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# Utilization of mensiang (Actinoscorpus grosus L.) as a cellulose-rich material for furfural synthesis

## Peluang pemanfaatan mensiang (Actinoscirpus grosus L.) yang kaya selulosa sebagai bahan baku furfural

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#### **ABSTRACT**

**ABSTRAK** 

The cellulose content of mensiang varies from 22% in the flower to 33% in the root. This could be used as a starting point for furfural synthesis. To separate furfural, several conditions are set for the distillation and evaporation processes. The conditions were rootstock part (A), upper stem part (B), whole stem part (C), whole stem with flowers part (D), and whole stem, flowers, and root part (E). Furfural content in the mensiang plant was conducted using Gas Chromatography-Mass Spectroscopy (GCMS) for the highest yield 11.78%, D treatment. Furfural synthesized from the mensiang plant has a furfural content of 96%. Furfural's density was 1.160 g/ml, boiling point was 161 °C, refractive index was 1.5120 °brix, and its viscosity was 1.46 cp.

#### mensiang

#### Kata kunci:

selulosa; furfural; mensiang Kandungan selulosa mensiang bervariasi dari 22% pada bagian bunga tanaman hingga 33% pada bagian akar. Berdasarkan hal tersebut mensiang dapat digunakan sebagai bahan baku untuk sintesis furfural. Untuk memisahkan furfural, tanaman mensiang dipisahkan kedalam beberapa perlakuan untuk proses distilasi dan evaporasi. Perlakuan tersebut adalah bagian batang bawah (A), bagian batang atas (B), bagian batang utuh (C), bagian batang utuh dengan bagian bunga (D), dan batang utuh, bunga, dan bagian akar (E). Kandungan furfural pada tanaman mensiang diperoleh dengan menggunakan Gas Chromatography - Mass Spectroscopy (GCMS) untuk rendemen tertinggi 11,78%, yaitu pada perlakuan D. Furfural yang disintesis dari tanaman mensiang memiliki kandungan furfural sebesar 96%. Massa jenis furfural adalah 1,160 g/ml, titik didih 161 °C, indeks bias 1,5120 °brix, dan viskositas 1,46 cp.

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#### 1. Introduction

Lignocellulosic biomass is a natural resource that is used for energy production or bioenergy. It is composed of three major elements: cellulose, hemicellulose, and lignin, as well as minor compounds (extractive substances) with low molecular weight, organic compounds, and ash as an inorganic compound (Phitsuwan et al., 2013). The most abundant and least expensive raw material on the planet is lignocellulosic biomass derived from agricultural and forest waste. Conversion of these materials into chemicals and other

industrial products has become a concern in recent decades (Zhang et al., 2018).

Furfural is produced by hydrolyzing lignocellulosic biomass containing pentosan compounds and then dehydrating with inorganic/mineral acid catalysts such as sulfuric acid and hydrochloric acid. It's a clear, colorless or yellow liquid that smells like almonds but quickly turns yellow, brown, and black when exposed to air; and it's commonly referred to as furfuraldehyde (Gebre et al., 2015).

Indonesia is a tropical climate country that has abundant of lignocellulosic material. One of the

lignocellulosic sources that we could easily find was *Cyperaceae*, a type of grass that is a herbaceous, grasslike plant found in wet areas all over the world. It has approximately 4,000 species and 70 genera (Butt et al., 2018). *Actinoscirpus grossus*, L. also known as *mensiang* or wlingi, is one of the many grass species found in Indonesia.

Mensiang grows abundantly in water or swamps, ponds, ditches, rice fields, and at an elevation of 900 meters above sea level. Mensiang is considered a weed in rice fields in Indonesia, where it can be controlled manually by plowing or with herbicides. It is commonly used to make mats or bags with a strong and durable texture that are also inexpensive. Mensiang post-harvest processing is done by taking the stalks, drying them in the sun, and then moving on to the next process for making products

Bagasse, corn cobs, and rice husks are common raw materials used to make furfural and contain pentosan at rates of 40.9% (Zhang et al., 2017), 32% (Pressure, 2014), and 8.9% (Suxia et al., 2012), respectively. Bagasse and corn cobs are the most commonly used raw materials in furfural production, with more than 98% of all furfural production using corn cobs as raw materials supplied by China, the world's largest raw materials producer (Services, 2006). Some researchers, such as (Andaka, 2011), obtained a furfural yield of 5.67% using baggase, while (Coniwanti et al., 2016) obtained a furfural yield of 7.74% using a mixture of bagasse and coconut-shell.

