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Comparison of Furfural Production from Corn Waste and Empty Fruit Bunch (EFB): Effect of Time, Temperature, and Solvent

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Abstract. Furfural is a compound derived from xylose, a building block material. Sources of furfural can come from various corn industry wastes such as corn cobs and corn husks, and palm oil industry wastes like Empty Fruit Bunch (EFB). This study aims to compare the potential of furfural production from corn industry waste and palm oil industry waste. The research method involves the hydrolysis and dehydration of hemicellulose using two types of solvents, sulfuric acid (SA) and Deep Eutectic Solvents (DES), at varying temperatures (160 °C, 170 °C, and 180 °C) and reaction times (60, 120, and 180 minutes). The highest furfural yield from corn cobs, 7.69 g/L, was obtained using 1 M sulfuric acid solvent at 160 °C for 60 minutes. The optimal condition for producing a high concentration of furfural from corn cobs was achieved using DES at 170°C for 60 minutes, yielding approximately 13.18 g/L. In contrast, furfural production from EFB resulted in a concentration of 4.70 g/L.

Keywords: Corn Cob, Corn Husk, Empty Fruit Bunch, Furfural, Lignocellulosic

INTRODUCTION

Furfural is a widely utilized compound, serving as a solvent in petroleum refining and nylon manufacturing (Dashtban *et al.*, 2012). Its demand has steadily increased in line with

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the growing needs of the petroleum industry. Furfural can be derived from lignocellulosic biomass, which is rich in hemicellulose, including corn cobs, husks, empty oil palm bunches, wheat, rice husks, wood, shells, and similar materials (Juwita *et al*., 2012; Muryanto *et al*., 2022; Panjaitan *et al*., 2017).

Corn is a staple food consumed globally as a primary food source and as animal feed. The high consumption of corn has led to a significant increase in corn waste. In 2020, Indonesia's corn production reached 29.02 million tons (Ditjetbun, 2019). The primary byproducts of corn agro-industrial processing are corn husks (produced before degraining) and cobs (produced after degraining). Research indicates that these materials together account for up to 65% of the total waste generated throughout the corn processing value chain, with their mass being comparable to that of the corn grains produced (Awosusi *et al*., 2017). The processing of corn waste, such as cobs and husks, remains underdeveloped. Corn cobs and husks are promising raw materials for furfural production due to their high hemicellulose content.

The synthesis of furfural from lignocellulosic biomass typically involves the hydrolysis of hemicellulose into xylose, followed by the dehydration of xylose to form furfural. Several factors influence these stages, including time, temperature, concentration, the nature of reactants, and stirring speed (Chen *et al*., 2019; Sun *et al*., 2019; Ye *et al*., 2021).

Sulfuric acid is commonly used as a solvent and a catalyst in furfural production. In addition to sulfuric acid, various chemicals such as metal chlorides, organic acids, and hydrochloric acid have been utilized (Lee and Wu, 2021). However, these solvents pose environmental hazards and can cause equipment corrosion, prompting extensive research into alternative solvents to replace sulfuric acid.

Deep eutectic solvents (DES) have recently gained popularity as green solvents for biomass conversion. It is easier to synthesize, generally more cost-effective, and less hazardous (Smith *et al.*, 2014). DES has been widely applied in biomass pretreatment, lignin isolation, chemical extraction, and other related applications (Ai *et al*., 2020). Several studies have also reported using DES in furfural production (Zhang *et al.*, 2014).

Although furfural production from lignocellulose biomass has been widely reported, this study introduces variations in process parameters under consistent environmental conditions to minimize the impact of differing external factors on furfural yield. This study aims to synthesize furfural by comparing different lignocellulosic feedstocks, such as corn cobs, corn husks, and empty oil palm bunches (EFB). Additionally, it evaluates the effect of temperature, reaction time, and solvent type on furfural yield.

