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Hydrothermal Production of Furfural from Corn Husk Biomass Using an $AlCl_3$–HCl Catalytic System

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ABSTRACT

Hydrothermal production of furfural from corn husk biomass was investigated using an $AlCl_3$–HCl–H_2SO_4 catalytic system. Hydrothermal treatment was carried out in a Teflon-lined reactor at 120 °C for 6 h with a solid-to-liquid ratio of 1:20 (g/mL). The catalytic system consisted of 200 mg $AlCl_3$, 330 µL HCl, and 110 µL H_2SO_4. Structural and chemical changes during the conversion process were analyzed using FTIR and HPLC. The results indicated partial degradation of lignocellulosic components, particularly hemicellulose, which promoted furfural formation during hydrothermal conversion. Quantitative analysis showed that furfural yield increased from 0.30% without catalyst addition to 2.37% with the $AlCl_3$-containing catalytic system, indicating the catalytic contribution toward hydrolysis and dehydration reactions. In addition, comparison of solvent systems showed that dimethyl carbonate (DMC) produced higher furfural yield (2.37%) than dichloromethane (DCM) (1.03%) under similar hydrothermal conditions, suggesting that solvent selection influenced furfural stabilization and conversion efficiency. Although the obtained furfural yield remained lower than values reported for optimized acid-catalyzed systems, the findings demonstrate the potential of corn husk biomass as a renewable feedstock for furfural production and provide preliminary insight into catalyst-assisted hydrothermal conversion.

Keywords: corn husk biomass, furfural production, hydrothermal process, $AlCl_3$–HCl catalyst, lignocellulosic conversion

1. INTRODUCTION

The increasing demand for chemical raw materials in Indonesia, combined with limited domestic production capacity, has encouraged the exploration of renewable biomass resources as alternative feedstocks

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for sustainable chemical production[1]. Furfural is an important platform chemical derived from lignocellulosic biomass and is widely utilized in the production of biofuels, resins, solvents, pharmaceuticals, and agricultural chemicals[2]. Due to its renewable origin and industrial importance, the development of efficient and environmentally friendly furfural production technologies has attracted considerable research interest[3].

Agricultural residues are considered promising feedstocks for furfural production because of their abundance, low economic value, and high hemicellulose content[4]. Among these residues, corn husk is a potential lignocellulosic biomass source due to its hemicellulose fraction, which can be converted into pentose sugars and subsequently dehydrated into furfural[5]. According to data from Badan Pusat Statistik (BPS), Indonesia produced approximately 15.1 million tons of corn in 2024, generating substantial amounts of agricultural waste, including corn husk[6]. Improper disposal of this biomass through open burning may contribute to environmental pollution and greenhouse gas emissions. Therefore, the valorization of corn husk into value-added chemicals such as furfural represents a sustainable strategy for biomass utilization and waste reduction[7].

The chemical composition of biomass strongly influences furfural production efficiency because hemicellulose acts as the primary precursor for furfural synthesis. Previous work by Dita Ariyanti et al. reported that corn husk contains 5.69% hemicellulose, 13.51% cellulose, and 40.06% lignin, whereas corn cob exhibited a higher hemicellulose content of 7.81%[7]. The relatively lower hemicellulose content and higher lignin fraction in corn husk may limit hydrolysis efficiency because lignin forms a rigid structure that restricts catalyst accessibility to polysaccharide components. Nevertheless, the presence of hemicellulose indicates that corn husk remains a potential renewable precursor for furfural production.

Furfural production generally involves acid-catalyzed hydrolysis of hemicellulose followed by dehydration of pentose intermediates[8]. Previous studies have reported furfural yields ranging from 35% to 84% using mineral acid catalysts under optimized conditions[9]. In recent years, combined Lewis acid–Brønsted acid catalytic systems have gained attention because of their synergistic effects during lignocellulosic conversion[10]. Lewis acids such as $AlCl_3$ can facilitate cleavage of glycosidic bonds and isomerization reactions, while Brønsted acids including HCl promote dehydration of pentose sugars into furfural[11]. The interaction of these catalysts may improve hydrolysis efficiency and furfural formation during hydrothermal treatment[12].

