Natural biopesticide from liquid rice hull smoke to control brown planthopper

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Abstract. Liquid smoke from rice hull has the potential to be developed as a natural pesticide, because of its contents of chemical compounds which is good for controlling insects. The purpose of this research was to find out the best formula from liquid rice hull smoke to control brown planthopper. The methodology used through several stages of activities, namely (1) purification of liquid smoke, (2) formulation of natural pesticide with liquid smoke as active compound, synergistic, adjuvant, and solvent; (3) toxicity of formula and (4) bioassay tested of formula to brown planthopper in the laboratory. The formulation was arranged completely randomized design with three replications. The results showed that 7 formulas of natural pesticide based on liquid rice hull smoke were highly effective to control brown planthopper assessed by mortality test. Furthermore, the toxicity test on those formulas has toxicity (LD₅₀) ranging from 128 to 725 ppm. All formulas are still categorized as toxic and can use as a pesticide. Mortality test results with 3 variations of concentration, i.e., 40%, 30% and 10% showed that the best results were in a concentration of 10%. In this case, for 7 days observations, pesticides with a formula of F1, F2 and F3 killed more than 90% of brown planthopper.

1. Introduction

Natural pesticides are natural products that are effective against bacteria, fungi, nematodes, viruses, and insect pests, that are derived from a natural source such as plant, animal, bacteria, and certain minerals [1][2]. The active compound of biopesticides is usually formulated in the same way as synthetic pesticides. Besides, it usually uses the same equipment for making the formulation [3]. One promising active compound to be formulated as a natural pesticide is liquid rice hull smoke. Liquid rice hull smoke was obtained from the condensation process and decomposition of organic compounds contained in the rice hull through the pyrolysis process. Pyrolysis is the process of organic material decomposition or complex compounds into substances in three forms, namely solids, liquids and gases. The use of liquid smoke as pest control has been reported by several researchers. Mortality test results with 3 variations of concentration, i.e., 40%, 30% and 10% showed that the best results were in a concentration of 10%. In this case, for 7 days observations, pesticides with a formula of F1, F2 and F3 killed more than 90% of brown planthopper.

The chemical components of liquid smoke of rice husks was acetic acid (33.15%), benzenoid 4-hydroxy (32.77%), benzeno 1,2-dimethoxy (2, 83%), 2-methoxyl-4-
Materials and Methods

2.1. Distillation of rice husk liquid smoke

For purification of liquid smoke 3 to grade 2, it was carried out by distillation. Liquid smoke was poured into a stainless-steel tank, then heated until it reaches its boiling point. The steam was condensed by a cooler, then collected as a grade 2 pure liquid smoke distillate.

2.2. Formulation of liquid smoke

The biopesticide was formulated in the form of an emulsifiable concentrate (EC). As a rule, emulsions usually have a “milky” appearance. The composition of natural biopesticide can be seen in Table 1. The liquid smoke was prepared by (1) preparing tween 20, clove/lemongrass oil, teepol, and oleic acid in a container, (2) mixing well by stirring the ingredient for 10 minutes at a speed of 300-500 rpm, (3) adding liquid smoke to the droplets while stirring using an electric stirrer at a speed of 300-500 rpm, (4) continuing the agitation at a speed of 300-500 rpm for 10 minutes. The quality of formulas was determined by specific gravity, pH, phenol content and LD50.

**Table 1.** The composition of natural biopesticide formula.

| Composition (%) | F1 | F2 | F3 | F4 | F5 | F6 | F7 |
|-----------------|----|----|----|----|----|----|----|
| Tween 20        | 5  | 5  | 5  | 5  | 5  | 5  | 5  |
| Glycerol        | 5  | 5  | 5  | 5  | -  | -  | -  |
| Teepol          | 5  | 5  | 5  | 5  | 10 | -  | 5  |
| Clove oil       | 10 | 10 | -  | -  | -  | -  | -  |
| Lemongrass oil  | -  | -  | 10 | 10 | -  | -  | -  |
| Oleic acid      | 5  | 5  | 5  | 5  | 5  | 5  | 5  |
| Liquid smoke grade 3 | - | 40 | 40 | -  | 75 | 75 | 75 |
| Liquid smoke grade 2 | 60 | -  | -  | 40 | -  | -  | -  |
| Solvent         | 10 | 30 | 30 | 30 | 5  | 5  | 10 |

