JOTCSA, Volume 3, issue 2, (2016), pp. 207-218. **Research article**

Journal homepage: http://dergipark.ulakbim.gov.tr/jotcsa

e-ISSN: 2149-0120

Phytochemical Profile and Insecticidal Activity of Essential Oil from Fresh and Dried Leaves of Nigerian Grown *Citrus meyerii*

Usman Lamidi Ajao[1]*, Olanipekun Bolatito Eunice[2], Ogundele Victor Ayorinde[2] and Musa AbdulRasak Kannike[3]

1. Department of Chemistry, University of Ilorin, P.M.B 1515, Ilorin, Nigeria.
2. Department of Chemical, Geological and Physical Science, Kwara State University, P.M.B 1530, Malete, Nigeria.
3. Department of Crop Protection, University of Ilorin, P.M.B 1515, Ilorin, Nigeria.

Abstract: Leaves of *Citrus meyerii* harvested fresh and dried for four consecutive days were separately hydro-distilled and yielded 0.11 – 0.24 %(w/w) of essential oils. Characterisation of the oils using Gas chromatography - mass spectrometry (GC-MS) revealed the predominance of hydrocarbon monoterpenoids 51.1 – 68.3%. Oxygenated monoterpenoids, hydrocarbon sesquiterpenoids, and oxygenated sesquiterpenoids constituted (17.4 - 24.9%), (12.2 - 19.8%) and (0.0 – 2.5%) of the oils respectively. Principal constituents of the oils were; 3-carene (10.1 - 30.7%), α-pinene (1.0% - 18.7%), d-limonene (5.2 - 6.4%), cis-β-ocimene (5.8 - 14.2%), citronellal (5.4 - 6.8%), and β-elemene (3.0 - 5.8%). The oils were of 3-carene and α-pinene chemotypes. Oils that were of 3-carene chemotype were those from fresh and the leaves dried for one and four days while the oils from leaves dried for two and three days were of α-pinene chemotype. Insecticidal activities of the oils were determined using contact toxicity test on *Callosobruchus maculatus*. Regardless of whether the leaves were fresh or dried, the oils were active against the insect. Interestingly, there was no significant difference in the activities of the oils against the insect.

Keywords: *Citrus meyerii*, Chemotype, 3-carene synthase, β-elemene synthase, insecticidal activities.

Submitted: May 30, 2016. **Revised:** June 30, 2016. **Accepted:** July 11, 2016.

Cite this: Lamidi Ajao *et al.* Phytochemical Profile and Incesticidal Activity of Essential Oil From Fresh and Dried Leaves of Nigerian Grown *Citrus meyerii*. Journal of the Turkish Chemical Society, Section A: Chemistry. 2016;3(2):207–218.

DOI: 10.18596/jotcsa.09331.

Correspondence to: Lamidi Ajao Usman. usmanlamidi@unilorin.edu.ng. Tel: +2348035032378.

INTRODUCTION

Citrus meyerii is a hybrid of *Citrus sinensis* and *Citrus limonium* gotten via budding of the parent plants. Just like the parent plants, the plant is odorous, hence, bearing essential oils. Several chemotypes of oil from the parent plants have been reported in various part of the world. For instance, limonene, α-fenchene, α-terpinolene, 3-carene, and β-pinene chemotypes of leaf essential oil of *Citrus sinensis* grown in Turkey, Pakistan, and Nigeria have been identified [1-3]. Similarly, limonene and α-pinene chemotypes of the leaf essential oil of *Citrus limonium* grown in Crete, Syria, and Iran have been discovered [4-6]. Occurrence of various chemotypes of the oils signified that the formations of its constituents were catalysed by different terpene synthase [7]. The activities of the synthases depend on biotic and abiotic factors which may affect phytochemical profiles and biological activity of the oils.

Usman *et al.*, 2016 investigated the effect of drying on the phytochemical profile and insecticidal activity of leaf essential oil of *C. sinensis* grown in Nigeria. In their investigation, α-fenchene, 3-carene, α-terpinolene and β-pinene chemotypes of the oil were detected. Despite the fact that the oils were of different chemotypes, they were active against *Callosobruchus maculatus* without any significant difference.

Phellandrene chemotype of leaf essential oils of *C. sinensis* from two different harvests (7:00 am and 4:00 pm) were characterised by Omoniwa *et al.,* 2014. The oils significantly ameliorated the impaired hepatic and renal functions. This implies that the time of harvest of leaves did not affect the oil's chemotype and its biochemical activity despite the fact that the quantity of most abundant compound differs appreciably [8]. Vekaria *et al.,* 2002 reported that seasonal variation did not affect the chemotype of leaf essential oil of *Citrus limonium* as they were of limonene chemotype. However, the quantities of the predominant compounds varied significantly [4].

