

- OKONKWO, E.U. UND W.I. OKOYE, 1996. The efficacy of four seed powders and the essential oils as protectants of cow-pea and maize grain against infestation by *Callosobruchus maculatus* (Fabricius) (Coleoptera: Bruchidae) and *Sitophilus zeamais* (Coleoptera: Curculionidae) in Nigeria. Int. J. Pest Manage., **42**: 143-146.
- PASCUAL, N., MARCO, M.P. UND X. BELLES, 1990. Azadirachtin induced imaginal moult deficiencies in *Tenebrio molitor* L. (Coleoptera: Tenebrionidae). J. Stored Prod. Res., **26**: 53-57.
- PHILLIPS, T.W. UND T.E. THRONE, 2010. Biorational approaches to managing stored-product insects. Ann. Rev. Entomol., **55**: 375-397.
- PHILOGENE, B.J.R., 1991. L'utilisation des produits naturels dans la lutte contre les insectes: problemes et perspectives. La lutte anti-acridienne. AUPELF-UREF (ed.). Paris. 269-278.
- RAHMAN, M.M. UND G.H. SCHMIDT, 1999. Effect of *Acorus calamus* (L.) (Araceae) essential oil vapours from various origins on *Callosobruchus phaseoli* (Gyllenhal) (Coleoptera: Bruchidae). J. Stored Prod. Res., **35**: 285-295.
- SAGHEER, M., YASIR, M., KHAN, B.S. UND M. HASAN, 2011. Ovicidal and reproduction inhibition activity of flufenoxuron, an acylurea insect growth regulator, against *Tribolium castaneum* (Herbst) (Coleoptera: Tenebrionidae). Pak. Entomol., **33**: 131-136.
- SCHMUTTERER, H., 1995. The neem tree, *Azadirachta indica* A. Juss. and other meliaceous plants: Sources of unique natural products for integrated pest management, medicine, industry and other purposes. VCH publishers Inc. pp: 696.
- SENTHIL-NATHAN, S., KALAIVANI, K., CHUNG, P.G. UND K. MURUGAN, 2006. Effect of biopesticides on the lactate dehydrogenase (LDH) of the rice leaf folder, *Cnaphalocrocis medinalis* (Guenee) (Insecta: Lepidoptera: Pyralidae). Ecotoxicol. Environ. Saf., **65**: 102-107.
- SENTHIL-NATHAN, S., CHUNG, P.G. UND K. MURUGAN, 2004. Effect of botanical insecticides and bacterial toxins on the gut enzyme of the rice leaf folder *Cnaphalocrocis medinalis*. Phytoparasitica, **32**: 433-443.
- SILVA, J. P., CROTTI, A.E.M. UND W.R. CUNHA, 2007. Antifeedant and allelopathic activities of the hydro alcoholic extract obtained from Neem (*Azadirachta indica*) leaves. Revista Brasileira de Farmacognosia, **17**: 529-532.
- SINGH, R.P., 1993. Neem for the management of stored grain insects in developing countries. World Neem Conf. Bangalore, India, Souvenir, pp. 69-80.
- STOLL, G., 2000. Natural Crop Protection in the Tropics, letting information comes to life. Margraf Verlag 2nd enlarged and revised edition; 104-243.
- SU, H.F.C., 1990. Biological activities of hexane extract of *Piper cubeba* against rice weevils and cowpea weevils (Coleoptera: Curculionidae). J. Entomol. Sci., **25**: 16-20.
- SUBBRAMANYAM, B. UND D.W. HAGSTRUM, 1996. Resistance measurement and management. In B. Subramanyam, D.W. Hagstrum (eds.) Integrated management of insects in stored products. New York, Marcel Dekker, Inc., 426, pp. 331-397.
- TREMATERRA, P. UND A. SCIARRETTA, 2002. Activity of chilli, *Capsicum annum* L. var. acuminatum, on stored product insects *Oryzaephilus surinamensis* (L.), *Sitophilus oryzae* (L.) and *Tribolium castaneum* (Herbst). Bull., **25**: 177-182.
- TRIPATHI, A.K., PRAJAPATI, V., AGRAWAL, K.K., KHANUJA, S.P.S. UND S. KUMAR, 2000. Toxicity towards *Tribolium castaneum* in the fraction of essential oil of *Anethum sowa* seeds. J. Med. Arom. Plant Sci., **22**: p. 40.
- VERMA, N., TRIPATHI, A.K., PRAJAPATI, V., BAHL, J.R., KHANUJA, S.P.S. UND S. KUMAR, 2000. Toxicity of essential oil from *Lippia alba* towards stored grain insects. J. Med. Arom. Plant Sci., **22**: p.50.
- WANG, J.J., TASI, H., DING, W., ZHAO, Z.M. UND L.S. LI, 2001. Toxic effects of six plant oils alone and in combination with controlled atmosphere on *Liposcelis bostrychophila* (Psocoptera: Liposcelididae). J. Econ. Entomol., **94**: 1296-1301.
- WEATHERSBEE, A.A. UND Y.Q. TANG, 2002. Effect of neem seed extract on feeding, growth, survival, and reproduction of Diaprepes abbreviatus (Coleoptera: Curculionidae). J. Econ. Entomol., **95**: 661-667.

