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RESEARCH ARTICLE

EFFECT OF PYROLYSIS TEMPERATURE AND DISTILLATION ON CHARACTER OF COCONUT SHELL LIQUID SMOKE

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ABSTRACT

A research on effect of coconut shell pyrolysis temperature and liquid smoke distillation on character of resulted liquid smoke has been done. Objective of the research is to identify effect of coconut shell pyrolysis temperature and liquid smoke distillation on character of coconut shell liquid smoke distillate. Liquid smoke was obtained with pyrolysis using pyrolysis reactor on temperature range of 150 to 450°C. Liquid smoke purification was done by decantation, filtering and distillation at various temperatures: <100 (D-I), 100-120 (D-II), 121-140 (D-III), 141-160 (D-IV), 161-180 (D-V) and 181-200 (D-VI). Liquid smoke characteristic was done by determining physical and chemical characteristic such as density, bias index, pH, acid level and phenol level. The result indicated that temperature of coconut shell pyrolysis and liquid smoke distillation affect concentration and liquid smoke characteristic. Highest liquid smoke concentration was obtained in pyrolysis temperature of 150-200°C (6.07% (v/m)) and the lowest one (0.85% (v/m)) at temperature of 351-450°C. highest tar concentration (1.03% (v/m)) was obtained at 276-350°C, and the lowest (0.20% (v/m)) at 150-200°C. Charcoal concentration was 28.75% (m/m). Characteristic (pH, acid level and phenol level) of liquid smoke before and after purification process and distillation are various. Highest pH (6.2) was D-I, while the lowest (4.1) at D-VI. The highest acid level (58.40%) was at D-VI, while the lowest (1.86%) was at D-I. at D-I liquid smoke does not contain phenol, while the highest phenol content (3.85%) was in D-VI and the lowest was liquid smoke residue (1.93%).

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INTRODUCTION

Coconut shell is one of coconut parts and is by product of coconut processing. Coconut farmer in North Sulawesi use it as fuel in making copra and for cooking. Direct burning of coconut shell result in some that is dangerous waste and when it is not managed seriously it can air pollution. Coconut shell can be processed to be product having high economic value. Pyrolysis of coconut shell result in charcoal and smoke that can be condensed to be liquid smoke, tar and uncondensed gasses. Charcoal can be processed further to be briquette or active charcoal while liquid smoke can be used as food preservation agent substituting formalin and as antibacterial agent (Milly *et al.*, 2008; Toledo, 2007). Wood pyrolysis result in liquid smoke that contains various complex chemical compounds (Budianto *et al.*, 2008). Coconut shell belongs to hard wood group that containing three main components: cellulose, hemicelluloses, and lignin. Cellulose decomposition by heat results in anhydroglucose, carbonyl compound and furan. Decomposition of hemicellulose is similar to that of cellulose, but resulting in acetate acid and carbon dioxide.

Partial pyrolysis of lignin results in various phenolic compounds (Wei, 2010). Composition of liquid smoke is affected by raw material type, duration of burning and burning temperature. Liquid smoke is used commonly by agent providing aroma, texture and taste of food product, such as mead, fish and cheese (Milly *et al.*, 2008). In Indonesia, liquid smoke is used in smoked milkfish industry in Sidoarjo. Liquid smoke of coconut shell (LS-CS) is also used in laboratory scale (Hadiwiyoto *et al.*, 2000). In pyrolysis process, to obtain quantity and quality of good liquid smoke require research to look for operational condition to maximize concentration and quality of liquid smoke. Pyrolysis temperature is one of operating parameters to achieve the goal. In addition, to obtain liquid smoke free from tar it is necessary a separation process to obtain good and safe liquid smoke to apply for food product.

MATERIALS AND METHODS

Materials

Coconut shell from byproduct of copra making, liquid smoke, chemical material with grade pro analyst from Merck: NaOH, Na₂S₂O₃, pp indicator, ethanol and reagent Follin-Ciocalteu, zeolite, filter paper Wathman no.42

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Coconut shell pyrolysis

Washed coconut shell with its dimension is reduced was put into pyrolysis reactor and closed well. Reactor was heated in high temperature (150-450°C) with heating rate of 30°C per minutes. After reaching desired temperature, it was let 2 hours and smoke flow through evaporation pipe, cooled through cooling media. Then its liquid was discharged through pipe placed in container. After there is no liquid, the heating is stopped and reactor is cooled. Condensate and charcoal was weighted to determine concentration. Condensate concentration is mix of liquid smoke and tar calculated as % volume/weight and charcoal is calculated as % weight/weight

Purification and characterization of liquid smoke

Liquid resulted from pyrolysis is heterogeneous mix of liquid smoke and tar and still contain toxic component. The liquid is stored some days to allow tar and other unsolved compounds to separate or settle, then decanted, filtered with filter paper and zeolite and distilled/re-distilled.

