

Phytochemical Content and Insecticidal Activity of Oil Palm Solid Waste Liquid Smoke

Dudi Haryadi ^{1*}, Fahri Rijal Giffari ², Neneng Sri Widayani ³, Khairunissa Ainun ⁴, Muhammad Fathussalam ⁵, Indra Yuwono ⁶, Danar Dono ⁷

^{1,2,4,5,6} Research Institutions, Universitas Langlangbuana, Bandung, Jawa Barat, Indonesia

^{3,7} Department of Plant Pests and Diseases, Faculty of Agriculture, Universitas Padjadjaran, Jatinangor, Jawa Barat, Indonesia

Abstract

Indonesia is one of the countries that relies on palm oil as a source of state income. However, palm oil waste has not been utilized optimally. Liquid smoke is one of the products that can be utilized from palm oil solid waste to control pests and diseases with many chemical compounds. This study aims to determine the content of chemical compounds in palm oil solid waste (fronds, mesocarp fiber, and shells) and the level of effectiveness against pests. Phytochemical analysis was carried out to determine the content of chemical compounds in our several liquid smoke. The solutions (liquid smoke) were analyzed by using UV-vis spectrophotometer and GC-MS. The experiment of liquid smoke against *Spodoptera frugiperda* was carried out by leaf-residual feeding method. Experiments on the effect of storage on toxicity were also carried out by testing liquid smoke fibers stored for 3 months (L). Leaf feeds (sized 4 x 4 cm) were dipped in a liquid smoke solution with a concentration of 0% (control); 2.5%; 5%; 7.5%; and 10% that mixed with emulsifier tween 80 (0.5 ml/l). The parameters observed in this experiment were the test larvae's mortality, the feed consumption area, the pupae's weight, and the larval stages' development time. Our research showed that the highest compounds in shell, fronds, and mesocarp fiber of palm oil waste liquid smoke were carboxylic acid derivatives (esters), phenols, alcohols, and furans, respectively. Liquid smoke originating from the fronds with a concentration of 7.5%, 10%, and fibers with a concentration of 10% cause the mortality of *S. frugiperda* larvae by 70%, 62.5%, and 60% respectively. Liquid smoke (from fronds, mesocarp fiber, and shells) also reduces feed consumption by 31-65% at a concentration of 10%. Meanwhile, the liquid smoke provided did not affect the development time and weight of the pupa. Thus, palm oil solid waste liquid smoke can be used as an alternative for pest control and a solution for utilizing waste.

Keywords: Botanical Insecticides, Chemical Compounds, Toxicity, Antifeedant

Introduction

Palm oil is an important and economically profitable crop commodity. Indonesia is one of the countries that relies on palm oil as a source of state income. Palm oil productivity in Indonesia will reach approximately 3.68 tonnes per hectare in 2022 (Badan Pusat Statistik, 2023). However, palm oil waste has not been utilized optimally. Liquid smoke is one of the products that can be utilized from palm oil solid waste. Meanwhile, liquid smoke can be defined as a product obtained from the process of chemical decomposition of organic matter by heating the material in a closed room with a lack or without oxygen to break the bonds of complex molecules into bonds of smaller molecules (Czernik & Bridgewater, 2004; Awaludin, 2007).

Several studies on the content of phytochemical compounds in liquid smoke have been widely carried out. A study conducted by Bottrell and Schoenly (2012), showed that the largest compound in liquid smoke of rice husks was acetic acid by 58.68%. The chemical components of liquid smoke of rice hull contained acetic acid (33.15%), benzenesulfonic acid, 4-hydroxy (32.77%), benzene, 1,2,3-trimethoxy (2, 83%), 2-methoxy-4-2 methylphenol (14.02%), phenol, 2,6-dimethoxy (1.35%), phenol, 2-methoxy (1.63%), phenol, 2-methyl (3.30%), phenol, 4-ethyl-2-methoxy (4.63%), and methyls (0.82%) (Wagiman *et al*, 2014).

Insects or pathogens that attack plants can result in economic losses. The use of pesticides made from synthetic chemicals can cause damage to the environment (soil, air, and water), causing pests and diseases to become resistant, pest resurgence, and toxicity to humans (Dhaliwal *et al*, 2004; Horowitz & Ishaaya, 2012; Diptaningsari *et al*, 2021). The use of pesticides made from natural ingredients can be used as an alternative to control pests and plant diseases that are relatively safe. Botanical pesticide has many advantages over chemical pesticides, such as minimal health hazards and environmental pollution, safety for non-target organisms, no negative effect on plant growth, and safety for human health (Pavela, 2014; Dimetry, 2014). A good botanical pesticide must meet several requirements before being developed, including being easier to produce in large quantities, not being toxic to non-target organisms, and having a lethal effect on insect pests (Prabowo *et al*, 2016).

The use of liquid smoke as pest control has been reported by several researchers. Liquid smoke from coconut shells at a concentration of 12.5% caused the mortality of the brown planthopper (*Nilaparvata lugens*) by 53% and is not phytotoxic to plants (Ariyani *et al*, 2015). Furthermore, liquid smoke from palm oil waste at a concentration of 1.25% caused the mortality of *N. lugens* by 91.67% (Hu *et al*, 2014). This study aims to determine the content of chemical compounds in palm oil solid waste (fronds, fibers, and shells) and the level of effectiveness against *Spodoptera frugiperda*. Furthermore, palm oil solid waste can be used as an alternative pest control and a solution in the utilization of palm oil waste.

