International Journal of ChemTech Research
CODEN (USA): IJCRGG, ISSN: 0974-4290, ISSN(Online):2455-9555
Vol.10 No.1 pp 12-26, 2017

# Chemical Components Analysis of Cinnamon Liquid Smoke with GC MS from Various Production of different Purification Method

I Ketut Budaraga[1]*, Arnim[2], Yetti Marlida[2] and Usman Bulanin[3]

[1]Agricultural Technology Department, Faculty of Agriculture, Ekasakti University, Veteran Dalam street 21b Padang city West Sumatera Indonesia,
[2] Animal Production Department, Faculty of Animal Husbandry, Andalas University, LimauManis street Padang city West Sumatra, Indonesia,
[3] Fisheries Cultivation Department, Faculty of Fisheries, Bung Hatta University, Sumatera street Padang city West Sumatra Indonesia

**Abstract :** This study aims to determine the chemical components of cinnamon liquid smoke obtained from different purification method. This research is performed experimentally by using GC MS on the cinnamon liquid smoke by purification on 100°C distillation temperature, 140°C distillation temperature, filtration purification by using active charcoal, active charcoal mixture with zeolite (50%:50%), purification by precipitation for 1,2 and 3 days) .The observed parameter is the chemical components of cinnamon liquid smoke from different purification method. The result of research shows that purification method treatment affects component structure of liquid smoke. Chemical component of liquid smoke with the biggest retention value obtained in cinnamon raw material treatment on purification method by distillation at 140°C temperature with chemical components of GC MS is phenol 2,6-Dimethoxy with retention value (RT) at 28.49, then the filtration treatment on purification treatment by using active charcoal is the compound of 2,4-Dimethyl-3-(Methoxycarbonil)-5 2,3,5-Trimetosytoluene Hydroquinone, Mono-TMS with retention value (RT) at 32.90, and decantation treatment for one day is Phenol,2,6-Dimethoxy with retention value (RT) 24,90. Based on this research, it can be concluded that the usage of cinnamon by purification method on distillation temperature of 140°C is better than the other purification method because it has better color result.
**Keywords:** chemical component; purification method; liquid smoke; cinnamon,GC-MS.

## Introduction

Liquid smoke is the condensation result of wood pyrolysis that contains lots of compounds formed by constituent pyrolysis process such as cellulose, hemicellulose and lignin that will produce organic acids, phenols, carbonyl that become the compound in food preservation. Those compounds have different proportions such as type, wood moisture and pyrolysis temperature[1].

Cinnamon is classified as hardwood because the lignin level is sufficient and the availability in West Sumatra province is quite large. Production data in 2014 has amounted to 107.3 thousand tons [10]. One of the results of pyrolysis is phenol. Phenol is the compound that has disinfectant and antiseptic characteristics, which is effective against bacteria vegetative forms of gram positive and gram negative, mycobacteria, fungi and virus are not effective against spore form. In the form of a solution on 1% phenol concentration has function as bacteriostatic while at higher concentrations it acts as bacterosidol, phenol at 0.5-1% concentration can be used as local anaesthetic and can be injected up to 10ml on the tissue as analgesic[2]. Asha et al.,[35] reported was observed that *A.niger*was most sensitive with ethyl acetate and acetoneextracts; *C.tropicalis*with n-hexane and methanol extracts; *S. cerevisiae*with ethanol extract and *C.albicans*with cool water and warm water extracts of *Cinnamon.*

Lignin is a complex polymer that has high molecular weight composed of phenyl propane units. The compounds derived from pyrolysis on lignin basic structure play important role in providing smoke scents to the products. This compound is phenol, phenol ethers such as *guaiakol*, *siringol* and *homologues* and its derivatives. Lignin begins to decompose at temperatures 300-350°C and on 400-450°C. The chemical components in the smoke is important in determining the quality of smoked products because besides from forming the flavor, texture and typical smoked color, it can also inhibit damage to the product[3].

All types of wood distillate contains compounds that can be extracted such as phenol derivatives which can inhibit microbes' growth. Liquid smoke from wood can be used as preservative because of the similarity of wood distillate chemical components contained in the certain kinds of preservatives, which are phenol and its derivatives. The problem in cinnamon liquid smoke result on pyrolysis temperature of 400°C, it still contains toxic compounds of *benzo(a)pyrene* compound of 0.04 ppm[4], so it needs to be purified from toxic and safe to be used as a preservative. The results of the study [5] states that benzo(a)pyrene concentration can be lowered by redistilled and adsorption by using active charcoal. Concentration that is reduced by redistilled technique is 33.1%, while the adsorption technique by using active charcoal is 54.3%.

According to [6], the decrease of *benzo(a)pyrene* levels on liquid smoke that is adsorbed by zeolite is different depending on the activation, the decline of natural zeolite is 26.41%, active zeolite 79.09%, chloride zeolite 49.06%, ammonium zeolite 39.26% and fluoride zeolite 44.28%. Based on this result, the decrease in *benzo(a)pyrene* by means of adsorption with active charcoal is very good compared by redistillation, however from the activity its antibacteria, then the liquid smoke that provides better activity is redistilled liquid smoke. The results of the study[7,8,9], indicate that liquid smoke made by the pyrolysis process still contains carcinogenic components, which is benzo(a)pyrene at various level depending on pyrolysis temperature.

Based on the potential of cinnamon, that the people usually only use cinnamon as firewood after the skin has been taken, it is necessary to provide added-value by processing cinnamon into liquid smoke. On the other hand, the produced liquid smoke still contains toxic that it needs to be purified. Based on the above problems, it is necessary to study the constituent chemical components by using GC MS from several different purification methods. The purpose of this study is to determine the liquid smoke chemical components by using GC MS from different purification method treatment.

## Experimental Section

### Materials

1. Raw materials: Cinnamon liquid smoke raw material on pyrolysis temperature 400°C as in (figure 1), zeolites, active charcoal.

**Figure 1.Cinnamon liquid smoke on pyrolysis temperature of 400°C**

2. Chemical material: The materials used in analyzing liquid smoke chemical properties. In analyzing chemical components of liquid smoke by using methanol, helium gas, Whatman filter paper No. 42

**Instrumentation**

The tools used in this study is GC MS, Shimadzu type QP 2010, by using operational conditions as follows: Gas: Helium, Column: RTX - 1MS, Stationary phase: Polymetilsiloxant, Temperature: Injector: 280°C, Oven: 80°C - 280°C, 20°C/minute, Interface: 300$^o$C, Control modes: Split, Column flow: 1:14 ml/minute, Split ratio: 200, ionization mode: Electron Impact, Solvent delay: 3 minutes, M/Z: 45.00 - 350.00, microliter syringes, syringe filters and sample bottles.

**Procedure**

This research uses complete random design (RAL) experiment with 8 purification treatments that are repeated three times to obtain 24 experimental units that include :

1. Purification by distillation of 100 ± 10°C,
2. Purification by distillation temperature of 140 ± 10°C,
3. Purification by active charcoal (AA) filtration,
4. Purification by active charcoal (AA) and zeolite (Z) filtration with ratio of 50:50,
5. Filtration by using zeolite (Z),
6. Purification by sedimentation/decantation for 1 day,
7. Purification by precipitation for 2 days, (8) purification by precipitation for 3 days.

Liquid smoke purification is performed on *cinnamon* raw materials with pyrolysis temperature of 400 ± 10°C because it meets the standard issued by [10]and the toxic content of *benzo(a)pyren* is lower than the other two raw materials. Purification activity is performed on cinnamon liquid smoke at 400$^o$C pyrolysis for 1 week to precipitate the tar, after 1 week, it is followed by administration of the purification treatment by distillation method at temperature of 100 ± 10°C and 140 ± 10°C for 1 hour, filtering (absorption) by using active charcoal, active charcoal mixture with zeolite (50:50) and zeolite and precipitation for 1,2 and 3 days.

