Deep Eutectic Solvents K_2CO_3–Ethylene Glycol As Extractor Agent for Sago Bark Waste (*Metroxylon sago* Rottb. L)

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Abstract: *Sago starch is a staple food for residents in Luwu Regency, North Luwu Regency, and East Luwu Regency. The increasing demand for sago starch is in line with the deposit of its waste. Unfortunately, sago bark waste has not been widely utilized appropriately. This study utilized a deep eutectic solvent (DES) K_2CO_3-ethylene glycol to extract versatile solvent candidates from sago bark waste. The research stages began with DES synthesis and followed by sample extraction with the help of sonication and DES solvent. DES synthesis was confirmed through FT-IR, while the compounds in the extract were confirmed through GC-MS. DES was successfully synthesized with the presence of characteristic functional groups of K_2CO_3 and ethylene glycol in the solvent functional group. From GC-MS analysis, there was a dioxolane compound with 90% similarity. It is assumed that there is an interaction between the DES constituent (ethylene glycol) and the sago bark constituent. Further research is needed to determine the interaction of DES K_2CO_3-ethylene glycol with other lignocellulosic materials.*

Keywords: *Deep Eutectic Solvents; K_2CO_3-Ethylene Glycol; green extraction; sago bark waste; lignocellulosic materials*

1. INTRODUCTION

Sago palm (Metroxylon Sagu) is a plant that grows abundantly in the Tana Luwu region, South Sulawesi Province, Indonesia. Sago plants are one of the plants that contain abundant lignocellulose, especially in the bark of sago trees (Shen, et al., 2019). The sago starch processing industry produces waste in the form of solids or liquids (Waluyo, 2024), where the solid waste produced is sago bark. Sago palm bark is a lignocellulosic biomass containing lignin (32.29%), cellulose (29.75%), and hemicellulose (23.58%) (Lestari et al., 2022), which has the potential as a source of renewable chemicals for industrial, pharmaceutical, and energy applications (Matsumura et al., 2000). However, sago palm bark waste has not been optimally utilized, causing environmental problems due to poor management.

Biomass waste extraction can be done in various ways, including ultrasonically, the process of extracting organic compounds in plants using organic solvents is faster to break down cell walls so that the compounds to be extracted are easier to obtain (Uluma, 2022). The sonication method speeds up the contact time between the solvent and the sample, even at room temperature. This process accelerates the mass transfer of bioactive compounds from the sample to the solvent (Zhou et al., 2024). Deep Eutectic Solvent (DES) is an environmentally friendly solvent consisting of two compounds, HBA (Hydrogen Bond Acceptor) and HBD (Hydrogen Bond Donor), mixed together in the right ratio to reach the eutectic point. DES is generally used as a solvent in the extraction process (Milenia et al., 2021). Potassium carbonate (K_2CO_3)-based DESs are also beginning to be explored, primarily because K_2CO_3 is a common and inexpensive inorganic base with environmentally friendly characteristics. Among these K_2CO_3-based DESs, the K_2CO_3-Ethylene Glycol (EG) mixture has received significant attention because EG is also a cheap and easily renewable compound. This supports the use of K_2CO_3-EG DESs as a solvent in various extraction processes (Zhou et al., 2024).

So far, there have not been many studies examining the effect of K_2CO_3-EG DESs in the extraction of sago bark waste. This study examined the effect of sonication time on the filtrate and residue during the extraction process, as well as the compound content of the extract.

2. MATERIALS AND METHODS

a. Sago bark powder preparation

The sago bark samples that have been dried at room temperature are then ground and sieved through 80 mesh and stored in an airtight container.

b. DES Synthesis

DES is made by mixing potassium carbonate and ethylene glycol at a molar ratio of 1:2, heated at

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80°C for 60 minutes or until homogeneous with a stirring speed of 200 rpm (Muryanto et al, 2024).

c. Extraction

3 g of sago bark and 30 ml of DES K_2CO_3-EG were mixed and then put into a sonicator at a time span of 5, 10, 15, 20, 25, 30 minutes at a temperature of 50°C and a frequency of 50 W. The extract was then filtered with Whattman filter paper no. 42. The filtrate and residue were analyzed using FT-IR. The compound content in the filtrate was analyzed through GC-MS results.

