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Determination of Phenol Content of Liquid Smoke of Palm Oil Shell: Characterizations by using of Gas Chromatography- Mass Spectra and Fourier Transformed Infra Red

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Abstract
The determination of phenol content of liquid smoke of palm oil shell has been carried out by using Gas Chromatography-Mass Spectra and Fourier Transformed Infrared. Pyrolisis was performed in the furnice that covered by white cement with a temperature of 500°C, 700°C, and 900°C, without and with nitrogen gas inlet with velocity flow of 10 mL/sec. The obtanined liquid smoke of palm oil shell cured for 7 days and then centrifuged at 3800 rpm for 60 minutes. The filtrate was distilled at a temperature of 125°C for 60 minutes. Redestilated liquid smoke analyzed by means of Gas Chromatography Mass Spectra to determine the levels of phenol content. Based on the Gas Chromatography Mass Spectra chromatogram, liquid smoke produced in free air pyrolysis at temperature 500°C, 700°C, 900°C containing phenol 35.09%; 27.92% and 14.49%, respectively, and in the air controlled pyrolysis were 7.86%; 9.25%, and 22.28%, respectively. FT-IR spectra of all liquid smoke product confirmed the presences of phenol functional groups.
Keywords: phenol, liquid smoke, palm oil shell, gas chromatography

Introduction

Liquid smoke is a product of the pyrolysis of organic material that performed at high temperatures without air. Organic materials such as palm kernel shells that primary contain of 26.6% cellulose, 27.7% hemicellulose and 29.9% lignin can undergo pyrolysis produces organic acids, phenol, carbonyl. The compounds content in the liquid smoke may differ depending on the type, moisture content and temperature pyrolysis of organic materials used (Darmaji, 2002 and Himawati, 2010).

To obtain the liquid smoke can be used hardwoods such as mangrove wood, teak wood sawing powder, coconut shell. Some researchers have reported the chemical compounds content in the liquid smoke vary according to the type of organic raw materials and temperature pyrolysis. Research on the content of phenol in liquid smoke from organic materials have been reported, such as corn cobs, stalks and leaves of corn (Fronthea Swastawati et al., 2007 and Matsu Zawa et al, 2007).

Darmaji, 2002, reported that liquid smoke of coconut shell with a phenolic compound content of 4.13%. Pyrolysis of organic materials containing hemicellulose, cellulose and lignin from palm shells can produce phenol derivatives such as guaiacol, siringol and homologues and derivatives (Girard, 1992). Liquid smoke obtained on pyrolysis of organic material rich in lignin compounds and aromatic compounds provide a distinctive aroma. Lignin begin to decompose at temperatures 300-350°C and on 400-450°C (Girard, 1992).

Liquid smoke obtained from palm shells containing of organic acid compounds and phenol derivatives. Pyrolysis process has previously been performed by many researchers such as Darmadji, et al (2002), the pyrolysis process at various temperatures and concluded that temperature affects the resulting liquid smoke. Girrard, 1992 performed pyrolysis with various raw materials and compare the liquid smoke component.

Components contained in the liquid smoke has been separated by a variety of methods based on the polarity, acidity and volatility (Putnam et al 1999). Liquid smoke component analysis by distillation method has been done by Astuti, (2000). Destillation was a way of separating mixtures based on differences in their boiling points when steam is obtained from a mixture containing many components that are volatile and phenol content.

In this research, the pyrolysis of palm oil shell performed by air controlled to produce liquid smoke and analyzed by gas chromatography-mass spectroscopy and Fourier Transformed Infrared.

Materials And Methods

Materials

Palm oil shells used in this study was obtained from The Research Institute of Palm Oil Agency, Medan, Indonesia. Reactor used a temperature-regulated furnice from 500°C to 900°C. Designed box-shaped container with a size 50x30 cm, smoke conduit made of iron 2 mm diameter designed spiral placed into drums with plate size ± 1,1mm thick, 48cm high and 40cm in diameter. Liquid smoke product were analyzed to determine the content of phenol by Gas Chromatography-Mass Spectroscopy (GC-MS) brands 201D Shimadzu GCMS QP using Rastek column stabilizer R-DA injection 215°C, interfaces 215°C Helium gas carrier, flow rate of 0.3 mL/min and Fourier Transformed Infrared (FT-IR) GALAXY 5000.