The chemical composition of biomass plays an important role in determining furfural production efficiency[13]. Therefore, characterization of the corn husk used in this study was conducted to determine its cellulose, hemicellulose, lignin, moisture, and ash contents. The hemicellulose fraction is particularly important because it serves as the primary precursor for furfural formation and forms the basis for yield calculations.

In addition to catalyst effects, solvent selection is also considered an important factor influencing furfural production efficiency during hydrothermal conversion. Organic solvents may improve furfural recovery by enhancing extraction efficiency and suppressing secondary degradation reactions under acidic conditions. Previous studies have reported that dimethyl carbonate (DMC) can act as a green solvent with favorable properties for furfural stabilization during biomass conversion processes. In contrast, solvent systems with lower stabilization capability may result in reduced furfural selectivity and recovery. Therefore, this study also compares the performance of DMC and DCM solvent systems under similar hydrothermal conditions to evaluate their influence on furfural formation from corn husk biomass.

Based on these considerations, this study investigates the hydrothermal conversion of corn husk biomass into furfural using an $AlCl_3$–HCl catalytic system. Rather than introducing a completely new catalytic

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approach, this work focuses on evaluating the effectiveness of the selected catalyst combination under hydrothermal conditions for corn husk conversion. FTIR and HPLC analyses were employed to evaluate structural changes and furfural formation. The reaction pathway involves hydrolysis of hemicellulose into pentose sugars followed by acid-catalyzed dehydration to produce furfural as the main product. However, this study has several limitations because the hydrothermal process was performed under fixed operating conditions without variations in temperature, reaction time, catalyst loading, and acid concentration. Therefore, further optimization studies are required to enhance conversion efficiency and improve furfural yield.

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

Corn husk biomass was used as the primary raw material. The chemicals used in this study included aluminum chloride ($AlCl_3$, ≥99%, Merck), hydrochloric acid (HCl, 37%, Merck), ethyl acetate ($C_4H_8O_2$, ≥99%, Merck), and distilled water (H_2O)[14]. Standard furfural ($C_5H_4O_2$, ≥99%, Sigma-Aldrich) was used for HPLC calibration and compound identification. All chemicals were used without further purification[15].

Standard laboratory glassware, including beakers, pipettes, watch glasses, spatulas, and glass stirring rods, was used throughout the experiment. The hydrothermal process was conducted using a Teflon-lined stainless steel autoclave reactor (100 mL capacity). The reaction was performed using a total reaction volume of 20 mL, corresponding to the biomass-to-liquid ratio used in the hydrothermal treatment. Solvent removal was performed using a rotary evaporator (Buchi Rotavapor R-300)[16].

Functional group analysis was performed using Fourier Transform Infrared Spectroscopy (FTIR; Shimadzu IRTracer-100). Furfural concentration was quantified using High Performance Liquid Chromatography (HPLC; Agilent 1260 Infinity equipped with a Coregel 87H3 column and UV detector).

2.2. Research Procedure

2.2.1 Time and Location of Research

The study was conducted at the Integrated Laboratory of Bioproducts (iLab), Research Center for Science and Technology (KST) Soekarno, National Research and Innovation Agency (BRIN), Indonesia, from July 2025 to January 2026.

2.2.2 Preparation of Biomass

Corn husk biomass was cleaned with distilled water and dried in an oven at 60 °C until constant weight was achieved. The dried biomass was ground and sieved through a 100-mesh sieve to obtain uniform particle size. Biomass characterization was conducted to determine cellulose, hemicellulose, lignin, moisture, and ash contents, since hemicellulose serves as the main precursor for furfural formation and as the basis for yield calculations.

2.2.3 Pretreatment Process

Approximately 1 g of prepared corn husk biomass was weighed and placed into a beaker containing 20 mL of distilled water, corresponding to a solid-to-liquid ratio of 1:20 (g/mL). Subsequently, 200 mg of $AlCl_3$ was added into the suspension and stirred until completely dissolved. Afterward, 330 µL of HCl was slowly

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introduced into the mixture under continuous stirring to ensure homogeneous distribution of the catalyst solution. The resulting mixture was continuously stirred for several minutes at room temperature to obtain a uniform reaction suspension before hydrothermal treatment.

