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Waste to wealth: Industrial raw materials potential of peels of Nigerian sweet orange (*Citrus sinensis*)

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Orange fruits have been part of human diet for ages due to its nutritional and medicinal values. But consumption of orange fruits generates orange peel wastes that could bring about environmental pollution if not properly handled. Towards recycling of wastes and avoiding littering and waste-related environmental degradation, this study was carried out to explore the components of orange peels with a view to establishing their raw material potentials. Orange peels cut into small bits were subjected to steam distillation process and the extracted essential oil was put through some chemical characterization procedures for purposes of identifying its components. Ultraviolet-visible spectrophotometric scan of the extract, revealed a single prominent peak at a wavelength of 300 nm, as was also the case with paper chromatography which showed one major band separation. Subsequent infrared spectroscopy for structural configuration gave three main identifiable peaks reflecting structural, functional and group/bond positions: C=C, C-H and =C-H at 1640 to 1680 cm^{-1} , 2850 to 2960 cm^{-1} and 3100 cm^{-1} bond positions, respectively, and these tallies exactly with those found in the structure of limonene, thus confirming the later (one of the terpenes), as a dominant component of the orange peel among others that were present in small amounts. Limonene is an essential oil with wide application in industrial and domestic domains. Thus, exploring essential oil is an additional way of evaluating the underlying economic value of citrus due to their usefulness as food nutrient and flavor, and their waste peel is a source of essential oil which is useful in cosmetics, pharmaceuticals, and other industrial and domestic applications. Processing of citrus peels into essential oils is a sure way of transforming these wastes with great potential for environmental pollution into a resource with great potential for economic prosperity, and also for securing the public health impacts of safer and healthier environment, likely to be obtained from the indirect waste management option so offered.

Keywords: Industrial raw materials, *Citrus sinensis* peels.

INTRODUCTION

Citrus is a term commonly used for genus of flowering plants in the family *Rutaceae* originating in tropical and sub-tropical south-east regions of the world. The taxonomy and systematic of the genus are complex, and the precise number of natural species is unclear (Ellis et al., 1985). The fruits are botanically classified as berries and could be termed as a hesperidium. Citrus fruits have a rough, robust and bright (green to yellow) coloured

skin. They are usually 4 to 30 cm long and 4 to 20 cm in diameter, with a leathery surrounding rind or skin known as epicarp (or flavedo) that covers the fruits and protects it from damages. Citrus fruits are notable for their fragrance, partly due to flavonoids and limonoids contained in the rind (Manthey, 2004). The endocarp is rich in soluble sugars and contains significant amounts of vitamin C, pectin, fibers, different organic acids and potassium salt which give the fruits its characteristics citrus flavor (Roger, 2002). Citrus juice also contains a high quantity of organic acids (citric, malic, acetic and formic acids) (Rogers, 2002).

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In many parts of the world, citrus fruits particularly those of the class *Citrus sinensis* (sweet orange) have always remained part of human diet for many years. In recent times, however, they have assumed greater importance in diets of both urban and rural dwellers. The increased interest in their consumption is not only due to their sweet refreshing properties but also as a result of increased knowledge of their nutritional and medicinal values. Orange fruit and its juice have several beneficial, nutritive and health properties (Okwu and Emenike, 2006). They are rich in vitamins especially ascorbic and folic acids. Over the last decades, many other virtues and medicinal benefits of orange fruits have been discovered besides their anti-scurvy property (Rapisararda et al., 1999). There is convincing epidemiological evidence that the consumption of orange fruits is beneficial to health and contributes to the prevention of degenerative process, particularly lowering incidence and mortality rate of cancer, cardio- and cerebro-vascular diseases (Rapisararda et al., 1999). The protection that orange fruits provide against these diseases has been attributed to the various antioxidant phytonutrients contained in citrus species (Okwu and Emenike, 2006; Rapisararda et al., 1999).

