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Utilization of Bamboo Waste by Engineering Acid Hydrolysis (H_2SO_4) to Produce Furfural Compounds

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ABSTRACT

Bamboo waste containing lignocellulosic can be used to form furfural compounds. Furfural is an intermediate product widely needed by the chemical industry to manufacture finished products such as resins, disinfectants, lubricating oils, synthetic rubber, etc. This product can be produced from materials containing pentosan. This study aimed to determine the effect of the delignification process on pentosan levels, yield and characterization of the resulting furfural, and the potential of pentosan to become furfural compounds. In this study, the formation of furfural from bamboo waste was carried out using the acid hydrolysis method, with the independent variables namely cooking temperatures of 90 °C, 100 °C and 110 °C, H_2SO_4 concentrations of 5%, 10%, and 15% and cooking times of 60 minutes, 90 minutes, and 120 minutes. Based on the results of research that has been done, it is known that the delignification process influences the pentosan level, where the sample without delignification obtained a pentosan level of 11.10%, and using the delignification process obtained a pentosan level of 14.67%. Characterization of furfural analysis results by color test showed a change in color to red and based on the results of GC-MS analysis at retention time 24.

1. Introduction

Indonesia is a country that has biodiversity; plants grow in almost every region of Indonesia, one of which is bamboo. The potential area of bamboo forests in Indonesia can reach 2 million ha with a yield of more than 3.8 tons ha-1 per year. Besides that, bamboo can only grow in 3-5 years and can be harvested. Bamboo is used daily as furniture and various crafts [1]. Bamboo has a reasonably high cellulose content, which is around 47.50%, hemicellulose 15.35%, and lignin 26.25% [2]. Other ingredients such as silica content 0.10-1.78%, ash content 1.24-3.77% [3]. In the utilization of bamboo, the yield of production used for furniture and crafts to become products is around 60%, while 40% becomes waste [1]. Only a few people know that several valuable compound components in bamboo can be utilized to increase the economic value of wasted bamboo waste.

Bamboo waste containing lignocellulose can be used to form furfural compounds. Furfural is a chemical compound with many uses, namely as a solvent in separating saturated and unsaturated compounds in the petroleum industry and as an intermediate compound in the manufacture of other industrial chemicals. Furfural has a broad function, especially to synthesize its derivative compounds. Furfural is widely used as a solvent in manufacturing lubricants, in the petroleum processing industry, shoe dyes, and raw materials for insecticides, herbicides, and fungicides [4]. Therefore, the country's need for furfural and its derivatives continues to increase, so the domestic need for furfural is obtained through imports. Furfural imports in Indonesia continue to increase.

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According to [5], the need for furfural in Indonesia in 2017 reached 805.626 kg/year. Hence, the utilization of bamboo waste biomass, which contains a lot of hemicellulose, cellulose, and lignin, into furfural is a solution to the problem of bamboo waste and meeting the needs of furfural.

Many studies have been carried out on biomass containing lignin, cellulose, and hemicellulose. Still, only a few have extracted it into furfural compounds, and many studies have only separated lignin from lignocellulosic, as was done by [6], extracting biomass to obtain lignin in a fast way of extraction pretreatment using *Ultrasonic-Microwave Assisted Extraction* (UMAE) [6] and delignification process with NaOH. Therefore, researchers will apply this research to the NaOH delignification process. The Furfural compound can be obtained by hydrolyzing pentosan contained in fruit and agricultural waste such as corn cobs and rice husks using acid and heating [7]. Based on the description above, this study aims to obtain furfural compounds from bamboo waste by hydrolysis reaction using an acid catalyst, namely sulfuric acid (H_2SO_4).

