Utilization of Liquid Smoke from Siwalan Fruit Skin Waste as an Organic Pesticide Against Armyworm Mortality

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Abstract
Pest attacks that result in reduced crop yields require alternative control that is able to prevent damage to agricultural land. Organic pesticides are environmentally friendly pesticides because the materials used are derived from natural ingredients, one of which is liquid smoke, namely liquid smoke made from natural ingredients containing cellulose, hemicellulose, and lignin. This study was to determine the effect of giving liquid smoke of Siwalan peel waste as an organic pesticide. Liquid smoke is obtained from the combustion process with a little oxygen at a certain temperature. The content of compounds in liquid smoke was analyzed using GCMS. Pyrolysis results obtained dark brown liquid smoke, clear, has a smoke-like odor (sangit), and has a watery texture which is used as an organic pesticide on Spodoptera litura with varying concentrations of 5%, 7%, 9%, 11%, and 13%. The results of the GCMS analysis found 18 components of the liquid smoke of Siwalan rind waste, among the compounds that function as organic pesticides are phenol, furfuran, acetone, and palmitic acid. The results of the analysis of the mortality of spodoptera litura sequentially showed the average value of mortality in 2 repetitions, namely 5%, 10%, 15%, 20%, and 35%. The greater the concentration of liquid smoke, the higher the mortality value. However, it is less effective to kill Spodoptera litura quickly, even mortality at a maximum concentration of only 35% or has not reached the death rate of 50% of test animals.

Keywords: Siwalan fruit skin waste, liquid smoke, organic pesticides, mortality, spodoptera litura

Abstrak
Serangan hama yang berakibat pada turunnya hasil panen membutuhkan alternatif pengendalian yang mampu mencegah kerusakan pada lahan pertanian. Pestisida organik merupakan pestisida yang ramah lingkungan karena bahan yang digunakan berasal dari bahan alami salah satunya yaitu asap cair, yaitu cairan asap yang terbuat dari bahan alami yang mengandung selulosa, hemiselulosa, dan lignin. Penelitian ini bertujuan untuk mengetahui pengaruh pemberian asap cair limbah kulit buah Siwalan sebagai pestisida organik. Asap cair diperoleh dari proses pembakaran dengan sedikit oksigen pada temperatur tertentu. Kandungan senyawa dalam asap cair dianalisis menggunakan GCMS. Hasil penelitian diperoleh asap cair berkarna coklat tua, jernih, memiliki bau seperti asap (sangit), dan memiliki tekstur encer yang digunakan sebagai pestisida organik pada ulat grayak dengan variasi konsentrasi 5%, 7%, 9%, 11%, dan 13%. Hasil Analisis GCMS didapatkan 18 komponen senyawa penyusun asap cair limbah kulit buah siwalan, diantaranya senyawa yang berfungsi sebagai pestisida organik adalah fenol, furfuran, aseton, dan asam palmitat. Hasil analisis mortalitas ulat grayak berurutan menunjukkan nilai rata-rata mortalitas dalam 2 kali pengulangan yaitu 5%, 10%, 15%, 20%, dan 35%. Semakin besar konsentrasi asap cair, nilai mortalitas semakin meningkat. Namun, kurang efektif untuk membumi ulat grayak secara cepat, bahkan mortalitas pada konsentrasi maksimal hanya 35% atau belum mencapai kematian 50% dari hewan uji.

Kata Kunci: limbah kulit buah siwalan, asap cair, pestisida organik, mortalitas, ulat grayak

1. Introduction
Pests and pathogens in agriculture can hamper productivity which results in loss of crop yields and decreased yield quality, so control is needed. Pesticides are toxic substances that function to kill living organisms that interfere with plants, livestock and so on. [1]. The widespread use of pesticides leaves residues that have been found to contaminate soil, drinking water, river water, well water, and air. This residue directly or indirectly causes land conditions in an environment to decrease because it is toxic.[2] Residual substances produced by the use of pesticides can cause biological magnification in the food chain. [3] The negative impact of using pesticides can be replaced with types of pesticides that come from environmentally friendly materials, such as organic pesticides that come from living things (plants, animals, or microorganisms). Liquid smoke is a liquid smoke vapor dispersion in water, or condensed liquid from
biomass that can be used as an organic pesticide, because it is antioxidative [4]. Liquid smoke is produced from the combustion of materials containing cellulose, hemicellulose, and lignin. This content will produce acid compounds, phenols, carbonyls, and other compounds that can kill nuisance organisms [5]. Based on the research of Isa, et al (2019), making organic pesticides using the coconut shell pyrolysis method, can produce liquid smoke products containing decomposed acids, phenols, and carbonyl compounds that are able to kill armyworms.

