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Extraction of Fragrance from Tangerine (Citrus Reticulata)

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Abstract- Extraction of essential oils/fragrance from dried tangerine peels by wet-steam distillation, dry-steam distillation and solvent extraction were investigated. Results indicated that wet steam distillation method gave the highest oil yield of 0.27% using simple weight of 200gm while the solvent extraction method gave the lowest yield of 0.21, 0.22 and 0.21 percent respectively using sample weight of 32gm. Analysis of the oil extracted from both wet and dry-steam distillation shows that tangerine oil contains high percentage of limonene (90.2% as terpene).

Keywords- *Citrus reticulata, distillation, Extraction, Fragrance, limonene.*

I. INTRODUCTION

Personal-care products are designated to satisfy certain consumer needs. Some of these ingredients perform specific physical functions, such as skin cleansing or hair conditioning. Others play more objective roles in helping the product achieve consumer satisfaction. Tangerine fruit is grown in Nigeria but it is native to china. It was brought to Europe in 1805. In 1845, it was brought to America and remarked tangerine. Fragrance is an important part of cosmetic formulations because of the psychological effects it can have. Branched aldehydes and alcohols are important flavor compounds in many food products (Salem, 2003). Various studies have showed that the tangerine - like smell was also suggested to be mainly based on carbonyl compounds, such as ∝-sinensal, β-sinensal, geranial, Citronella and decanal (Darjazi et al 2009). Literature survey reveals that extensive studies on the citrus peel oil have been carried out (Ravi et al., 2010).Chemically speaking, a fragrance is best described as a complex mixture of ingredients specially blended to produce specific scent. The peel of citrus reticulata has been used to kill mosquito, larvae and mites. (Nwaiko., 1992).

A more systematic, scientific approach which described fragrance by top, middle and bottom notes has been developed. The top note made of the most volatile material is the first element smelled when a fragrance is applied. Citrus notes are common top notes ingredients. The middle note or body of fragrance is composed of somewhat less volatile material. These components come more into play after the top notes have dissipated. Floral scents such as violet or lube rosé are typical middle notes. The bottom or base note is made from the least volatile materials like musk scents, woods and vanillins. The long lasting portion of the fragrance. The chemicals responsible for the aromatic character of a fragrance can be divided into three broad categories: lerpenoids, aliphatics and benezenoid. Essential oils are made up of different volatile compounds and aromatic oily liquid obtained from different plant parts (Amal et al, 2010). The study of individual components of different essential oil has shown that many terpenes containing oils possess antiradical and antioxidant activity (Misharina et al, 2010).Relative percentages of the identified components depend on the plant part studied. However, it should be kept in mind that the isolation method has an effect on some of the component of oil Darjazi B (2011c).The quality of oil may be influenced by the amount of oxygenated compounds present in the honey.(Alissandrakis etal.,2003).These influence may have consequence in determining the yield of some agricultural products like tangerine (Kite et al.,1991).

Since fragrance has such a strong influence on consumer perception, formulators must select them with care. The process of formulation is concerned with performance, stability, cost and safety. Both delicately balance creative artistry with technical expertise. The tangerine is a citrus fruit in the mandarin Orange family. Although similar in fragrance and taste to the standard orange, the tangerine is considerably smaller and can be peeled much easier. The tangerine has a light bright and sweetly scent that is used in various household products.

To create successful fragrance, perfumers require certain key information from the product familiar, typically relayed in the form of "fragrance brief. Particulars regarding cost packaging, stability requirements and project completion time constraints are also important consideration to be addressed. Since creating a fragrance is very subjective, several fragrance houses may be given the same brief and asked to submit sample.

The aim of this work is to identify optimally viable techniques of extracting essential oils from local (tangerine) considered for fragrance production.

II. MATERIALS AND METHODS

Fresh tangerine was collected in 2011 season from a private orchard in Umuahia, Abia state South-Eastern Nigeria, Peeled by hand and dried in an oven at 45℃ until constant was obtained. The samples were ground into powders of 0.2mm in particle size.Hydrodistillation (wet and dry steam) by using Dean-Stark assembly (Scatter, 1989). The distillate was removed and separated from water by using a separating funnel. The distillate was dried over anhydrous sodium sulphate and stored in a refrigerator for further analysis. Soxhlet Extractor was used in the in the case of solvent extraction of oil from the same tangerine peel. Solvent used are heptane, acetone, hexane and methyl carbonyl. The method of extraction was the same for the four solvents.