Toxicity and repellence of *Citrus jambhiri* Lush rind essential oil against maize weevil (*Sitophilus zeamais* Motschulsky 1855) (Coleoptera: Curculionidae)

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Abstract

Rind of matured fruits of *Citrus jambhiri* Lush was hydro-distilled to obtain essential oil (EO) which was subjected to Gas chromatography-Mass spectrometry (GC-MS) analysis. The EO was evaluated for fumigant toxicity (at 27-107 µL/L air) and repellence against maize weevil (*Sitophilus zeamais* Motschulsky). Area preference methodology was used to evaluate the repellence of the EO at 0.15-0.9 µL/cm², while isopropanol served as control for both bioassays. The experiments were set up in a completely randomized design and data were subjected to analysis of variance and probit analysis. Fifty-two compounds were identified in the EO with the

predominant compounds being α -terpineol (8.03%), citral (7.00%), 4-terpineol (6.52%), caryophellene (4.58%), cis-geraniol (4.44%), citronellal (4.38%), β -bisabolene (4.01%) and n-hexadecanoic acid (4.70%). Others were α -bergamotene (3.74%), lemonol (3.23%), precocene I (3.33%) and β -copaene (3.09%). Toxicity progressed with EO dose and exposure period and application of EO at 80 and 107 μ L/L air caused significantly higher mortality (33.75-100.00%) than isopropanol (0.00-22.50%). Lethal time for 50% assayed weevil (LT_{50}) for the EO application at 107 μ L/L air {7.51 (6.95-8.13) h} was significantly lower than the values obtained for 27 and 53 μ L/L air {44.78 (27.49-312.61) and 21.87 (11.91-45.96) h, respectively}. EO caused significantly higher repellence (75.00-90.00%) than control (15.00%) at 24 hours after treatment. The results indicate that *C. jambhiri* rind EO has prospects as effective biorational formulation for control of maize weevil.

Keywords: *Citrus jambhiri*, Essential oil, Fumigant toxicity, GC-MS, Lethal time, Maize weevil

1. Introduction

Maize weevil (*Sitophilus zeamais* Motschulsky 1855) (Coleoptera: Curculionidae) is a cosmopolitan tropical postharvest pest attacking cereals, processed tubers and their products (Haines, 1990; Babarinde et al., 2008, 2013). Its infestation can cause quantitative loss like weight reduction, or qualitative losses like reduction of aesthetic/market value, nutritional values and loss of seed germination ability, especially when weevils feed on the seed's embryo. Its control with synthetic chemicals, although very effective, can cause several economic, ecological and health risks (Arthur, 1996); hence the need for bio-rational control options for the pest. Several formulations of botanicals had been studied (Dales, 1986), but essential oils (EOs) are receiving renewed attention due to the fact that they are effective at low concentrations, even without direct contact with the target organisms (Babarinde et al., 2015). Due to the multiple bioactive compounds present in EOs and their multiple sites and modes of action, the tendency for a pest to develop resistance against EOs is comparatively low (Venkitanarayanan et al., 2013)

Several authors have worked on different *Citrus* species as protectants of stored produce against postharvest pests (Don-Pedro, 1996; Dutra et al., 2016; Fouad and Camara, 2017; Lu, 2017; Oboh et al., 2017). The need to convert agricultural waste product into useful by-products is a major concern in many developing countries. This is primarily because of poor waste management practices peculiar to the region. *C. jambhiri* rind usually constitute a nuisance compared to other agricultural wastes that can be converted to positive use like animal feed, for instance. Therefore, any positive use of the waste product will be embraced by rural dwellers to whom its handling is an ecological or economic burden. To a very great extent, evaluation of the insecticidal properties of *C. jambhiri* seems to be scarce in the literatures. Different parts of *C. jambhirir* have differs uses. In India, its ground root is orally administered to control vomiting (Tiwari et al., 2017) In Nigeria, it is formulated into concoction for medicinal purposes like tooth whitening and human weight loss. The fruit is also used for making local juice. Despite its several uses, the rind id usually thrown away after peeling without any productive utilization. In some rural areas of south western Nigeria, *Citrus* species rinds are often dried and put on indoor charcoal fire to produce smoke which acts either as toxicant or repellent to mosquitoes This ethno botanical practice was one of the major thrusts for this research. The study was therefore designed with the following objectives (a) To evaluate the fumigant toxicity and repellence of *Citrus jambhiri* rind EO against *Sitophilus zeamais*, and (b) To identify the chemical components of the EO using Gas chromatography-Mass spectrometry.

