African Crop Science Journal, Vol. 31, No. 3, pp. 301 - 318
ISSN 1021-9730/2023
eISSN 2072-6589/2023

DOI: https://dx.doi.org/10.4314/acsj.v31i3.3

INSECTICIDAL ACTIVITIES OF VOLATILE OILS OF LIME FRUIT PEELS AND AFRICAN BLACK PEPPER SEEDS ON ADULT KOLA WEEVIL AND THEIR CHEMICAL COMPOSITIONS

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(*Received 15 March 2023; accepted 16 June 2023*)

ABSTRACT

Balanogastris kolae reduces the quality of stored kolanuts; yet, plants with insecticidal properties could be used to manage this insect pest. The objective of this study was to evaluate the efficacy of plant oils against *B. kolae,* a storage pest of kolanuts. The *Citrus aurantifolia* fruit peel and *Piper guineense* seed volatile oils obtained *via* hydro-distillation of the dried, pulverised samples were tested. Their chemical composition was determined using Gas Chromatography-Mass Spectrometry (GC-MS) analysis. Adult mortality was generally low (0.0%) across treatments and exposure times, though not significant ($P > 0.05$) in the fumigant tests The highest mortality (47.5%) was recorded at 24 hours in 1.0% *P. guineense*, while the mortality was 13.1% occurred at 12 hours in the 15.0% *C. aurantifolia* concentration. In contact toxicity tests, none of the weevils treated with 10.0% *C. aurantifolia* survived after 24 hours of exposure. The highest mortality (85.0%) was obtained from the 10.0 and 15.0% *P. guineense* volatile oil after 96 hours. *Citrus aurantifolia* elicited contact toxicity against the weevil more than *P. guineense* across all concentrations. The GC-MS analysis gave 33 compounds in *C. aurantifolia* and 43 in *P. guineense* volatile oil samples. The prominent compounds were limonene (3.222%), linalyl isobutyrate (2.484%), gamma-caryophyllene (1.197%) and Patchoulene (6.529%). Therefore, *C. aurantifolia* fruit peel and *P. guineense* seed volatile oils are efficacios in controlling *Balanogastris kolae* insect pest infestation on kolanuts, though *C. aurantifolia* fruit peel oil is the best as it compares closely with the synthetic pesticide (2,2-dimethyl dichlorovinyl phosphate (DDVP)).

Key Words: *Balanogastris kolae*, *Citrus aurantifolia, Piper guineense*

RÉSUMÉ

Balanogastris kolae réduit la qualité des noix de kola stockées ; pourtant, des plantes aux propriétés insecticides pourraient être utilisées pour lutter contre cet insecte ravageur. L'objectif de cette étude était d'évaluer l'efficacité des huiles végétales contre *B. kolae*, un ravageur des entrepôts de noix de cola. Les huiles volatiles d'écorce de fruit de *Citrus aurantifolia* et de graines de *Piper guineense* obtenues par hydro-distillation des échantillons séchés et pulvérisés ont été testées. Leur composition chimique a été déterminée par analyse par chromatographie en phase gazeuse-spectrométrie de masse (GC-MS). La mortalité des adultes était généralement faible (0,0 %) selon les traitements et les durées d'exposition, mais non significative (P > 0,05) dans les tests de fumigation. La mortalité la plus élevée (47,5 %) a été enregistrée à 24 heures chez 1,0 % de *P. guineense*, tandis que la mortalité était 13,1 % se sont produits à 12 heures à la concentration de 15,0 % de *C. aurantifolia*. Dans les tests de toxicité par contact, aucun des charançons traités avec 10,0 % de *C. aurantifolia* n'a survécu après 24 heures d'exposition. La mortalité la plus élevée (85,0 %) a été obtenue avec 10,0 et 15,0 % d'huile volatile de *P. guineense* après 96 heures. *Citrus aurantifolia* a provoqué une toxicité de contact contre le charançon plus que *P. guineense* à toutes les concentrations. L'analyse GC-MS a donné 33 composés dans *C. aurantifolia* et 43 dans des échantillons d'huile volatile de *P. guineense*. Les principaux composés étaient le limonène (3,222 %), l'isobutyrate de linalyle (2,484 %), le gamma-caryophyllène (1,197 %) et le patchoulène (6,529 %). Par conséquent, l'écorce de fruit de *C. aurantifolia* et les huiles volatiles de graines de *P. guineense* sont efficaces pour contrôler l'infestation d'insectes nuisibles par *Balanogastris kolae* sur les noix de kola, bien que l'huile d'écorce de fruit de *C. aurantifolia* soit la meilleure car elle se compare étroitement au pesticide synthétique (2,2-diméthyl phosphate de dichlorovinyle (DDVP)).

Mots Clés: *Balanogastris kolae*, *Citrus aurantifolia*, *Piper guineense*

INTRODUCTION

Kolanuts (Cola *nitida* (Vent) Schott & Endl.) are considerably vulnerable to infestation caused by a weevil called *Balanogastris kolae* Desbr., particularly in storage. The loss due to this infestation can be as high as 100% of the stored nuts if not duly managed (Azeez, 2015). Synthetic pesticides are used to control this pest (Popoola *et al*., 2020). Though more often than not, these synthetic insecticides have proved to be effective, their repeated use may inflict damage to both the environment and human wellbeing. They are often highly toxic to human health as they cause a host of noninfectious diseases (Chengala *et al*., 2017). The adverse effects of synthetic pesticides and their residues have necessitated the quest for effective alternatives from plants. Many such alternatives that are non-persistent in the environment, selective towards beneficial insects and less toxic to humans have been reported to be used traditionally (Grdisa *et al*., 2013; Oben *et al*., 2015). Botanical plants used either in powdered form or extracts such as crude extract or volatile oil have been successful against a number of pests in Africa (Pinto *et al*., 2015; Chand *et al*., 2017; Gharsan *et al*., 2018; Rahayu *et al*., 2020).