MATERIALS AND METHODS

Materials

The corn cobs and husks utilized in this research were collected from Sumbawa, West Nusa Tenggara, and the empty fruit bunches were obtained from a palm oil plantation in Sumatra, Indonesia. The furfural solution, used as a reference standard and sulfuric acid, was sourced from Merck. Analytical-grade chemicals, including xylose, choline chloride, and oxalic acid, were purchased from Sigma-Aldrich.

Solvent Preparation

The acidic solvent used in this research was 1M sulfuric acid, prepared by diluting concentrated sulfuric acid. Additionally, a Deep Eutectic Solvent (DES) was also used. The DES was generated by combining oxalic acid as the hydrogen bond donor and choline chloride as the hydrogen bond acceptor in a 1:1 molar ratio. The mixture was heated for 60 minutes at 80–100°C using an oil bath until a clear and homogeneous solution was obtained (Abbott, 2022).

Furfural production

The synthesis of furfural from corn cobs and corn husks was carried out by introducing 20% of the substrate into 1M sulfuric acid solution. The process was conducted using an autoclave reactor with a stirring mechanism in a heated oil bath. Once the oil bath reached 160°C, the hydrothermal autoclave was inserted, and the magnetic stirrer was set to 300 rpm. The hydrolysis and dehydration of the corn cobs and corn husks were performed at 160°C with varying durations of 60, 120, and 180 minutes. Similar experiments were conducted at temperatures of 170°C and 180°C. The study also explored solvent variations using DES and substrate variations by incorporating empty fruit bunches (EFB).

After the hydrolysis and dehydration processes were completed, ice rapidly cooled the reactor. The reaction mixture was then removed from the reactor and filtered through a sieve. The resulting filtrate was transferred into a centrifuge tube and centrifuged at 3000 rpm. The supernatant was collected, placed in glass bottles, and stored for further analysis using UV-Vis spectrophotometry.

Analytical Methods

Furfural analysis was performed using an aniline acetate reagent. This reagent was prepared by mixing aniline, acetic acid, and 98% ethanol in a volume ratio of 1:1:8. A standard calibration curve for furfural solutions was established based on absorbance measurements. For sample analysis, the aniline acetate reagent was added to the samples, inducing a color change to red. The absorbance of the samples was then measured using a UV-Vis spectrophotometer at a wavelength of 515 nm.

RESULTS AND DISCUSSION

Furfural can be produced from lignocellulosic biomass through hydrolysis and dehydration processes. Hydrolysis converts hemicellulose present in lignocellulosic into xylose, while subsequent dehydration of xylose results in furfural formation with the release of water. The conversion of lignocellulose to furfural can be facilitated using acidic solvents, such as sulfuric acid. The large hemicellulose molecules are broken down during hydrolysis into simpler components, specifically pentosan, which is further hydrolyzed into pentose sugar. The mechanism involves proton (H^+) interaction with the glycosidic oxygen (glycosidic bond) connecting the hemicellulose monomers. This reaction is followed by the glycosidic bond (C-O) cleavage, forming a carbonium ion. The carbonium ion then reacts with H_2O and undergoes deprotonation to form xylose (Tuas and Lerrick, 2017).

During the dehydration process, the pyranose ring of xylose undergoes protonation by H^+, followed by the removal of water (dehydration) to form a carbonium ion. The process continues with the ring's opening in the carbonium ion, which undergoes further deprotonation by acid to form polyalcohol. This is followed by sequential deprotonation and dehydration steps catalyzed by acid, eventually forming the aromatic furfural ring (Tuas and Lerrick, 2017).

Effect of Temperature and Time on Furfural Concentration

Corn cobs can be converted into furfural using acidic solvents, as shown in Figure 1. Based on Figure 1, the furfural concentration obtained from corn cobs using sulfuric acid at different temperatures and reaction times indicates that prolonged hydrolysis and dehydration times decrease in furfural concentration for both corn cobs and corn husks. The highest furfural concentration was achieved at 160°C for 60 minutes, yielding 7.96 g/L. These results are consistent with the study conducted by Ramadhan *et al.* (2022) (Ramadhan *et al.*, 2022), which reported that furfural yield declined when the reaction time. This is because prolonged operation leads to the degradation of furfural into simpler organic compounds. This study produced the most significant furfural concentration within 60 minutes.

