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Pyroligneous Acid Derived from *Ficus benjamina* Wastes Synergize Deltamethrin against *Sitophilus oryzae*

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ABSTRACT

In this study, the pruning residues of *ficus benjamina* tree were used to prepare the wood vinegar through the carbonization process then its effectiveness was evaluated against the rice weevil *Sitophilus oryzae* (L.) (Coleoptera: Curculionidae) as single treatment by two bioassay techniques; contact and fumigation, then mixed with the sublethal dose of deltamethrin (LC_{20}) and re-evaluated using the contact technique. The GC-MS analysis was used to analyze the chemical composition of wood vinegar. The wood vinegar did not have insecticidal activity although it reached the highest concentration (100%), while after mixing with LC_{20} of deltamethrin, the toxicity of both was greatly increased, when a low concentration of wood vinegar (5%) mixed with LC_{20} of deltamethrin, the mortality reached about 90%. The GC-MS analysis showed that the phenolic compounds were the major component in the wood vinegar followed by the benzene derivatives. The obtained results clearly showed the ability to use the wood vinegar as a natural compound mixed with chemical insecticides successfully so that we can reduce the concentrations of chemical insecticides involved in our environment.

INTRODUCTION

One of the most grain pests is the rice weevil, *Sitophilus oryzae* (L.) (Coleoptera: Curculionidae), This species infests cereal kernels, causing losses in both quality and quantity during grain storage (Hong et al., 2018), due to feeding inside and outside the grain during the larval and adult stages (Ertürk *et al.,* 2020). The mainly control tools that used stored product pests are conventional insecticides and fumigants. As a result of resistance development in many stored product pests toward many insecticides, the managing process of these pests has become more difficult in recent years(Arthur, 1996; Benhalima *et al.*, 2004; Pimentel *et al.*, 2010; Zettler & Keever, 1994).

It has been established that the use of pesticides must be minimized to reduce the resulting hazards to human health and the environment (Tiilikkala *et al.*, 2010a).

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During the production of biochar under elevated temperature from different agricultural and forestry wastes. During the pyrolysis process about 60-75 wt % of bio-oil, also known as pyroligneous acid, wood vinegar and crude bio-oil,15- 25 wt% of solid char, and 10-20 wt% of non-condensable (Kim *et al.*,2020; Mohan *et al.*,2006; Aly, 2017) . wood vinegar is a liquid produced as a result of the condensation of carbonized flue gas. From the point of view of environmental safety, wood vinegar is a green biomass material with activity toward the control of insects (Grewal *et al.,* 2018; Tiilikkala *et al.*, 2010b; Zhang *et al.*, 2012). Many products of wood vinegar have been inserted into wide forms of markets. It has many uses, for example, used in wood preservation, regulation of plant growth, fertilizer for soil or foliar, insect repellant also as animal feed additives and other uses (Baimark & Niamsa, 2009; Ferreira *et al.*, 2005; Mu *et al.*, 2004; Shiny & Remadevi, 2014; S. Y. Wu *et al.*, 2014) .

As a plant protection agent, the wood vinegar or Pyroligneous acid has been used against fungi and bacteria (Radhakrishnan *et al.*, 2002; Nakai *et al.*, 2007; Seo *et al.*, 2000) also its activity against termites and insects has been reported (Kim et al., 2008; Pangnakorn *et al.*, 2011; Yatagai *et al.*, 2002).

More than two hundred compounds have been found in the composition of wood vinegar produced from different sources, the organic compounds occupy from 10% to 20% of the total composition (Bilehal *et al.*, 2012).Mainly based on its composition, both the properties and uses of the wood vinegar are determined (Wu *et al.*, 2015**).** for example, wood vinegar with high content of organic acids has been reported to have antimicrobial activity (Ma *et al.*, 2011) while that with high content of phenolic compounds exhibited antioxidant activity(Loo *et al.*, 2007, 2008)**.**

In terms of its insecticidal activity, in this research we have been evaluated the activity of wood vinegar prepared from the residue of *ficus benjamina* trees by carbonization process against the adult of the rice weevil, *Sitophilus oryzae* (L.) using the residual film and fumigation techniques alone and mixed with the sublethal dose of deltamethrin and the chemical composition of the produced wood vinegar has been analyzed using GC-MS.

