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Extraction of Phenol from Bio-oil Produced by Pyrolysis of Coconut Shell

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ABSTRACT: *Bio-oil from coconut shell pyrolysis has a very acidic pH and is corrosive due to the presence of phenolic compounds. Phenol is corrosive and can cause damage to the engine. If the bio-oil is intended to be used as an alternative diesel fuel, the phenol content needs to be removed. Phenol actually has an economic value that can be used as disinfectants, resins, pesticides, explosives, drugs and dyes. Separation of phenol from bio-oil can be carried out using liquid-liquid extraction method by utilising solvent as separator, where the liquid phase separation utilises the different solubility of compound to be separated between carrier and solvent solution. In this work, bio-oil produced by pyrolysis of coconut shell was extracted using aqueous methanol as a solvent. The extraction process was carried out for 60 min, and then separated using separating funnel through two phases, i.e., the extract phase and the raffinate phase. The extract phase and the raffinate phase of each extraction processes are analysed with gas chromatography (GC) to obtain the concentration of each component. The objective of this work is to study the effects of the temperature and speed of stirring on the distribution coefficient and the yield of phenol extraction from bio-oil produced by pyrolysis of coconut shell. The analysis results show that the highest distribution coefficient and the yield of phenol extraction was obtained at 50°C and 250 rpm stirring speed.*

Keywords: Bio-oil, phenol, methanol, extraction, extraction of phenol, coconut shell

1. INTRODUCTION

Indonesia has considerable renewable energy potential as a substitute for fossil energy, especially from biomass. One of the available biomass sources is coconut shell. Coconut shells, for now, are untapped waste. Therefore, one of the ways is to utilise coconut shell waste as a base material in the manufacture of bio-oil. The results of proximate, ultimate and thermogravimetric analysis (TGA) show that coconut shells have a high potential for producing fuel fluid through pyrolysis conversion process.[1]

Coconut shell can be transformed into bio-oil through heating process in pyrolysis. Bio-oil made from coconut shell by using slow pyrolysis at the temperature of 250°C–300°C produces liquid which contains a very acidic pH and is corrosive.[2] The largest component of bio-oil is lignin derivatives, namely phenol, alcohol, organic acids and carbonyl compounds such as ketones, aldehydes and esters.[3] Phenol compounds are acidic compounds, therefore the use of this bio-oil will directly cause corrosion on the machine.[4]

Phenol needs to be taken from bio-oil to reduce the corrosiveness of bio-oil. Method that can be used to take phenol compounds from bio-oil is by liquid-liquid extraction method using a solvent such as methanol.[5] This has been done in a study by Jazbinsek et al. on the isolation of phenol compounds from bio-oil resulted from pyrolysis of forestry waste, and Mantilla et al. on the extraction of phenol compounds from bio-oil from pyrolysis of agricultural waste.[6,7] Dahlia conducted a research on liquid-liquid extraction of phenol compound from bio-oil resulted from pyrolysis of palm oil empty bunches using methanol solvent.[8] The yield of this extraction process was 40%.

The aim of this research is to separate the phenol compounds from bio-oil solution (from coconut pyrolysis) using liquid-liquid extraction method. The purpose of phenol extraction is to improve the quality of bio-oil in order to make it not corrosive. Therefore, when used as fuel, it will not damage the machine.

2. EXPERIMENTAL

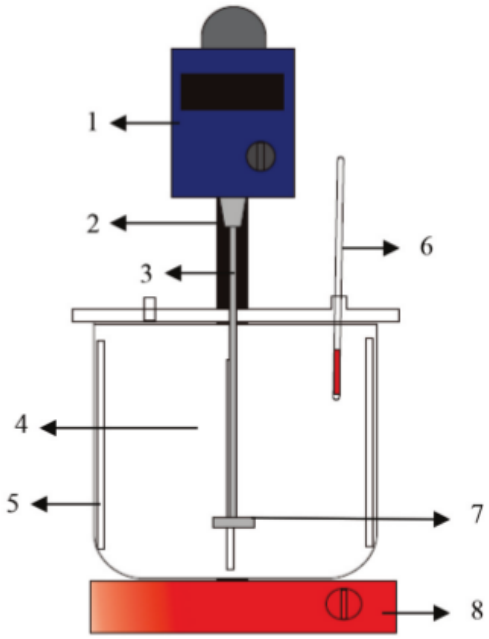
2.1 Materials

Materials used in this research are bio-oil, methanol (e-MERCK), chloroform (e-MERCK) and distilled water. Bio-oil used in the present work was prepared from our previous work.[9]

2.2 Methods

Apparatus used in this research are hot plate stirrer, thermometer, gas chromatography (GC) product by GC 6820 Agilent Technologies, and gas chromatography-mass spectroscopy (GC-MS) product by GCMS-QP2010S Shimadzu.

