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DETERMINATION OF PRINCIPAL VOLATILE COMPOUNDS OF NANOENCAPSULATED COCONUT SHELL-LIQUID SMOKE AS A FOOD BIOPRESERVATIVE

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AUTHORS' CONTRIBUTIONS

This work was carried out in collaboration between all authors. Author SS designed the study, wrote the protocol and interpreted the data. Author PD anchored the field study, gathered the initial data and performed preliminary data analysis. Authors YP and SW managed the literature searches and produced the initial draft. All authors read and approved the final manuscript.

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ABSTRACT

Nanoencapsulated liquid smoke was made to enhance the physicochemical qualities, ease of handling and application of liquid smoke (LS) prepared from pyrolysis of coconut shells. A descriptive experiment was conducted to compare redistilled LS (sample F1), homogenised dispersion of nanoparticles (sample F2) and spray dried nanoencapsulated preparation of LS (sample F3). Encapsulants used were chitosan (1.5% w/v) and maltodextrin (8.5% w/v). The qualitative and quantitative composition of volatile compounds were studied by using a gas chromatograph-mass spectrometer (GC-MS) techniques. Results showed that sample F1 consisted primery of as acetic acid (57.70%) and phenol (24.03%). Sample F2 contained 18 compounds dominated by acetic acid (50.49%) and phenol (24.55%), and sample F3 contained 6 compounds dominated by acetic acid (11.10%) and phenol (70.79%). Nanoencapsulated liquid smoke (sample F3) was reduced an of acetic acid and increased in phenol content. It is suggested that nanoencapsulated liquid smoke will have enhanced as biopreservative activity compared with redistilled LS and enhanced sensory acceptability.

Keywords: Volatile compounds; coconut shell-liquid smoke; nanocapsules; chitosan; maltodextrin.

1. INTRODUCTION

Smoking technology has a long history in food preservation methods used either in household practices or food industries [1]. Preservative effects of smoke are due to the presence of antimicrobial and antioxidant compounds such as aldehydes, carboxylic acids and phenols [2]. Phenol derivatives resulting from lignin pyrolysis such as guaiacol (2-methoxyphenol) and syringol (2,6-dimethoxyphenol) have been identified as the most frequent compounds found in smoke [3,4].

Liquid smoke (LS) is a solution obtained through a series of processed involving burning material, collecting smoke, smoke condensation, and refining (two or three steps of distillation). Distillation allows the removal of undesirable polycyclic aromatic

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hydrocarbons (PAH) from the liquid [5]. Therefore, compared to original form of smoke, LS is considered much safer for health and is also easier to apply to food products as it is easier to control the smoky flavour and colour intensity [4].

The volatile bioactive compounds of LS which are responsible for biopreservative activity can evaporate during storage. Therefore, the application of nanoencapsulation might be important to protect these principal compounds. The technique represents an efficient approach in increasing the physical stability of the active substances, protecting them from interactions with food ingredients, and enhancing their effectiveness because of their nanosized formulation [6,7].

Various biocompatible and degradable natural polymers can be used as carriers in nanoencapsulation technique. Chitosan (CS) has been used as a wall material for encapsulation of sensitive core ingredients such as vitamin D_2 [8], astaxanthin [9] and ampicillin [10]. A previous study demonstrated the ability of CS assembled into nanoparticles in the size range of 400 - 600 nm [11]. Preparing nanoparticles in this size range is facilitated by the use of adequate cross-linking agents. TPP is a non-toxic polyanion known for its capacity to cross-link CS, a reaction mediated by electrostatic forces, resulting in the formation of ionic cross-linked networks [12]. Maltodextrin (MD) is the most commonly preferred wall material because of its low cost and effectiveness. Some studies have explored the use of MD to protect sensitive compounds like vitamin C in fruit juice and to increase product stability in acerola powder [13]. MD is water-soluble and able to protect encapsulated ingredients from oxidation. The aims of this study were to investigate the use of CS and MD as polycationic and anionic matrixes to protect volatile compounds of nanoencapsulated coconut-shell liquid smoke.

