The Characteristics of Volatile Compounds of Kenari (Canarium indicum L.) Shell Liquid Smoke

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Abstract. Kenari (Canarium indicum L.) shell is a potential raw material for liquid smoke. The quality of liquid smoke depends on the volatile compounds of it. This study was aimed to qualify the volatile properties of Kenari (Canarium indicum L.) shell liquid smoke. The characteristic of volatile compounds of Kenari shell liquid smoke was determined in qualitative study of it’s volatile components. Kenari shell liquid smoke was produced using pyrolysis method. The pyrolysis process was carried out at temperature 420°C for 100 mins. Volatile compounds of Kenari shell liquid smoke was analyzed using GC-MS. The GC-MS detected 58 peaks of Kenari shell liquid smoke sample. It was consisted of 32.8% phenolic compounds, 48.3% carbonyl compounds, 10.3% acidic compounds, and 8.6% unknown compounds. Major volatile compounds of it were consisted of acetic acid, 2,6-dimethoxyphenol (syringol), 2-furanecarboxaldehyde (furfural), phenol, and 2-methoxyphenol (guaiacol).

1. Introduction

Liquid smoke had been made by various raw material such as woods [1], coconut shell, coconut fiber, cinnamon [2], corn cob [3], palm kernel shell [4], cashew nut shell [5], durian peel [6], nutmeg shell [7], cocoa shell [8], and tobacco steam [9]. Liquid smoke has been used as antibacterial and antimicrobial [10]-[12], antioxidant [12]-[14], preservative [10], [15], [16], enhance organoleptic quality [17]-[20], enhance texture [21], [22] and enhance physico-chemical matters [23], [19], [11]. Kenari trees are one of Indonesia native plant. It grows well in East Indonesia [24], such as in North and South Sulawesi, Seram island, North Maluku, and Maluku. Kenari shell is waste product that have not utilized well because of its small shape. Kenari shell is a potential raw material to produce liquid smoke other than coconut shell which already well known as raw material of mostly liquid smoke product.

Kenari shell liquid smoke can be made using pyrolysis method [18]. Pyrolysis process is carried out to degrade organic compounds using heat in absence of oxygen condition. Pyrolysis process is referred to incomplete thermal degradation which produced charcoal, condensable liquid or tar, and gaseous products [25]. The chemical components of liquid smoke made from palm kernel shell was consisted of 41.03% acetic acid, 8.18% phenol, and 50.79% other compounds [10]. Other study reported liquid smoke made from cashew nut shell consist of 36.6% phenol, 7.1% carbonyl, and 18.8% [26]. Chemical components of liquid smoke widely used to enhance the flavour of foods, as antioxidant,
antibacterial, and antimicrobial. Liquid smoke has a lot of benefit. Kenari shell can be alternative raw material of liquid smoke and to utilize it as zero waste industry application. Identification of volatile compounds of liquid smoke made from Kenari shell is needed to be done. This study was carried out to measure the amount of liquid smoke produced from kenari shells and its volatile compounds.

2. Material and Method

Kenari shell kindly obtained from Moti, Ternate, North Maluku. Reagents used in this study were CH2Cl2 and catridge Sep-Pak Florisil. Equipments used in this study were pyrolysis reactor, rotary evaporator (IKA Werke HB4 Basic with vacuum evaporator Ikavac VC-2), and gas chromatography-mass spectrometer (GC-MS).

Kenari shell was analysed to determine water, ash, cellulose, hemicellulose, and lignin content using method by Ref. [27]. Kenari shell liquid smoke was produced by pyrolysis process [18]. Pyrolysis furnace used in this study was equipped with electric heater encircling reactor (1500 watt), diameter 20 cm, height 40 cm, with maximum 4 kg sample or material. Pyrolysis process was carried out at temperature 420°C for 100 mins. Liquid smoke then centrifuged in 4000 rpm for 20 mins. Volatile compounds of Kenari shell liquid smoke was extracted using CH2Cl2. The procedure had done according to method [28,29].

