



Research Article

Optimization of Vanillin Production from Lignin Using Catalytic Depolymerization over a CuO/Al₂O₃ Catalyst

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Abstract

The optimal conditions for vanillin production from lignin depolymerization using CuO/Al₂O₃ catalysts were determined by combining Box-Behnken design (BBD) and response surface methodology (RSM). Independent variables, including temperature (80–140 °C), NaOH loading (0.5–1.5 g), time (30–90 min), and catalyst weight (0.5–1.5 g), were investigated to determine the optimal conditions, with the concentration of vanillin being the dependent variable. A CuO/Al₂O₃ catalyst was prepared by impregnation method. The vanillin obtained from the reaction was analyzed using high-performance liquid chromatography (HPLC). The maximum obtained vanillin concentration of 59.14 mg L⁻¹ was achieved with a temperature of 80 °C, a reaction time of 90 min, NaOH loading of 1.5 g, and 1.5 g of catalyst. The amount of NaOH was the most influential factor governing the obtained vanillin concentration. Regression analysis was performed to determine the formula describing the vanillin concentration in terms of the independent variables with a reasonable degree of accuracy ($R^2 = 0.87$). This study shows that the optimal conditions for the depolymerization of lignin to vanillin over a CuO/Al₂O₃ catalyst can be achieved under milder conditions than those reported previously.

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Introduction

Recently, the conversion of biomass into valuable products or high-value substances to replace petroleum has received considerable attention. Among the available biomasses, lignin is the second-most abundant [1]. Lignin has a large, complex, and amorphous structure consisting of three central units: coniferyl, p-coumaryl, and sinapyl alcohols [2–4]. This structure is composed of smaller parts connected by ethers or carbon-carbon bonds and high-value substances such as vanillin [5]. Vanillin (4-hydroxy-3-methoxy benzaldehyde) is an aromatic compound with a sweet fragrance that is extracted from vanilla beans. This compound has widespread applications, which can be classified into

three main categories: flavoring agents in the food industry, perfume ingredients, and pharmaceutical intermediates [6–7]. Vanillin is widely produced using non-renewable petrochemical sources, but it can also be synthesized by the oxidation of lignin with the Kraft pulping process. However, the production of vanillin from lignin requires further study to achieve better performance under milder conditions.

Worldwide, approximately 50 tons of lignin are produced annually, it is mainly used for energy purposes. And only 10% of Kraft lignin obtained from pulp production is used for high-value-added chemical production [8]. Moreover, only 10% of Kraft lignin is a high-value substance; the residue is burned to produce

heat for generating steam as the power source in pulp production. Reports on lignin decomposition to produce high-value substances have shown that hydrothermal processes can decompose lignin and convert it to vanillin via advanced oxidation reactions [9–10]. According to Sangnak et al. [11], Ni/Al₂O₃ is a suitable catalyst for degrading lignin to vanillin, but a high temperature of 275 °C is required. This is because the use of Ni catalysts requires severe conditions, such as high temperatures. Other studies have shown that using Fe/Al₂O₃ as the catalyst can reduce the reaction temperature, but the vanillin yield remains low [12–13]. Kang et al. [14] reviewed the catalytic thermal decomposition of lignin, reporting that Cu shows the highest reactivity for lignin degradation in the temperature range of 100–320 °C. Moreover, CuO is highly active for lignin decomposition, producing compounds including phenolic compounds, chlorophenol, and lignin derivatives [15]. Therefore, CuO/Al₂O₃ is a potentially promising catalyst for decomposing lignin into vanillin. Furthermore, according to Sriprom et al. [16–17], the amount of NaOH affects vanillin formation via the partial oxidation of lignin. Lignin decomposition yields carboxylic acid, which causes a decrease in pH, resulting in the precipitation of lignin. Hence, increasing the amount of NaOH prevents lignin precipitation. In addition to these factors, the reaction time also affects vanillin production. A longer reaction time may reduce the vanillin yield because the vanillin can decompose into other products [18].

This study aims to optimize the depolymerization of lignin to vanillin using CuO/Al₂O₃ as a catalyst. The potential factors governing the catalyst concentration, including the temperature, amount of catalyst, amount of NaOH, and reaction time, were investigated.

Methods and materials

1) Materials

The chemicals used for lignin degradation to vanillin were copper (II) nitrate nonahydrate (Cu(NO₃)₂ · 9H₂O, Loba Chemie, India), alumina (Al₂O₃), lignin powder (analytical grade, Sigma-Aldrich, product number 370959, lot# 04414PE), sodium hydroxide (NaOH, Loba Chemie Pvt. Ltd.), sulfuric acid (H₂SO₄ 98%, RCI Labscan Ltd.), acetic acid (CH₃COOH 99.8%, RCI Labscan Ltd.), methanol (CH₃OH, HPLC grade, RCI Labscan Ltd.), deionized water, and vanillin (C₈H₈O₃, Sigma-Aldrich).