Many studies have been done on the control of *B. kolae* attack on kolanuts using different plant sample formulations and extracts (Akunne *et al*., 2018; Ugwu *et al*., 2019; Ifebueme *et al.,* 2020). However, information on the use of genus *Citrus* and *Piper* for this purpose is scanty, yet, there are indicators that the plant materials have potency in controlling insect pests, hence, the need to exploit these opportunities.

The genus *Citrus* belonging to the family *Rutaceae* is one of the most commonly consumed and widely distributed fruits worldwide (Lin *et al*., 2019; Sarma *et al*., 2019). Citrus essential oils are characterised by mixtures of many components such as terpenes, sesquiterpenes, aldehydes, alcohols, and esters (Lin *et al*., 2019).

Previous reports showed that volatile oils from the fruit peels and aerial parts of lime (*Citrus aurantifolia)* present strong insecticidal activities on *Aedes aegypti* (the yellow fever mosquito), cowpea weevil, *Callosobruchis maculatus* (F.) and carpenter ants (*Camponotus nearcticus*) (Adusei- Mensah *et al*.,2014; Olonisakin, 2014; Sarma *et al*., 2019).

Piper guineense (Schum.& Thonn.) commonly called the West African Black pepper, is a climbing perennial plant belonging to the genus *Piper* and the family Piperaceae (Ojinnaka *et al*., 2016). This genus (*Piper*) comprises about 1000 species of tropical lianas, small trees and shrubs, many of which are used as spices, flavouring agents and medicines (Ojiako *et al*., 2018). This plant is found in tropical regions of Central and West African countries like Nigeria. The seeds of *P. guineense* are rich in a good number of natural products such as lignans, amides, alkaloids, flavonoids and volatiles oils (Rodolfo *et al*., 2013) and can be used in treating cough, bronchitis, intestinal disease and rheumatism (Ojinnaka *et al.,* 2016). They are also used in traditional African medicine, as a result of their various pharmacological effects such as antibacterial, insecticidal, anticonvulsant, antioxidant, antihypertensive, aphrodisiac, sedative and anti-inflammatory (Ojinnaka *et al*., 2016; Chukwunonso *et al.,* 2020).

Presently, information on the use of the volatile oils from *C. aurantifolia* and *P. guineense* seems unavailable to enable effective exploitation of their bio-control levels. Therefore, the objective of this study was to evaluate the efficacy of the plant oils against *B. kolae,* a major storage insect pest of kolanuts in sub-Saharan Africa.

MATERIALS AND METHODS

Preparation of plant materials. The plant materials, *Citrus aurantifolia* (CA) fruits and *Piper guineense* (PG) seeds were purchased from Bode market in Ibadan, Oyo State in Nigeria. The *C. aurantifolia* fruits were first rinsed in clean tap water and the back thinly peeled off with a sterilised razor. The *P. guineense* fruits, on the other hand were obtained from Bodija market, in Ibadan, Oyo State in Nigeria. They were sorted to remove dirt, dried under shade for two weeks and then crushed with the aid of a silver crest (SC – 1589) blender.

Extraction of the essential oils. Fresh peels of *C. aurantifolia* fruit (200 g) were thinly sliced and *P. guineense* seeds (300g) were crushed and then subjected to hydro-distillation for 3 hours in a Clevenger-type apparatus. The essential oil (EO) of each of the plant samples obtained was stored at -4 ^{o}C for analyses.

Kola nut samples for insect culture. Three plastic containers (4 L capacity each) of both infested and clean kola nuts were purchased from a local market in Osogbo, Osun State in Nigeria. The infested nuts were kept inside plastic bags in the laboratory for two weeks to enable adult weevil emergence. The clean nuts were preserved in a paper wrapper and placed inside a black polythene bag to keep them fresh. The kola weevil (*Balanogastris kolae*) culture was raised in the laboratory from adult weevils obtained from the infested kola nut following the method described by Asogwa *et al*. (2009).

Twenty male and female (10:10) weevils were introduced into a black polythene bag containing fifty nuts and left on a laboratory bench for 30 days to facilitate oviposition and development of F_1 adult weevil emergence. Teneral adult weevils obtained from this culture were used for the bioassays.

Bioassays of volatile oils against kola weevil

Contact toxicity test. Ten teneral (newly emerged) adults *Balanogastris kolae* male and female (5: 5) of the same age were placed inside 11.5 cm by 4.5cm transparent dishes. A volume of 0.2 ml of the different concentrations namely, 1, 5, 10, 15 and 20% of each volatile oil of *C. aurantifolia* fruits peel and *P. guineense* seeds were introduced into the insects-containing dishes to permit contact with the oil. Two kola nuts were placed inside each of the containers for the treated insects as food. Lid of the containers were perforated for aeration purposes. Each of the treatments was replicated four times in a completely randomised design. There were two controls, one in which only hexane was used against the insect and the other without treatments. Mortality counts were recorded after 24, 48, 72 and 96 hours of exposure (Azeez, 2015) and corrected based on the control count using Abbott's formula (Abbott, 1925). 2,2-dimethyl dichlorovinyl phosphate (DDVP) was used as standard in which 0.5 ml of the stock pesticide was diluted in 100 ml of water and applied following the same procedure used for the oil samples.

Fumigant toxicity test. Ten adults of *B. kolae* male and female (5: 5) of the same age were placed inside a 16 cm by 10 cm cylindrical container. Then, 0.2 ml of the different concentrations (1, 5, 10, 15 and 20%) of each of the volatile oil of *C. aurantifolia* fruits peel and *P. guineense* seeds was introduced on a filter paper strip of 2.5 cm diameter. The volatile oil-impregnated filter paper strip was dropped inside the cylindrical container. Two kola nuts were placed inside the container to serve as food for the treated insects. Each fumigation test was replicated four times along with the control which received no essential oil. Adult mortality was recorded after 1, 6, 12 and 24 hr of the exposure and corrected for mortality using Abbott's formula (Abbott, 1925). Then 2 ml of 0.5% concentration of 2,2-dimethyl dichlorovinyl phosphate (DDVP) was used as standard following the same procedure.

