

Pyrolysis of Corncob Waste to Produce Liquid Smoke

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Abstract. Corn cobs are the main waste of corn farming. Corncobs biomass waste chemically contains carbon and hydrogen which can be pyrolysed to produce liquid smoke for food preservation. The aim of this study is to observe the effects of pyrolysis time and temperature as well as the particle size of corncobs to the yield of pyrolysis products. The discussion is limited to liquid smoke products. The experiments were performed by pyrolysing sun-dried corncobs at a certain weight and particle size in a slow heating rate pyrolysis equipment at a targeted residence time and temperature. The results of this study showed that the optimum yield of the liquid smoke was 48% and achieved at an optimum time, temperature and particle size of corncobs of 2.5 hours, 400 °C and 1 inch, respectively. The effect of pyrolysis time (t) on the yield of liquid smoke (Y) from corn cobs in this study could be represented quite well by an empirical equation of $Y (\%) = 33.57 + 0.024t + 9.10 \cdot 10^{-4}t^2 - 4.10 \cdot 10^{-6}t^3$. The empirical equation of the effect of pyrolysis temperature (T) on liquid smoke yield (Y) could be quite well represented by the equation $Y (\%) = 21.19 + 0.071T + 6.10 \cdot 10^{-5}T^2 - 2.10 \cdot 10^{-7}T^3$. The characteristics of the liquid smoke produced from this study have a density of 1.05 g / mL, pH of 1.9, brown-red color, and corn-smoke like smell.

Keywords: liquid smoke, pyrolysis, corn cobs

1. Introduction

Corn farming products are one of the second most significant sources of carbohydrates after rice in Indonesia. Corn is mainly consumed as food. It is also used as feed for livestock and raw materials of chemical and pharmaceutical industries. Corncobs are considered as a waste from the corn farming, the waste is accounted approximately 20% of the corn production. The production of corn in 2017 was ca 11 million tons [1], therefore the corncob waste was ca 2.2 million tons. Corncob biomass waste is used mainly as animal feed or compost fertilizer. Corncobs contain carbon and hydrogen in the form of furfural and lignocellulosic compounds. The lignocellulosics are as a combination of cellulose, hemicellulose, and lignin [2]. The content of lignocellulosic compounds in corncobs makes this waste as a potential source to be utilized as a source of energy and a raw material for liquid smoke through pyrolysis [3]. Biomass pyrolysis is a thermochemical process in the absence of oxygen at a specified heating rate to transform solid biomass into solid bio-char, liquid smoke and pyrolysis gas [4]. During pyrolysis process, the long chains of carbon, hydrogen and oxygen compounds in biomass thermally decompose into smaller molecules, releasing a vapour phase and a residual solid bio-char phase.

The pyrolysis vapour is then cooled down to condense out polar and high molecular weight compounds as liquid (liquid smoke) while low molecular weight volatile compounds remain in the gas phase [5]. The pyrolysis products have many applications as energy sources, raw materials for industries and other industrial applications. For example, bio-char of corncob pyrolysis can be made into bio-briquettes, an alternative solid fuels for domestic and industry, or activated carbon adsorbents for water purification or waste treatment processes.



Liquid smoke has wide applications and is usually produced through a slow pyrolysis process [6]. Slow pyrolysis is run at a temperature range of 300-700 °C with a slow heating rate [7]. The yield of liquid smoke is dependent of raw materials and pyrolysis operating conditions. The liquid smoke could be utilized into different grades, namely grade 1, 2 or 3 [8]. Liquid smoke may act as a food additive, either as an antioxidant or preservative or as a flavor that can increase the aroma and taste of food or preserved food products [9]. Thus, liquid smoke could be use as an alternative to conventional meat preservation products. Liquid smoke has antioxidant effects, resulting in inhibitory effects on the growth of *Escherichia coli*, *Salmonella choleraesuis*, *Staphylococcus aureus* and *Listeria monocytogenes* bacteria. The use of liquid smoke in meat preservation is reported to reduce a processing time by about 43%, thereby significantly reducing the cost of meat processing. Processing of meat preservation with liquid smoke will be safer, economical and yielding more uniform yet qualified processed products [10]. The compounds in liquid smoke are determined by the raw materials and thermal degradation process. The compounds could be identified as carbohydrates such as ketones, carbonyls, acids, furans, and pyran. Liquid smoke is also contained components derived from lignin thermal degradation, such as phenol, guaiacol and its derivatives, syringols and derivatives and also alkyl aryl, with phenol being the most significant component in liquid smoke [11].