2. Research Methodology

2.1. Materials

The tools used in this study were an analytical balance (Shimadzu), distillation, evaporator, biuret and flask, filter paper, oven, furnace, thermometer, porcelain cup, hotplate, vacuum filter, measuring pipette, volume pipette, beaker glass (Pyrex), Erlenmeyer (Pyrex), desiccator, volumetric flask, measuring cup, GC-MS (Acquisition). The materials used are bamboo waste (Mayan), sulfuric acid (H_2SO_4) (Merck), sodium hydroxide (NaOH), chloroform (Merck), distilled water, chloride acid (HCl) (Merck), starch, phloroglucinol, potassium iodide (KI) (Merck), KIO_3, sodium thiosulphate (Merck).

2.2. Procedures

In this study, the software used was Minitab version 2017, a statistical method used to determine the optimum formulation [6], [8]. The independent variables in this study were three factors, namely the cooking temperature of 90, 100, and 110 °C, respectively, the concentration of sulfuric acid 5, 10, and 15%, the variations in the cooking time of 60, 90, and 120 minutes, and the response was furfural yield value. The experimental design using RSM can be seen in Table 1.

Table 1. Experimental design using the response surface method (RSM)

Run Orders	Temperature (°C)	H_2SO_4 Concentration (%)	Time (Minute)
1	90	5	90
2	110	5	90
3	90	15	90
4	110	15	90
5	90	10	60
6	110	10	60
7	90	10	120
8	110	10	120
9	100	5	60
10	100	15	60
11	100	5	120
12	100	15	120
13	100	10	90
14	100	10	90
15	100	10	90

The raw material used is bamboo waste, which is used as a source of pentosan in the formation of furfural at the hydrolysis stage. The raw material used in this study is bamboo waste from bamboo craftsmen assisted by PT. Bukit Asam Tbk. Before use, bamboo waste is dried, then crushed, and sieved with a pass size of 80 mesh [6], [9]. Then, a delignification process is carried out; this delignification process refers to research carried out by [6]; however, there will be changes regarding the ratio of NaOH used in this study. The bamboo powder sample was added with 1% NaOH (1:30) and then heated at 105 °C for 120 minutes. Filtered and taken the solids, then washed using distilled water until they reached a pH of 7. Washed samples were dried in an oven at 45 °C for 18 hours. After the delignification stage, then it enters the hydrolysis stage. The hydrolysis process was carried out as much as 10 grams of sample was put into a 500 ml round flask and added 250 ml of H_2SO_4 with variations of 5, 10, and 15%, respectively, then stirred until evenly distributed and refluxed with variations of cooking time of 60 minutes, 90 minutes and 120 minutes, with cooking temperatures of 90, 100, and 110 °C, respectively. The reaction product was allowed to cool to room temperature and filtered using filter paper. The filtrate obtained was added with 100 ml of chloroform so that furfural chloroform and sulfuric acid would separate. Separation was done using a separatory funnel; the upper layer was sulfuric acid and water, and the chloroform-furfural layer was at the bottom. The chloroform and furfural layers were separated by an evaporator at 60-70 °C.

2.3. Analysis Lignocellulose Bamboo

Analysis was performed using the Chesson-Datta method to determine the concentrations of cellulose, lignin, and cellulose. A dry sample weighing one gram (weight a) was cooked for one hour at 100 °C when combined with 150 ml of distilled water. After filtering, the mixture is then rinsed with hot distilled water. The solid was dried at 105 °C in an oven (weight b) for one hour. The solid was then combined with 150 ml of 1 N H_2SO_4 and heated for an hour at 100 °C. With distilled water, the solid is filtered and cleaned. The solid was then dried for one hour (at a weight of c) at 105 °C. At room temperature, 10 ml (72% H_2SO_4) of the dry substance was added to it for 4 hours. The mixture was then given 150 ml of 1 N H_2SO_4 and refluxed for an additional hour. The solid was then baked in the oven for 1 hour at 105 °C after being rinsed with 400 cc of distilled water. Heating continues until the weight (weight d) is constant. The solid was then heated for 1 hour at 550 °C and weighed (weight e).