**Distillation**

In the process of distillation: cinnamon liquid smoke sample from result of pyrolysis at 400$^o$C temperature of 100 ml is put in distillation flask where the container uses lubricating oil as good heat conductor and keeps being heated by using electric heater. The distillation process is performed when the temperature of heating medium (lubricating oil) has shown the desired temperature according to treatment of 100$^o$C and 140°C. The purpose of distillation is to take all of the factions and is set at 100°C and 140°C temperatures. At each temperature treatment is performed three repetitions. The temperature that is shown is the temperature of liquid smoke in distillation flask. The steam is formed and then it enters the coolant pipe (*condenser*) and the distillate is collected in a flask. In this purification process, it obtains liquid smoke quality II. The result of liquid smoke purification is then analyzed its chemical components by using GC-MS.

**Filtering (*adsorption*) by using active charcoal, mixture of AA+ zeolite and zeolite**

Cinnamon liquid smoke as the result of pyrolysis at temperature of 400°C of 100 ml is mixed with active carbon of 3.5% [11], it is conducted by using a funnel and is further shaken and put aside for 15 minutes. The same activities is performed on zeolite materials and the mixture of both is ready to be used, after putting it aside for 15 minutes, then it is filtered through Whatman filter paper No. 42. The result of purification that has been performed is further analyzed its chemical components by using GC_MS.

**Precipitation**

Cinnamon liquid smoke is prepared in a measuring cup of 100 ml each, then it is deposited/decanted for 1, 2 and 3 days with three repetitions. This treatment refers to the results of study [12]. Furthermore, the analysis of the chemical components is performed by using GC MS.

### Chemical Component Analysis (Gas)

Liquid smoke gas component analysis (*aromatic compound*) uses variety of methods [5,7,10]. Measurement uses GC MS Shimadzu type QP 2010, with operational conditions as follows: Gas: Helium, Column: RTX-1MS, Stationary phase: Polymetilsiloxant, Temperature: Injector: 280°C, Oven: 80°C - 280°C, 20oC/minute, Interface: 300oC, Control modes: Split, Column flow: 1:14 ml/minute, Split ratio: 200, Ionization mode: Electron Impact, Solvent delay: 3 minutes, M/Z: 45.00 - 350.00.

Analysis method by using GC MS is performed on liquid smoke cinnamon result of pyrolysis at temperatures of 400$^{o}$C with 8 different ways purification. Previously the samples are prepared, which are: liquid smoke to be analyzed is deposited for one week then iit is filtered using Whatman filter paper No.42. Filtration is repeated again by using microliter syringes and syringe filters. The filtration result is diluted with methanol up to 1000 times, then checking the operating conditions of GC MS QP2010, namely: Operating conditions of GC MS QP2010 when used for analysis are as follow: oven temperature 75$^{o}$C maintained for 5 minutes, then increased to 130°C with the rate of temperature increase at 8°C/min and then maintained for 3 minutes, then it is increased again to 290°C with temperature increase speed at 10°C/minute maintained for 3 minutes, and then it is increased once again to temperature of 300°C for 24 minutes. Ion source temperature is 200$^{o}$C. Helium that is used in this analysis has the purity of 99.999%. Gas pressure is regulated to 75.0 kPa and gas flow rate at 0.57 ml/min, while the injector temperature at 250$^{o}$C. When the condition of GC MS is ready to be operationalized then it is inserted into sample tube and then injected into GC MS QP2010 with the procedure of liquid smoke sample of 0.2 µl is injected in GC MS (tool condition has already been programmed) with specification: column that is used is capillary column GL science TC 17 (0.25 mm x 30 m, helium carrier gas, the initial temperature is 50°C (hold for 5 minutes), the final temperature is 250°C with the rate of temperature rise at 4°C per minute, 40-45 Kpa pressure, interface temperature of 230$^{o}$C with ionization energy of 120 kv (mass range 33-400). The analysis results of mass spectrum interpretation is performed by comparing mass spectrum in the data base NIST 62.

## Results and Discussion

### The results of the liquid smoke components by using GC MS

GC MS is a combination tool of gas chromatography and mass spectrometry, that the result of analysis on GC is directly linked to the system on mass spectrum of several compounds in the system, so that it will obtain retention time, name and structure of the compounds in the analyzed samples. From the chromatogram of liquid smoke analysis results with GC MS dominant in 8 (eight) purification method is predicted as derivative compounds of acetic acid and phenols. The results of chemical components 10 (biggest ten) of wood liquid smoke with different purification method can be seen in table 1. Then, peak chart on cinnamon liquid smoke components analysis result with different purification method by using GC MS is presented in (Figure 2,3,4,5,6,7,8,9). The result of analysis 10 (ten) chemical components of liquid smoke to the results of eight different methods of purification can be seen in (Table 1).

**Table 1. The result of chemical components of cinnamon liquid smoke with different purification**

| Purification | Purification Method | Component | RT | (%) area |
|---|---|---|---|---|
| Distillation | | 1.Acetic Acid | 3.18 | 34.99 |
| | | 2. Acetic Acid | 3.42 | 23.42 |
| | | 3. Phenol, 2-methoxy- | 20.32 | 9.65 |
| | | 4. 2-Furancarboxaldehyde | 7.33 | 6.98 |
| | | Furfural,2-Formylfuran | | |
| | | 5. 2-Methoxy-4-Methylphenol | 23.78 | 2.98 |
| | Temperature | 6. Phenol | 16.65 | 2.4 |
| | 100+10$^{o}$C | 7. 2-Furancarboxaldehyde | 7.74 | 1.97 |
| | | Pyrazole, 1,4-Dimethyl | | |

| | | | | |
|---|---|---|---|---|
| | | 8.Phenol,4-Ethyl-2-Methoxy | 26.36 | 1.77 |
| | | 9.2-Cyclopenten-1-one-,2-Methyl- | 12 | 1.31 |
| | | 2-Methyl-2-Cyclopenten-1-one | | |
| | | 3-Methylcyclopenten-2-Enone | | |
| | | 10.Propanoic Acid, 2-Methyl,2-Ethyl | 3.84 | 1.21 |
| | | Furan,tetrahydro-2-methoxymethyl | | |
| | | Propanoic Acid,2-Methyl-,Pentyl | | |
| | | 1.Acetic Acid | 4.33 | 33.42 |
| | | 2.Acetic Acid | 4.48 | 17.34 |
| | | 3.Acetic Acid | 4.66 | 13.95 |
| | | 4.Acetic Acid | 4.6 | 3.84 |
| | | 5. Acetic Acid | 4.55 | 3.29 |
| | Distillation Temperature | 6.Phenol,2-Methoxy | 20.36 | 2.38 |
| | 140+10$^{o}$C | 7.Phenol, 2,6-Dimethoxy | 28.49 | 2.25 |
| | | 8.2(3H)-Furanone, Dihydro | 13.75 | 1.65 |
| | | Gamma.Butyrolactone | | |
| | | 9.Cyclotene | 18.76 | 1.58 |
| | | Corylone 2-Hydroxy-3-Mrthyl-2-c | | |
| | | 2-Cyclopenten-1-one,2-Hydroxy-3-m | | |
| | | 10. Acetic Acid , Ethyl Ester | 4.92 | 1.47 |
| | | Ethyl actate | | |
| Filtration | | 1.Acetic Acid | 3.96 | 31.49 |
| | | 2.Phenol, 2,6-Dimethoxy | 28.58 | 8.19 |
| | | 3.1,2-Benzenediol | 24.99 | 5.04 |
| | | 4.Benzene, 1,2,3-Trimethoxy | 30.99 | 4.61 |
| | | 4-Methoxy-2-Methyl-1-(Methylthio)b | | |
| | | Benzoid Acid, 4-Hydroxy-3-Methoxy | | |
| | | 5. Phenol,2-Methoxy | 20.39 | 3.95 |
| | Active Charcoal | 6. 2-Butanamine,Hydrocloride | 21.05 | 3.33 |
| | | Acetamide, N,N-Dimethyl | | |
| | | 4H-Pyran-4-one,2,6-Dimethyl | | |
| | | 7. Acetid Acid | 2.96 | 2.58 |
| | | 8.2-Furancarboxaldehyde | 7.38 | 2.56 |
| | | Furfural 2-Formilfiran | | |
| | | 1H-pyrazole,3,5-Dimethyl | | |
| | | 9.Phenol,2,6-Dimethoxy | 28.33 | 2.39 |
| | | 10.2,4-Dimethyl-3-(Methoxycarbonil)-5 | 32,90 | 2.27 |
| | | 2,3,5-Trimetosytoluene | | |

