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A MICROWAVE-ASSISTED ACID HYDROLYSIS OF CORNCOB: RAPID FURFURAL PRODUCTION AND ITS KINETIC

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ABSTRACT

The microwave-assisted hydrolysis of corncob was successfully performed using H_2SO_4 *as an acid catalyst for rapid furfural production. The influences of reaction time and the catalyst amount on the yield of furfural production were discussed comparatively. The hydrolysate obtained from the reaction was characterized by an aniline-acetate test and FT-IR to confirm furfural generation. The degradation of the cell wall structure of corncobs during the reaction was investigated by SEM and FT-IR analysis of the solid before and after the reaction. The results showed that hemicelluloses were released from the cell wall to be converted into furfural. The highest yield of furfural was reached after a 5 min reaction under microwave irradiation, which was 9-fold faster than a reaction with conventional heating methods for the same amount of furfural (a 45 min reaction). It signifies that the chemical reaction could be performed efficiently using microwave irradiation which provides better heating distribution, faster heating, and a more controllable heating mechanism compared to conventional heating methods.*

Keywords: biomass, hemicellulose, xylose, dehydration, sulfuric acid, irradiation.

INTRODUCTION

Biomass, as a material derived from agricultural crops, algae, and organic waste, has been widely used as a raw material in the process of conversion and manufacture of chemical compounds. One way to protect natural resources is through the utilization of materials that are derived from nature, coupled with the practice of waste reduction and reuse [1]. From an economic perspective, it is crucial for the conversion process to utilize a biomass source that is readily accessible and affordable. Plant biomass waste, such as the waste generated by the wood industry, agricultural sector, and paper industry, fulfils these criteria [2].

Lignocellulose as the main biomass can be derived from natural resources such as wood and agricultural waste, which contain cellulose, hemicellulose, and lignin [3]. Hemicellulose, which is a polysaccharide found in plant cell walls, is the second most abundant after cellulose. Its structure lacks a distinct shape and accounts for approximately 15 to 30 % of the weight of lignocellulosic compounds. Hemicellulose hydrolyses more easily than crystalline cellulose, resulting in a higher concentration of C_5 monosaccharides, specifically xylose (a pentose sugar). Chemical compounds like furfural, ethanol, xylitol, and other small molecules are produced through the conversion of xylose [4].

Hemicellulose and cellulose can be hydrolysed into fermentable sugars; the process of cellulose hydrolysis produces glucose, which is used in alcohol fermentation, whereas hemicellulose hydrolysis produces xylose as the product. Some parameters affecting sugar hydrolysis include solvent volume, amount of catalyst used, temperature, and reaction time [5]. Hemicellulose-rich biomass is used as a primary source for furfural production. Furfural can be synthesized in one or two stages, with either batch or continuous processes. Wheat straw, corn cob, and sugarcane bagasse are examples of hemicellulose-rich biomass that have been successfully converted into furfural [6 - 8]. Several physical, chemical, and biological approaches have been used to pre-treat lignocellulosic biomass. The combined physicochemical method, which included acid catalysis as well as enhanced temperature and pressure has been the most effective [2].

Furfural is produced through a two-step process involving the conversion of pentosan or hemicellulose into pentose through hydrolysis and then the dehydration of pentose into furfural. Acid hydrolysis can produce higher sugar yields and good reproducibility when compared to other methods [9]. Under acidic conditions, hemicellulose can be hydrolysed to produce furfural as an intermediate product, xylose [10]. The process of producing furfural through the hydrolysis method using homogeneous acid catalysts of mineral acids is conducted in an integrated process where the two main steps are not separated but move from one to another in a continuous process [11]. Despite its high efficiency in furfural production, this method has some disadvantages, including the generation of large amounts of acidic wastewater, the use of non-recyclable homogeneous catalysts, and the risk of equipment corrosion [12].

Several studies have been conducted on producing furfural using mineral acids such as sulfuric acid and hydrochloric acid [13, 14]. The experiments were conducted at temperatures of about 140 - 275^{o}C and acid concentrations of 1.5 - 2 M. These operating conditions can increase furfural production while also increasing the dense black suspension known as humin [15]. Acid catalysts can break down large molecules into smaller ones. However, they may have a negative impact on the quality of the resulting products. In addition, the effects of using low levels of acid in the hydrolysis process have not been thoroughly investigated [16].