Earlier works on the leaf essential oil of *C. meyerii* revealed the existence of oil of thymol and limonene chemotypes of the plants grown in Florida and Cameroon [9, 10]. The antioxidant assay of the oil from Cameroonian grown *C. meyerii* showed that, the oil demonstrated higher antioxidant property than the synthetic antioxidant (Butylhydroxyltoluene (BHT)). The oil also has higher inflammatory activity than the reference drug nordihydroguaieretic acid (NDGA). Higher biochemical activity of the oil may be due to synergistic actions of the constituents of the oil.

To the best of our knowledge, there are no reports on the effect of dryness of *C. meyerii* leaves on the chemical composition and insecticidal activity of its essential oil. It is on the basis of this that this work aims at monitoring the effect of drying on the chemical composition and insecticidal property of essential oil from the leaf of the plant.

MATERIALS AND METHODS

Sample Collection

Leaves of *Citrus meyerii* were harvested from Park and garden, University of Ilorin, Ilorin, Kwara state. The leaves were air-dried at ambient temperature for four consecutive days and subsequently pulverised.

Oil Isolation

500 g of each of the pulverised leaves of *C. meyerii* was hydro-distilled for 3 hours in a Clevenger-type apparatus, according to the British Pharmacopoeia specification [11]. The resulting oil from each sample was collected, preserved in a sealed sample tube, and stored under refrigeration until analysis.

GC-MS Analysis

An Agilent 19091S gas chromatograph coupled with a quadruple focusing mass spectrometer 433HP-5 mass detector was used. Helium was used as the carrier gas at a flow rate of 1.5 mL/min; all analyses were performed at constant flow. The GC was fitted with a 30 m x 0.25 mm fused silica capillary column coated with phenyl methyl siloxane at a split ratio of 1:50. The film thickness was 0.25 µm. Oven temperature was initially kept at 100 °C for 5 min then 150 °C at a rate of 4 °C for 8 min and to 250 °C at a rate of 20 °C /min. Mass detector conditions were as follows: Transfer line temperature at 300 °C, ionisation mode electron impact at 70 eV. The percentage composition of the oils was computed in each case from GC peak areas. The identification of the components was done based on comparison of retention indices (determined relative to the retention times of series of n-alkanes) and mass spectra with those of authentic samples and with data from literature [12-14].

Insecticidal Activity

Insect Culture

Cowpea beetles (*Callosobruchus maculatus*) were obtained from heavily infested cowpea. They were reared on clean beans and maintained under ambient environmental condition (28 ± 2 °C). The rearing jars were covered with muslin fabric to allow aeration and prevent escape of the insects. The jars were placed inside a wire-netted shelf in the laboratory.

Mortality Determination

10 g of clean, uninfected sweet cowpea grains were separately put into sixteen plastic containers (4 cm diameter) including the one meant for control. 0.1 mL of essential oil from fresh and the leaves dried for four consecutive days were separately added to the beans in the containers except the one meant for the control in triplicate. Apart from the control, the samples were agitated thoroughly for uniform distribution of the oil. Ten active adult *Callosobruchus maculatus* insects were introduced into each container. The control consisted of ten active adult *Callosobruchus maculatus* insects kept in a plastic container containing 10 g of clean, uninfected beans without any portion of the oil in it. The percentage mortality was reported at 6 hours interval for 42 hours.

Data Analysis

The adult toxicity experiments were carried out in a Completely Randomised Design by which the percentage mortality data from the experiment was subjected to one way Analysis of Variance (ANOVA) and where there was a significant difference, the mean separation was done using Duncan Multiple Range Test at 1% level of significance (0.01 probabilities). This statistical analysis was performed using SPSS software, version 21.

RESULTS AND DISCUSSION

Fresh and dried leaves of *Citrus meyerii* afforded oils in the range of 0.11-0.24% (w/w). The yield increased with increase in day of drying (Fig 1). The highest yield was obtained in the leaves dried for four days (0.37%) while fresh leaves afforded the lowest yield (0.11%). The increase in oil yield could be attributed to the decrease in moisture content as the day of drying increases. The oil yields from the fresh and dried leaves were lower than the yield from Cameroonian grown *C. meyerii* [10]. This could be attributed to the variation in agro-climatic conditions of the two countries.