Siwalan fruit is a palm plant (areca nut) that grows a lot in Tuban Regency [6]. Currently, the use of Siwalan in the community is limited to fruit, leaves, stems, and flowers, so that a lot of Siwalan skin waste is collected into organic waste and then burned, while the skin of Siwalan fruit waste according to ref. [7] contains 68, 94% cellulose, 5.37% lignin, and 14.03% hemicellulose which have the potential to be used as raw materials for liquid smoke. [8]

Armyworm with the Latin name Spodoptera litura F is a type of leaf-eating pest that is very detrimental to farmers. Loss of crop yields due to this pest attack can reach 80% and if no control measures are taken, it can result in crop failure. This pest is reported to be able to attack many crops including vegetables (cabbage, chili, tomatoes, beans, tobacco, eggplant, potatoes) and food (rice, corn, peanuts, soybeans). Armyworms are spread in areas with hot and humid climates from the subtropics to the tropics such as Asia, the Pacific and Australia. This pest spreads in Indonesia from Nangroe Aceh Darussalam, Jambi, South Sumatra, Java Island, Central Sulawesi, South Sulawesi, Bali, West Nusa Tenggara, Papua and Maluku. [9]

2. Material and Methods

2.1. Making of Siwalan Fruit Peel Waste Liquid Smoke

The equipment intended for making liquid smoke is a modified pyrolysis reactor with a tar catcher, a condenser in which there is a spiral-shaped pipe, and is equipped with a liquid smoke reservoir. Combustion of raw materials will produce steam to flow following the connecting pipe to the condenser, the temperature difference will make the smoke liquid and will be expelled through the pipe connected to the condenser and then accommodated in a holding container.

2.2. Analysis of components of the liquid smoke of Siwalan fruit peel waste using GCMS

Analysis of the components of liquid smoke compounds qualitatively and quantitatively can be done with GCMS. According to ref. [10], the treatment was carried out by extracting liquid smoke using dichloromethane solvent, with a composition of 5 ml of liquid smoke and 5 ml of dichloromethane solvent and then homogenized for 5 minutes using a separating funnel. The upper fraction (dichloromethane) was separated from the lower fraction by short precipitation. The upper fraction was accommodated and 5 mL of dichloromethane was added to the lower fraction, homogenized again in a separatory funnel as in the initial stage. The resulting upper fraction from the second extraction is mixed with the upper fraction resulting from the initial separation. Then it is concentrated by blowing nitrogen gas until a volume containing 1 mL is detected. GCMS operating conditions are ionizing type: EI (Electron Impact), injector temperature: 280°C, column type: Agilent HP 1MS, length 30 m, column temperature 75°C, and carrier gas: helium with a pressure of 13.0 kPa.[7].

2.3. Liquid smoke test of Siwalan fruit peel waste on armyworm mortality

Analysis of armyworm mortality with parameter LC50.24 hours was carried out by preparing 10 instar 3rd armyworms in healthy condition and acclimatized (starved) for 1-2 hours before testing. Made 5 variations of liquid smoke and aquadest 5%, 7%, 9%, 11%, and 13% with 1 control containing only aquadest which was applied to young corn leaves using leaf dipping methods as feed for test animals. Furthermore, as many as 10 armyworms were placed in a container with clean conditions, given leaf feed that had received dyeing treatment and repeated twice. Observations were made for 24 hours. Percentage of mortality is calculated by equation 1 [10].

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\text{% Mortality} = \frac{\text{Total number of dead larvae}}{\text{Total number of larvae}} \times 100\%
\]

3. Results and Discussion

3.1. Making Liquid Smoke from Siwalan Fruit Peel Waste

The raw material for Siwalan fruit peel waste before pyrolysis combustion is to reduce the water content in the Siwalan fruit peel. Then the waste of Siwalan rind is put into the pyrolysis reactor. The pyrolysis process is carried out by using a small amount of oxygen which is used to ignite a fire in the
combustion process. The results immediately liquid physically have a dark brown color, clear, has a smell like smoke (sangit), and has a watery texture like water. The results of making Siwalan fruit peel waste smoke can be seen in Figure 1.

![Figure 1. Liquid smoke of Siwalan fruit peel](source)

Source: Production results (2022)

The pyrolysis process lasts about 8 hours with a less stable combustion temperature, which is around 200°C this is caused by the type of tool that cannot set the temperature automatically. According to ref. [11] the decomposition of cellulose, hemicellulose, and lignin compounds occurs at a temperature of 200-400°C.[12]

3.2. Results of Compound Component Analysis Results of Liquid Smoked Waste of Siwalan Peel Using GCMS

The results of the analysis of liquid smoke compounds from Siwalan fruit peel waste using GCMS (Gas Chromatography-Mass Spectroscopy) showed 18 peaks. The liquid smoke GCMS chromatography is presented in Figure 2 and spectrum results in Table 1.