2. Materials and methods

2.1 Insect culture

Sitophilus zeamais was reared on Tsolo variety maize under laboratory conditions of $28\pm 2^{\circ}\text{C}$ temperature and $70\pm 3\%$ relative humidity as described by Babarinde et al. (2008).

2.2 Essential oil Extraction and Chromatographic analysis

Rinds of freshly harvested *C. jambhiri* fruits were manually removed and pounded in a mortal with the aid of a pestle. EO was extracted from the rind using hydro distillation method (British

Pharmacopoeia, 1988; Babarinde *et al.*, 2017b). The EO was subjected to Gas chromatography-Mass spectrometry (GC-MS) using the following procedures. EO (1.0 μL) was injected into a GC-MS machine (GCMS-QP2010SE^o, a product of Shimadzu, Kyoto, Japan), equipped with an AOC-20i auto sampler and a split injector (split ratio 1:50). The description and conditions of the GC-MS machine are as follow. Column used: Optima^o 5MS (a product of Macherey – Nagel, USA) (30 m \times 0.25 mm internal diameter \times 0.25 μm film thickness) coated with 95% dimethylpolysiloxane 5% diphenyl packing materials; helium was the carrier gas at 56.2 kPa inlet pressure and 36.2 cm/s linear velocity, 3 and 0.99 ml/min purge flow rate, respectively. Oven temperature began at 60 °C and ramp of 10 °C/min up to 180 °C held for 2 min, and subsequent increase to 280 °C with a 15 °C/min heating ramp at 280 °C for 4 min. Injection temperature was 250 °C. The MS operating conditions were as follows: ionization with an ion trap detector in full scan mode under electron impact ionization (EI) at 70 eV, ion source temperature 200 °C; interface temperature 250 °C, scan range, 40–700 m/z. The identification of the components was done as earlier described (Adams, 2001; Joulain and Koenig, 1998; Babarinde *et al.*, 2017b).

2.3 Entomological bioassays

2.3.1 Fumigant toxicity bioassay

Varying doses of *C. jambhiri* EO (27, 53, 80 and 107 $\mu\text{L/L}$ air) were separately dissolved in 0.2 mL isopropanol and applied to 8 cm² Whatman filter paper attached to the inner surface of the cork of 750 mL capacity fumigation chamber. Isopropanol (0.2 mL) served as control. Twenty 1-3-day old mixed sex *S. zeamais* adults were introduced into the fumigation chamber and covered. Mortality data were collected at 1, 3, 6, 12, 24 and 48 h after treatment (HAT). The weevils were adjudged to be dead when they were unable to move their body parts after a gentle shaking of the fumigation chamber.

2.3.2 Repellence bioassay

Area preference methodology was adopted for the repellence bioassay. Whatman filter paper (9 cm diameter) was used following the method of McDonald *et al.* (1970), which was modified by Zhang *et al.* (2015) and Babarinde *et al.* (2017b) using the following doses: 0.15, 0.30, 0.45 and 0.90 $\mu\text{L/cm}^2$. Each dose was separately dissolved in 0.2 mL isopropanol, while isopropanol served as control. Twenty insects similar in all respects with those used for fumigant toxicity bioassay were introduced into the repellence chamber. Numbers of insects on the treated (Nt) and untreated (Nc) discs of the filter paper were counted at 24 hours after treatment and percentage repellence (PR) was calculated using the formula:

$$\text{PR} = \{(\text{Nc}-\text{Nt})/(\text{Nc}+\text{Nt})\} \times 100$$

2.4 Experimental design statistical analyses

The experiments were set up in completely randomized design and data were subjected to analysis of variance (ANOVA), while significant means were separated using Studentized Neuman Keuls (SNK) post-hoc test at 5% probability level. Lethal times (LT₅₀ and LT₉₀) for each of the EO doses were determined using probit analysis. All statistical analyses were done with the aid of SPSS software package version 16 (SPSS, 2006).

3. Results

3.1 Chromatographic analysis

A total of 52 chemical compounds were identified in *C. jambhiri* rind EO. Among the identified chemical groups were monoterpenoid and sesquiterpenoid compounds (hydrocarbon and oxygenated or alcohols), fatty acids and aldehydes. Major compounds were α -terpineol (8.03%), citral (7.00%), 4-terpineol (6.52%), citronellal (4.38%), β -bisabolene (4.01%) and n-hexadecanoic acid

(4.70%). Others were caryophellene (4.58%), lemonol (3.23%), precocene I (3.33%) and β -copaene (3.09%) (Table 1).

