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Production of Coconut Shell Liquid Smoke from Pilot Plant of Pyrolysis Process

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Nur Sakinah Mohd Yusoffa[1,2], Maizirwan Mel[1], Wan Bazli Wan Ishak[2], Azlin Suhaida Azmi[1,*]

[1] Department of Biotechnology Engineering, International Islamic University Malaysia, P.O. Box 10, Kuala Lumpur 50728, Malaysia
[2] One Tech Supply, No 2 Kedai Jalan Besar, Jalan Kampung Kalong, Kampung Kalong Hilir, 44300 Hulu Yam, Selangor, Malaysia

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ABSTRACT

Liquid smoking in preserving protein-based foods such as meat, fish and cheese has been increasingly utilized, giving a pleasant flavour and inhibit the effects of pathogens. Liquid smoke is the product of pyrolysis system and distillation process. The smoke produced by incomplete combustion reaction involves the decomposition of polymers into low molecular weight of organic compound due to the effect of oxidation reaction, polymerization and condensation. Coconut Shell Liquid Smoke (CSLS) has been reported to be safe and not toxic to animals and human health. In this study, coconut shells are used to produce the liquid smoke using pyrolysis and condensation processes. The main objective of this research is to produce liquid smoke from coconut shell using 230 L pilot plant of pyrolysis system. The process conditions and characteristics for high yield of coconut shell liquid smoke production were determined. The composition and characteristics of coconut shell liquid smoke was also analysed. Result shows that the pyrolysis of coconut shell using the designed system produced the highest liquid smoke yield of 36.93 wt% obtained from run 1 at reaction temperature of 220°C in five hours.

1. Introduction

In the era of global food science, technology and nutritional health, preservation by liquid smoke is becoming more convenient, efficient and acceptable. The smoke flavour has being used for years to enhance and modify the flavour, aroma and colours of foods as well as to preserve meats. The use of liquid smoke for food preservation and also as flavoring agent is acceptable as it has more benefits compared to that using natural smoke. It is reported to be cheaper; healthier; more usable and practical; and easy to measure the smoke concentration in the product [1].

* *Corresponding author.*
E-mail address: azlinsu76@iium.edu.my (Azlin Suhaida Azmi)

Liquid smoke is produced in from pyrolysis process system. The smoke produced by incomplete combustion reaction involves the decomposition of polymers into low molecular weight of organic compound due to the effect of oxidation reaction, polymerization and condensation. The amount of solid particles and liquid in the gas medium will determine the density of smoke.

In this study, coconut shells were used to produce filtered liquid smoke from pyrolysis and condensation process. The coconut shell liquid smoke has huge potential as natural food preservative which able to increase the shelf life of proteinaceous food products.

2. Materials and Methods

2.1 Sample Preparation and Experimental Run

Coconut shells were collected from Pasar Tani Batang Kali, Malaysia. The shells were crushed with average surface area between 20 and 30 cm^2. Pyrolysis system consists of 230 L of carbon steel pyrolyzer and condenser as shown in Figure 1. Five experimental runs were conducted as shown in Table 1. Some of the feedstocks were cleaned by removing coconut husk and dust from the shell followed by sun dried.

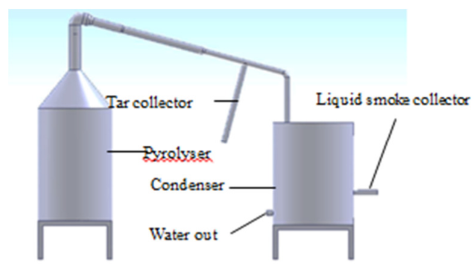

Fig. 1. Pyrolysis System

Table 1
Experimental runs

Run 1	100% clean and dry feedstock with diameter size between 4 – 6 cm
Run 2	100% clean and dry feedstock with diameter size between 2 – 4 cm
Run 3	Char product of first and second pyrolysis batch with diameter size between 2 – 5 cm
Run 4	Mixed of 90% clean and dry feedstock and 10% unclean and wet feedstock with diameter size between 2 – 4 cm
Run 5	100% unclean and wet feedstock with diameter size between 2 – 4 cm

The feedstock was pyrolyzed in a carbon steel reactor chamber which was externally heated by flame from liquefied petroleum gas (LPG). Temperature of the reactor was increased to the maximum value with constant gas pressure. The temperature reading was taken every 15 minutes to determine the heating rate using thermocouple probe that was inserted to the reactor chamber before the process started. Each run lasted for 5 hours.