$$\%\ Hemicellulose = \frac{(b-c)}{a} x\ 100\% \tag{1}$$

$$\%\ Cellulose = \frac{(c-d)}{a} x\ 100\% \tag{2}$$

$$\%\ Lignin = \frac{(d-e)}{a} x\ 100\% \tag{3}$$

2.4. Analysis Pentosan Content

Analysis was done to identify the amounts of cellulose, lignin, and cellulose. Chesson-Datta technique [6]. A dry sample weighing one gram (weight a) was combined with 150 ml of distilled water and heated at 100 °C for one hour. The mixture is then filtered and cleaned with hot, distilled water. The solid was dried for 1 hour at 105 °C in an oven (weight b). The material was then combined with 150 ml of H_2SO_4 (1 N) and heated at 100 °C for 1 hour. Filtered water is used to cleanse the solid after filtering. After that, the solid was dried for 1 hour (weight c) at 105 °C. The dried substance was left to soak at room temperature for four hours in 10 ml of 72% H_2SO_4. The mixture was then given 150 ml of H_2SO_4 (1 N), which was added and refluxed for an hour. Following a 400 ml wash with distilled water, the solid was cooked in the oven for an hour at 105 °C. Heat is applied until the weight (weight d) becomes constant. The solid was heated for an additional hour at 550 °C before being weighed (weight e).

2.5. Analysis Furfural Content

The volumetric approach can be used to determine the content of furfural. An Erlenmeyer was filled with 5 ml of hydrolysate and 5 ml of distilled water, and the mixture was agitated until homogenous. The solution was combined with 5 ml of 0.1 N KIO_3 solution and 5 ml of 4 N sulfuric acid and was then agitated for 5 minutes. Potassium iodide (KI) (10 ml, 0.05 M) was added. Titrated using sodium thiosulfate 0.1 N Starch standard is the indicator in use. The same approach was used to create a blank without adding samples as a comparison.

3. Results and Discussion

3.1. Effect of delignification on Bamboo Waste

Bamboo powder was delignified using a 1% NaOH solution (1:30) and heated at 105 °C. The heating process allows more cellulose to be degraded because the lignin has been completely dissolved so that the remaining delignification will degrade cellulose, while at room temperature, the lignin has not decomposed and still protects cellulose and hemicellulose so that cellulose and hemicellulose are still difficult to access [6]. In this delignification process, using an alkaline solution is preferable to using an acid solution because, when compared to using an acid solution, an alkaline solution has better results for the delignification process. Based on the result conducted by [10], it has been reported that using NaOH is proven effective for various biomass feedstocks. A previous study conducted by [6] has performed the delignification of citronella with NaOH solution and can separate the content of lignin, cellulose, and hemicellulose.

1) Influence on Lignocellulosic Content

Based on the results of a comparative analysis of the lignocellulose content of the samples with delignification and without delignification treatment are shown in Table 2.

Table 2. Lignocellulose content without delignification and delignification process

Sample	Lignin	Celulose (%)	Hemicellulose
No Delignification	20.36	40.86	18.74
Delignification	12.82	58.89	16.56

a) Lignin

Table 2 shows a decrease in lignin content; the lignin content without delignification was 20.36% and decreased after the delignification process was carried out to 12.82%. The effect of alkali on lignin removal could be due to the ability of the ether bond between cellulose and the lignin complex [11], [12]. The released lignin will bind to the alkali to form a lignin-alkali complex. Adding an alkali or base would cause a high concentration of hydroxyl ions in the soaking solution, transferring hydrogen ions from the lignin hydroxyl group to the basic hydroxyl group [6]. The delignified bamboo powder was then washed to remove the remaining soaking solution. In addition, washing the residue aims to remove the lignin still contained in the bamboo powder. During soaking, there may still be much lignin that does not dissolve in the soaking solution and sticks back to the bamboo powder after washing.