3. RESULTS AND DISCUSSION

This section is divided into four parts. The parts are the synthesis of DES K_2CO_3-EG, the effect of sonication time on the functional groups of the filtrate, the effect of the highest sonication time on the functional groups of the extract residue and the extract compounds from the filtrate that were successfully identified using GC-MS. For the first parts, DES K_2CO_3-EG was successfully synthesized.

3.1 Synthesis of DES K_2CO_3-EG

Based on Figure 1, it is known that the typical vibrations of ethylene glycol (EG) include stretching O-H (a, 3437 cm^{-1}), stretching C-H (b, 2927 cm^{-1}), stretching carbonyl C=O (1672 cm^{-1}), stretching C-O (e, 1085 cm^{-1}) and typical vibrations of carbonate ion from K_2CO_3 namely stretching C-O (1060 cm^{-1}) and bending O-C-O (885 cm^{-1}) are present in the DES spectrum. Meanwhile, 2 new vibrations were also found, stretching conjugated C=O (c, 1668 cm^{-1}) and bending CH_2 (d, 1448 cm^{-1}). Along with these facts, the synthesized DES is in a single liquid phase after heating. In this study, the storage treatment at low temperature also showed that DES is in a stable single phase. Thus, we conclude that DES K_2CO_3-EG was successfully synthesized.

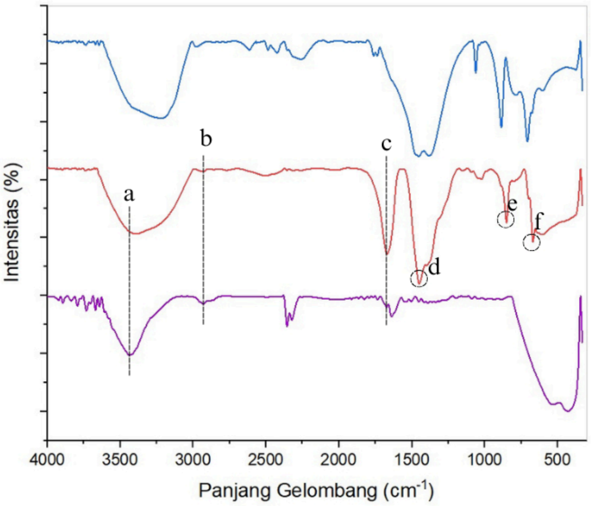

Figure 1. Comparative IR Spectra of K_2CO_3, DES, and EG

3.2 Effect of Sonication Duration

Based on Figure 2, it is known that the difference in sonication time has little effect on the spectrum of each extraction filtrate. However, each spectrum still displays the characteristic lignin vibrations. The analysis results show absorptions of functional groups similar to those of lignin. However, in some vibration ranges, the spectrum still appears faint. This could be due, among other things, to the purity of the extracted sample.