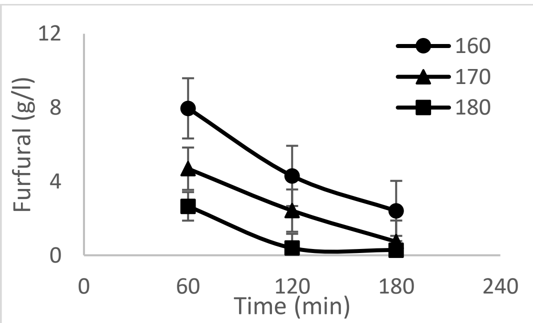

Fig. 1: Furfural concentration from corn cob in a variation of time and temperature

Temperature is another factor influencing the furfural concentration produced. Based on Figure 1, it is clear that the highest furfural concentration was obtained at 160°C. However, the furfural concentration declined as the temperature increased beyond this point. At higher temperatures, the decomposition reaction of hemicellulose into furfural tends to be faster but may also accelerate side reactions, such as the degradation of furfural into unwanted by-products. This is due to furfural's thermal sensitivity, as it easily degrades under extreme conditions (Cousin *et al.*, 2022). Consequently, although higher temperatures may initially enhance furfural formation, the degradation rate surpasses the production rate, resulting in lower furfural concentrations.

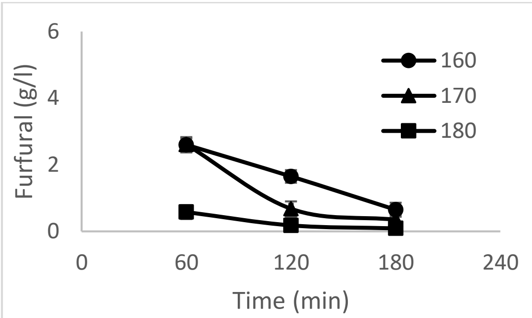

Fig. 2: Furfural concentration from corn husk in a variation of time and temperature

Corn husks, a byproduct of corn kernel processing for concentrate feed, were also treated similarly to assess their potential as a furfural feedstock. The furfural concentration from corn husks is shown in Figure 2. Figure 2 shows a similar trend was observed, where prolonged reaction times and higher temperatures led to lower furfural concentrations. The highest furfural concentration from corn husks, 2.61 g/L, was obtained at 160°C for 60 minutes. The furfural concentration produced by corn husks is lower than that of corn cobs. This difference is likely due to corn husks' lower hemicellulose content than corn cobs (Awosusi *et al.*, 2017).

Considering the trend observed in previous experiments, where increasing temperatures reduced furfural concentration, an additional experiment was conducted at a temperature lower than 160°C for 60 minutes using corn cob substrates, as shown in Figure 3. When the temperature was reduced to 150°C, the furfural concentration decreased to 7.28 g/L. This finding confirms that this study's optimal temperature for furfural production was 160°C.

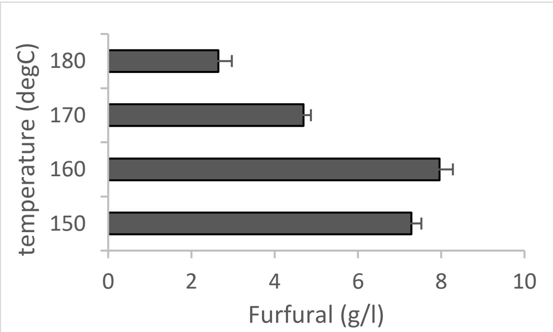

Fig. 3: Variation temperature in furfural production from corn cob

Effect of Substrate on Furfural Concentration

The substrate variation was conducted to evaluate the potential of other lignocellulosic materials as feedstock for furfural production. Empty Fruit Bunches (EFB) were used in this study. Based on Figure 4, it can be observed that the type of substrate and the duration of the process significantly affect the furfural concentration produced. Corn cob yielded the highest furfural concentration after 60 minutes, at 7.96 g/L, followed by EFB at 4.7 g/L, and corn husk with the lowest concentration of 2.6 g/L. These results indicate that corn cob exhibits the highest potential for furfural production among the three substrates within a shorter processing time. Additionally, the furfural yield from EFB remained lower than that from corn cob.