Materials And Methods

Liquid Smoke Production

Palm oil solid waste biomass was obtained from palm oil plantations and mills owned by PT Condong Garut, West Java, Indonesia. The types of waste used are palm fruit fibers and palm fronds. Palm waste biomass, each dried first in the sun. The design of a cyclone-distillation-based liquid smoke-making machine is modified and is different from the existing distillation-based liquid (Figure 1). Making liquid smoke uses a modified distillation device by installing a centrifugal blower and a cyclone-shaped separator to filter the ash and particles carried by the smoke formed. The filtered smoke is then channelled to the condenser tube for condensation. The resulting liquid smoke droplets are collected in a container, closed tightly and stored in the refrigerator.

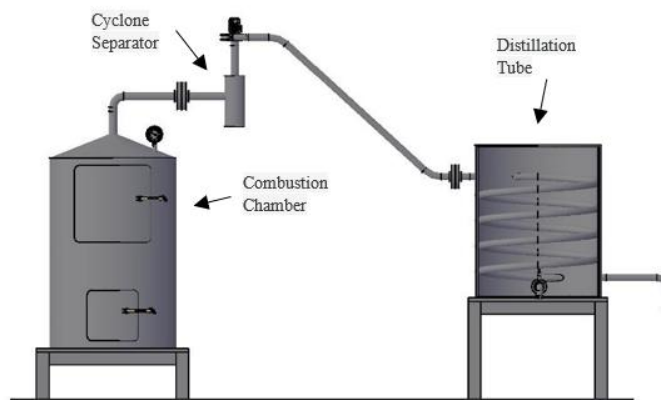


Figure 1. Design of a Cyclone-Distillation-Based Liquid Smoke-Making Machine

Phytochemical Screening

Analysis of the content of chemical compounds produced from palm oil waste liquid smoke was carried out based on research by Maulina & Nurtahara (2020). The extraction tool was a set of reflux condensers equipped with a hot plate heater. The liquid smoke obtained was filtered first to remove tar. Then, the solvent and liquid smoke were adjusted. Liquid smoke was dissolved using a measured volume. Afterwards, the mixture was put into a three-neck decoction. Stirring was carried out with the stirrer speed at 250 rpm for a certain time (t). The extraction was set at a certain temperature (T). After that, the raffinate and extract phases were removed from the separating funnel, then the compounds were precipitated for 2 hours. Both compounds were put into different

Erlenmeyer flasks. Next, the extract phase was distilled, and then the volume of the remains in the flask was measured. Finally, the solution was analyzed using UV-visible spectrophotometer and GC-MS brand Shimadzu QP 2100.

Treatment of Palm Oil Waste Liquid Smoke Against *S. frugiperda*

The research was conducted at the Laboratory of Pesticide and Environmental Toxicology at Universitas Padjadjaran. There are several observation parameters in this research. The observation parameters are divided into several sub-chapter points, including the content of chemical compounds in palm oil waste liquid smoke. The content of chemical compounds recorded was included in the research report as additional information that is useful in the development of further research.

The research design was carried out based on research conducted by Permana *et al* (2021). The experiment of liquid smoke against *S. frugiperda* was carried out by dipping the leaf feed into a solution of liquid smoke from the kernel shell (C), fronds (P), and mesocarp fibers (S). Experiments on the effect of storage on toxicity were also carried out by testing liquid smoke fibers stored for 3 months (L). Leaf feeds from corn var. Talenta F1 (sized 4 x 4 cm) were dipped in a liquid smoke solution with a concentration of 0% (control); 2.5%; 5%; 7.5%; and 10% that mixed with emulsifier (0.5 ml/l). Feed treatments were only given for the first 48 hours. On the third day, the feed used baby corn and the test insect was placed individually. The parameters observed in this experiment were the mortality of the test larvae, the area of feed consumption, the weight of the pupae and the development time of larval stages. Post-treatment caterpillar population data were calculated at 2, 4, 6, and 12 days after treatment.

Data Analysis

All averages of collected data were analyzed using IBM SPSS 26. The comparison of efficacy of various insecticides was analyzed using ANOVA at $P = 0.05$. Significant F values were further analyzed using the Duncan test to separate treatment means with a real level of 5%.

Result And Discussion

Phytochemical

The results of liquid smoke chromatography from pyrolysis of palm oil shell material show that there are twenty-seven compounds contained in this material (**Table 1**). GC-MS chromatogram of oil palm frond liquid smoke can be seen in **Figure 2**. The highest concentrations of compounds detected were carboxylic acid derivatives (esters), phenols, alcohols, and furans, respectively. The lignin, cellulose, and hemicellulose content in palm oil shells is degraded into several phenol derivative compounds with a total concentration of 35%, and carboxylic acids of 60%, the rest are furans and alcohol.

Liquid smoke from palm kernel shells and empty palm fruit bunches is known to contain phenol, acetic acid, 2,6-dimethoxyphenol (Syringol), acetic acid, oxalic acid, dihydroxybenzene, 2-methoxyphenol, 2-methoxymethyl phenol, furfural (Fauziati and Sampepana, 2021). These compounds are known to act as compounds that are repellent for insects and also have toxic effects. Experiments with palm frond material were also carried out. The fronds' high lignin content can produce phenolic compound output because the lignin content tends to be high (Maulina & Silia, 2018). The compound content of liquid smoke produced from the pyrolysis process of palm oil solid waste, namely shells, fibers and fronds processed by a cyclone distillation device, has a phenol concentration high enough to be used as a natural insecticide.

