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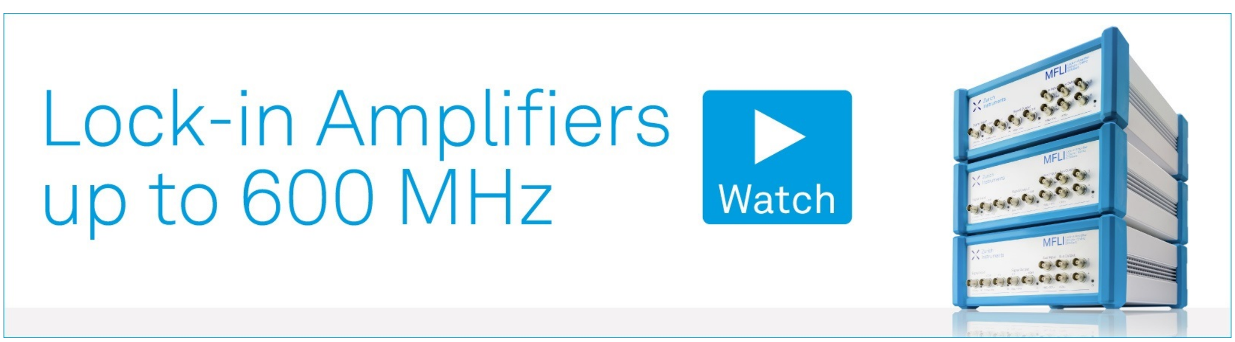

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The Separation of Phenolic Compounds from Bio-oil Produced from Pyrolysis of Corncobs

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Abstract. Corncob is an agricultural waste which has a potential to be utilized as a raw material for bio-oil. One of many processes to convert the agricultural waste (biomass) into bio-oil is pyrolysis. The pyrolysis process for this study was carried out using fixed bed pyrolysis reactor, performed under temperature of 500°C and 600°C with residence time of 4 seconds. The bio-oil was analyzed for its components content using Gas Chromatography-Mass Spectrometry (GCMS). The separation of phenolic compounds from bio-oil was done by a liquid-liquid extraction which carried out for 60 minutes using methanol 80% and chloroform as solvent at various extraction temperatures of 30°C, 40°C, 50°C, and stirring speeds of 200 rpm, 250 rpm, 300 rpm. Then, the extract phase and the raffinate phase were analyzed using Folin-Ciocalteu method. The aim of this study was to determine the effect of temperatures and stirring speeds of the liquid-liquid extraction to the distribution coefficient and the yield of phenolic compound. The result showed that the main component of bio-oil obtained is phenolic compounds. The highest distribution coefficient and the highest yield of phenolic compound were obtained at 50°C and 300 rpm. The highest yield was 66.31% and 58.9% obtained from pyrolysis process at 500°C and 600°C, respectively. Meanwhile, the highest distribution coefficient was 11.08 and 8.92 for pyrolysis at 500°C and 600°C.

INTRODUCTION

Petroleum, a non-renewable source of energy, is still being used as the main fuel in most of our activity, especially in Indonesia. Therefore, efforts are needed to replace petroleum through the use of renewable energy to overcome the sustainability problems. One of many renewable energy kinds that can be developed in Indonesia is biomass. Biomass refers to non-fossil organic materials derived from plants, animals and microorganisms. Biomass includes products, by-products, and also wastes from agriculture, forestry, and industry [1].

Being a big commodity in Indonesia, corn produces an enormous amount of agricultural wastes along with its production, which includes some parts (40-50% of total corn weight) called corncobs. The availability of corncobs can be predicted from corn production in Indonesia. Table 1 showed the data of corn production in 2012-2016 which was obtained from Badan Pusat Statistik Indonesia [2].

TABLE 1. Corn production in Indonesia

Year	Amount (ton)
2012	19,387.022
2013	18,511.853
2014	19,008.426
2015	19,612.435
2016	23,578.413

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Corncob is a waste containing a hemicellulose, cellulose, and high level of lignocellulose up until 39.99% [3]. With that much content of lignocellulose, corncob can be easily processed to be utilized as a raw material for bio-oil production [4]. Bio-oil is a dark colored liquid and has smoke-like smell. Bio-oil can be produced from biomass, such as corncob, wood, bark, and other celluloses [5]. Corncob can be converted into bio-oil by pyrolysis process.

