

# Characteristics of Cinnamon Liquid Smoke Produced Using Several Purification Techniques

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## To cite this article

I. Ketut Budaraga, Arnim, Yetti Marlida, Usman Bulanin. Characteristics of Cinnamon Liquid Smoke Produced Using Several Purification Techniques. *American Journal of Food Science and Nutrition Research*. Vol. 3, No. 2, 2016, pp. 16-21.

**Received:** May 5, 2016; **Accepted:** May 14, 2016; **Published:** June 2, 2016

## Abstract

This research aims to discover the physical and chemical quality characteristics of liquid smoke obtained from liquid smoke from a number of different purification techniques. The research experiments used Complete Random Design with eight blocks repeated three times. The process used was the purification involving distillation at  $100\pm 10^{\circ}\text{C}$ ; at  $140\pm 10^{\circ}\text{C}$ ; using activated carbon, using a (50:50), mixture of activated carbon and zeolite, using decantation for a day, for two days and for three days. Changes that were observed in the physical characteristics of the liquid smoke included yield, specific density and colour. The chemical characteristics observed were water content, pH, total acid number, phenol content, carbonyl content and the content of benzo(a) pyrene. The results indicated that there was a very significant influence ( $P<0,01$ ) of the process on the quality of the liquid smoke. The best quality resulted from distillation at  $140 \pm 10^{\circ}\text{C}$  resulting in a specific gravity of 1,05 g/ml, yield of 88%, pH 2,36, total acid number titration 8,12%, phenol 0,22%, carbonyl 4,33% and no detectable benzo(a) pyrene. Based on these results it can be concluded that use of distillation at  $140 \pm 10^{\circ}\text{C}$  is the best purification process amongst those studied.

## Keywords

Purification Methods, Liquid Smoke, Physical Characteristics, Chemical Characteristics, Distillation

## 1. Introduction

Purification is a process that separates and removes foreign material from a desired product. There are several ways to do physical, mechanical and chemical process on their own or in combination. Purification is frequently used in food production or agricultural products. Purification of liquid smoke is generally carried out using precipitation filtration, adsorption, distillation, on their own or in combination [1].

Absorption is a purification method that utilizes sorbent. The general term in this process is called sorbsi covering adsorption and absorption. Adsorption is a process related to a solute on the surface of a solid, while absorption is the process where a solute diffuses into a solid with pores and is

bound to the surface inside the pores. The solid that absorbs the material is called a sorbent. A sorbent that is often used in the food industry is activated carbon and zeolite. The absorbent characteristics of activated carbon are due to its physical structure. Activated carbon is an amorphous form of carbon that consists mostly of free carbon containing an internal surface. The functional groups often found on the surface of activated carbon are carboxylate, hydroxyphenol, quinine, lactone and cyclic peroxide. Each type of activated carbon has different composition and activity [2].

According to Tahir [3] the pyrolysis process produces three classes of products; gas that is given off as carbonization occurs, chiefly  $\text{CO}_2$  and other flammable gases such as  $\text{CO}$ ,  $\text{CH}_4$ ,  $\text{H}_2$  and other short chain hydrocarbons. Distillates in the form of liquid smoke are primarily composed of floating products; methanol and acetic acid and

activated carbon. Liquid smoke according to Darmadji [4] consists of a liquid mixture of wood ash in water that is made by making the smoke from the pyrolysis of wood condensate. It contains a number of compounds that are formed from the pyrolysis of the three components of the wood; cellulose, hemicellulose, and lignin. More than 400 chemical compounds have been identified in wood smoke. Some of these components are acids that can influence the taste, pH, and shelf life of the smoked product. Wood smoke also contains carbonyls that react with proteins and produce browning and phenol which is a major source of aroma and demonstrates antioxidant activity. Fatimah [5] states that the composition of liquid smoke includes water (91-92%), phenol (0.2-2.9%), acid (2.8-9.5%), carbonyl (2.6-4.0%) and tar (1-7%). Liquid smoke that can be used with food has been distilled repeatedly and purified using adsorbent zeolite or activated carbon. Research about the purification of liquid smoke using an adsorbent has been conducted by Novita [6] using activated carbon. Zeolite is a member of a group of minerals formed by hydrothermal processes from alkaline igneous rocks by [7]. A zeolite has a structure characterized by canals and pores. Because of this porous structure the surface area is very large and can be made even larger by an activation process [8]. [9] studied some of the characteristics of a liquid smoke made from empty oil palm fruit bunches using zeolite as the adsorbent but did not examine the antibacterial properties of this product. This research aims to extend the information available about the characteristics of liquid smoke including its antibacterial.