2. MATERIALS AND METHODS

2.1 Materials

Raw crude coconut-shell liquid smoke (LS) was obtained from Tropica Nucifera Industry, Yogyakarta, Indonesia. LS was purified two times using redistillation methods in the laboratory at 98±2^{o}C. Chitosan (CS) was purchased from Biotech Surindo, Indonesia (deacetylation degree 91.5%, moisture 10.43%, ash 0.71%). Maltodextrin (MD) with dextrose equivalent (DE) 10.8% was from Grain Processing Corp. (Iowa, USA), Sodium tripolyphosphate (TPP) and diethyl ether were supplied by Sigma Chemicals Ltd. (Munich, Germany). The other chemicals used for analysis were of analytical grade.

2.2 Preparation of Nanocapsules

CS-MD mixed nanoparticles were prepared with a slight modification of previously described method [11], based on the polyelectrolyte complexing of CS with MD and additional ionic gelation of chitosan with sodium tripolyphosphate (TPP) anions CS (1.5% w/v) and MD (8.5% w/v) dissolved in coconut shell liquid smoke. TPP (1.0 mg/mL) was added in these mixtures and agitated using a magnetic stirrer at 200 rpm for 30 min at room temperature. Liquid smoke alone (without treatment) was referred to as F1. Nanoparticles were isolated by centrifugation (Centrifuge Damon/IEC Division, Connecticut, USA) at 3,000 rpm in a 50 mL conical tube for 30 min at room temperature. Supernatant was discarded and nanoparticles were vacuum filtered (Gast, USA) using Whatman # 2. The dispersion of nanoparticles was heated at 50°C in a waterbath for 15 min and homogenized using homogenizer (Ultraturrax T50 Basic IKA Werke, Germany) at 4,000 rpm for 2.5 min and referred to as F2. Subsequently, the sample dispersion was fed into a Büchi B-290 mini spray dryer (Flawil, Switzerland). The operating conditions were aspirator rate 50%, drying inlet air temperature 150±2°C, outlet air temperature between 70 and 82°C, feed flow rate 5.1 mL/min and referred to as F3. The result spray-dried powders were collected and stored in amber bottles and stored under desiccation at room temperature prior to analysis of volatile compounds.

2.3 GC-MS Analysis

Volatile compounds in the coconut shell liquid smoke nanocapsules were identified using gas chromatography-mass spectrometry (GC-MS). Three g of sample was diluted in water (1:1) into a 10 mL separatoring funnel which was vortex-mixed and diethyl ether was added (1:3). An ultrasonic bath was used to extract solutes for 10 min and then the two phases were allowed to separate. The separated organic phase was dried over anhydrous sodium sulfate and analyzed in a gas chromatograph coupled to a quadrupolar mass spectrometer (Shimadzu QP 2010; Shimadzu, Tokyo, Japan) with a flame ionization detector (260°C). The analytes were separated on a Restex Stabilwax-DA capillary column (30 m × 0.25 mm i.d., 0.25 μm film thickness). Column temperature was held at 60°C for 2 min, increased to 215^{o}C at 5°C/min. Injector, transfer line, and ion source temperatures were 250, 250, and 200°C, respectively. Injection was conducted in the splitless mode (2 min), and the volume was 0.2 μL. Helium was used as the carrier gas at a flow rate of

81.5 mL/min at 12 kPa pressure. Electron impact mass spectra were recorded at 70 eV ionization energy.

Coconut shell-liquid smoke nanocapsule components were identified by comparison of their mass spectra and retention times with those of standard compounds or by comparison of the mass spectrum with those of the mass spectra library Wiley 229 [14].

3. RESULTS AND DISCUSSION

3.1 Physicochemical Characteristics of Coconut-shell Liquid Smoke

The redistilled coconut shell liquid smoke (LS) at 98±2°C gave clear yellowish white, acidic liquid (pH 2.54), containing total carbonyl 10.83%, total phenolic 2.08%, acetic acid 9.97%. Acids contained in coconut shell-LS influence food stuff flavor, pH and shelf life. Phenolic compounds provide aromatic and antioxidant effects as well as enhancing the shelf-life of the product [15]. Carbonyls generally contribute to the colour of the product once reacted with amino groups. [16] mentioned that bioactive compounds in liquid smoke gave not only preservative and antioxidative effects but also contribute colour, flavour and taste to the product [15,17]. Furthermore, compounds of polyaromatic hydrocarbons family were not identified, indicating that the redistillation of the LS at 98±2°C was effective in eliminating carcinogenic PAHs. GC-MS analysis on liquid smoke has been also performed a previous study [6,18].