| | | | | |
|---|---|---|---|---|
| | | Hydroquinone,Mono-TMS | | |
| | | 1. Phenol,2,6-Dimethoxy | 28.85 | 10.34 |
| | | 2. Acetic Acid | 4.04 | 9.71 |
| | | 3. Acetic Acid | 3.72 | 7.95 |
| | | 4. 1,2-Benzenediol | 25.68 | 6.75 |
| | | 5. Acetic Acid | 4.09 | 5.78 |
| | | 6. Acetic Acid | 3.57 | 4.6 |
| | Active Charcoal | 7. 4-Methoxy-2-Methyl-1-(Methylthio)b | 31.17 | 3.69 |
| | + zeolite | Benzene, 1,2,3-Trimethoxy | | |
| | | Benzoic Acid, 4- hydroxy-3-Methoxy | | |
| | | 8. Acetic Acid | 3.26 | 3.4 |
| | | 9.Propanenitrile, 3-(Methylamino) | 21.87 | 2.93 |
| | | 2-Butanamine,Hydrochoride | | |
| | | Butanal,3-Methyl | | |
| | | 10. 1.2-Benzenediol, 3-Methoxy | 26.49 | 2.91 |
| | | 2-Methoxyresorcinol 1,3-Benzene | | |
| | Zeolite | 1. Acetic Acid | 4.43 | 22.61 |
| | | 2. Acetic Acid | 3.44 | 16.92 |
| | | 3. Phenol,2,6-Dimethoxy | 28.71 | 7,84 |
| | | 4. 1,2-Benzenediol | 25.37 | 7.1 |
| | | 5.2-Propinamide | 21.48 | 5.48 |
| | | 1,6-Heptadien-4-Ol | | |
| | | Decane, 2- Methyl | | |
| | | 6. Phenol, 2-Methoxy | 20.39 | 3,13 |
| | | 7.1,2-Benzenediol,3-Methoxy | 26.35 | 3.02 |
| | | 2-Methoxyresorcinol 1,3-Benzene | | |
| | | 8. 4-Methoxy-2-Methyl-1-(Methylthio)b | 31.06 | 2.98 |
| | | Benzene, 1,2,3-Trimethoxy | | |
| | | Benzoid Acid 4- Hydroxy-3-Methoxy | | |
| | | 9. 1,6-Anhydro-Beta-De-Glucopyranose | 33.91 | 2.74 |
| | | Hexanoid Acid | | |
| | | 10.2,4-Dimethyl-3-(Methoxycarbonil)5 | 32.96 | 2.47 |
| | | 2,3,5-Trimethoxytoluene | | |
| | | Methylphloracetophenone | | |
| Decantation (Precipation) | Decantation 1 day | 1. Acetic Acid | 2.07 | 25.56 |
| | | 2. Acetic Acid | 3.24 | 25.04 |
| | | 3. Acetic Acid | 1.69 | 20.5 |
| | | 4. Acetic Acid | 2.79 | 12.67 |

| | | | | |
|---|---|---|---|---|
| | | 5. Acetic Acid | 1.53 | 6.47 |
| | | 6.Phenol,2,6-Dimethoxy | 24.9 | 1.5 |
| | | 7. Phenol | 12.32 | 1.38 |
| | | 8. Ethanol 2-Phenoxy | 19.45 | 1.27 |
| | | 1-Fluoro-3,4,5-Trimethylbenzene | | |
| | | 9. Phenol | 14.24 | 1.18 |
| | | 10. Phenol | 11.98 | 0.81 |
| | Decantation 2 day | 1. Acetic Acid | 2.76 | 22.95 |
| | | 2. Acetic Acid | 4.38 | 20.87 |
| | | 3. ethyl acetate | 1.84 | 16.65 |
| | | Acetic Acid | | |
| | | 4. Acetic Acid | 1.67 | 3.87 |
| | | 5. Phenol | 13.15 | 3.79 |
| | | 6. Phenol | 14.67 | 3.72 |
| | | 7. Phenol | 14.05 | 3.58 |
| | | 8. Phenol | 13.33 | 2.98 |
| | | 9. Phenol | 14.88 | 2.97 |
| | | 10. Phenol | 12.21 | 2.3 |
| | Decantation 3 day | 1. Acetic Acid | 2.16 | 39.85 |
| | | 2. Acetic Acid | 1.54 | 28.97 |
| | | 3. Acetic Acid | 2.82 | 21.75 |
| | | 4. Formic acid, 2-Methylpropyl | 1.4 | 8.45 |
| | | Formic Acid, 1-Methylpropyl | | |
| | | Acetic Acid | | |
| | | 5. Phenol, 4-methoxy | 15.17 | 0.98 |
| | | Phenol, 3-methoxy | | |
| | | Phenol, 2-methoxy | | |
| | | 6. - | | |
| | | 7. - | | |
| | | 8. - | | |
| | | 9. - | | |
| | | 10. - | | |

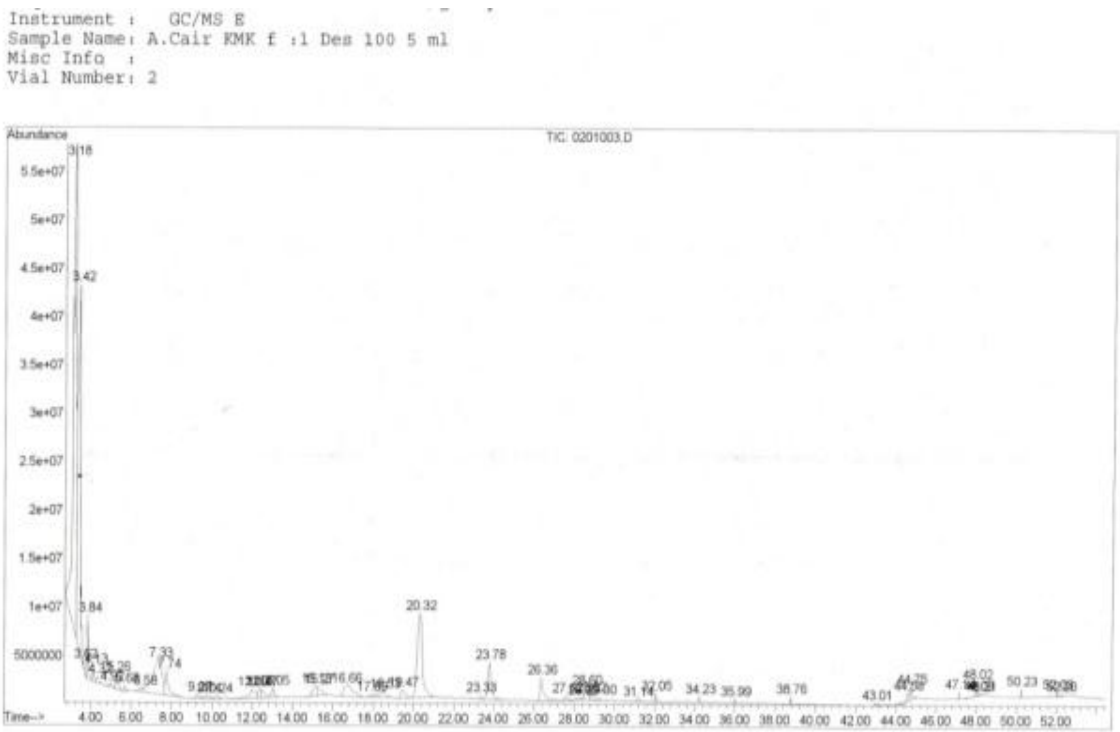


**Figure 2. Graphic Peak of Chemical Components Results in cinnamon liquid smoke with distillation purification at temperature 100°C**