Statistical analysis. Mortality data were corrected for the mortality that occurred in the control treatment using Abbott's formula. Data collected were analysed using analysis of variance (ANOVA) of R statistical software, version 4.1.3.

Gas Chromatography-Mass Spectrometry analysis. Gas Chromatography - Mass Spectrometry analysis of the volatile oils of *C. aurantifolia* fruits peel and *P. guineense* seeds were conducted at the Biocontrol Centre, International Institute of Tropical Agriculture, Republic of Benin. Capillary gas chromatography was performed using Hewlett–Packard 8890 gas chromatograph equipped with a flame ionisation detector and fused silica capillary column HP-5 MS (30 m × 0.25 mm, 0.25 μm film thickness); injector and detector temperatures were 270 °C and 300°C, respectively. The components of the volatile oils of *C. aurantifolia* fruits peel and *P. guineense* seeds were separated by the GC and identified by mass spectrometry (GC–MS) using Agilent 8890 gas chromatography coupled to Agilent 5977B mass spectrometry detector. The GC settings were as follows: the initial oven temperature was held at 60°C for 1 min and ramped at 10°C min^{-1} to 180°C where it was held for 1 min, and then ramped at 20°C min^{-1} to 280°C and held there for 15 min. The total run time was 42 minutes. The sample (1 μL, diluted 1:100 in acetone) was injected, with a split ratio of 1:10. The carrier gas was helium at a flow rate of 1.0 $mLmin^{-1}$. Spectra were obtained over the scan range of 20 to 550 m/z at 2 scans s^{-1}. The constituents of the oils were identified *via* gas chromatography by comparing their retention times using the library (Adams 2. L and NIST 11. L). Further identification was made by comparison of their mass spectra with those stored in the libraries.

RESULTS

Bioassays of the insecticidal activity

Fumigant toxicity test. Tables 1a - 1e shows the percentage mortality of the volatile oils as fumigants against adult *B. kolae*. After 1 hour

TABLE 1a. Fumigant toxicity of 1% volatile oils of *C. aurantifolia* (CA) and *P. guineense* (PG) against kolanuts weevil at different exposure times

Fumigant	1% Concentration			
	1 hour	6 hours	12 hours	24 hours
PG	0.0±0a	10.0±4.08a	25.0±10.4a	47.5±15.5a
CA	2.78±2.78a	7.78±2.61a	10.28±4.09a	10.0±0a
DDVP	100.0±0b	100.0±0b	100.0±0b	100.0±0b
Untreated	0±0	0±0	0±0	0±0

Mean±SE. Means within a column followed by the same letters are not significantly different using Tukey Test at $P < 0.05$. PG = 47.5±15.5a, CA = 10.0±0a, DDVP = 100.0±0b

TABLE 1b. Fumigant toxicity of 5% volatile oils of *C. aurantifolia* (CA) and *P. guineense* (PG) against kolanuts weevil at different exposure times

Fumigant	5% Concentration			
	1 hour	6 hours	12 hours	24 hours
PG	0.0±0a	0.0±0a	0.0±0a	2.50±2.5a
CA	0.0±0a	2.50±2.5a	2.05±2.5a	5.0±2.89a
DDVP	100.0±0b	100.0±0b	100.0±0b	100.0±0b

Mean±SE. Means within a column followed by the same letters are not significantly different using Tukey Test at $P < 0.05$. PG = 2.50 ±2.5a, CA = 5.0±289a, DDVP = 100.0±0b

TABLE 1c. Fumigant toxicity of 10% volatile oils of *C. aurantifolia* (CA) and *P. guineense* (PG) against kolanuts weevil at different exposure times

Fumigant	10% Concentration			
	1 hour	6 hours	12 hours	24 hours
PG	0.0±0a	2.50±2.5a	7.50±4.79a	10.0±5.77a
CA	0.0±0a	2.50±2.5a	10.0±7.07a	15.63±11.8a
DDVP	100.0±0a	100.0±0b	100.0±0b	100.0±0b
Untreated	0±0	0±0	0±0	0±0

Mean±SE. Means within a column followed by the same letters are not significantly different using Tukey Test at $P < 0.05$. PG = 10.0±5.77a, CA = 15.63±11.8a, DDVP = 100.0±0b

TABLE 1d. Fumigant toxicity of 15% volatile oils of *C. aurantifolia* (CA) and *P. guineense (*PG*)* against kolanuts weevil at different exposure times

Fumigant	15% Concentration			
	1 hour	6 hours	12 hours	24 hours
PG	2.50±2.5a	15.0±6.45a	17.50±6.29a	22.50±10.3a
CA	2.50±2.5a	2.5±2.5a	13.06±3.06a	10.63±4.13a
DDVP	100.0±0b	100.0±0b	100.0±0b	100.0±0b
Untreated	0±0	0±0	0±0	0±0

Mean±SE. Means within a column followed by the same letters are not significantly different using Tukey Test at P < 0.05. PG = 22.50±10.3a, CA = 10.63±4.13a, DDVP = 100.0±0b

TABLE 1e. Fumigant toxicity of 20% volatile oils of *C. aurantifolia* (CA) and *P. guineense (*PG) against kolanuts weevil at different exposure times