In West Sumatra, common cellulose sources include rice husks (Helmiyati and Suci, 2019), hemp (Yulfa et al., 2019), corn cobs (Winarti et al., 2018), and water hyacinth (Asrofi et al., 2018). With a cellulose content of 36.21 percent (Bidin et al., 2015), Mensiang has the potential to become one of West Sumatra's sources of cellulose plants, one of which was used as a raw material in furfural production.

Mensiang containing 20-21.2% pentosan (Brink and Escobin, 2016) could be used as a raw material for furfural production. Every part of the plant contains different chemical contents, as well as hemicellulose, which is the primary ingredient in the production of furfural. Cellulose, hemicellulose, and lignin are all components of cells, so the higher the hemicellulose content of plants, the closer to growth they are. Based on the description above, this study was conducted to determine the yield and quality of furfural produced by the conversion of mensiang plants.

#### 2. Method

Mensiang (Actinoscirpus grossus, L.) (fig.1) were obtained from Aia Pacah in Padang and divided into lower stem, upper stem, flowers, and roots. The parts are then dried using a solar thermal method, cut into small pieces with a hammer mill, and sieved through an 60 mesh sieve before being placed in a ziplock plastic bag for further analysis. Sulfuric acid obtained from SMART-LAB as a catalyst, chloroform as an organic solvent, sodium chloride, and the materials used for chemical identification and analysis, which include furfural standard, analin, glacial acetic acid, and alcohol

were obtained from MERCK, Germany, and distilled water are the chemicals used in the main process of furfural synthesis.



Figure 1. Washed mensiang

#### 2.1. Preparation of raw material

Mensiang was prepared based on the treatment; rootstock (A), upper stem (B), whole stem (C), whole stem with flower (D), and whole stem, flowers, and root (E). From 128 cm height, Mensiang was separated into four part (flower, upper stem, rootstock, and root) and then prepared for the five treatments above. Mensiang was then dried and sieved through a 60 mesh sieve.

#### 2.2. Furfural synthesis

50~g of *mensiang* powder was prepared and placed in the distillation flask, along with 62.5~g of NaCl and 250~ml of  $12\%~H_2SO_4$ , while Erlenmeyer was filled with 50~ml chloroform. For two hours, the distillation was carried out at  $120^{\circ}C$ . The distillate will be separated into two layers. The top layer is water, and the bottom layer is chloroform mixed with furfural, which is produced during the process. A separator was used to separate the chloroform from the water. The furfural was then separated from the chloroform using a vacuum evaporator set to  $60\text{-}70^{\circ}C$ 

#### 2.3. Characteristic analysis

Raw materials and crude furfural were analyzed in this study. Raw material analysis included moisture content (Official methods of analysis of AOAC international 18th Ed, 2005), extractive substance content (TAPPI-T 280), holocellulose, cellulose (TAPPI-T 429), hemicellulose, and lignin (TAPPI, 2011) and for crude furfural was carried out on processing yield, determination of furfural content using GC-MS (Senila et al., 2013), identification of furfural using aniline acetate, density, refractive index, boiling point and furfural viscosity (Lomba et al., 2013).

#### 3. Result and discussion

#### 3.1. Raw material composition

The raw materials in this study were parts of the mensiang plant (Actinoscirpus grossus) including

flowers, upper stem, rootstock, and roots. Analysis of the raw materials of the *mensiang* plant including extractive

substances, cellulose, hemicellulose, and lignin content could be seen in Table 1.

