Fig. 4 N_2 adsorption–desorption isotherm curves of (a) SiO_2, (b) Ni–SiO_2, (c) $Ni_3(PO_4)_2$–SiO_2, (d) $NiHPO_4$–SiO_2 and (e) $Ni(H_2PO_4)$–SiO_2 catalysts

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

drastically decreased after being loaded by nickel and nickel phosphate species, which was probably caused by the pore's obstruction. Furthermore, it can be seen that the incorporation of phosphate ions into nickel-silica increased the surface area of catalysts from 62.77 to 63.76 m^2/g, which implied the generation of new pores by phosphate ions, thereby increasing the surface areas along with the total pore volume and pore diameter [59]. It appears that various catalysts had different surface areas depending on phosphate precursors. The highest surface area of 67.68 m^2/g was found in the $Ni(H_2PO4)_2$–SiO_2 catalyst. The high surface area prompts an increment in the number of catalytic site availability, thereby increasing the productivity of the catalyst [23]. Nonetheless, there was a reduce in the total pore volume and pore diameter of the $Ni(H_2PO_4)_2$-SiO_2 catalyst which might be due to the bulk phase partially blocking the surface pores [60].

The total acidity of the nickel-silica catalyst and nickel phosphate-silica catalysts of different precursors are shown in Table 3. The catalysts synthesized using different precursors had different total acidities. Low acidity values were observed

Table 3 Total acidity analysis of the catalysts

Catalysts	Total acidity (mmol/g)
Ni-SiO_2	0.015
Ni_3PO_4–SiO_2	0.017
$NiHPO_2$–SiO_2	0.070
$Ni(H_2PO_4)_2$–SiO_2	0.126

in Ni_3PO_4–SiO_2 and Ni–SiO_2 catalysts as both catalysts had lewis acid sites from the empty d orbitals of Ni metal that could accept a lone pair of electrons from the pyridine gas [61], and low Brønsted acid sites. Generally, the modification of nickel-silica catalyst using phosphate increases the total acidity of the catalyst. The difference in the total acidity of $Ni(H_2PO_4)_2$–SiO_2 and $NiHPO_2$–SiO_2 catalysts was not very substantial since both catalysts exhibited Brønsted acid sites from the hydrogen atoms of the phosphate groups [20], and Lewis acid sites from metal [36]. The silanol group in silica also provided another form of active site for pyridine adsorption, though not as strongly as it has a weak acid strength [62]. Remarkably, the $Ni(H_2PO_2)_2$–SiO_2 catalyst had the highest total acidity due to the high presence of Brønsted acid and Lewis acid sites [63].

Fig. 5 reveals the FTIR spectra of the catalysts after the adsorption of pyridine as indications of the Lewis and Bronsted acid sites. The FTIR spectra in Fig. 5 showed absorption bands at 1558 cm^{-1}, 1542 cm^{-1}, as well as 1508 cm^{-1}, representing the intense mode of association of the pyridine cation with the transfer of the protons from the surface OH group of the Brønsted acid sites to the corresponding molecule

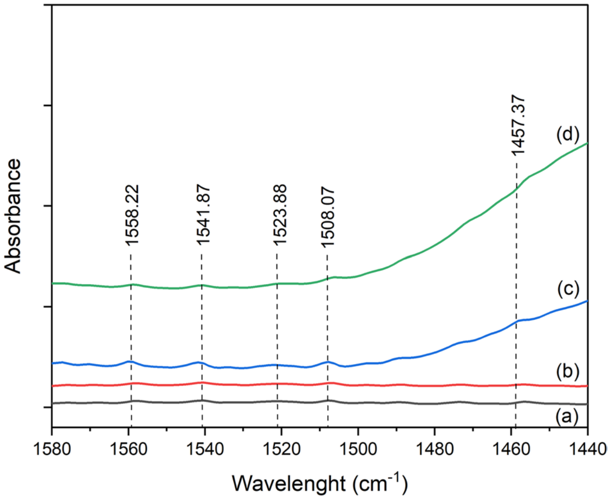

Fig. 5 FTIR spectra of (a) Ni–SiO_2, (b) $Ni_3(PO_4)_2$–SiO_2, (c) $NiHPO_4$–SiO_2 and (d) $Ni(H_2PO_4)$–SiO_2 after the adsorption of pyridine gas

[64]. According to Ni et al. [52], the absorptions are characteristic of the pyridinium ion (PhH^+), corroborating the existence of Brønsted acid site from the hydrogen in the phosphate group. The absorption peaks observed in $Ni(H_2PO_4)_2$–SiO_2 and $NiHPO_4$–SiO_2 catalysts indicated absorption through the hydrogen phosphate group. Whereas, the absorption peaks in $Ni_3(PO_4)_2$–SiO_2 and Ni–SiO_2 catalysts originated from the hydroxyl group of the Si–OH bonding. The absorption band at 1457 cm^{-1} indicated the molecularly coordinated pyridines on Ni^{2+} cations acting as Lewis acid sites [65]. Fig. 5 confirmed that $Ni(H_2PO_4)_2$–SiO_2 catalyst provided the highest absorption intensity allowing the catalyst to have the highest acidity [61]. These results are in line with the findings from the gravimetric analysis.