The use of microwaves in furfural synthesis is deemed a highly efficient approach for producing furfural. Furfural production in two steps has been conducted using acid hydrolysis followed by microwave treatment. These techniques enhance the dissolution of hemicellulose and thus the efficiency of furfural production [1]. Acid hydrolysis as the first step was carried out with a solid/liquid ratio of 20 % w/v for 60 min at 130^{o}C and 2.59 % w/v H_2SO_4. The second step, employing the previously produced hydrolysate carried out in a microwave reactor at 200^{o}C and 850 W with the addition of 0.1 M $FeCl_3$, produced a solution with 18 g L^{-1} furfural and a yield of 63.3 %, equivalent to almost 90 kg of furfural per ton of processed solids [17].

One of the crucial steps in furfural production from biomass is the pentose dehydration reaction. $FeCl_3$ in various quantities and sulfuric acid were used as catalysts. The best operational conditions were at 210°C for 0.5 min, and 0.05 M $FeCl_3$ in a microwave reactor for the highest furfural yield of 57 % [18]. Another experiment for furfural production from xylose and hemicellulose in bamboo was carried out in a biphasic 1-butyl-3-methylimidazolium hydrogen sulphate medium with microwave radiation. The addition of a small amount of water can increase the total furfural yield, and the highest furfural yield from bamboo xylose and hemicellulose can reach 63.87 % mol and 85.69 % mol [19].

The microwave method of heating biomass samples can accelerate the rate of degradation of complex carbohydrates into simple sugars when compared to conventional heating; this process will automatically reduce the reaction time [20]. The increase in furfural yield and shorter hydrolysis time are the advantages of using microwaves in furfural production [21]. Microwave heating is both faster and more energy-efficient compared to traditional heating methods. The process can be stopped immediately, allowing for better control of the reaction temperature [2]. Microwave radiation has been applied successfully for the thermochemical conversion of cellulose, conversion of wood waste to levulinic acid, extraction of lignin from plant biomass, and ethanol production [22].

The duration of the microwave heating process has an impact on the results obtained. If the biomass is heated for a short duration of 15 minutes, it can break down the lignocellulosic material into various components. However, if the heating process is conducted for a longer duration, along with a high catalyst concentration and high temperatures, it can result in biomass combustion [23]. Hydrolysis of polysaccharides into sugar monomers can be inhibited by the presence of lignin, but the breakdown into glucose is slowed by the crystallinity of cellulose, so amorphous cellulose and xylan greatly affect the amount of glucose and xylose released [24 - 26]. Optimal conditions will be reached after heating for a certain time; if these conditions are passed, the furfural yield will decrease. Furfural can be decomposed back into other compounds through the breakdown of the aldehyde group in the subsequent reaction of the hydrolysis process [21].

The selection of solvent system is another crucial element that can impact both the productivity of furfural and the sustainability of the process. These solvents function by lowering the activation energy and enhancing the ability of the biomass to dissolve. The use of biphasic solvents can minimize side effects and drag effects [8]. Microwave heating occurs at less severe reaction conditions and has the potential to greatly decrease reaction duration, expedite the process with enhanced productivity, and conserve energy [27].

Corn cobs contain the highest amount of fiber compared to rice husks and sawdust. This high amount of fiber influences the high moisture content of corn cobs [11]. Preliminary tests on the raw material show that it contains pentosan which is present in hemicellulose and plays an important role in furfural production [28]. This raw material is considered a good source of furfural production. Different raw materials provide different amounts of furfural. This can be influenced by the pentosan content in different materials in producing pentose for furfural production. Corn cob has the highest pentosan level compared to rice husk and sawdust [11]. Furfural production with corn cob conversion was conducted with a very low water/solids ratio (< 1). A furfural yield of 65.67 % mol was achieved in 10 min. Pressure significantly increased the furfural yield and shortened the reaction time [29]. This study was conducted to determine the efficiency of using microwaves in the hydrolysis of pentosan from corn cob with an acid catalyst to produce furfural. The optimal operating conditions for furfural production will be found. The use of microwaves is expected to increase furfural production compared to conventional methods.