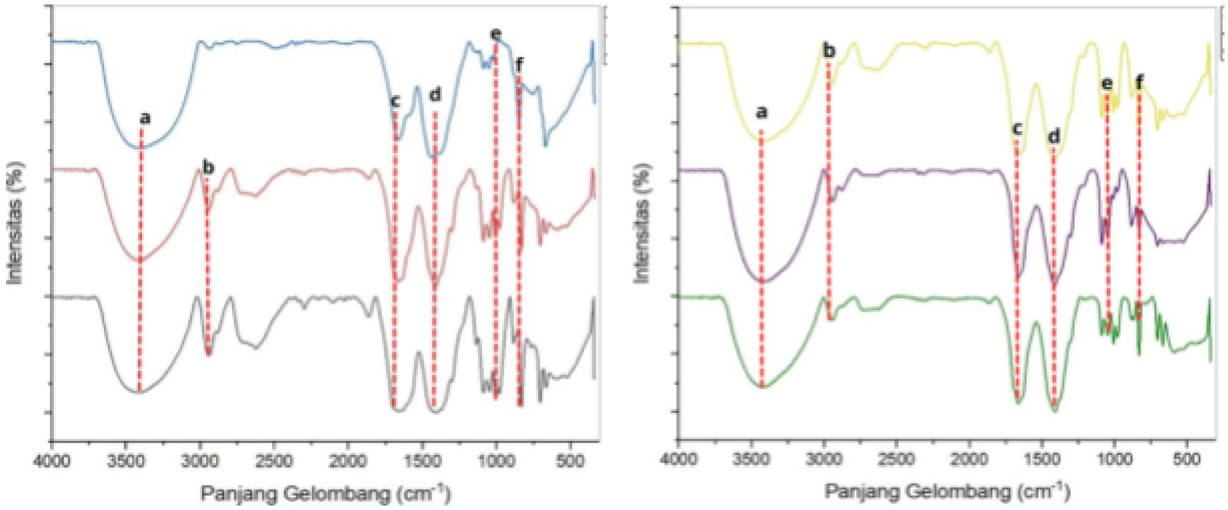

Figure 2. IR absorption of each filtrate with different sonication durations. sonication durations of 5 minutes (bottom left), 10 minutes (middle left), 15 minutes (top left), 20 minutes (bottom right), 25 minutes (middle right) and 30 minutes (top right).

Several functional groups whose intensity becomes clearer with increasing sonication time include a) O–H stretching vibration (3412–3416 cm^{-1}), b) C–H stretching vibration (2941–2945 cm^{-1}), d) C=O stretching vibration (1631–1664 cm^{-1}), e) CH_2 bending vibration (1404–1440 cm^{-1}), and C–O Bending Vibration (1006–1087 cm^{-1}). especially Aromatic C–H bending vibration (f, 831–849 cm^{-1}), it is known that the intensity decreases with increasing sonication time as shown in Figure 2. It is assumed that the amount of lignin compounds in the sample is getting lower due to the breakdown by the solvent and sonication applied. However, visually, the sonication process caused a color change in the sample, which became darker with increasing sonication duration. This indicates the presence of a complex compound release process, which was then confirmed through functional group analysis using FT-IR.

3.3 Effect Sonication on Residue

The difference in functional groups of sago bark before and after sonication is shown in Figure 3. The notation (g) is the absorption band in KBS before extraction in the range of 3414.00 cm^{-1} with moderate to strong intensity shifted slightly to 3446.79 cm^{-1} indicating the stretching vibration of the hydroxyl group (-OH) of cellulose, hemicellulose, and lignin compounds (Sim et al., 2012). The notation (h) is the absorption band of KBS before extraction in the range of 2926.01 cm^{-1} with strong intensity indicating the stretching vibration of the C-H group of the lignocellulose structure. and after extraction, it only shifted to 2927.94 cm^{-1} with the same intensity, which indicates that the aliphatic structure of lignin is still present and has not been completely degraded.

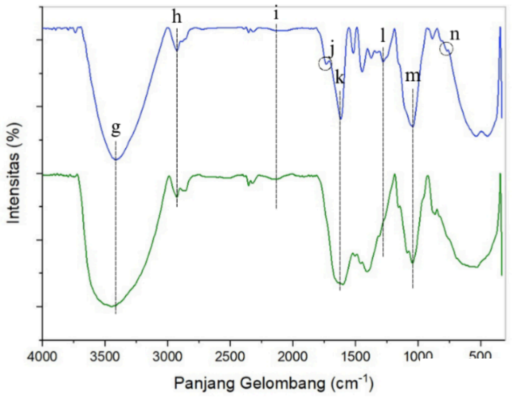

Figure 3. Comparative spectrum of sago bark before (top) and after extraction (bottom).

Notation (i) is the KBS absorption band before extraction in the range of 2112.05 cm^{-1} with strong intensity indicating the stretching vibration of the C≡C group while in the KBS absorption band after extraction in the range of 2148.70 cm^{-1}. This can occur because when KBS is extracted using DES, where this group forms a new interaction with the DES compound so that there is a shift in the wave number towards higher (Pavia et al., 2015). Notation (j) is the KBS absorption band before extraction in the range of 1732.08 cm^{-1} with strong intensity indicating the stretching vibration of the C=O group of the ester compound contained in lignin, but in the extracted KBS this group is no longer visible. This occurs because extraction with DES and the sonication process can dissolve and remove the compound (Quek et al., 2019). The notation (k) is the KBS absorption band before extraction in the range of 1616.35 cm^{-1} with strong intensity indicating the stretching vibration of the C=C group which is a characteristic aromatic compound of lignin (Wan, et. al, 2017) and after extraction it is at 1631.78 cm^{-1} with strong intensity confirming that after harsh treatment by sonication, the aromatic group remains in the residue, but does not appear in the filtrate. The notation (l) is the KBS absorption band before extraction in the range of 1278.81 cm^{-1} with strong intensity indicating the stretching vibration of the C-O-C group which is an ether bond of cellulose and lignin (Hammado et al., 2020), and after extraction it appears at 1155.36 cm^{-1}.