Fumigant	20% Concentration			
	1 hour	6 hours	12 hours	24 hours
PG	0.0±0a	5.0±2.89a	10.0±5.77a	15.0±6.46a
CA	2.50±2.5a	2.50±2.5a	7.78±2.61a	2.50±2.5a
DDVP	100.0±0b	100.0±0b	100.0±0b	100.0±0b
Untreated	0±0	0±0	0±0	0±0

Mean±SE. Means within a column followed by the same letters are not significantly different using Tukey Test at P< 0.05 PG = 15.0±6.46a, CA = 2.50±2.5a, DDVP = 100.0±0b

of exposure of *B. kolae* to the fumigants, the percentage mortality was generally low across the treatments ranging from 0% mortality (PG at 1%; CA and PG at 5%, CA and PG at 10% and PG at 20% to CA at 2.78% at 1%). The observed percentage mortality at those concentrations was significantly lower (P<0.05) than the synthetic insecticide (DDVP) where 100% mortality was recorded. After 6 and 12 hour, mortality remained at 0% with PG at 5% concentration and increased at 1, 10 15 and 20% concentrations as the exposure time increased, but the increase was significantly lower when compared with the synthetic insecticide. With the fumigant CA, percentage mortality increased from 6 hours to 24 hours of exposure for all the concentrations except 5%, which remained at 2.50% mortality. However, there were no significant differences (P<0.05) between the percentage mortality recorded for all the concentrations of CA and PG through all the exposure time.

Table 2 shows the percentage mortality of *B.kolae* after 24 hours of exposure to fumigant toxicity from *Citrus aurantifolia* (CA) *and Piper guineense (*PG*)* volatile oils and DDVP.

Contact toxicity test. Tables 3a to 3e presents the effect of *Citrus aurantifolia* (CA) *and Piper guineense (*PG*)* as contact insecticide on *Balanogastris kolae* at different concentrations and time of exposure. Although, the volatility effect were not significantly different (P>0.05)

TABLE 2. Percentage *B.kolae* mortality after 24 hours of exposure to fumigant toxicity from *Citrus aurantifolia* (CA) *and Piper guineense (*PG*)* volatile oils and DDVP

Concentrations	Weevil's mortality (%)				
	1	5	10	15	20
PG	47.5±15.5a	2.50 ±2.5a	10.0±5.77a	22.50±10.3a	15.0±6.46a
CA	10.0±0a	5.0±289a	15.63±11.8a	10.63±4.13a	2.50±2.5a
DDVP	100.0±0b	100.0±0b	100.0±0b	100.0±0b	100.0±0b

Mean±SE. Means within a column followed by the same letters are not significantly different using Tukey Test at P< 0.05

TABLE 3a. Contact toxicity of 1% volatile oils of *Citrus aurantifolia (*CA*)* and *Piper guineense* (PG) against kolanuts weevil at different exposure times

Contact	1% mortality			
	24 hour	48 hours	72 hours	96 hours
PG	0.0±0a	10.0±4.08a	25.0±10.4a	47.5±15.5a
CA	2.78±2.78a	7.78±2.61a	10.28±4.09a	10.0±0a
DDVP	100.0±0b	100.0±0b	100.0±0b	100.0±0b
Untreated	0±0	0±0	0±0	0±0

Mean±SE. Means within a column followed by the same letters are not significantly different using Tukey Test at P < 0.05. PG = 47.5±15.5a, CA = 10.0±0a, DDVP = 100.0±0b

TABLE 3b. Contact toxicity of 5% volatile oils of *Citrus aurantifolia (*CA*)* and *Piper guineense* (PG) against kolanuts weevil at different exposure time

Contact	5% concentration			
	24 hours	48 hours	72 hours	96 hours
PG	38.06±7.08a	56.39±10.3a	69.44±13.4a	77.50±16.5a
CA	69.44±17.2ab	77.78±18.7a	77.78±18.7a	83.33±16.8a
DDVP	100.0±0b	100.0±0a	100.0±0a	100.0±0a

Mean±SE. Means within a column followed by the same letters are not significantly different using Tukey Test at P < 0.05. PG = 77.50±16.5a, CA = 83.33±16.8a, DDVP = 100.0±0b

TABLE 3c. Contact toxicity of 10% volatile oils of *Citrus aurantifolia (*CA*)* and *Piper guineense* (PG) against kolanuts weevil at different exposure times

Contact	10% concentration			
	24 hours	48 hours	72 hours	96 hours
PG	28.06±4.52a	48.61±7.21a	72.50±11.1a	85.0±8.66a
CA	100.0±0b	100.0±0b	100.0±0b	100.0±0a
DDVP	100.0±0b	100.0±0b	100.0±0b	100.0±0a

Mean±SE. Means within a column followed by the same letters are not significantly different using Tukey Test at P < 0.05. PG = 85.0±8.66a, CA = 100.0±0a, DDVP = 100.0±0a

TABLE 3d. Contact toxicity of 15% volatile oils of *Citrus aurantifolia (*CA*)* and *Piper guineense* (PG) against kolanuts weevil at different exposure times

Contact	15% concentration			
	24 hours	48 hours	72 hours	96 hours
PG	51.67±9.86a	64.44±11.7a	79.72±13.5a	85.0±11.9a
CA	94.44±5.56a	97.22±2.78b	100.0±0a	100.0±0a
DDVP	100.0±0b	100.0±0b	100.0±0a	100.0±0a

Mean±SE. Means within a column followed by the same letters are not significantly different using Tukey Test at P < 0.05. PG = 85.0±11.9a, CA = 100.0±0a, DDVP = 100.0±0a

TABLE 3e. Contact toxicity of 20% volatile oils of *Citrus aurantifolia (*CA*)* and *Piper guineense* (PG) against kolanuts weevil at different exposure times

Contact	20% concentration			
	24 hours	48 hours	72 hours	96 hours
PG	35.28±10.2a	47.78±14.7a	63.06±16.0a	68.33±14.2a
CA	88.89±6.42b	94.10±3.42b	96.88±3.12a	96.88±3.12a
DDVP	100.0±0b	100.0±0b	100.0±0a	100.0±0a

Mean±SE. Means within a column followed by the same letters are not significantly different using Tukey Test at P < 0.05. PG = 68.33±14.2a, CA = 96.88±3.12a, DDVP = 100.0±0a across concentrations and exposure time , PG volatile oil tended to cause the mortality on the weevil with a rise in concentration, most especially from 5% through to 15% concentration across all the times of exposure. On the other hand, mortality of *B. kolae* tended to be higher with CA for all the concentrations and exposure time than for PG.