MATERIALS AND METHODS

Wood Vinegar (Pyroligneous acid):

The raw material for wood vinegar was branches of pruning residue of *Ficus benjamina* trees grow in the forestry research sector of Antoniades botanical garden, Alexandria governorate, Egypt. The pruning branches were air-dried in the open air for about three months, after that the branches were stored in the lab. at room temperature. The branches were debarked and sawn into suitable pieces.

Insecticide:

Deltamethrin (98%) as a technical grad was used in this study and obtained from the National Company for Agrochemicals &Investment (Agrochem.), Egypt.

The Preparation and Refinement of Pyroligneous Acids from *Ficus benjamin* Wood:

The cylindrical stainless-steel reactor measured 300 mm in height and 120 mm in internal diameter. Temperature control and an electrically heated furnace were included. In the related test, 50 g of sample was heated in the reactor at a rate of roughly 5 °C/min from ambient to target temperature. The samples were heated to the pyrolyzing temperature of 400 ° C. The required temperature was maintained for 1 hour. For the separation of pyroligneous acids, the pyrolyzed vapors were passed through a cooled condenser. The pyroligneous acid was stored in a refrigerator after the pyrolysis experiment, and as the storage period extended, the crude pyroligneous acid was progressively separated into three different layers. The top layer was thin oil, the middle layer was high-quality liquid, and the bottom layer was viscose wood tar mixed with a variety of other materials. It was possible to separate the liquid in the intermediate fraction, which will be pyroligneous acids.

Chemical Components Analysis of *Ficus benjamina* Wood Vinegar:

GC-TSQ mass spectrometer (Thermo Scientific, Austin, TX, USA) with direct capillary column TG-5MS was used to analyses the chemical components of *Ficus benjamina* wood vinegar (30 m x 0.25mm film thickness). The column oven temperature was initially kept at 50°C, then raised at a rate of 5°C/min to 250°C, held for 2 minutes, and then raised at a rate of 30°C/min to 300°C, held for 2 min. The temperature of the injector and MS transfer line were fixed at 270 and 260 ° C, accordingly, and helium was utilized as the gas at a flow rate of 1 ml/min. The solvent delay was 4 minutes, and 1 µl diluted samples were injected automatically using an Autosampler AS1300 and a split mode GC. In full scan mode, EI mass spectra were acquired at 70eV ionisation voltages spanning the m/z 50-60 range. The temperature of the ion source is set at 200°C. The chemical components of wood vinegar were identified using their retention time (RT) and mass spectra from the Wiley 09 and NIST 14 mass spectral databases. The GC peak area was used to compute the proportion of components.

Insects:

Sitophilus oryzae has been raised in whole sterilized wheat for the past ten years at the Faculty of Agriculture, Alexandria University (Laboratory conditions were 28°C, 70% relative humidity, and a photoperiod L/D of 12:12hr.). 2-3 weeks-old adults were used for all experiments.

Bioassay of Wood Vinegar:

Fumigation Bioassay:

Transferring twenty adults into glass jars (250 mL) containing 20 g of sterilized wheat and exposing them to various volumes (50,100, and 200 µl) of wood vinegar sprayed to filter paper (2cm diameter) was used to evaluate the vapor toxicity of the wood vinegar against the adults of *S. oryzae*. The treated filter papers were adhered to the inside surface of the jar's screw lids using sticky tape, which was made airtight after 30 min of WV evaporation. Water was used to treat the control jars. Three times, all of the treatments and controls were reproduced. After three days, the mortality percentage (M %) was calculated (Jayakumar *et al.*, 2017).

