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Article

Bioactivity of liquid smoke of rice husk against *Spodoptera exigua*

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Abstract

The characteristics and bioactivity of liquid smoke produced from rice husk as antifeedant activity against *Spodoptera exigua* larvae were investigated. The carbonation process of rice husk was conducted into low heating rate (100-450^{o} C). The organic components in the liquid smoke were analyzed to determine the major composition. Bioassay of *S. exigua* was carried out by using crude liquid smoke and its fractions to find out the feeding inhibitory. The feeding inhibitory of *S. exigua* on liquid smoke fraction increased significantly with increasing temperature of the carbonation process. It was clearly observed that liquid smoke fraction at 250-350^{o} C was the most effective as antifeedant against *S. exigua*. In addition, two major toxic compounds, i.e., phenolic and acid were much concentrated in that fraction. We suggest that those compounds might responsible to feeding inhibitory activities of *S. exigua* larvae.

Keywords : *liquid smoke, rice husk, Spodoptera exigua, antifeedant activity*

Introduction

Carbonation process is decomposition of lignocellulosic biomass components, i.e., cellulose, hemicelluloses and lignin to generate liquid smoke. Such process can be divided into tree modes based on heating rate: fast, intermediate, and slow (Wu et al., 2015). Zhai et al. (2015) also reported that heating temperature influenced the proportion of yielded compound in pyrolysis.

Some earlier studies reported that liquid smoke has an antifeedant activity (Wu et al., 2015; Yatagai et al., 2002). Gani et al. (2012) identified γ-butyrolactone as antifeedant compound from the liquid smoke of organic waste. The compound caused detrimental effect against *Spodoptera litura* larvae. Research on antifeedant activity of plant extracts have been immensely conducted. Chinnamani and Jeyasankar (2018) reported that *Pseudocalymma alliaceum, Solanum pseudocapsicum* and *Barleria buxifolia* showed significant antifeedant activity against the fourth instar larvae of *S. litura* and *Helicoverpa armigera.* Some antifeedant compounds have been isolated from *Gnaphalium affine* (Morimoto et al., 2003). Arivoli and Tennyson (2012) also reported the antifeedant activity in hexane washed leaf extracts of *Zanthoxylum limonella* against *S. litura* (77.52%).

On the other hand, rice husk is abundantly, and typically of lignocellulosic biomass, rice husk can be used as a source of liquid smoke. Besides that rice husk is an organic waste that has not been fully utilized. Tajali (2015) reported that chemical contents of rice husk are carbon (charcoal) 1.33%, hydrogen 1.54%, oxygen 33.64% and silica 16.98%. Based on the literature survey, little is known regarding the bioactivity of liquid smoke produced from rice husk against pest insects. Therefore, the present study was conducted to assess the bioactivity of liquid smoke produced from rice husk as antifeedant for *S. exigua* larvae.

Material and Methods

Liquid smoke production

In this research, rice husk was used as raw material to produce liquid smoke. The carbonation process was carried out using a pyrolysis reactor. Rice husk sample was weighed and then put into pyrolysis reactor. The pyrolysis reactor was closed tightly. The pyrolysis process has lasted for 4 hours at temperatures between 100–450°C. Liquid smoke was accommodated in the three temperature ranges, i.e., 100-250 °C, 250-350°C and 350-450°C.

Total phenolic content

One mL of liquid smoke was diluted in 500 mL water. One ml of dilution concentration and 5 mL of 15% sodium carbonate were applied to a test tube, and allowed to stand at room temperature for 10 minutes. Folin-Ciocalteau reagent (0.5 mL) was then added and mixed with vortex - shaker. The solution was incubated at room temperature for 30 minutes. UV - VIS Spectrophotometer was used to measure absorbance of samples at 750 wavelengths. Phenolic content was calculated based on a standard curve of pure phenol solution.