Table 1. Chemical composition of corn waste and EFB

	Lignin	Cellulose	Hemicellulose
Corn cob	26.47 ±0.25	31.69 +0.16	24.39 ±0.26
Corn husk	33.35 ±0.35	28.95 +0.24	15.15 ±0.21
EFB	39.13 ±0.28	32.15 +0.17	20.88 ±0.09

The differences in chemical composition among the substrates, particularly in hemicellulose, lignin, and cellulose content, are likely the primary factors affecting the final furfural yield. Table 1 shows the chemical composition of corn cob, corn husk, and EFB. The hemicellulose content of EFB is lower compared to corn cob. Previous studies have shown that the hemicellulose content in corn cob can reach 30-40% (Awosusi *et al.*, 2017; Pointner *et al.*, 2014), while in EFB, it only reaches 14-25% (M Muryanto *et al.*, 2022). Zou *et al.* (2021) stated that corn cob has a sponge-like microstructure in the pith, an irregular elliptical shape, and several small micropores in the cellulose microfibrils (Zou *et al.*, 2021). Corn cob, with its more porous structure and higher hemicellulose content, facilitates better diffusion of intermediates during hydrolysis.

In contrast, EFB has a denser structure with a surface covered by waxes and silica (Triwahyuni *et al.*, 2021). This structure and a higher lignin content may hinder the hydrolysis of hemicellulose and reduce the efficiency of furfural formation in the early stages of the reaction.

The difference in structural composition plays a critical role in determining the effectiveness of substrate conversion to furfural (Ye *et al.*, 2021). Corn cob, with its higher hemicellulose availability and porous structure, allows for a more efficient breakdown of its components during the hydrolysis and dehydration. This leads to a higher furfural yield within a relatively shorter time frame. On the other hand, EFB, despite being a suitable lignocellulosic material, presents more challenges in terms of hemicellulose accessibility due to its potentially higher lignin content, which acts as a barrier to the hydrolysis process.

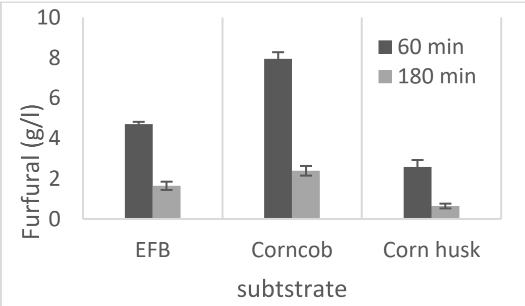

Fig. 4: Comparison of furfural production from EFB, corn cob, and corn husk at 160ºC

Corn husk also showed the lowest furfural concentration, possibly due to its lower hemicellulose content than corn cob and EFB. Corn husk may also possess a less favorable structure for the breakdown of lignocellulosic material, further contributing to its lower furfural yield. This suggests that the substrate selection for furfural production should be carefully considered, with particular attention to the hemicellulose content and structural characteristics that influence the hydrolysis and dehydration processes.

Effect of Solvent on Furfural Concentration

Figure 5 shows the furfural concentrations produced using DES and sulfuric acid (SA) at two temperature variations (160°C and 170°C). It can be observed that the use of DES solvents and sulfuric acid, as well as temperature variation, significantly affects the production of furfural from two types of substrates, corn cob and corn husk. In general, DES solvents produce higher furfural concentrations than sulfuric acid (SA), especially at higher temperatures (170°C).

