COMPARATIVE STUDY OF ESSENTIAL OIL COMPOSITION OF FRESH AND DRY PEEL AND SEED OF *CITRUS SINENSIS* (L) OSBECK VAR SHAMUTI AND *CITRUS PARADISI* MACFADYEN VAR MARSH

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(Received: 2nd June, 2014; Accepted:17th July, 2014)

ABSTRACT

Citrus essential oils have an impressive range of food and medicinal uses. In this study investigation has been conducted on the variation in the yield, chemical composition and their identities in oils isolated from fresh and air-dried peel and seed of orange (*Citrus sinensis*) and grape(*Citrus paradisi*) planted in a cocoa farm. The yield of solvent-extracted essential oils from the fresh peel and seed ranged between 0.31 and 1.01%, while the yield in the air-dried peel and seed of the two different citrus samples ranged between 0.98 and 2.30%. The four major compounds present in all the oils are limonene, myrcene, alpha terpinene and camphene which ranged between 74.97 - 90.58%, 5.19 - 10.41%, 0.14 - 4.00% and 0.05 - 3.87%, respectively in fresh peel and seed. In the air-dried peel and seed their values ranged between 58.64 - 77.30%, 0.08 - 5.04%, 0.05 - 3.68% and 0.02-4.88%, respectively for the four compounds. The fresh peel and seed have lower yield but contain higher percentage concentrations of major compounds that serve as compound identification for the citrus family. Air-dried peel and seed have alcohol components like spathulenol (9.78 - 15.13%), linalool (5.05 - 9.27%), nerol (7.98 - 8.60), alpha terpeniol (1.06 - 1.15%) and farnesol (1.54 - 1.66%) which were not present in fresh samples of the two different citrus samples. Apart from concentration differences, the results of this study are similar to other research work in other regions of the world on citrus; the only difference was the identification of camphene in this study but which was not found in citrus located in other regions of the world under consideration.

Keywords: Citrus peel, Seed, Fresh, Air-dried, Essential Oil.

INTRODUCTION

Citrus essential oils are obtained from various *Citrus* species found within the family Rutaceae and the subfamily Aurantioideae; these include about 17 species distributed throughout the tropical and temperate regions (Davies and Albrigo, 1994; Shaw, 1977). They are evergreen trees that give fruits of different forms and sizes (from round to oblong), which are full of fragrances, flavor and juice. The citrus genus includes various species; oranges (*Citrus sinensis*), limes (*Citrus aurantifolia*), tangerine (*Citrus reticulate*), lemons (*Citrus limon*) and grapefruit (*Citrus paradisi*) (Burcu *et al*., 2011; Mohammed *et al*., 2010). Citrus is one of the most important commercially-grown crops in the world. The world production of citrus was estimated to be 51.8 million metric tonnes during 2013 with Brazil, China and United States as the first three leading producers in the world and Egypt, South Africa and Morocco, respectively as the leading producers in the African continent (USDA, 2014). Citrus essential oil are a mixture of volatile compounds and mainly consists of monoterpene hydrocarbons of about 70-95% along with smaller amount of sesquiterpene hydrocarbons which are responsible for a characteristic flavor (Muhammad, 2006).

Although, the fruits are mainly used for dessert, it has significant economic value for its essential oil (EO) due to their aromatic compounds (Minh *et al*., 2002). Citrus flavours are used in beverage, confectionary, cookies and desserts (Buchel, 1989; Dharmawan *et al*., 2007). The exocarps of *C. reticulata* and *C. sinensis* are used for flavorings of liquor. The composition of the oil is significantly affected by the ripeness of fruits, vegetative stage of plant, storage condition and extraction method (Njoroge *et al*., 2006; Venkateshwarlu and Selvaraj, 2000). The quality and the odour of the oil are influenced by the limonene content which may vary in the different agro-climatic conditions (Dharmawan *et al*., 2007).

Like many other plant families, Rutaceae has been reported to be a source of potent botanical insecticide. Peel and seed solvent extract of citrus plant have shown insecticidal activity against several coleopteran and dipteran species (Su *et al*.,

1972; Greany *et al.*, 1983; Sheppard 1984; Salvatore *et al.*, 2004). Previously it has been established that *C. aurantium* peels contain secondary metabolites with insecticidal activity against *B. oleae* adult (Siskos *et al.*, 2007).