Identification of volatile compounds of Kenari shell liquid smoke had done by GC-MS. As much as 1µl of sample was injected to GC-MS (6890N 5975B MSD) with column (AGILENT 19091s-433 HP-5MS 5% phenyl methyl siloxane capillary column) 30m i.d. 0.25 mm, helium as the carrier gas and ionization EI 70 Ev. GC operational condition was used oven temperature 50°C at the beginning for 0.5 mins then increased 7°C/muns until reached 290°C, injector temperature 250°C, and detector temperature 280°C. Volatile compound were identified according to their retention times and based on the data base on the MS tools.

3. Results and Discussion

3.1. Chemical composition of kenari shells

Chemical composition of kenari shells was dominated with cellulose (39.24%) and lignin (38.00%) (Fig 1). Kenari shells composition were same with other plant shells. Almond, coconut, chestnut, walnut, and pistachio shells also were dominated with cellulose. The difference was at the amount of lignin and hemicellulose. Kenari shells had high amount of lignin, almost as many as cellulose, but had low amount
of hemicellulose. Other plant shells (coconut, chestnut, walnut, and pistachio shells) mostly had almost the same amount of cellulose, hemicellulose, and lignin [30].

3.2. The yield of pyrolysis processing of kenari shells

![Fig 2. The yield of pyrolysis processing of kenari shells](image)

The amount of liquid smoke produced from kenari shells was 42.58% (Fig 2). The yield of liquid smoke from kenari shells was higher than coconut shells, fibers, rice husks, and corncobs [31]. But in other study, the yield of liquid smoke from kenari shell was almost the same as corncob (48%) [32] and less than coconut shell (51%) [33].

3.3. Volatile compounds of kenari shell liquid smoke

![Fig 3. Chromatogram of kenari shell liquid smoke](image)

GC chromatograms of kenari shell liquid smoke were presented in Figure 3. It showed kenari shell liquid smoke consisted of 58 components. It occurred from the peak that showed in the chromatograms. Identification of the components was conducted by comparing the mass spectra with MS data base. Based on these data, it could be predicted that the aromatic components were detected in kenari shell liquid smoke.
Volatile compounds detected from kenari shell liquid smoke were consisted of 32.8% phenolic compounds, 48.3% carbonyl compounds, 10.3% acidic compounds, and 8.6% unknown compounds (Table 1). Research liquid smoke made from cashew nut shell consist of 36.6% phenol, 7.1% carbonyl, and 18.8% acid [26]. Liquid smoke from coconut shell consist of 58.40% acid and 3.85% phenol [34]. Liquid smoke made from palm kernel shell were reported consist of 41.03% acetic acid, 8.18% phenol, and 50.79% other compounds [10]. Kenari shell liquid smoke had almost the same characteristic with cashew nut shell liquid smoke, but different than coconut shell and palm kernel shell liquid smoke. Kenari shell liquid smoke had higher phenolic compound than acidic compound.

Table 1. The Volatile Compounds Detected In Kenari Shell Liquid Smoke And The Description Of The Flavour