A total of twenty-three peaks were detected in the chromatography results of liquid smoke from palm fronds from the pyrolysis process using a cyclone distillation apparatus (**Table 2**). The phenol and acetic acid content of the pyrolysis process for each shell, fiber and fronds material will differ depending on the composition of the chemical components. The production results from thermal degradation of lignin components produce phenols (including guaiacyl and syring derivatives) and mostly produce tar (Maulina *et al*, 2018; Muzyka *et al*, 2023). Meanwhile, hemicellulose and cellulose are degraded to form ketones, aldehydes, furans and carboxylic acids (Muzyka *et al*, 2023). The shell contains the highest lignin (47.68%) compared to cellulose (27.56%) and

hemicellulose (22.32%) (Chang *et al*, 2020). Meanwhile, fiber has higher cellulose (43.00%) than hemicellulose (33.00%) and lignin (22.00%) (Yasim-Anuar *et al*, 2016). The frond also has cellulose (42.20%) which is higher than hemicellulose (26.40%) and lignin (22.30%) (Abdul Khalil *et al*, 2010).

The results of analysis of liquid smoke compound components from the three dry waste materials showed that the phenol content was quite high (Table 3). However, its toxicity ability against pests will have different effects depending on the type of pest to be controlled, so further testing needs to be carried out. In experiments on the quality of liquid smoke based on storage time, it was discovered that there were differences in the components of liquid smoke compounds in new samples and old samples that had been stored for three months. The diversity of compound components was higher in samples of liquid smoke made from palm fiber which were still new, with 23 peaks, whereas in samples of liquid smoke from fiber that had been stored for a long time at room temperature for three months only 11 peaks of chemical compounds were identified (Table 3 and Table 4).

Compared with the new sample, only the compounds methanol, acetone-oxime, acetic acid, 2-propanone, 1-hydroxy-pyridine, acetol, phenol, and guaiacol were still found in the old samples. However, new compounds were identified, namely, tetra deuterio methane and ethanol, which were formed in the old samples. The acetic acid content (4.88%) in this sample tends to be lower than in the new sample (30.23%). The phenolic content found in the old sample was higher (57.96%) compared to the phenolic compounds in the new sample (23.77%) (Table 3 and Table 4).

The difference in compound components occurs because the liquid smoke resulting from the pyrolysis process is unstable during storage so the viscosity of the solution increases which causes oxidation reactions of the active compounds in the solution (Theapparatt *et al*, 2018). Longer storage time for liquid smoke has the effect of increasing the content of phenol and guaiacol compounds. A similar thing also happened in the research of Cheng *et al* (2021) where the formation of 2-methoxy-phenol (CAS) Guaiacol comes from the breaking reaction of the O-CH₃ bond from aromatic compounds or also the impact of the oxidation process that occurs during storage.