Citrus essential oils have been applied in many products, such as foods, beverages, cosmetics and medicines, as flavouring agents as well as for aromatherapy. They are also used for their germicidal, antioxidant and anticarcinogenic properties (Guenther, 1948; Mukhopadhyay, 2000). The active constituents exist in citrus EOs, such as limonene, α-pinene, β-pinene and α-terpinolene which exhibit a wide spectrum of antimicrobial activity, as reported by many studies in other plants (Jirovetz *et al.*, 2005; Magwa *et al.*, 2006; Skocibusic *et al.*, 2006; Matasyoh *et al.*, 2007).

The biological properties of EOs extracted from medicinal plants may show differences depending upon the distinctness in cultivation, origin, vegetative stage and growing seasons of the plants and geographical location (Deans *et al.*, 1992; Muller-Riebau *et al.*, 1995; Kustrak *et al.*, 1996; Leung and Foster, 1996; Milos *et al.*, 2000; Jerkovic *et al.*, 2001). In addition, other factors that can affect chemical composition are extraction process, stage of harvesting and post-harvest processing (Dean *et al.*, 1992).

In this study, the main aim was to obtain essential oils from orange (*C. sinensis*) and grapefruit (*Citrus paradisi* L.) peel and seed using solvent extraction method under fresh and air dried conditions in order to compare the yield and chemical composition. Also this study will obtain information about the chemical variability of the peel and seed from this region of Nigeria and compare to result from other regions.

MATERIALS AND METHODS

Sample Collection and Preparation

The fresh fully matured ripened fruits of two species of Citrus: Sweet orange (*C. sinensis*) (CS) and grape (*C. paradisi*) (CP) were collected from a cocoa farm in Isinbode-Ekiti, Ekiti-State. Nigeria. The citrus varieties were identified in the Department of Plant Science and Biotechnology of Adekunle Ajasin University, Akungba Akoko. Ondo State, Nigeria. The two citrus fruit species were washed free of sand over tap water. The fruits were then peeled off carefully with the help of a sharp razor blade to avoid any damage of oil glands. Due to practical reasons, the *Citrus* peels under testing were processed under two categories: one portion used as fresh, the other air-dried at ambient temperature (30 ^{0}C) were ground and extraction took place immediately while the other portion was air-dried.

Extraction

Precisely, 200 g of each of the samples was packed into a Soxhlet apparatus and extracted exhaustively with 750 ml of Hexane for 3 h. The solvent was recovered using a rotary evaporator. The oils were made to be moisture free by filtering through the anhydrous sodium sulphate. The oils were transferred into brown sample bottles and stored in the refrigerator until ready for analysis

Analysis of the Components

The volatile constituents were identified by gas chromatography technique using direct injection in the split mode with a split ratio of 20:1 under the following conditions: Hewlet-Packard 6890 equipped with a quartz capillary column; 30 mm x 0.25 mm internal diameter and 0.25 μm film thickness was used. Hydrogen was the carrier gas at 1.0 ml/min flow rate; oven temperature, 40^{o}C to 200^{o}C at a rate of 5^{o}C/min then held isothermal for 2 min. Injector temperature and volume are 150^{o}C and 1.0 μl, respectively. The ionization of the sample components was performed on E.I mode (70eV). The identification of different constituents was performed by comparison of their retention time and mass spectra with those of the library.

RESULTS AND DISCUSSION

The yield and colour of the essential oil from the peel and seed samples are presented in Table 1. The yield ranged between 0.31-1.01% in fresh peel and seed with peel having the lowest values. The air-dried peel and seed had the yield ranged between 0.98 - 2.30%. The fresh samples generally had low yield when compared to dry samples, this may be due to the high water content of the materials. The seed of both citrus had higher yield when in dry condition than in fresh. According to Williams, 1996, depending on the variety of the fruit peel or seed, citrus yields essential oil content

of between 0.5 and 5.0%.

Table 1: Essential Oil Yield and Colour of Essential Oil from Peel and Seed of CS and CP

Species	Condition	Yield %v/w	Colour
C. sinensis (peel)	Fresh	0.40	Light green
C. sinensis (seed)	Fresh	0.67	Light yellow
C.paradisi (peel)	Fresh	0.31	Light green
C.paradisi (seed)	Fresh	1.01	Light yellow
C. sinensis (peel)	Dry	1.09	Dark green
C. sinensis (seed)	Dry	1.73	Dark yellow
C.paradisi (peel)	Dry	0.98	Dark green
C.paradisi (seed)	Dry	2.30	Dark yellow