Pyrolysis Process
Pyrolisis was performed in the furnice that covered by white cement with a temperature of 500°C, 700°C, and 900°C, without and with nitrogen gas inlet with velocity flow of 10 mL/sec. Palm oil shells dried for 3 days to reduced the moisture content, weighed about 10 kg and then put into a reactor/container and heated at a temperature of 500°C. The obtanined liquid smoke of palm oil shell cured for 7 days and then centrifuged at 3800 rpm for 60 minutes. The filtrate was distilled at a temperature of 125°C for 60 minutes. Redestilated liquid smoke analyzed by means of Gas Chromatography Mass spectra and Fourier Transformed Infrared to determine the levels of phenol content. Pyrolysis was repeated with temperature of 700°C and 900°C.

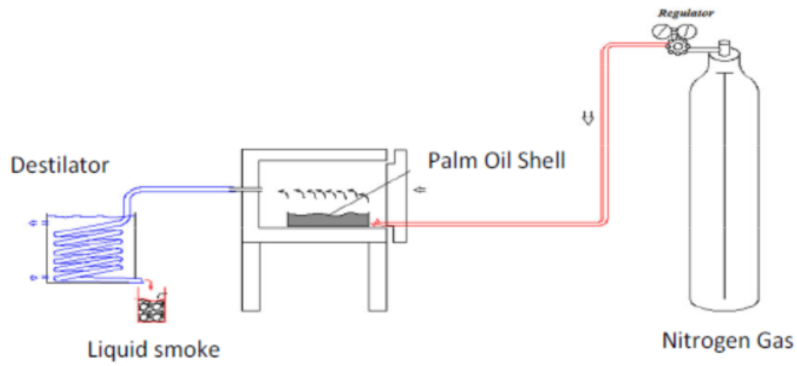
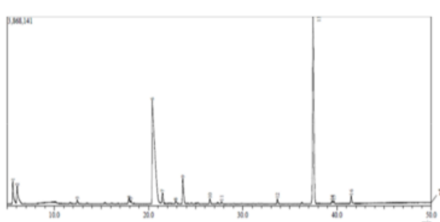


Figure 1. Reactor equipment and pyrolysis process

Characterizations by GC-MS
GC-MS instrument -QP2010 optimized at a temperature of 100°C for 4 minutes, then the temperature was increased to 200°C with the increase in retained 20°C for 2 minutes then increase the temperature to 300°C again with the increase in temperature of 20°C per minute and maintained for 16 minutes. The temperature on the ion source temperature set at 230°C injector set at 260°C. This analysis using pure helium gas which has a purity level of 99.99% with a gas pressure 62,7kPa. The amount of 1µl sample was injected into a gas chromatograph, analyzed from 50.00 to 500.00 molecular weight within 3 to 32 minutes.

Characterization by FT-IR
Liquid smoke characterized by placing the sample in the compartment. Then put on the tool towards the infrared rays. The results will be recorded on the wave number 4000 - 200 cm^{-1}. The recorded graph then analyzed by using OMNIC software.

Results and Discussions
Characterization by Gas Chromatography
The palm shell liquid smoke were characterized by a spectrophotometer Gas Chromatography-Mass Spectra (GC-MS). GC-MS chromatograms obtained from the analysis of the temperature of 500°C liquid smoke can be seen in Figure 2.

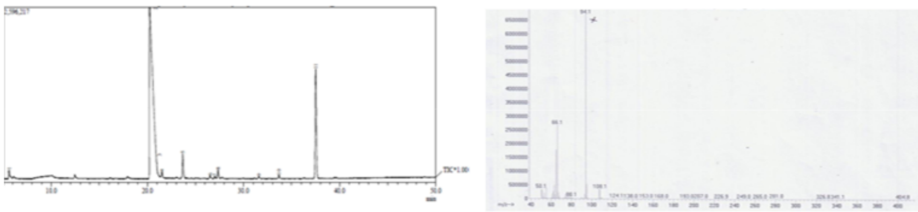


(i)

(ii)

Figure 2. Chromatogram gas chromatography of liquid smoke at temperature of 500°C, without nitrogen gas (i) and with nitrogen gas (ii)