Beyond these, essential oil is another citrus by-product which attracts keen interests. Essential oils are volatile odoriferous oils occurring in buds, flowers, leaves, fruits, seeds, roots, etc. of plants (Heath, 1981). They represent the total odour principle of any single botanical species. In recent years, the citrus essential oil has been identified in different parts of the fruit (preferably present in fruit flavedo) and is generally isolated by some physical process (by expression, solvent extraction or distillation). They are mostly volatile and are made up of a wide variety of organic compounds of many different functional groups and molecular structures. The organic constituents present in essential oils include the terpenes and their oxygenated derivatives, aromatic compounds of benzenoid structure, aliphatic hydrocarbons and their oxygenated derivatives as well as nitrogen- or sulphur-containing compounds (Rogers, 1981). Other compounds also present in essential oils are alcohols, aldehydes, acids and other aromatic compounds (Stashenko et al., 1996; Caccioni et al., 1998; Vekiari et al., 2002; Pultrini et al., 2006; Sharma and Tripathi, 2006).

The actual functions of the essential oils in plants are not yet fully understood. What is certain however is that flowers (rich in essential oils) attract insects involved in pollination by their odour, suggesting that these oils may have a role in natural selection and preservation. The chemical constituents of the essential oils may participate in physiological reactions. For instance, balsams and resins contain essential oils and they act as protective seals against diseases or parasites, and also prevent loss of sap when the three trunks are damaged. These aromatic compounds are however, important raw materials with wide applications particularly in the food

and flavor industries (Reische et al., 1998). They can also serve as an excellent starting material in the synthesis of fine chemicals including new fragrances for the cosmetics industry (Lis-Balchin and Hart, 1999).

Citrus, a major plant source of essential oils is one of the most important commercial fruit crops grown in all continents of the world (Tao et al., 2007). In China alone, according to the FAO report (2005), citrus covers a cultivating area of 17.1 million hm^2 , with a production capacity of almost 16 million tons. Natural and cultivated hybrids of citrus include commercially important fruits such as the oranges, grape fruit, lemons, some limes and some tangerines. Sweet orange (*C. sinensis*) is one of the native citrus cultivars grown in China, constituting about 60% of total citrus yields and keeping a stable development (Shan, 2006). In current citrus industry, emphasis are laid only on orange fruits harnessed and marketed fresh or as processed (and canned) juice, while fruit peels produced in great quantities during the process are mainly discarded as waste. For this reason, researchers have focused on the utilization of citrus products and by-products (Kubo et al., 2005; Wu et al., 2007). Thus, the peels of sweet orange are not only left out as waste but also considered as one of the major factors that hamper the development of citrus industry. In Florida alone, citrus processing yields about 1.2 million tonnes of dry orange waste most of which is currently marketed as low value feed for cattle. Of the 36.0 metric tonnes world production, Nigeria produces 0.3 million tonnes and has the potential to produce more orange wastes in high proportion. Though Nigeria is not well noted for the exportation of citrus fruits, she has the potential to produce more for both local and international markets. Presently, the local processing of citrus fruits is on the increase to meet increasing local demands for fruit juice that was previously met by large-scale importation. Of all the citrus fruits, sweet orange is the commonest and the most widely cultivated and consumed in the major 15 citrus growing states in Nigeria, namely: Cross River, Imo, Anambra, Osun, Ondo, Lagos, Ogun, Oyo, Kwara, Benue, Abia, Plateau, Kogi, Kaduna, Enugu and Bauchi states (Odbanjo and Sangodoyin, 2002). This also means quantum increases in the volume of waste derived from its fruit juice production.

Given the array of chemical contents of orange peel as reviewed briefly earlier, it will amount to huge economic waste to consider the orange peels as waste garbage meant only for the waste bin. Therefore, there is need to explore ways of reducing the environmental menace that may arise from huge orange peel wastes. In this regard, the possibility of converting these waste peels to industrial raw materials is being considered, and forms the central interest behind the conception of this study. The major interest in this regard is to explore the contents of the Nigerian sweet orange peel with a view to harnessing the plenipotential essential oils hopefully abundant in these supposed wastes. Exploring and

exploiting the abundant essential oil, seemed to be an additional way to evaluate the underlying economic values of citrus due to the special roles it plays in food, flavor and cosmetics industries. Extraction, identification and separation of the components of Nigerian sweet orange peel is another way of enhancing the economic value and industrial application of this cultivar, providing a cheaper and safer alternative source of raw material for industrial and other useful purposes, as well as providing a safer and cheaper means of waste management through transformation of orange peel wastes to a source of industrial wealth. The significance of this study could be seen from the fact that: successfully accomplishing the objectives of this study would mean a secured and safer environment for our enjoyment, boost to job/employment opportunities for the unemployed, increase in productivity and buoyancy of industrial economy as well as general economic growth.