EXPERIMENTAL

Materials

The materials used in this research included acetic acid, NaOH 5 %, sulfuric acid (H_2SO_4) 98 %, HCl 12 %, potassium iodide 10 %, sodium thiosulfate 0.1 N, potassium bromate, potassium bromide, indicator amylum, aquadest were purchased from Sigma-Aldrich, Singapore. The corncob used in this study was obtained from a farm in South Kalimantan Province, Indonesia. It was first washed thoroughly to remove any undesirable particles before dried naturally. When the sample was completely dried, physical pre-treatment was performed by chopping and crushing the materials to increase the reaction surface and reduce the size of the raw material. Prior to the experiments, corncob was ground into particles with a 60-size mesh and then oven-dried at 100^{o}C to a constant weight. The dried corncob was delignified with a 10 % NaOH solution (solid-to-liquid ratio of 1:100 g.mL^{-1}) for 2 h at 50^{o}C. After the delignification process, the solid fraction was washed with warm distilled water at 40^{o}C and dried to obtain delignified corncob at 80^{o}C to a constant weight.

Microwave-assisted hydrolysis

Microwave irradiation was carried out utilizing a microwave oven with a thermocouple and a temperature control. Microwave intensity and irradiation time were set by a control panel. According to the experimental design, 10 g of delignified corncob were suspended in 250 mL of a dilute sulfuric acid solution in each run. A microwave irradiation power of 400 W was applied to heat the suspension to 100^{o}C and held for the desired time (30, 45, 60, 75, and 90 min).

The kinetic of furfural production

The Saeman model was used to estimate the conversion of corn cobs into furfural. In this study, a simplified version of the model was applied, focusing on furfural formation and disregarding the production of intermediate compounds. Subsequently, first-order irreversible consecutive reactions were defined, encompassing furfural production from hemicellulose and the degradation of furfural into by-products. The model is expressed as in Eq. (1):

Hemicellulose (HE) $\overset{k_1}{\rightarrow}$ Furfural (Fu) $\overset{k_2}{\rightarrow}$ Degradation Product (DP) (1)

The reaction rate equations used for the Saeman model of the kinetics are presented in Eq. (2 - 4):

$$\frac{d[HE]}{dt} = -k_1[HE], t = 0 \rightarrow |HE| = [HE]_0 \quad (2)$$

$$\frac{d[Fu]}{dt} = k_1[HE] - k_2[Fu], t = 0 \rightarrow |HE| = [HE]_0, [Fu] = 0 \quad (3)$$

$$\frac{\mathrm{d[DE]}}{\mathrm{dt}} = -\mathrm{k_2[Fu]}, \mathrm{t} = 0 \ \rightarrow |\mathrm{Fu}| = 0 \qquad (4)$$

where [HE], [FU], and [DE] represent the concentrations of corn cobs (primarily composed of hemicellulose), furfural, and degradation products, respectively. $[\mathrm{HE}]_0$ refers to the initial concentration of hemicellulose, while k_{H1} and k_{H2} are the rate constant for the degradation of hemicellulose and furfural, respectively. The influence of reaction time on furfural concentration is described in Eq. (5):

$$[\mathrm{Fu}] = \frac{\mathrm{k_1[HE]_0}}{k_2 - k_1}\left[e^{-k_1 t} - e^{-k_2 t}\right] \qquad (5)$$

Characteristics of raw materials and products

Analysis of hemicellulose, cellulose, and lignin content was conducted on corn cob powder. The morphology of corn cob powder before and after hydrolysis was analysed by scanning electron microscopy (SEM) on a JEOL JSM-6500 LV microscope at 5 kV. Qualitative analysis for furfural formed was done with aniline acetate. FT-IR analysis was conducted to identify the functional groups formed in the furfural product produced. This characterization was executed in the range of 450 - 4500 $\mathrm{cm^{-1}}$ using FT-IR, Bio-rad, Digilab FTS-3500.

RESULTS AND DISCUSSION

Product characterization

Furfural could be produced from xylose-rich lignocelluloses such as corncob, baggase, rice husk, and pine wood [29 - 31]. Corn cob is suitable as raw material for furfural production since the amount of hemicellulose in corn cob is quite high, almost similar to its cellulose content. Table 1 shows the lignocellulose composition of corn cob, which was utilized in this study. Hemicellulose could be released from the cell wall and then continued into xylose formation by the hydrolysis process. As a target product, furfural could be generated by the dehydration of xylose using an acid catalyst [4].