Liquid smoke distillation (Darmadji, 2006)

Liquid smoke resulted from decantation and filtering (750 ml) was put into 1000 mL round-bottom flask then distilled fractionation using fractionation column of 60 cm equipped with condenser and heated using oil heater at 0-200°C. Distillate was taken in collector in various temperatures: <100 (D-I); 100-120 (D-II); 121-140 (D-III); 141-160 (D-IV); 161-180 (D-V); 180-200 (D-VI). Resulted distillates were characterized by determining physical and chemical characteristic.

Characterization of liquid smoke

Liquid smoke is characterized with standard method according to (LTP, 1994), which includes determination of specific mass, bias index, pH value, total acid analysis and phenol content

Determination of specific mass and bias index of liquid smoke

Measurement of specific mass of liquid smoke and tar was done using 10 ml pycnometer, while bias index was determined using refractometer. Procedure of specific mass determination is as follow. Cleaned and dried pycnometer was weighted carefully and its weight was recorded. Distilled water was put into pycnometer until calibration mark and weighted. Its weight was recorded. Distilled water is discharged, and then pycnometer is re dried. Then liquid smoke was put into calibration mark and its weight was recorded. Specific mass of liquid smoke was calculated using following equation:

$$\text{Specific mass} = \frac{(\text{sample mass} + \text{empty pycnometer mass}) - \text{empty pycnometer mass (gr)}}{(\text{water mass} - \text{empty pycnometer mass}) - \text{empty pycnometer mass (gr)}} \quad (\text{SNI 06-2388-1998})$$

pH value

pH value of resulted liquid smoke is determined using digital pH meter Metrohm type 692 pH/ion meter by dipping electrode into distilled water and wiped with tissue. Then, electrode is put into liquid smoke sample. pH value appearing in screen is recorded as pH value of liquid smoke.

Analysis of total acid (AOAC, 2005)

Liquid smoke 5 mL was added with 100 mL distilled water and shake until homogenous. Then 3 drips of pp indicator were added. Then it was titrated with NaOH 0.1 N until light red. Total acid measured is considered as acetate acid. Acetate acid content:

$$Y = \frac{\text{amount of ml titer} \times N \text{ NaOH} \times 60}{\text{sample volume (mL} \times 1000)} \times 100$$

Phenol content (Senter *et al.*, 1989)

Sample of 10 mL was placed in reaction tube containing 12 mL ethanol 95% and 5 mL distilled water. Then 0.5 mL reagent Follin-Ciocalteu was added into each tubes. They are let for 5 minutes and added 1 ml Na₂S₂O₃ 5% into sample, and shaken in Vortex Shaker, then stored in dark room for 60 minutes. Then, sample was shaken again using Vortex Shaker and its absorbance is measured at 725 nm wave length.

RESULTS AND DISCUSSION

Pyrolysis result

Coconut shell from byproduct of copra making has 6.16% water content. Pyrolysis product of 8 kg coconut shell in various temperatures is presented in table 1.

Table 1. Yield recovery of liquid smoke, tar and charcoal

No	Temperature (°C)	The yield of		
		Liquid smoke (%v/m)	Tar (%v/m)	charcoal (%m/m)
1	150-200	6,07	0,20	28,75
2	201-275	3,21	0,23	
3	276-350	4,58	1,03	
4	350-450	0,85	0,83	