Pyrolysis is a decomposition process of a substance or material carried out at a relatively high temperature. This process produces liquid (bio-oil), solid (bio-char) and gas [6]. Bio-oil can be used as an alternative fuel instead of diesel fuel. Furthermore, it is also easier to be stored. Bio-oil contained a lot of organic components which are derivatives of lignin such as phenols, alcohols, organic acids, and carbonyls such as ketones, aldehydes, and esters [7].

The presence of phenolic compound will reduce the quality of bio-oil because it causes low heating value, corrosiveness, high acidity, high viscosity, and unstable product that damaging the machine [8]. So, it is necessary to separate the phenolic compounds to obtain a better quality of bio-oil. One of the most economical methods for separating the phenolic compounds from bio-oil is liquid-liquid extraction method.

Extraction is a liquid phase separation process by utilizing the solubility difference of substances that will be separated [9]. In this study, the purification of phenolic compounds was being done through a liquid-liquid extraction method with the help of solvents such as methanol 80% as polar solvent and chloroform as non-polar solvent.

RESEARCH METHODOLOGY

Materials

All reagents used here were A.R. grade and used as received without further purification. Methanol, gallic acid, and chloroform were purchased from e-Merck (Germany). Corncob was obtained from public market in Semarang.

Corncob Pyrolysis Process for Bio-Oil Producing

Pyrolysis was performed using fixed bed pyrolysis reactor operated at 500°C and 600°C with residence time of 4 seconds. Gas which was produced by pyrolysis process was condensed using the condenser. This liquid which was produced by condensation process is called bio-oil. Bio-oil was collected in the dark bottle and settled for an hour. Then, the bio-oil was analyzed for its components content using Gas Chromatography-Mass Spectrometry (GC-MS), GC Clarus 680 and MS Clarus SQ 8T from Perkin Elmer. The GC-MS analysis conditions were already reported in our previous work [10]. The characteristics of bio-oil such as pH, viscosity, density, acid number, flash point, and heating value were also analyzed. The series of fixed bed pyrolysis equipment is shown in Figure 1.

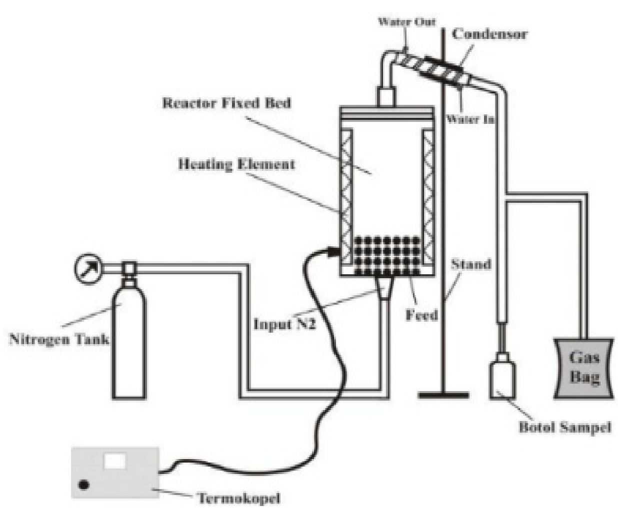

FIGURE 1. Pyrolysis Equipment

Extraction of Phenolic Compounds from Bio-Oil

First, the bio-oil was neutralized using NaOH solution for pH of 5. Then, the extraction was carried out for 60 minutes using methanol 80% and chloroform solvent at various extraction temperatures of 30°C, 40°C, 50°C, and stirring speeds of 200 rpm, 250 rpm, and 300 rpm.

After the extraction process, the solution was separated using separating funnel and settled for 60 minutes to form the extract and raffinate phase. Then the extract phase and raffinate phase were analyzed using Folin-Ciocalteu Method to determine the concentration of phenolic compounds.

Folin-Ciocalteu Method

The extracts and raffinates were taken 1 mL each into 10 mL measuring flasks. Each of the solution were added by 0.5 mL Folin-Ciocalteu reagent and shaken until it homogeneously mixed. About less than 8 minutes, 4 mL of 7.5% Na_2CO_3 solution was added and followed by addition of distilled water and then settled for 15 minutes. Subsequently, the total phenolic compound was analyzed using a UV-Vis spectrophotometer with λ = 765 nm.