2.3. Toxicity of formula

Toxicity of formula was carried out on freshly hatched brine shrimp (Artemia salina Leach) [12]. The LD50 was calculated based on the toxicity effect analyzed from observations with the percentage of deaths on 50%.

2.4. Bioassay for laboratory testing

Application of formula was carried out by preparing plants and insects to test plant-based pesticides against brown planthopper at the gauze house. Rice seeds are sown in a plastic tray, seedlings aged 14 days after the seedlings are planted in a plastic bucket (diameter 35 cm) containing a mixture of soil and fertilizer. Rice plants that were used in this research from TN1 varieties after 30 days of planting. The brown planthopper insects used in this study were taken from Sukamandi field population that has been nurtured and propagated in a greenhouse. Eggs and brown planthopper nymphs were nurtured and propagated on rice plants placed in mylar plastic cages. Mylar plastic sides are perforated by 10 x 10. When the plants have wilted and dried out, they were replaced with new plants which were put in confinement and then covered with gauze cloth and at the top also covered with a gauze cloth. The brown planthopper nymph used in this study was an instar-4 nymph. A total of 100 planthoppers were put in a cage and sprayed with the test material. The number of insect mortality was calculated using Abbott’s formula by counting the
number of dead insects in each gauze house at 24 hrs, 48 hrs, 72 hrs, 96 hrs, 120 hrs and 168 hrs after treatment.

Abbot’s formula: \[ P_t = \frac{P_0 - P_c}{100 - P_c} \times 100\% \] (1)

where \( P_t \) = percent (%) mortality; \( P_0 \) = observed mortality; \( P_c \) = control mortality

3. Results and Discussion

3.1. Quality of formula

In the process of making liquid rice hull smoke, the resulting combustion temperature was reached to 357°C, the burning process time 3-4 days, the condensate flow rate in 1 minute was 57 ml, and the yield ranging from 22.12% to 28%. Purification of liquid rice hull smoke from grade 3 to grade 2 was carried out by distillation, in term to remove undesirable and hazardous compounds such as polyaromatic hydrocarbons (PAH) and tar [13].

The 7 natural pesticides have been formulated from liquid rice hull smoke with the types of emulsifiable concentrate/EC (Figure 1). The EC formula can make it easier in the application, high level of active ingredients, and high biological activity [13]. A formula which uses the liquid rice hull smoke grade 2 has a milky appearance. This is because liquid rice hull smoke grade 2 has a clearer colour compared with grade 3 (black colour).

![Figure 1. The natural biopesticides with different formulas](image)

As seen in Table 2 that every formula of natural biopesticide has different pH, phenol content and LD50 values. The F1 formula has the lowest pH, the highest phenol content and the lowest LD50 compared to other formulas. Its phenol content was the highest due to the addition of clove oil which contains phenol compounds. LD50 with the lowest value comes from the F1 formula, followed by F3, and F2, which is 128.48; 271.93 and 418.91 ppm. If the LD50 has valued around 500 and 1000 µg/ml, this is considered weakly toxic. Those between 100 and 500 µg/ml as moderately toxic, and those <100 µg/ml as strongly toxic [14]. The formulas in this study indicated moderately toxic for F1, F2, and F3, while the others included in weakly toxic. On the other hand, the toxicity not only from the LD50 value but also from phenol content. The F5 formula showed the highest values probably affected by liquid rice hull smoke with teepol and oleic acid.