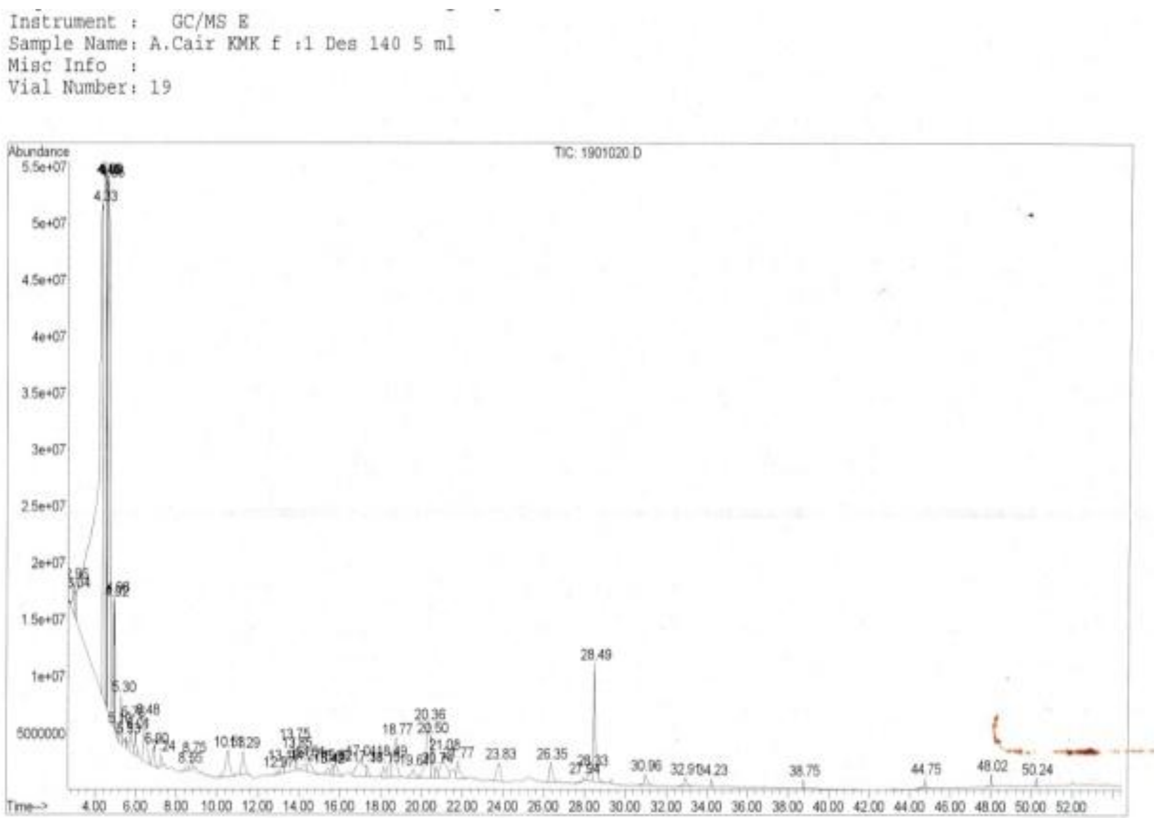


**Figure 3. Graphic Peak of Chemical Components Results in cinnamon liquid smoke with distillation purification at temperature 140°C**

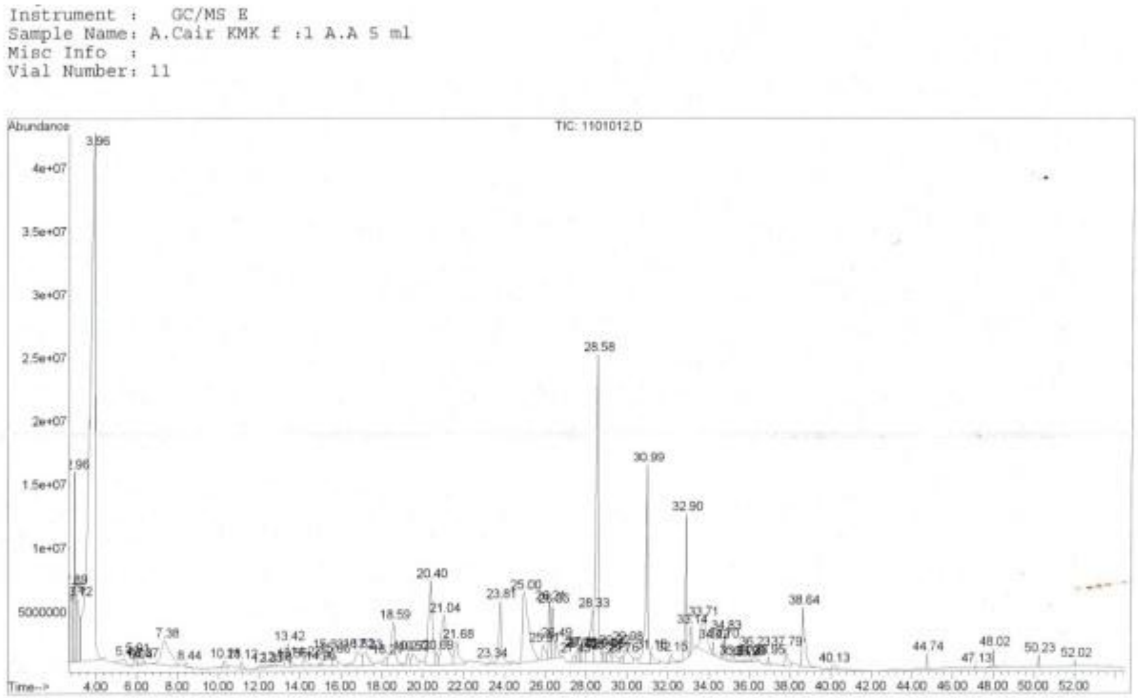


**Figure 4. Graphic Peak of Chemical Components Result in cinnamon liquid smoke with filtration purification method by using active charcoal (AA)**