RESULTS AND DISCUSSION

Characterization of Bio-oil Produced from Pyrolysis of Corncob

Bio-oil produced from pyrolysis were analyzed and characterized in order to measure its viscosity, density, acid number, and pH. Bio-oil resulted from the corncob pyrolysis at 500°C and 600°C has yellowish brown color and smoky smell. The physical properties of bio-oil are shown at Table 2.

TABLE 2. The physical properties of bio-oil

Parameters	Pyrolysis at 500°C	Pyrolysis at 600°C
Density (kg/m^3)	1.0227	1024.7
Viscosity at 40^{o}C (cSt)	0.9832	1.0343
Acid Number (mg KOH/gr)	99.4031	82.4930
pH	3.5	4
Flash Point (^{o}C)	151	147
Heating Value (MJ/kg)	3.7882	3.9708

TABLE 3. Physical properties of corncobs bio-oil compared to other biomass-based bio-oil and petroleum products

Raw Material	Density (kg/m^3)	Viscosity (cSt)	pH	Flash Point (^{o}C)	Calor Value (MJ/kg)	Reference
Corncobs	1,022.7	0.9823	4	151	3.7882	Experimental
Corncobs	1,162	20.80	5.2	-	16.54	Ogunjobi and Lajide [11]
Sugar Baggase	1,039	14.20	2.17	62	27.75	Varma et al. [12]
Coconut shell	1,053	1.47	-	-	19.75	Rout et al. [13]
Saal seeds	921	1.32	3.7	53	23.75	Singh et al. [14]
Neem seeds	961	22.6	3.9	42	32.3	Nayan et al. [15]
Rice straw	1,153	10.81	3.21	103	18,34	Islam et al. [16]
Diesel	820-850	2-5.5	5.6	53-80	42-45	Rout et al. [13]
Bio-diesel	880	4-6	-	100-170	37-40	Rout et al. [13]

TABLE 4. The composition of bio-oil produced by pyrolysis of corncob at 500°C

Retention Time	Percentage (%)	Compounds
4.479	13.24%	Furfural
5.544	7.24%	2-Furanmethanol
6.494	1.41%	2-Cyclopenten-1-one, 2-methyl-
6.739	1.04%	Ethanone, 1-(2-furanyl)-
8.68	4.77%	2-Cyclopenten-1-one, 3-methyl-
9.886	1.70%	1,6-Heptadien-4-ol
10.951	23.86%	Phenol
12.977	10.91%	Ethanone, 1-(1-cyclohexen-1-yl)-
13.982	10.59%	Phenol, 4-methyl-
16.583	8.54%	Phenol, 3-ethyl-
18.514	2.57%	Phenol, 4-ethyl-2-methoxy-
19.124	6.10%	1,2-Benzenediol
20.745	2.04%	Phenol, 2,6-dimethoxy-
21.51	3.26%	3-Chloro-4-hydroxyiminocarane
23.136	0.99%	Phenol, 4-methoxy-3-(methoxymethyl)-
23.626	1.74%	3-Buten-2-one, 3-methyl-4-(1,3,3-trimethyl-7-oxabicyclo[4.1.0]heptan-1-yl)-

TABLE 5. The Composition of Bio-oil Produced by Pyrolysis of Corncob at 600°C

Retention Time	Percentage (%)	Compounds
4.449	9.18%	3-Cyclopentene-1-acetaldehyde, 2-oxo-
5.434	8.18%	2-Furanmethanol
6.449	1.02%	2-Cyclopenten-1-one, 2-methyl-
6.699	1.27%	Ethanone, 1-(2-furanyl)-
7.045	1.04%	Butyrolactone
8.605	3.76%	2-Cyclopenten-1-one, 3-methyl-
9.816	2.60%	1,6-Heptadien-4-ol
10.896	21.22%	Phosphonic acid, (p-hydroxyphenyl)-
12.882	11.18%	Phenol, 2-methoxy-
13.502	2.18%	Sulfurous acid, isobutyl pentyl ester
13.907	7.78%	Phenol, 4-methyl-
15.818	1.14%	Phenol, 2,5-dimethyl-
16.073	1.05%	2-(2-Isopropenyl-5-methyl-cyclopentyl)-acetamide
16.528	6.90%	Phenol, 4-ethyl-
17.484	1.91%	2-Vinyl-9-[3-deoxy-β-d-ribofuranosyl]hypoxanthine
18.474	2.10%	Phenol, 4-ethyl-2-methoxy-
19.019	8.84%	1,2-Benzenediol
20.68	2.86%	Phenol, 2,6-dimethoxy-
21.46	5.76%	3-Chloro-4-hydroxyiminocarane