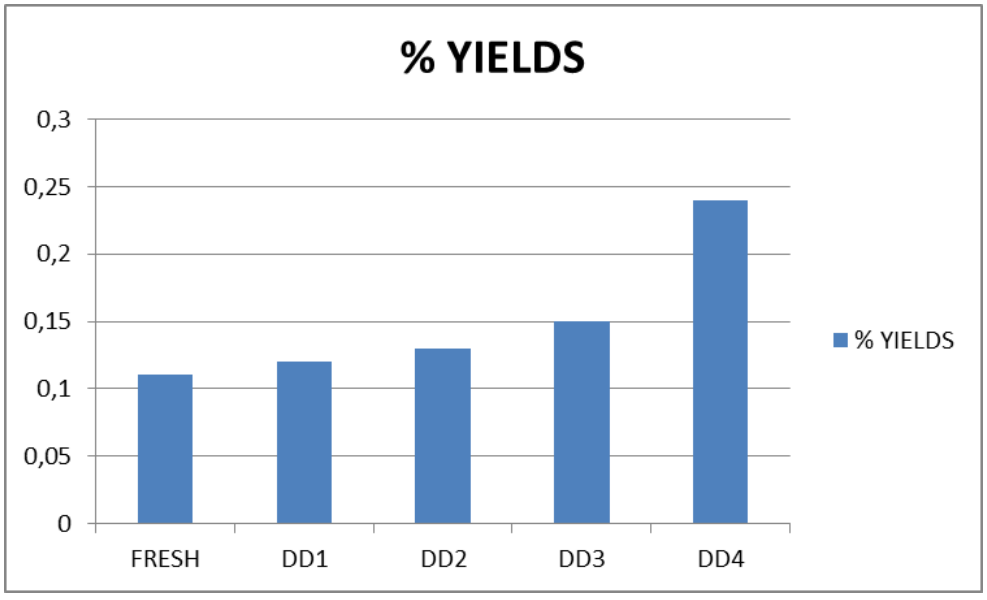


Figure 1. Yield of *Citrus meyerii* oils.

KEY TO FIGURE 1

FRESH: Fresh leaves
DD1: Leaves dried for one day
DD2: Leaves dried for two days
DD3: Leaves dried for three days
DD4: Leaves dried for four days

Table 1 (see Supplementary File) shows the identities, kovats indices and percentage composition of the constituents of essential oils obtained from fresh and dried leaves of *Citrus meyerii*. In the Table, 25-35 compounds that represented 92.0 - 99.9% of the oils were identified. Hydrocarbon monoterpenoids constituted 51.1 - 68.3% of the oils while the percentage composition of oxygenated monoterpenoids ranged from 17.4 - 24.9%. Meanwhile, 9.4 - 19.8% of the oils were hydrocarbon sesquiterpenoids. 0.5 - 2.5% of the oil, were oxygenated sesquiterpenoids which were not detected in the oil obtained from fresh and leaves dried for three days.

The principal constituents of the oils were; α-pinene (1.0 – 18.9%), 3-carene (10.1 – 30.7%), D-limonene (5.2 – 6.4%), Cis-β-ocimene (5.8 – 14.9%), 2-carene (0 – 4.4%), terpinolene (0 – 5.4%), Citronellal (0 – 6.8%), geraniol (0.9 – 5.4%), neral (0.3 – 9.1%), terpinen-4-ol (3.1 – 4.5%), β-elemene (3.0 – 5.5%), γ-elemene (0.0 – 4.8%), cis-β-farnesene (0.6 – 4.4%) and eremophilene (0 – 4.0%). α-Thujene (0 – 1.4%), β-pinene (2.2 – 3.6%), α-phellandrene (1.0 – 1.5%), o-cymene (0.2 – 1.8%), γ-terpinene (0 – 2.7%), 1,3,8-p-menthatriene (0.0 – 1.8%), citronellol (0.0 – 3.7%), geranial (0.0 – 3.5%), humulene 1.0 – 1.6%), β-caryophyllene (2.3 – 3.6%), and phytol acetate (0.5 – 1.4%) were also detected in appreciable quantities. However, some of the compounds that existed in significant quantities in the oils occurred as minor constituents in oil of leaves dried for one and four days. For instance, isopulegol (0.4% and 0.3%) was detected as minor constituent of the oils. α-Terpinene (0.3%), decanal (0.4%), β-copaene (0.4%), aromadendrene (0.2%), spathulenol (0.2%) and caryophyllene oxide (0.3%) were also identified as minor constituents in the oil of leaves dried for one day. Furthermore, geranial (0.4%), methyl geranate (0.3%), β-copaene (0.4%) and γ-muurolene (0.3%) were found in minor quantities in the oil of leaves dried for four days. Their existence as minor constituents signifies that their formations were prematurely terminated by synthase that mediates the transformations of their respective precursors to the compounds.

Comparison of the oils revealed that the composition patterns of the oils differ significantly. Qualitatively, geranyl butyrate, γ-elemene, guaia-9,11-diene that existed in the oil from fresh leaves were not found in the oils from the dried leaves. α-Terpinene, isoterpinolene, decanal, α-copaene, aromadendrene, spathulenol and caryophyllene oxide were identified in the oil from the leaves dried for one day but were not detected in other oils. However, δ-cadinene and eremophilene existed in the oil of leaves dried for three days and did not exist in other oils. γ-muurolene, geranyl acetate, and geranyl linalool were detected in the oils from leaves dried for four days but were not detected in other oils.