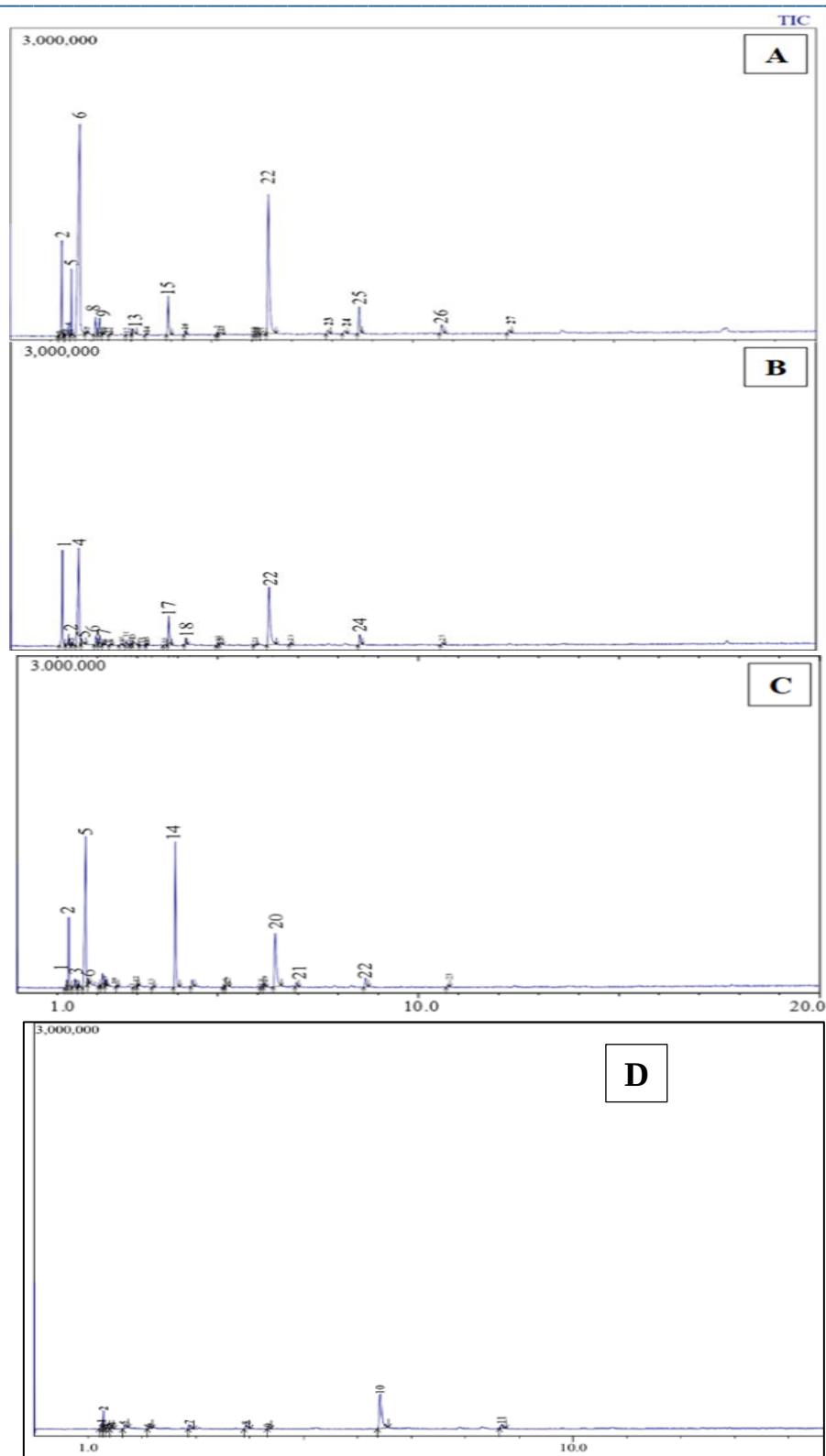


Figure 2. Chromatogram of Liquid Smoke from Palm-Oil Kernel Shell (A); Mesocarp Fiber (B); Frond (C), and Mesocarp Fiber three months after storage (D).

Table 1. The Phytochemical composition of liquid smoke from palm kernel shell (PKS)

Peak	Compounds	Time of Retention tR (minutes)	Percent of composition (%)
1	Carbon dioxide (CAS) Dry ice	1.211	0.12
2	Methanol (CAS) Carbinol	1.289	7.10
3	Formic acid, methyl ester (CAS) Methyl formate	1.340	0.27
4	2-Propanone (CAS) Acetone	1.442	0.77
5	Acetic acid, methyl ester (CAS) Methyl acetate	1.527	4.24
6	Acetic acid (CAS) Ethylic acid	1.732	43.05
7	Propanoic acid, methyl ester (CAS) Methyl propanoate	1.919	0.35
8	2-Propanone, 1-hydroxy- (CAS) Acetol	2.126	2.12
9	Propanoic acid (CAS) Propionic acid	2.228	2.10
10	2,3-Pentanedione (CAS) 2,3-Pentanedione	2.330	0.20
11	2-Butanone, 3-hydroxy- (CAS) Acetoin	2.475	0.12
12	Pyridine (CAS) Azine	2.885	0.07
13	1-Hydroxy-2-Butanone	3.056	1.08
14	Cyclopentanone (CAS) Dumasine	3.377	0.17
15	2-Furancarboxaldehyde (CAS) Furfural	3.929	4.41
16	2-Propanone, 1-(acetyloxy)- (CAS) Acetol acetate	4.352	0.47
17	2-Cyclopenten-1-one, 2-methyl- (CAS) 2-Methyl-2-cyclopentenone	5.141	0.25
18	2(3H)-Furanone, dihydro- (CAS) Butyrolactone	5.228	0.59
19	2-Butanone, 1-(acetyloxy)- (CAS) 1-Acetoxy-2-butanone	6.057	0.16
20	2-Furancarboxaldehyde, 5-methyl- (CAS) 5-Methyl-2-furfural	6.150	0.21
21	2-Cyclopenten-1-one, 3-methyl- (CAS) 3-Methyl-2-cyclopentenone	6.220	0.05
22	Phenol (CAS) Izal	6.417	25.19
23	Phenol, 2-methyl- (CAS) o-Cresol	7.892	0.67
24	Phenol, 3-methyl- (CAS) m-Cresol	8.304	0.55
25	Phenol, 2-methoxy- (CAS) Guaiacol	8.672	3.65
26	2-Methoxy-4-methylphenol	10.722	1.47
27	Phenol, 4-ethyl-2-methoxy- (CAS) p-Ethylguaiacol	12.383	0.57

Table 2. The Phytochemical composition of liquid smoke from palm oil fronds

Peak	Compounds	Time of Retention tR (minutes)	Percent of composition (%)
1	Formaldehyde (CAS) BFV	1.244	1.27
2	Methanol (CAS) Carbinol	1.291	7.97
3	2-Propanone (CAS) Acetone	1.446	1.13

4	Acetic acid, methyl ester (CAS) Methyl acetate	1.532	0.40
5	Acetic acid (CAS) Ethylic acid	1.712	34.68
6	3-Pentanone (CAS) Diethyl ketone	1.775	1.04
7	2-Butenal (CAS) Crotonaldehyde	2.054	0.09
8	2-Propanone, 1-hydroxy- (CAS) Acetol	2.136	2.39
9	Propanoic acid (CAS) Propionic acid	2.219	1.20
10	2-Butanone, 3-hydroxy- (CAS) Acetoin	2.483	0.21
11	Pyridine (CAS) Azine	2.905	0.17
12	Acetic acid, methyl ester (CAS) Methyl acetate	3.014	0.35
13	Cyclopentanone (CAS) Dumasine	3.388	0.18
14	2-Furancarboxaldehyde (CAS) Furfural	3.943	24.92
15	2-Propanone, 1-(acetyloxy)- (CAS) Acetol acetate	4.367	1.15
16	2-Cyclopenten-1-one, 2-methyl- (CAS) 2-Methyl-2-cyclopentenone	5.158	0.33
17	2(3H)-Furanone, dihydro- (CAS) Butyrolactone	5.246	0.87
18	2-Butanone, 1-(acetyloxy)- (CAS) 1-Acetoxy-2-butanone	6.065	0.15
19	2-Furancarboxaldehyde, 5-methyl- (CAS) 5-Methyl-2-furfural	6.168	0.93
20	Phenol (CAS) Izal	6.435	17.02
21	2-Furanmethanol, tetrahydro- (CAS) Tetrahydrofurfuryl alcohol	6.966	0.87
22	Phenol, 2-methoxy- (CAS) Guaiacol	8.689	2.11
23	2-Methoxy-4-methylphenol	10.734	0.55