This study is focused on slow pyrolysis of corncobs with the aims to study the effects of pyrolysis time and temperature as well as particle size into the yields of liquid pyrolysis. It is expected that the research could provide important informations on the optimum conditions of corncob pyrolysis to produce liquid smoke. The liquid smoke then later will be implemented in food industries as a food preservative..

2. Methodology

2.1. Materials and Equipment

The pyrolysis equipment used in this was specifically designed to produce char and liquid smoke products simultaneously. The pyrolysis devices (Figure 1) comprises of reactors, condensers, control temperatures, thermocouples, pressurized gas stoves, gas-fueled containers, tar containers, liquid smoke containers, cooling water circulation pumps and cooling water containers. The pyrolysis reactor and tcondenser 1 were made of stainless steel, in which the reactor was designed to prevent there is no air flow back into the reactor. Nitrogen was flown into the reactor to and the gas with volatile gases were driven out from the reactor through a small tube. The reactor walls were double-layered walls. Between the two walls, there was a slightly- spaced annulus hole. This hole has a role to stabilize and maximize the heating process. The reactor was designed with the principle of energy-saving, safe and environmentally-friendly.

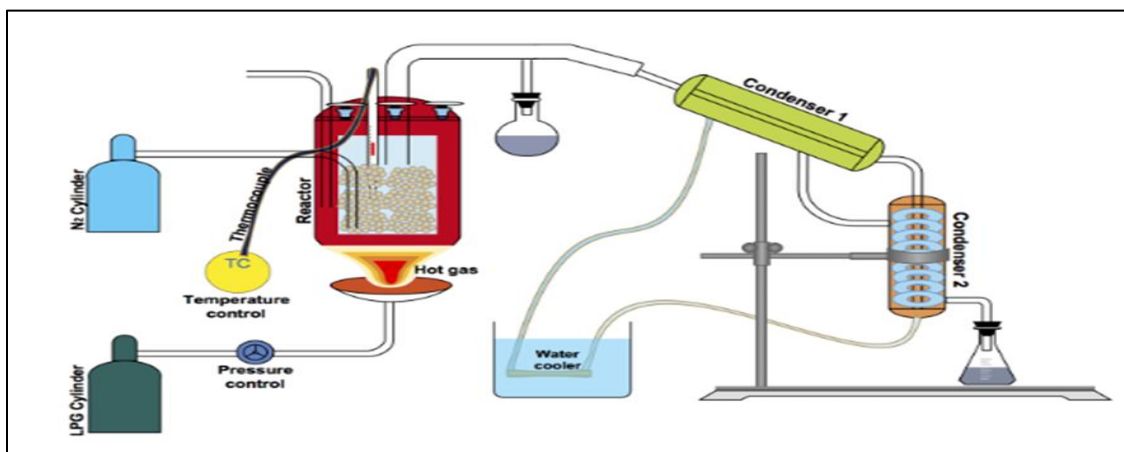


Figure 1. A set of pyrolysis equipment

Raw materials used in this research were corncobs obtained from Bone Regency, South Sulawesi, Indonesia. The fresh corncobs were cleaned from impurities and then sundried for three (3) days. The dried corncobs are then chopped to large size (± 1.5 inches), medium size (± 1 inch) and small size ($\pm 3/8$ inches) using sieves (0.1-inch equivalent 20 mesh).. Then the materials were kept in an airtight container.