In pyrolysis process, coconut shell is broken and put into pyrolysis reactor connected with smoke link pipe and equipped with tar collector, cooling and liquid smoke collector. Pyrolysis process of coconut shell occurred in pyrolysis reactor after heating in high temperature and took 5 hours. Pyrolysis product of coconut shell consisted of charcoal, tar, liquid smoke distillate and uncondensed gases. Pyrolysisate is created or drip at 150°C and at higher temperature from 200°C tar is formed. End of pyrolysis process is known from no distillate created and from discharged gases or not condensed gas. Charcoal product is carbon composing coconut shell solidified after volatile compound of coconut shell change to be liquid smoke and gas in pyrolysis process. Gas volume was obtained from law of mass conservation assumption, in which mass before reaction is same as mass after reaction (Soldera *et al.*, 2008). After let sometime the resulted Tar and liquid smoke would be separated by forming two layers due to its phase nature, polarity and different specific gravity; liquid phase was in upper layer and solid phase was in lower layer. Tar is non polar and very concentrated, while liquid smoke is polar because it can mix with water and contain non polar component solved within it. Liquid smoke of pyrolysis process has strong smell and contain smoke particle. According to (Girard, 1992; Maga, 1988) in temperature below 200°C is pyrolysis reaction that disappear water from wood, while above

400°C it is not decomposition of wood component to organic compounds but heating process and charcoal ripening. In addition, the temperature range is maximal burning temperature in liquid smoke making process.

Effect of temperature on liquid smoke and tar concentration

Concentration is one of important parameter to identify result of a process (Wijaya *et al.* 2008). Figure 1 indicate graphic of association between pyrolysis temperature and % liquid smoke and tar concentration resulted from pyrolysis process of coconut shell. The figure shows that highest liquid smoke volume is obtained at 150-250°C. In the temperature range, water discharge process occurs in coconut shell.

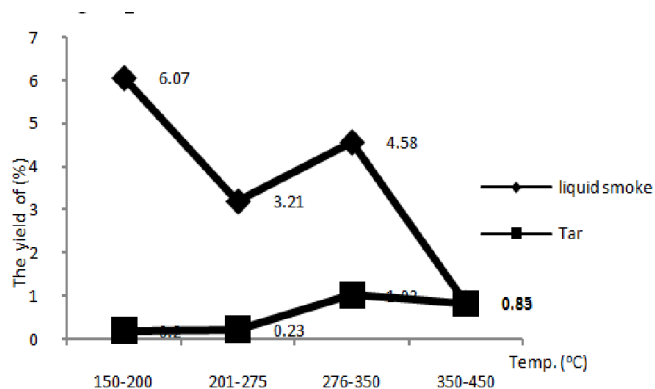


Figure 1. Graph relationships % yield volumes of liquid smoke and tar with pyrolysis temperature

At 201-275°C, it occur decrease in liquid smoke volume due to decrease in water in coconut shell accompanied with discharge of CO and making CO₂, and charcoal residue making. So, result of pyrolysis decrease. At temperature 276-350°C result of pyrolysis rise due to lignocellulose decomposition and lignin disproportionation reaction what still result in volatile compound with low molecule weight that is evaporated and condensed to be liquid smoke. Decrease in significant number of pyrolysis is indicated at 350-450°C. It is caused by component formed in pyrolysis process is degraded to result in CO, CO₂, H₂ (low and middle chain hydrocarbon) such as research by stating that higher pyrolysis temperature will decrease amount of organic monomer degradation product. Decrease in degradation product will be accompanied with increase in carbon dioxide and other gas (Wijaya *et al.*, 2008) Tar volume at 150-275°C is not relatively increase, while at 276-350°C increased and at 351-450°C it decreased again. It may due to addition of vapor product of low volatile compound due to temperature rise, so push of vapor faster to be liquid smoke and result of tar is fewer. Maximal tar volume occur at 276-350°C, because at the temperature range lignocellulose and lignin degradation process occur that resulted in anhydrate and tar. So, maximal tar volume is accompanied with increase in pyrolysis product. At 350-450°C, there occurred decrease in tar volume and also very significant decrease in pyrolysis product due to end of degradation process of coconut shell component. Total concentration of liquid smoke and tar produced in this research was 14.71% and 2.29%, respectively. Table 1 indicated that solid product of shell charcoal of 28.75 what show in coconut shell there is volatile component of 71.25% that change into pyrolysis, tar

and uncondensed gas. Total liquid smoke and tar concentration is 17% so there is 54.25% gas disappear or uncondensed. In this pyrolysis process, percentage of loss gas uncondensed is very high. According to (Tranggono, *et al.*, 1997), lost chemical component in pyrolysis process is volatile compound and cannot be condensed with water as cooler in form of gas such as CO₂, CO, H₂, CH_n and some hydrocarbon. Compared with some previous research, percentage of liquid smoke concentration in this research was far different. It is due to coconut shell used in this research is coconut shell from copra making remaining that have underwent baking process that has very low water content. In addition, the difference may be due to type of raw material, reactor shape and pyrolysis process. Concentration percentage also depends on condensation system and instrument used. In this research condensation system used to form liquid smoke use water flowed continuously as cooler media in order to make heat changing process can occur fast and temperature in cooler system did not increase. Liquid smoke resulted of coconut shell pyrolysis can be obtained maximally when its condensation process run perfectly.