Table 3. The Phytochemical composition of liquid smoke from mesocarp fiber (new)

Peak	Compounds	Time of Retention tR (minutes)	Percent of composition (%)
1	Methanol (CAS) Carbinol	1.290	16.00
2	acetone-oxime	1.442	1.98
3	Acetic acid, methyl ester (CAS) Methyl acetate	1.529	0.55
4	Acetic acid (CAS) Ethylic acid	1.697	30.23

5	2-Butanone (CAS) Methyl ethyl ketone	1.760	1.97
6	2-Propanone, 1-hydroxy- (CAS) Acetol	2.127	2.54
7	Propanoic acid (CAS) Propionic acid	2.214	2.24
8	2,3-Pentanedione (CAS) 2,3-Pentanedione	2.330	0.35
9	2-Butanone, 3-hydroxy- (CAS) Acetoin	2.474	0.26
10	Ethanone, 1-cyclopropyl- (CAS) Cyclopropyl methyl ketone	2.752	0.51
11	Pyridine (CAS) Azine	2.876	1.10
12	2-Propanone, 1-hydroxy- (CAS) Acetol	3.000	0.34
13	1-HYDROXY-2-BUTANONE	3.057	1.97
14	Propanoic acid, 2-oxo-, methyl ester (CAS) Methyl pyruvate	3.220	0.30
15	Cyclopentanone (CAS) Dumasine	3.377	0.39
16	Pyrazine, methyl- (CAS) Methylpyrazine	3.810	0.39
17	2-Furancarboxaldehyde (CAS) Furfural	3.931	7.75
18	2-Propanone, 1-(acetyloxy)- (CAS) Acetol acetate	4.354	1.41
19	2-Cyclopenten-1-one, 2-methyl- (CAS) 2-Methyl-2-cyclopentenone	5.145	0.58
20	2(3H)-Furanone, dihydro- (CAS) Butyrolactone	5.229	0.74
21	2-Butanone, 1-(acetyloxy)- (CAS) 1-Acetoxy-2-butanone	6.050	0.26
22	Phenol (CAS) Izal	6.420	23.77
23	2-Furanmethanol, tetrahydro- (CAS) Tetrahydrofurfuryl alcohol	6.953	0.51
24	Phenol, 2-methoxy- (CAS) Guaiacol	8.677	3.12
25	2-Methoxy-4-methylphenol	10.725	0.73

Peak	Compounds	Time of Retention tR (minutes)	Percent of composition (%)
1	Tetra Deutero Methane	1.250	6.57
2	Methanol (CAS) Carbinol	1.288	11.54
3	Ethanol (CAS) Ethyl alcohol	1.365	1.41
4	acetone-oxime	1.441	1.52
5	Acetic acid (CAS) Ethylic acid	1.674	4.88
6	2-Propanone, 1-hydroxy- (CAS) Acetol	2.120	1.26
7	Pyridine (CAS) Azine	2.876	3.77

Peak	Compounds	Time of Retention tR (minutes)	Percent of composition (%)
8	2-Furancarboxaldehyde (CAS) Furfural	3.934	5.00
9	2-Propanone, 1-(acetyloxy)- (CAS) Acetol acetate	4.354	1.21
10	Phenol (CAS) Izal	6.421	57.96
11	Phenol, 2-methoxy- (CAS) Guaiacol	8.677	4.89

Table 4. The Phytochemical composition of liquid smoke from mesocarp fiber after three months of storage

Toxicity

The mortality rate for *S. frugiperda* caterpillars was highest, namely 70% on the 6th day after treatment with fronds liquid smoke (P) with a concentration of 10%. Meanwhile, the treatment of palm oil fiber liquid (L) smoke that had been stored at a concentration of 2.5% caused the lowest mortality rate. The P 10%, P 7.5%, and S (fiber liquid fresh) 7.5% treatments had a mortality effect of more than 50% on day 6 after treatment. However, the effects of the liquid smoke that we had provided began to appear 2 days after application. The research results also showed that in several treatments we carried out on days 4 and 5, there was no increase in larval mortality. Furthermore, on day 9 after treatment, there was no increase in mortality from all treatments. This concludes that the effect of solid palm oil waste liquid smoke on *S. frugiperda* larvae only has an effect approximately one week after treatment (Figure 3).