2.2. Research Procedures

The dried and chopped raw material of corn cobs were weighed for 1 Kg, then the cobs were put into the reactor which was then closed tightly by tightening all the bolts so there was no air-leaking. The thermocouple wire was connected to the reactor through the available holes. The condensers, the hose and the liquid smoke container were ensured to be correctly installed. The pyrolysis process was started by turning the pressurized gas stove on and the temperature was set at targeted temperature. It was important to maintain the temperature of cooling water flowing through the condenser at maximum 20 ° C. The pyrolysis time was started from when the target temperature was reached. Once the pyrolysis completed, the liquid smoke was filtered using Whatman filter paper No. 10. The filtered liquid smoke was then measured and characterized. The pyrolysis processes were performed at various pyrolysis times, namely 30, 60, 90, 120, 150 and 180 min, respectively; pyrolysis temperature of 300, 350, 400, 450 and 500 oC, respectively; and corncob raw material particle sizes of rough and smooth.

3. Results and Discussion

3.1. Effect of pyrolysis time

To study effects of temperatures to the yield of liquid smoke, slow pyrolysis was performed at a temperature of 300 °C at pyrolysis time ranges of 30 - 180 minutes. The results are presented in Figure 2.

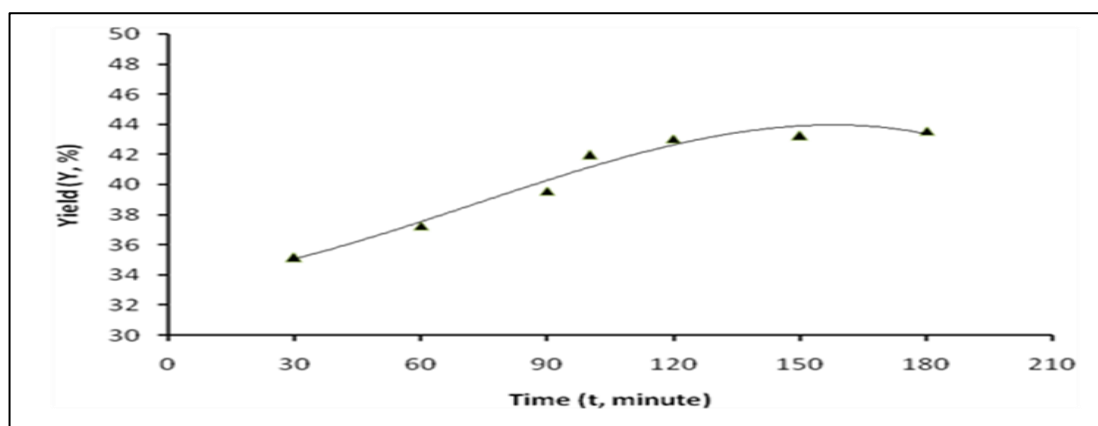


Figure 2. Effect of pyrolysis time on the yield liquid smoke product

It can be seen from Fig. 2 that the yield of liquid smoke increases linearly with increase in pyrolysis time from 30 minutes to 120 minutes. However, above 120 minutes, the increase in pyrolysis does not significantly increase the liquid smoke yield. The yield of liquid smoke at pyrolysis time of 120 minutes was 43.05% and the yield at 180 minutes only give a slight increase to 43.58%. Extending the pyrolysis time above 120 minutes is considered economically not viable, since extending the length of the pyrolysis would require more energy to be supplied into the process. For the case of extending pyrolysis time from 120 minutes to 180 minutes, there was only 0.53% increase in the liquid pyrolysis. Thus, from the results of this study, it is suggested that the optimum pyrolysis time was 120 minutes.

Based on Fig.2, the relationship between pyrolysis time (t) and the liquid smoke yield (Y) of corncob slow pyrolysis at 300oC could be predicted using a 3rd order polynomial equation as follows:
 $Y (\%) = 33.57 + 0.024t + 9.10 \cdot 10^{-4}t^2 - 4.10 \cdot 10^{-6}t^3$ (Eq.1)

The equation (Eq.1) is satisfactory enough to predict the liquid smoke yield in the range of pyrolysis time of 30 - 180 minutes, as indicated by the value of the correlation coefficient which is very close to 1 ($R^2 = 0.970$), the value of the revision which is quite small (1.1%) and the sum of squared errors which is also quite small ($SSE = 2.14$).