MATERIALS AND METHODS

Sample

Oranges used were bought from a local (Ihiagwa) market in Owerri West L.G.A, Imo State, Nigeria, and taken to the Laboratory of the Department of Industrial Chemistry, Federal University of Technology, Owerri, Nigeria. All chemicals used for the study (anhydrous sodium sulphate salt, acetone, ethanol, acetaldehyde, acetic acid, n-hexane and distilled water) were of reagent grade and were sourced from the laboratories of the Department of Industrial Chemistry, Federal University of Technology, Owerri, where the extraction procedure was carried out.

Extraction of orange oil from the peel

The extraction of orange peel oil was done at the Industrial Chemistry laboratory, Federal University of technology, Owerri, using steam distillation method. The oranges were washed with water, cleaned and peeled thinly. In all, 4980 g (4.98 kg) of the thin peels were weighed out using a digital weighing balance. This was cut into small bits and then transferred into a 1000 ml flat bottom flask, with a large amount of water added to cover the peels. The flask was then connected to a Liebig extraction/condenser system. The distillation set up involving a local source of heat (kitchen stove), distillation/condenser apparatus and their tubing is shown in Figure 1. The 1000 ml flat bottom flask was introduced through a connection pipe to the flask containing the peels. The steam generated from the boiling water (as the flask was being heated) extracted the volatile oil which condensed as part of steam into the distillation flask as it passed through a cooling system. The distillate (a mixture of oil and water) was then poured into a separating funnel where the mixture separated into two layers: oil at the upper layer and water at the lower layer. Water was then run off and the oil was collected in a bottle, while any remaining water droplets in the oil were then removed by the addition of anhydrous sodium sulphate.

Characterization of the orange oil

Several tests were carried out to characterize the properties of the orange oil:

Sensory analysis of the orange oil

The sensory analysis was carried out to find out the properties of the oil. It was done using the sense organs of sight, smell and touch.

Physio-chemical analysis of orange oil

Saponification value

Saponification value is defined as the weight of potassium hydroxide expressed in milligrams required to saponify 1 g of fat or oil. 2 g of the oil was weighed into a 200 ml conical flask, to which 50 ml 0.5 M alcoholic KOH was added. This was refluxed for 30 min, followed by the addition of 3 drops of phenolphthalein indicator and was titrated with 0.5 M HCl until the pink coloration disappeared. This was repeated without the oil and the titre value was determined as the blank.

$$\text{Saponification value} = \frac{(t_1 - t_2) \times 28.1}{W}$$

Where, t_1 = blank value, t_2 = sample value and w = weight of sample.

Acid value

2 g of the sample was weighed into a conical flask containing 50 ml of isopropyl alcohol. To the mixture, 3 drops of phenolphthalein indicator was added. This was titrated with 0.1 M NaOH from the burette.

$$\text{Acid value} = \frac{5.61 \times \text{titre value}}{\text{Weight of sample}}$$

Melting point

The oil was solidified at -78°C and part of it was used to fill the capillary tube of the melting point apparatus. The apparatus was connected and the temperature at which the solidified oil melted was noted.

Boiling point

The capillary tube of the boiling point apparatus containing some amount of oil was connected and the temperature at which the oil boiled was noted.

Specific gravity

The specific gravity of the extracted oil was determined using a hydrometer. The oil was poured into a cylindrical long tube, and the hydrometer was immersed into it and allowed to float.

Refractive index

Refractometer was used to determine the refractive index of the extracted oil. One drop of the oil was dropped on the cell compartments of the instrument. The necessary adjustments were made and the result was recorded when the lower part became darker.



Figure 1. Steam distillation set-up.

Solubility test

2 ml of the oil was poured into a test tube, containing 4 ml of water, and the content of the test tube were vigorously shaken to mix them and allowed to stand for 5 min. At the end of the 5th minute, the solubility of the oil in water was noted.

Identification of components of the orange peel extract

This was done using various tests including chromatographic separation analysis and instrumental analytical methods using both ultraviolet-visible and infrared spectroscopy, and carefully interpreting the displayed bands and peaks.