LA production

The production of LA from glucose was executed in a reflux system at 120 °C for 4 h, using 2 g of glucose feed and 0.5 g of catalyst in an aqueous medium. 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 to 99.82%, with the LA and FA observed as a major product. The maximum glucose conversion was attained by the $Ni(H_2PO_4)_2$–SiO_2 (99.82%), whereas other catalysts showed no significant difference in the glucose conversion. As can be seen from Fig. 6, the Ni–SiO_2 catalyst produced the lowest levulinic acid yield at around 1.24%. In this catalyst, the production of LA was only aided by the metal, i.e., nickel, acting as Lewis acid active sites [66, 67]. By using $Ni_3(PO_4)_2$–SiO_2 catalyst, the levulinic acid yield was increased to 3.72%. The increase in LA yield could be attributed to the Lewis acid sites formed by metals (M) bound to the phosphate groups (M–O–P), which enhanced the catalytic activity [26, 68].

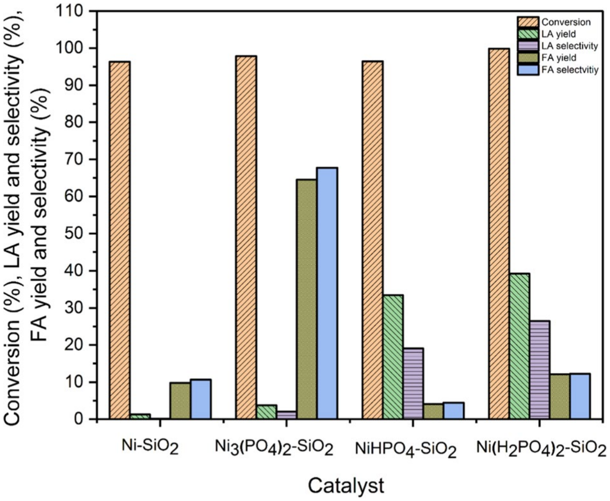

Fig. 6 Catalytic activities resulted from different catalysts (catalyst weight = 0.5 g, glucose weight = 2 g dissolved in 50 mL of DW, T = 120 °C for 3 h, stirred under reflux system)

There was a prominent increase observed in LA yield with the increment in hydrogen atoms according to the different phosphate precursors used. In general, $NiH_2(PO_4)$–SiO_2 catalyst exhibited the highest levulinic yield at 39.27%, whereas $NiHPO_4$–SiO_2 catalyst showed a 33.44% yield percentage. This increase in levulinic acid yield indicated that the hydrogen atom performed as a Brønsted acid site and played a major role in catalyzing the transformation of levulinic acid through glucose conversion. New Brønsted acid active sites were formed, generating improved catalytic activity and inherently increased the levulinic acid yield. According to Sinhamahapatra et al. [69], Brønsted acid sites mainly come from the hydrogen phosphate group. Other studies have also reported that metal phosphate catalysts provide both Lewis and Brønsted active sites [20]. Additionally, the hydrogen atom in the phosphate could form hydrogen bonds with the OH groups in glucose and strengthen the interaction among glucose and the catalyst [14]. The results showed that more hydrogen atoms were associated with more Brønsted acid active sites that, thus, would yield a higher amount of levulinic acid. Based on the acidity analysis, $NiH_2(PO_4)$–SiO_2 had the highest total acidity, indicating a positive correlation between catalyst acidity and levulinic acid yield. These findings are consistent with a previous study [12]. Similarly, the Ni–SiO_2 exhibited selectivity towards LA (0.10%), followed by the $Ni_3(PO_4)_2$–SiO_2, which only had 2.05% selectivity towards LA. The highest LA selectivity (26.49%) was achived by $Ni(H_2PO_4)_2$–SiO_2 catalyst. Weingarten et al. [20] reported that high catalyst acidity could exhibit high catalytic activity towards LA formation. Interestingly, the $Ni_3(PO_4)_2$–SiO_2 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]. 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. [70] reported that the 98% glucose conversion with LA yield of 63% was achieved by ionic liquid catalyst of [C4SO_3HPhim] [Cl] under 3 h reaction at 180 °C. This result suggests that the $Ni(H_2PO_4)_2$–SiO_2 catalyst had comparable catalytic activity towards LA production compared with other catalysts. As $Ni(H_2PO_4)_2$–SiO2 catalyst produced the highest LA yield, it was further studied and optimized using RSM-CCD. The variables observed were temperature, reaction time, and catalyst weight.