Mortality of *S. frugiperda* larvae due to liquid smoke treatment was similar to several previous studies. Indriati and Samsudin (2018) demonstrated the effect of liquid smoke from several materials including cocoa pod husks, sawdust, coconut shells, and rice husks on the coffee berry borer *Hypothenemus hampei*. Liquid smoke derived from coconut shells (concentration of 12.5%) has also been proven in controlling *Nilaparvata lugens* with a mortality rate of 53% in 72 HAT or with an LC₅₀ value is 12,87% (Wagiman *et al*, 2014). Meanwhile, liquid smoke from coconut shells can suppress the population of *Odontotermes sp* termites (in concentration 50%) and *Ferrisia virgata* (in concentration 10%) respectively 81.71% and 95.12% (Wititsiri, 2011). Apart from causing mortality to pests, liquid smoke from tree branches has also been reported to inhibit the laying of *Callobruchus maculatus* eggs in cowpeas (Chalermnan & Peerapan, 2009).

Mortality of *S. frugiperda* larvae began to occur two days after treatment. Each treatment has a different effect on mortality rates. The constant administration of liquid smoke made from palm fiber with a concentration of 10% (P 10%) and palm fiber with a concentration of 10% (S 10%) had a significant effect and had the highest mortality rate for *S. frugiperda*. However, the use of palm frond liquid smoke with a concentration of 7.5% can be used as an alternative to control *S. frugiperda* caterpillars with the mortality rate on day 12 not being significantly different from the P 10% and S 10% treatments (Table 5).

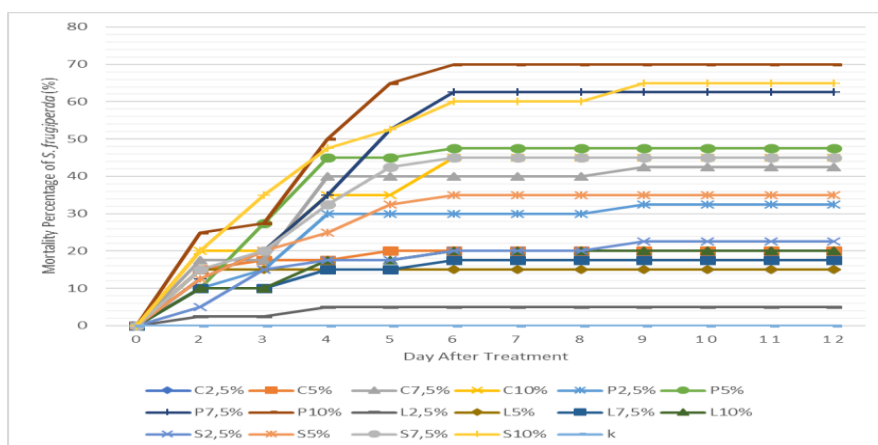


Figure 3. *S. frugiperda* cumulative mortality graph after treatment with smoke liquid from Shells (C), Fronds (P), and Fiber (S: new fiber; L: old fiber), control (K) of palm oil waste

The mortality that occurred in *S. frugiperda* larvae is thought to be due to the toxic properties of the liquid smoke that we provided. The high phenol content in the liquid smoke solution is one of the causes of larval mortality. Phenol is a secondary metabolite compound that is structurally diverse and is needed by plants for resistance to insect pests and pathogens (De Moraes *et al*, 1998; Agrawal *et al*, 2012; War *et al*, 2016). Plants can produce metabolite compounds which are divided into two, namely primary and secondary. Primary compounds are ingredients that are directly involved in plant growth, development, and reproduction (Gajger & Dar, 2021). Meanwhile, secondary compounds have a large role in defence from insect threats (Dar, 2012). These compounds can also be called allelochemicals which play a crucial role in insect-plant interactions (Ibanez *et al*, 2012).

Table 5. Mortality of *S. frugiperda* after treatment with smoke liquid from shells, fonds, and fiber of palm oil waste.

Treatment	Mortality of <i>S. frugiperda</i> (X±SE) (day after treatment) (%)											
	2 ^{*ns}			4			6			12		
C2.5%	10.00	±	3.54	15.00	±	4.33abc	15.00	±	4.33abc	15.00	±	4.33abc
C5%	15.00	±	7.50	17.50	±	4.15bcd	20.00	±	3.54bcd	20.00	±	3.54bcd
C7.5%	17.50	±	4.15	40.00	±	3.54efgh	40.00	±	3.54ef	40.00	±	3.54ef
C10%	20.00	±	3.54	35.00	±	2.50efgh	45.00	±	2.50efg	45.00	±	2.50efg
P2.5%	10.00	±	5.00	30.00	±	3.54cde	30.00	±	3.54cde	30.00	±	3.54cde
P5%	10.00	±	3.54	45.00	±	5.59fgh	47.50	±	4.15fg	47.50	±	4.15fg
P7.5%	12.50	±	4.15	35.00	±	2.50efgh	62.50	±	2.17h	62.50	±	2.17h
P10%	25.00	±	5.59	50.00	±	6.12h	70.00	±	5.00h	70.00	±	5.00h
L2.5%	2.50	±	2.17	5.00	±	2.50ab	5.00	±	2.50ab	5.00	±	2.50ab
L5%	15.00	±	5.59	15.00	±	5.59abc	15.00	±	5.59abc	15.00	±	5.59abc
L7.5%	10.00	±	5.00	15.00	±	5.59abc	17.50	±	6.50bc	17.50	±	6.50bc
L10%	10.00	±	6.12	17.50	±	4.15bcd	20.00	±	5.00bcd	20.00	±	5.00bcd
S2.5%	5.00	±	2.50	17.50	±	2.17bcd	20.00	±	3.54bcd	20.00	±	3.54bcd
S5%	12.50	±	5.45	25.00	±	5.59cde	35.00	±	2.50def	35.00	±	2.50def
S7.5%	15.00	±	2.50	32.50	±	2.17cdef	45.00	±	2.50cefg	45.00	±	2.50cefg
S10%	20.00	±	6.12	47.50	±	6.50gh	60.00	±	7.91gh	60.00	±	7.91gh
K	0.00	±	0.00	0.00a	±	0.00	0.00a	±	0.00a	0.00a	±	0.00a