The liquid smoke resulted from condensation process was left for two weeks to produce two layers product. The upper layer was the liquid smoke while the bottom organic layer was the light tar. After separating both layers, the liquid smoke (upper layer) was centrifuged at 10000 rpm for 25 minutes to make sure all light tars was totally removed, and finally it was filtered with microfiltration unit.

2.2 Analysis of Product

The product sample was analysed to determine the pH, density and chemical composition. The pH value of liquid smoke was determined by pH meter and the density was determined by density meter. The composition of carbonyls, acidic and phenolic compounds of coconut shell liquid smoke was determined by GC-MS analysis using DB-WAX column (30 m length, 0.25 mm diameter and 0.25 µm film). Liquid smoke sample of 100 mg of liquid smoke was dissolved in 10 mL hexane. Then, 100 µL of 2 N potassium hydroxide in methanol (2.8 g of potassium hydroxide + 25 mL of methanol) was added. The test tube was closed tightly and vortex for 30 seconds before it was centrifuged at speed of 8000 rpm in 15 minutes. The clear supernatant was transferred to a 2 mL auto sampler vial.

3. Results and Discussion

Figure 2 shows operating temperature obtained from the pilot plant unit for respective runs. All runs operated at above 150^{o}C after the first 1 h, and achieved maximum temperature of higher than 200^{o}C except for run 3. This is due to the feedstock of run 3 was utilizing char that was produced from runs 1 and 2. All in all, the temperatures indicated that the processes only achieved intermediate stage (primary pyrolysis). At this stage, it was reported that lignin is slowly decomposed (at wider temperature between 150-750^{o}C) followed by hemicellulose and later by cellulose at narrow temperature interval between 200-400^{o}C [2, 3].

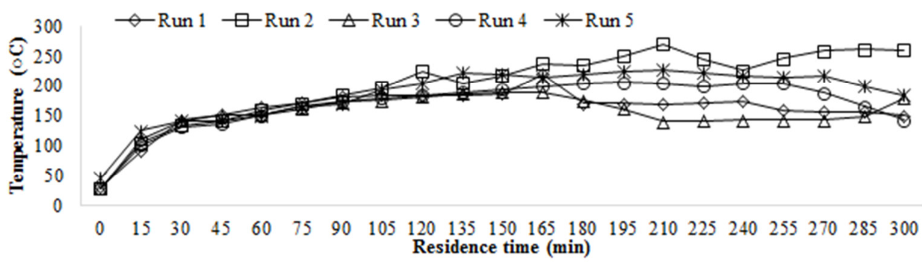

Fig. 2. Maximum operating temperature for every experimental run

3.1 Product of Pyrolysis and its Characteristics

The pyrolysis process produced four types of products which were incondensable gases, tar, char and liquid smoke. The proportion yield of each product varied with the parameters of the system. Figure 3 presents the product yield distribution from slow pyrolysis process of coconut shell from 5 different experimental runs.

The highest liquid smoke yield was produced from Run 1 (0.36 L/ kg of coconut shell), followed by Run 5, Run 4, Run 2 and Run 3. Run 1 also produced the highest char yield which was 0.5 kg/kg of coconut shell.The highest yields from run 1 might be due the size of the particle which was also shown by Sundaram *et al*. [4]. They have reported that liquid and solid yields from their pyrolysis products were increasing as the particle size increasing whereas the gas yield is reducing. The density of liquid smoke obtained from runs 1, 2, 4 and 5 is 1.016 g/cm^3, which is comparable to as obtained by Budaraga *et al*. [5] (i.e. 1.0167 – 1.0467 g/cm^3). Density liquid smoke obtained from run 3 however was lower than the rest which was 0.992 g/cm^3. The tar density for all runs was 1.029 g/cm^3.

More than 30% of liquid smokes were obtained from all experimental runs of coconut shell pyrolysis except from Runs 2 and 3. The yield is in accordance to Budaraga *et al*. [5] study, where they have observed the liquid smoke yield which is between 30- 34% as the pyrolysis temperature increase from 100°C to 400°C using coconut shell as the feedstock. Joardder et al [6] has also reported about the same yield (30-35%) obtained from coconut shell but at higher processing temperature of 400 – 600°C.