3.2. Effect of temperature

The optimum pyrolysis time obtained from this study was 120 minutes. Based on this optimum time, a study on the effects of pyrolysis temperatures to the yield of liquid smoke was performed. The pyrolysis temperatures studied were in the range of 300 - 500 °C using a medium particle size of corncobs. The results of the study are at Figure 3.

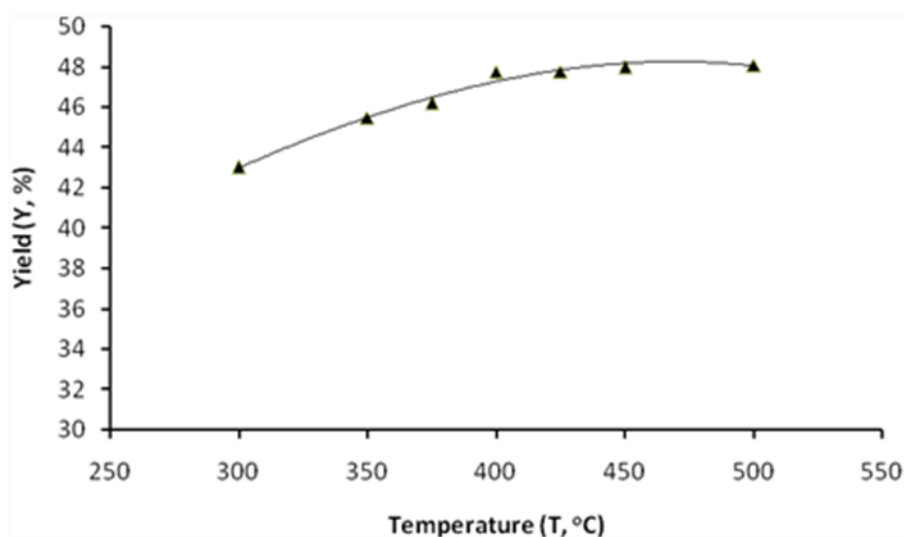


Figure 3. Effect of pyrolysis temperature on liquid smoke product (yield)

It can be seen from Fig. 3 that the yield of the liquid smoke reached 43% at 300 oC. Increasing the pyrolysis temperatures from 300 ° C to 400 ° C gives a significant increase in the yield of smoke liquid. However, further increased the temperature to 500 °C only slightly increases the yield from 47.78% to 48.09%. It has been reported that temperature has played a significant effect on the yield of pyrolysis liquid. During pyrolysis of pine sawdust in a fixed bed reactor , it was found that the maximum pyrolysis liquid yield was at 550°C [12]. It was also reported in the study that the condensable gas decreased and non-condensable gas increased with increasing temperature during pyrolysis.

In a practical implementation, although the maximum yield of liquid smoke was obtained at 500oC at this research, however to produce economical liquid smoke it is suggested that the pyrolysis should be run at 400oC. The energy which would be consumed to increase the temperature from 400oC to 500oC would not be significant with the increase in the yield of liquid smoke.

Based on Fig.3, a profile of the effect of temperature (T) on the yield of liquid smoke (Y) from slow pyrolysis of a medium size of corncobs was predicted using a 3rd order polynomial equation. The equation is as follows:

$$Y (\%) = 21.19 + 0.071T + 6.10 \cdot 10^{-5}T^2 - 2.10 \cdot 10^{-7}T^3$$
(Eq.2)

Eq.2 is satisfactory enough to predict slow pyrolysis process in the temperature range of 300 - 500 oC. The equation has the value of correlation coefficient of 0.981, the value of the revision 1.92% and the sum of squared error of 6.56.