The prepared reaction mixture was then transferred into a 100 mL Teflon-lined stainless steel autoclave reactor, ensuring that all biomass particles and catalyst solution were completely introduced into the reactor. The reactor was tightly sealed and subjected to hydrothermal treatment at 120 °C for 6 h under autogenous pressure.

After completion of the reaction, the reactor was allowed to cool naturally to room temperature. The reaction mixture was filtered to separate the liquid fraction from the solid residue. Furfural extraction was performed from the liquid filtrate using ethyl acetate as the extraction solvent. The organic phase was subsequently concentrated using a rotary evaporator to remove residual solvent prior to FTIR and HPLC analyses. The solid residue was washed several times with distilled water to remove residual catalyst and impurities.

The present study was designed as a preliminary investigation to evaluate the effect of the $AlCl_3$-containing catalytic system under fixed hydrothermal conditions. Therefore, variations in temperature, reaction time, catalyst loading, and acid concentration were not explored in this work and are proposed for future optimization studies.

2.2.4 Characterization and Analysis

FTIR analysis was performed within the wavenumber range of 4000–400 cm^{-1} to identify functional group changes before and after hydrothermal treatment.

Furfural quantification was conducted using High Performance Liquid Chromatography (HPLC) equipped with a Coregel 87H3 column. The analysis was carried out using an appropriate mobile phase under controlled operating conditions at a flow rate of [flow rate] mL/min and a column temperature of [temperature] °C. Detection was performed using a UV detector at [wavelength] nm with an injection volume of [volume] µL. Standard furfural solutions were used to construct a calibration curve with a linear regression coefficient (R^2) of [R^2 value]. The furfural yield was calculated using the following equation:

$$Yield \text{ furfural } (\%) = \frac{mass\ of\ furfural}{initial\ hemicellulose\ mass}\ x\ 100\% \quad (1)$$

3. RESULTS AND DISCUSSION

3.1. FTIR Analysis of Corn Husk Biomass

Fourier Transform Infrared (FTIR) spectroscopy was employed to evaluate the structural changes in corn husk biomass before and after hydrothermal pretreatment. The analysis focused on the modification of lignocellulosic components, particularly hemicellulose, which plays an important role in furfural formation. The FTIR spectra before and after pretreatment are presented in Figure 1.

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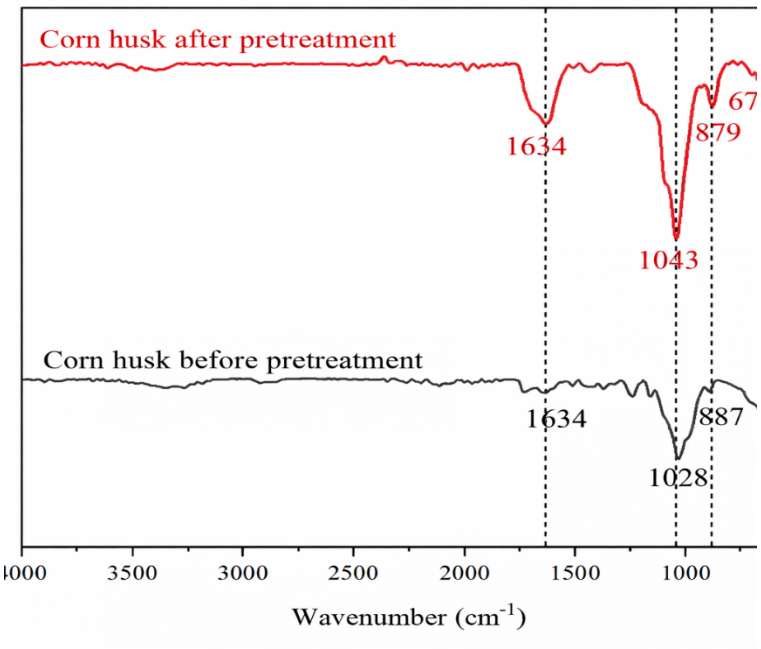

Figure 1. Corn Husk FTIR Results

The FTIR spectra presented in Figure 1 show several changes in peak intensity and position after pretreatment, indicating structural modification of the lignocellulosic matrix. The absorption peak observed at 1634 cm^{-1} corresponds to the stretching vibration of carbonyl (C=O) groups associated with lignin and hemicellulosic components. The persistence of this peak after pretreatment indicates that portions of the lignocellulosic structure remained present, although chemical modifications had occurred.