Limonene and ortho-cymene were identified in the oil of fresh leaves but not detected in the oils from dried leaves. Furthermore, β-pinene, citronellyl acetate, and methyl geranate were found in the oil of leaves dried for two days but not found in other oils. Similarly, Cis-β-ocimene and phytol were identified in the oils except the oil from leaves dried for three days. α-Thujene and eremophilene were detected in the oils but were not found in the oil of the leaves dried for five days. Absence of some compounds in the oils of the leaves may be due to the fact that the physiological conditions of the plant did not favour their biosynthesis.

Quantitatively, α-pinene, α-phellandrene, 3-carene, o-cymene, d-limonene, cis-β-ocimene, trans-β-ocimene, α-terpineol, neral, geraniol, β-elemene, humulene, β-caryophyllene and cis-β-farnesene were detected in the oils but at varying quantities. α-Terpineol, geraniol, humulene and β-caryophyllene were detected in appreciable quantities in the oil of fresh leaves than other oils. Neral predominates the oil of the leaves dried for one day than other oils. α-Pinene and d-limonene were of greater abundance in the oil of the leaves dried for two days than other oils. Furthermore, terpinen-4-ol and cis-β-ocimene occurred in higher amounts in the oil of the leaves dried for three days than other oils. Similarly, β-elemene, trans-β-ocimene, and 3-carene occur in appreciable quantities in the oil of the leaves dried for four days than other oils. The variations in the quantities of the constituents of the oil are attributable to difference in activity of synthases that mediate the formation of the compounds from their respective precursors [15].

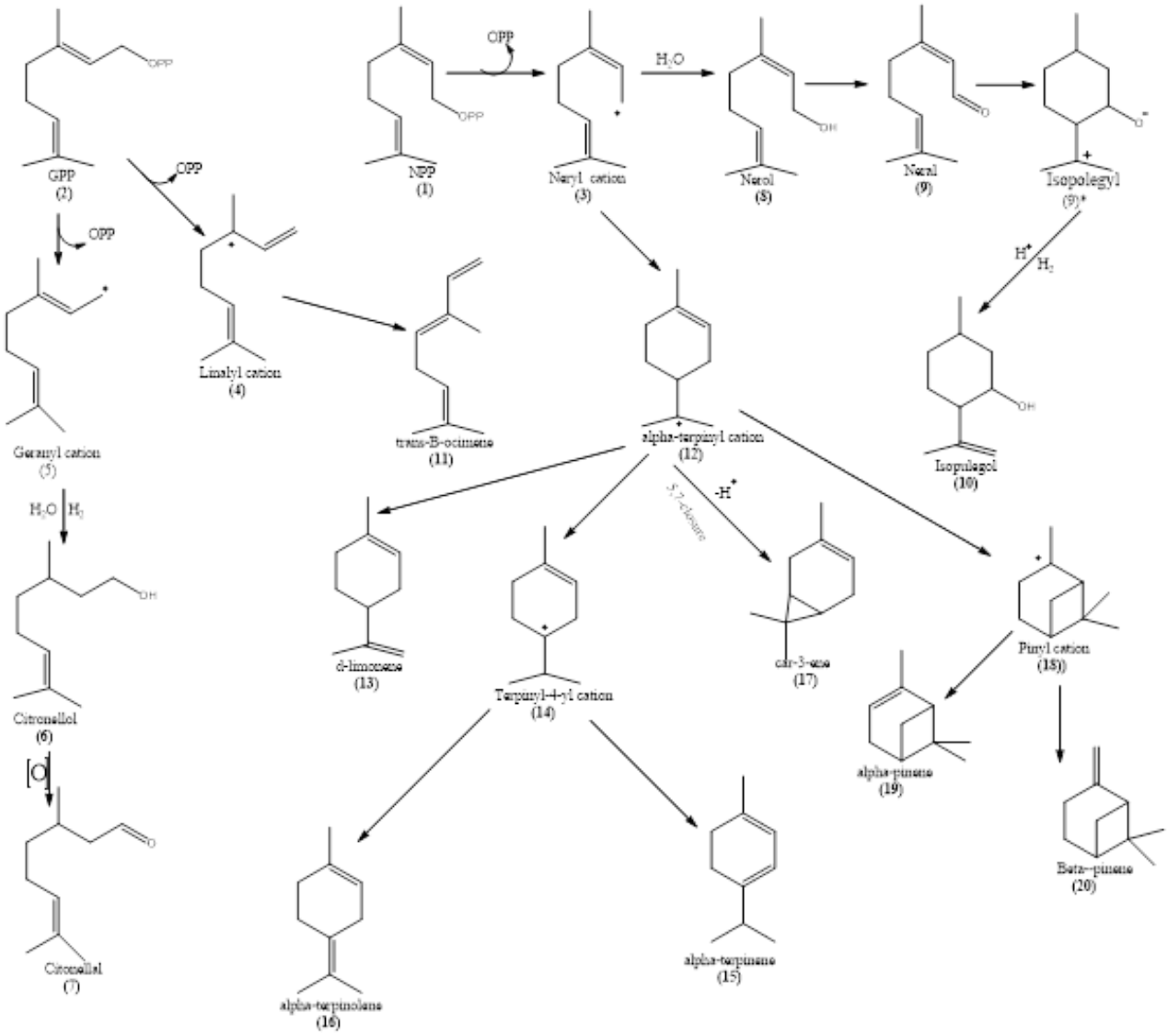
Oils of the fresh leaf of Nigerian and Cameroonian grown *C. meyerii* were of 3-carene and d-limonene chemotypes respectively. Interestingly, 3-carene was not detected in the oil of Cameroonian grown *C. meyerii* which implies that the physiological conditions of the plant did not favour its biosynthesis. The conditions depend on the agro-climatic conditions which determine the activity of the enzyme that aid the formation of the terpenoids. Meanwhile, the biosynthesis of terpenoids is usually catalysed by the most active enzyme that forms the most abundant mono- and sesqui-terpenoids. [7, 15].