A. Solubility Measurment

The solubility of the essential oil was determined with a known weight of th oil in various % ethanol solution.1m of oil was introduced into a 15ml glass cylinder .Ethanol of required concentration was added with the aid of 10ml pipette in small amounts into the glass. The glass cylinder was thoroughly shaken after each addition at a point when the clear solution was obtained. The volume of ethanol was noted. Addition of the ethanol in small increments was continued until a total of 10ml has been added. At this stage the opalescence or cloudiness of the resulting solution was observed.

B. Limonene Determination

The hydroxylamine method was used. 27.5g of recrystallized hydroxylamine hydrochloride was dissolved in 30ml of distilled water and warmed to 65℃ on a steam bath in order to yield a clear solution. This prepared hydroxylamine hydrochloride solution was diluted in 1 litre volumetric flask with 95% isopropyl alcohol. The PH of this solution was then adjusted to 3.5 with 0.5 sodium hydroxide solution, 35ml of the hydroxylamine hydrochloride solution was added to 1gm of sample oil and stirred for 15minutes at room temperature. The Ph of this mixture was adjusted to 3.5 with the standard 0.5sodium hydroxide solution and the volume of sodium hydroxide solution used were noted.

C. Acid Value Measurement

A combination of 1g of the sample oil and equal volumes of 95% ethanol and ether was titrated against 0.5N sodium hydroxide .The volume of sodium hydroxide solution used was noted.

III. RESULTS AND DISCUSSION

Table 1: Percentage Yield Using Wet And Dry Steam Distillation Method. Weight of sample (Tangerine peels) =200gm.

No	Extraction Method	Oil yield (g)	% Oil	Time
1	Wet-Steam Distillation	0.54	0.27	2hrs
2	Dry-Steam Distillation	0.46	0.23	4hrs

The result obtained from the experiment in table 1 above showed that wet steam distillation extraction method of oil gave higher yield of oil than the dry steam distillation.Tangerine peel oil is collected as quickly as possible so as to obtain a better yield and to reduce the time of contact between oil and peel to reduce the oxidation of terpene content.

Table 2: Percentage Yield Using Solvent Extraction. Weight of sample (Tangerine)=32gm.

No	Solvent used	Weight of Yield	%Extract	Time
1	Heptane	0.066	0.21	4hrs
2	Acetone	0.070	0.22	5hrs
3	Hexane	0.068	0.25	3.5hrs

From the result obtained from the experiment in table 2 showed that acetone has the highest extract than hexane and heptane. This consequently implies that acetone is a better solvent in extraction of Tangerine oil except for its shortcomings such as its chief impurity of one percent of water and he total extraction time.

Table 3: Solubility In 95% Ethanol

No	Sample oil	Volume of ethanol	Observation
1	Wet-steam distillation	3ml and 10ml	Clearly soluble, No cloudiness
2	Dry-steam distillation	3ml and 10ml	Clearly soluble, No cloudiness.

The solubility measurement using ethanol solution showed that both wet and dry steam distillation were very soluble in ethanol. This is due to the absence of higher boiling components in the oil extracted.

Table 4: Percentage Limonene Value

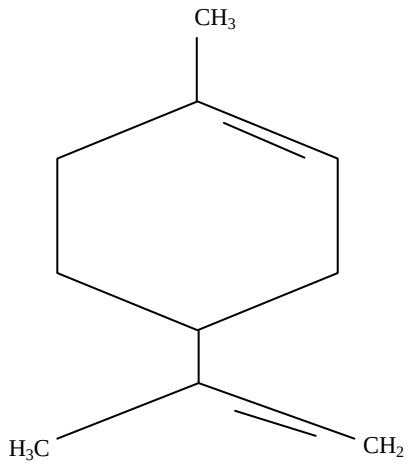
No	Sample Oil	Volume of Ethanol
1	Wet-steam distillation	90.2
2	Dry-steam distillation	90.2

The percentage limonene value from tangerine peel oil of 90.2% using both wet and dry steam distillation agrees with result of macra, 1993 that gave the range of limonene in tangerine peel oil to be between 80-95%. This also confirms the presence of limonene as the dominant fraction in tangerine oil.