A shift in the absorption band from 1028 cm^{-1} before pretreatment to 1043 cm^{-1} after pretreatment was observed and attributed to C–O–C stretching vibrations. This shift suggests partial cleavage of glycosidic linkages in hemicellulose during hydrothermal treatment, which may improve the accessibility of pentose sugars for subsequent furfural formation.

Additional absorption bands detected at 879 cm^{-1} and 670 cm^{-1} correspond to out-of-plane C–H bending vibrations. The persistence of these bands indicates that certain cellulose and lignin structures remained undegraded, suggesting that the hydrothermal pretreatment under the applied operating conditions was not sufficient to completely disrupt the lignocellulosic network.

Although the shift in the C–O–C stretching region suggests structural modification of hemicellulose, this evidence alone is insufficient to fully confirm hemicellulose degradation. In lignocellulosic biomass, hemicellulose degradation is commonly associated with changes in several characteristic FTIR regions, including attenuation of xylan-associated bands near 1730 cm^{-1} and changes in the broad O–H stretching region around 3200–3600 cm^{-1}. In this study, these changes were not clearly observed, possibly due to overlapping absorption bands and the complex composition of corn husk biomass. Therefore, the FTIR results mainly indicate partial structural alteration of lignocellulosic components rather than complete hemicellulose removal. The assignment of the observed functional groups is summarized in Table 1.

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Table 1. FTIR Vibration Assignments of Corn Husk Biomass Here

Functional Group	Absorption Region (cm^{-1})	Wavenumber (cm^{-1})	Reference
C=O stretching vibration	1820–1620	1634	(K Fitri et al., 2024)[18]
C–O–C stretching vibration	1033	1043	(Ariyanti et al., 2025)[17]
Out-of-plane C–H bending	900–560	879 and 670	(K Fitri et al., 2024)[18]

The FTIR data presented in Table 1 are generally consistent with reported FTIR characteristics of lignocellulosic biomass. Overall, the results indicate that hydrothermal pretreatment promoted partial modification of the corn husk structure, which may facilitate the conversion of hemicellulose-derived sugars into furfural.

3.2. FTIR Characterization of Furfural

FTIR analysis was conducted to identify the functional groups present in the liquid product obtained from the hydrothermal conversion of corn husk biomass. The FTIR spectrum of the obtained product is presented in Figure 2.