Table 1. Analysis of *mensiang* plant raw materials

Parameter	Treatments (%)			
Tarameter	Extractive substances	Hemicellulose	Cellulose	Lignin
Root part	$2.189 \pm 0.397$	$27.945 \pm 0.714$	$33.64 \pm 2.319$	$34.28 \pm 4.299$
Rootstock part	$5.305 \pm 1.85$	$26.687 \pm 3.248$	$32.63 \pm 1.357$	$27.053 \pm 1.552$
Upper stem part	$4.792 \pm 0.040$	$23.993 \pm 0.372$	$24.605 \pm 3.542$	$28.669 \pm 3.601$
Flower part	$9.355 \pm 4.348$	$30.237 \pm 4.572$	$22.46 \pm 1.584$	$37.66 \pm 0.707$

The raw material used has been dried and passes through a 60 mesh sieve. Extractive substances were found in 2.189% of the roots, 5.305% of the rootstock, 4.792% of the upper stem, and 9.335% of the flowers. Extractive substances are plant substances other than cellulose, hemicellulose, and lignin. Plants typically 10% extractive substances less than (Lukmandaru, 2011). Plant extractives, in general, dissolve easily in ether, alcohol, or water solvents. Extractive substances in plants can be used for wood identification because the extractive substances differed depending on the type of wood.

For roots, rootstocks, upper stem, and flowers, the cellulose content was 33.64%, 32.63%, 24.605%, and 22.4%, respectively. The cellulose content of the Mensiang plant is 36.21 percent (Bidin et al., 2015), but the results vary because they are from different parts of the plant. Cellulose in plants was useful in the formation of plant cell walls, and it was also widely used in the production of pulp and paper. The rootstock has a higher cellulose content than the upper stem. This is due to the fact that growth begins at the bottom of the stem. Cellulose content was found to be related to the formation of cell walls, which is consistent with plant growth.

The hemicellulose content (Glucuronoxylan) of the dry weight of wood generally ranges from 15-30% (Rowell et al., 2021). In this study, the root hemicellulose yield was 27.945%, the rootstock yield was 26.687%, the upper stem yield was 23.993%, and the flower yield was 30.237%. The presence of hemicellulose in raw materials was critical because pentosan, a component of hemicellulose, was required for the production of furfural. Hemicellulose is made up of various sugar monomers.

The difference in raw material component content was caused by the location of these components in the plant. In plants, cellulose was a component of the cell wall. Cellulose, a glucose homopolymer with no branches, stiffens the plant cell walls. Cellulose, hemicellulose, pectin, and protein form a network that strengthens plant cell walls, making cellulose difficult to isolate (Rowell et al., 2021). Hemicellulose is made up of sugar and modified sugar heteropolymers that are bonded together with cellulose to strengthen the secondary cell wall (Zhang et al., 2015).

According to Brink & Escobin (2016), *mensiang* contained 21.2% pentosan, whereas (Bidin et al., 2015) states that it has 49.88% hemicellulose. Because raw material determination was carried out for parts of the

*mensiang* plant, the results obtained differ from those obtained by previous researchers.

#### 3.2. Furfural yield

The yield of furfural could be seen in Table 2. The highest yield was obtained in treatments C and D with a yield of 11.78%, while the lowest yield was obtained in treatment E with a yield of 5.76%. The results differed from those obtained in previous studies, such as (Wang et al., 2018), which obtained 65.67% furfural yield with corn cob as raw material, and (Andaka, 2011) which obtained 5.67% furfural yield with bagasse as raw material at a reaction time of 120 minutes. (Hambali et al., 2016) obtained a furfural yield of 1.87% from rice husk. Because of differences in the raw materials used, the results obtained varied. According to the research, different parts of the mensiang plant contain different amounts of hemicellulose. The higher the hemicellulose content of the material, the closer it is to the point of growth. According to the results, the rootstock and upper stem produced the highest yield.

Table 2.
Furfural Yield on Different Plant Parts of Mensiang

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Treatments	Yield (%) ± Standard Deviation
A	$9.97 \pm 0.40$
В	$8.75 \pm 0.31$
C	$11.78 \pm 0.92$
D	$11.78 \pm 0.35$
E	$5.76 \pm 0.70$

However, due to the saponification process that occurs during the distillation process, treatment E had the lowest yield of 5.76%. Because each part of the plant contains hemicellulose, the combination of parts of the *mensiang* plant was expected to yield the highest yield. Saponification was a reaction between fats (fatty acids or glycerol) and alkaline substances The root part of the plant was used in treatment E, where the root part of the plant contained a tuber. Triterpenoids are found in the tubers of *mensiang* plants, where triterpenoids were constituents of saponin compounds (Triterpenoid saponins) (Zhao and Li, 2018), which are thought to cause the saponification process during the distillation process (Ganapathi et al., 2017).