The yield obtained from this study with respect to the effects of drying condition on peel and seed essential oil are in agreement with the findings of Asekun *et al.* (2007) who also investigated higher oil contents from oven-dried samples of *Helichrysum odoratissimum* in comparison with fresh material. Asekun *et al.* (2006) had earlier studied the effects of drying on the yield and chemical composition of essential oil from the aerial parts of *Leonotis leonurus* and found that the oils derived from sun-dried plant material had better yield than those from the air and oven-dried materials. Some other reports in the literature also revealed considerable effects of drying on the yield and characteristics of the essential oils (Rahula *et al.*, 1973; Laranja *et al.*, 2003). Furthermore, the yield obtained in this work which ranged between 0.31 - 2.30% is slightly higher than the one obtained by Tu *et al.* (2002) (0.20 -2.0%) and Kamal *et al.* (2001) (0.24 – 1.07%). This may be due to the method of extraction in their studies, steam distillation was used and solvent extraction was used in this study. In essential oil extraction methods, solvent extraction technique has been found to have higher yield than other methods.

The results of the essential oil composition of the fresh and air-dried peel and seed of *C. sinensis* and *C. paradisi* are shown in Table 2. Peel and seed oils consisted almost exclusively of hydrocarbons, with limonene as major component. Limonene, myrcene, α-pinene and camphene were the main components in the EO. Linalool is the most abundant oxygenated monoterpene. The fresh peel and seed were found to contain high concentration of limonene in all the samples when compared to air-dried samples, for instance limonene content in the fresh peel ranged between 88.32 to 90.58% in CP and CS, respectively, while in fresh seed limonene content ranged between 74.97 – 81.73% for CP and CS, respectively. Limonene percentage concentration ranged between 58.64 - 65.95%, respectively for air-dry peel of CP and CS, while it ranged between 67.92 to 77.30% for CP and CS, respectively for air-dried seed. Myrcene is another compound that is present in all the samples and it ranged between 0.08% minimum in *C. sinensis* dry peel (CSDP) to maximum of 10.41% in *C. paradise* fresh seed (CPFS). Camphene ranged between 0.02 to 4.88%, respectively in CSDP and CPDS. The fresh peel sample is however higher in limonene content than the fresh seed. Moreover the fresh peel of CS was found to contain more number of terpenoids than CP fresh peel, for instance α-thujene, γ-terpinene, citral and neral were present in C. *sinensis* fresh peel (CSFP) but not in *C. paradise* fresh peel (CPFP) and the fresh seed of the two citrus almost have the same number for terpenoids except linalool that is present in CSFS. In all the samples, it was observed that air-dried samples of CS and CP peel have the highest number of compounds. Compounds like nerol, α-terpineol, spathulenol and farnesol (alcohol) were present in these two oils at higher concentration in *C. paradise* dry peel (CPDP) than CSDP but were absent in all the remaining oils. This alcohol content ranged between 28.91% to 35.81% in CS and CP, respectively. The general result indicates that fresh sample has more main components in

higher concentration than the dry one. This may be due to the volatile nature of terpenoids during the course of its drying. The alcohol content that was not found in the fresh samples was seen in the dry sample of the citrus most especially the dry peel.

Table 2: Essential Oil Composition (%) of Fresh and Air-dry Peel and Seed of *C.sinensis* and *C. paradisi.*

Compound	CSFP %	CPFP %	CSFS %	CPFS %	CSDP %	CPDP %	CSDS %	CPDS %
Cymene	-	-	-		2.09	2.26	1.82	1.37
α-phellandrene	-	-	0.27	0.04	-	-	0.03	0.72
α- terpinene	0.14	1.86	4.00	6.62	0.05	0.06	1.59	3.68
β-ocimene	-	-	0.27	0.02	-	-	0.03	0.02
Camphene	0.17	0.05	3.87	3.74	0.02	0.03	1.38	4.88
Terpinolene	-	-	0.22	0.02	-	-	0.03	0.02
Sabinine	-	-	0.36	0.03	-	-	0.04	0.02
Limonene	90.58	88.32	81.73	74.97	65.95	58.64	77.30	67.92
α-pinene	2.27	2.08	0.93	3.33	-	1.36	3.17	2.31
β-pinene	-	0.01	0.58	0.05	-	-	0.08	0.05
Myrcene	5.19	7.00	5.35	10.41	0.08	0.86	2.65	5.04
Thujene	0.10	-	0.21	0.01	0.14	0.16	6.38	6.84
γ-terpinene	0.02	-	0.96	0.90	-	-	0.21	0.67
Citral	0.03	-	-	-	0.03	0.03	-	-
Geranial (nera)	0.49	-	-	-	0.57	0.62	-	-
Linalool	-	-	0.04	-	8.57	9.27	5.05	6.78
Nerol	-	-	-	-	7.96	8.60	-	-
α-terpineol	-	-	-	-	1.06	1.15	-	-
Spathulenol	-	-	-	-	9.97	15.13	-	-
Farnesol	-	-	-	-	1.54	1.66	-	-