Across all the concentrations of the volatile oil (CA), percentage mortality tended to increase with increasing exposure time, although, 100 percent mortality was obtained with CA at 10% concentration (24 to 96 hours) and 15% concentration (72 and 96 hours). Across all the concentrations of the CA volatile oil, percentage mortality tended to increase with increasing exposure time; although, 100 percent mortality was obtained with CA at 10% concentration (24 to 96 hours) and 15% concentration (72 and 96 hours).

Table 4 shows the percentage mortality of *B.kolae* after 96 hours of exposure to contact toxicity from *Citrus aurantifolia* (CA) *and Piper guineense (*PG*)* volatile oils and DDVP.

Chemical composition of the volatile oils. The volatile oil obtained from CA fruit peel was yellowish to greenish in colour with a strong pungent smell while that of (PG) was pale yellow (Tables 5 and 6). The oils extracted from each of the plant samples gave 1.15 and 0.9% yields respectively.

The result of the GC- MS analysis showed the presence of 33 compounds in the volatile oil of CA fruit peels (Table 5) and the chromatogram (Fig. 1). Based on the area percentage of the GC- chromatogram, hydrocarbons such as tricyclo[5.2.1.0(4,8)] decane (14.458), 8-methylene-Bicyclo[5.1.0] octane (11.958), 7,8-Dioxabicyclo[3.2.1]oct-2-ene (4.665) and terpenes such as limonene (3.222), linalyl isobutyrate (2.484), caryophyllene (1.197) were identified as the major compounds present in the *Citrus aurantifolia* fruit peels. Picolinamidoxime (6.983), which is an oxime and 4-propyl-1,3-Benzenediol (4.689), an alcohol were equally found as major constituents though the terpenes are more abundant in number in the oil.

For the PG volatile oil, the GC-MS analysis identified 43 compounds comprising of about 70% terpenes/terpenoids (Table 6) and the chromatogram (Fig. 2). The prominent ones among these were sesquiterpene ν-Patchoulene (6.529), β-Selinene (5.3892), 7-epi-silphiperfol-5-ene (5.3647), cis-cadina-1(6),4-diene (5.0566), Iso-daucene (4.263), among others. Also, present were a few hydrocarbons which included

TABLE 4. Percentage *B.kolae* mortality after 96 hours of exposure to contact toxicity from *Citrus aurantifolia* (CA) *and Piper guineense (*PG*)* volatile oils and DDVP

Concentrations	Weevil's mortality (%)				
	1	5	10	15	20
PG	47.5±15.5a	77.50±16.5a	85.0±8.66a	85.0±11.9a	68.33±14.2a
CA	10.0±0a	83.33±16.8a	100.0±0a	100.0±0a	96.88±3.12a
DDVP	100.0±0b	100.0±0a	100.0±0a	100.0±0a	100.0±0a

Mean±SE. Means within a column followed by the same letters are not significantly different using Tukey Test at P < 0.05

TABLE 5. Chemical constituents of the volatile oils of *Citrus aurantifolia* fruit peels

S/N	RT	Area Pct	Library/ID	Types of compound
1	4.5513	0.8607	o-Xylene	Aromatic hydrocarbon
2	5.2052	2.2314	1-methylene-3-(1-methylethenyl)-, (R)-Cyclohexane	Hydrocarbon
3	5.4615	0.3294	Camphene	Monoterpene
4	5.8562	1.1603	(+)-3-Carene	Monoterpene
5	5.9211	14.858	Tricyclo[5.2.1.0(4,8)] decane	Hydrocarbon
6	6.6913	0.8185	Benzene, 1-methyl-3-(1-methylethyl)-	Aromatic hydrocarbon
7	6.7507	2.802	Bicyclo[6.1.0]non-1-ene	Hydrocarbon
8	6.8192	11.9583	8-methylene-Bicyclo[5.1.0]octane	Hydrocarbon
9	7.0391	0.8074	β-Ocimene	Monoterpene
10	7.2476	6.9831	Picolinamidoxime	Oxime
11	7.4809	0.1343	3-Amino-1,2,4-triazole-5-carboxylic acid	Acid
12	7.8721	2.4837	Linalyl isobutyrate	Monoterpenoid
13	8.1593	0.1882	Fenchol, exo-	Monoterpenoid
14	8.265	0.3107	ν-Terpinene	Monoterpene
15	8.7107	0.6981	3,7-dimethyl-, (R)-6-Octenal	Aldehyde
16	8.8799	0.1113	Isoneral	Monoterpene
17	8.9796	0.3489	Isoborneol	Monoterpene
18	9.1448	4.6654	7,8-Dioxabicyclo[3.2.1]oct-2-ene	Hydrocarbon
19	9.3346	4.1992	9-methyl-9-Borabicyclo[3.3.1] nonane	Hydrocarbon
20	9.4888	0.5116	Decanal	Aldehyde
21	9.5771	0.1268	3-methyl-6-(1-methylethyl)-, cis-2-Cyclohexen-1-ol	Alcohol
22	9.8357	3.2218	Limonene	Monoterpene
23	10.4612	4.6488	4-propyl-1,3-Benzenediol	Alcohol
24	11.9644	0.5431	Geranyl propionate	Hydrocarbon
25	12.3126	0.073	Tetradecanal	Aldehyde
26	12.489	0.0479	trans-.α-Bergamotene	Monoterpene
27	12.6281	1.1968	Caryophyllene	Sesquiterpene
28	12.747	1.1208	(Z,Z)-.α-Farnesene	Sesquiterpene
29	12.9969	0.0323	Propanamide, 3-(3,4-dimethylphenylsulfonyl)-	Amide
30	13.0687	0.1936	Humulene	Sesquiterpene
31	13.4045	0.2016	Germacrene D	Monoterpene
32	13.4818	0.1079	Di-epi-.alpha.-cedrene	Sesquiterpene
33	16.1582	0.1575	Levomenol	Sesquiterpenoid