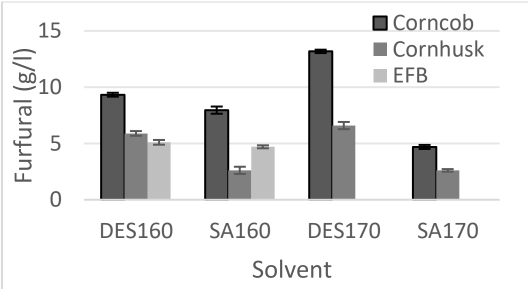

Fig. 5: Variation of solvent in furfural production from corn cob

Using DES on corn cob at 160°C (DES160) yields 9.33 g/l of furfural, whereas sulfuric acid at the same temperature (SA160) only produces 7.96 g/l. This difference indicates that DES is more effective in facilitating hemicellulose decomposition at moderate temperatures. DES, which is typically a combination of salts and hydrogen donors, can dissolve lignocellulosic components more efficiently, allowing better access to hemicellulose for furfural production (Muryanto *et al.*, 2023; Yong *et al.*, 2022).

The disparity between DES and SA becomes more significant when the temperature is increased to 170°C, corn cob processed with DES at 170°C produces the highest furfural yield, at 13.18 g/l, compared to 4.69 g/l when using SA at the same temperature. This temperature increase appears to favor DES solvents, which can accelerate hemicellulose hydrolysis and optimally enhance furfural production. In contrast, using SA results in a decrease in furfural concentration with increasing temperature, likely due to the accelerated degradation of furfural at higher temperatures (Lee and Wu, 2021; Mittal *et al.*, 2017). Furfural can still be produced at lower temperatures but requires a higher acid concentration. Additionally, the furfural yield at lower temperatures is lower than that obtained at higher processing temperatures (Amraini *et al.*, 2023).

A similar trend is observed for corn husk, although furfural concentrations are generally lower than those obtained from corn cob. At 160°C, DES yields 5.89 g/l of furfural, whereas SA only produces 2.61 g/l. This indicates that corn husk contains lower hemicellulose content than corn cob, it can still generate a significant amount of furfural when DES is used as the solvent.

Corn husk processed with DES produces 6.59 g/l of furfural when the temperature is raised to 170°C, slightly higher than at 160°C. However, corn husk processed with SA at 170°C produces the same amount of furfural as at 160°C, 2.61 g/l. This suggests that the temperature increase positively affects DES but has no significant impact on SA in improving furfural yields from corn husks.

The effectiveness of DES solvents compared to sulfuric acid (SA) in producing furfural may be due to DES's ability to maintain furfural stability during the reaction process. DES dissolves hemicellulose effectively, and helps prevent furfural degradation at high temperatures (Lee and Wu, 2021). Meanwhile, sulfuric acid, while effective at breaking down hemicellulose structures, tends to accelerate side reactions that lead to furfural degradation, particularly at higher temperatures such as 170°C. This highlights the superior performance of DES in supporting furfural formation from biomass materials.

Furfural production from Empty Fruit Bunches (EFB) using DES follows a similar trend to that observed with corn-based biomass. In this study, hydrolysis of EFB was conducted at 160°C, yielding a furfural concentration of 5.89 g/L. This yield is notably higher than that obtained through conventional hydrolysis using sulfuric acid, further demonstrating DES's potential as a more effective and sustainable solvent for furfural production.

CONCLUSIONS

The study highlights the significant effects of temperature, processing time, substrate type, and solvent selection on furfural concentration derived from corn cobs, corn husks, and Empty Fruit Bunches (EFB). The optimal conditions for furfural production were 160°C for 60 minutes, where corn cobs yielded the highest concentration of 7.96 g/L. Prolonged processing times and increasing temperatures beyond this point led to decreased furfural concentrations due to thermal degradation. Substrate variation revealed that corn cobs facilitated superior furfural yields compared to corn husks and EFB. The lower hemicellulose content in EFB and corn husks limited their potential, underscoring the importance of selecting suitable substrates based on their chemical composition. Furthermore, Deep Eutectic solvents (DES) demonstrated a marked improvement in furfural yields compared to sulfuric acid, particularly at elevated temperatures. DES enhanced the efficiency of hemicellulose breakdown and mitigated furfural degradation during the reaction process, confirming its superior performance as a solvent in furfural production.

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