RSM-CCD analysis

RSM-CCD with three variable inputs was formulated to assess the catalytic activity of the developed catalyst. All 20 runs were performed (Table 4), and the outcomes were examined through multiple regression analysis. The coefficients were calculated and tested for their significance in view of the P-value and F-value as well.

Table 4 Experimental data of levulinic acid yield percentage

Run	Temperature (°C) Actual (coded)	Reaction time (min) Actual (coded)	Catalyst weight (g) Actual (coded)	LA yield (%)
1	110 (− 1)	120 (− 1)	0.35 (− 1)	40.34
2	130 (1)	120 (− 1)	0.35 (− 1)	25.07
3	110 (− 1)	240 (1)	0.35 (− 1)	15.54
4	130 (1)	240 (1)	0.35 (− 1)	10.31
5	110 (− 1)	120 (− 1)	0.65 (1)	30.26
6	130 (1)	120 (− 1)	0.65 (1)	34.82
7	110 (− 1)	240 (1)	0.65 (1)	30.28
8	130 (1)	240 (1)	0.65 (1)	22.29
9	103.2 (− 1.682)	180 (0)	0.5 (0)	33.44
10	136.82 (1.682)	180 (0)	0.5 (0)	35.11
11	120 (0)	79.09 (− 1.682)	0.5 (0)	42.81
12	120 (0)	280.9 (1.682)	0.5 (0)	25.50
13	120 (0)	180 (0)	0.25 (− 1.682)	23.63
14	120 (0)	180 (0)	0.75 (1.682)	23.32
15	120 (0)	180 (0)	0.5 (0)	35.67
16	120 (0)	180 (0)	0.5 (0)	35.57
17	120 (0)	180 (0)	0.5 (0)	35.48
18	120 (0)	180 (0)	0.5 (0)	35.47
19	120 (0)	180 (0)	0.5 (0)	35.66
20	120 (0)	180 (0)	0.5 (0)	35.63

The quadratic polynomial equation (in the form of coded values) from the multiple linear regression analysis is presented in the Eq. 5 as follows:

$$\begin{aligned} Y = 35.72 - 1.55A - 5.94B + 1.89C - 0.3143AB + 2.13AC \\ + 3.38BC - 1.35A^2 - 1.39B^2 - 5.17C^2 \end{aligned} \tag{5}$$

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. The positive signs in the regression coefficients indicate synergistic effects, whereas the negative signs indicate antagonistic effects on the LA yield [71].

ANOVA was performed to assess the measurable of the model significance (Table 5). The significant degree of the model and all factors (A, B, and C) was given out according to the P-value and F-value, where P-values less than 0.050 (95% confidence level) were considered significant [72]. Statistical significance could be indicated by the F-value, where a high F-value indicates that the data provided a statistically significant contribution [73]. Table 5 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 statistical diagnostic by RSM-CCD to optimize LA yield are shown in the Supplementary Information. Internal studentized of the residues with normal

Table 5 ANOVA of response surface quadratic model for LA yield

Source	Sum of square	df	Mean square	F-value	P-value
Model	1097.97	9	122.00	5.94	0.0051
A	32.65	1	32.65	1.59	0.2359
B	482.62	1	482.62	23.51	0.0007
C	49.02	1	49.02	2.39	0.1533
AB	0.7903	1	0.7903	0.0385	0.8484
AC	36.45	1	36.45	1.78	0.2122
BC	91.41	1	91.41	4.45	0.0610
A^2	26.33	1	26.33	1.28	0.2838
B^2	28.01	1	28.01	1.36	0.2699
C^2	385.29	1	385.29	18.77	0.0015
Residual	205.30	10	20.53		
Lack of fit	205.27	5	41.05	5462.82	<0.001
Pure error	0.0376	5	0.0075		
Cor total	1303.27	19			

probability showed that all points were close to the line, indicating no major problems with the design normality [74]. According to Tan et al. [41], 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 predicted 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 [75].

Behera et al. [43] 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 [37], 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 [40]. 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. [74], 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.