Chromatographic analysis

This was carried out to determine the number of components present in the oil. A drop of the oil was spotted at the origin of the chromatographic paper and dipped into a chromatographic tank containing ethanol and then was allowed to run for about 5 min for components to be separated. The paper was subsequently stained with iodine to identify the separated components. At the end of the separation time, only one component was observed.

Ultraviolet-visible scanning

This was done using ultraviolet-visible spectroscopy method (Jenway, model 6305) (Hirayama, 1967; Robert, 1974). In this method procedure, the instrument was switched on and allowed to run for about 3 min in order to stabilize; 1 ml of the extract was

measured out and made up to 10 ml with n-hexane (1 in 10 dilution); n-hexane served as the blank. The solution of the extract and blank was fed into the system and was scanned for wavelength of maximum absorption.

Infrared analysis of the extract

To identify the functional groups present, the extract was put through infrared spectroscopy method (Brand and Egliton, 1965; James et al., 1982) using Shimadzu model of the instrument.

RESULTS

In all, 30 ml of orange oil was extracted from 4.98 kg of the orange peel, giving only 0.6% extraction success rate. The results of the sensory evaluation showed that the extracted oil was orange in colour, pungent in smell, had orange smell, and was watery in viscosity. The results of physico-chemical tests performed on the extracted oil revealed that while it was insoluble in water, some of its important properties included: a specific gravity of 0.843, melting point of -96°C but with a tendency to thicken at a temperature of -78°C , a boiling point of 176°C , a saponification value of 177.03 and an acid value of 12.34 among others (Table 1). The result of the ultraviolet-visible (Uv-visible) scanning of the extract over various absorption wavelengths is shown in Table 2. Following this scanning, the wavelength of maximum

Table 1. Results of some physico-chemical tests performed on the extract.

Property	Result obtained
Specific gravity	0.843
Refractive index	1.474
Melting point	-96°C, thickens at -78°C
Boiling point	176°C
Solubility	Insoluble in water
Acid value	12.34
Saponification value	177.03

Table 2. Result of ultraviolet-visible (U-V) scanning of the orange peel extract over various wavelength ranges.

Wavelength (nm)	Absorbance
200	0.009
210	0.005
220	0.006
230	0.004
240	0.001
250	0.007
260	0.004
270	0.013
280	0.053
290	0.506
300*	0.749
310	0.611
320	0.426
330	0.310
340	0.226
350	0.173
360	0.142
370	0.103
380	0.075
390	0.049
400	0.040
410	0.034
420	0.031
430	0.015
440	0.028
450	0.025
460	0.024
470	0.023
480	0.022
490	0.021
500	0.021

* Wavelength of maximum absorption (0.749 = 300 nm).

absorption was observed to be 300 nm, and optical density of this maximum absorbance at this wavelength was 0.749. In Figure 2, the separation pattern of the peel

extract on paper chromatogram is shown, while Figure 3 shows the result of the infra-red analysis revealing peaks at different identifiable band positions outside the finger print regions, depicting the major functional groups and bond types that are present: Carbon to carbon double bond (C=C) was identified at 1640 to 1680 cm^{-1} , carbon to hydrogen single bond (C-H) was identified at 2850 to 2960 cm^{-1} and carbon to hydrogen bond with a double bond attachment to the carbon atom (=C-H) was identified at 3100 cm^{-1} band regions respectively, as well as an aromatic component indicated by the stretches around the C-H (alkane) functional group at 2850 to 2960 cm^{-1} band position.

DISCUSSION

The method of extraction (steam distillation) employed in this study was only 0.6% successful. Though small in the consideration, it was however quite considerable when compared to the 0.3 to 0.5% yielded by cold-pressed extraction method of Pultrini et al. (2006). It is equally possible that a stronger and better heat source other than stove used in this study for steam distillation may have improved the extraction success rate. That as much as 4.98 kg of orange peel yielded as little as 30 ml of orange oil meant that very large quantity of this waste would be needed to generate a larger quantity of this important raw material. This surely would have meant a plus for the waste management sector given that very large volumes of orange wastes are always available (particularly during orange season) to generate any amount of this raw material needed at any given time; still there is great need to develop alternative extraction methods that will optimize the generation of this important raw material from as little waste as possible, thereby maximizing the use of orange waste.