Table 1. The composition of lignocellulosic compounds in corn cob.

Lignocellulosic compound	Composition, %
Cellulose	36.35
Hemicellulose	31.19
Lignin	19.22

It is quite challenging to produce furfural from biomass. In many cases, the xylose, a wood sugar compound that was yielded by the hydrolysis of hemicellulose, was degraded into other compounds instead of furfural. Two qualitative analyses have been performed to confirm the existence of furfural as a product from the hydrolysis of corn cob, namely aniline-acetate analysis and FT-IR analysis.

The aniline-acetate analysis is a qualitative test for the presence of furfural after a hydrolysis reaction. The furfural, which is contained in hydrolysate, could react with aniline-acetate to produce a bright pink colour. The aniline-acetate test can detect furfural at low concentrations [32]. The negative control was composed of corn cob powder and distilled water. The result showed no colour generation after the aniline-acetate reagent was added to the mixture (Fig. 1a). The analysis revealed that the sample containing hydrolysate was pink (Fig. 1b). It indicates the furfural had been produced after the hydrolysis reaction of corn cob which was catalysed by H_2SO_4 under specified reaction conditions.

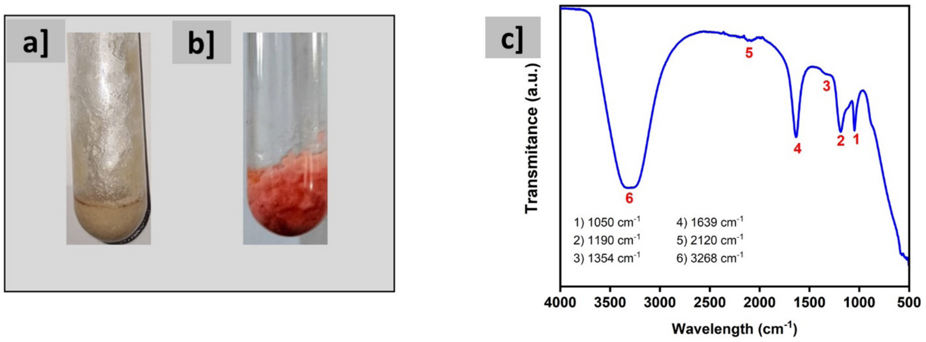

Fig. 1. Colour generation of the hydrolysate in aniline-acetate analysis, (a) before and (b) after the hydrolysis reaction; (c) FT-IR analysis of hydrolysate after a 5-min reaction at 100°C with 1 % H_2SO_4 under microwave-assisted hydrolysis.

Furthermore, the hydrolysate was analysed by Fourier-transform infrared spectroscopy (FT-IR) to confirm the furfural as the target product (Fig. 1c). The broad absorption peak was detected at a range area of 3400 - 3200 cm^{-1}, which indicated phenolic groups. The saturated aliphatic C-H stretching bands range from 3000 - 2850 cm^{-1}. The α-β unsaturated ketone was indicated by strong C=C stretching at 1639 cm^{-1}. An aldehydic C-H bending peak was detected at 1354 cm^{-1}. The small peak at 1190 - 1050 cm^{-1} is depicted as CO stretching, which represents carboxylic acid [16, 33]. According to both qualitative analyses, furfural was successfully produced and was well detected in the hydrolysate after the acid hydrolysis of corn cob.

Microwave-assisted process for furfural production

Recently, the utilization of microwave irradiation to accelerate chemical reactions has gained attention as a less energy-intensive and environmentally benign process. In this study, microwave-assisted acid hydrolysis could be convinced to be a very effective technology for accelerating furfural production from corncob due to the rapid heat generation [34].