Contact Bioassay:

Using the residue film technique, the insecticidal activity of various doses of WV dilution with water was evaluated (Urrutia *et al.*, 2021). Filter paper was used for bioassays. The filter paper (9 cm in diameter) was covered with 1 mL of the dilution. For 15 minutes, the diluted WV was allowed to evaporate. The filter papers were then transferred into Petri dishes (9 cm in diameter). Water was used to treat the control Petri dishes. Twenty adults were placed into each Petri dish separately and then covered. Each treatment and control had three replicates set up. After three days, the mortality rate was determined.

Treatments With Deltamethrin:

Deltamethrin was determined in adult *S. oryzae* using a residual contact bioassay with filter papers. A range of concentrations were prepared acetone (solvent). Several filter papers (Whatman No. 1) were put in 9 cm glass Petri dishes and treated with 1ml insecticide solutions or acetone solvent (control). Before being utilized in the assay, the treated filter papers were allowed to dry for 15 minutes. Each glass Petri dish contained 20 adults (2 weeks old) of *S. oryzae*. Insect mortality was recorded after three days; three replicates were used for each concentration.

The Mixture of Wood Vinegar + Deltamethrin:

LC_{20} ($0.24x10^{-5}$ %) of deltamethrin were mixed with 0.1, 0.5, 1, 3 and 40% of the WV. Firstly, the solution of the WV is being applied to filter papers (Whatman No. 1) after evaporation the deltamethrin was applied and allowed to evaporate for a few minutes. The filter papers were then placed into Petri dishes (9 cm in diameter). Positive control treatments were treated with LC_{20} of deltamethrin, and the control were treated with water. 20 adults were placed into each Petri dish separately and then covered.

Statistical Analysis:

Data was analyzed to one-way ANOVA at a probability level of 0.05%, with individual pairwise comparisons done using Tukey's HSD in a Co-Stat software.

RESULTS

Chemical Composition of The Wood Vinegar:

The chemical composition of wood vinegar is listed in (Table1) was analyzed by GC-MS. As shown in the table the major component of the wood vinegar is the phenolic compounds represented by Syringol (48.98%), phenol 4,5 dimethoxy -2-methyl (4.16%), Guaiacol-4-Ethyl (3.05%) and Mequinol (2.61). the next major component is benzene derivatives including Estragole (33.09%), Benzene,1,2,5 -trimethoxy-3-methyl (2.65%) and Butylated hydroxytoluene (1.69%) and the minor component is ketones which are represented by one compound, corylone (3.76%).

Table 1: Component (%) of *Ficus benjamina* trees Wood vinegar.

Components	Retention time (min)	Peak area%	Molecular weight (g /mol)
Syringol (Phenol, 2,6 dimethoxy)	11.73	48.89	154
Estragole	8.28	33.09	148
Phenol,4,5 dimethoxy- 2-methyl	3.68	4.16	168
Corylone	5.36	3.76	112
(Guaiacol -4-Ethyl)	10.09	3.05	152
Benzene,1,2,5-Trimethoxy-3-Methyl	15.24	2.65	182
Mequinol	6.32	2.61	124
Butylated Hydroxytoluene	14.73	1.69	220

Bioassay:

The results of *S. oryzae* mortality using a film residue method on filter papers with the wood vinegar from *Ficus benjamina* trees at 350 °C. And the mixture of wood vinegar and LC_{20} deltamethrin (Table 2). From the average percentage mortality data obtained, it was evident that WV alone doesn't have very little toxic effects on *S. oryzae*. At 100% concentration of WV, the mortality average was 32.5%, WV. but the *S. oryzae* adults showed increasing percent mortality after application of wood vinegar and LC_{20} (0.024X10-4 %) of deltamethrin. In general, mortality percentage increased with increasing concentration of WV. The treatment of mixed wood vinegar (5 %) and LC_{20} of deltamethrin showed (90%) mortality with had significant differences ($P<0.05$).