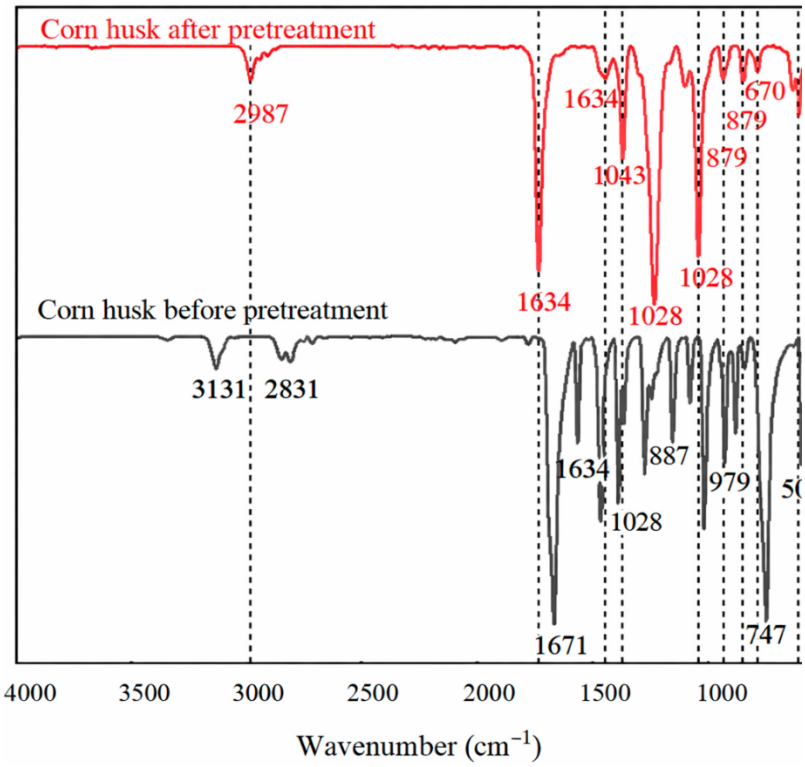

Figure 2. Wash FTIR Furfural

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The FTIR spectrum shown in Figure 2 exhibits several absorption bands associated with furfural-related compounds. A prominent absorption band was observed at 1733 cm^{-1}, indicating the presence of carbonyl-containing compounds generated during the hydrothermal process. However, this value is higher than the typical conjugated aldehyde C=O stretching vibration of pure furfural, which is commonly reported in the range of approximately 1670–1685 cm^{-1} due to conjugation between the aldehyde group and the furan ring. The observed shift may indicate the presence of residual impurities or side products such as organic acids and other oxygenated compounds formed during biomass degradation.

An absorption band at 2987 cm^{-1} was observed and is more appropriately assigned to C–H stretching vibrations associated with aldehydic or furan-ring C–H groups rather than O–H stretching vibrations. This assignment is consistent with previously reported FTIR spectra of furfural and related furanic compounds.

The absorption band at 1371 cm^{-1} corresponds to C–H bending vibrations, while the peak at 1042 cm^{-1} is attributed to C–O stretching vibrations associated with the furan ring structure. Additional peaks observed at 846 cm^{-1} and 609 cm^{-1} correspond to out-of-plane C–H bending vibrations characteristic of heterocyclic aromatic compounds.

In acid-catalyzed hydrothermal conversion, furfural is not the only product generated from lignocellulosic biomass. Several side products, including acetic acid, formic acid, levulinic acid, and 5-hydroxymethylfurfural (HMF), may also be formed through sugar degradation and secondary reactions. The relatively strong carbonyl absorption observed in the FTIR spectrum may partially originate from these oxygenated compounds. Therefore, although the FTIR results indicate the formation of furfural-related compounds, the presence of side products or incomplete purification cannot be excluded. The assignment of the observed functional groups is summarized in Table 2.

Table 2. FTIR Vibration Assignments of Hydrothermal Product Here

Functional Group	Absorption Region (cm^{-1})	Wavenumber (cm^{-1})	Reference
O–H stretching vibration	2850-3160	2987	(Ariyanti et al., 2025)[16]
C=O aldehyde stretching vibration	16700–1735	1733	(K Fitri et al., 2024)[18]
C–H bending vibration	1650–1450	1371	(Ariyanti et al., 2025)[16]
Aromatic C–H bending	1234	1234	(Ariyanti et al., 2025)[16]
C–O stretching vibration	1040	1042	(Ariyanti et al., 2025)[16]
Out-of-plane C–H bending	900–560	846 and 609	(K Fitri et al., 2024)[18]

The FTIR data presented in Table 2 generally correspond to the characteristic functional groups of furfural-containing products. The presence of carbonyl and furan-related functional groups supports the formation of furfural during hydrothermal conversion. However, the obtained product is more appropriately described as a furfural-containing hydrothermal product rather than highly purified furfural, since the presence of side products and residual impurities could not be completely excluded.

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3.3. Effect of Catalyst on Furfural Yield

The effect of catalyst addition on furfural yield was evaluated using High Performance Liquid Chromatography (HPLC). The results are presented in Figure 3 and Figure 4. Figure 3 compares furfural yields obtained under identical operating conditions (120 °C, 6 h) with and without catalyst addition, while Figure 4 presents the comparison between DMC and DCM solvent systems under similar hydrothermal conditions.

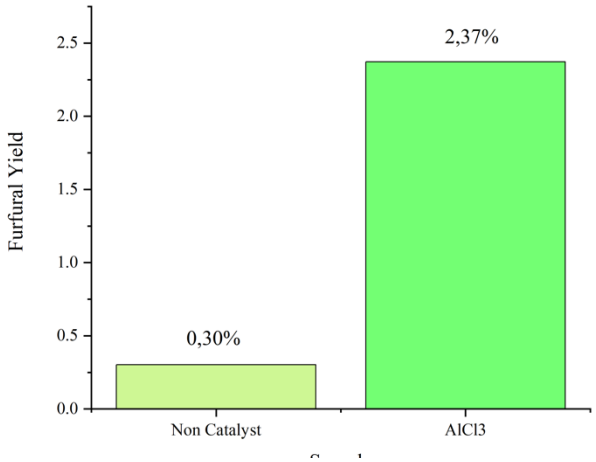

Figure 3. Catalyst Influence Graph

The results presented in Figure 3 show that the furfural yield obtained without catalyst addition at an operating condition of 120 °C for 6 h was only 0.30%, whereas the addition of $AlCl_3$ under acidic conditions increased the furfural yield to 2.37%. Although the overall yield remained relatively low, the increase indicates that catalyst addition contributed to improving the conversion of corn husk biomass into furfural-related products.