b) Cellulose

Table 2 shows an increase in cellulose content; the cellulose content without delignification was 40.86%, and it increased after the delignification process was carried out to 58.89%. The increase in cellulose levels occurred due to decreased levels of lignin and hemicellulose in the samples due to the delignification process. The bond between cellulose, hemicellulose, and lignin can be broken using NaOH. Besides that, delignification can also break the bonds in each component, such as hydrogen and covalent bonds [11]. This can be seen from changes in the levels of these compounds, such as that the percentage of cellulose increased while that of hemicellulose and lignin decreased.

c) Hemicellulose

Table 2 shows a decrease in the hemicellulose content, where the hemicellulose content of the samples without delignification was 18.74% and decreased after the delignification process was carried out to 16.56%. Delignification with NaOH will reduce hemicellulose levels. The decrease in hemicellulose content shows that the delignification treatment carried out in this study caused lignin degradation and depolymerization of hemicellulose [1], [13]. This is because hemicellulose is a building block of lignocellulosic cells but has a lower degree of polymerization than cellulose. Hemicellulose is very susceptible to high temperatures, acids, and bases compared to cellulose [14].

The NaOH molecule will dissolve the hemicellulose in the bamboo powder and break the hydrogen bonds, especially the intermolecular bonds of cellulose, so the cellulose is unbound. When hemicellulose fiber is reacted with NaOH at high temperatures, it can be oxidized into simple units into pentosan sugars such as D-xylose and L-arabinose and hexose sugars such as D-glucose, D-galactose, and D-mannose, which are easily soluble in water [15].

2) Influence on Pentose Levels

Based on the results of a comparative analysis of pentosan levels in samples with delignification and without delignification treatment it was shown in Table 3.

Table 3. Pentose levels without delignification and the delignification process

Sample	Pentosan Weight (g)	Pentosan Level (%)
No Delignification	0.5549	11.10
Delignification	0.7334	14.67

Pentosan is a major component of hemicellulose. Table 3 shows differences in pentosan levels without delignification and with delignification. The pentosan content without delignification was 11.10%, while the pentosan content with delignification was 14.67%. The pentosan content of delignified bamboo powder is higher due to decreased lignin content. In addition, when hemicellulose fiber is reacted with NaOH at high temperatures, it can be oxidized into simple units, pentosan sugar groups such as D-xylose and L-arabinose, and hexose sugars such as D-glucose, D-galactose, and D-mannose, which are easily soluble in water [15].

3.2. Furfural Analysis with Color Test

The results of refluxing bamboo powder using sulfuric acid were then identified by a qualitative test using aniline-acetate reagent with a ratio (1:1). Theoretically, furfural is determined by a color change from yellow to red. The color change of the identification results is shown in the Fig. 1.

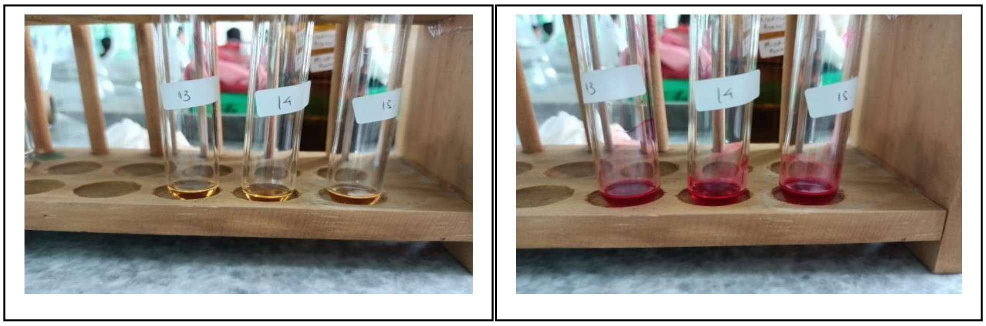

Fig. 1. Results of furfural compounds by color test, (a) before dropping aniline acetate and (b) after dropping aniline acetate