The furfural production was carried out at 100^{o}C in a microwave-assisted reactor with a liquid-solid ratio (LSR) of 10:1. The influence of the reaction time on furfural yield is presented in Fig. 2. The reaction was conducted in the time range of 5 - 90 min with 1 % sulphuric acid. Initially, corncob, as a hemicellulose source, was hydrolysed into xylose which belongs to pentose, and then further dehydrated to generate furfural. In the microwave heating method, furfural yield reached its highest value, 2.26 % at a 5-min reaction time. The high yield of furfural was achieved within a short time, which indicates that microwave heating may provide uniform heating for chemical reactions and accelerate the reaction rate [35]. Compared to the conventional reaction system, the formation rate of furfural is slower than that of the microwave system. The highest yield of furfural in the conventional system was reached at 45 min (2.21 % yield). The result showed that the reaction time to produce the highest furfural using the microwave heating method was 9-fold faster than the conventional heating method. It was also found that for both reaction systems, after the highest point, the yield of furfural tended to decrease due to further degradation product in prolonged reaction time [36].

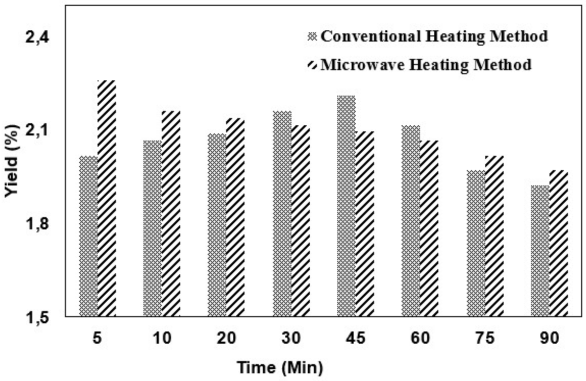

Fig. 2. Effect of reaction time on furfural yield at 100^{o}C with 1 % H_2SO_4 under microwave and conventional heating methods.

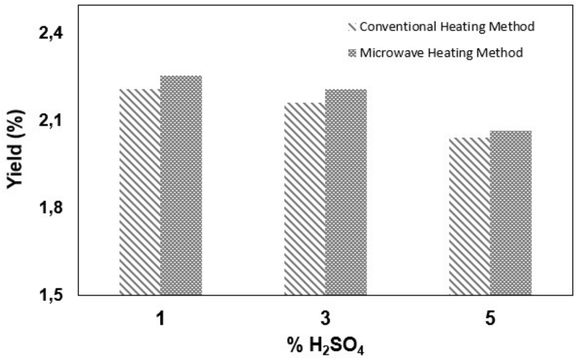

Fig. 3. Effect of acid catalyst concentration on furfural yield at 100^{o}C for 5 min-reaction time for the microwave heating method and 45 min-reaction time for the conventional heating method.

Furthermore, a sequential reaction was performed to investigate the effect of sulphuric acid concentration on furfural production. The sulphuric concentration was varied to 1 %, 2 %, and 3 %-w/v with LSR 10:1 under 100^{o}C. The reaction was carried out at the optimum reaction time of 5 min and 45 min for the microwave system and conventional system, respectively. The addition of sulphuric acid to the reaction system promotes furfural formation. The acid condition of the solution induces the release of polysaccharides from the cell wall of corncob; thus, both hydrolysis and dehydration processes occur in the reaction system to generate furfural [17, 37]. As a control, a catalyst-free reaction system was also tested. Only a trace amount of furfural was detected in the hydrolysate. It proves that the presence of an acid catalyst is required in furfural production from biomass.

The result shows the highest yield is achieved at 1 % w/v of catalyst for both systems, then significantly decrease at 2 % and 3 % w/v of catalyst (Fig. 3). It indicates a high concentration of sulphuric acid as catalysts in the reaction system has a negative effect on furfural yield. Further addition of sulphuric acid might cause the formation of degradation products such as formic acid [38]. The lower pH value also promotes furfural polymerization in the reaction system, resulting in the other by-products [36, 38].