The catalytic activity of $AlCl_3$ may be attributed to its role as a Lewis acid, which facilitates the cleavage of glycosidic bonds in hemicellulose and promotes hydrolysis of polysaccharides into pentose sugars. These intermediates may subsequently undergo dehydration reactions under acidic conditions to form furfural. In the absence of catalyst addition, hemicellulose degradation proceeded less efficiently due to the relatively high activation energy required for biomass hydrolysis.

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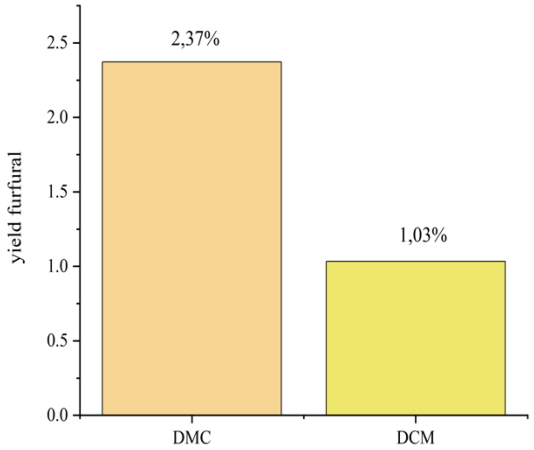

Figure 4. Effect of Solvent Type on Furfural Yield

The comparison of solvent-assisted hydrothermal systems presented in Figure 4 shows that the DMC-based system produced higher furfural yield than the DCM-based system. Under similar hydrothermal conditions at 120 °C for 6 h, the furfural yield obtained using the $AlCl_3$-containing catalytic system with DMC reached 2.37%, whereas the HCl–DCM system produced a lower furfural yield of 1.03%.

The higher furfural yield observed in the DMC-based system may be associated with the ability of dimethyl carbonate (DMC) to stabilize furfural and suppress secondary degradation reactions during biomass conversion. In contrast, the DCM-based system may provide lower stabilization efficiency under acidic hydrothermal conditions, resulting in lower furfural recovery. However, because the catalyst compositions between both systems were not identical, the observed differences cannot be attributed solely to solvent effects.

Compared with previous studies, the furfural yield obtained in this work (2.37% at 120 °C for 6 hours) was lower than yields reported under optimized operating conditions. Several studies using stronger acid systems, higher temperatures, or different catalyst configurations have reported significantly higher furfural yields from lignocellulosic biomass. The relatively low yield observed in this study may be associated with the mild operating temperature, incomplete hemicellulose conversion, and the occurrence of secondary degradation reactions.

Compared with other solvent systems under similar hydrothermal conditions, the use of dimethyl carbonate (DMC) showed higher furfural yield than dichloromethane (DCM). Under identical operating conditions at 120 °C for 6 h, the furfural yield obtained using the $AlCl_3$-containing catalytic system with DMC reached 2.37%, whereas the use of HCl with DCM resulted in a lower furfural yield of 1.03%, as presented in Figure 4. This result suggests that solvent selection significantly affects furfural formation and stabilization during hydrothermal conversion.

The higher furfural yield obtained in the DMC-based system may be attributed to the better extraction capability and stabilization effect of DMC toward furfural during the reaction process. In contrast, the DCM system may have provided lower stabilization efficiency, leading to furfural degradation or side reactions under acidic hydrothermal conditions. In addition, DMC has been reported as a greener solvent with favorable

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physicochemical properties for biomass conversion processes, particularly in suppressing undesired secondary reactions.