Based on Fig. 1, the results of identifying the furfural color test from the hydrolysis of bamboo powder showed that the color of the test results was the same as the theoretical color of the furfural test, namely furfural, which was initially yellow. Then, after adding aniline acetate, it turned red. This indicates the yield tested was furfural. The red color is caused by a condensation reaction between furfural and aniline to form the compound dialdehyde hydroxyglutaconic dienyl. It is this compound that forms the red color in the reaction. The formation of the dialdehyde-hydroxyglutaconic compound takes place in two stages. The first stage is a reductive amination reaction where the N electrons in aniline attack the carbonyl group to form imine compounds with the release of water, then react with the second aniline, resulting in the breakdown of the furfural ring and the formation of dialdehyde [16]. The following is the aniline-acetate reaction to furfural, as shown in Fig. 2.

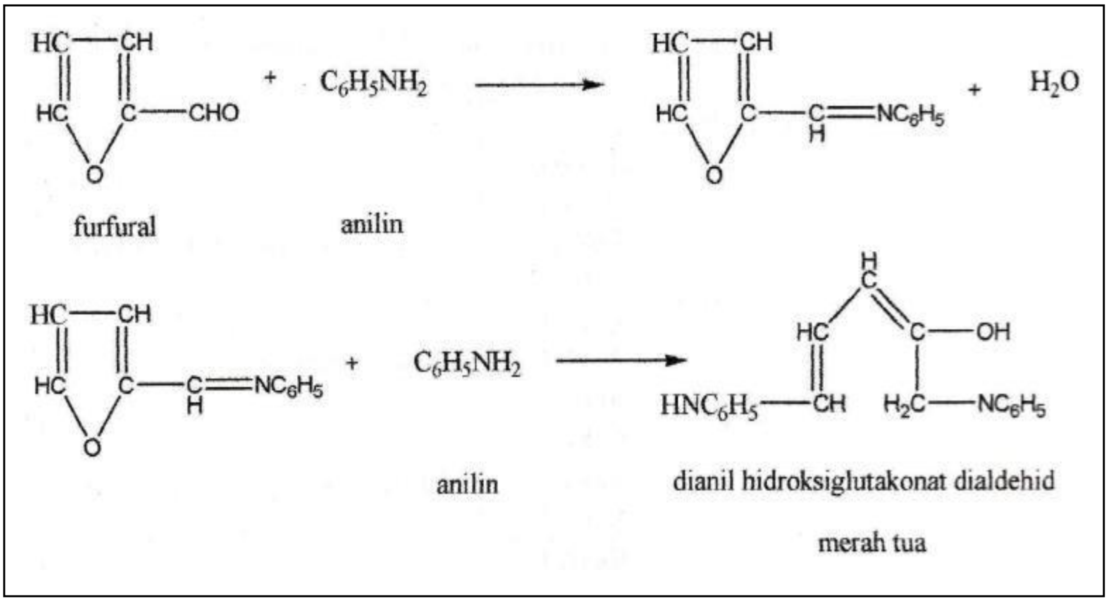

Fig. 2. Reaction of aniline acetate to furfural samples

3.3. Furfural Yield Analysis

The following is the furfural yield obtained, as shown in the Fig. 3.

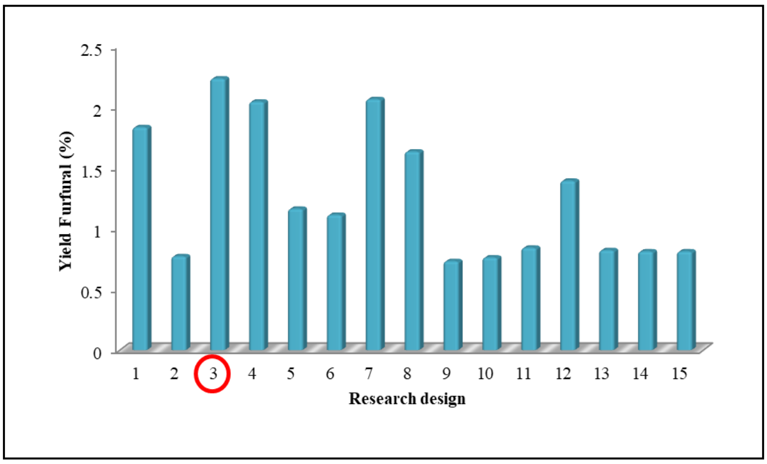

Fig. 3. Yield furfural from bamboo waste with delignification of NaOH

Furfural yield yield data were optimized using RSM to obtain optimum temperature, H_2SO_4 concentration, and time variables. The results of optimizing the formation of furfural compounds can be seen in Fig. 4.