3.2 Entomological bioassays

At 1 Hour after treatment (HAT), there was no mortality due to the applied treatments. Observed mortality in all EO doses (5.00-100.00%) was significantly higher than mortality observed in isopropanol (0.00-22.50%). At 3 HAT, treatment had no significant ($df=4,19$; $F=3.29$; $p=0.051$) effect on weevil's mortality; thereafter, toxicity increased with increase in EO doses. At 6 HAT, mortality observed when 80 $\mu\text{L/L}$ air (33.75%) and 107 $\mu\text{L/L}$ air (40.00%) was significantly ($df=4,19$; $F=10.317$; $p<0.0001$) higher than the observed mortality when lower doses of EO and isopropanol were applied (0.00-16.25%). At 12-48 HAT, all EO doses caused significantly ($p<0.0001$) higher mortality (28.75-100.00%) than 5.00-22.5% mortality observed in isopropanol (Table 2).

The results of probit analysis followed similar pattern as ANOVA results. LT_{50} for EO applied at 107 $\mu\text{L/L}$ air {7.51(6.95-8.13) h} was significantly lower than the values for 53 $\mu\text{L/L}$ air {21.87(11.91-45.96) h} and 27 $\mu\text{L/L}$ air {44.78(27.49-312.62) h}. LT_{90} values followed the same pattern; application of EO at 107 $\mu\text{L/L}$ air had significantly lower value {11.7(10.82-12.85) h} than 18.33(13.02-42.72) - 87.53(53.25-804.05) h observed in other EO doses (Table 3). All EO doses caused significantly ($df=4, 19$; $F=5.173$; $p=0.008$) higher percentage repellence (75.00-90.00%) than isopropanol (15.00%) (Fig. 1).

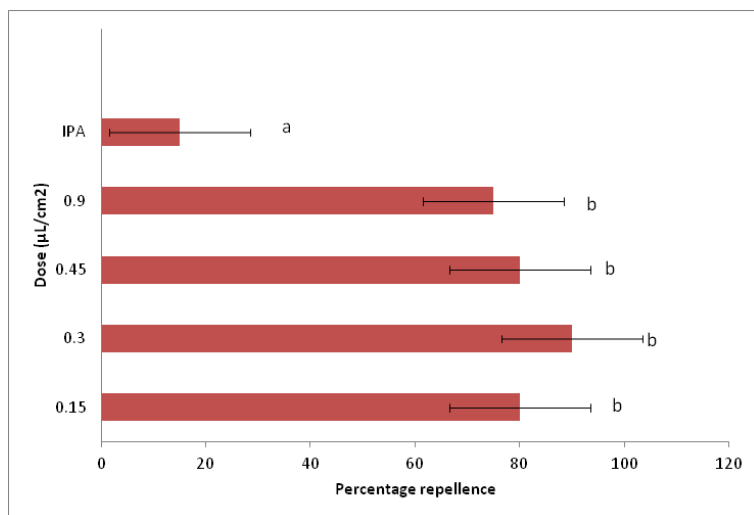


Fig. 1: Percentage repellence of *Citrus jambhiri* rind essential oil against *Sitophilus zeamais*

Means with the same letters of alphabet are not significantly different using SNK at 5% probability level.

IPA: Isopropanol (used as spreading agent for the essential oil and control).

ANOVA Results: $df=4, 19$; $F=5.173$; $p=0.008$

Tab. 1: Chemical composition of *Citrus jambhiri* rind essential oil

S/N	Retention Time	Name	% Composition
1.	4.215	1-nonanol	0.89
2.	4.303	4-pentadecyne, 15 chloro	0.8
3.	4.431	4-terpineol	6.52
4.	4.684	α -terpineol	8.03
5.	5.002	Citronellal	4.38
6.	5.137	Cis-geraniol	4.44