3.2 Volatile Compounds

The GC-MS analysis on redistilled coconut shells liquid smoke (LS) identified some chemicals as shown in Table 1. From GC-MS spectra were identified 17 peaks of a higher proportions that identified components grouped by families of compounds, together with the proportion of the major components in the sample. The highest constituent (57.7%) was Acetic acid, followed by Phenol (24.03%), that have more than 90% similarity with those in GC-MS instrument's library.

Fig. 1 shows the total ion chromatogram of the dispersed nanoparticle (sample F2) and nanocapsules (sample F3). It can be observed that the compounds in appreciable concentrations in the sample eluted between 9 and 22 min, and only a few that were eluted under 7 min in very small concentrations.

From GC-MS spectra data, the dispersed nanoparticles (sample F2) consisted of 18 components dominated by acetic acid (50.85%), phenol (32.50%), 2-metoxyphenol (guaiacol) (5.55%), methyl acetetat (2.16%), propionic acid (1.32%), 2-methoxy-4-methylphenol (1.32%), 4-methylphenol (1.30%), 4-ethyl-2-methoxyphenol (1.05%), 2-methylphenol (0.92%), 2-furancarboxaldehyde (0.81%), 1-hydroxy-2-propane (0.46%), 2-ethylphenol (0.33%), 2-cyclopenten-1-one (0.30%), methyl propanoate (0.28%), beta tumerone (0.28%), pentacosane (0.24%), 2-ethylphenol (0.18%), and 2,5-xylenol (0.14%). The results were in agreement with the report by [14,18] showed that highest compounds in liquid smoke was the Acetic acid followed by Phenol.

Table 1. GC-MS analysis on volatile compounds of coconut shell liquid smoke (sample F1)

Peak no.	Family (compound)	Area (%)	MW	Similarity (%)
Acid and alcohol derivatives		**64.23**		
1.	Methyl acetate	0.59	74	96
2.	Methanol	2.14	32	98
6.	Acetic acid	57.70	60	99
7.	2-(1-methylethoxy)-Ethanol	0	104	63
10.	Propionic acid	3.14	74	95
13.	1.4.dimethoxy-benzene	0.66	138	89
Carbonyl derivatives		**7.53**		
3.	1.Hydroxy-2-propanone	2.34	74	97
4.	2-Cyclopenten-1-one	0.70	82	91
5.	1-Hydroxy-2-butanone	2.13	88	89
8.	2-Furancarboxaldehyde	1.68	96	97
9.	1-(2-Furanyl) ethanone	0.20	110	91
11.	2(3H)-dihydro-Furanone	0.48	86	96
Phenol derivatives		**28.25**		
12.	2-Methoxyphenol	2.68	124	95
14.	Phenol	24.03	94	95
15.	4-Ethyl-2-methoxyphenol	0.32	152	88
16.	2-Ethylphenol	0.74	108	88
17.	3-Methylphenol	0.48	108	92