The predominance of α-pinene in the oils obtained from the leaves dried for two and three days implied that its synthase mediates the transformation of its precursor to all monoterpenoids. However, the most abundant monoterpenoids in the oils obtained from fresh and the leaves dried for one and four days was 3-carene, its synthase catalysed the formation of all monoterpenoids in the oils. Differences in monoterpenoid synthases that aided the formation of the compounds implied that the period of drying affects the activity of the synthase which depend on the physiological condition of the leaves. Despite the fact that β-elemene was the most abundant sesquiterpenoid in the oils from the fresh and dried leaves and its quantity varied significantly, its synthase catalysed the formation of all sesquiterpenoids in the oils. Quantitative difference in β-elemene content is attributable to variation in physiological condition of the leaves that determine the activities of the enzyme. This variation connotes that the period of drying of the leaves affect the activity of the enzyme. The enzymes catalysed the transformation of mono- and sesquiterpenoid precursor to the respective terpenoids via cationic mechanisms (Schemes 1 and 2).

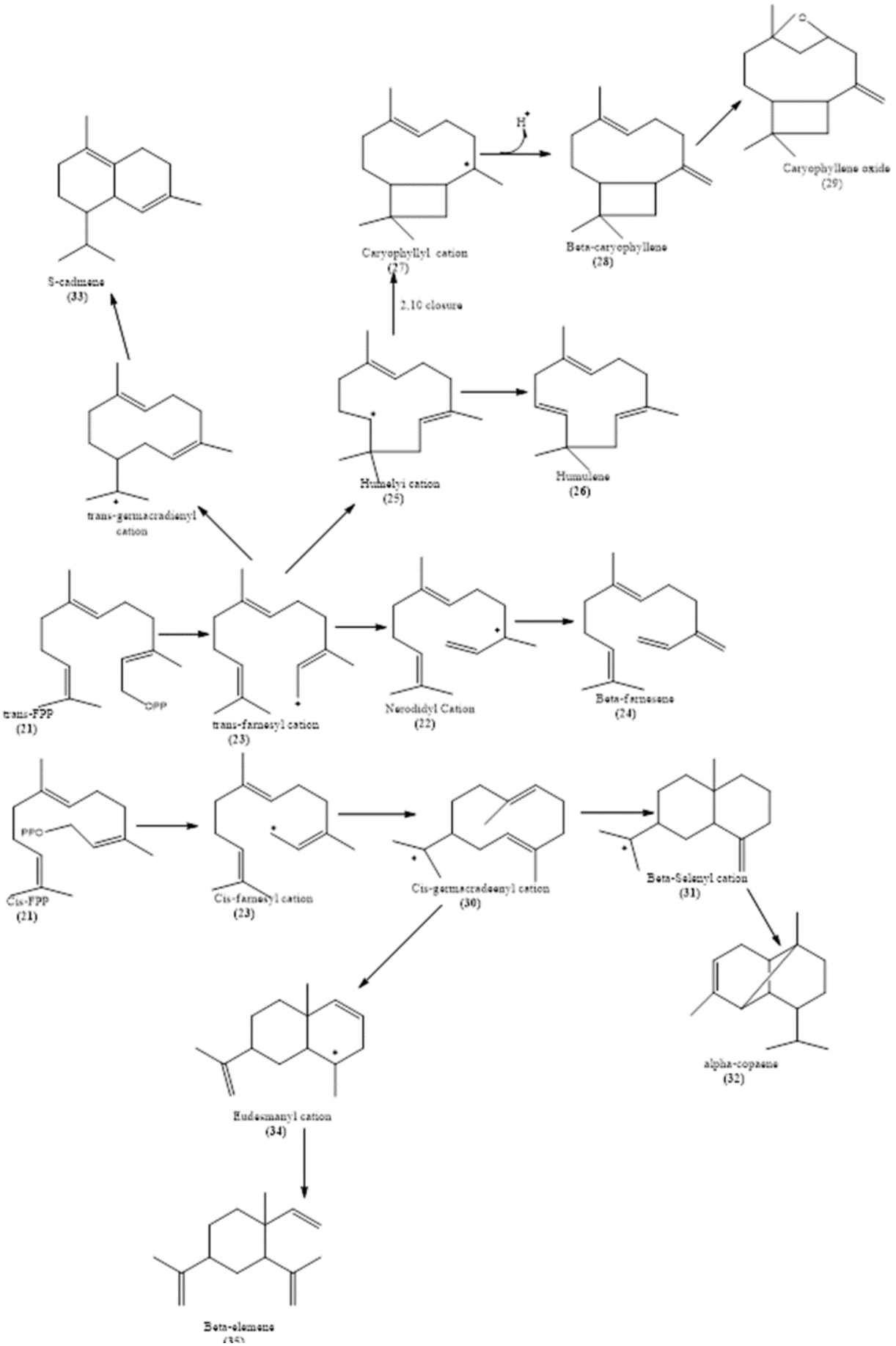
Reaction mechanisms

In Reaction Scheme 1, 3-carene and α-pinene synthases aided the transformation of neryl **(1)** and geranyl pyrophosphate **(2)** to their respective cationic intermediates (neryl **(3)**, linalyl **(4)** and geranyl **(5)** cations). Hydration of geranyl cation with subsequent hydrogenation at C2 and C3 gives citronellol **(6)** which further undergoes oxidation to give citronellal **(7)**. Neryl cation **(3)** undergoes hydration to give nerol **(8)** which subsequently oxidises to neral **(9)**. Nucleophillic attack of carbonyl carbon of the compound **(9)**, by C6-C7 double bond form isopulegyl cation. Deprotonation of the ion at C7 follow by protonation of alkoxide intermediate with hydrogenation at C2-C3 produce isopulegol **(10)**. Linalyl cation **(4)** deprotonate at C1 to form trans-β-ocimene **(11)**. The ion **(3)** undergoes electrophilic attack on C6-C7 double bond to give α-terpinyl cation **(12)**. C8 deprotonation of the ion **(12)** gives limonene **(13)**. 