The purpose of the study was to determine the physical and chemical properties of liquid smoke cinnamon performed purification of different ways.

## 2. Materials and Equipment

### 2.1. Research Materials

- a. Raw Materials: Liquid smoke produced from cinnamon at pyrolysis temperature 400°C, zeolite, activated carbon.
- b. Chemicals for analysing the chemical characteristics of liquid smoke. Methanol, helium gas, Whatman filter paper No 42 for analyzing chemical composition.

### 2.2. Research Equipment

Research equipment included scales, flasks, petri dishes, electric stove, filter paper, oven, autoclave, incubator, distillation apparatus, analytical balance, oven, porcelain dish, desiccator, filter, thermometer, pH meter, Erlenmeyer 125 ml and 500 ml, beaker, filter paper, Soxhlet, test tubes, centrifuge tubes, micro burette, pipette, volumetric pipette, 250 ml flask, centrifuge, spectrophotometer, Pyrometer, Equipment for determining phenol content included flasks, pipettes, Erlenmeyer, burettes and sample bottles.

### 2.3. Method

Complete randomized design was used with 8 purification methods and 3 repeats resulting in 24 trials. Purification

techniques used were (1) distillation at 100±10°C, (2) distillation at 140±10°C, (3) filtration using activated carbon, (4) filtration using a (50:50) mixture of activated carbon and zeolite, (5) filtration using zeolite, (6) precipitation then decantation after a day, (7) precipitation then decantation after two days and (8) precipitation then decantation after three days.

Data were analyzed by analysis of variance at the 5% level, if there was a significant difference data were further subjected to a Tukey test at the 5% level [10].

## 2.4. Implementation of Research

Liquid smoke from cinnamon was chosen for analysis as it produces the lowest level of the poison benzo(e) pyrene and results in a product that fulfills the standards established by [11]. It was produced using pyrolysis at 400±10°C. Purification was conducted using the following methods :

1. left for a week to allow precipitation of tar then distilled at 100 ±10°C for one hour
2. as above but at 140 ±10°C
3. filtered using absorption with activated carbon,
4. as above but with a (50:50) carbon/zeolite mixture
5. as above with zeolite
6. decanted after one day
7. decanted after two days
8. decanted after three days

#### a. Distillation

A 100 ml sample of the liquid smoke was placed in a distillation flask in a container of oil to transfer the heat then the apparatus was heated with an electric heating element. Once the oil (and also the liquid smoke) reached the desired temperature distillation was carried out to yield the whole fraction. The steam produced entered the condenser, and the distillate was collected in a flask. This process produced grade 2 liquid smoke. The physical and chemical characteristics were then determined and compared with the standards established by Japanese Wood Vinegar Association, [11].

#### b. Filtration

A 100 ml sample of the liquid smoke was mixed with 3.5% activated carbon [12] using a funnel then shaken and left to rest. The same process was used with the zeolite and the carbon zeolite mixture, after 15 minutes these were filtered through whatman no. 42 filter paper. As before physical and chemical characteristics were then determined.

#### c. Decantation

100ml samples of liquid smoke were measured into a glass, allowed to precipitate for 1, 2 and three days then decanted following the method of [13]. Parameters observed were:

1. Physical characteristics
  - a. Yield [14]
  - b. Specific gravity [15]
  - c. Colour (*Hunter Lab Color Flex EZ spectrophotometer*)
2. Chemical characteristics
  - a. Total acid number [16]
  - b. Phenol content [17], [18]
  - c. Carbonyl [17], [19]
  - d. pH [20]

### 3. Results

#### 3.1. Physical Characteristics

Table 1. Average values for each purification method.