7.	5.349	Decanol	1.97
8.	5.441	Lemonol	3.23
9.	5.574	Alfol 10	1.73
10.	5.636	Cyclohexene, 2-ethenyl-1,3,3-trimethyl	3.8
11.	5.774	Allyl trisulphide	0.31
12.	5.863	Mentha-1,8-dien-7-yl acetate	0.65
13.	6.047	P-menth-3-ene, 2-isopropenyl-1-vinyl-, (15,2R)-(-)	1.72
14.	6.283	(-) Carvone	2.11
15.	6.440	Citral	7.0
16.	6.726	Perillaaldehyde	1.30
17.	6.978	Cyclohexene, 1-ethenyl-1-menthyl-2,4 bis(1-methyle nemy)-(15-1-apha)	1.8
18.	7.182	Neryl acetate	3.19
19.	7.366	α -bergamotene	3.74
20.	7.472	Genanyl acetate	2.44
21.	7.578	Caryophyllene	4.58
22.	7.975	Acetic acid, chloro- decyl ester	0.64
23.	8.062	Humulene	1.07
24.	8.298	β -bisalolene	4.01
25.	8.434	β -copaene	3.09
26.	8.629	Precocene 1	3.33
27.	8.727	β -sesquiphollendene	0.22
28.	9.371	Squalene	0.57
29.	9.649	Nerolidyl acetate	2.27
30.	10.019	Dodecanoic acid	0.51
31.	10.985	Spathulenol	0.45
32.	11.241	Viridiflorol	0.42
33.	11.372	α - bisabolol	0.65
34.	11.856	Farnesol	0.15
35.	12.036	(E)- stilebene	0.35
36.	12.256	3-methyldiadamenthane	0.21
37.	12.491	2,6,10 Trimetnyl-2,6,9,11-dodecatetraenal	0.44
38.	13.077	Isocarveol	0.22
39.	13.382	Eicosquonic acid	0.14
40.	13.648	Spiro (andnot-5-ene-17	0.08
41.	13.851	Methyl 16-hydroxy-hexadecanoate	0.06
42.	14.249	n-hexadecanoic acid	4.7
43.	14.424	(6E)-nerolidol	0.64
44.	14.932	Dimethoxybicyclo(3.3.1) nonane-2-4-dione	0.51
45.	15.052	2-4-diflorobenzene,1-benzylosy	0.29
46.	15.234	Phytol	0.23
47.	15.483	Oleic acid	3.37
48.	15.615	Octadecanoic acid	3.34
49.	16.699	Dipalmitin	0.69
50.	18.000	Palmitin,2-mono	2.15
51.	19.208	Oleic acid chloride	1.21
52.	19.367	α -monostearin	1.0

The components are listed in ascending order of retention time

Percentage composition is the percentage peak area relative to total peak area obtained from total ion chromatogram peak report

Tab. 2: Percentage mortality of *Sitophilus zeamais* exposed to *Citrus jambhiri* rind essential oil in fumigant bioassay.

Treatment ($\mu\text{L/L air}$)	Duration after treatment (h)					
	1	3	6	12	24	48
Isopropanol	0.00 \pm 0.0	0.00 \pm 0.0	0.00 \pm 0.0a	5.00 \pm 3.5a	10.00 \pm 5.8a	22.5 \pm 4.3a
27	0.00 \pm 0.0	5.00 \pm 3.5	15.00 \pm 3.5a	28.75 \pm 3.1b	35.00 \pm 2.9b	46.25 \pm 3.8b
53	0.00 \pm 0.0	0.00 \pm 0.0	16.25 \pm 3.8a	45.00 \pm 7.4c	63.75 \pm 10.3c	90.00 \pm 4.6c
80	0.00 \pm 0.0	3.75 \pm 1.25	33.75 \pm 8.9b	72.50 \pm 2.5d	93.75 \pm 6.3d	100.00 \pm 0.0c
107	0.00 \pm 0.0	7.50 \pm 1.4	40.00 \pm 4.1b	88.75 \pm 2.4e	100.00 \pm 0.0d	100.00 \pm 0.0c
ANOVA	-	df=4,19	df=4,19	df=4,19	df=4,19	df=4,19
Result		F=3.29 p=0.051	F=10.317 p<0.0001	F=63.379 p<0.0001	F=39.398 p<0.0001	F=116.621 p<0.0001

Values are means of four replicates \pm S.E. Means followed by the same letters of alphabet are not significantly different using SNK at 5% probability level.

Tab. 3: Lethal time (h) of *Citrus jambhiri* rind essential oil against *Sitophilus zeamais*.

Dose ($\mu\text{L/L air}$)	LT ₅₀ (FL)	LT ₉₀ (FL)	χ^2	P	DF	Slope
27	44.78(27.49-312.62)	87.53(53.25-804.05)	31.617	0.001	4	-14.483
53	21.87(11.91-45.96)	40.93(27.75-109.19)	53.135	<0.0001	4	-15.116
80	10.51(6.18-19.23)	18.33(13.02-42.72)	42.93	<0.0001	4	-13.633
107	7.51(6.95-8.13)	11.7(10.82-12.85)	6.26	0.041	4	-13.084

FL: Fiducial limits

4. Discussion

The result of the GC-MS analysis of *C. jambhiri* rind oil shows that the oil was predominated by terpenoid compounds. Apart from the terpenes, other chemical groups identified were aldehydes, alcohols and fatty acids. Monoterpenes and sesquiterpenes have been associated with the bioactivity of EO against many invertebrate pests (Obeng-Ofori and Reichmuth, 1999; Yildirim *et al.*, 2013; Saad *et al.*, 2018). Previous studies (Usman *et al.*, 2016; Fouad and Camara, 2017) on chemical components of different parts of *Citrus* species show the dominance of terpenoid compounds. The variations in the constituents of EOs are attributable to the difference in the activity of the synthases that mediate the formation of the compounds from their respective precursors (Degenhardt, 2009). Basically, however, the disparity in the chemical composition of any plant could be the differences in the species studied and the environmental factors involved in its cultivation (Jemaa *et al.*, 2012; Fouad and Camar, 2017).