#### 3.3. Furfural content

The furfural content in the sample was determined using Gas Chromatograph - Mass Spectrophotoscopy (GC-MS). Fig. 2 showed the results of the furfural content. According to the chromatogram of the crude furfural sample, 7 peaks were read in the data analysis,

with retention times of 1.143; 1.190; 1.303; 1.585; 3.193; 5.235; and 5.981. The furfural compound was read at a retention time of 3.193 seconds and a concentration of 96.23%. The result for the furfural content in sample was read around the same peak as the standar furfural for the retention time 3.114 seconds and concentration 92.10%.

Table 3. Chemical content of furfural samples

Peak	Retention time (min)	Concentration (%)	Molecular weight (Mol)	Compound name
1	1.143	0.41	46	Ethanol
2	1.190	0.17	46	Formic Acid
3	1.303	0.26	60	Acetic Acid
4	1.585	0.36	179	Pyrolidine,1-(6-Ethil-1-Cyclohexan-1-Yl)
5	3.193	96.23	96	Furfural
6	5.235	0.61	98	2(5H)-Furanone,5-Methyl
7	5.981	1.91	110	2-Furanocarboxaldehyde

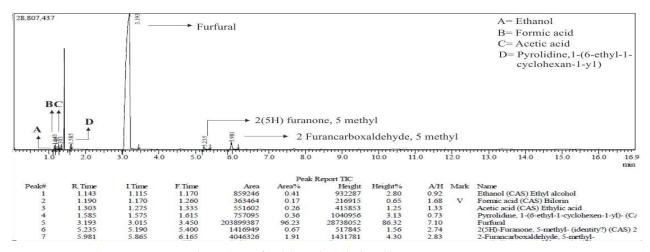


Figure 2. Furfural Sample Solution Chromatogram

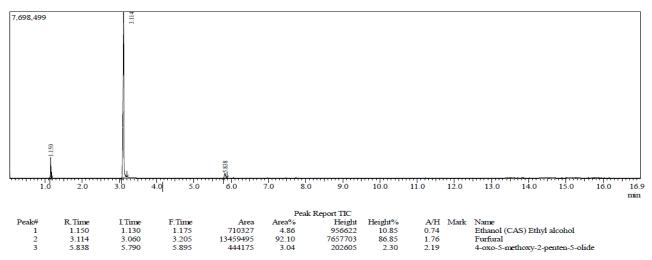


Figure 3. Furfural standard solution chromatogram

According to (Farmakope Indonesia Edisi V., 2014), the retention time difference between the test result chromatogram and the comparison chromatogram is typically less than a tenth of a minute. The probability of

correct identification is quite high if the peaks have the same time under the same conditions.

The furfural sample has a higher concentration than the analyzed standard furfural solution. Table 3 contains additional data from observations. There are several non-

furfural compounds detected, which may result in some differences in the observations of furfural properties. A reactive distillation could be used as a method to overcomes some disadvantages as for lack in purity and results in a higher yield of furfural (Metkar et al., 2015).

Tabel 4. Properties of furfural

Treatment	Density (g/ml)	Boiling Point (°C)	Refractive index (°brix)	Viscosity (cp)
A	1.140	161	1.512	1.29
В	1.130	160	1.502	1.46
C	1.153	1154.5	1.497	1.30
D	1.147	159	1.506	1.31
E	1.160	159	1.501	1.35

#### 3.4. Aniline acetate for furfural identification

To determine the results obtained were furfural, furfural identification with aniline acetate was performed. The sample is tested by sprinkling an aniline:acetate (1:1) solution on it. Table 5 shows the results of furfural identification with aniline acetate.

Table 5. Identification of furfural using aniline acetate

Treatments		Aniline acetate identification		
		Colour	result	
A	2	Dark red	+	
В	2	Dark red	+	
C	2	Dark red	+	
D	2	Dark red	+	
E	2	Dark red	+	

(+) contains furfural

Based on the observations, each furfural sample tested produced a red color when reacted with aniline acetate, as shown in Table 5. This demonstrates that all of the samples obtained were furfural. A condensation reaction between furfural and aniline produces dianil hydroxy gluconate dialdehyde, which causes the color to change to dark red.