Sample code	Purification method	Yield (%)	Specific gravity (ml/g)	Colour		
				L	a	b
1. (Distillation 100±10°C)		58.00±0.57 c	1.03±0.01 c	3.73±0.01 d	-0.27±0.01 cd	1.23±0.02 c
2. (Distillation 140±10°C)		88.00±0.87 b	1.05±0.01 abc	3.92±0.01 c	-0.31±0.0265 d	0.99±0.02 e
3. (Filtration AA)		90.00±0.89 b	1.04±0.01 bc	3.23±0.01 f	-0.14±0.0173 b	1.03±0.02 de
4. (Filtration AA+Z)		98.00±0.97 a	1.0567±0.0153 abc	3.90±0.01 c	-0.1967±0.0153 b	1.03±0.02 de
5. (Filtration Zeolite)		96.00±0.95 a	1.04±0.021 bc	4.74±0.021 b	0.43±0.02 a	2.81±0.02 a
6. (Decantation 1 day)		98.00±0.97 a	1.0767±0.0153 a	5.01±0.021 a	-0.25±0.0173 bc	2.11±0.02 b
7. (Decantation 2 days)		98.00±0.97 a	1.0767±0.0153 a	2.92±0.01 g	-0.24±0.0265 bc	1.08±0.02 d
6. (Decantation 3 days)		98.00±0.97 a	1.07±0.01 ab	3.51±0.01 e	-0.28±0.02 cd	1.22±0.02 c
Japan Standard **)			>1,001	Brownish yellow and transparent		

Key:

\* Different superscripts indicate statistically significantly different values (P<0,05)

\*\* from Yatagai (2002)

#### 3.2. Chemical Characteristics

Table 2. Average values for each chemical characteristic after each purification method.

Sample code	Total acid number	Phenol content(%)	Carbonyl (%)	Benzoe-pyrene (ppb)	pH
1. (Distillation 100±10°C)	4.38±0.04 f	0.20±0.10 d	3.9567±0.1686a	ND***	2.51±0.02 a
2. (Distillation 140±10°C)	8.17±0.08 b	0.22±0.01 d	4.33±0.1836 bc	ND	2.32±0.02 c
3. (Filtration AA)	7.09±0.07 c	0.50±0.01 c	4.56±0.1916 ab	ND	2.44±0.02 b
4. (Filtration AA+Z)	6.85±0.07 d	0.09±0.02 e	4.56±0.1916 ab	ND	2.4±0.020 b
5. (Filtration Zeolite)	7.27±0.07 c	0.47±0.021 c	4.75±0.2007 ab	ND	2.44±0.02 b
6. (Decantation 1 day)	8.47±0.08 a	2.49±0.02 a	4.76±0.2007 ab	ND	2.3±0.023 c
7. (Decantation 2 days)	5.65±0.06 e	2.30±0.02 b	4.88±0.2088 ab	ND	2.31±0.02 c
6. (Decantation 3 days)	5.55±0.05 e	2.25±0.02 b	4.9767±0.2122 a	ND	2.30±0.02 c
Japan Standard **)	1-18				1,5 - 3,7

Key:

\* Different superscripts indicate statistically significantly different values (P<0,05)

\*\* from Yatagai (2002)

\*\*\* ND = No Detected

### 4. Discussion

#### 4.1. Physical Characteristics

##### 4.1.1. Yield (%)

Average yield ranged between 58% and 98%. The size of the variance indicated that the purification method has a significant influence on yield. (P<0.05). Subsequent Tukey test at 5% in Table 1 further confirms that purification method influences yield. Filtration with a mixture of activated carbon and zeolite produces an average yield of 98%. Distillation at 100°C produces the lowest average yield of (58%). This is due to the evaporation and escape of many of the compounds in the liquid smoke with the steam during the distillation process.

The use of (4) the activated carbon zeolite mixture produced the highest yield at (98%) which was not statistically different from using zeolite (5), decantation after one day (6), decantation after two days (7), or three days (8). However there was a statistical difference with the yield from activated carbon filtration and distillation at 140±10°C

(2) and 100± 10°C (1). The value of the yield of liquid smoke is influenced by losses due to evaporation during distillation at 140±10°C (2) and 100± 10°C (1) and absorption during filtration particularly when active carbon is used. The losses during the distillation process reflect the results of [21]. They found it important to use a continuous flow of cold water to keep the distillate at a constant temperature and reduce evaporation loss to optimize yield when producing liquid smoke.

##### 4.1.2. Specific Gravity

Specific gravity is the comparison between the weight of a certain volume of a substance compared to that same volume of water at a particular temperature and depends on the components that make up the liquid smoke. A large number of molecules with high molecular weight in the liquid smoke will result in a high specific gravity. The specific gravity of cinnamon liquid smoke ranged from 1.03 to 1.07. The value of the variance of these values indicates that purification method has a significant influence on specific gravity (P<0.01). A subsequent Tukey test at 5% further confirms that purification

method influences specific gravity. Decantation for a day gave the highest specific gravity of (1.07) which was significantly different from values for samples filtered through activated carbon or zeolite and distillation at 100°C. This last method resulted in the lowest specific gravity of 1.03.