Total acid content

One mL of liquid smoke was put into 100 mL of distilled water, then homogenized. Phenoptaline indicator (0.3 mL) was added into the solutions and titrated with 0.1 N NaOH. Titration was stopped when color changes in solution from clear yellow to yellowish brown color. The total acid content was calculated by the following formula.

$$\text{Total acid (\%)} = \frac{(V \times N \times MW) \times 100}{(SW \times 1000)}$$

Where:

V : volume (mL)
N : normality of NaOH (N)
MW : molecular weight of acetic acid (g/mol)
SW : weight of sample (g)

Bioassays against S.exigua larvae

Bioassay used in this experiment is a modified method developed by Arivoli and Tennyson (2012). Onion leaves (15 x 20 mm) were used for bioassay test. The leaves were dipped into the solution for 20 seconds and air dried at room temperature. Two pieces of onion leaves (*Allium cepa*), i.e., treated and untreated were put into the Petri dish. One of third instar larvae of *S.exigua* was then placed in each dish after being starved for 2 hours. Observations were conducted after 24 hours by measuring the consumed leaf area with millimeter block. The percentage of antifeedant activity was calculated by the feeding inhibitory formula:

$$\text{FI (\%)} = \frac{\text{(leaf area consumed (control - treatment))}}{\text{(leaf area consumed (control + treatment))}}$$

FI = Feeding inhibitory activities

Table 1. The yield of rice husk liquid smoke

Sample Weight (gram)	Temp (°C)	Volume (ml)	Yield (%)	pH	Colour
1468.65	100-250	315	29.90	-	-
	250-350	30	2.98	2.77	Brown
	350-450	23	2.09	3.05	Dark Brown

Results and Discussion

In this study, we first carbonated of rice husk in low moisture content (8.13%) to maximize the liquid smoke`s yield. We applied around 1064 g of rice husk as maximum capacity of a pirolisator, and allowed the pyrolysis process for 4-5 hours. As mentioned above, the carbonation was divided into three fractions based on the differences of temperature, i.e., at 100-250°C, 250-350°C and 350-450°C. Our data demonstrated that the third fraction produced different characteristic of liquid smoke with more intense color of the liquid smoke or darkening (Table 1). We speculate that this phenomenon may be induced by phenol and tar content, in which phenol and tar content in the third fraction likely higher than that in the other fractions. Supporting this, an earlier study suggested that phenol and tar are the result of lignin decomposition at high temperature (Loo A.Y, 2008).

As shown in Table 1, the largest liquid smoke was produced at 100-250°C (29.9%) while the smallest liquid smoke was yielded at 350-450 °C (2.09%). This condition was generated by evaporation of water and degradation of cellulose and hemicellulose, as major compounds of rice husk, at 100-250 °C, Rice husk consists of three major components, i.e., cellulose, hemicelluloses, and lignin. Since the degradation of hemicellulose, cellulose and lignin occurred at 200-280°C, 280-320°C and 320-450°C, respectively (Loo, 2008), the highest yield of liquid smoke was possibly produced at 200-250°C. In carbonation process, the cellulose produced acetic acid, hemicellulose produced acetic acid, furan and furfural while lignin generated phenol, phenol derivatives and tar formation (Darmadji, 1996).

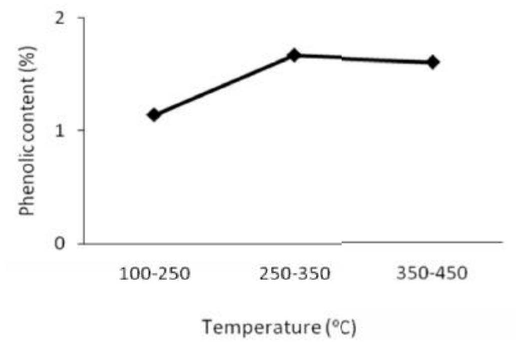

Figure1. Total phenolic content of liquid smoke

Thus, it has been well understood that the determination of liquid smoke`s yield is the one of the most important parameters to determine the output of the process (Prianto, 2015). The present study noted that total yield of carbonation process was 34.65%. Gani *et al.* (2007) revealed that the amount yield of liquid smoke produced in the carbonation process depends on the process conditions and the type of used raw materials. In addition, percentage of yields obtained from the carbonation are also highly depend on carbonation temperature and condensation systems (Wijaya et al., 2008), therefore the high of carbonation temperatures may resulted in reducing the liquid smoke. Besides that, our liquid smoke`s yield is consistent with the previous study by Halim et al. (2004). In this study, we also observed the degree of acidity (pH) of liquid smoke. The result showed that pH of liquid smoke was 2.77 to 3.11 (Table 1). Wijaya et al. (2008) reported that low pH value indicate the high quality of liquid smoke especially in its use as a preservative.