The extraction equipment is showed in Figure 1.



Legends:

1. Motor
2. Slab stand
3. Stirrer
4. Flask
5. Baffle
6. Thermometer
7. Impeller
8. Hot plate

Figure 1: Extraction equipment.

Phenol was extracted from bio-oil using two-stage extraction:

1. The first stage of extraction used a distilled water as polar solvent and chloroform as a non-polar solvent. This extraction used ice-bath method at 4°C–5°C, 200 rpm stirring speed and stirring time for 2 h. The extract phase which was obtained at this first stage was used as the feed in the second stage extraction.

2. For the second stage, each extract phase was extracted using an aqueous methanol solvent. The material systems and experimental conditions for the equilibrium extraction are summarised in Table 1.

Table 1: Material systems and conditions for equilibrium extraction.

	Quantity
Feed volume	2.5 ml
Solvent	Aqueous methanol (according to the literature)[9]
Mole fraction of water in solvent	0.2 (according to the literature)[9]
Solvent-feed mass ratio	1
Shaking time	60 min
Amplitude of shaking	150, 200, 250 rpm
Temperature	25°C, 40°C, 50°C

After the equilibrium had been attained (60 min), the mixtures were poured into a separating funnel, settled for an hour and separated into two phases. Then the two phases were weighed. The extract phase and the raffinate phase of each extraction processes were analysed by GC to obtain the concentration of each component. The principal conditions of this analysis are shown in Table 2.

Table 2: Conditions of analysis using GC.[9]

Column: Rastek RXi-5MS		
Column:		
Inner diameter	[m]	3.2×10^{-4}
Length	[m]	30
Carrier gas		He
Split ratio	[–]	153
Flow rate	[cm s^{-1}]	26.6
Sample volume	m^3	1.10^{-9}
Injection temp.	[K]	553
Column temp.	[K]	313–573
Pressure column	kPa	10.0
Column flow	ml min^{-1}	0.54
Detector (FID) Temperature	[K]	573

The distribution coefficient was calculated by using Equation 1:[9]

$$Distribution\ of\ Coefficient = \frac{mole\ fraction\ of\ solute\ in\ the\ extract\ phase}{mole\ fraction\ of\ solute\ in\ the\ raffinate\ phase} \quad (1)$$

3. RESULTS AND DISCUSSION

Bio-oil was analysed for its components and concentration by using GC-MS. From the analysis by GC-MS on bio-oil, the concentration of each component will be obtained. The chromatograms of the GC-MS analysis are shown in Figure 2.

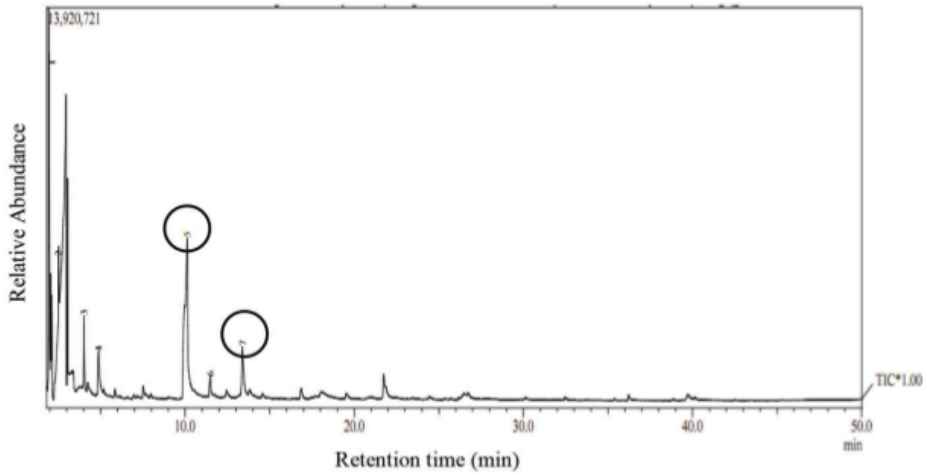


Figure 2: GC-MS spectra components produced from the pyrolysis of coconut shell.

The results of the GC-MS analysis show that the bio-oil contained more than seven chemical compounds, such as ethyl ester, phenol, furfural and others. The main components of bio-oil from coconut shell are presented in Table 3.