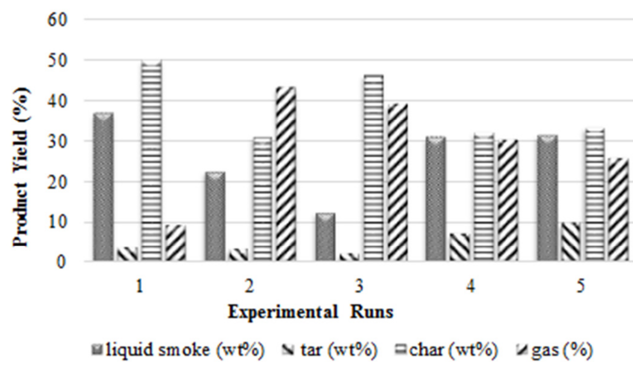

Fig. 3. Yield of pyrolysis products for every experimental run

The feedstock from Run 3 consisted of char obtained from Runs 1 and 2. The liquid portion of 12.4 wt% from Run 3 could not be considered as "liquid smoke" since the liquid contains very high amount of water and high carbon content of black particulates resulted from the pyrolysis of char. This was compared with the commercialized liquid smoke which was yellowish-brown in color. Thus, char from pyrolysis is not suitable to be used as feedstock for liquid smoke production.

Feedstock from Runs 2, 4 and 5 were different in terms of its quality which the feedstock from Run 2 had been cleaned and dried before it was pyrolised while Runs 4 and 5 were uncleaned and wet. The result obtained indicated that the unclean- and wet-feedstock led to the decreased of gaseous yield but increased in the liquid smoke and tar yield although the char proportions were both similar. The increased in liquid yield might be due to the high water content and volatile matter like coconut husk in the feedstock.

The pH of liquid smoke produced from different experimental runs is presented in Figure 4. Liquid smoke produced from Runs 3 and 5 were the highest and lowest pH value, respectively. However, these results are in accordance to Montazeri et al. [7] which is comparable to the commercialized liquid smoke (1.5 to 5.5 of pH value).

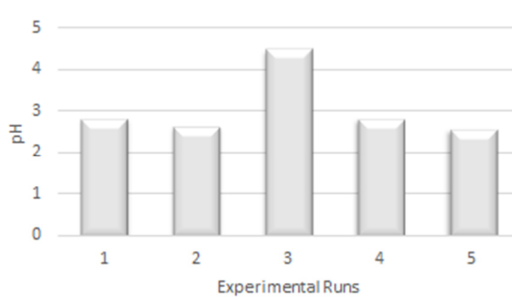

Fig. 4. pH of liquid smoke for every experimental runs

3.2 Chemical Composition of Liquid Smoke

More than 200 intermediate compounds were detected in the liquid smoke. Table 2 summarized identified compounds which were more than 80% probability match with the GC-MS library. The composition is comparable to commercialised liquid smoke as reported by Montezari *et al*. [7] with some missing compound. This might be due to different feedstock and process conditions were applied.

4. Conclusions

Pyrolysis of coconut shell using the available system design produced the highest liquid smoke proportion at 36.93wt% when using clean and dry feedstock at reaction temperature of 220°C in five hours. The liquid smoke pH and density were comparable to previous studies using coconut shell as feedstock. The chemical composition was also determined while its potential as alternative to other chemical food preservative could be further studied.

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Table 2
Chemical compound identified by a probability match >80% using the GC-MS library

Experimental Runs	**Run 1**	**Run 2**	**Run 3**	**Run 4**	**Run 5**
Alcohols	Isopropyl Alcohol (80%)	1-(2-methoxy-1-methylethoxy)-2-propanol (83%)	NA	Isopropyl Alcohol (80%)	NA
Furans	Furfural (81%)	NA	NA	3-furanmethanol (83%)	NA
Aromatics with N	Pyridine (91%)	Pyridine (91%)	Pyridine (91%)	Pyridine (91%)	Pyridine (91%)
Aromatics	Benzene (87%) Ethylbenzene (91%) P-xylene (94%), O-xylene (93%), 1,3-dimethylbenzene (90%)	Benzene (83%) Ethylbenzene (90%) P-xylene (87%) O-xylene (87%)	Benzene (80%), Ethylbenzene (91%), P-xylene (93%)	Ethylbenzene (91%), P -xylene (94%), 1,3-dimethylbenzene (93%)	Benzene (80%) Ethylbenzene (91%) 1,3-dimethylbenzene (90%) P -xylene (87%) O-xylene (87%)
Phenols	Phenol (91%) Creosol (97%) 2-methoxy phenol (95%), 2,6-dimethoxy phenol (89%),	Phenol (91%) 2,4-bis(1,1-dimethylethyl)-phenol (81%) Cresol (93%) 2-methoxy phenol (95%), 2,6-dimethoxy phenol (89%),	2,4-bis(1,1-dimethylethyl)-phenol (94%)	Phenol (91%) Mequinol (87%) 2-methoxy phenol (94%)	Phenol (91%), 2,4-bis(1,1-dimethylethyl)-phenol (89%) Creosol (87%) Mequinol (90%) 2-methoxy phenol (93%)