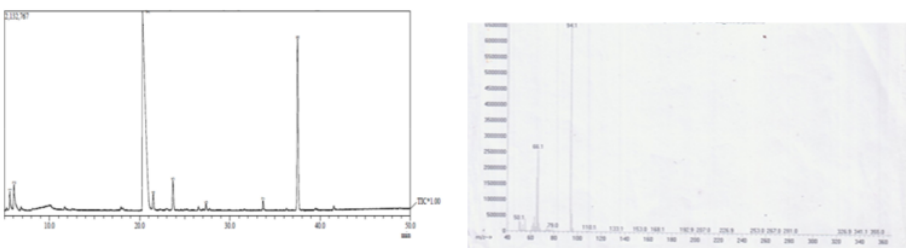
In figure 2 (i) it can be seen that amount of 16 compounds identified in the liquid smoke, the peak with a retention time of 37.49 min peak contained 35.09% with a peak of fragmentation 94. In figure 2 (ii) also showed identified 8 compounds. At a retention time of 10.21 minutes, 7.86 peak and peak fragmentation fragmentation 94. Peak 94 shows the phenol compound (Silverstein et al., 1991). Phenol has the largest peak area, which is in line with those reported by Soldera (2008) stated that the phenol and its derivatives is the largest component constituent liquid smoke is mainly derived from pyrolysis oil palm shells.



(i) (ii)

Figure 3. Chromatogram gas chromatography of liquid smoke at temperature of 700°C, without nitrogen gas (i) and with nitrogen gas (ii)

In figure 3 (i) it can be seen that the compounds identified as many as 9, the peak with a retention time of 37.48 min, 30.24% peak and peak fragmentation 94. In Figure 3 (ii) identified 8 compounds with retention time of 9.89 minutes, 9.25% peak and peak fragmentation fragmentation 94. peak 94 shows the phenol compound (Silverstein et al., 1991). This is consistent with the reported Guillen et al, (2001) phenol is the component with the highest proportion of 24.11% resulting from thermal degradation of cellulose and hemicellulose and is also present in commercial liquid smoke.



(i) (ii)

Figure 4. Chromatogram gas chromatography of liquid smoke at temperature of 900°C, without nitrogen gas (i) and with nitrogen gas (ii)

In Figure 4 (i) it can be seen that the compounds identified as many as 8, the peak with a retention time of 37.50 min, 21.77% peak and peak fragmentation 94. In Figure 3 (ii) identified eight compounds with a peak of 9.25% and peak fragmentation 94. Peak fragmentation 94 indicates phenol compounds (Silverstein et al., 1991). This is consistent with the reported Guillen et al, 1999 stated that the phenol and its derivatives is the largest component constituent liquid smoke. In pyrolysis with different methods and different temperatures can be concluded that the most dominant content is phenol. From the graph above can be described in terms of a comparison table of liquid smoke analysis results of the two methods of pyrolysis.

Table 1. Phenol Content of Liquid Smoke of Palm Oil Shells, without nitrogen gas (i) and with nitrogen gas (ii)

Temperature (oC)	Phenol, without Nitrogen gas (%)	Phenol, with Nitrogen gas (%)
500	35,09	9,86
700	27,92	9,25
900	14,49	22,28

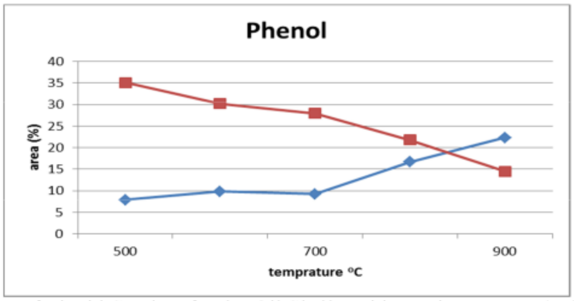


Figure 5. Phenol Content of Liquid Smoke of Palm Oil Shells, without nitrogen gas (red) and with nitrogen gas (blue)

Characterization by Fourier Transformed Infra-Red

GC data is supported by the data FTIR indicating phenol in liquid smoke palm shell, it can be seen from the graph; catchment area 3435.0 cm^{-1} probably indicate -OH group. at a temperature of 700°C catchment area of about 3400 cm^{-1} showed -OH group and a temperature of 900°C catchment area 3435 cm^{-1} probably indicate -OH group. Whereas the data controlled air liquid smoke FT-IR absorption area 3320.19 cm^{-1} indicates OH, the temperature of 700°C area uptake 3318.07 cm^{-1} indicates the presence of OH and temperature of 900°C-absorption area 3332.28 cm^{-1} shows OH.