TABLE 6. Chemical composition of the volatile oils of *Piper guineense* seeds

S/N	RT	Area Pct	Library/ID	Type of compound
1	4.5559	0.5993	p-Xylene	Aromatic hydrocarbon
2	5.2139	1.5026	p-Mentha-1(7),8-diene (limonene)	Monoterpene
3	5.4607	0.1707	Camphene	Monoterpene
4	5.9306	2.8949	α-Fenchene	Monoterpene
5	6.1069	0.8097	β-Pinene	Monoterpene
6	6.3654	0.9146	a- Phellandrene	Monoterpene
7	6.4594	2.3504	(cis-)m- Mentha-2,8-diene	Monoterpene
8	6.6944	0.2132	Ortho-Cymene	Monoterpene
9	6.7649	2.0384	o-Carene	Monoterpene
10	6.8589	0.9844	Santolina triene	Hydrocarbon
11	7.0469	0.2267	β-Ocimene(E)	Monoterpene
12	7.2466	0.2143	ν-Terpinene	Monoterpene
13	7.8929	6.6507	Tricyclo[5.3.0.0(3,9)]decane	Hydrocarbon
14	8.1749	0.0588	2,2,6,6-tetramethyl-4-Piperidinone	Lactam
15	8.2689	0.0337	Dihydrotecomanine	Hydrocarbon
16	8.6801	0.0705	Camphor	Terpennoid
17	8.8564	0.0795	Isoborneol	Terpennoid
18	9.1501	0.1906	Terpinen-4-ol	Terpennoid
19	10.5366	0.0066	Cathinone	Monoamine alkaloid
20	11.6293	0.7386	o-Amorphene	Terpennoid
21	12.1228	0.2327	β-Elemene	Sesquiterpene
22	12.2051	4.263	Iso-daucene	Sesquiterpene
23	12.3343	0.1859	α-Zingiberene	Sesquiterpene
24	12.6281	5.3647	7-epi-Silphiperfol-5-ene	Sesquiterpene
25	12.7456	2.6306	Germacrene B	Sesquiterpene
26	12.8161	0.9839	β-Gurjunene	Sesquiterpene
27	12.9571	6.529	ν-Patchoulene	Sesquiterpene
28	13.0746	3.4446	g-Elemene	Sesquiterpene
29	13.3213	2.4293	ν-Himachalene	Sesquiterpenoid
30	13.6503	5.3892	β-Selinene	Sesquiterpene
31	13.8618	5.0566	cis-Cadina-1(6),4-diene	Sesquiterpene
32	13.9558	1.5799	Naphthalene	Aromatic hydrocarbon
33	14.2848	0.6787	<E-> Nerolidol	Sesquiterpenoid
34	14.3788	0.9865	Premnaspirodiene	Sesquiterpene
35	14.5903	0.3983	ν-Muurolene	Sesquiterpene
36	14.7313	0.5955	Caryophyllene oxide	Sesquiterpenoid
37	15.0133	0.1169	β-Longipinene	Sesquiterpene
38	15.2248	0.1205	5-Aminoisoxazole	Amino
39	15.401	0.0389	cis-Aconitic anhydride	Tricarboxylic acid
40	22.3922	0.9736	Di-n-octyl phthalate	Ester
41	24.8714	0.0293	Propanamide	Amide
42	26.1404	0.0364	3-Hydroxy-N-Cyclobutanol	Hydrocarbon
43	34.1068	0.0046	Methylphenethylamine	Amine