Therefore, condenser length used also determine product of liquid smoke. Theoretically, longer condenser may make smoke resulted from imperfect burning in extraction process of smoke distillate will be more optimal (Wijaya, *et al.*, 2008). According to (Tranggono, *et al.*, 1997) liquid smoke concentration resulted from some woods varied from 39.15% to 61.55% with average value of 50.09%. Meanwhile charcoal product varied from 21.5% to 37.62% with average value of 31.54%. According to (Sari *et al.*, 2006), pyrolysis temperature affect concentration or percentage of pyrolysis resulted. Higher pyrolysis temperature causes higher distillate percentage but with lower charcoal product. However, too high pyrolysis temperature will decrease amount of organic monomer degradation product. Less charcoal product relates to decrease in organic component in coconut shell cell due to higher pyrolysis temperature. Increase in pyrolysis temperature also increase lost component. It is indicated at pyrolysis execution in which increase in smoke temperature discharged and uncondensed is greater. Based on the result, it may be concluded that liquid smoke concentration resulted in pyrolysis process depend on raw material type, pyrolysis reactor and pyrolysis temperature.

Effect of pyrolysis temperature on liquid smoke and tar specific mass

Specific mass is ratio of mass of a sample and its volume. Specific mass of liquid smoke does not relate directly to liquid smoke quality resulted, but can indicate amount of component within it. Determination of specific mass LS-CS and tar was done using pycnometer. Data of specific mass measurement indicated that change in pyrolysis temperature did not affect specific mass of liquid smoke and tar significantly. In this research, lowest result of analysis of liquid smoke specific mass was 1.022 gr/mL and the highest 1.042 gr/mL. Specific mass of liquid smoke resulted from pyrolysis increase with increase in temperature although its increase very small. Highest specific mass is liquid smoke obtained from temperature 350-450°C, while its lowest in 150-200°C. The lowest specific mass of tar is 1.086 and the highest one is 1.101 gr/mL. Specific mass of tar rise with increase in

pyrolysis temperature although its increase is very small as liquid smoke. Specific mass of liquid smoke in each temperature is relatively low than that off specific mass of tar, so increase in pyrolysis temperature did not give significant effect on specific mass of liquid smoke and tar.

Liquid smoke distillation

Liquid smoke distillation was done to remove dangerous compound and not desired such as polyaromatic hydrocarbon (PAH) and tar, with regulation of boil temperature to obtain clean liquid smoke, free of tar and benzopirene. The distillation process used Vigreux column with 60 cm length and 4 cm diameter. Use of Vigreux column is expected to separate component of liquid smoke more effective particularly carcinogenic component so purer liquid smoke may be obtained and its compound is isolated based on its characteristic. Data of volume concentration of distilled result of 750 mL LS-CS in certain temperature range is presented in graphic of association of % distillate concentration and distillate temperature as presented in figure 2. Liquid smoke distillation process for 187 minutes resulted in liquid smoke distillate with different time volume and distillate percentage. Data and graphic in figure 2 indicated that D-II has most volume percentage (71.3%) and longest process (125 minutes). While at D-I, the concentration is leas (1.6%) with distillation duration of 15 minutes. D-III to D-V has relative similar concentration (3.7% and 3.6%) with distillation duration of 10, 12, and 15 minutes. D-VI has concentration of 1.7% with 10 minutes distillation period. Remaining liquid smoke residue (un-distilled) was 5.3%. The data indicated that dominant D-II contain water while D-I is composed by volatile compounds having low boil point.