The toxicity of *C. jambhiri* EO against *S. zeamais* conforms to recent studies (Campolo *et al.*, 2014; Dutra *et al.*, 2016; Heidari *et al.*, 2017; Oboh *et al.*, 2017) on the bioactivity of EO obtained from *Citrus* species against stored product pests. According to Don-Pedro (1996), toxicity of *Citrus limon* against *Callosobruchus maculatus*, *S. zeamais* and *Dermestes maculatus* depended on strong fumigation action. Kumar and Tiwari (2016) reported the fumigant toxicity of *Citrus reticulata* against *Sitophilus oryzae*. Apart from *Citrus* species, other botanical EOs which have been reported to be toxic or repellent against *Sitophilus* species include *Hoslundia opposita* (Babarinde *et al.*, 2017a), *Lippia javanica* (Kamanula *et al.*, 2017), *Teucrium capitatum* and *Salvia pomifera* subsp. *calycina* (Koutsaviti *et al.*, 2018). Although the mechanism of toxicity of the EO against *S. zeamais* was not covered in the scope of this study, fumigant toxicity of another EO (*X. parviflora*) against *C. maculatus* was suggested to be due to the inhalation of the EO which led to neurotoxicity and eventual mortality (Babarinde *et al.*, 2015). The fumigant toxicity of *C. jambhiri* implies that the EO can be used to protect infested maize against the damage of *S. zeamais*.

Whereas Dutra *et al.* (2016) classified the repellence of the EO from four *Citrus* species against *C. maculatus* as neutral; our study shows *C. jambhiri* to be repellent against *S. zeamais*. The disparity in the result of Dutra *et al.* (2016) and the present study was due to the differences in the studied Coleoptera species and their physiological responses to the exposures to the chemical compounds in the different EOs. Repellence of *Citrus reticulata* EO against *Cryptolestes ferrugineus* has been reported by Lu (2017). The repellence of the *C. jambhiri* rind EO against *S. zeamais* implies that the EO

is effective to prevent re-infestation after an initial effective control of *S. zeamais* in stored maize. It also indicates the ability of the EO to prevent the infestation of non-resident pest population (Lale and Alaga, 2001; Babarinde *et al*, 2014).

5. Conclusion

C. jambhiri EO was effective as a toxicant and repellent against *S. zeamais*. With the appreciable number (52) of identified chemical compounds in the EO, the tendency of the development of resistance against the EO by maize weevil is low. Therefore, the EO can be incorporated into Integrated Weevil Management scheme. Since citrus rind is often thrown away after peeling, the results of this research have established a potential of the waste product of *C. jambhiri* in the pest control segment. The scope of the study did not extend to the evaluation of the bioactivity of the prominent chemical constituents, we therefore suggest that the observed bioactivity of the EO could be due to the combined effects of the chemical constituents identified in *C. jambhiri*.