6,7-hydride shift of the ion **(12)** gives terpinyl-4-yl cation **(14)**. Subsequent deprotonation of the latter **(14)** at C1 and C7 gives α-terpinene **(15)** and α-terpinolene **(16)** accordingly. Deprotonation of ion **(12)** at C5 followed by electrophilic attack on C7 forms car-3-ene **(17)**. Folding of the ion (12) towards C2-C3 double bond followed by electrophilic attack of the ion via C2 gives pinyl cation **(18)**. Deprotonation of the ion at C4 and C10 give α-pinene **(19)** and β-pinene **(20)** respectively.

The abundance of β-elemene in the oils implied that its synthase mediated the biosynthesis of all the sesquiterpenoids (Reaction Scheme 2). In the scheme, β-elemene synthase catalysed the ionisation of farnesyl pyrophosphate **(21)** to nerolidyl **(22)** and farnesyl **(23)** cations. Loss of proton at C15 by the ion **(22)** forms β-farnesene **(24)**. Electrophilic attack of the ion on C10-C11 double bond give humulyl cation **(25)** which further under goes deprotonation at C9 to form humulene **(26)**. Electrophyllic attack of the ion **(25)** on C2-C3 double bond form caryophyllyl cation **(27)**. Deprotonation of the ion **(24)** at C15 form β-caryophyllene **(28)**. Epoxidation of the compound **(28)** at C5-C7 double bond produces caryophyllene epoxide **(29)**. Electrophyllic attack of ion **(23)** on C10-C11 double bond gives germacrenyl cation **(30)**. Ion **(30)** undergoes 2, 7-ring closure to form β-selinyl cation **(31)**. Subsequent hydride shift from C11-C10, C10-C1 and deprotonation at C6 followed by electrophilic attack of the ion **(31)** on C6 forms α-copaene **(32)**. Hydride shift from C11-C10, C10-C1 followed by deprotonation of the ion **(30)** at C6 gives δ-cadinene **(33)**. This ion **(30)** undergoes 2, 11- hydride shift and 2,7-cyclisation to give eudesmanyl cation **(34)** which undergoes 4,5-cleavage and deprotonation at C15 to form β-elemene **(35)**.