2) Preparation and catalyst characterization

A CuO/Al₂O₃ catalyst consisting of copper oxide supported on alumina (10 wt% Cu content) was prepared by wetness impregnation method using copper nitrate as a copper precursor. First, copper nitrate was dissolved in deionized water. Next, alumina was added to the copper solution under thorough mixing by a magnetic stirrer. The sample was dried in an oven at 100 °C for 12 h and then calcined in a muffle furnace at 350 °C for 4 h.

The specific surface area of the catalyst was measured by nitrogen adsorption–desorption analysis in a gas adsorption instrument (ASAP 2010) at -196 °C and was calculated using the Brunauer, Emmett, and Teller (BET) equation, as shown in Eq. 1 [19].

$$\frac{P}{V(P_0 - P)} = \frac{1}{V_m C} + \frac{(C - 1)P}{V_m C P_0} \quad (\text{Eq. 1})$$

where P is the vapor pressure of nitrogen gas, P_0 is the saturated vapor pressure of the gas at temperature (T), P/P_0 is the relative vapor pressure of the gas, V is the volume of the gas adsorbed at pressure (P) and temperature (T), V_m is the volume of the gas adsorbed to form a saturated monolayer per gram of the adsorbent (monolayer capacity, cm³ g⁻¹), and C is the BET constant.

The phase structure of the catalyst was determined by X-ray diffractometry (XRD, PANalytical, Model EMPYREAN). Scanning electron microscopy/energy-dispersive X-ray spectroscopy (SEM/EDS, FEI, Model: Helios NanoLab G3 CX) was used to analyze the copper dispersion and catalyst morphology. The CuO loading was determined by wavelength dispersive X-ray fluorescence spectroscopy (XRF, Rigaku, Model ZSX Primus II).

3) Experimental design

The factors influencing lignin degradation to vanillin, including temperature, amount of catalyst, amount of NaOH, and time, were determined by Box-Behnken design (BBD) [20–21] using MINITAB 16 statistical software (Minitab, Inc., Pennsylvania, USA), as shown in Table 1. In addition, 27 experimental conditions were used, as listed in Table 2.

Table 1 Factors and levels

Factor	Unit	Levels		
		Low (-1)	Medium (0)	High (+1)
Temperature (X ₁)	°C	80	110	140
Catalyst weight (X ₂)	g	0.5	1	1.5
Sodium hydroxide (X ₃)	g	0.5	1	1.5
Time (X ₄)	min	30	60	90

Table 2 Set and results of the experiments using Box-Behnken design (coded)

Run number	Manipulated variables				Response
	X ₁	X ₂	X ₃	X ₄	Vanillin concentration (mg L ⁻¹)
1	-1	-1	0	0	48.46
2	1	-1	0	0	48.30
3	-1	1	0	0	48.58
4	1	1	0	0	48.97
5	0	0	-1	-1	36.02
6	0	0	1	-1	57.08
7	0	0	-1	1	35.98
8	0	0	1	1	59.35
9	-1	0	0	-1	48.66
10	1	0	0	-1	49.26
11	-1	0	0	1	48.82
12	1	0	0	1	35.44
13	0	-1	-1	0	39.93
14	0	1	-1	0	36.54
15	0	-1	1	0	56.63
16	0	1	1	0	55.45
17	-1	0	-1	0	33.33
18	1	0	-1	0	38.26
19	-1	0	1	0	54.10
20	1	0	1	0	58.33
21	0	-1	0	-1	49.07
22	0	1	0	-1	35.78
23	0	-1	0	1	48.38
24	0	1	0	1	51.29
25	0	0	0	0	44.37
26	0	0	0	0	45.78
27	0	0	0	0	44.85

4) Lignin depolymerization to vanillin using a CuO/Al₂O₃ catalyst

Tables 1 and 2 show the BBD experimental design. A batch reactor was used to carry out the conversion of lignin to vanillin via depolymerization following the methods described by Patraporn et al. [22]. Lignin solution (1000 mg L⁻¹) was prepared by dissolving 1,000 mg of lignin powder in distilled water in a 1 L volumetric flask. The pH of the solution was adjusted to 9 using NaOH pellets. Then, 250 mL of the lignin solution was poured into a two-necked flask, followed by the addition of various amounts of catalyst and NaOH, as described in Tables 1 and 2. The mixture was

stirred with a magnetic stirrer at a constant speed (400 rpm) and heated to the desired temperature. Each experiment was carried out under the conditions designed explicitly by the BBD. A sample of the liquid-solid mixture was collected at the end of the reaction. The sample was neutralized with sulfuric acid to separate the remaining lignin, and the resulting mixture was filtered using a 0.22 µm syringe filter. Next, the mixture was analyzed by high-performance liquid chromatography (HPLC, Waters, Model e2695) to determine the concentration of vanillin using an ACE 5 C-18 PFP column (150 × 4.6 mm id, ACE-1210-1546) and a UV detector at a wavelength of 274 nm. A

methanol/acetic acid solution with a volumetric ratio of 40/60 (v/v) was used as the mobile phase. The flow rate of the mobile phase was set at 0.75 mL min^{-1} at room temperature [23].