| Peak | RT    | Area (%) | Compound                    | Qual | Flavour Description                  |
|------|-------|----------|-----------------------------|------|--------------------------------------|
| 1    | 2.058 | 0.645    | Nt                          | 9    | -                                    |
| 2    | 2.220 | 0.584    | 2-propanone                 | 86   | -                                    |
| 3    | 2.324 | 12.176   | Dichloromethane             | 96   | -                                    |
| 4    | 2.539 | 0.466    | 2,3-butanedione (diacetyl)  | 53   | soft, caramel [35]                   |
|      |       |          |                             |      | butter [36],[37]                     |
|      |       |          |                             |      | cheese [37]                          |
| 5    | 2.587 | 0.787    | 2-Butanone (acetyl)         | 46   | sap acetone [38]                     |
| 6    | 2.952 | 12.074   | Acetic acid                 | 91   | acid [39],[40]                        |
|      |       |          |                             |      | spicy [39]                           |
|      |       |          |                             |      | vinegar [40],[41]                    |
| 7    | 3.085 | 2.195    | 1-hydroxy-2-Propanone       | 86   | vinegar, sharp [38]                  |
| 8    | 3.277 | 0.234    | 2,3-pentanedione             | 70   | Caramel [35]                         |
|      |       |          |                             |      | cheese [37]                          |
|      |       |          |                             |      | Acid [42]                            |
| 9    | 3.351 | 0.398    | Hydroxyl acetic acid methyl ester | 80   | -                                    |
| 10   | 3.447 | 0.570    | 3-hydroxy-2-Butanone (Acetoin) | 59   | butter [35],[37],[40], [42],[43]     |
|      |       |          |                             |      | Acid [41]                            |
|      |       |          |                             |      | cheese [37]                          |
| 11   | 3.697 | 1.448    | Propanoic acid              | 96   | fume [39]                            |
| 12   | 3.942 | 0.262    | 2-Methoxytetrahydrofuran    | 90   | -                                    |
| 13   | 4.164 | 2.218    | 1-Hydroxy-2-butanone        | 91   | -                                    |
| 14   | 4.337 | 0.502    | Propanal                    | 86   | -                                    |
| 15   | 4.490 | 0.493    | Cyclopentanone              | 93   | caramel [39]                         |
| 16   | 5.238 | 7.205    | 2-Furancarboxaldehyde (Furfural) | 96   | Sweet and caramel [36]               |
|      |       |          |                             |      | Frosted peanut [44]                  |
| No | Value | Retention Time | Compound                           | Detection Limit | Notes                        |
|----|-------|----------------|------------------------------------|-----------------|------------------------------|
| 17 | 5.608 | 2.031          | Nt                                 | 12              | -                            |
| 18 | 5.796 | 1.177          | 1-(acetyloxy)-2-propanone           | 80              | -                            |
| 19 | 6.291 | 0.257          | 1-isothiocyanato-propane            | 74              | -                            |
| 20 | 6.536 | 0.702          | 2-methyl-2-cyclopenten-1-one        | 96              | -                            |
| 21 | 6.635 | 0.257          | 1-(2-furanyl)-ethanone (Acetylfuran)| 91              | -                            |
| 22 | 6.768 | 1.500          | Dihydro-2(3H)-furanone (α-butyrolactone) | 91              | -                            |
| 23 | 7.021 | 1.155          | 2-Hydroxy-2-cyclopenten-1-one       | 90              | -                            |
| 24 | 7.628 | 0.445          | 2-methyl-3-pentanone               | 59              | -                            |
| 25 | 7.706 | 1.025          | Nt                                 | 22              | -                            |
| 26 | 7.793 | 0.433          | 3-methyl-2-cyclopenten-1-one        | 97              | -                            |
| 27 | 8.271 | 4.470          | Phenol                             | 97              | Sea, vinegar, metal, sulphur [44] phenolic, drugs, smoke [45] |