Analysis using GC-MS shows that the main component of the bio-oil was phenolic compounds which were about 48.59% (for pyrolysis temperature at 500°C) and 31.96% (for pyrolysis temperature at 600°C). Additionally, the main components of bio-oil produced via pyrolysis of corncob at pyrolysis temperature of 500°C and 600 °C are listed in Table 4 and Table 5, respectively. These findings are in accordance with another research study reported by Sun et al. [17], which studied the content of bio-oils from various biomasses. They found that the main chemical compounds of bio-oil were phenol, aldehyde, ketone, and organic acids. The presence of alcohols, aldehydes, ketones, furfurals, and furans compounds in the obtained bio-oil was due to the decomposition of cellulose and also hemicellulose from the biomass [17]. Meanwhile, aromatic compounds, cresol, and derivatives of phenol appeared as the lignin fraction was degraded [18]. In the bio-oil obtained from corncob pyrolysis results, phenol compounds and their derivatives become the majority of the compound content, leading to a high acid number. So that, it needs further processing for its application. The chemical composition of the bio-oil produced was determined by analyzing the percentage area of the peaks in the GC-MS analysis chromatograms.

TABLE 6. Comparison of components in bio-oil from corncobs pyrolysis with reference

Component	Percentages (%)		
	Temperature 450°C [19]	Temperature 500°C	Temperature 600°C
Furfural	1	13.24	-
3-Cyclopentene-1-acetaldehyde, 2-oxo-	-	-	9.18
2-Furanmethanol	4.1	7.24	8.18
2-Cyclopenten-1-one, 2-methyl-	-	1.41	1.02
Ethanone, 1-(2-furanyl)-	-	1.04	1.27
Butyrolactone	-	-	1.04
2-Cyclopenten-1-one, 3-methyl-	2.6	4.77	3.76
2-Cyclopenten-1-one, 2-hydroxy-3-methyl-	3.9	-	-
2-Cyclopenten-1-one, 3-ethyl-2-hydroxy-	1.8	-	-
1,6-Heptadien-4-ol	-	1.70	2.60
Phenol	6	23.86	-
Creosol	2.0	-	-
Catechol	5.2	-	-
Benzofuran, 2,3-dihydro-	9.1	-	-
Phosphonic acid, (p-hydroxyphenyl)-	-	-	21.22
Phenol, 2-methoxy-	12.4	-	11.18
Ethanone, 1-(1-cyclohexen-1-yl)-	-	10.91	-
Sulfurous acid, isobutyl pentyl ester	-	-	2.18
Phenol, 2-methyl-	3.1		
Phenol, 4-methyl-	-	10.59	7.78
Phenol, 3-ethyl-	-	8.54	-
Phenol, 4-ethyl-2-methoxy-	4.9	2.57	-
Phenol, 2,5-dimethyl-	-	-	1.14
2-(2-Isopropenyl-5-methyl-cyclopentyl)-acetamide	-	-	1.05
Phenol, 4-ethyl-	6.0	-	6.90
2-Vinyl-9-(3-deoxy-β-d-ribofuranosyl) hypoxanthine	-	-	1.91
Phenol, 4-ethyl-2-methoxy-	-	-	2.10
1,2-Benzendiol, 3-methyl-	2.7	-	-
1,2-Benzendiol, 4-methyl-	1.4	-	-
1,2-Benzenediol	-	6.10	8.84
2-Methoxy-4-vinylphenol	7.9	-	-
Phenol, 2,6-dimethoxy-	7.2	2.04	2.86
3-Chloro-4-hydroxyiminocarane	-	3.26	5.76
Phenol, 4-methoxy-3-(methoxymethyl)-	-	0.99	-

