

# The Effect of Distillation Temperature in Liquid Smoke Purification Process: A Review

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## ABSTRACT

Liquid smoke is generated by condensing smoke from pyrolysis process of wooden material which contains lots of lignocellulose (lignin, cellulose, and hemicellulose). Liquid smoke is usually used as food preservation because of their antibacterial ability. There are two important compounds that make antibacterial properties of liquid smoke, i.e. phenolic and acid compound. In the other hand, liquid smoke from pyrolysis of wooden material also contains carcinogenic material like Polycyclic Aromatic Hydrocarbons (PAH). It must be purified in order to be used as food preservation safely. Fractional distillation is chosen as the separation process for liquid smoke purification to remove the PAH content. Fractional distillation occurs in the fractionation column with plates or trays. The variety of temperature in distillation will affect to the phenolic, acid compound, and PAH on the end product of liquid smoke. The objective of this study was to obtain the optimal temperature of distillation based on the phenolic and acid compound. Reviewing of the literature was chosen as the method to know the effect of variety of temperature in distillation for liquid smoke. used to determine the correlation of the literature.

**Keywords:** liquid smoke, phenolic, acid compound, PAH, temperature, distillation

## 1. INTRODUCTION

Liquid smoke is a suspension of solid and liquid particles in a gas medium obtained from biomass pyrolysis smoke condensation. The biomass which is being used in pyrolysis contains lots of lignocellulose (lignin, cellulose, and hemicellulose) [1]. Liquid smoke contains an antibacterial compound such as phenolic and acid compound [2] which is used as preservation for food, wood, etc. Phenol, one of component in phenolic compound is generated from lignin decomposition, acetic acid from the hemicellulose, and cellulose will be broken down into furan during the pyrolysis. [3].

Liquid smoke obtained from pyrolysis of wooden material also contains a Polycyclic Aromatic Hydrocarbons (PAH) constituent such as benzo[a]pyrene [4] which are carcinogenic and mutagenic for living things [5]. Using liquid smoke for food preservation require the certainty of PAH free. So that it must be purified before used. The purification method in this review was fractional distillation because it was easy and effective to separate the components by the boiling points [6]. The fractionating column aimed to be more effective to separate hazardous material from liquid smoke [7].

Fractional distillation occurs in the fractionating column that consists of plates or tray. In the fractionating column, liquid will be evaporated and contacted with condensed vapor from the higher plates. On each plate equilibrium between the liquid and the vapor is established, the vapor leaving the tray will be enriched with volatile components [8]. The equilibrium in each plate makes the composition of the boiling liquid and the distillate is constantly changing as the distillation proceeds. The exchange of material and heat is a physical process taking place when the condensed vapor and the vapor in contact, and plates placed one above the other make the surface area for exchange larger than simple distillation [9]. The desired product from fractional distillation is the first grade of liquid smoke that does not contain any hazardous components and safe to be consumed. It is usually used for food preservatives [10]. The following table is the quality standards for liquid smoke in Japan.

**Table 1.** Quality Standard for Liquid Smoke [11].

Parameter	Pyrolysis	Distillation
pH	1.5-3.7	1.5-3.7
Specific gravity	>1.005	>1.001
Acid Compound (%)	1-18	1-18
Color	Yellow	Colorless
	Pale Radish	Pale Yellow
	Brown Reddish brown	Pale Reddish Brown
color	Colorless	Colorless
Floating matters	No floating matters	No floating matters

## 2. METHODS

### *Identifying the Literature*

This study reviews the pyrolysis of wood material with fractional distillation as the purification process. The literature reviewed based on the liquid smoke with pyrolysis and distillation at the temperature range 100-

200°C. Besides the separation of PAH in liquid smoke, phenol and acetic acid content before and after distillation process become the parameters observed.

## 3. RESULT AND DISCUSSION

Distillation is a liquid smoke purification which generally aims to remove PAH and a portion of the tar that cannot be precipitated so that a clear yellow liquid smoke is obtained. In addition, this process will also affect to the component concentration of the liquid smoke. The main components in liquid smoke are water, a group of acidic compounds, a group of phenol compounds, and a group of carbonyl compounds. One of the factors that influence the distillation process and the characteristics of the resulting distillate is the operating temperature. Table 2 and 3 showed some studies about fractional distillation of liquid smoke with the phenol and acetic acid concentration. The effect of distillation temperature on liquid smoke purification is generally carried out at a temperature of 100-200°C with a variety of different temperature ranges.

**Table 2.** Relative Concentration of Phenol and Acetic Acid Before and After Distillation

Ref	Material	Temp (°C)	Phenol (%)		Acetic Acid (%)	
			Before Distillation	After Distillation	Before Distillation	After Distillation
[7]	Coconut Shell	<100	-	-	-	1,86
		100-120		2,25		9,6
		121-140		2,42		29,3
		141-160		2,53		39,66
		161-180		2,82		56,41
		180-200		3,85		58,4
[12]	Coconut Husk 300°C	<100	-	0,39	-	4,26
		100-125		0,65		8,19
		125-150		-		-
		150-200		-		-
	Coconut Husk 500°C	<100	-	0,37	-	4,15
		100-125		0,62		8,08
		125-150		-		-
		150-200		-		-
	Coconut Shell 300°C	<100	-	0,47	-	9,65
		100-125		0,59		18,75
		125-150		0,64		43,96
		150-200		0,78		59,93
	Coconut Shell 500°C	<100	-	0,44	-	9,58
		100-125		0,66		18,92
		125-150		0,54		44,24
		150-200		0,64		58,63
[14]	Sawdust	<100	-	-	2,6	4,2
		101-125				7,37
		126-150				10,58
		151-200				42,31