The kinetic of furfural production

Fig. 4 shows the comparison between the predicted value by the proposed model (solid lines) and experimental data (points). The value of the proposed model fitted well with the experimental data for both heating method of microwave and conventional ($R^2 \geq 0.99$) as presented in Table 2. The k_1 values were significantly higher than those of k_2, indicating that furfural production was the dominant reaction throughout the entire transformation process. The k_1/k_2 ratios were utilized to compare the values of k_1 and k_2, as well as to assess the suitability of the reaction conditions [39]. Higher value of $[HE]_0$ for the microwave method compared to those for conventional method indicated that higher furfural obtained for microwave heating method. This finding was also supported by the value of k_1 for microwave method is much higher (three-fold) than those for the conventional one. The value of k_1 describes the rate of the kinetics process of hemicellulose decomposition to produce furfural; therefore, the reaction process using microwaves is more efficient than the conventional heating method. However, a smaller value of k_2 for the microwave heating method was observed than those for the conventional one indicating that the rate of decrease in furfural production with respect to time (after optimum production) is greater in the microwave process than in the conventional heating method. It was due to further degradation product in prolonged reaction time.

The solid characterization before and after reaction

A qualitative observation of the structural morphologies of untreated and treated materials (conventional and microwave method) is exhibited in Fig. 4a-c. The raw corncob has a dense and smooth surface (Fig. 5a), while both treated samples have a looser, random, and rough surface. The irregular shape

Table 2. Kinetic rate constants for the catalyst hydrolysis of corn cobs.

Heating method	$[HE]_{0,}$ g L^{-1}	$k_{1,}$ min^{-1}	$k_{2,}$ min^{-1}	R^2
Conventional	0.8749	0.5038	0.0011	0.9901
Microwave	0.8919	1.8560	0.0015	0.9983

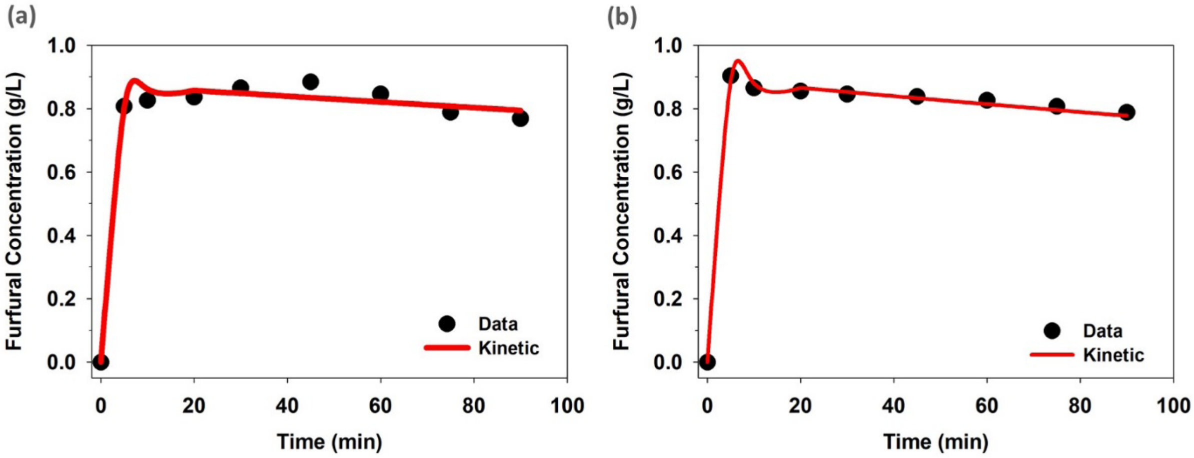

Fig. 4. Kinetic model for conventional heating method (a) microwave heating method (b).