The wood vinegar from *Ficus benjamina* does not achieve any fumigant toxicity toward S. *oryzae*. Whereat the highest concentration (200 µl/l) mortality was 10%.

Table 2: Contact effect of WV deltamethrin and LC_{20} deltamethrin + WV on *S. oryzea* mortality after three days exposure periods.

Material	Conc. (mg/cm²)	Conc. %	Mortality%±SE after 3dayes
Wood Vinegar	control	0	3.3±3.3b
	0.63	40	5.0± 2.9b
	1.26	80	10.0± 5.0b
	1.57	100	32.5± 1.4a
deltamethrin	control	0	3.33±3.3c
	0.0003	$0.023X10^{-4}$	20 ±2.3c
	0.0007	$0.5 X10^{-4}$	60±8.6b
	0.023	$1.5 X10^{-4}$	75±2.8ab
	0.031	$2 X10^{-4}$	80±2.8ab
	0.062	$4 X10^{-4}$	84±2.3a
LC_{20} ($0.024X10^{-4}$ %) deltamethrin+ wood vinegar	control	0	0.6±0.6f
	0.002	0.1	25.0±5.7e
	0.008	0.5	35.0±2.8d
	0.02	1	60.0±5.0c
	0.05	3	65.0±0.0c
	0.08	5	90.0±0.0b
	0. 2	10	100.0±0.0a

DISCUSSION

The GC-MS analysis of the WV prepared from *Ficus benjamina* and this work showed that the phenolic compounds present the most abundant component in the wood vinegar prepared from *Ficus benjamina* followed by the benzene derivatives as listed in table (1) which agree with the results of a study conducted by Yang *et al.,* 2016 who found that the GC-MS analysis of WV prepared from *Litchi chinensis* mainly consist of the syringe, guaiacol and 3,5 dimethoxy toluene as the major component (Yang *et al.,* 2016). As reported by many studies, wood vinegar is rich in phenolic compounds (Baimark & Niamsa, 2009; Ma *et al.,* 2011; Velmurugan *et al.,* 2009) which can be produced from the thermal composition of lignin (Yang *et al.,* 2016). The major compound Syringol is known to have antioxidant activity (Loo *et al.,* 2008). The next major component in the wood vinegar is estragole which was reported to have insecticidal activity when tested against three tephritid fruit fly species *Bactrocera cucurbitae* (Coquillett), *Ceratitis capitata* (Wiedemann) and *Bactrocera dorsalis* (Hendel), in a study conducted by Chang *et al.,* 2009 as the results showed that estragol caused knockdown effect recorded by the value of LT90 faster in *Bactrocera dorsalis* than *Ceratitis capitata*and *Bactrocera cucurbitae* (Chang et al., 2009).

The toxicity results show the low insecticidal activity of wood vinegar alone against the tested insect, *Sitophilus oryzae* although the concentration of wood vinegar rich 100% the recorded mortality was only 32.5%, the treatment of insect with wood vinegar at low concentrations mixed with the LC_{20} of deltamethrin give very high mortality which record to be 100% with only 10% of wood vinegar which agree with the work of Kim et al 2008 who found the low insecticidal activity of wood vinegar against two species of planthoppers, while when mixed with carbosulfan, the mortality was significantly increased (Kim *et al.,* 2008a). Similar results were recorded by Hashemi *et al.,* 2014 as the treatment of *Lasioderma serricorne* with WV alone didn’t show insecticidal activity while when mixed with the methanol extract of *Salvia leriifolia*, the mortality increased greatly compared with the individual treatment methanol extract (Hashemi *et al.*, 2014).

Also, the highest mortality was recorded against the housefly (*Musca Domestica*) when treated with the mixture of wood vinegar and citronella extract according to Pangnakorn and Kanlaya 2014 (Pangnakorn & Kanlaya, 2014). Another study showed the low larvicidal activity of wood vinegar against the armyworm, *Spodoptera litura* (Rahmat *et al.*, 2015)

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Consent for publication: Not applicable' for that section.

Availability of data and materials: The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

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