Optimization of LA yield

Fig. 7 depicts the response surface and contour plots that demonstrated the interaction effect between the two factors and LA yields. The interactions influenced by the input variables were observed to demonstrate the quadratic effect of the pertinent variable input on the LA yield. The effect of interaction between temperature and reaction time (at a constant

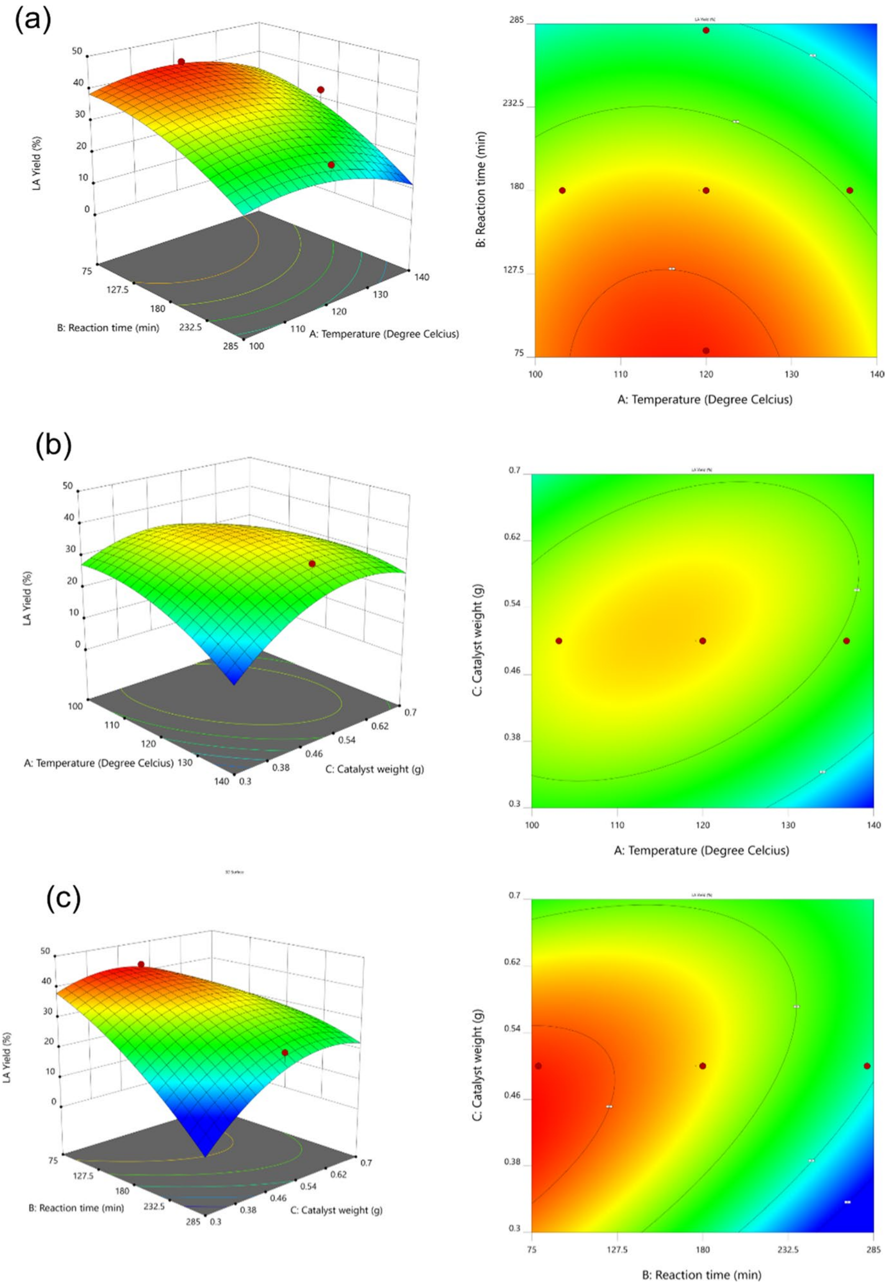

Fig. 7 3D Response fitted surface area plots and contour plots of LA yield with different variables, i.e. **a** temperature vs reaction time at a constant catalyst weight (0.5 g) **b** temperature vs catalyst weight at a constant reaction time (180 min), and **c** reaction time vs catalyst weight at a constant temperature (120 °C)

catalyst weight of 0.5 g) on LA in Fig. 7a showed that the maximum LA yield obtained was more than 40% when the temperature was in the range of 103.2–130 °C, correspondingly with a reaction time of fewer than 130 min. It can be seen from the 3D surface contour in Fig. 7a that the LA yield increased linearly with temperature and reaction time until it reached an optimum condition. This implied that an increase in temperature could cause an acceleration of the reaction rate [76]; hence under these conditions, it was very feasible for glucose to be converted into LA and for the LA yield to be increased as well. However, higher temperatures and longer reaction times allowed the side reactions to increase simultaneously, thus decreasing the LA yield. Similar findings have been reported previously [77]. From the dynamic 2D surface contour, the reaction time significantly contributed up to 30–40 of the LA yield, whereas prolonged reaction time did not increase the LA yield.