Results and discussion

1) Characterization of the CuO/Al₂O₃ catalyst

The BET surface area of the catalyst, as determined by nitrogen adsorption–desorption analysis, was $154.3 \text{ m}^2 \text{ g}^{-1}$. The catalyst had a pallet morphology, as shown in Figure 1 (a). All elements were evenly dispersed throughout the catalyst surface, as indicated by Figure 1b. The copper was not agglomerated and was well-distributed, as shown in Figure 1c.

The XRD pattern of CuO/Al₂O₃ showed a crystalline CuO phase, as displayed in Figure 2. The peaks at $2\theta = 35.5^\circ, 38.7^\circ, 48.7^\circ, 53.4^\circ, 58.3^\circ, 61.5^\circ, 72.6^\circ$ and 75.2° were consistent with the standard peaks of CuO (PDF#48-1548) [15, 24]. Al₂O₃ peaks were also observed. The CuO and Al₂O₃ content of the catalyst was determined using XRF. As shown in Table 3, this

catalyst contained 12.8 wt% CuO and 84.90 wt% Al₂O₃. Thus, the catalyst was confirmed to be CuO/Al₂O₃.

2) Lignin degradation to vanillin

Lignin has a polymer structure consisting of three monomers: cross-linked p-hydroxyphenyl, siringyl, and guaiacyl, which can be transformed into an aromatic oxygenate by catalytic alkaline hydrothermal treatment. Vanillin is typically produced from guaiacol via an almost by-product-free process, thus simplifying product separation. However, the guaiacol route is completely dependent on petroleum-derived compounds. On the other hand, the synthesis of vanillin from renewable sources is expected to be greener and more sustainable. Vanillin can be produced from biomass through the controlled oxidation of lignin. However, there are limitations to this production route, as lignin depolymerization requires a large amount of energy. Hence, vanillin production from lignin was studied in this work to improve production under milder conditions.

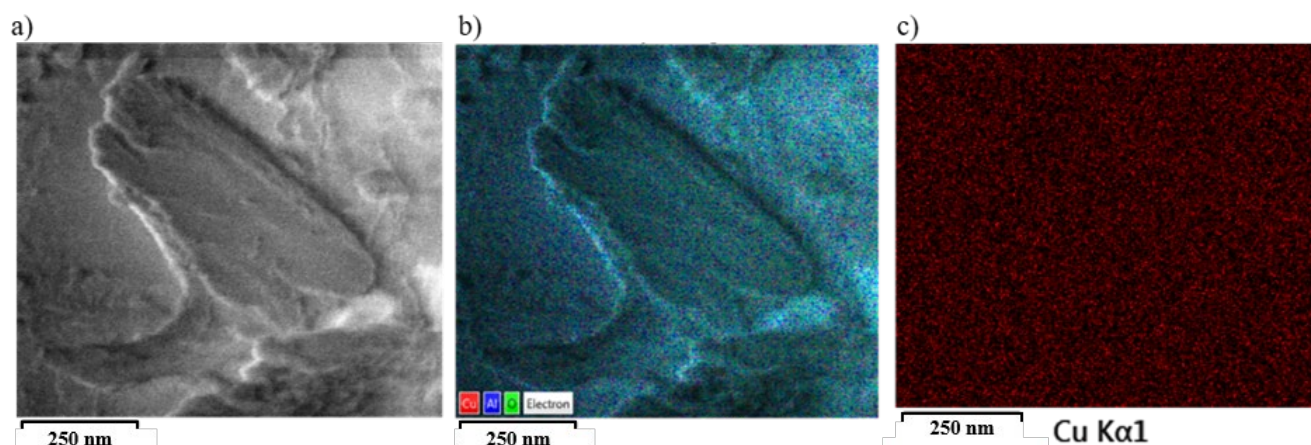


Figure 1 SEM analysis of CuO/Al₂O₃: (a) surface morphology, (b) surface elemental dispersion on the surface, and (c) Cu mapping of CuO/Al₂O₃.