#### 4.1.3. Colour (L,a,b) Hunter Lab Color Flex System

The colour of each liquid smoke sample was measured using a *Hunter Lab Color Flex EZ* tool to produce values for the parameters L, a\*(-), and b\*(+). Parameter L (Lightness) indicates degree of clarity, a\*(-) greenness and b\*(+) yellowness. The L value of the liquid smoke had values ranging between (+) 3.51 - 3.73. The parameter a\* ranged from (-) 0.27 to (-) 0.28 and b\* from (+) 0.99 to 2.81 after conversion with reference to the scale the colour of the liquid smoke could be classified as a greenish yellow-brown. The lightness was not particularly high due to the presence of phenol and carbonyl. The brownish yellow colour could also be due to these two compounds and possibly enzymatic browning during production. Enzymatic browning can be overcome by using opaque storage bottles to reduce oxidation.

The value of the variance of these values indicates that purification method has a significant influence on colour (L,a,b) This was further confirmed with the Tukey test at the 0,05 level. Liquid smoke decanted after one day is significantly lighter in colour than other purification methods. Liquid smoke filtered through zeolite was the reddest (least green) and there is a statistically significant difference between this method and the others. Filtration using zeolite results in the highest values for (b) and results in a reddish brown colour that is statistically different from samples purified using other methods.

### 4.2. Chemical Characteristics

#### 4.2.1. Acid Total Number

Total acid number is the amount of free acid in a liquid. These compounds are formed from the degradation of esters by water. In liquid smoke, the acid is a catalyst that speeds the breakdown of esters into acid and alcohol. It also causes oxidation of the alcohols into aldehydes and ketones. Total acid number ranged between 4.38 and 8.47.

The value of the variance of these values indicates that purification method has a significant influence on total acid number of liquid smoke ( $P < 0.05$ ). This may be because each process results in a different composition of free acid. Average values of total acid number for each process (Table 2) indicate that decantation after one day produces the liquid smoke with the highest total acid number at (8.47). Distillation at 100 ±10°C produces the lowest value (4.38) possibly because of losses due to evaporation due to heating.

#### 4.2.2. Phenol Content (%)

Phenols are aromatic ring compounds that are produced by plants containing one or two hydroxyl groups. Flavonoids make up a large number of these along with simple monocyclic phenols, phenylpropanoid and phenolic quinine.

Aromatic groups on phenolic compounds are often strong UV absorbers. Phenol compounds tend to be highly soluble in water because they are often bonded to glycosides that are found in the cell vacuole [22].

The test for total phenol uses Folin-Ciocalteu solution and gallic acid as a comparison [23]. The Folin-Ciocalteu method involves a reaction with this reagent and involves oxidation of the phenolic group (ROH) with a mixture of phosphotungstic acid and molybdate acid in the reagent forming a quinoid (R=O). This Folin-Ciocalteu produces a blue color according to the levels of total phenols react. Furthermore, this color is calculated intensity at a wavelength of 765 nm. Gallic acid can be used as a standard as it is a polyphenolic compound that occurs in almost all plant material and contains a pure and stable organic phenol acid.

The results of the phenol content measurements (Table 2) indicate that purification method has a significant influence ( $\alpha = 0,1$ ) on phenol content with a range of 0.09% - 2.49%. Filtration with decantation after one day gave a significantly ( $\alpha = 0,05$ ) higher phenol content than other methods. Total phenol content is not related to water content of the raw materials or the yield. However, yield does depend on the characteristics of the raw materials and the temperature reached during purification as was discovered by [24]. [25] by stating that evaporation, decomposition or breakdown of the chemical components of the wood during pyrolysis is gradual, i.e. at a temperature of 100-150°C only water evaporates, degradation of hemicellulose at 140°C. Cellulose and lignin breakdown need much higher temperatures to breakdown.

Phenol is a colorless crystalline substance with colour varying from colourless to bright pink and has a sharp and distinctive odor. The largest group of phenols are flavonoids and tannins. Phenol compounds have antibacterial properties, interacting with bacterial cells through an absorption process involving hydrogen bonds and so hindering the activity in the cytoplasmic membrane [22]. Phenol content in liquid smoke that was obtained during this research was smaller than that obtained by [26] who used pyrolysis on a variety of woods at 350-400°C and obtained an average phenol content of 2.90%. [27] obtained 1.28% phenol content from liquid smoke made from coconut shells and [28] 3.24% from tusam wood. Highest phenol content was obtained by [29], at 2.10 -5.13%.