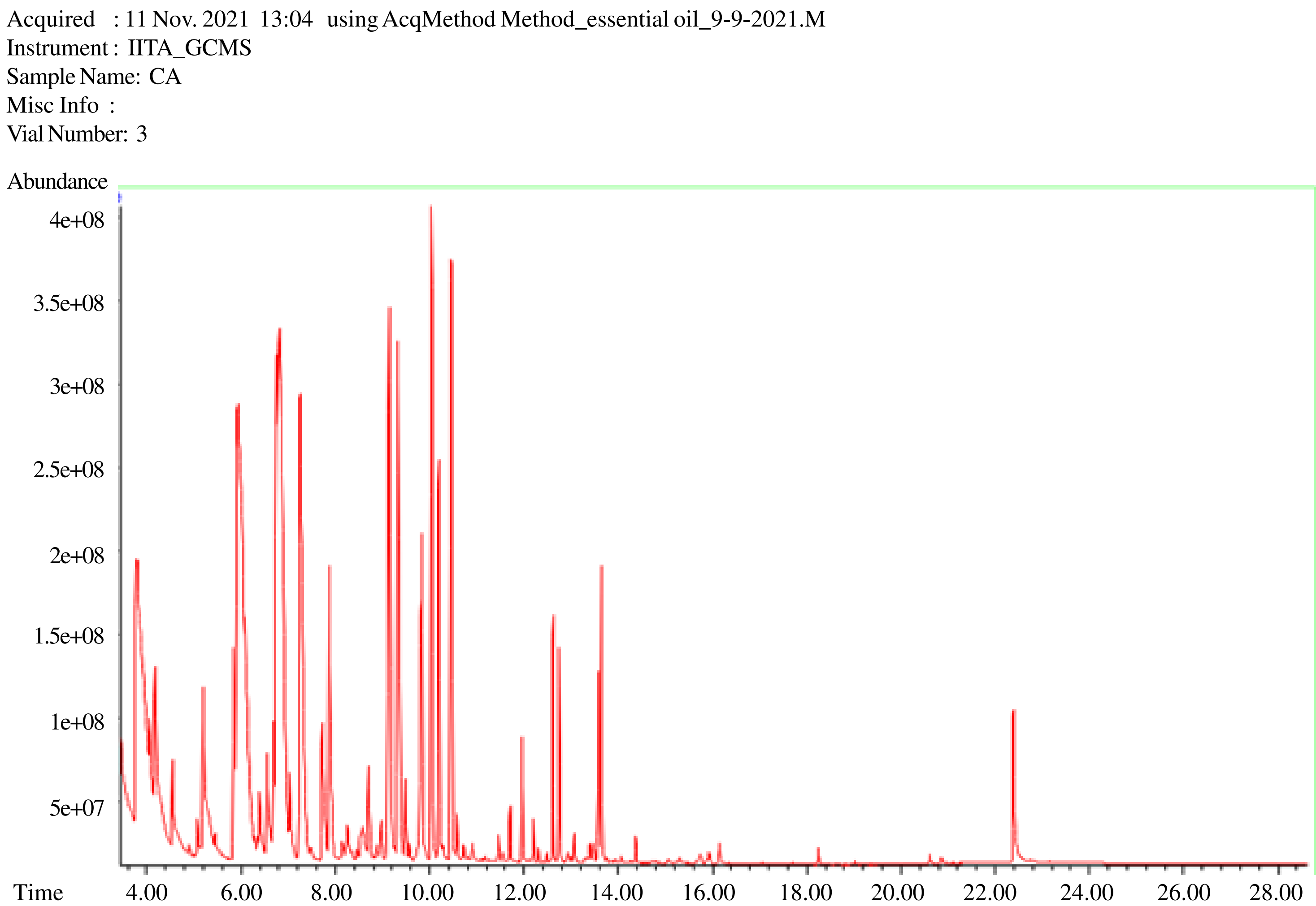

Figure 1. Gas chromatogram of *Citrus aurantifolia* fruit peel volatilitic oil.

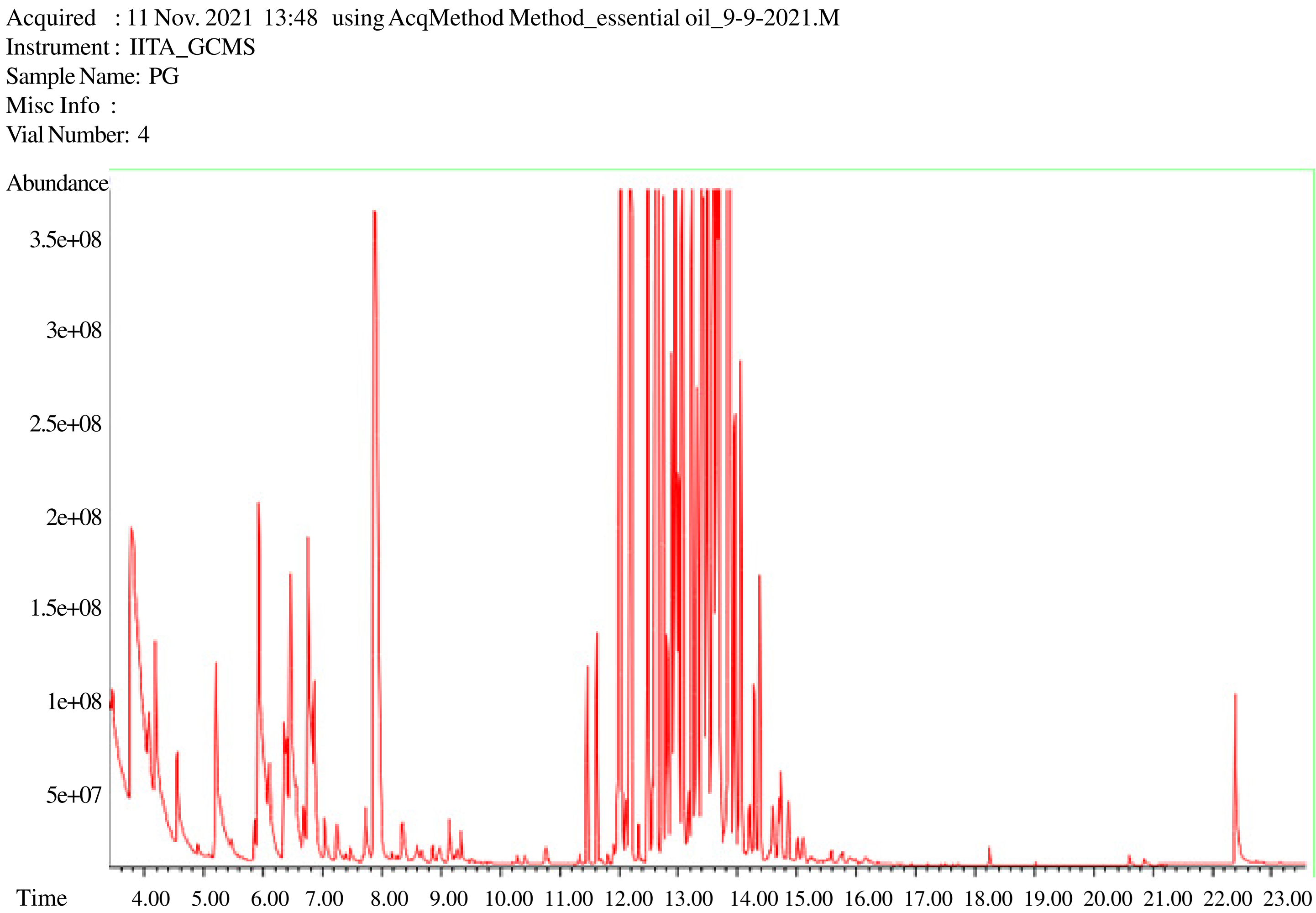

Figure 2. Gas analysis of chromatogram of *Piper guineense* seed oil.

tricyclo[5.3.0.0(3,9)]decane (6.6507), naphthalene - an aromatic hydrocarbon (1.5799), santolina triene (0.9844) and di-n-octyl phthalate (0.9736) which is an ester.