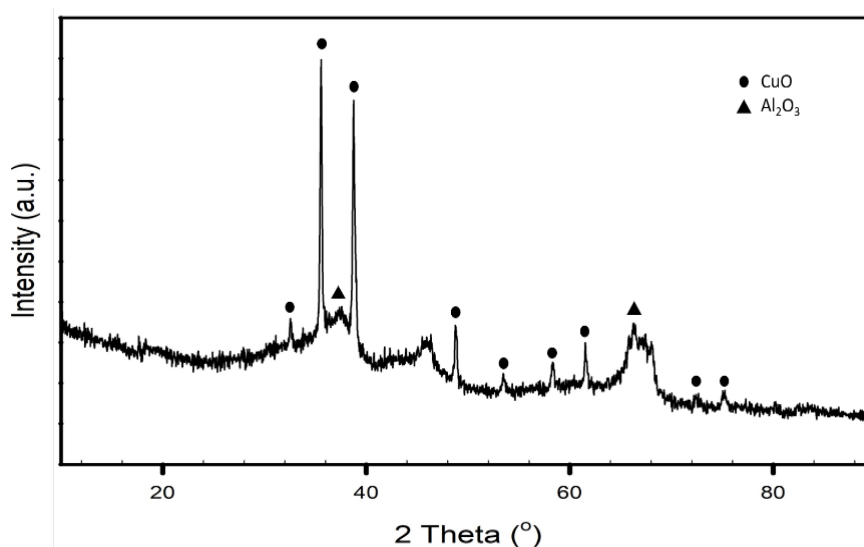


Figure 2 XRD pattern of CuO/Al₂O₃.

Table 3 Chemical composition of CuO/Al₂O₃ catalyst

Component	Result (wt%)	Detection limit	El. line	Intensity	w/o normal
Al ₂ O ₃	84.90	0.083	Al-K	261.921	84.291
CuO	12.80	0.005	Cu-K	2333.478	12.667
SiO ₂	1.41	0.017	Si-K	3.732	1.395
Cl	0.647	0.017	Cl-K	4.996	0.643
MgO	Trace	0.128	Mg-K	0.043	0.085
P ₂ O ₅	0.07	0.009	P-K	0.748	0.070
CaO	0.041	0.003	Ca-K	0.551	0.040
SO ₃	0.035	0.011	S-K	0.318	0.034
Fe ₂ O ₃	0.027	0.003	Fe-K	1.193	0.027
Ga ₂ O ₃	0.008	0.002	Ga-K	1.338	0.008
NiO	0.003	0.002	Ni-K	0.420	0.003
ZrO ₂	0.002	0.001	Zr-K	1.031	0.001

The lignin depolymerization to vanillin experiment was designed using BBD to determine the optimal conditions based on four factors: temperature, catalyst amount, NaOH amount, and reaction time, at three levels. The experiment was set up for 27 runs using BBD, and the vanillin concentration was in the range of 33.33–59.35 mg L⁻¹, as shown in Table 2. According to the HPLC spectra of vanillin (Figure 3), the highest vanillin concentration was 59.35 mg L⁻¹.

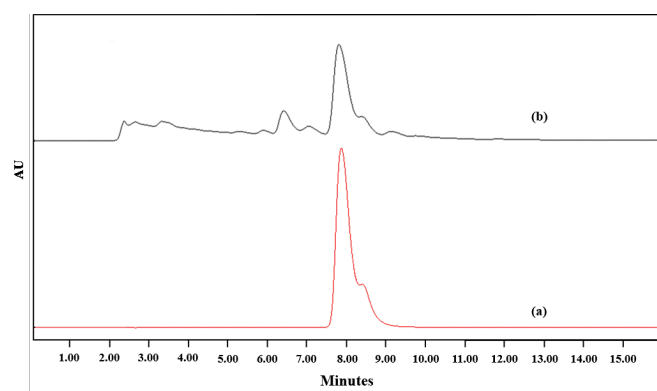


Figure 3 HPLC chromatogram of (a) the vanillin standard, and (b) vanillin produced from lignin depolymerization.

The experimentally obtained vanillin concentrations shown in Table 2 were used to generate a full quadratic model for predicting the optimum vanillin concentration. The four main BBD steps are: (1) conducting statistically designed experiments specific to the experimental goals; (2) recommending a statistical model based on the experimental findings and analysis of variance (ANOVA); (3) using diagnostic plots to confirm model suitability; and (4) predicting the response variables and verifying the model. The full quadratic model is given in Eq. 2 [25].

The vanillin concentrations (Table 2) were used in a quadratic model to obtain a mathematical formula for determining the vanillin concentration as a function of the four variables, as shown in Eq. 3.