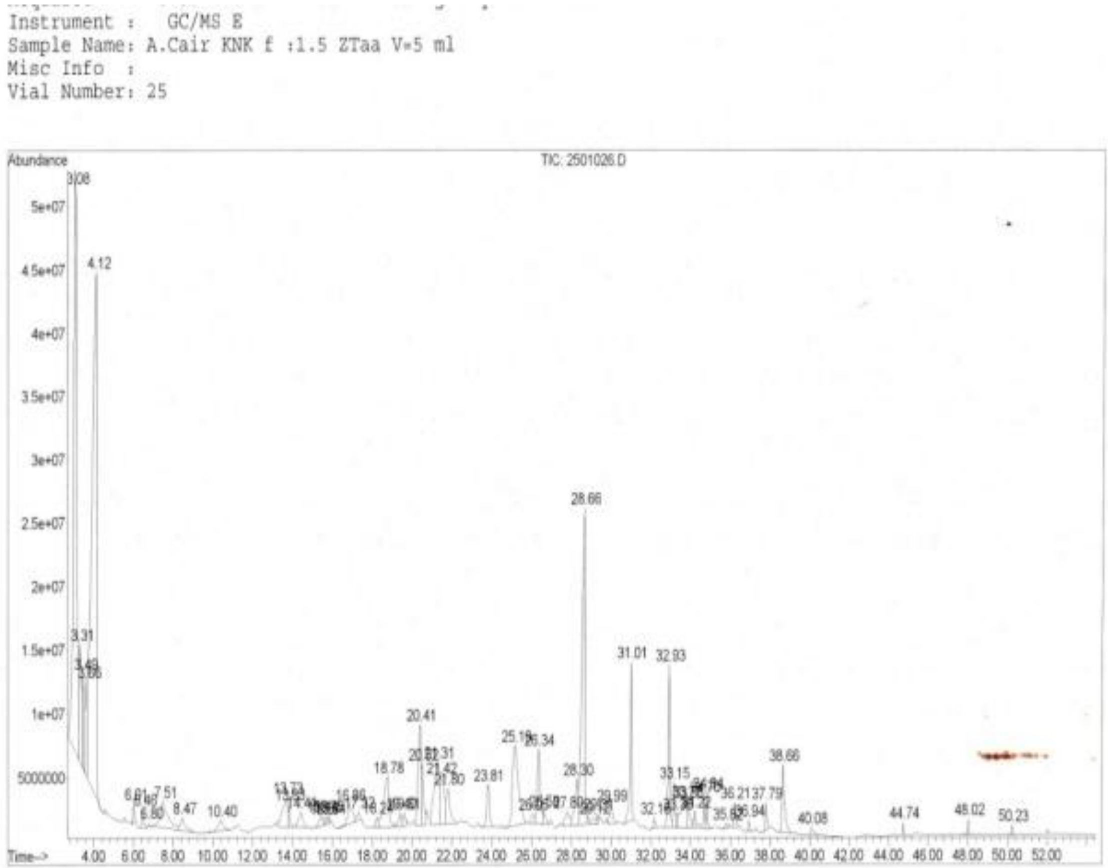


**Figure 5. Graphic Peak of Chemical Components Result in cinnamon liquid smoke with filtration purification method by using active charcoal (AA) + Zeolite (Z)**

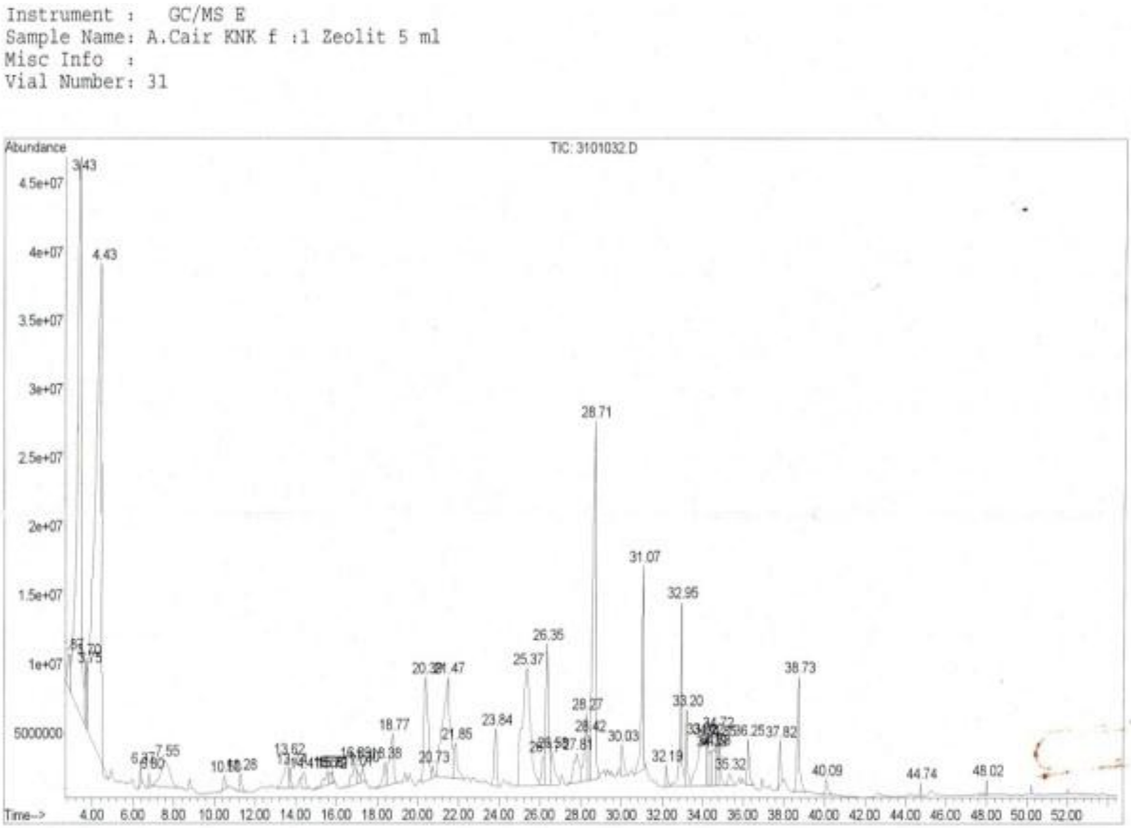


**Figure 6. Graphic Peak of Chemical Components Result in cinnamon liquid smoke with filtration purification method by using Zeolite (Z)**

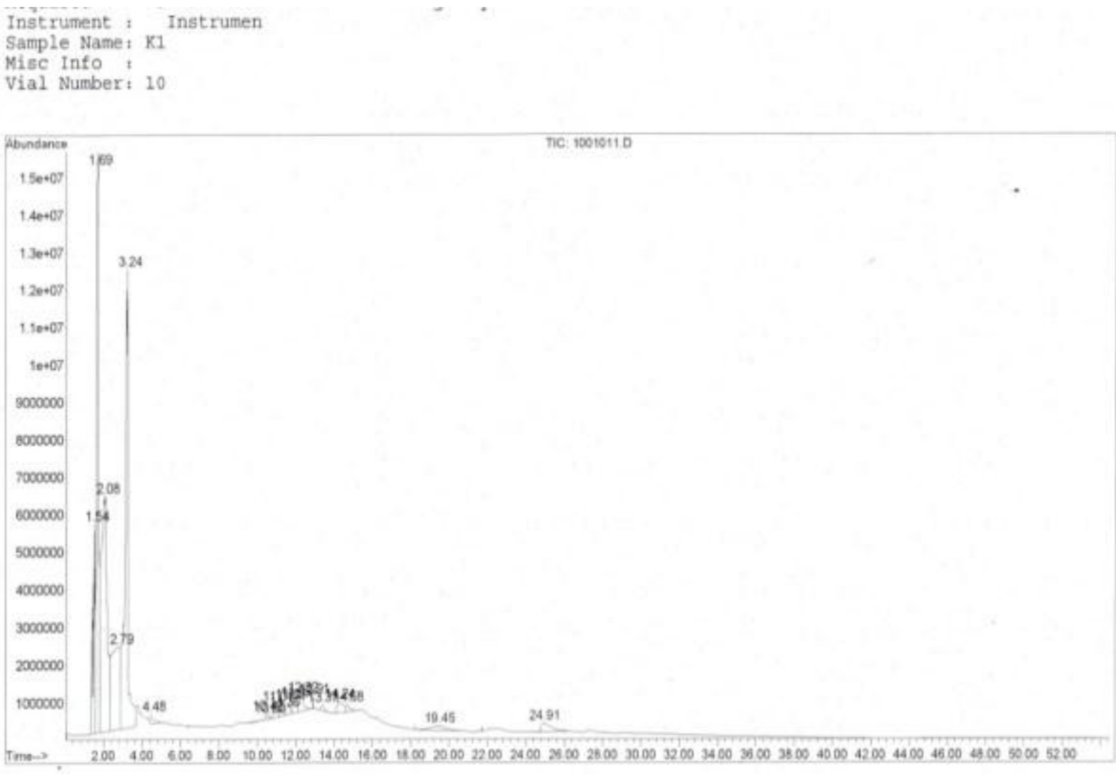


**Figure 7. Graphic Peak of Chemical Components Result in cinnamon liquid smoke with decantation purification method for 1 day**