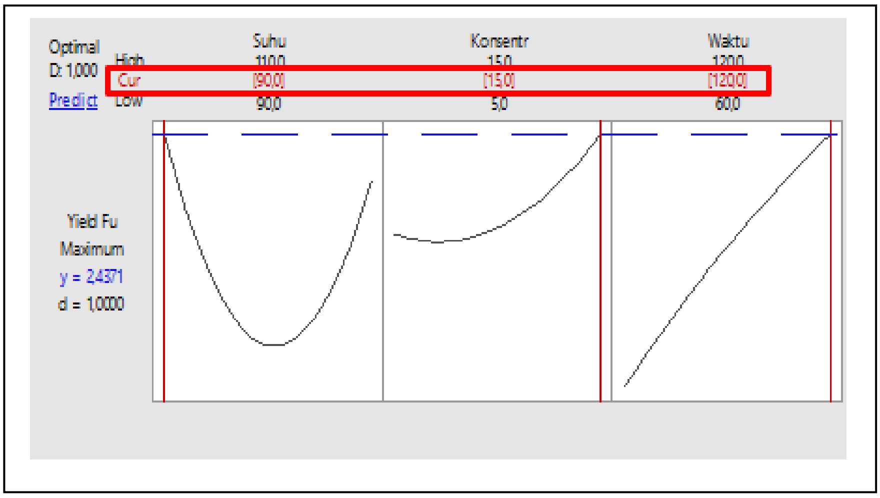

Fig. 4. Furfural production optimization of RSM

Fig. 4 shows that the optimum condition is at 90 ^{o}C for 120 minutes with an H_2SO_4 concentration of 15%. Under these optimum conditions, the resulting furfural yield is estimated to be 2.44%. Based on the study's results, the furfural yield value was influenced by temperature, time, and H_2SO_4 concentration. In Fig. 4, the furfural yield tends to increase with increasing catalyst concentrations of H_2SO_4 and reaches its highest value at a 15% H_2SO_4 concentration, equal to 2.23%. According to [16], This occurs because bamboo waste will hydrolyze at a higher rate at higher concentrations; consequently, the higher the catalyst concentration, the higher the yield of furfural that can be produced. This is due to an increasing number of H^+ ions in H_2SO_4, resulting in better breaking of the pentosan bond, which causes the reaction rate to increase [17]. Increasing the reaction speed will lower the activation energy, so the furfural obtained will also be higher. However, if the concentration of H_2SO_4 has reached the optimum concentration, the furfural yield will decrease. This is because furfural will undergo the breakdown of the aldehyde group so that it will produce furoic acid [16].

A linear regression analysis was performed to prove that the concentration of H_2SO_4, temperature, and time influence the furfural yield—analysis and data processing using Minitab software version 17 with the model obtained as a full quadratic model. The regression analysis of furfural yield on H_2SO_4 concentration, temperature, and hydrolysis time can be seen in Table 4.