CSFP = *C. sinensis* fresh peel, CPFP = *C. paradisi* fresh peel, CSFS = *C. sinensis* fresh seed, CPFS = *C. paradisi* fresh seed, CSDP = *C. sinensis* dry peel, CPDP = *C. paradisi dry peel*, CSDS = *C. sinensis* dry seed, CPDS = C. *paradisi* dry seed.

Table 3: Essential Oil Composition of Fresh Peel and Seed of Present Study and Fresh Peel of Other Region

Compounds	CSFP %	CSFS %	CPFP %	CPFS %	ITCS %	FLCS %	TUCS %	PKCP1 %	IRCS %	PKCP2 %	GRCS %
α-terpinene	0.14	4.00	1.86	6.62	-	-	1.0	2.11	0.20	-	-
Camphene	0.17	3.87	0.05	3.74	-	-	-	-	-	-	-
Sabinene	-	0.36	-	0.03	1.00	0.24	0.06	-	0.5	-	0.2
Limonene	90.50	81.73	88.32	74.97	93.67	95.17	88.60	86.27	90.50	76.28	94.20
α-Pinene	2.27	0.93	2.08	3.33	0.67	0.42	0.70	1.26	1.40	1.26	0.80
β-Pinene	-	0.58	0.01	0.05	-	-	1.20	-	-	5.45	-
Myrcene	5.19	5.35	7.00	10.41	2.09	1.86	0.90	6.28	2.50	1.30	2.80
Thujane	0.10	0.21	-	0.01	-	-	-	0.15	-	-	-
Octanal	-	-	-	-	-	-	-	-	-	0.80	-
linalool	-	0.04	-	-	0.31	0.25	0.70	0.02	0.80	2.32	0.80
3-carene	-	-	-	-	0.31	-	0.50	-	-	-	-
Decanal.	-	-	-	-	0.27	o.28	-	-	-	-	-

CSFP = *C.sinensis* fresh peel, CSFS = *C. sinensis* fresh seed, CPFP = *C. paradis* fresh peel, CPFS = *C. paradise* fresh seed, ITCS = Italian *C. sinensis* (Verzera et al., 2004), FLCS = Florida *C. sinensis* (Vora, 1983), TUCS = Turkey *C.sinensis*(Burcu*, et al*., 2011) PKCP1 = Pakistan *C. paradisi* 1(Muhammad, *et al*., 2006), IRCS = Iran *C. sinensis (*Parvizetal,2006), PKCP2 = Pakistan *C. paradis* 2 (), GRCS = Greece *C.sinensis*(Filitsa, *et al*., 2013)

Table 3 shows the chemical composition of fresh CS and CP peel and seed of this study and that of some fresh peels from other regions of the world. In all the essential oil under consideration, it was noticed that limonene, myrcene, pinene and linalool were present in all the geographical locations in the order of limonene > myrcene > pinene. Limonene percentage value ranged between 74.97- 95.17% in CPFS and Florida sample (Bauer and Surburk, 2001), respectively and this is in agreement with Muhammad *et al.* (2006) that stated that limonene value of citrus range between 75- 97%. Myrcene in this study is higher in percentage concentration (2.09-10.41%) than the values of other essential oils from other regions which ranged between 0.90-6.20% in Turkey and Pakistan, respectively (Burcu *et al*., 2011; Muhammad *et al*., 2006). The alpha pinene in this study is in higher concentration (0.65 - 3.33%) than other regions which ranged between 0.42-1.26% in Florida and Iran, respectively (Burcu *et al*., 2011; Parviz *et al*., 2011).

CONCLUSION

The essential oil yields in dry samples are higher than the fresh but the percentage concentration of the major compounds are higher in the fresh than the dry. Limonene, myrcene, α-terpinene, pinene and camphene were all present in the oils under study but when compared with the other region only camphene were absent in other region but present in this study. Linalool was found in other oils from other region but is only on the dry samples that have linalool in this study with higher concentration than other regions.

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