The regression coefficients, standard error, and P values of each variable in Eq. 3 were calculated, as shown in Table 4. The R² value of the quadratic regression model was 0.87, indicating a reasonably good fit between the experimental results and the model. The regression model was tested using ANOVA (Table 4). The linear model term representing the amount of NaOH (X₃) significantly affected lignin depolymerization to vanillin (P < 0.05).

The predicted values of the vanillin concentration from lignin depolymerization using CuO/Al₂O₃ as the catalyst were obtained using Eq. 3. The regression model was checked by graphical analysis of the residuals, which were calculated from the differences between the observed and predicted values. The residual plots of the regression model demonstrating the reliability of the data are shown in Figure 4 (a-d). The normal probability plots of the residuals in Figure 4a show that the data are distributed in a straight line, indicating that they are accurate and reliable. Figure 4b shows the standardized residual and fit values (vanillin concentration), depicting a random scattering plot. This plot suggests a relatively constant variance across the fitted values. The histogram in Figure 4c shows the standard curve. As shown in Figure 4d, the plot of the standardized residual versus the run order of vanillin concentration displays fluctuations around the zero line in the range of ±2. These results indicate that the data are well distributed. Furthermore, these plots show that the regression model for predicting the vanillin concentration obtained from lignin depolymerization is accurate and reliable.

$$Y = \gamma_0 + \sum_{i=1}^3 \gamma_i X_i + \sum_{i=1}^3 \gamma_{ii} X_i^2 + \sum_{i=1}^2 \sum_{j=i+1}^3 \gamma_{ij} X_{ij} \tag{Eq. 2}$$

where Y is the vanillin concentration, X_i and X_{ij} are uncoded independent variables, γ_0 is the offset term, and γ_i , γ_{ii} , and γ_{ij} are the regression coefficients.

$$Y = 45 - 0.28(X_1) - 1.18(X_2) + 10.07(X_3) + 0.28(X_4) + 0.82(X_1^2) + 1.67(X_2^2) + 0.87(X_3^2) + 0.14(X_4^2) + 0.14(X_1X_2) - 0.18(X_1X_3) - 0.35(X_1X_4) + 0.55(X_2X_3) + 4.05(X_2X_4) + 0.58(X_3X_4) \tag{Eq. 3}$$

where Y represents the predicted vanillin concentration (mg L^{-1}) and X_1 , X_2 , X_3 , and X_4 are the temperature, amount of catalyst, amount of NaOH, and time, respectively.

Table 4 Regression coefficients for vanillin concentration model

Term	Coefficient	Standard error coefficient	p-value
Constant	45.00	2.38	0.000
X_1	-0.28	1.19	0.817
X_2	-1.18	1.19	0.340
X_3	10.07	1.19	0.000
X_4	0.28	1.19	0.816
X_1^2	0.82	1.78	0.656
X_2^2	1.67	1.78	0.367
X_3^2	0.87	1.78	0.634
X_4^2	0.14	1.78	0.938
X_1X_2	0.14	2.06	0.947
X_1X_3	-0.18	2.06	0.933
X_1X_4	-3.50	2.06	0.116
X_2X_3	0.55	2.06	0.793
X_2X_4	4.05	2.06	0.073
X_3X_4	0.58	2.06	0.784