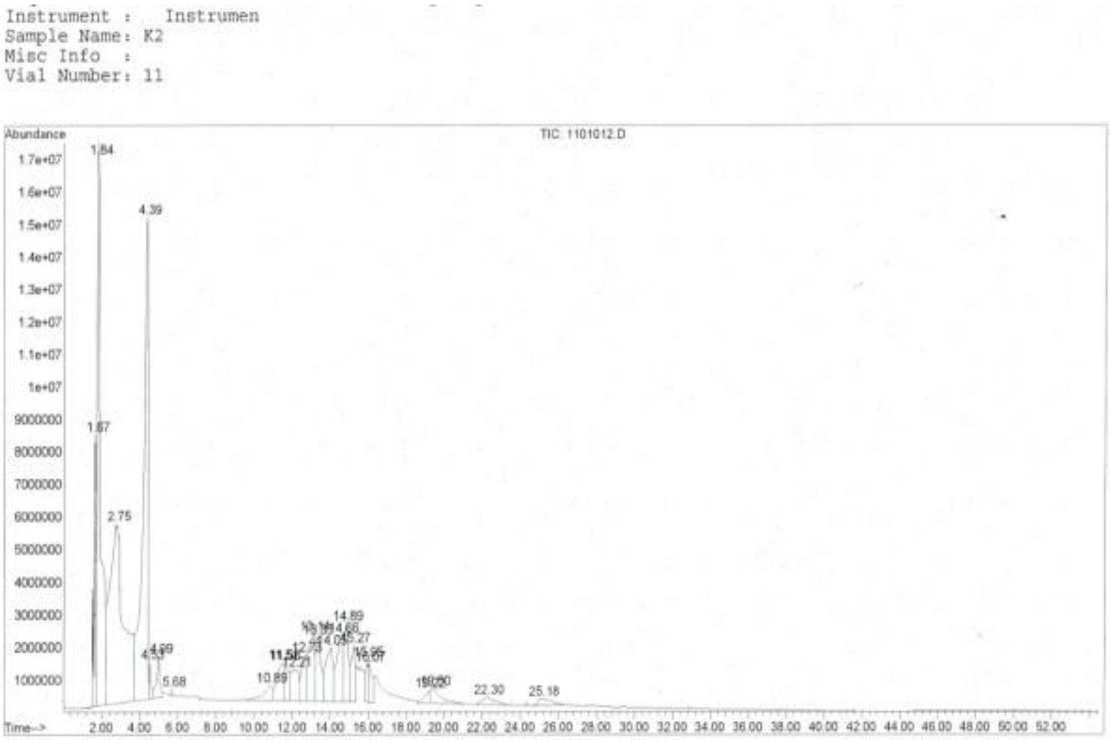


**Figure 8. Graphic Peak of Chemical Components Result in cinnamon liquid smoke with decantation purification method for 2 day**

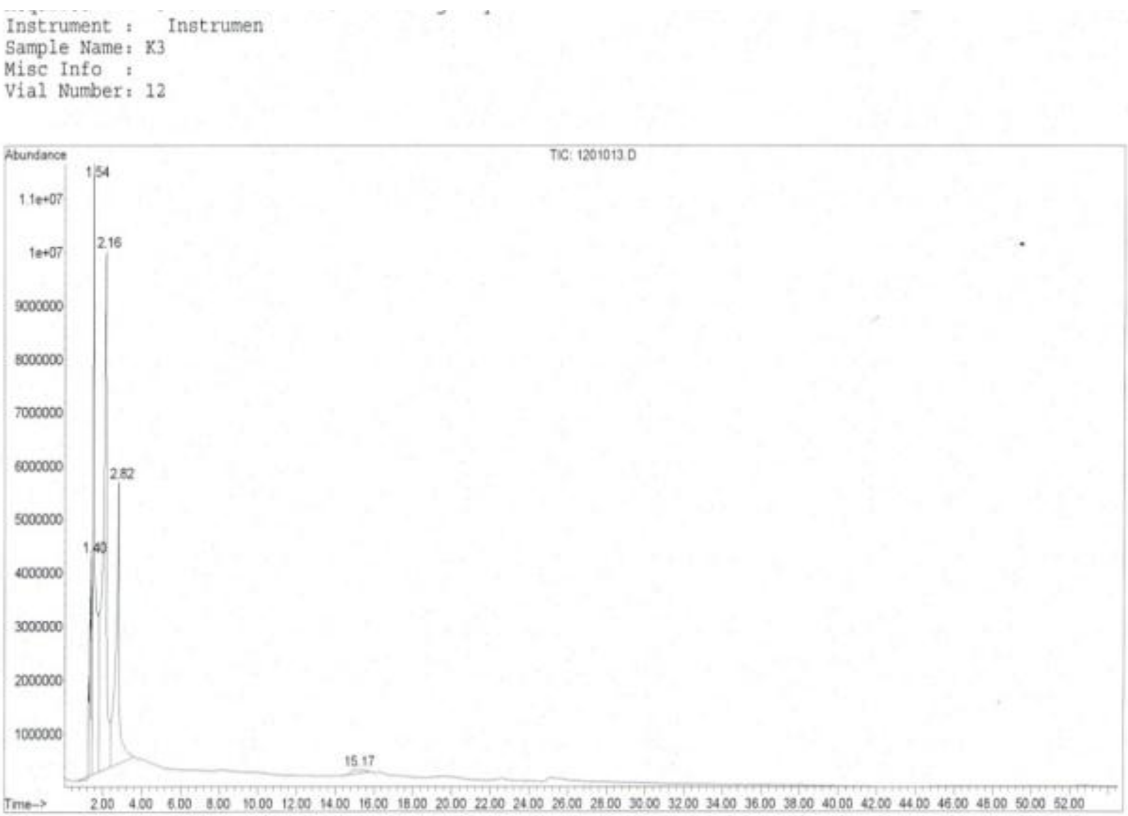


**Figure 9. Graphic Peak of Chemical Components Result in cinnamon liquid smoke with decantation purification method for 3 day**

The analysis results with GC MS shows that components of cinnamon liquid smoke with eight purification method contains dominant chemical compounds of acetic acid and phenol compound. Acetic acid class compound with the largest area can be found in liquid smoke purification method by decantation for 3 days at 39.85%, and the smallest can be found in liquid smoke filtration purification method by using active charcoal mixture with zeolite at 9.71%. The small area percentage is because acetic acid is absorbed by active carbon and zeolite. For phenol compound, there is a big area percentage, on liquid smoke purification method by using active charcoal + zeolite at 10.34%, and the smallest in purification method by decantation for 3 days at 0.98%.