Phenol compounds in bio-oil are formed from the decomposition of lignocellulose in corncobs during the pyrolysis process [20]. The content of large phenol compounds is influenced by pyrolysis temperature. The higher the pyrolysis temperature, the more lignocellulose content would be decomposed [21]. But in this study, the results of pyrolysis of corncobs at 500°C produced more phenol compounds than the results of pyrolysis of corncobs at 600°C. It was stated in a study conducted by Zhang et al. [22] that the optimum temperature in the pyrolysis of corncobs is 550°C. If pyrolysis is carried out at temperatures above the optimum, the lignin decomposition process cannot be completely condensed into bio-oil, but instead more of non-condensable gases such as CO, CO_2, methane, ethane, propane and propene [23] will be produced. Thus, the total phenol compound produced at 500°C is greater than that produced at 600°C.

The Effects of Extraction Temperatures and Speeds of Stirring for the Yields of Phenolic Compounds

The extraction process was carried out at temperature of 30°C, 40°C, 50°C and stirring speed of 200, 250, and 300 rpm. The experimental data of extraction with various temperatures and stirring speeds showed that the extraction process at 50°C and 300 rpm produced the highest yield, about 66.31%. The lowest yield about 13.4% was obtained from extraction at temperature of 30°C and stirring speed at 200 rpm.

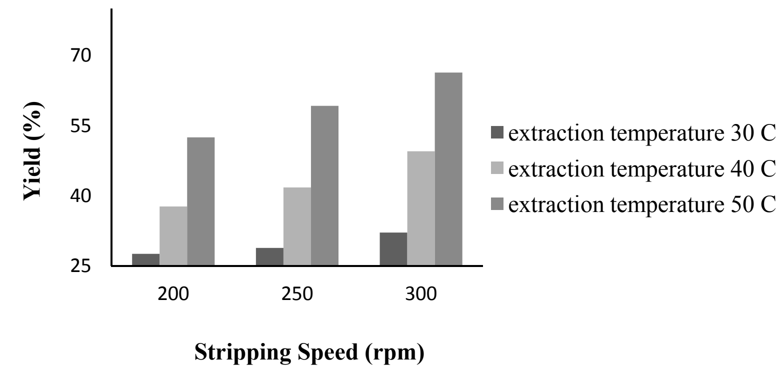

FIGURE 2. The effects of extraction temperatures and stirring speeds for the yield of phenolic compounds in the extraction of bio-oil produced from corncob pyrolysis at 500°C

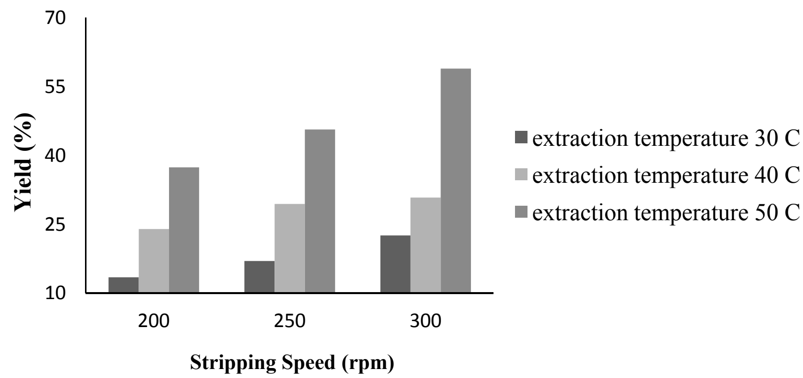

FIGURE 3. The effects of extraction temperatures and stirring speeds for the yield of phenolic compounds in the extraction of bio-oil produced from corncob pyrolysis at 600°C

Figure 2 and 3 shows that the yield of phenolic compound for the extraction of bio-oil produced from corncob pyrolysis at 500°C and 600°C was obtained at the optimum extraction temperature of 50°C. The increasing of the extraction temperature affects in the increasing of phenolic compounds produced. Higher extraction temperature can make solvent has better ability to dissolve into solute [24]. It influences the amount of phenolic compounds moved into solvent from diluent. However, the higher extraction temperature can also decreased the ability of solvent to extract phenolic compounds from bio-oil. It depends on characteristics and the boiling point of the solvent [9].