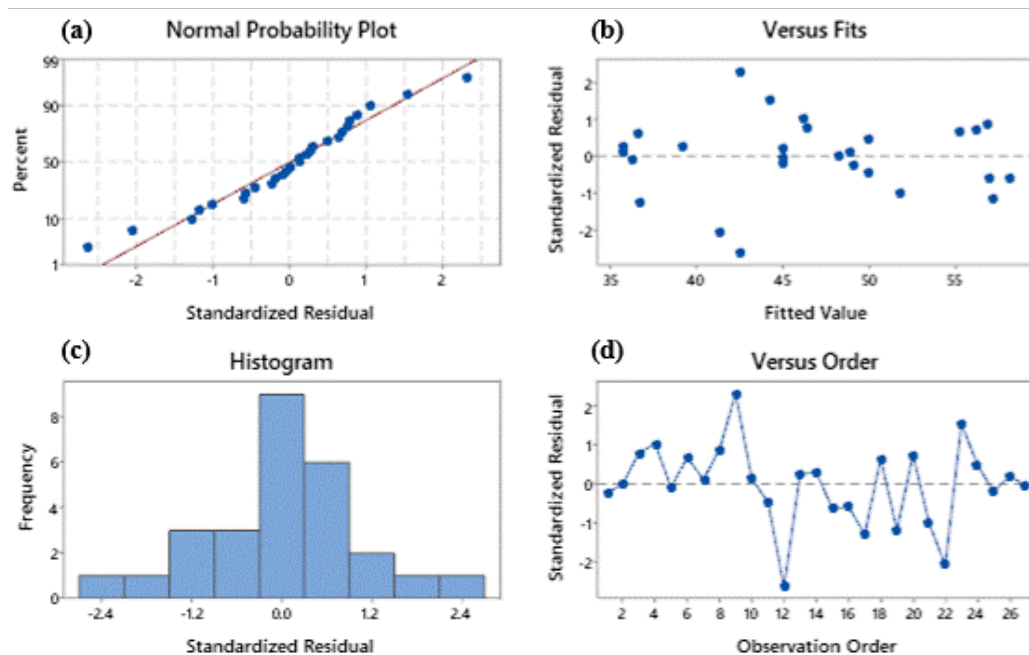


Figure 4 Residual plots of vanillin concentration: (a) normal probability plot, (b) versus fit, (c) histogram, and (d) versus order.

The effects of the four variables (temperature, amount of catalyst, NaOH amount, and time) on the vanillin concentration from lignin depolymerization via catalytic alkaline hydrothermal depolymerization are shown in Figure 5. The vanillin concentration was largely a function of the amount of NaOH: the concentration of vanillin increased with increasing NaOH amount. These results indicate that NaOH-based alkaline media can catalyze the hydrothermal depolymerization of lignin under all the reaction conditions. Furthermore, Na^+ and OH^- can react with ether bond terminals and promote the cleavage of β -O-4 and 4-O-5 linkages, resulting in the production of vanillin from lignin depolymerization [24].

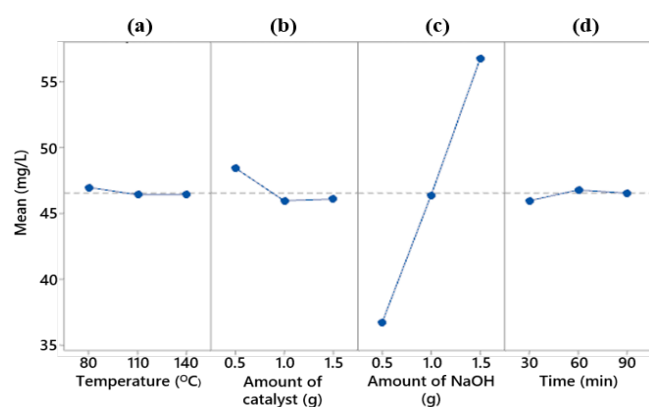


Figure 5 Main effect plots of the independent variables on the vanillin concentration: (a) temperature, (b) amount of catalyst, (c) amount of NaOH, and (d) time.

Figure 5 also reveals that between 80 °C and 140 °C, the vanillin concentration slightly decreased as the temperature increased. This change might be ascribed to higher H^+ and OH^- ion concentrations in the water, caused by a decrease in the dielectric constant and an increase in the ion products under subcritical conditions with increasing temperature. At higher temperatures, lignin depolymerization result in greater amounts of phenol than vanillin [26]. As higher temperatures enhance the reaction rate, this could affect the cleavage of the weak C–O bonds, resulting in the generation of monomer radicals. Thus, the selectivity for vanillin was low at high temperatures. Moreover, it has been reported that catalysts with higher Cu content have higher surface oxygen concentrations, leading to a decline in vanillin concentration because vanillin is oxidized to vanillic acid [24]. In other words, higher Cu loading increases the number of CuO sites on the catalyst surface, thus increasing the overoxidation of vanillin. The influence of CuO sites on vanillin oxidation has been reported at temperatures over 150 °C. As shown in Figure 5 (d), the vanillin concentration increased

with time until 60 min, after which it slightly decreased. Thus, these results indicate that prolonging the reaction time increased the engagement and activity of H^+/OH^- ions produced by water, and the main reactions in water, such as C–O hydrolysis, stable C–C bond cleavage, and demethoxylation, facilitated the cracking of lignin.

The effects of the lignin depolymerization factors on the production of vanillin with the $\text{CuO}/\text{Al}_2\text{O}_3$ catalyst are explained by the contour plots shown in Figure 6. Figure 6 (a–c) shows plots of temperature versus the amount of catalyst for constant vanillin concentrations, amount of NaOH, and time. Figure 6 (a) shows that greater catalyst amounts and higher temperatures results in lower vanillin concentrations, thus affecting vanillin production. Jeon et al. reported a wet alkaline oxidation process in which the highest vanillin yield (6.8 %) was obtained at 150 °C. The vanillin yield achieved by this process notably decreased at 180 °C, indicating that vanillin was unstable at high temperatures. At these elevated temperatures, the vanillin was converted into vanillic acid and various organic acids.