Various analyses to ascertain the qualitative characteristics of orange peel extracts and its raw-material potentials revealed some important physical and chemical properties of the extracted oil in terms of specific gravity, refractive index, melting point, boiling point, solubility, acid and saponification values (Table 1). The values obtained for these parameters agreed with those of previous characterization of the orange oil (Kesterson et al., 1971). A particular plant essential oil is characterized by certain physical and chemical properties important for its identification. The physical properties include specific gravity, optical rotation, refraction index, melting point, boiling range, colour and solubility; while the chemical properties include acid value, saponification value and ester content, total alcohol, aldehyde and ketone contents (Macrae et al., 1993).

Sensory and other characterizations revealed that orange essential oil is yellow to orange in color, watery in viscosity, has a sweet, fresh and tangy smell, and a shelf life of approximately 6 months. The ultraviolet-visible

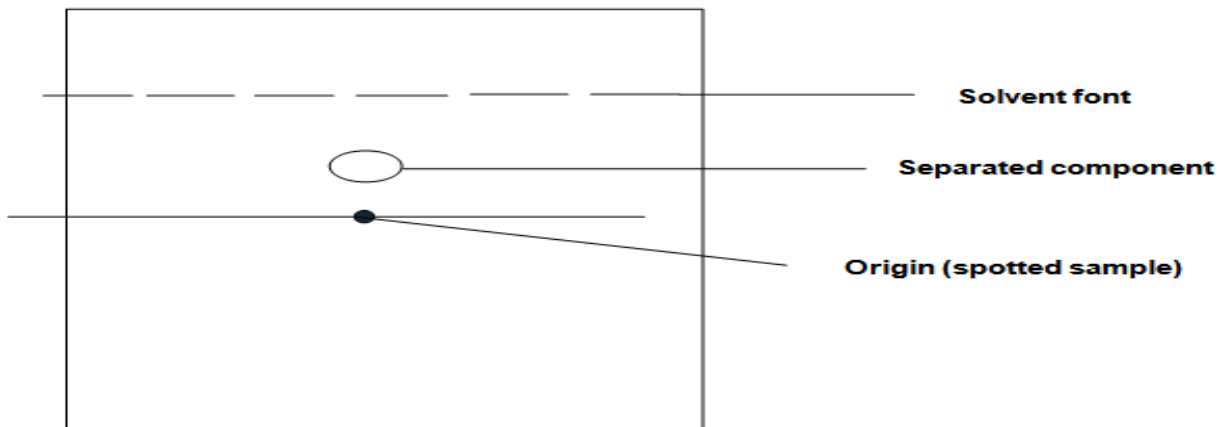


Figure 2. The separation pattern of the orange peel extract using paper chromatogram. Only one prominent band separation was observed