| 28 | 8.624 | 1.404          | Tetrahydro-2-furannethanol (Butanoic acid) | 59              | Rotten cheese, hard [37]    |
| 29 | 9.288 | 2.143          | 2-hydroxy-3-methyl-2-cyclopenten-1-one | 97              | spicy, green [44]           |
| 30 | 9.424 | 0.415          | 2,3-dimethyl-2-cyclopenten-1-one    | 81              | -                            |
| 31 | 9.808 | 0.904          | 2-methylphenol (o-cresol)           | 98              | smoke [43],[44] Burning rubber [43] |
| 32 | 10.299| 1.904          | 3-methylphenol (m-cresol)           | 95              | spicy [29]                   |
| 33 | 10.522| 4.094          | 2-methoxyphenol (guaiacol)          | 97              | drugs [39],[44] smoke [38],[44] Vaniline [44] |
| 34 | 11.104| 0.268          | 3-hydro-2-methyl-4H-pyran-4-one (maltol) | 95              | Smell sweet [39]            |
| 35 | 11.176| 0.389          | 3-ethyl-2-hydroxy-2-cyclopenten-1-one | 94              | -                            |
| 36 | 12.350| 0.267          | 2-methoxy-3-methylphenol (2-methoxy-m-cresol) | 87              | -                            |
| 37 | 12.680| 2.208          | 2-methoxy-4-methylpheno (2-methoxy-p-cresol) | 98              | Wood, smoke, sweet [43]     |
| 38 | 13.243| 1.850          | 1,2-Benzenediol (pyrocatecol)       | 97              | -                            |
| 39 | 13.510| 0.383          | [(1-methylthio)-thio] Benzenephthalene | 59              | -                            |
|   |   |   |   |   |
|---|---|---|---|---|
| 40 | 13.726 | 0.383 | 5-hydroxy-2,3-dimethyl-2-cyclopenten-1-one | 78 | - |
| 41 | 14.258 | 2.410 | 3-methoxy-1,2-Benzenediol (3-methoxypyrocatecol) | 97 | - |
| 42 | 14.420 | 2.269 | 2-methoxybenzeethanol | 94 | - |
| 43 | 15.006 | 0.963 | 4-methyl-1,2-benzenediol (4-methy|pyrocatecol) | 95 | - |
| 44 | 15.944 | 8.827 | 2,6-dimetoxyphenol (Syringol) | 95 | Light smoke [43],[44], spicy [44] |
| 45 | 16.150 | 1.324 | 3,4-dimetoxyphenol | 60 | Burnt smell [46] |
| 46 | 16.902 | 0.273 | 3-hydroxy-4-methoxy-benzaldehyde (vanillin) | 96 | Vaniline [41], sweet [41],[47] |
| 47 | 17.672 | 4.087 | 4-methoxy-2-methyl-1-(methylthio)benzene | 83 | - |
| 48 | 17.779 | 0.538 | 1-(2,3,4-trihydroxyphenyl)-ethanone | 87 | - |
| 49 | 18.456 | 0.281 | 1-(4-hydroxy-3-methoxyphenol)-ethanone (Acetovanillone) | 95 | Vaniline, sweet, honey [41] |
| 50 | 19.031 | 1.543 | 3-hydroxy-5-(hydroxymethyl)-2-methyl-4-pyridinecarboxaldehyde | 87 | - |
| 51 | 19.203 | 0.634 | 4-hydroxy-3-methoxy-benzenecacetid acid (Homovanillic acid) | 72 | - |
| 52 | 20.419 | 0.532 | (3S)-2-chloro-1-phenyl-1-penten-3-ol | 64 | - |
| 53 | 21.181 | 0.388 | Methyl-(2-hydroxy-3-ethoxy-benzyl)ether | 90 | - |
| 54 | 21.376 | 0.344 | 4-hydroxy-3,5-dimethoxy-benzaldehyde (Syringaldehyde) | 97 | - |
| 55 | 21.932 | 0.461 | 2,6-dimethohy-4-(2-propenyl)phenol | 87 | - |
| 56 | 22.529 | 0.768 | 1-(4-hydroxy-3,5-dimethoxyphenyl)ethanone (acetosyringone) | 97 | - |
| 57 | 23.089 | 1.700 | Nt | 80 | - |
| 58 | 23.884 | 0.228 | Nt | 53 | - |
The major components of kenari shell liquid smoke were acetic acid, 2-furanecarboxaldehyde (furfural), phenol, 2-methoxyphenol (guaiacol), and 2,6-dimethoxyphenol (syringol). Furfural component has a sweet taste like bread and caramel [48] and soft smell [49]. Derived phenol compound has strong, cresolic, and smoke smell [50]. It also reported has acidic, spicy, and soft smoking smell [49]. Derived carbonil has sweet, baked, and caramel taste [50].

Volatile compounds of kenari shell liquid smoke were product of combusting process of cellulose, hemicellulose, and lignin. Furane and phenol compounds were the products of cellulose through pyrolysis process. Hemicellulose also would decompose into furane and its derivates during the combustion process. Lignin produced derivate of phenolic compound such as methyl ester, mixture guaiacol, crecol, and phenol compounds and also tar [49],[51].

4. Conclusion
Kenari shell is a potential raw material to be made as liquid smoke. The number of components detected by GC-MS were 58 components. It was consisted of 32.8% phenolic compounds, 48.3% carbonyl compounds, 10.3% acidic compounds, and 8.6% unknown compounds. The major components of kenari shell liquid smoke consisted of acetic acid, 2,6-dimethoxyphenol (syringol), 2-furanecarboxaldehyde (furfural), phenol, and 2-methoxyphenol (guaiacol).

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