Table 3: Main chemical components of bio-oil from coconut shell.

Peak	Component	Content (%)
1	L-Alanine, methyl ester	37.60
2	Formic acid, ethylene	4.02
3	Propionic acid, 1-hydroxy-2-butanone	3.89
4	Furfural	5.45
5	Phenol	40.01
6	Cyclopentane	2.01
7	Phenol compounds	7.02

The percentage (%) of the compositions of the bio-oil analysis by GC-MS was obtained from the percentage area of the peak or the height of the peak in the chromatograms. The total phenolic compounds contained in bio-oil was approximately 47.03%.

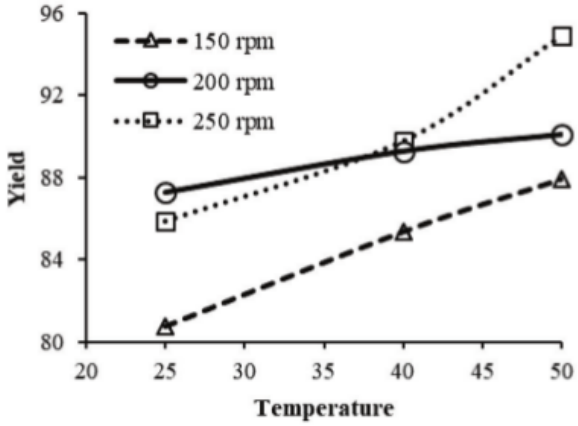


Figure 3: The effect of stirring speed and temperature of extraction for the yield of phenol.

The increase in temperature will increase phenol concentration in the extract phase. It is because the increasing temperature increases the kinetic energy of the solution and the solvent diffusion into the solute tissue cells.[10] It affects the amount of the extracted phenol from diluent, which means that more phenol is produced. However, if the extraction temperature is too high it can decrease the phenol concentration in the extract phase, according to the nature and boiling point of the solvent.[1] Figure 3 shows the effect of temperature and stirring speed for the yield of phenol extraction. The highest yield of phenol extraction (i.e., 94.89%) was obtained at 50°C and 250 rpm, and the lowest yield of phenol extraction (i.e., 80.73%) was obtained at 25°C and 150 rpm. For the extraction of phenol using methanol solvent, the yield value increases along with the greater stirring rate used because greater the stirring rate will increase the driving force which cause the extraction process, so that the solution dissolved from diluent can be maximised. In addition, greater speed of stirring will enlarge the contact area between the solution and the solvent.[7]

Figure 4 shows the effect of temperature and stirring speed on the distribution coefficient of phenol extraction. The highest distribution coefficient of phenol extraction (7.29) was obtained at 50°C and 250 rpm and the lowest distribution coefficient of phenol extraction (1.47) was obtained at 30°C and 150 rpm. The distribution coefficients of phenol extraction are more than 1. It shows that methanol solution had successfully separated phenol from the diluent.[5] The distribution coefficient increases along with the increasing speed of stirring. It indicates that the increase in stirring speed raises the amount of phenol that moves into the extract phase because the diffusion rate is influenced by the distance travelled by

the diffused compound. Longer distance leads to the lower diffusion rate, thus shortening the distance is done by increasing the stirring speed.[11]

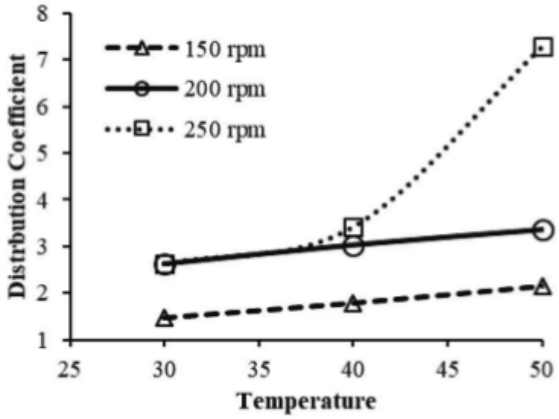


Figure 4: The effect of stirring speed and temperature for the distribution coefficient of phenol extraction.

4. CONCLUSION

From the results of phenol extraction from artificial bio-oil solution, it can be concluded that the best conditions of the extraction process are temperature of 50°C and stirring speed of 250 rpm, which give the highest yield of phenol extraction (i.e., 94.89%) as well as the highest distribution coefficient (i.e., 7.29).

5. ACKNOWLEDGEMENTS

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