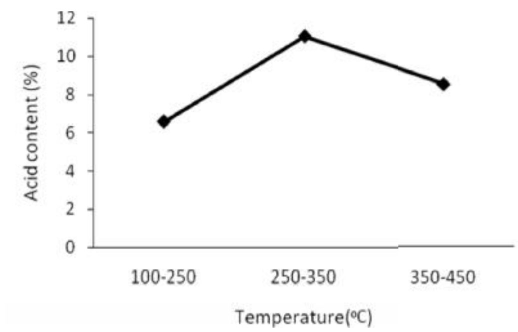

Figure 2. Acid content of liquid smoke

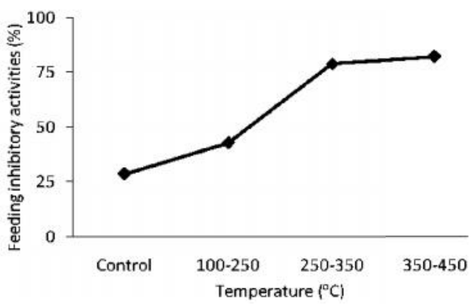

Figure 3. Feeding inhibitory activities

Next, we analyzed the chemical compounds in the liquid smoke produced from rice husk. The present study detected two major compounds in the liquid smoke, i.e., phenolic and acid compounds (Figs. 1 and 2). Our data noted that 1.6% phenolic, and 11.03% acid compounds were consisted in the liquid smoke at 250-350°C (Fig. 1 and Fig. 2). We also detected that largest acid compound in the liquid smoke is acetic acid. Yatagai et al. (2002) suggested that acid acetic acid is the largest component in the liquid smoke. Another study by Mustikawati et al. (2016) also reported that cconcentration of acetic acid and phenol in liquid smoke from rice husk were 72.22%, and 12.39%.

Lastly, we tested the liquid smoke and its fractions against *S.exigua* larvae. Our data presented here, showed that the highest of feeding inhibitory activities occurred at 250-350°C with time observation was 24 hours (91.71%) (Fig. 3). As discused above that phenolic and acid compounds are consisted in liquid smoke with carbonation process at 250-350°C. We suggest that feeding inhibitory activities of *S.exigua* were caused by the presence of phenolic compounds and organic acids, and the synergistic effect of phenolic compounds with organic acids has a higher effect in inhibiting feeding level. Supporting this, some earlier studies suggested that phenolic and acid compounds have detrimental effect to pest insect (Yatagai, 2002). Liquid smoke contains some compounds that are suspected to be toxic, repellent and antifeedant activity such as phenols and organic acids, so that liquid smoke has an opportunity as a biopesticide to control pests (Ma et al., 2011; Prianto, 2015; Yatagai et al., 2002). Gani (2007) also reported that methanol and water fractions of liquid smoke have potential as antifeedant activity more than 50%.

Conclusions

Overall, the feeding of *S. exigua* on liquid smoke fraction increased significantly with increasing temperature of carbonation. We observed that liquid smoke fraction at 250-350°C was the most effective as antifeedant against *S. exiqua* with 94.71% of the feeding inhibitory. In addition, our chemical components analysis clearly confirmed that two major compounds i.e., phenolic and acid compounds are consisted in the liquid smoke. We suggest that those compounds have synergetic effect in the feeding inhibitory of *S. exigua*. This finding contributes to improve the value-added of rice husk as a source of liquid smoke as agent for controlling pest insects.

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