The relationship between the amount of NaOH and the temperature is shown in Figure 6b. This plot indicates that a high concentration of NaOH to maintain a pH above 9 prevents lignin precipitation. With increasing NaOH concentration, the vanillin yield significantly increased. However, temperature did not significantly influence vanillin yield.

The relationship between the temperature and reaction time is shown in Figure 6 (c). With decreasing reaction time, an increasing temperature is required for a constant vanillin concentration. A long heating time might result in shorter lignin fragments or oligomer formation, resulting in pore blocking and coke formation [27]. However, the long reaction time would be advantageous for producing vanillin at the temperature in the range of 80 °C to 90 °C.

The relationship between the amount of Cu and NaOH, as depicted in Figure 6d, shows that the amount of catalyst has a less significant effect than the amount of NaOH on the vanillin concentration. On the other hand, the interaction between the amount of catalyst and the reaction time, shown in Figure 6 (e), demonstrates that an increasing amount of catalyst is required to achieve a constant vanillin concentration as the reaction time decreases. The interaction between the amount of NaOH and reaction time is shown in Figure 6f, which reveals that the vanillin concentration is independent of time after 30 min. Thus, after 30 min, the vanillin concentration is solely dependent on the amount of NaOH.

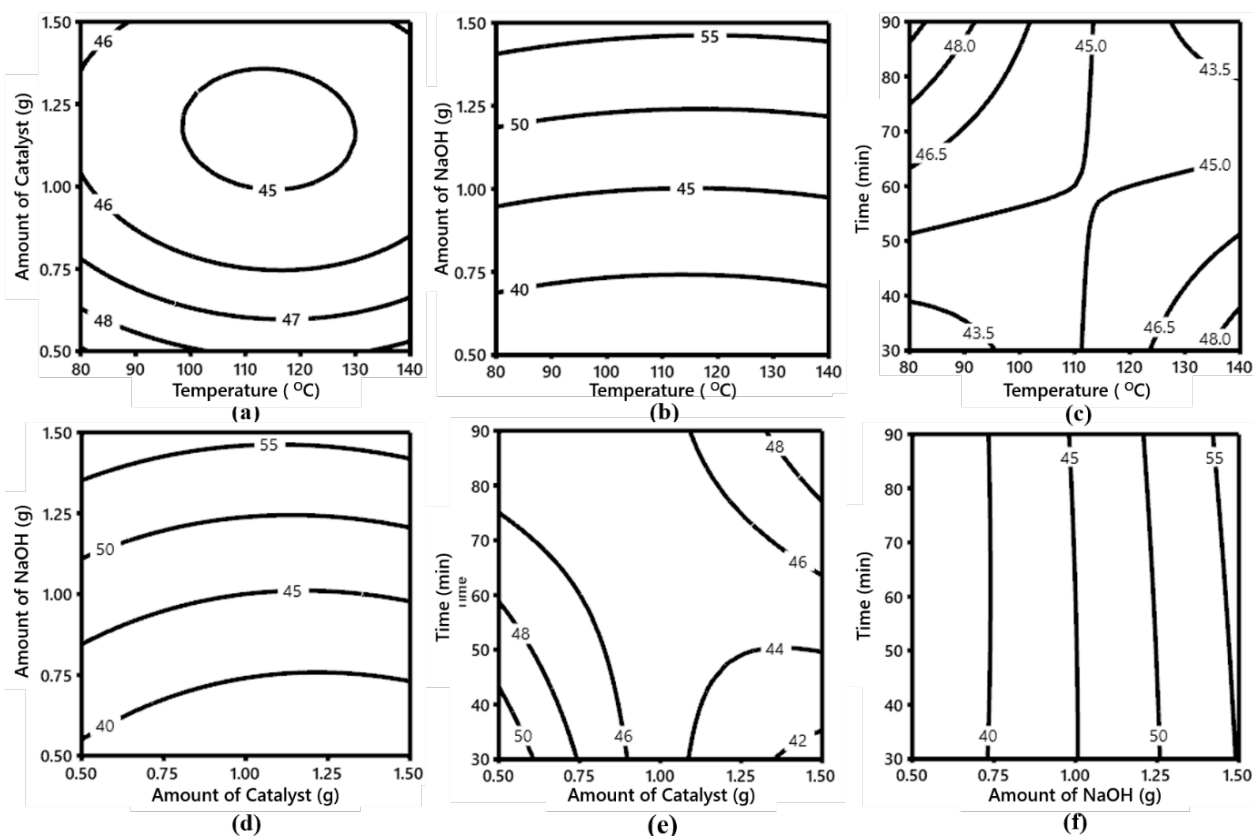


Figure 6 The effects of variables on the vanillin concentration: (a) temperature and amount of catalyst, (b) temperature and amount of NaOH, (c) temperature and time, (d) amount of catalyst and amount of NaOH, (e) amount of catalyst and time, and (f) amount of NaOH and time.