![Figure 2. Chromatogram of GCMS liquid smoke of Siwalan fruit peel](source)

Source: Analysis results (2022)

<table>
<thead>
<tr>
<th>No.</th>
<th>Ret Time Min</th>
<th>Peak Name</th>
<th>SI</th>
<th>Area Counts*Min</th>
<th>Rel. Amount %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>2.028</td>
<td>Acetoin</td>
<td>794</td>
<td>445482.706</td>
<td>1.82</td>
</tr>
<tr>
<td>2.</td>
<td>2.351</td>
<td>1-Hydroxy-2-butanone</td>
<td>756</td>
<td>383547.128</td>
<td>1.57</td>
</tr>
<tr>
<td>3.</td>
<td>2.966</td>
<td>Furfural</td>
<td>904</td>
<td>1725668.636</td>
<td>7.05</td>
</tr>
</tbody>
</table>
Based on the results of the analysis, there are 5 components of the compound with the largest percentage distribution, namely phenol compounds (19.24%), 2-Heptadecanone (19.14), Cis-9-Hexadecenal (13.50%), cyclopentadecanone (11.94%), and furfural (7.05%).

The content of compounds that function as organic pesticides is obtained from phenol, furfural, acid, and carbonyl compounds. Phenol compounds are formed from the pyrolysis of lignin contained in Siwalan rind waste. Phenolic compounds are repellent and anti-insect eating. The presence of phenol is able to provide an odor that insects do not like. Furfuran is basically derived from pentose which is part of hemicellulose. In agriculture, Furfuran is used as a herbicide, fungicide, and insecticide.[13]

The acid compound produced is palmitic acid (Hexadecanoic acid) which acts as an anti-bacterial and microbial which can be used as a vegetable pesticide [11]. The acid content in liquid smoke is influenced by the cellulose content of the raw material used. The carbonyl compound in this study is acetone (Acetoin) which functions as insect repellent.

3.3. The results of the armyworm mortality test from liquid smoke of Siwalan fruit peel waste

The results of the armyworm mortality test from both repetitions obtained that the average mortality at a concentration of 5% was 5%, a concentration of 7% was 10%, a concentration of 9% was 15%, a concentration of 11% was 20%, and a concentration of 13% was 35%. The results showed that the overall treatment had a mortality rate of less than 50%. From the results of the analysis, it can be concluded that the higher the concentration used, the higher the mortality value of the armyworm tested. However, the overall treatment had a mortality result of less than 50% so it can be said that the liquid smoke of Siwalan peel waste has a slow killing power, this is in line with reference [14] that organic pesticides do not directly kill insects (low toxicity) or have a slow effect [2].

**Figure 3. Death of Armyworm**

Source: Analysis results (2022)
Visual observation of armyworm death due to liquid smoke of Siwalan peel waste generally shows physical characteristics such as the absence of body movement in armyworms which indicates death, its body excretes yellowish green liquid due to stomach toxins that enter through the digestive tract of food or stomach [14], as well as making the body of armyworms that experience death sticky or sticking to the food provided, the body color becomes darker besides that the caterpillar's body looks like it is malnourished, weak, and some shows the characteristics of the caterpillar becoming thinner or shrinking. This is caused by anti-eating compounds and toxins contained in the liquid smoke of Siwalan peel waste. Armyworm death is caused by toxins that enter through corn leaves which further inhibit cell metabolism in the body, then inhibit electron transport in mitochondria so that energy formation from food does not occur as an energy source in cells and cells cannot function, and armyworms die.[8]. In addition, some of the caterpillars' bodies were bent or irregular due to the active compound of liquid smoke which gave a convulsive effect before death as with the research of ref [14], insect toxic compounds will penetrate the intestinal wall which in turn will disrupt the insect's metabolism and cause a lack of energy needed for life activities, seizures and will eventually cause death [15].

4. Conclusion

The results of the research conducted concluded that the pyrolysis process was carried out for 8 hours at a temperature of around 200°C with a little oxygen producing liquid smoke that was dark brown, clear, smelled like smoke (sangit), and had a watery texture like water.

The liquid smoke of Siwalan peel waste contains 18 compounds detected with 5 components having the highest concentration, namely phenol compounds (19.24%), 2-Heptadecanone (19.14), Cis-9-Hexadecenal (13.50%), cyclopentadecanone (11.94%), and furfural (7.05%). Compounds that act as organic pesticides are phenol, furfuran, palmitic acid, and acetone which are antifeedant, repellent, attractant, stomach poison and inhibit growth.

The greater the concentration of liquid smoke of Siwalan skin waste, the higher the percentage value of armyworm mortality. However, it is less effective in killing armyworms quickly, even mortality at a maximum concentration of only 35% or has not reached the mortality rate of 50% of test animals.

5. References