| Formula | pH  | Density (g/l) | Phenol content (%) | LD50 (ppm) |
|---------|-----|---------------|--------------------|------------|
| F1      | 1.43| 1.02          | 10.12              | 128.48     |
| F2      | 2.80| 1.03          | 9.97               | 418.91     |
| F3      | 2.69| 1.03          | 0.62               | 271.93     |
| F4      | 1.57| 1.01          | 0.18               | 644.96     |
| F5      | 2.74| 1.07          | 1.07               | 725.82     |
| F6      | 4.64| 1.06          | 0.95               | 511.38     |
| F7      | 2.44| 1.03          | 3.02               | 560.36     |
3.2. Laboratory test to brown planthopper

All tested formulas showed high efficiency in mortality of the tested pests (Figure 2a). Besides, significant differences in efficiency were also found. In a 40% concentration, the highest efficiency was F1, and F2 formula with 100% mortality on 1st day and followed by F3 and F7 on 2nd days. This means that those concentrations were very toxic. The other formulas tend to be similar. It can be seen in the rice plant (Figure 3), that the plants looked yellow and dry. This is because of insect mortality effect of natural biopesticide was depended on dose formulations [15]. Furthermore, if the concentration of exposure was reduced to 30% (Figure 2b) that it has not significantly different from the 40% concentration. The mortality of the insect is still too high.

![Figure 2. Mortality for concentration of (a) 40% and (b) 30%](image)

![Figure 3. The mortality test of brown planthopper in various formulas at 40% concentration.](image)

Then, the concentration of exposure was reduced to 10% concentration. The mortality of brown planthopper was shown a significant difference in 1st application Table 3. The mortality rates of the brown planthopper have appeared on day 3 reaching 80% and continued to day 7 achieving more than 90%. It means that the F1, F2, and F7 are very effective by the mortality of the brown planthopper. These were correlated with phenol content in that formula higher than other formulas. Phenol is known as a toxic substance that can be used as an insecticide. According to Soediyo [9] that brown planthopper nymph death is caused by the presence of phenolic compounds.
Liquid smoke may synergize with clove oil, this is indicated by the increase in phenol levels. There was an increase in phenol levels in the formula because clove oil also contains high phenolic compounds Table 1. Furthermore, it was also found out that the mortality effect of formulas was dependent on the dose of concentration [15].

### 4. Conclusions

The formula based on liquid rice hull smoke has types of emulsifiable concentrate with the form milky appearance. In the laboratory tested to brown planthopper, the mortality test was observed in 7 days. The best efficacy was found at 10 % concentration with the best formula F1, F2, and F7. These correlated with phenol content which is ranging from 3.02 to 10.12%. Based on the laboratory that treatment, for the formula of F1, F2 and F7 will be beneficial to control brown planthopper in the field with 10% concentration.

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**Table 3.** Percentage of the death of the brown planthopper after application with 10% concentration.

| Formula | Percentage mortality brown planthopper on observation (%) (days) |
|---------|---------------------------------------------------------------|
|         | 1               | 2               | 3               | 4               | 5               | 6               | 7               |
| F1      | 64.33e           | 80.00c           | 83.70bc         | 88.00de         | 89.00bc         | 89.33cd         | 92.67b          |
| F2      | 75.00d           | 87.00b           | 89.30b          | 91.00cd         | 91.67b          | 92.67bc         | 95.33b          |
| F3      | 67.33e           | 71.33            | 79.30c          | 82.70e          | 85.33c          | 87.00d          | 87.67c          |
| F4      | 50.67f           | 55.00e           | 62.00eh         | 64.30g          | 65.00e          | 65.33f          | 74.33d          |
| F5      | 15.00h           | 16.67h           | 33.00d          | 42.70j          | 46.67h          | 50.00h          | 56.67h          |
| F6      | 65.00e           | 69.33d           | 71.70b          | 74.70f          | 79.67d          | 80.67e          | 85.33c          |
| F7      | 84.33bc          | 87.33b           | 88.70b          | 91.70bcd        | 92.67b          | 93.33b          | 95b             |
| Control | 0.00i            | 0.00i            | 0.00k           | 0.00l           | 0.00j           | 0.00j           | 0.00i           |
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