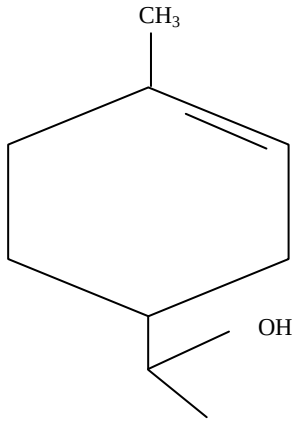
Citrus fruit quality and extractive methods generally determines the yield of terpenes got from tangerine. Generally, the oil field from both wet and dry steam distillation was low but more acceptable compared to other method. The low oil content of the tangerine peels was due to the volatility of the essential oil. Maintenance of good sanitary practices and limited holding time is important for centralizing microbial growth in the oil recovery process (Murdock et al, 1976). Result obtained showed that the aldehyde content of the oil decreases as the amount of aqueous phase in contact with the

oil during processing increases. Low quality oils lack high boiling components like citral (Guenther, 1969).

-d- limonene is a major constituent of Tangerine oil which terpineol is an important constitution of the lilac family.



d-Limonene



∝ -terpineol

Most essential oils generally contain a wide variety of mono-terpenes such as – pinene, carvone, borneol. A few oils are rich in one compound only, and are invariably used as a commercial source of these compounds. E.g. lemongrass oil

Contains 75%-80% of citral while citrus oils (Tangerine) contain 90% of (+) - limonene. Progress has been made rapidly during the past two decades due to better purification and characterization techniques, particularly chromatographic and spectral methods. Citrus fruits quality and extraction methods generally determines the yield of terpenes got in a particular citrus: Production of volatiles from green fruits was lower in quantity and variety from yellow fruits than from lemons, and was much greater from both green and yellow treated with ethylene (1:1000 parts). The principal components detected were – terpinene, terpineole, d-limonene and citral from (citrus-lemon).Limonene has a weak citrus-like aroma (Sawamura et al., 2004).

A. Concrete and Absolutes

Because of some draw backs of the distillation process such as the one used to extract essential oil, other methods can be used such as volatile solvent extraction The plant feedstock is submerged in a solvent (typically hexane) for several hours. The solvent is then separated from the plant feedstock and removed by vacuum distillation .The resulting solution is known as "concretes", which contains essential oils and some waxes, glycerides and some similar materials. Volatile solvent extraction is typically used to get flower oils that cannot be isolated through steam or water distillation. Concretes can be further processed to create "absolutes" by removing the waxes, glycerides and other materials. Concretes and absolutes include jasmine, rose oil, ylangylang oil, orange blossom (neroli), lavender, naracissus and oakmoss.

B. Resinoids

In addition to essential oils, certain plants also produce resins that can be extracted to produce resinoids. Since these materials have low volatility, they are used as fragrance fixatives. Fixatives help a fragrance last longer because they reduce the volatility of other perfume ingredients. Important resin types include gums which are resinous materials usually obtained by boiling the backs twigs or leaves of trees and shrubs in water. Consequently, the most important gums are water- insoluble. Example includes labdanum, olibanum, opopanaux and myrrh, which is used in soap fragrance.

IV. CONCLUSION

In general, the wet-steam distillation gave an oil yield of 0.27%, Dry-steam distillation gave a yield of 0.23% while solvent extraction method gave 0.21, 0.22 and 0.21 when non-polar solvent such as heptanes, acetone and hexane is used respectively. On the analysis of terpene composition, steam distillation gave a yield of 90.2% limonene, showing limonene as the dominating fraction of the oil composition. The optimum oil yield was obtained with wet-steam distillation while the lowest oil yield was obtained using solvent extraction method. It was equally shown that the oil is very soluble in ethanol. The wet-steam distillation method is recommended based on the percentage oil yield, especially if the oil extracted is to be used as a raw material for soft drink industries. Where the oil is to be used in cosmetic industries, the solvent extracted method could be preferable due to high percentage of chlorophyll which could be used as a colorant such as in soaps and perfumes.

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