DISCUSSION

Bioassays on the insecticidal activity of the volatile oils

Fumigant toxicity. The lack of significant effect of fumigant toxicity displayed by the volatile oils of *Citrus aurantifolia* (CA) and *Piper guineense* (PG) (Tables 1a - e) probably implies that the fumes emitted by these oils were below threshold level and could not cause the insects to absorb the components of the oil *via* their spiracles. It may also be that antagonistic effects may be existing between some of their constituents as observed by Sun *et al.* (2020}. This corroborates the findings of Rayanna *et al.* (2019) in which *Callosobruchus maculatus* adults were exposed to essential oils of *Citrus limon*, *Citrus aurantifolia* and *Piper nigrum* and found that *Citrus aurantifolia* oil exhibited the lowest fumigant activity. Chaubey (2019) further supports this observation as neither *C. aurantifolia* nor *P. guineense* were listed among the plant species that have fumigant toxicity against some insects. Nevertheless, a few species in each family of these plant samples such as *C. aurantium* and *P.nigrum* were reported elsewhere to be toxic against some insect pests (Changbunjong *et al*., 2022, Sleem, 2021). Sahla *et al*., 2020 equally discovered that essential oil from *Citrus limon* is highly effective as fumigant and repellant in controlling adult beetles of *Callosobruchus maculatus*.

Contact toxicity. The presence of contact toxicity effectiveness of *C. aurantifolia* (CA) and *P. guineense* (PG) on percentage mortality of *B. kolae* through all the exposure time (Tables 3a to e) could be attributed to high toxic effect the volatile oils imposed on the insect pests. This toxicity may make feeding on the treated kolanuts difficult, which then leads to starvation and eventual death of the insect pests (Alabi *et al*., 2017). This is supported by previous studies conducted on the insecticidal activity of these volatile oils which presented positive results on their biological activities. Olonisakin (2014) reported acute toxicity and strong repellence of the volatile oil of CA on cowpea weevil, *Callosobruchis maculatus* (F.) (Coleoptera: Chrysomelidae). A study by Adusei- Mensah *et al*. (2014) corroborated this having reported that CA had the highest lethal activity at low concentration on carpenter ants (*Camponotus nearcticus*). Ugwu *et al. (*2019) also conducted a study on insecticidal potency of ethanol extracts of the seeds of *Azadiractha indica, Jatropha curcas, Piper guineense* and *Afframomum melegueta* against larvae and adult *B.kolae* on stored kolanuts. It was observed that the extract caused the mortality of the larvae and consequently reduced the adult weevil emergence. Babarinde *et al*. (2017) also revealed that the essential oil obtained from *P. guineense* protected some varieties of cowpea from *C. maculatus* infestation. Similarly, Ojiako *et al*. (2018) found that *P. guineense* extract compared very well with the synthetic pesticide (cypermethrin) in the control of the insect pests of cucumber.

Chemical composition of the volatile oils. The occurrence of insecticidal ability of oils from *Citrus aurantifolia* fruit peels and the *Piper guineense* seeds as contact toxicants can be attributed to the high content of the monoterpenes in their volatile oils (Pagare *et al*., 2015). In *C. aurantifolia* (CA) and *P. guineense* (PG) oils, there is a prominence of limonene, gamma-terpinene and caryophyllene which reportedly possess high insecticidal potency (Liu *et al.* 2012; Wang *et al.,* 2015; Zhu *et al*., 2017). Abdullahi *et al*., (2017) reported that limonene ruptures the wax layer of the insect respiratory system resulting in suffocation of the insects as soon as it is applied.

The superiority of the contact toxicity of *C. aurantifolia* to that of the *P. guineense* in this study appears to be due to synergy among its various constituents. Liang *et al.* (2020) reported a synergistic activity of Carvone and limonene against *Tribolium Castaneum*, a food storage pest. More so, the predominance of hydrocarbons such as Tricyclo[5.2.1.0(4,8)] decane,8-methylene-Bicyclo[5.1.0]octane and 9-methyl-9-Borabicyclo[3.3.1]nonane in the *C. aurantifolia* fruit peel oil can equally aid the observed higher insecticidal property (Heipieper *et al.,* 2010; Shaheed *et al*., 2019). This is the first report on using the volatile oils obtained from *C. aurantifolia* fruit peels and *P. guineense* seeds against *B. kolae* infestation on kolanuts.

CONCLUSION

The insecticidal potency of the volatile oils of *Citrus aurantifolia* fruit peels and the *Piper guineense* seeds against *Balanogastris kolae* as contact toxicants have been confirmed. The synergy among the components of the volatile oils, most especially *Citrus aurantifolia* (CA) is highly suspected to be the cause of the efficacy observed in this study. Farmers and traders may consider protecting the kolanuts with CA fruit peel extract to mitigate the effect of *Balanogastris kolae* infestation and preserve the nuts for future purposes. Therefore, *C. aurantifolia* fruit peel oil and *Piper guineense* seed oil can be introduced into pest management scheme to effectively manage the insect pests of kolanuts, most especially *Balanogastris kolae*.

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