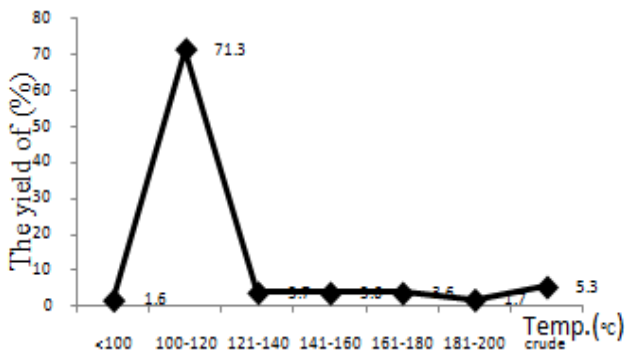


Figure 2. Graph relationships% yield of distillate volume of liquid smoke with distillation temperature

Characteristic of liquid smoke distillate

Liquid smoke quality

Liquid smoke quality in taste and aroma is determined by composition and compound contained within it because the component is made as criteria of liquid smoke quality. According (Gani, *et al.*, 2012) quality of liquid smoke was determined by its chemical compound because the compounds are criteria for taste and aroma as characteristic of some. In this research, test of liquid smoke quality was done with physical and chemical test. Physical characteristic observed include color, aroma, specific mass and bias index, while chemical characteristic include pH, acid content and phenol content.

Specific mass and bias index of liquid smoke distillate

Specific mass of liquid smoke distillate was determined using pycnometer, while bias index was determined using refractometer. Result of specific mass and bias index measurement indicated that distillate collected in various temperatures did not affect specific mass and bias index. Resulted specific mass and bias index of distillate and residue indicated not significantly different value. The lowest specific mass is LSBD (1.01238), while the highest specific mass is liquid smoke residue (1.10369). specific mass of D-I to D-VI are relatively similar (1.07 – 1.09). Bias index of LSBD and distillate indicate not significantly different value (1.344-1.357). The lowest bias index is D-I (1.344), while the highest is in liquid smoke residue (1.673).

pH, acid content and phenol content

pH value is one of parameters of liquid smoke quality and was determined by chemical composition. pH value of liquid smoke related to process level of decomposition of chemical component of coconut shell resulting in organic acid in liquid smoke. When liquid smoke has low pH, quality of liquid smoke resulted is very high because at whole in food preservation process it effect greatly preservation value and storage value of smoked process and its organoleptic characteristic (Wijaya, *et al.*, 2008). Result of pH measurement phenol contain and total acid content from liquid smoke of coconut shell pyrolysis before and after purification process and distillation are different. pH of liquid smoke before distillation (LSBD) was 4.82. After distillation at a certain temperature distillate yield with varying pH values. The highest Ph was in D-I (6.20) while D-II to D-VI and residue have no significantly different ph value (4.10 -4.63). First distillate has lowest acidity (highest pH). It is due to distilled compound in the temperature range is a volatile compound having low acidity level. D-I liquid smoke obtained at 100-120°C was predicted having high pH near seven due to the temperature is boil point of water so the distillate has water content, but based on measurement result it has pH 4.63. based on the data, the distillate may contain acid compound particularly acetate acid having boiling point of 118°C.

Table 2. pH, acid and phenol content of distillate and residual liquid smoke coconut shell

Distillate	pH	Acid Content (%)	Phenol Content (%)
D-I	6,20	1,86	-
D-II	4,63	9,60	2,25
D-III	4,40	29,30	2,42
D-IV	4,36	39,66	2,53
D-V	4,22	56,41	2,82
D-VI	4,10	58,40	3,85
Residue	4,12	48,86	1,93
LSBD	4,82	9,81	2,35

pH value of liquid smoke distillate is increasingly low along with increase in distillation temperature. It may be due to in the temperature water component increasingly less with increase of distilled organic acid. Presence of other compound having high acidity in each distillate is also predicted causing low pH or increase in acidity level of liquid smoke distillate. The lowest pH was D-VI and liquid smoke residue (4.10 and 4.12, respectively). Comparison with pH value of liquid smoke before distillation indicated pH of liquid smoke distillate

decreasing, except for D-I having pH 6.2. Acid content of LSBD compared with distillate and residue is relatively different. Acid content of D-II liquid smoke is almost same as LSBD. D-I LS-CS has the lowest acid content (1.86%), while acid content of D-III to D-VI increase with increase in distillation temperature. The difference is predicted due to temperature of D-I and DII, boiling point of acid compound has not been reached. At the temperature, distilled component is dominated by volatile compound having low acidity content. Result of acid content measurement indicated that higher distillation temperature reveal higher acid content. Increase in acid level is expected due to increase on acid component content along with characteristic of acids composing liquid smoke such as acetic acid having boiling point of 118°C, propionate acid (141°C) and butyrate acid (162°C) (Darmadji, 2006). Acidity data of liquid smoke distillate suit to pH data in which higher distillation temperature result in distillate with lower pH that means increase in acidity.