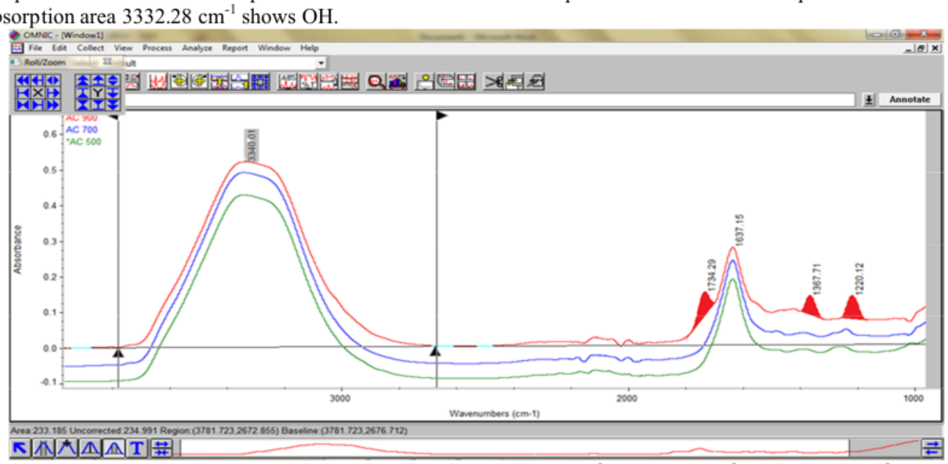


Figure 6. Overlay FT-IR spectra of Liquid Smike of Palm Oil at 500°C (green), 700°C (blue) and 900°C (red)

From Figure 6. can be explained that the liquid smoke (free air pyrolysis) optimum phenol formed in the pyrolysis temperature of 500°C and decreased with increasing pyrolysis temperature. In free air pyrolysis of lignin decomposition occurs at a temperature of 300°C-400°C-350°C and 450°C so that the maximum composition of phenol is at 500°C temperature, it is supported by previous investigators Girrard, (1992) which says that the lignin begins to decompose at a temperature range 300°C- 450°C.

Can also be explained that the phenol has a major wake formula of benzene rings bonded to a hydroxyl group. Benzene ring is really a lignin monomers, which generally have a cluster/ aromatics (which berbangun cyclic). Hydroxyl groups bonded to aromatic compounds (monomers lignin) donated from oxidation (burning) during the pyrolysis process. At a temperature rise of 500°C to 900°C. Broad peak area of phenol decreased due to the aromatic group bonded to be continuously oxidized as a result of increasing pyrolysis temperature.

While the method of controlled air pyrolysis higher pyrolysis temperature increased levels of phenols. This can be explained because lignin is difficult to decompose at lower temperatures and requires a long time so that even if the temperature pyrolysis at high research with nitrogen gas effects that are non-flammable and does not contribute to combustion (inert) resulted running slow pyrolysis process, nitrogen gas will binds oxygen present in the pyrolysis process so as to prevent premature oxidation. Inert nitrogen gas is a substance that is independent and not easily react with other elements or compounds, colorless, odorless and tasteless as well as the most important nitrogen stable in any condition so as not to increase the combustion and is not affected even at high temperature and other properties of nitrogen gas can expel other gases such as oxygen.

Yang, YB, et al, 2007 stating that the lignin decomposition takes a long and difficult terdekompisis at low temperature. Lignin is a component of palm shell macromolecules other than cellulose and hemicellulose. In hemicellulose will start easily decomposed at a temperature of 220°C-315°C, with a peak point at a temperature of 268°C, while the cellulose decomposes at a temperature of 350°C. Events decomposition of each component is different because of differences in the chemical structure of each of the components in the organic material.

Conclusions

Liquid smoke can be produced by pyrolysis of oil palm shell at a temperature of 300°C to 900°C. Liquid smoke produced in free air pyrolysis at temperature 500°C, 700°C, 900°C containing phenol 35.09%; 27.92% and 14.49%, respectively, and in the air controlled pyrolysis were 7.86%; 9.25%, and 22.28%, respectively.

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