Liquid smoke redistillation is intended to reduce the levels of tar and *benzo(a)pyrene* contains in liquid smoke. The result of research shows that on liquid smoke, it cannot be performed fractional distillation at some temperature level. Liquid smoke is a fluid that consists mostly of water, so the presence of semi polar organic compounds such as phenol will form azeotrop in water that causes the inability for fractional distillation at several temperature fractions. Maga and Girarrd[13,14] reported that cellulose pyrolysis occurs at temperature 260-310°C and lignin undergoes pyrolysis at temperatures 320-400°C. Pyrolysis of cellulose produces simple *glucose* (sugar), oligosaccharides, acetic acid, furan, carbonyl (glyoxal, metilglioksal, diacetyl, formadehid, evaporation of water, *carboxyl, hidropiroksid*, production of CO and $CO_2$ (and other gas phases) as well as charcoal waste. Shahidi[15] reported that the result of *cellulosic* pyrolysis is *alcohol* with concentration of 2.6%. While lignin at that temperature produces phenol (*guaiacol, siringol, eugenol, pyrocatechol), hydroquinone, vanillin* and vanilat and water and above 400$^{o}$C generates *poliksiklis aromatic hydrocarbons* (PAH) and other complex compounds.Budaraga*et al*., [34] reported *cinnamon* raw material at different pyrolysis temperature shows results that there are lots of chemical components and the measuring result of liquid smoke using GC-MS also shows that chemical component of *cinnamon* liquid smoke at pyrolysis temperature of 100$^{o}$C, 200$^{o}$C, 300°C and 400$^{o}$C has different chemical component area with liquid smoke of coconut fiberand coconut shell.

The following chart peak of liquid smoke components at 8 (eight) different purification methods can be seen in the (figure diagram 2,3,4,5,6,7,8,9).The results of liquid smoke fractional distillation at temperature 100$^{o}$C is the fluid that still smells of smoke but do not stink, more clearer than the original smoke and almost colorless. At liquid smoke distillation at temperature 100$^{o}$C, the distillate that is obtained has RT 20.32 with area of 9.65%, which is phenolic compounds (Figure 2). The missing part in this process is largely semi-solid viscous liquid that remains on distillation tube as tar and *benzo(a)pyrene*. At °C distillation temperature of 140$^{o}$C, distillation that is obtained has RT 28.49 with total area of 2.25%, which is also dominant on phenol compounds (Figure 3). Based on the results of distillation at above temperature that wood liquid smoke shows dominant phenol compounds compared to the other compounds.

In this study, active charcoal is used at 3.5%, meaning that for absorbing 100g liquid smoke, it needs active charcoal at 3.5 grams. The use of this ratio is based on the reason that on the ratio, the produced liquid smoke is colorless like liquid smoke from pyrolysis results. This indicates that tar remaining in the liquid smoke has begun to decrease and it is expected that benzo(e)pyrene is also no longer exist. The results of adsorption by using active charcoal to produce liquid smoke with smell of smoke has begun to decrease. Retention (RT) of liquid smoke obtained from this process is 28.58 with total area of 8.19% is Phenol compound, *2,6-Dimethoxy* (Figure 4). If phenol compound is dominant then it is assumed that antibacterial activity from liquid smoke from adsorption will be reduced compared to the original liquid smoke. In figure 5 shows retention (RT) at 28.85 with total area of 10.34% with compound *Phenol, 2,6-Dimethoxy*. This means that the tool entraps active charcoal in combination with zeolite to produce the same type of compound. Active charcoal is one of the adsorbent that is widely used in the food sector. This is due to reason that active charcoal contains carbon that is inert, so it is safe to be used [5].

(Figure 6) shows retention (RT) at 28.71 with total area of 7.84% with Phenol compound, *2,6-Dimethoxy*. Based on the data that filtration method uses active charcoal, active charcoal and zeolites, and zeolite, it produces the same type of compound that is phenol compound. According to [16]that the mechanism of forming HPA compound is still not certain, some experts conclude that HPA is formed through the reaction of free radicals, intramolecular, or polymerization reactions of small molecules. Liquid smoke that can be used for food is liquid smoke that has been processed repeatedly through distillation and purification by using zeolite or active carbon adsorbents. Research on purification of liquid smoke by using adsorbents has been performed by [17]. That study uses active carbon adsorbent. Zeolite is part of mineral group that is the result of hydrothermal processes in alkaline igneous rocks [18]. The typical structure of zeolite is that it has lots of canals and pores. This causes the pores to have large surface area. Zeolite surface area can be enlarged with the activation [19].

The results of dominant compound found in liquid smoke on decantation treatment with MS GC measurement is acetic acid. This shows that liquid smoke purified by decantation produced dominant compounds of acetic acid. Decantation treatment is performed by letting the liquid smoke in precipation so it is expected that compounds such as tar and *benzo(a)pyrene* can also be put in precipation. Goldenberg [20] reported that decantation activity is the volumetric analysis of previously shaken sample and afterwards putting it aside.Payamara J [36]reported the major component of wood vinegar products are *acetic acid, methanol, propanoic acid, phenolic and carbonyl* compounds.

The chemical composition of liquid smoke that has been purified in (Table 1) shows that the distillation purification at temperature of 100°C on a *cinnamon* is capable of producing liquid smoke distillate that contains acetic acid and phenol compounds. Henrickson[21] reported that the lower of compound boiling point, then the higher of that compound volatile because the vapor point also becomes higher, on the contrary, the higher of compound boiling point then the lower it volatility. Liquid smoke as the result of distillation at temperature 100°C produces high levels of acid in liquid smoke. In addition, the thermal degradation process is faster and occurs at relatively low temperatures when compared with the dominant raw materials degradation of lignin such as local coconut shell and coconut fiber. Chen *et al*. [22] reported that the results of hemicellulose and cellulose thermal degradation causes carbonyl and acid contents in liquid smoke is higher than phenol content obtained from the degradation of lignin. Furthermore, Girard [14] reported that the degradation of lignin occurs at relatively higher temperatures > 350°C when compared with the degradation of hemicellulose and cellulose that takes place at temperatures >270°C. The total phenol obtained contained in this liquid smoke research result comes from phenolic compound dispersed in rough liquid smoke from pyrolysis at 400°C.

Liquid smoke derived from different raw materials and different pyrolysis method, will produce different chemical components[23,24,25]. Commercial liquid smoke that is widely used in industrial and laboratory scale, has been studied its compositions[23,24,25,26], the activity of its antimicrobial [27,28,29], and the effect on organoleptic characteristic of fishery products[30,31]. The composition of liquid smoke is very complex and consists of components that come from different chemical compound groups, such as *aldehydes, ketones, alcohols, acids, esters, furan* derivatives and *pyran, phenolic* derivatives, *hydrocarbons*, and *nitrogen*[32]. Based on chemical components with the biggest acetic acid (34.99%) and phenol (9.65%) contents are in the purification method by distillation at temperature 100$^{o}$C. The presence of acetic acid content in liquid smoke can be used in food preservation.Budaraga*et al*., [33]reported the percentage of the largest inhibition is got in thecombination of *cinnamon* raw materials treatment on pyrolysis temperature of 400 ± 10 °C of23.865% with $IC_{50}$ value of 35.52 ppm.

## Conclusion

The chemical components of liquid smoke with the greatest retention value contained in cinnamon raw material treatment on purification method by distillation at temperature of 140°C with chemical components of GC MS is *phenol 2,6-Dimethoxy* with retention value (RT) at 28.49, then on filtration treatment by using active charcoal is compound*2,4-Dimethyl3 (Methoxycarbonil) 5,2,3,5 Trimetosytoluene Hydroquinone, Mono-TMS* with retention value (RT) at 32.90, and also on the decantation treatment for one day is Phenol, 2,6-Dimethoxy with retention value (RT) at 24.90.

The use of cinnamon liquid smoke with purification method by using distillation at temperature 140°C is better to be used than the other purification method because the color is better.