Reaction Scheme 1. Biosynthesis of monoterpenoids catalysed by 3-carene and α-pinene synthases.



Reaction Scheme 2. Biosynthesis of Sesquiterpenoids catalysed by β-elemene synthase.

Insecticidal Activity

Contact toxicity data of the oils of fresh and dried leaves of *C. meyerii* against adult *Callosobruchus maculatus* are shown in Table 2 (see Supplementary File). The table revealed that at the end of 42 hours of exposure, oils from the fresh leaves and leaves dried for a day caused 85% mortality of *C. maculatus*. Meanwhile, the oils from the leaves dried for two, three, and four days caused 95% mortality of *C. maculatus*. However, irrespective of the days of drying, the oils were highly active, that is, caused very high percentage adult mortality with no significant difference between them at 0.01 probabilities. Rajendra *et al.,* 2007 linked the toxicity of essential oils to stored insect pests with the individual and synergistic actions of the compounds present in the oils [16]. Such compounds include; α-pinene, β-pinene, phellandrene, limonene, γ-terpinene, citronellal, and geraniol. The presence of these compounds in substantial amounts in the oil may be responsible for their activity against *C. maculatus*. However, the contact toxicity activity of the oil compared favourably with activities of the leaf oils of other Citrus species.

CONCLUSION

Oil yields from the leaves varied significantly. It increases with increase in period of drying. This can be attributed to the loss of moisture by drying from the leaves samples which subsequently increase the yields. Phytochemical profile of the oil was also affected by drying as it affects the chemical composition of the leaf oil of *Citrus sinensis* [3]. This could justify the presence of both plants in citrus family. However, the drying did not affect their insecticidal activity against *C. maculatus*. Since the oils have significant activities against the insect. Hence, they can be use as alternative to synthetic insecticide for *C. maculatus*. Meanwhile, it is preferable to extract the oils from dried samples as they afforded more oils than fresh leaf sample.

REFERENCES

1. Kırbaşlar F, Tavman A, Dülger B, Türker G. Antimicrobial activity of Turkish citrus peel oils. Pakistan Journal of Botany. 2009;41(6):3207–12. URL: www.pakbs.org/pjbot/PDFs/41(6)/PJB41(6)3207.pdf.

2. Kamal G, Anwar F, Hussain A, Sarri N, Ashraf M. Yield and chemical composition of Citrus essential oils as affected by drying pretreatment of peels. Int Food Res J. 2011;18(4):1275–82.

3. Usman LA, Watti OI, Ismaeel RO, Ojumoola AO. Effect of Drying on Yield, Chemical Composition and Insecticidal Activity of Leaf Essential Oils of Sweet Orange (*Citrus sinensis*). Journal of the Turkish Chemical Society, Section A: Chemistry. 2015 Dec 14;3(1):1. DOI: 10.18596/jotcsa.98689.

4. Vekiari SA, Protopapadakis EE, Papadopoulou P, Papanicolaou D, Panou C, Vamvakias M. Composition and Seasonal Variation of the Essential Oil from Leaves and Peel of a Cretan Lemon Variety. Journal of Agricultural and Food Chemistry. 2002 Jan;50(1):147–53. DOI: 10.1021/jf001369a.

5. Odeh F, Rahmo A, Alnori A, Chaty M. The Cytotoxic Effect of Essential Oils *Citrus Aurantium* Peels on Human Colorectal Carcinoma Cell Line (LIM1863). The Journal of Microbiology, Biotechnology and Food Sciences. 2012;1(6):1476–87.

6. Oshaghi M, Ghalandari R, Vatandoost H, Shayeghi M, Kamali-Nejad M, Tourabi-Khaledi H, et al. Repellent effect of extracts and essential oils of Citrus limon (Rutaceae) and Melissa officinalis (Labiatae) against main malaria vector, Anopheles stephensi (Diptera: Culicidae). Iranian Journal of Public Health. 2003;32(4):47–52. URL: http://ijph.tums.ac.ir/index.php/ijph/article/download/1940/1921.

7. Wise ML, Savage TJ, Katahira E, Croteau R. Monoterpene Synthases from Common Sage (*Salvia officinalis*): cDNA Isolation, Characterization, AND Functional Expression of (+)-Sabinene Synthase, 1,8-Cineole Synthase, and (+)-Bornyl Diphosphate Synthase. Journal of Biological Chemistry. 1998 Jun 12;273(24):14891–9. DOI: 10.1074/jbc.273.24.14891.