Descriptions: Shells (C), fronds (P), fiber (S: new fiber, L: old fiber), and Control (K). X – average of the mortality (day), SE – standard error, The average number followed by the same letter in the same column is not significantly different according to the Duncan test at 5% level

Apart from phenol, mortality in larvae can also be caused by other metabolite compounds such as methanol, carboxylic acids, and several other types of toxic compounds in palm oil waste liquid smoke. The methanol extract in the *Fagopyrum esculentum* plant is effective in controlling the aphid pest *Myzus persicae* (Gajger & Dar, 2021). The content of carboxylic acid compounds in liquid smoke was thought to be one of the factors in the mortality of *S. frugiperda* caterpillars. Hastuti *et al* (2019), reported that acetic acid at a concentration of 30% was effective in controlling the brown planthopper *Nilaparvata lugens* on rice plants. Propanoic acid in palm oil waste liquid smoke also determines the survival of *S. frugiperda* larvae. Application of propionic acid will effectively suppress the mass development of *S. granarius* (Lorenz *et al*, 2010). Propionic acid serves for prevention of fungal growth and control of insects, especially in storage of moist feed grain (Reichmuth and Richter, 1991)

The results of the research show that liquid smoke from palm oil waste has a significant influence on the level of feed consumption provided and the area of feed consumed by *S. frugiperda*. The provision of a liquid smoke solution made from stored shells, shells, fronds, and fibers generally significantly reduces feed

consumption levels with a concentration of $\geq 5\%$. This provides evidence that there is a tendency for *S. frugiperda* not to consume food that has been treated (Table 6).

Giving a low treatment concentration gives a different effect. Treatment with liquid smoke from all ingredients with a concentration of 2.5% caused the larvae to tend to consume more food, although, in the end, the larvae died on a certain day. The tendency of larvae to consume feed is thought to contain the compound 2-methoxy-4-ethylphenol or guaiacol. Several previous studies have shown that there is a response that attracts larvae to eat but is repellent to the female imago in the decision to oviposition (Ainun *et al*, 2023; Revadi *et al*, 2020). Previous research also showed that there are olfactory receptors in *Spodoptera littoralis* that are physiologically sensitive to guaiacol (Revadi *et al*, 2020).

This research shows that the administration of liquid smoke from solid palm oil waste does not have a real effect on the length of development time for *S. frugiperda* larvae. The duration of larval development was relatively the same in all treatments including the control. In general, larval development time from instars II to VI is around 10-11 days. Even though the larval development time that we observed was relatively the same, the larvae's ability to consume the food we provided varied for each treatment (Table 7).

Table 6. Leaf area consumed by *S. frugiperda* larvae after treatment with smoke liquid from Shells, fronds, and fiber of palm oil waste

Treatment	The leaf area consumed ($X \pm$ SE) (mm)			Percentage of leaf area consumed ($X \pm$ SE) (%)			Inhibition of leaf consume (%)
C2.5%	168.88	$\pm$	15.84 g	10.55	$\pm$	0.99 g	-17.79
C5%	157.88	$\pm$	10.51 ef	9.87	$\pm$	0.66 ef	-10.11
C7.5%	98.50	$\pm$	5.77 bc	6.16	$\pm$	0.36 bc	31.30
C10%	83.88	$\pm$	4.81 bc	5.24	$\pm$	0.30 bc	41.50
P2.5%	151.00	$\pm$	13.52 fg	9.44	$\pm$	0.85 fg	-5.32
P5%	122.00	$\pm$	4.39 de	7.63	$\pm$	0.27 de	14.91
P7.5%	91.13	$\pm$	4.83 bc	5.70	$\pm$	0.30 bc	36.44
P10%	87.38	$\pm$	6.40 bc	5.46	$\pm$	0.40 bc	39.06
L2.5%	166.50	$\pm$	5.86 g	10.41	$\pm$	0.37 g	-16.13
L5%	104.63	$\pm$	7.15 cd	6.54	$\pm$	0.45 cd	27.03
L7.5%	95.75	$\pm$	2.54 bc	5.98	$\pm$	0.16 bc	33.22
L10%	97.88	$\pm$	1.20 bc	6.12	$\pm$	0.07 bc	31.73
S2.5%	136.38	$\pm$	10.58 ef	8.52	$\pm$	0.66 ef	4.88
S5%	81.75	$\pm$	2.69 bc	5.11	$\pm$	0.17 bc	42.98
S7.5%	76.75	$\pm$	7.45 b	4.80	$\pm$	0.47 b	46.47
S10%	49.63	$\pm$	2.51 a	3.10	$\pm$	0.16 a	65.39
K	143.38	$\pm$	5.97 ef	8.96	$\pm$	0.37 ef	-

Description: The average number followed by the same letter in the same column is not significantly different according to the Duncan test at 5% level;. Shells (C), fronds (P), and fiber (S: new fiber, L: old fiber, K: Control). X – average of the leaf area consumed (mm dan %); SE – standard error. Shells (C), fronds (P), and fiber (S: new fiber, L: old fiber)

Table 7. Length of development time of *S. frugiperda* larvae after treatment with smoke liquid from Shells, fronds, and fiber of palm oil waste.