Table 3. Comparison of Furfural Production from Various Lignocellulosic Biomass Here

Biomass	Experimental Condition	Furfural Yield (%)	Reference
Corn husk	$AlCl_3$ under acidic condition, 120 °C, 6 h	2.37	Present study
Corn cob	DMC solvent, acidic condition, one-pot hydrothermal process, 120 °C, 2 h	93.26	Ariyanti et al. (2025)
Corn-cob, sugarcane bagasse, rice-straw, and corn-straw	DMC solvent, $AlCl_3$ and HCl under pressurized hydrothermal conditions, 180 °C, 6 h	61–98	Bains et al.
Corn cob	Butyl acetate + saturated NaCl (3:1), 12.5 wt% p-sulfonic acid calix[4]arene, 160 °C, 1 h	56	Ariyanti et al. (2025)
Corn cob	$Al_2(SO_4)_3$, 100 °C, 2 h	13.2	Ariyanti et al. (2025)
Corn stalk	0.05 mol/L H_2SO_4, $LiBr{\cdot}3H_2O$, DCM, 120 °C, 90 min	72.5	Ariyanti et al. (2025)
Sugarcane bagasse	8.3 wt% H_3PO_4, 25% $AlCl_3$, acetone/water (7:3), 170 °C, 5 min	72.7	Ariyanti et al. (2025)

Compared with previous studies, the furfural yield obtained in this work was considerably lower than those reported under optimized reaction conditions. Ariyanti et al. reported a furfural yield of 93.26% from corn cob biomass using a one-pot hydrothermal process with dimethyl carbonate (DMC) as a green solvent at 120 °C within 2 h[16]. In addition, Bains et al. reported furfural yields ranging from 61% to 98% from various lignocellulosic biomass feedstocks under hydrothermal conditions using DMC solvent with $AlCl_3$ and HCl catalysts at 180 °C for 6 hours[17]. These studies demonstrate that catalyst systems, solvent selection, reaction temperature, and operating conditions strongly influence furfural production efficiency.

The relatively low yield obtained in the present study may be associated with the mild operating temperature (120 °C), incomplete hemicellulose conversion, and the occurrence of secondary degradation reactions that reduced furfural selectivity. In addition, the absence of an organic extraction solvent such as DMC may have limited furfural stabilization during the hydrothermal conversion process.

Overall, the results indicate that catalyst addition improved furfural formation from corn husk biomass under hydrothermal conditions. Nevertheless, further optimization of catalyst composition, reaction temperature, reaction time, and purification procedures is still required to improve furfural yield and product selectivity. One limitation of this study is that detailed product selectivity analysis was not comprehensively performed; therefore, the exact composition and concentration of side products could not be fully determined.

4. CONCLUSION

The hydrothermal conversion of corn husk biomass under acidic conditions with the addition of $AlCl_3$ produced furfural-related compounds, as indicated by FTIR analysis showing the presence of carbonyl and furan-associated functional groups. The FTIR results also suggested partial structural modification of lignocellulosic components after pretreatment, particularly in the hemicellulosic fraction. Under the operating

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condition of 120 °C for 6 h, the furfural yield increased from 0.30% without catalyst addition to 2.37% with the addition of $AlCl_3$ under acidic conditions, indicating that catalyst addition contributed to improving biomass conversion. However, the obtained yield remained relatively low compared with previously reported acid-catalyzed hydrothermal systems in the literature. The comparison between solvent systems also indicated that DMC provided higher furfural yield than DCM under similar hydrothermal conditions, suggesting that solvent selection influences furfural stabilization and overall conversion efficiency. The low yield may be associated with mild operating conditions, incomplete hemicellulose conversion, and the occurrence of secondary degradation reactions that reduced furfural selectivity.

This study only compared systems without catalyst and with combined catalyst conditions; therefore, the individual catalytic contributions of $AlCl_3$ and acidic conditions could not be distinguished, and no direct conclusion regarding synergistic effects can be established. In addition, detailed product characterization and selectivity analysis were not comprehensively performed, so the presence and composition of side products could not be fully determined. Overall, the results indicate that corn husk biomass has potential as a feedstock for furfural production through hydrothermal conversion. Nevertheless, further studies involving catalyst optimization, separate catalyst evaluation, higher reaction temperatures, and more comprehensive product characterization are necessary to improve furfural yield and process selectivity.

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