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References

- ADAMS, R.P. 2001: Identification of essential oil components by gas chromatography and mass spectrometry. Carol Stream, IL: Allured Publishing Corporation.
- ARTHUR, F.H. 1996: Grain Protectants: Current status and prospects for the future. *Journal of Stored Product Research* **37**, 291-302.
- BABARINDE, S.A., AKINYEMI, A.O., USMAN, L.A., ODEWOLE, A.F., SANGODELE, A.O., IYIOLA, O.O., AND OLALERE, O.D. 2014: Toxicity and repellency of *Hoslundia opposita* Vahl (Lamiaceae) leaves' essential oil against rust red flour beetle, *Tribolium castaneum* Herbst (Coleoptera:Tenebrionidae). *Natural Product Research* **28**, 365-371.
- BABARINDE, S.A., BABARINDE, G.O., ODEWOLE, A.F., AND ALAGBE O.O. 2013: Effect of the prevalent insect species of yam chips on consumers' acceptability of yam paste. *Agricultura Tropica et Subtropica* **43**, 97-101.
- BABARINDE, S.A., OLANIRAN, O.A., USMAN, L.A., ESAN, E.O., AFOLABI, A., SANMORI, O., AND LOMOWU, J.D. 2017a: Comparative sensitivity of maize weevil to essential oil of *Hoslundia opposita* Vahl leaves subjected to different drying regimes. *Acta Fytotechnica et Zootechnica* **20**, 54-59.
- BABARINDE, S.A., PITAN, O.O.R., AJALA, M.O., AND OLATUNDE, G.O. 2017b: Insectifugal and insecticidal potentials of two tropical botanical essential oils against cowpea seed bruchid. *Environmental Science and Pollution Research*, **24**, 19785-19794.
- BABARINDE, S.A., PITAN, O.O.R., OLATUNDE, G.O., AND AJALA, M.O. 2015: First report of toxicity of *Xylopija parviflora* (A. Rich.) Benth (Annonaceae) root bark's essential oil against cowpea seed bruchid, *Callosobruchus maculatus* Fabricius (Coleoptera: Chrysomelidae: Bruchinae), *Natural Product Research* **29**, 349-352.
- BABARINDE, S. A., SOSINA, A., AND OYEYIOLA, E. I. 2008: Susceptibility of the selected crops in storage to *Sitophilus zeamais* Motschulsky in south western Nigeria. *Journal of Plant Protection Research* **48**, 541-550.
- BRITISH PHARMACOPOEIA. 1988: Vol. 2. Her Majesty's Stationery Office HMSO, London.
- CAMPOLO, O., MALACRINO, A., ZAPPALÀ, L., LAUDANI, F., CHIERA, E., SERRA, D., RUSSO, M., AND PALMERI, V. 2014: Fumigant bioactivity of five Citrus essential oils against *Tribolium cconfusum*. *Phytoparasitica* **42**, 223-233.
- DALES, M.J. 1996: A review of plant materials used for controlling insect pests of stored products. Natural Resources Institute (NRI) Bulletin 65 Chatham United Kingdom.
- DEGENHARDT J, KELLNER TG, AND GERSHENZON J. 2009: Monoterpene and sesquiterpene syntheses and the origin of terpene skeletal diversity in plants. *Photochemistry* **70**, 1621-37.
- DON-PEDRO, K.N. 1996. Fumigant toxicity of *Citrus* peel oils against adult and immature stages of storage insect pests. *Pest Management Science* **47**, 213-223.
- DUTRA, K.A., OLIVEIRA, J. V., NAVARRO, D.M.A., F., BARBOSA, D.R.S., AND SANTOS, J.P.O. 2016: Control of *Callosobruchus maculatus* (Fabr.) (Coleoptera: Chrysomelidae: Bruchinae) in *Vigna unguiculata* (L.) Walp. with essential oils from four *Citrus* spp. plants. *Journal of Stored Product Research* **68**, 25-32
- FOUAD H.A. AND CAMARA, C.A.G. 2017: Chemical composition and bioactivity of peel oils from *Citrus aurantiifolia* and *Citrus reticulata* and enantiomers of their major constituent against *Sitophilus zeamais* (Coleoptera: Curculionidae). *Journal of Stored Product Research* **73**, 30-36.
- HAINES C.P. 1991: Insects and arachnids of tropical stored products: Their biology and identification: A training manual. Natural Research Institute, Kent, UK, 246 pp.