Fig. 7b appears the effect of the interaction between the catalyst weight and the reaction temperature (at a constant reaction time of 180 min) on LA yield. Through this interaction, a maximum LA yield of more than 35% was obtained at a temperature between 106 and 125 °C and a catalyst weight between 0.44 and 0.58 g. The LA yield increased with increasing catalyst weight. This is associated with more available active sites in the reaction system, thus increasing LA yield [78]. According to Dharne and Bokade [5], the increase in LA yield attributed to the increasing amount of catalyst loading is also due to the availability of a large surface area, which favors the dispersion of the active species. Therefore, the accessibility of reactant molecules at the active site of the catalyst is preferable. A further increase in catalyst weight was negatively correlated with LA yields, presumably due to the mass transfer resistance of the catalyst [79]. After the catalyst weight met the ideal necessities, a prolonged reaction temperature resulted in a lower LA yield. In this condition, by-products such as formic acid were likely more dominant, thereby reducing the LA yield. Furthermore, it was feasible to realize that reactive compounds resulting from the glucose conversion presumably collided with 5-HMF and caused cross-polymerization to produce undesired products [80]. The highly dynamic interactions between A and C were seen in the 3D surface plots showing the effectiveness of the catalyst performance in this study in the low-temperature range.

Moreover, Fig. 7c appears that the maximum LA yield was obtained by more than 40% when the reaction time was less than 125 min and catalyst weight was 0.34–0.55 g (at a constant temperature of 120 min). As illustrated in Fig. 7c, at a certain point, an increase in either of these two variables did not result in a prominent response of LA yield. A further increase in the amount of catalyst did not show a significant increase in the LA yield, which might be ascribed to the emergence of a by-product called humin being predominant. The formation of humin may be responsible for the condensation reactions of glucose, fructose, and 5-HMF [70]. After 125 min, it was found that the active center of the catalyst could be covered by humin and other polymeric compounds that reduce LA yield [81]. Similar findings were reported by Ramli and Amin [82] previously.

Process variables were optimized using RSM-CCD based on LA yield and compared with experimental results. Optimum process parameters were predicted at the temperature of 113.20 °C, the reaction time of 120 min, and the catalyst weight of 0.4573 g. The LA yield for this optimum processing parameter was estimated at 40.89%. The desirability of the applied model was 94% for this predictive value. Experiments were conducted and it was found that the predicted LA yield was close

Table 6 Reusability performance of $Ni(H_2PO_4)_2$–SiO_2 catalyst for LA fornation

Run	LA yield (%)
1st	40.70
2nd	40.25
3rd	39.43
4th	36.53
5th	34.46

to the experimental value, which had an error below ± 5%. This proved the RSM-CCD design is a great tool to specify optimum conditions. The stable process performance can be ascribed to the synthesized of nickel phosphate-silica with suitable physicochemical properties, good interaction between all the selected factors, and preferable experimental design.

As presented in Table 6, 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.

Conclusions

Nickel phosphate-silica catalyst had been synthesized with different phosphate precursors and used to convert glucose into levulinic acid. Catalyst characterization showed that the phosphate ion had been successfully incorporated onto nickel-silica catalyst and increased the acidity of the catalyst. The present study showed that catalytic activity increased significantly with increased in hydrogen atoms from the different phosphate precursors. $Ni(H_2PO_4)_2$–SiO_2 catalyst exhibited the highest catalytic activity and surface area compared to other catalysts. Furthermore, the optimum levulinic acid production using $Ni(H_2PO_4)_2$–SiO_2 catalyst by RSM-CCD was achieved at a temperature of 113.20 °C, a reaction time of 120 min, and a catalyst weight of 0.4573 g, with LA yield of 40.89%. The obtained model was statistically significant with good adequate precision. Overall, the results of this work can provide preliminary report regarding the potential of nickel phosphate-silica catalysts for the production of levulinic acid as high-value products.

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Declarations

Conflict of interest The authors have no competing interests to declare that are relevant to the content of this article.

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