3) Optimization

The optimal conditions for alkaline lignin depolymerization using CuO/Al₂O₃ as the catalyst were determined with response surface methodology to obtain the highest concentration of vanillin. The optimal conditions were a reaction temperature of 80 °C, a reaction time of 90 min, 1.5 g of NaOH, and 1.5 g of CuO/Al₂O₃. Using these conditions, an average vanillin concentration of 59.14 mg L⁻¹ ± 0.4 (5.91% yield) was experimentally obtained, with the results verified thrice. Thus, a low temperature and normal atmospheric pressure were suitable for obtaining a high vanillin yield. This vanillin yield was compared with several reported works, as shown in Table 5. Rawat et al. [28] used a MoPO/CeO₂ catalyst to achieve a 9.00% vanillin yield from lignin under 5 bar O₂ at 150 °C, demonstrating that their novel metal oxide-based catalyst had excellent performance for the conversion of lignin to vanillin. Zhang et al. obtained a vanillin yield of 1.90%

by the catalytic depolymerization of lignin using FeCl₃/NaNO₃ under 10 bar O₂ at 100 °C [29]. Totong et al. [30] reported the conversion of lignin to vanillin with a yield of about 2.70% by depolymerization using Ni/CeO₂-ZrO₂ at 250 °C. They also showed that lignin conversion to vanillin decreased with increasing temperature. Sangnak et al. [11] used 10% Ni/Al₂O₃ as a catalyst for lignin depolymerization, reporting a vanillin yield of 6.56% under 10 bar N₂ at 275 °C. Chaiyasat et al. [13] used a hydrothermal process to convert lignin to vanillin over an Fe₂O₃/Al₂O₃ catalyst under 10 bar O₂ at 120 °C, reporting a vanillin yield of 6.46%. This literature review shows that vanillin yields between 1.90–9.00% can be obtained at high temperatures and high pressures. However, this work shows that a similar vanillin yield can be obtained at a low temperature and atmospheric pressure using a CuO/Al₂O₃ catalyst. Thus, CuO/Al₂O₃ can catalyze lignin depolymerization to vanillin under mild conditions.

Table 5 Comparison of lignin reaction conditions and vanillin yields reported in the literature

Process	Catalyst	Conditions	Vanillin yield (%)	Ref
Depolymerization	MoPO/CeO ₂	- Alkaline lignin, 0.5 g L ⁻¹ - 0.1 g catalyst - 15 mL of 2 M NaOH - 5 bar O ₂ - 150 °C - 3 h	9.00	[28]
Depolymerization	FeCl ₃ /NaNO ₃	- Alkaline lignin 10 g L ⁻¹ - 0.025 g catalyst - 10 bar O ₂ - 100 °C - 30 min	1.90	[29]
Depolymerization	Ni/CeO ₂ -ZrO ₂	- Alkaline lignin 2 g L ⁻¹ - 1 g catalyst - 250 °C with autogeneous pressure of helium - 3 h - 25 mmol NaOH	2.70	[30]
Hydrothermal	Ni/Al ₂ O ₃	- Alkaline lignin 0.5 g L ⁻¹ - 1 g catalyst - 275 °C - 2 h - 1 g NaOH - 10 bar N ₂	6.56	[11]
Hydrothermal	Fe/Al ₂ O ₃	- Alkaline lignin 0.5 g L ⁻¹ - 1 g catalyst - 120 °C - 0.5 h - 1 g NaOH - 10 bar O ₂	6.46	[13]
Depolymerization	CuO/Al ₂ O ₃	- Alkaline lignin 0.25 g L ⁻¹ - 1.5 g catalyst - 80 °C - 1.5 h - 1.5 g NaOH - Atmospheric pressure	5.91	This work

Conclusions

The optimization of vanillin production by the depolymerization of alkaline lignin as a renewable material was experimentally demonstrated, and the optimal conditions were determined with Box-Behnken design. Four crucial factors were studied: temperature, amount of NaOH, a reaction time, and amount of catalyst. The obtained data were statistically analyzed to create a regression equation for predicting the vanillin concentration from lignin depolymerization. The optimal lignin depolymerization conditions to achieve the highest vanillin yield were a reaction temperature of 80 °C, reaction time of 90 min, 1.5 g of NaOH, and 1.5 g

of CuO/Al₂O₃ catalyst. The average amount of lignin decomposed into vanillin under these conditions at atmospheric pressure was 59.14 mg L⁻¹. These optimal conditions are milder than those of other processes reported in the literature.

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