The highest yield in phenol extraction was obtained from extraction process performed at temperature of 50^{o}C with a yield as much as 58.9% and the lowest yield obtained was 13.47% which was result of extraction at temperature of 30^{o}C. The increase in extraction temperature is directly proportional to the increase in phenol yield produced, due to the longer the contact between the material to be extracted with the solvent [25]. The same thing was said by Wijanarko et al, [24] that operating conditions with high extraction temperatures would make the solvent to have greater ability in dissolving the solute. This affects the amount of phenol extracted from the diluent, so that more phenol would be produced. However, if the extraction temperature is too high, it can reduce the concentration of phenol produced according to the nature and boiling point of the solvent [26]. Based on the yield value produced, it is shown that the most optimum temperature used in phenol extraction is at temperature of 50^{o}C. However, if the extraction temperature is increased the yield will not always follow the trend to be increased, as the boiling point of methanol and chloroform which were used as solvents in this study is 64.5^{o}C and 61.2^{o}C, respectively. Thus, if the extraction is carried out at temperatures above 60^{o}C, the yield produced will be small.

The Effect of Extraction Temperatures and Stirring Speeds for the Distribution Coefficient of Phenolic Compounds

The effect of extraction temperature and stirring speed for the distribution coefficient of phenolic compounds in the extraction of bio-oil from corncob pyrolysis at 500°C and 600°C showed that the highest distribution coefficient of phenolic compound was obtained at 30°C and 300 rpm. They were 3.66 and 3.61 for pyrolysis temperature 500°C and 600°C, respectively.

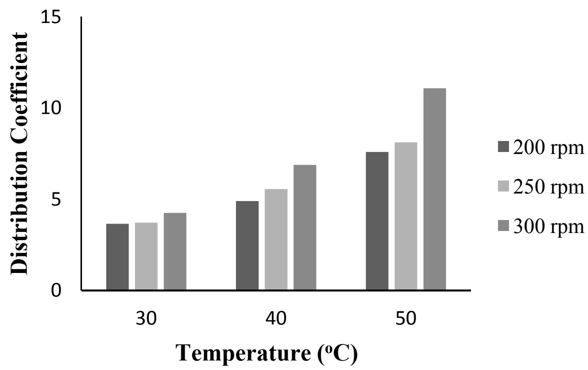

FIGURE 4. The effects of extraction temperatures and stirring speeds for the distribution coefficient of phenolic compounds in the extraction of bio-oil produced from corncob pyrolysis at 500°C

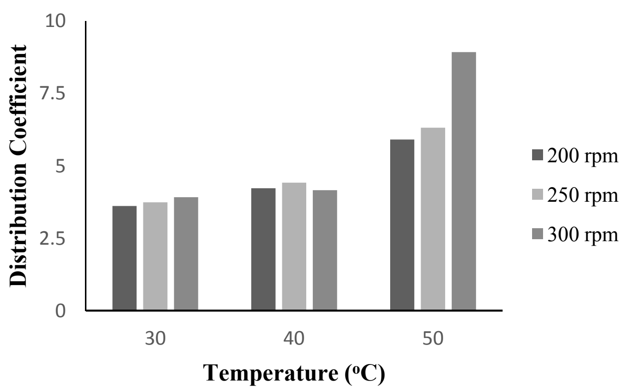

FIGURE 5. The effects of extraction temperatures and stirring speeds for the distribution coefficient of phenolic compounds in the extraction of bio-oil produced from corncob pyrolysis at 600°C

If the distribution coefficient is more than 1, it indicates that the extraction process can be done to extract phenolic compound from bio-oil [4]. The increasing of extraction temperature affects the increasing of the distribution coefficient of phenolic compound. Higher temperature of extraction process can cause the increasing of reaction rate leading to the increase of particle movement from the solute into the solvent [27].

CONCLUSION

From the results and discussion, it can be concluded that the main component of bio-oil is phenolic compounds which was 48.59% for pyrolysis at 500°C and 31.96% for pyrolysis at 600°C. The highest distribution coefficient and the highest yield of phenolic compound were obtained from extraction process at 50°C and 300 rpm. The highest yield produced was 66.31% and 58.9% for pyrolysis at 500°C and 600°C. The highest distribution coefficient was 11.08 and 8.92 for pyrolysis at 500°C and 600°C.

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