## Acknowledgements

We would like to thank Directorate General of High Education, Ministry of Education, in correspond toImplementation Agreement of Competitive Research Grant, National Priority, Rector of Ekasakti University,The Head of LPPM Ekasakti University, The Dean of Agriculture Faculty Ekasakti University, Supervisors of Andalas University and Bung Hatta University Laboratory team and staff that have provided help in thisactivity.

## References


1. Pszczola, D.E., 1995. Tour Highlights Production and Users of Smoke Based flavours. Food Technology (1)70-74.
2. Pastor-Villegas, J., Pastor-Valle, J.F., MenesesRoddriguez, J.M., & Garcia Garcia, M. 2006. Study of Commercial Wood Charcoals for The Preparation of Carbon Adsorbents Journal of Analytical and Applied Pyrolysis. (76) 103-108.
3. Branen, A.L., P.M. Davidson and B.Katz. 1980. Antimicrobial Properties of phenolic Antioxidants and Lipids. Food Tech. (34) : 42-53.
4. Budaraga I K., Arnim,YettiMarlida,UsmanBulanin,2016, Liquid Smoke Production Quality from Raw Materials Variation and Different Pyrolysis Temperature. International Journal. Of Advanced Science Engineering Information Technology (IJASEIT) Vol.6 (2016) No.3.
5. Feti Fatimah.,2009. Study about how to make *1-(3,4-methylenedioxyphenil)-2-propylalcohol*. Chem. Prog. Vol. 2, No. 2. November 2009
6. Tranggono,SuhardidanA.H.Setiadji, 1997.ProduksiAsapCairBeberapaJenisKayudanPenggunaanyapadaPengolahanBeberapaBahanMakanan Indonesia, LaporanAkhirRisetUnggulanTerpadu, Kantor KementeriNRisetdanTeknologi Jakarta.
7. Maga, J.A., 1986, Polyciclic Aromatic Hydrocarbon(PAH) Composition of Mesquite Smoke andGrilled Beef, J.Agric. Food Chem. (34), 249.
8. O'hara,J.R.,MS.Chin,B.Danius.,1974. Determination*Benzo(a)pirena* in Smoke Condensates byHigh Pressure Rapid Liquid Chromatography,J.Food Science (39):38-41
9. Fatimah, F., 1998, AnalisisKomponen-KomponenPenyusunAsapcairTempurungKelapa, Thesis S-2, Pascasarjana UGM Yogyakarta
10. Yatagai, M.MadokaNishimoto,Keko Hori, TatsuroOhira, Akira Shibata (2002).Termiticidal Activity of Vinegar, its Components and their Homologues. Journal Wood Science (2002) 48:338-342.
11. Murhadi. 1994. IdentifikasidanKetahananPanasBakteripadaProdukRendangDagingSapi. Tesis IPB, Bogor
12. Setiyawati, MoriscodanPrayitno, TA. 2008. Pengaruh Ekstrak Tembakau Terhadap Sifatdan Perilaku Mekanik Laminasi Bambu Petung.Tesis Program PascaSarjanaUniversitas Gajah Mada.
13. Maga, J. (1988). Smoke in Food Processing.Florida: CRC Press-Inc Boca Rotan.
14. Girarrd, J.P., 1992, Smoking in Technology of Meatabd Meat Product, J.P.Girarrd, New York, EllisHorwood
15. Shahidi, F. 1997, Natural Antioxidant, Chemistry, Health Effect and Application, AOCS. Press Champaign, Illinois.: 1-23.
16. Chen, B.H. danYS.Lin, 1997, Formation of Polycyclicof Duck Meat, J.Agric.Food Chem. Vol.45,1394-1395
17. Novita, S.A., 2011, KinerjadanAnalisisTekno-EkonomiAlatPenghasilAsapCairDenganBahan Baku LimbahPertanian, Program PascasarjanaUniversitasAndalas Padang, (Artikel).
18. Saputra, R,.2006, PemanfaatanZeolitSintesisSebagaiAlternatifPengolahanLimbahIndustri, Paper.
19. Lestari.D., 2010, KajianModifikasidanKarakterisasiZeolitAlam Dari Berbagai Negara, Proseding National Kimia danPendidikan Kimia.
20. Goldenberg, Harry., 1959.Decantation as Precision Step in Colormetric Analysis, Anal Chem., 28 (6), 1003–1006
21. Henrickson. 2005. Chemistry. Canada Wiley Publishing.Inc.
22. Chen, C.A., Pakdel H. dan Roy, C. (2001). Production of monomeric phenols by thermochemical conversion of biomass: A Rreview. Bioresource Technology 79: 277-299.
23. Guillen, M.D., M.J. Manzanos and L. Zabala. 1995.Study of commercial liquid smoke flavoring by means of Gas Chromatography- Mass Spectrometry and Fourier transporm Infrared Spectroscopy. J Agric Food Chem 43:463-468.
24. Guillen, M.D. and M.L. Ibargoitia. 1998. New components with potential antioxidant and organoleptic properties, detected for the first time in liquid smoke flavoring preparations. J Agric Food Chem 46: 1276–1285.
25. Guiilen, M.D., M.J. Manzanos and M.L. Ibargoitia. 2001. Carbohydrate and nitrogenated compounds in liquid smoke flavorings. J Agric Food Chem 49:2395-2403.

26. Guillen,M.D. and Ibargoita,L. 1999. Influence of the Moisture Content on The Composition of The Liduid Smoke Produced in Pyrolysis of fagussylvatica L, Wood. Jurnal Agric. Food Chem, 47:4126-4136
27. Faith N.G,A.EYousef and J.B. Luchansky.1992. Inhibition of listeria monocytogenes by liquid smoke and isoeugenol, a phenolic coumponentfound in smoke.J.Food Safety 12: 303-314.
28. Munoz, R.E., E.A.E. Boyle and J.L. Marsden. 1998. Liquid Smoke Effects on Escherichia coli O157:H7. and its antioxidant properties in beef products. J Food Sci 63:150-153.
29. Milly, P.J., R.T. Toledo and S. Ramakrishnan. 2005. Determination of minimum inhibitory concentrations of liquid smoke fractions. J Food Sci 70:12-17.
30. Cardinal,M., J.Connet, T.Serot and R. Baron. 2006. Effects of the smoking process on odor characteristic of smoke herring (*Clupeaharingus*) and relationshipandfenolic compound content. Food Chem96 : 137-146
31. Martinez. O. J.Salmeron, M.D.Guillen, C. Casas, 2007. Textural and Physicochemical Changes in Salmon (SalmoSalar) Treated with Commercial Liquid Smoke Flavourings. Science Direct. Food Chemistry 100 (2007) 498 – 503.
32. Soldera, S., N. Sebastianutto and R. Bortolomeazzi.2008. Composition of phenolic compounds and antioxidant activity of commercial aqueous smoke flavorings. J Agric Food Chem 56: 2727–2734.
33. Budaraga I K., Arnim, YettiMarlida,UsmanBulanin, 2016. Antioxidant Properties of Liquid Smoke Production Variationof Pyrolysis Temperature Raw and Different Concentration.International Journal of PharmTech ResearchVol.9, No.6, pp 366-379.
34. Budaraga I K., Arnim,YettiMarlida,Usman Bulanin,2016. Analysis of Liquid Smoke Chemical Components with GC MSfrom Different Raw Materials Variation Production andPyrolysis Temperaturelevel.International Journal of ChemTech ResearchVol.9, No.06 pp 694-708.
35. Asha,S., Nithisha,K., BharathKumar,R. and Ravi Kumar,V,2014. Deciphering the Antimicrobial Potential of Cinnamonzeylanicum Bark. International Journal of PharmTech ResearchVol.6, No.4, pp 1226-1235..
36. Payamara J., 2011. Usage of Wood Vinegar as New OrganicSubstance. International Journal of ChemTechResearchVol. 3, No.3,pp1658-1662.

*****