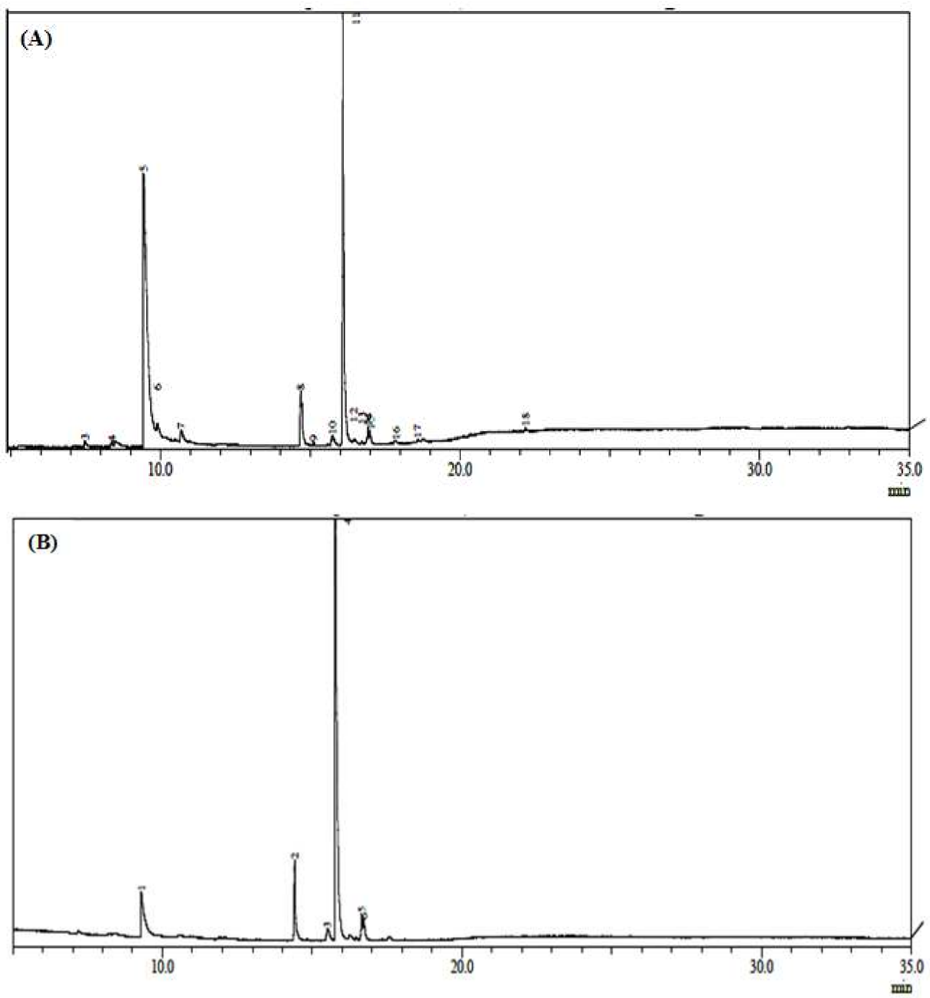

Fig. 1. Total ion chromatogram illustrating the separation of components A). Dispersed nanoparticles (Sample F2), and B). Nanocapsules (sample F3)

Coconut-shell liquid smoke nanocapsules (sample F3) showed 7 peaks in a higher proportion of acetic acid than sample F1. They were identified as phenol (70.79%), acetic acid (11.10%), 2-methoxyphenol (guaiacol) (9,92%), 4-methylphenol (2.81%), 2-methylphenol (2.74%), and 2-methoxy-4-methylphenol (2.64%) responsible for aromatic and antioxidant effects [1,15]. This result was similar to earlier reports [1,19] showing that phenolic compounds are prominent in liquid smoke and play a major contribution to antibacterial activity.

Compared to sample F1 (only LS), the percentage of phenol (70.79%) from sample F3 was higher and the amount of acetic acid was lower because of the heat treatment applied during the spray drying technique. The volatile compounds of nanocapsules (sample F3) were evided which very increase antibacterial and antioxidant properties of LS [4].

These volatile compounds were present in sample F1, as shown in Table 1. However, methyl propanoate, 2,5-xylenol, 2-methoxy-4-methylphenol, 4-methylphenol, 2-methylphenol, beta tumerone and pentacosane were not detected in redistilled LS of coconut shell (sample F2) perhaps as a results of thermal degradation during spray drying.

4. CONCLUSIONS

Coconut shell-liquid smoke nanocapsules contained selected volatile compounds of acetic acid and phenols. Nanoencapsulated liquid smoke (sample F3) revealed reduced concentration of acetic acid and increased phenol content. It is suggested that nanoencapsulated liquid smoke will have increased biopreservative activity compared to redistilled LS while also enhancing sensory acceptability of smoke products.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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