According to (Wijaya *et al.*, 2008), acid content is one of chemical characteristic determining quality of liquid smoke. Acid compound formed from wood burning process is organic acid compound resulted from pyrolysis process of wood components such as hemicellulose and cellulose in certain temperature. According to (Girard, 1992) some in liquid form also affect total acid of smoke condensate (40%) with 35 acid types. Acidity in some determine natural pH of smoke that have preservation characteristic, particularly in smoked product. Organic acid having important role in liquid smoke is acetic acid. Acetic acid is formed from pyrolysis process of lignin and part of pyrolysis of carbohydrate component, cellulose. Compared with result of research (Darmadji, 2006) there is not significant different between acid levels from distillate LS-CS purified with re-distillation except in D-I that is collected in temperature below 100°C. Acid content of liquid smoke distillate obtained in this research range from 1.86 to 58.40%, while research Darmadji indicated range of acid content from 8.68 -55.72%. In this research, first distillate acid content obtained in temperate <100°C (D-I) was 1.86% while research Darmadji indicated result of 12.34%.

The difference may be due to different distillation process condition and temperature of distillate storing range, where in this research D-I is stored in temperature below 100°C so only volatile components have been distilled, while in research Darmadji D-I is stored up to 100°C so it is possible much water and acid components distilled. Liquid smoke acidity is also influenced by wood type or raw material used. According to (Tranggono *et al.*, 1997) liquid smoke acidity of some woods varied from 4.27 to 11.39% with average value of 6.59%. According to (Darmadji, 2006) liquid smoke acidity of some woods varied from 4 to 29%. Research (Porter *et al.*, 1965) indicated presence of acids till 10 carbon chain but the most is format acid, acetic acid and propionate acid and butyrate acid. Different acid content in liquid smoke may be due to difference on chemical composition particularly cellulose. Data of phenol content indicates presence of different phenol content between distillate, residue and liquid smoke before distillation. Increase in temperature in distillation process cause increase in phenol content. The research indicates that D-I did not contain phenol. It may due to boiling point of phenol and its derivative is relatively high so it is not distilled below 100°C. Phenol compound begin

being detected at distillate collected at 100-120°C although its boiling point is high. It may be due to phenol form azeotrope with other compound such as water and organic acid so it is distilled. The highest phenol content in this research is D-V and D-VI collected in temperature range of 161-200°C (2.82 and 3.85%). It is due to the temperature range is boiling point of phenol compound and its derivative (18°C). Based on result of previous researches, phenol content in this research is not significantly different except phenol content in D-I. Darmadji (2006) reported that liquid smoke re-distillation at 100-200°C resulted in distillate with higher phenol content at 175-200°C (3.1%), while the lowest one is distillate collected below 100°C (1.14%). the result indicted that distilled collected below 100°C did not contain phenol. It is supported with result of identification with GC-MS analysis that did not indicate phenol compound in GC chromatogram or on MS spectrum data (Lombok *et al.*, 2012). According to (Arizona *et al.* 2011), phenol containing LS-CS was 2996.48 ppm or about 2.99%, while (Yu and Zun, 2006) stated that concentration and composition of phenol in liquid smoke varied between 1.10 to 5.13% depending on type and shape of wood as material of making liquid smoke.

Conclusion

1. Concentration of liquid smoke and tar resulted from pyrolysis process of coconut shell is greatly influenced by pyrolysis temperature. Highest liquid smoke concentration is obtained at 150-200°C (6/06%), while the lowest is obtained at 350-450°C (0.85%).
2. Distillation process using fractionation column can separate and purify composing component of liquid smoke of coconut shell. The highest liquid smoke distillate concentration is obtained at temperature range of 100-120°C (71.3%), while the lowest at temperature below 100°C (1.6%)
3. pH value, acid content, and phenol content of liquid smoke before and after purification are various. The highest pH is D-I liquid smoke, while the lowest is in D-VI liquid smoke. The highest acid level is D-VI (58.4%), while the lowest is D-I (1.86%). The highest phenol content is D-VI (3.85%), while the lowest is liquid smoke residue (1.93%). D-I liquid smoke did not contain phenol.

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