**Table 3.** Content of Phenolic and Acid Compound Before and After Distillation [15]

Temp (°C)	Condensor	Acetic Acid (%)		Phenol (%)		Carbonil (%)		Color	Aroma
		Before	After	Before	After	Before	After		
90-100	Pre	10,38	6,2	1,55	2,9	18,4	22	yellow +++	smoky +++
	Tk1		5,8		1,7		24	yellow +	smoky +++++
	Tk2		10,2		1,8		18	yellow +++++	smoky +++++
	Tk3		1		2,6		16	yellow +++++	smoky +++++
100-110	Pre		11,9		3,1		24	yellow +++	smoky +++++
	Tk1		3,9		2,7		18	colorless	smoky +++++
	Tk2		9,6		2,6		18	colorless	smoky +++++
	Tk3		8		2,4		23	colorless	smoky +++++
110-120	Pre		17,8		1		17	yellow +++++	smoky ++
	Tk1		13,4		2,4		16	colorless	smoky +++++
	Tk2		15		2,2		23	colorless	smoky +++++
	Tk3		13		2,4		24	colorless	smoky +++++

The studies in a lab-scale fractionation column increase the amount of the distillation equilibrium due to the limited tools. Generally, in the research conducted, fractionation was carried out by increasing the distillation operating temperature after no distillate was produced at a fractionation temperature. Muhammad. 2011 [15] made the research with modified tools so that the separation of fractions occurs in the column but this is rarely done. The fractionation column is modified with triple condenser to avoid the *carried over* of PAH in the distillate. The first condenser namely with TK1, second condenser with TK2, and TK 3 for the last condenser took place at the top of fractionation column. Yield is amount of the liquid smoke generated from pyrolysis and the purification process. The highest yield of liquid smoke produced in process with distillation temperature of 100-110°C. It showed that the liquid smoke from the pyrolysis have high water content because in that temperature range water start to evaporate.

Acetic acid is one of the compounds that determine the quality of liquid smoke which gives antibacterial properties. To obtain the optimal acetic acid yield of the liquid smoke, it is necessary to know the right distillation temperature by analyzing the previous studies of other researchers in table 2 and 3. Based on the analysis that has been carried out in each study regarding the purification of acetic acid, all of them show that the acetic acid concentration in the distillate will increase by increasing distillation temperature. The acetic acid is already detected at the distillation temperature of 100°C in small concentration. This is because the equilibrium of liquid acetic acid vapor with other components in the liquid

smoke causes acetic acid to evaporate and is found in the distillate. The highest concentration of acetic acid was obtained at the fractionation temperature of 200°C but the highest yield of liquid smoke was resulted in fractionation temperature of 100-110°C. It made the amount of acetic acid (volume of acetic acid in liquid smoke) in this temperature range was well produced. Fachraniah's study [14] produced a yield of 62.5% at 101-125°C, Lombok's [7] produced a yield of 71.3% at a temperature of 100-120°C, Rusdi's [13] produced a yield of 61.5 at a temperature of 91-130°C, and Muhammad's [15] produced a yield of 60% at a temperature of 100-110°C. From these results, increasing the distillation temperature will increase the acetic acid concentration although the yield's decrease. The boiling point of acetic acid is 118°C at a pressure of 1 atm. So that distillation at 100-130°C will obtain the acetic acid with high water content because at this temperature range not only the acetic acid evaporated but also the water.

The other important compounds in liquid smoke are phenols and their derivatives which have an antibacterial function and useful on the food preservation. Some study showed the highest phenol concentration was obtained from the higher fraction in distillate. The highest phenol concentration got 3.85% at distillation temperature range 180-200°C [7], and 0.78 at 150-200°C [12]. Their study is also showed that increasing the distillation temperature will increase the phenol concentration. The phenolic compound had the boiling point at 181.8°C [12].

Phenolic compounds and their derivatives have the same anti-bacterial function so that if a phenol derivative is obtained in the distillate like has the same anti-bacterial function as phenol. Purification of liquid smoke by distillation to be used as a food preservative must take into account the phenol concentration allowed in the food. According to the Food and Agriculture Organization of the United Nations (FAO) the composition of liquid smoke in food includes total carbonyl acids (ketones and aldehydes) and phenols, respectively (2- 20%), (2-25%) and (0.1-16%) [16]. The phenol concentration up to a fractionation temperature of 200°C is usually not more than 16%.

The setting the temperature range of fractional distillation of liquid smoke not only considers the concentration of compounds obtained, but also the concentration of hazardous substances such as tar. Tar concentration indication is the color of liquid smoke, the color of liquid smoke which does not contain tar is clear yellow. The clear yellow color was obtained at a fractionation temperature of 100-150°C [14], another research got the liquid smoke with an almost clear yellow color for condenser 1, 2, 3 at a temperature of 130°C, except for the pre condenser output because it was the bottom fraction of the column [15], while Rusdi's study [13] found clear yellow at the maximum fraction temperature of 111-130°C. Based on these data, the best temperature for tar separation and getting clear liquid smoke is at a temperature of 100-130°C at that temperature also produces the highest yield, and the highest amount of acetic acid and phenol derivatives.

#### 4. CONCLUSION

From previous study, it can be concluded that liquid smoke has high water content. It can be seen that in temperature 100-110°C has the highest yield. It is because most of the water are evaporated in that range of temperature. Thus, the higher yield is obtained. The best temperature for tar separation and getting clear liquid smoke is at a temperature of 100-130°C at that temperature also produces the highest yield, and the highest amount of acetic acid and phenol derivatives.

It can be concluded that the best distillation temperature to purify liquid smoke is in range 100-130°C.

#### ACKNOWLEDGMENT

This work was supported by Internal Research Funding of Politeknik Negeri Bandung in order to Applied Research No: B/ 186.64/ PL1.R7/ PG.00.03/ 2020.

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