3.3. Effect of material size

Based on the optimum time and temperature of pyrolysis of corn cobs biomass which has been obtained in the previous sections, effects of particle sizes of corncobs on the yield of liquid smoke during slow pyrolysis were studied. Three particle sizes, namely rough size (± 1.5 inch), medium size (± 1 inch) and fine size ($\pm 3/8$ inch), were chosen. The results are presented in Fig.4.

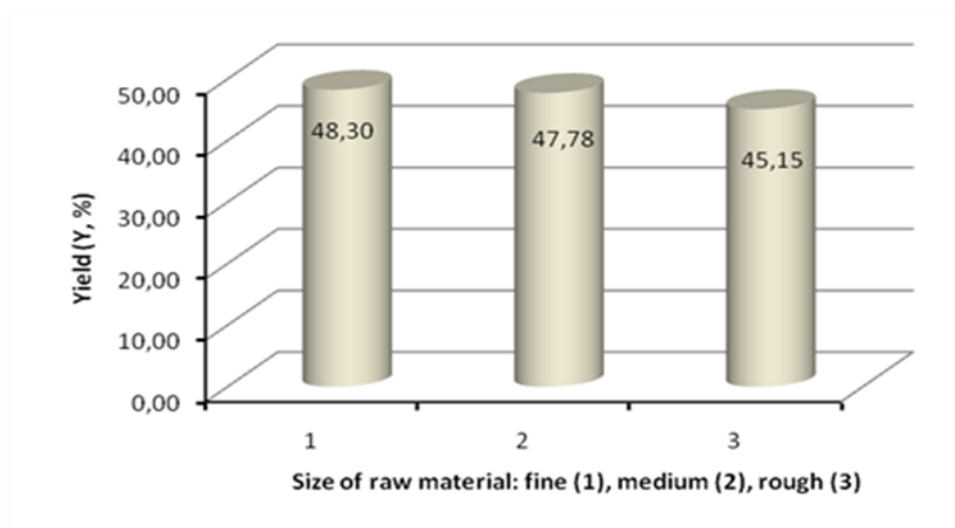


Figure 4. Effect of raw material particle size on liquid smoke yield

It can be seen from Fig.4 that the yield of liquid smoke increases with the decrease in particle sizes. The yield of liquid smoke using a rough particle was 45.15%. This result was relatively lower than if using medium and fine materials which were 47.78 and 48.30%, respectively. It was reported that during slow pyrolysis of woody biomass, the yield of pyrolysis liquid decreased with increase in particle size (Benhajji, 2014). In a large particle, during devolatilization process, the volatiles have a longer residence time than in a smaller particle. The condensable volatiles may then trap inside the solid char, resulting a lower yield of liquid smoke.

It is observed that a smaller particle size produces a higher liquid smoke yield. However, reducing the particle size of the material requires energy. Therefore, for a practical application, it is suggested that the optimum particle size for producing liquid smoke from corncobs was the medium particle size. The characteristics of the liquid smoke is presented at Table 1. Based on the characteristics, the liquid smoke is categorised as grade 3 liquid smoke [13].

Table 1 Characteristics of liquid smoke

Characteristics	
Density	1.05 g/mL
pH	1.9
Color	Brownish red
Smell	corn-like smell

Grade 3 liquid smoke may be used as a raw material for insecticides. Grade 3 liquid may be further processed to produce a higher quality of liquid smoke (grade 2 and 1) via distillation and other purification processes. The higher quality of liquid smoke is expected to have a better application in food industries.

4. Conclusions

Slow pyrolysis of corncobs has been performed to produce liquid smoke. It was observed that the optimum yield of liquid smoke was achieved at the following operating conditions, namely temperature of 400 oC, a medium size particle size of ca 1 inch and pyrolysis time of 120 minutes, respectively. The optimum yield of the liquid smoke was 48%. The liquid smoke was categorized as a grade 3 liquid smoke which could be directly applied as a raw material for pesticides industries and could be further processed to yield higher quality liquid smoke.

5. Acknowledgement

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6. References

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