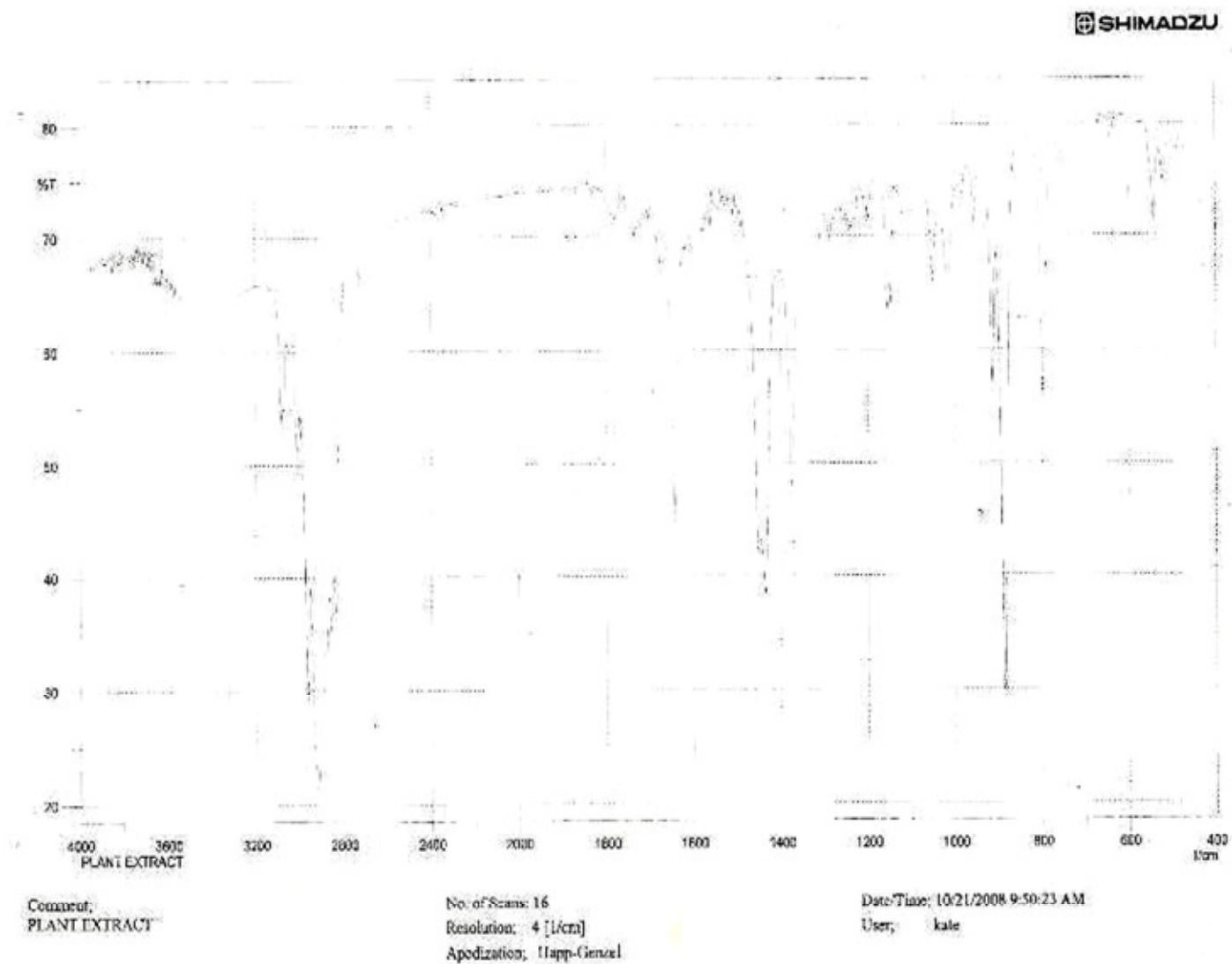


Figure 3. Result of Infrared (IR) analysis showing peaks at different identifiable band positions outside the finger print regions, depicting the major functional groups and bond types present in the orange peel extract.

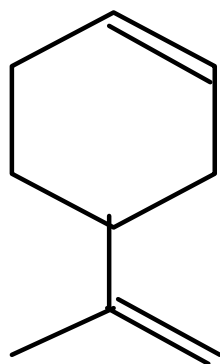


Figure 4. Limonene structure. Source: Vekiari et al. (2002).

spectroscopic scanning of the oil revealed one prominent peak, with the wavelength of this maximum absorbance located at 300 nm (Table 2). Again, when the orange peel extract was subjected to paper chromatographic analysis, only a major single band separation was observed (Figure 2), which is perhaps a confirmation of the result of the uv-visible scan, thus revealing a relatively prominent presence of a particular component of the oil among others. Vekiari et al. (2002) reported that the percentage composition of the main component of the orange essential oil was as high as 90 to 95%. This main component of the Nigerian sweet orange peel extract that consistently displayed this prominent peak is suspected to be limonene (a terpene). This is because the infrared spectroscopic analysis of the peel extract (Figure 3) revealed peaks at different identifiable band positions outside the finger print regions that are consistent with what is found in the structure of limonene in terms of major functional groups and bond types that are present: carbon to carbon double bond (C=C) identified at 1640 to 1680 cm^{-1} , carbon to hydrogen single bond (C-H) identified at 2850 to 2960 cm^{-1} and carbon to hydrogen bond with a double bond attachment to the carbon atom (=C-H) identified at 3100 cm^{-1} band regions, respectively. The C=C, C-H and =C-H indicated the presence of alkene, alkane and alkene-alkane functional groups. The stretches around the C-H (alkane) functional group at 2850 to 2960 cm^{-1} band position indicated the presence of an aromatic component. The presence of these functional groups and their associated bonds, alongside an aromatic structural component, all revealed by infrared analysis of the extract, is consistent with the structure of limonene as shown in Figure 4; thus confirming this to be the substance present in prominent amount in the orange peel oil extracted.