Table 4. ANOVA Results Based on Furfural Yield

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	3.96547	0.44061	7.85	0.0180
linear	3	1.59012	0.53004	9.45	0.017
Temperature	1	0.37411	0.37411	6.67	0.049
H_2SO_4 concentration	1	0.63281	0.63281	11.28	0.020
Time	1	0.58320	0.58320	10.40	0.023
Square	3	2.08242	0.69414	12.37	0.009
Temperature*Temperature	1	1.97888	1.97888	35.28	0.002
Concentration H_2SO_4* Concentration H_2SO_4	1	0.10934	0.10934	1.95	0.222
Time*Time	1	0.01134	0.01134	0.20	0.672
2-Way Interactions	3	0.29293	0.09764	1.74	0.274
Temperature*H_2SO_4 concentration	1	0.18923	0.18923	3.37	0.126
Temperature*Time	1	0.03610	0.03610	0.64	0.459
H_2SO_4*Time concentration	1	0.06760	0.06760	1.21	0.322
Error	5	0.28049	0.05610		
Lack-of-Fit	3	0.28042	0.09347	2804.25	0.001
Pure Error	2	0.00007	0.00003		
Total	14	4.24596			

Table 4 shows that the concentration of H_2SO_4, temperature, and time can affect the furfural yield. According to [6], If the P value is <0.05, the treatment given has a significant regression coefficient to the model. In contrast, if the P value is >0.05, it indicates that the treatment given is not significant. The probability value (p-value) can be interpreted as the magnitude of the error value obtained by the researcher. While the value of α is the maximum error that has been determined, namely $\alpha = 0.05$, Table 4 shows that the p-value at temperature is 0.049, H_2SO_4 concentration is 0.020, and time is 0.023 ($P < 0.05$), which means that temperature, H_2SO_4 concentration, and time have a significant effect on furfural yield.

Lack of Fit analysis produces a p-value of 0.001; this test is a test to determine the suitability of the model being carried out and whether there is a match between the response data and the model. The area of rejection is when the p-value > α with a significant degree of $\alpha = 0.05$ because the p-value of 0.001 lack of fit < α means a discrepancy between the response data and the model. Based on the statistical summary model, the R^2 value is 0.9339. The R^2 model of 0.9339 indicates that 93.39% of the total data is appropriate or close to correct.

3.4. Furfural Content Analysis with GC-MS

The molecules being investigated in this study were identified as furfural compounds using GC-MS analysis, and this identification was confirmed. The GC-MS are displayed in Fig. 5.