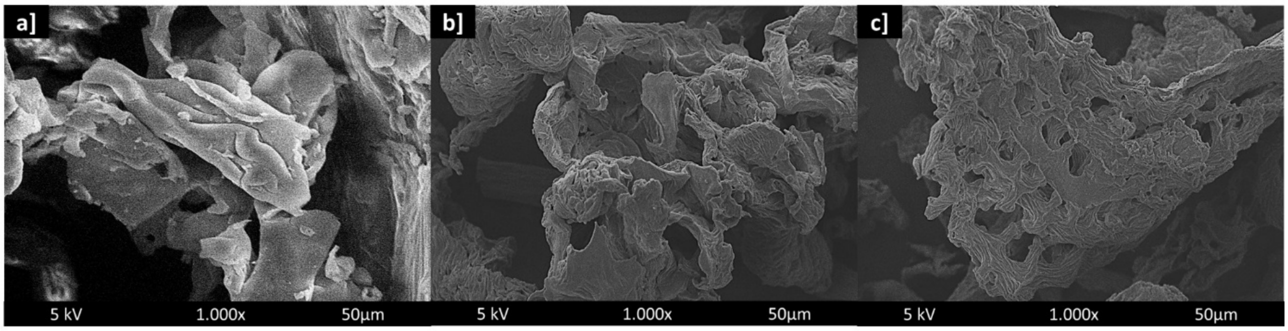

Fig. 5. SEM images of untreated and treated corncob for surface morphology: (a) raw corncob; (b) treated corncob after conventional hydrolysis at 100°C for 45 min with 1 % H_2SO_4; (c) treated corncob after microwave-assisted hydrolysis at 100°C for 5 min with 1 % H_2SO_4.

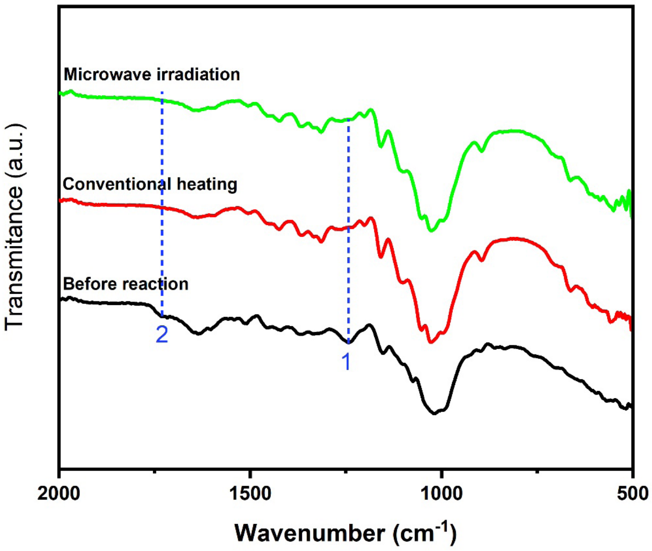

Fig. 6. FT-IR spectra of the raw corncob, conventionally treated corncob (100°C, 45 min with 1 % H_2SO_4), and microwave-treated corncob (100°C, 5 min with 1 % H_2SO_4).

indicated the release of hemicellulose and lignin during the hydrolysis reaction [35]. The microwave-treated corncob showed a more damaged surface due to more cellulose, hemicellulose, and lignin release from the cell wall of the corncob (Fig. 5c). This condition could be verified by more furfural in the hydrolysate.

FT-IR analysis was used to verify the structural change in untreated and treated corncob (Fig. 6). For the untreated corncob, the band around 1735 cm^{-1} is assigned to the stretching vibration of the C=O bonds of the acetyl group in hemicellulose [40]. The band at 1253 cm^{-1} is attributed to the deformation vibration of C-H in polysaccharose.

The decreased intensity of the bands at 1735 cm^{-1} and 1253 cm^{-1} indicates the structural change of hemicellulose during the hydrolysis process, both in the conventional method and the microwave-assisted method. The cleavage of acetyl groups from hemicellulose causes a decrease in the intensity of the band at 1735 cm^{-1}. While the decreased intensity of the band at 1253 cm^{-1} exhibits the release of hemicellulose from the cell wall of the corncob.

CONCLUSIONS

Microwave-assisted acid hydrolysis has been successfully employed to produce furfural from corncobs. The highest furfural yield (2.26 %) was produced at a 5-min reaction time of 100°C with 1 % H_2SO_4. Microwave irradiation is more effective than conventional heating methods in the enhancement of furfural yield at short reaction times. The results demonstrate that the reaction time required to achieve the highest furfural production in the microwave-assisted reaction is 9-fold faster than that of the conventional heating method. In the present research, microwave-assisted acid hydrolysis demonstrated a promising less reaction time and less energy-intensive process for carrying out the furfural production process from biomass compared to a commonly employed process.

Authors' contributions

R.J., P.M., M.D.P.: conceptualization; H.W., R.J., I.F.N.: methodology; M.D.P., P.M.: validation; R., M., P.M.: formal analysis; R.J., M.D.P., P.M. : investigation; R.J., P.M., M.D.P.: writing-original draft preparation; P.M., I.F.N., H.W.: writing-review and editing; P.M., M.D.P. s: supervision. All authors have read and agreed to the published version of the manuscript.

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