Limonene is a monoterpene found in many citrus oils. Limonene is what gives citrus fruit their familiar aroma. This major constituent of orange peel essential oil has

been reported valuable for the flavoring of foods (Reische et al., 1998) and so may find usefulness where floral-fresh-fruity aromas are required, such as chewing gums, sweets, teas, soft and energy drinks as well as milk products. In the cosmetic industries, orange peel essential oil with characteristic fresh orange-fruity odour impressions may be used in shampoos, soaps, shower gels, body lotions and tooth pastes, while an application of the oils in fine perfumery seems to be interesting top-notes in perfumes and deodorants. Combinations of essential oils are used in every fragranced product such as toothpaste, mouthwash, and room fresheners, paper, printing ink, paint, candles, soap and floor wash. A proper mixture of essential oils in room fresheners could create pleasant odour, improve the moods of people (as shown in psychology), disinfects the air and help indoor plants grow. Essential oils are widely used for flavouring baked goods, soft drinks, liquors, tobacco, sauces, gravies, salad dressings and many such food products. It could also be used in food preservation due to high percentage of well-known antimicrobial compounds reportedly present in limonene (Caccioni et al., 1998; Williams et al., 1998; Filipowicz et al., 2003; Bajpai et al., 2007). These oils have also been used in aromatherapy, which is the therapeutic use of fragrances to cure, mitigate or prevent diseases and infections by means of inhalation only. It also has wide applications as insect repellants and insecticides. Most essential oils have been termed as safe natural herbicides and insecticides.

Apart from this major component (Limonene), other numerous industrially and domestically useful substances, belonging essentially to one of the three chemical groupings- the terpenes, benzenoid compounds and compounds containing nitrogen or sulphur (Kesterson et al., 1971), are said to be present in orange essential oil. These include such industrially important large chemical groups as the terpenoids, alcohols, aldehydes, oxides, acids, ketones, esters, α , β -diacylacroleins and paraffin waxes (Kesterson et al., 1971). Other main chemical components are α -pinene, sabinene, myrcene, limonene, linalool and citronellal, neral and geranial (Pultrini et al., 2006). The exact chemical composition of the oils is characteristic of a given species, and the different constituents are synthesized by the plant during its normal growth. Some plant oils are rich in only one compound and are invariably used as a commercial source of that particular compound. For example, lemon grass oil contains 50 to 70% of citral, while citrus oil contains 90% of limonene (Stashenko et al., 1996; Vekiari et al., 2002). Sweet orange oil produced by glands inside the rind (located mainly within the peel area) of an orange fruit contains over 90% d-limonene and is therefore often used in place of pure d-limonene that can be further extracted from the oil by distillation (Williams et al., 1999). In this study, only the presence of limonene was prominently revealed as a major component as noted previously (Stashenko et al., 1996;

Vekiari et al., 2002). The inability to detect these other possible constituents could be due to their insignificant presence in this citrus specie, or was as a result of the lack of access to certain desired analytical chemicals and equipments as well as other limitations and inhibitions suffered in the course of this work. For instance, the needed standard salts and/or solutions of these substances that would have enabled concretely the fingerprint of their actual presence in the Nigerian sweet orange peel extracts could not be sourced, just as was the case with services of a gas liquid chromatographic system, which we believed could have offered a better resolution of the components of the extract than the paper chromatography used. All these make the Nigerian sweet orange peel a candidate for further evaluation in order to harness all the industrial raw material potentials inherent in it.

Given that the peels of Nigerian sweet orange (*C. sinensis*) have been proven to be a good source of essential oil, which in addition to other substances, has great preponderance of limonene- a chemical with very wide industrial applications, and given that this substance is extractable from ever superabundantly available orange peel wastes needing urgent disposal to avert health hazards from environmental disruptions/degradations, it was concluded that the extraction of essential oil from this source will not only provide a cheap source of this plenipotential industrial raw material but also a complimentary method for the management of wastes generated from orange fruit consumption. In this way, these wastes with great potential for environmental pollution and degradation are converted into veritable source of industrial raw material with great potential for stimulating industrial and economic growth, employment generation and national wealth. Added to these are the public health impacts of securing safer and healthier environment from this indirect waste management option.

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