Furthermore, (Pressure, 2014) describes the reaction that occurs when aniline acetate is reacted with furfural. The reaction occurs in two stages. The first stage involves the formation of a yellow color, which then reacts with aniline, resulting in the breakdown of the furfural ring to form dialdehyde. This reaction was used as a furfural test because furfural has chemical properties that cause a distinct color when reacted with aniline.

#### 3.5. Density of furfural

The furfural density from parts of the *mensiang* plant can be seen in Table 4. Furfural density ranges from 1.155 g/ml to 1.161 g/ml at 20°C, according to (States and Toxicology, 1989). According to Table 4, the furfural densities obtained ranged from 1.130 g/ml to 1.160 g/ml. Densities included in standards are 1.153°C for treatment C and 1.160°C for treatment E. The density of the plant parts differs slightly, as shown in table 4. This is influenced by the mass of wood at the base, which is dominated by latewood with thick cell walls and small cell cavities, resulting in a higher density. This

influences the density of plant parts from the plant's base to the plant's tip (flowers) (Uar et al., 2018).

#### 3.6. Boiling point

More than 95% of furfural is distilled at temperatures ranging from 159°C to 162°C, according to (*Farmakope Indonesia Edisi V.*, 2014). According to Table 4, the furfural boiling point ranges from 154°C to 161°C, so the average furfural boiling point of the research results was still within the predetermined range. Furfural has a boiling point of 161°C, according to (Gebre et al., 2015).

The boiling point of furfural is affected by the density of furfural, plant part that has higher density affected the boiling point of the furfural. Table 4 shows that the higher the density, the lower the boiling point (Gao et al., 2017).

Treatment C had a boiling point of 154.5°C, which was lower than Pharmacopeia and Barenbers standards. When making observations, the air pressure caused this. A difference in pressure can cause a difference in the boiling points of a solution. When the pressure in a pressure chamber varies slightly from 760 mmHg, the boiling point of the organic liquid deviates from the specified value (Firdaus, 2011).

#### 3.7. Refractive Index

Based on (Mitarlis et al., 2011), the refractive index of furfural at 20 °C was 1.5261 °Brix and at 25 °C was 1.5235 °brix. According to Table 4, the furfural sample obtained has a refractive index value lower than the standard, with the highest refractive index obtained in treatment A at 20.4°C being 1.5120 °brix. This is due to the presence of substances other than furfural in the samples under investigation. GC-MS analysis revealed that the samples obtained contained compounds other than furfural, such as formic acid, ethanol, acetic acid, and pyrrolidine. These compounds can cause the sample's refractive index reading to differ from the literature.

Firdaus (2011) stated that a compound's refractive index is extremely sensitive to the presence of impurities. Aside from the hemicelllulose used in furfural synthesis, different plant parts contain a variety of chemical compounds. In particular, chlorophyll in the plant's stem and other companies. Meanwhile, those compounds were removed during the distillation process.

We can see from the table that the refractive index of the furfural is mostly the same.

#### 3.8. Viscosity

Furfural viscosity was 1.36 mpa.s-1, according to (Lomba et al., 2011) Meanwhile, the furfural viscosity observations obtained did not meet the existing literature value. According to Table 4, treatment B had the highest furfural viscosity (1.46 cp), while treatment A had the lowest (1.29 cp). This is due to the presence of other compounds in the sample besides furfural. GC-MS observations revealed that there were other compounds in the sample besides furfural. According to (Lide and Baysinger, 2005), the formic acid compound has a viscosity of 1.607 mpa.s-1, which was thought to affect the furfural viscosity tested.

The viscosity of the furfural will be affected by the part of the plant with a higher boiling point. Furfural has a high mobility because of its high boiling point. As a result, the viscosity of furfural decreases. Table 4 shows that the sample with the highest boiling point has the lowest viscosity.

#### 4. Conclusion

Following the research findings, *mensiang* plant can be used as a source of cellulose, with cellulose content ranging from 22 to 33% in parts of the plant, and it can also be used as a raw material for furfural production, with the highest yield of 11,78 % in treatments C and D. The results also revealed that the obtained furfural content is 96% higher than the commercial furfural standard, whereas the commercial furfural standard has 92,10 %. More research should be done on the saponification that occurs during the distillation process in order to increase the furfural yield.

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