Treatment	Length of larval development at test concentration ($X \pm$ SE) (days)											
	N			instar II-III			N			instar II-IV		
C2.5%	36	2.53	$\pm$	0.08	34	5.59	$\pm$	0.08	34	7.94	$\pm$	0.09
C5%	33	2.58	$\pm$	0.09	32	5.31	$\pm$	0.08	32	7.97	$\pm$	0.10
C7.5%	33	2.70	$\pm$	0.08	24	5.58	$\pm$	0.10	24	7.75	$\pm$	0.09
C10%	32	2.56	$\pm$	0.09	22	5.50	$\pm$	0.11	22	7.59	$\pm$	0.10

P2.5%	34	2.65	±	0.08	28	5.54	±	0.09	28	7.86	±	0.12	27	10.44	±	0.10
P5%	29	2.62	±	0.09	21	5.43	±	0.11	21	7.90	±	0.15	21	10.67	±	0.10
P7.5%	32	2.63	±	0.09	15	5.80	±	0.10	15	7.60	±	0.13	15	10.87	±	0.09
P10%	29	2.66	±	0.09	12	5.56	±	0.12	12	7.50	±	0.14	12	10.83	±	0.11
L2.5%	39	2.69	±	0.07	38	5.55	±	0.08	38	7.95	±	0.08	38	10.32	±	0.08
L5%	34	2.62	±	0.08	34	5.53	±	0.09	34	7.97	±	0.10	34	10.38	±	0.08
L7.5%	36	2.53	±	0.08	33	5.76	±	0.07	33	7.82	±	0.07	33	10.36	±	0.08
L10%	36	2.50	±	0.08	32	5.78	±	0.07	32	7.72	±	0.11	32	10.25	±	0.08
S2.5%	34	2.56	±	0.09	32	5.75	±	0.08	32	7.97	±	0.13	31	10.42	±	0.09
S5%	32	2.88	±	0.06	26	5.38	±	0.10	26	7.96	±	0.13	26	10.50	±	0.10
S7.5%	32	2.69	±	0.08	22	5.64	±	0.10	22	7.95	±	0.15	22	10.50	±	0.11
S10%	26	2.77	±	0.08	16	5.44	±	0.12	16	7.56	±	0.12	14	10.57	±	0.13
K	40	2.60	±	0.09	40	5.38	±	0.08	40	7.80	±	0.06	40	10.33	±	0.07

Description: X – average of the length development time (day), SE – standard error, N – number of larvae. Shells (C), fronds (P), and fiber (S: new fiber, L: old fiber, K: Control)

The difference in development time is usually caused by the quality and nutrients contained in the given substrate (Permana *et al*, 2022). Previous research showed that food quality can also influence the length of development time, total insect lifespan, life expectancy, and fecundity (Giffari *et al*, 2021). The shell, fiber, and fronds have relatively high contents of lignin, cellulose, and hemicellulose (Chang *et al*, 2020; Yasim-Anuar *et al*, 2016; Abdul Khalil *et al*, 2010).

This research shows that the application of solid palm waste liquid smoke pesticide did not have a real effect on the weight of the pupae. There was no significant difference in the average weight of the pupae calculated (Table 8). This is thought to be because the shell, fronds, and fibers contain relatively the same compounds and nutrients. Differences in body weight in insects are usually determined by the feed given and the nutritional content in the feed (Permana *et al*, 2022).

Table 8. Average weight of *S. frugiperda* pupae after treatment with smoke liquid from Shells, fronds, and fiber of palm oil waste.

Treatment	N	Pupae weight (g) (X±SE)			
C2.5%	34	0.2042	±		0.0034
C5%	32	0.2114	±		0.0035
C7.5%	23	0.2098	±		0.0042
C10%	22	0.2019	±		0.0041
P2.5%	27	0.2076	±		0.0051
P5%	21	0.2005	±		0.0060
P7.5%	15	0.2075	±		0.0064
P10%	12	0.2036	±		0.0110
L2.5%	38	0.2188	±		0.0029
L5%	34	0.2312	±		0.0052
L7.5%	33	0.2118	±		0.0051
L10%	32	0.2210	±		0.0047
S2.5%	31	0.2063	±		0.0034
S5%	26	0.2118	±		0.0057
S7.5%	22	0.2158	±		0.0061
S10%	14	0.2431	±		0.0049
k	40	0.2164	±		0.0036

Description: X – average of the weight the pupae (g); SE – standard error; N – number of pupae. Shells (C), fronds (P), and fiber (S: new fiber, L: old fiber, K: Control)

Conclusions

The results of the research showed that liquid smoke made from solid palm oil waste (shells, fibers, and fronds) contains some compounds that can cause mortality of *Spodoptera frugiperda* larvae and antifeedant effect. Liquid smoke originating from the fronds with a concentration of 7.5%, 10%, and fibers with a concentration of 10% cause the mortality of *S. frugiperda* larvae by 70%, 62.5%, and 60% respectively. Meanwhile, the liquid smoke did not affect the development time and weight of the pupa. Thus, palm oil solid waste liquid smoke can be used as an alternative for pest control and a solution for utilising waste.

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