- HEIDARI, F., SARAILOO M., GHASEMI, V., AND NADIMI, A. 2017: Toxic and oviposition deterrence activities of essential oils from *Citrus sinensis* (L.) Osbeck and *Citrus paradisi* (Macfarlane) fruit peel against adults of *Tribolium castaneum* (Herbst). *Journal of Crop Protection* **6**, 79-88
- JEMAA, J.M.B, TERSIM, N., TOUDERT, K.T, AND KHOJA, M.H. 2012: Insecticidal activity of essential oils from leaves of *Laurus nobilis* from Tunisia, Algeria and Morocco, and comparative chemical composition. *Journal of Stored Product Research* **48**, 97-104.
- JOULAIN D. AND KOENIG WA. 1998: The atlas of spectra data of sequiterpene hydrocarbons. Hamburg, Germany E.B. Verlag Stream.
- Kamanula, J.F., Belmain, S.R., Hall, D.R., Framan, D.I., Goyder, D.J., Brighton, M.M., Masumbu, F.F., and Stevenson, P.C. 2017: Chemical variation and insecticidal activity of *Lippia javanica* (Burm. f.) Spreng essential oil against *Sitophilus zeamais* Motschulsky. *Industrial Crops and Products* **110**, 75-82.
- KOUTSAVITI, A., ANTONOPOULOU, V., VLASSI, A. ANTONATOS, S., MICHAELAKIS, A., PAPACHRISTOS, D.P., AND TZAKOU, O. 2018: Chemical composition and fumigant activity of essential oils from six plant families against *Sitophilus oryzae* (Col: Curculionidae). *Journal of Pest Science* **91**, 873-886.
- KUMAR, R. AND TIWARI, S.N. 2016: Fumigant toxicity of essential oils and their combination against *Sitophilus oryzae* (Coleoptera: Curculionidae) at different days interval in stored wheat, *Journal of Postharvest Technology* **4**, S06-S10.
- LALE, N.E.S. AND ALAGA, K.A. 2001: Exploring the insecticidal, larvicidal and repellent properties of *Piper guineense* schum et Thonn. Seed oil for the control of frust-red flour beetle *Tribolium castaneum* (Herbst) in stored pearl millet *Pennisetum glaucum* (L) R. Br/ *Journal of Plant Diseases and Protection* **108**, 305-313.
- LÜ J.H. 2017: Effect of *Citrus reticulata* Blanco essential oil on *Cryptolestes ferrugineus* (Stephens) adults. *Journal of Food Protection* **80**, 2090-2093.
- LUND, E.D., SHAW, P.E., KIRKLAND, C.L. 1981. Composition of rough lemon leaf oil. *Journal of Agricultural and Food Chemistry* **29**, 490-494
- MCDONALD LL, GUYRH, SPIERS RD. 1970: Preliminary evaluation of new candidate materials as toxicants, repellents and attractants against stored-product insects -1. Marketing Research Report No. 882. Washington (DC): Agriculture Research Service, USA Department of Agriculture.
- OBENG-OFORI, D. AND REICHMUTH CH. 1999: Plant oils as potentiation agents of monoterpenes for protection of stored grains against damage by stored product beetle pests. *International Journal of Pest Management* **45**, 155-159.
- OBOH, G., ADEMOSUN, A.O., OLUMUYIWA, T.A., OLASEHINDE, T.A., ADEMILUYI, A.O., AND ADEYEMO, A.C. 2017: Insecticidal activity of essential oil from orange peels (*Citrus sinensis*) against *Tribolium confusum*, *Callosobruchus maculatus* and *Sitophilus oryzae* and its inhibitory effects on acetylcholinesterase and Na⁺/K⁺-ATPase activities. *Phytoparasitica* **45**, 501-508.
- SAAD, M.M.G., ABOU-TALEB, H.K., AND ABDELGALEIL, S.A.M. 2018: Insecticidal activities of monoterpenes and phenylpropenes against *Sitophilus oryzae* and their inhibitory effects on acetylcholinesterase and adenosine triphosphatases. *Applied Entomology and Zoology*, <https://doi.org/10.1007/s13355-017-0532-x>
- SPSS. 2006. Statistical Package for Social Sciences. Version 15.0 for Windows, SPSS Inc. 2335, Walker Drive, Chicago, Illinois 60606.
- TIWARI, V., NEGI, K.S., RAWAT, R. AND MEHTA, P.S. 2017: *In-situ* conservation and Traditional Uses of Medicinal Plants: A Case Study of Home Gardens in Nainital, Uttarakhand. *Asian Agri-History* **21**, 47-61.
- USMAN L. A., OLANIPEKUN B. E., OGUONDELE V. A. AND MUSA A. K. 2016: Phytochemical profile and insecticidal activity of essential oil from fresh and dried leaves of the Nigerian grown *Citrus meyeri*. *Journal of Turkish Chemical Society (Section A)* **3**, 207-218.
- VENKATANARAYANAN K., KOLLANOOR-JOHN Y, DARRE M. J., DONOGHUE A. M., AND DONOGHUE D. J. 2013: Use of plant-derived antimicrobials for improving the safety of poultry products. *Poultry Science* **92**, 493-501.
- YILDIRIM, E., EMOSEN, B., AND KORDALI, S. 2013: Insecticidal effects of monoterpenes on *Sitophilus zeamais* Motschulsky (Coleoptera: Curculionidae). *Journal of Applied Botany and Food Quality* **86**, 198-204
- ZHANG, W.J., YANG, K., YOU, C.X., WANG, C.F, GENG, Z.F, SU, Y, WANG, Y, DU, S.S., AND DENG, Z.W. 2015: Contact toxicity and repellency of the essential oil from *Mentha haplocalyx* Briq. against *Lasioderma serricorne*. *Chemistry and Biodiversity* **12**, 832-839

Binary mixture efficacy of NeemAzal and *Plectranthus glandulosus* leaf powder against cowpea and maize weevils

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Abstract

The aim of this study was to determine the insecticidal efficacy of mixture of NeemAzal a commercial neem product and *Plectranthus glandulosus* leaf powder against *Callosobruchus maculatus* and *Sitophilus zeamais*. Mixed at various proportions (100 + 0, 75 + 25, 50 + 50, 25 + 75 and 0 + 100%, these powders were tested on adult mortality, inhibition of offspring production and their persistence on *C. maculatus* and *S. zeamais*. All the mixed NeemAzal and *P. glandulosus* caused significant mortality to adult *C. maculatus* and *S. zeamais*. No