The notation (m) is the KBS absorption band before extraction in the range of 1041.56 cm^{-1} with moderate to strong intensity indicating the stretching vibration of the C-O group is the main bond in the structure of the cellulose compound in KBS (Hammado et al., 2020), and after extraction shifted slightly to 1043.49 cm^{-1}. The notation (n) is the KBS absorption band before extraction in the range of 767.67 cm^{-1} with strong intensity indicating the bending vibration of the long chain CH_2 group (Silverstain, et. al., 2014) long chain fatty acid compounds, natural waxes, the absorption band of this group does not appear after KBS is extracted. we assume that these compounds are also dissolved by sonication treatment and DES solvent. Thus, based on the description of the IR vibration of its functional group, the DES solvent together with sonication treatment directly succeeded in damaging the lignocellulose cell wall of sago bark.

3.4. Compound Identification from Extract

We identified compounds in the filtrate using GC-MS. Figure 4 shows a GC-MS chromatogram of the filtrate from a 30-minute sonication period. This filtrate was selected considering the longest the sago bark could withstand sonication and DES solvent treatment. Based on this chromatogram, we grouped several compounds with >90% and 80% similarity, all of which share an aromatic ring in the suggested structure.

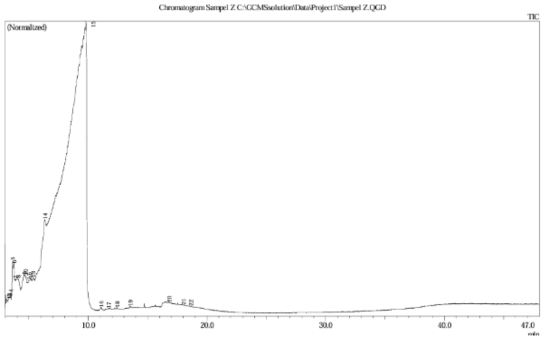

Figure 4. Chromatogram GC-MS of extract

Based on the analysis, it is known that 5 of the 6 compounds with a similarity level above 90% are 1,3-Dioxolane, 2 methyl- (similarity index up to 96%), while the rest are Borane-methyl sulfide complex compounds (similarity index 91%). While at a similarity level between 80-90%, there are geraniol compounds (similarity index 84%) and simple organic compounds such as acetic acid (similarity index 87%), butane (similarity index 81%) and methoxyacetic acid (similarity index 81%) in the filtrate. at a similarity level of 70-79%, there are hydroperoxide compounds (similarity index 79%) and butane (similarity index 75%). groups of compounds with a similarity level of >70%, namely Urethane (similarity index 67%), nordazefam (similarity index 63%), 3-trans-(1,1-dimenthylethyl)-4-trans-methoxycyclohexanol (similarity index 61%), and 5-Methyl-2-hexanone oxime (similarity index 59%). Based on these results, it is known that in 30 minutes of sonication with DES solvent, the filtrate begins to contain large aromatic molecular fragments such as lignin as evidenced by the presence of simple organic acids with a similarity index of >80%. In addition, the fragments produce essential aromatic compounds such as geraniol with a fairly high similarity index.

4. CONCLUSIONS

Based on the analysis results, it was concluded that the DES K_2CO_3-EG solvent combined with sonication treatment in sago bark extraction was able to break down lignocellulose compounds in the sample. The resulting compounds included dioxolane, geraniol, and simple organic acids. However, this study needs to be continued further to check the level of lignocellulose damage caused by sonication treatment with DES solvent through other, more complex methods.

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