8. Soji-Omoniwa O, Muhammad N, Usman L, Omoniwa B. Effect of leaf essential oil of Citrus sinensis at different harvest time on some liver and kidney function indices of diabetic rats. Int J Biol Vet Agric Food Eng. 2014;8:484–488. URL: http://www.waset.org/publications/9998274.

9. Moshonas MG, Shaw PE, Veldhuis MK. Analysis of volatile constituents from Meyer lemon oil. Journal of Agricultural and Food Chemistry. 1972 Jul;20(4):751–2. DOI: 10.1021/jf60182a018.

10. Dongmo PMJ, Tchoumbougnang F, Boyom FF, Sonwa ET, Zollo PHA, Menut C, et al. Antiradical, antioxidant activities and anti-inflammatory potential of the essential oils of the varieties of Citrus limon and Citrus aurantifolia growing in Cameroon. Journal of Asian Scientific Research. 2013;3(10):1046–1057. URL: http://www.aessweb.com/pdf-files/jasr%203(10),%201046-1057.pdf.

11. Pharmacopoeia B. Vol. 11, London: HM Stationary Office. PA; 1980.

12. Adams RP. Identification of essential oil components by gas chromatography/mass spectrometry. 4th ed. Carol Stream, Ill: Allured Pub. Corp; 2007. 804 p. ISBN: 978-1-932633-21-4.

13. Joulain D, König WA. The atlas of spectral data of sesquiterpene hydrocarbons. EB-Verlag; 1998.

14. Jennings W, Shibamoto T. Qualitative analysis of flavor and fragrance volatiles by glass capillary gas chromatography. San Francisco: Academic Press; 1980. 472 p. ISBN: 978-0-12-384250-3.

15. Degenhardt J, Köllner TG, Gershenzon J. Monoterpene and sesquiterpene synthases and the origin of terpene skeletal diversity in plants. Phytochemistry. 2009 Oct;70(15–16):1621–37. DOI: 10.1016/j.phytochem.2009.07.030.

16. Rajendran S, Sriranjini V. Plant products as fumigants for stored-product insect control. Journal of Stored Products Research. 2008 Jan;44(2):126–35. DOI: 10.1016/j.jspr.2007.08.003.

Research article

Türkçe Öz ve Anahtar Kelimeler

Nijerya Bölgesinde Yetişen *Citrus meyerii*'nin Taze ve Kurutulmuş Yapraklarından Elde Edilen Esansiyel Yağın Fitokimyasal Profili ve İnsektisit Aktivitesi

Usman Lamidi Ajao, Olanipekun Bolatito Eunice, Ogundele Victor Ayorinde ve Musa AbdulRasak Kannike

Öz: *Citrus meyerii* yaprakları taze olarak ve dört ardışık gün kurutularak ayrı ayrı hidrodistilasyona uğratılmış ve ağırlıkça %0,11-0,24 esansiyel yağ vermiştir. Yağların Gaz kromatografisi - kütle spektrometrisi (GC-MS) ile karakterizasyonu %51,1 - 68,3 oranında baskın bir hidrokarbon monoterpenoidlerini göstermiştir. Oksijenli monoterpenoidler, hidrokarbon seskiterpenoidler ve oksijenli seskiterpenoidler yağların sırası ile (%17,4 - 24,9), (%12,2 - 19,8) ve (%0,0 - 2,5) kısmını oluşturmuştur. Yağların baskın bileşenleri 3-karen (%10,1 - 30,7), α-pinen (%1,0 - 18,7), d-limonen (%5,2 - 6,4), cis-β-osimen (%5,8 - 14,2), sitronellal (%5,4 - 6,8) ve β-elemen (%3,0 - 5,8) olarak tespit edilmiştir. Yağlar 3-karen ve α-pinen kemotiplerinden oluşmaktadır. 3-Karen kemotipi olan yağlar taze ve bir ve dört gün kurutulan yapraklardaki esansiyel yağdan oluşmaktadır, buna karşılık iki ve üç gün kurutulan yapraklardaki yağlar α-pinen kemotipi ağırlıklıdır. Yağların insektisit aktiviteleri *Callosobruchus maculatus* üzerindeki temas toksisite testleri ile belirlenmiştir. Yaprakların taze veya kurutulmuş olmasından bağımsız olarak, yağlar böceklere karşı aktivite göstermiştir. İlginç bir şekilde, yağların böceğe karşı aktivitesinde belirgin bir farklılık tespit edilmemiştir.

Anahtar kelimeler: *Citrus meyerii*, Kemotip, kar-3-ene sentaz, β-elemen sentaz, insektisit aktivitesi.

Sunulma: 30 Mayıs 2016. **Gözden geçirme:** 30 Haziran 2016. **Kabul:** 11 Temmuz 2016.