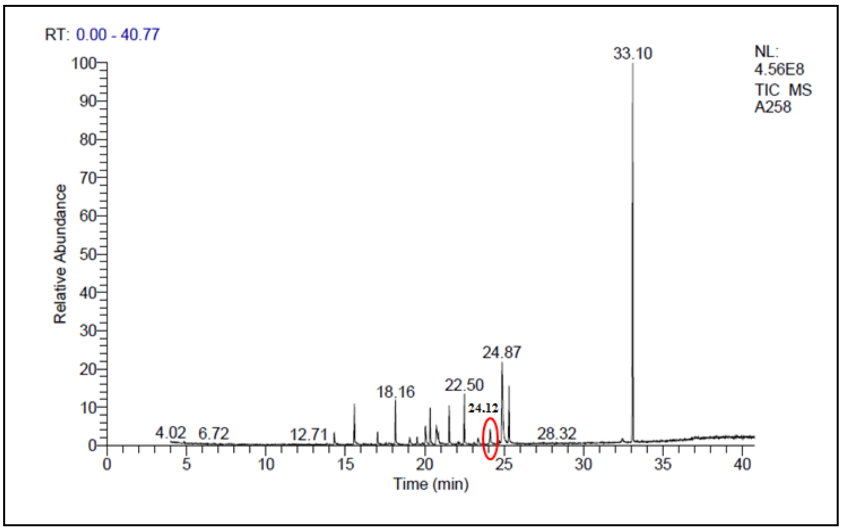

Fig. 5.GC-MS Test Results at the Highest Yield

GC-MS analysis aims to confirm that the hydrolyzed compound is a furfural compound. The GC-MS analysis of samples under 90 °C for 90 minutes with a 15% H_2SO_4 concentration revealed several compound fragments. The furfural compound resulting from the hydrolysis of bamboo powder with a sulfuric acid catalyst was shown at peak with a retention time of 24.12 with the identified compound, namely furane-3-carboxylic acid, which indicates the furfural group. Furane-3-carboxylic acid is a compound derived from furfural. Furfural is thought to be formed due to the influence of the high catalyst concentration and reaction time. This is due to the decomposition of furfural into furoic acid because of breaking down the aldehyde group and forming a black resin. Besides that, furfural can be oxidized to produce furoic acid and form a kind of black resin [16]. Several furfural derivative compounds are shown in Table 5. The highest peak was at a retention time of 33.10 with the identified compound, dioctyl phthalate. Dioctyl phthalate is a compound widely used as an auxiliary material in plastic industries, such as the cable industry, imitation leather, and shoe soles. Doctyl phthalate is also used for various resins and elastomers [18].

Table 5. GC-MS Analysis Results for Furfural Content in the Sample

Retention Time	**Compound Name**	***Areas (%)***	**Compound Potential**
14.31	*Vanillin*	1.33	Vanillin is a compound that can be used as a synthetic vanilla flavor, an ingredient for pharmaceutical compounds, and an antioxidant for cosmetics.
15.57	*Homovanillyl alcohol*	4.57	It can be used as a permitted food additive, namely as milk products and their analogs, ready-to-eat snacks except for infant formula, follow-on formula, growth formula, and food for infants and children in their infancy.
17.03	*2-Propanone, 1-(4-hydroxy-3-ethoxyphenyl)*	1.33	An active compound that can be used as a biomedicine, antiseptic, antioxidant, and anti-inflammatory drug.
18.16	*Apocynin*	4.72	*Apocynin*it can be used as a NADPH oxidase inhibitor without interfering with other aspects of the immune system.
18.16	*Vanilic acid hydrazide*	4.72	This compound can generally be used as a flavoring agent, which is said to have activity as an antioxidant.
19.06	*Mandelic acid, 4-hydroxy-3-methoxy*	0.93	This compound can be used in the beauty industry; mendalic acid is one AHA class, which can exfoliate the skin.
19.52	*Benzaldehyde, 4-hydroxy-3,5-dimethoxy*	0.85	A compound found in liquid smoke, liquid smoke can be used for chemical synthesis, food preservatives, coagulants in the rubber industry, antibacterial antioxidants, anti-termites, and others.
20.04	*1-Propanone, 3-hydroxy-1-(4-hydroxy-3-methoxyphenyl)*	2.32	An active compound that can be used as a biomedicine, antiseptic, antioxidant, and anti-inflammatory drug.
20.35	*3,5-Dimethoxy-4-hydroxyphenyl acetic acid*	3.82	A compound found in liquid smoke, liquid smoke can be used for chemical synthesis, food preservatives, coagulants in the rubber industry, antibacterial antioxidants, anti-termites, and others.
20.73	*2-Propanone, 1-hydroxy-3-(4-hydroxy-3-methoxyphenyl)*	3.07	An active compound that can be used as a biomedicine, antiseptic, antioxidant, and anti-inflammatory drug.
21.54	*Mandelic acid, 3,4-dimethoxy, methyl ester*	4.14	This compound can be used in the beauty industry; mendalic acid is one of the AHA groups that can exfoliate the skin.
10:50	*Aspidinol*	5.65	A compound that is used as an herbal medicine that can overcome tapeworms, anti-virus,

4. Conclusion

Based on the results of the research that has been carried out, it can be concluded that the delignification process influences the pentosan level; that is, samples without delignification obtained a pentosan level of 11.10%, and samples using the delignification process obtained a pentosan level of 14.67%. Furfural characterization based on the results of the analysis with a color test showed a change in color to red, and based on the results of the GC-MS analysis on the retention time (24.12 minutes), the compound identified was furane-3-carboxylic acid, which is a derivative of furfural. The resulting furfural yield was 2.23% under operating conditions at room temperature (90 ^oC) for 90 minutes with a 15% H_2SO_4 concentration. As much as 14.67% pentosan can produce 2.23% furfural compounds.

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