Decantation after one day results in the highest values for phenol content and this is statistically different from samples purified using other methods. This higher value is thought to be related to the lack of loss of phenol during precipitation.

#### 4.2.3. Carbonyl Content (%)

The carbonyl in liquid smoke is a product of the pyrolysis of cellulose. The value for carbonyl is higher than that for phenol because phenol molecules that contain carbonyl groups are measured as carbonyl also. Carbonyl contained in coconut shell liquid smoke was measured to be 13.28% by [26] which is much higher than the 4.97% measured for cinnamon liquid smoke in this study. Carbonyl content results are displayed in Table 2. The carbonyl content is significantly influenced by purification method ( $P < 0.01$ ). Based on advanced test Tukey 5 percent (Table

2) shows that the purification decantation for 3 days gives the results of the highest levels of carbonyl (4.97%) is not significant different by distillation temperature of 140°C and 100°C distillation. The result of lowest carbonyl levels in the liquid smoke purification using distillation temperature of 100°C (3.95%) is significantly different by filtration and decantation.

These values are relatively small compared to liquid smoke made from coconut shells (9.3%) or cacao shells (11.32%), coffee husks (12.26%), oil palm shells (12.48%) and clove leaves (36.95%) but similar to oil palm fiber (3.06%). [30]; [4]; [31]; [32].

Decantation after three day results in the highest values for carbonyl content and is statistically different from distillation at 140°C (table 2). Carbonyl is a polar group hence molecules containing carbonyl groups such as aldehydes and ketones have a higher boiling point than hydrocarbons similar molecular weight. Even so, as aldehydes and ketones are unable to form strong intermolecular hydrogen bonds they have a lower boiling point than alcohols with similar molecular weight [33]. A ketone contains two alkyl groups that are bonded to the carbonyl carbon. Other groups in an aldehyde can take the form of alkyl, aryl or H. Aldehyde and ketones are frequently found in living cells. A reaction using the reagent 2,4-dinitrophenylhydrazine is usually used to identify the presence of aldehyde and ketone groups. If these are present a precipitate 2,4-dinitrophenylhydrazone is formed.

#### 4.2.4. Benzo(a)Pyrene (ppb) Content

Benzo(a) pyrene is pro-carcinogen. The enzymatic metabolism of this compound produces the mutagen benzo(a) pyrenediol-epoxide. Benzo(a) pyrene and benzo[a] anthracene are formed from incomplete combustion of carbon containing materials such as wood. This can be because of the type of wood or the burning process or the amount that is used. The presence of this carcinogenic compound in liquid smoke presents a health hazard.

No benzo(a) pyrene was observed in any of the purified samples. If the liquid smoke is made from dry cinnamon less potentially carcinogenic PAH such as benzo(a) pyrene are formed. It appears that all purification processes studied are effective in removing benzo(a) pyrene from liquid smoke making it safe for use in food processing and as an alternative preservative as the amount is below the maximum limit set by Indonesian National Standards of 0.2–10 ppb [34] and also below the FAO and WHO guidelines of 10 µg/kg for food [35] and the Turki Food Codex which sets 1 µg/kg and the European Commission standard of 5 µg/kg [36].

#### 4.2.5. Acidity (pH)

Acidity is a measure of liquid smoke quality. The size of the variance indicated that the purification method has a significant influence on pH. ( $P < 0.01$ ). Application of the tukey test at the 0.05 level (table 2) indicates that purification method tends to have a significant influence on pH. Distillation at 100±10°C produced the highest pH (2.51) which was significantly different from other purification methods. Decantation after 3 days produces liquid smoke with the lowest pH (2.30) which was not significantly different

from decantation after one or two days or distillation at 140°C±10°C or filtration methods. Purification tends to reduce then pH. The acidic nature of liquid smoke is caused by the breakdown of chemical components during each purification method. The low overall pH contributes to the value of liquid smoke as a preservative and the shelf life of the product and organoleptic properties.

## 5. Conclusion

The best way to purify cinnamon liquid smoke is distillation at 140±10°C because of all the methods trialed it produces the best combination of physical and chemical properties. It has a high yield (88%), a low total acid number of 4.38, a low phenol content of 0.20%, and a good carbonyl level of 3.96%. The colour of the liquid smoke produced was transparent enough to fulfill the Japanese wood vinegar quality standards [11].

## Acknowledgements

The writer would like to say a very great thank to the ministry of research of technology of higher education which has given very valuable horizons to this paper. We also would like to impress our great thank to the Rector of Ekasakti university which has given a big support so that the paper can be finished.

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