EUROPEAN JOURNAL OF BIOMEDICAL AND PHARMACEUTICAL SCIENCES
http://www.ejbps.com

ISSN 2349-8870
Volume: 10
Issue: 6
187-192
Year: 2023

EXTRACTION OF ESSENTIAL OIL FROM ORANGE PEEL

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Article Received on 27/3/2023 **Article Revised on 17/4/2023** **Article Accepted on 07/5/2023**

ABSTRACT
Presently, in orange peels are discarded as wastes after consumption of edible parts of orange fruits. To get the highest quality and quantity of orange peel oil, it is necessary to know the suitable methods for drying and the appropriate form of the peel, whether in the form of pieces, grated or powder. Essential oils are highly concentrated substances, which are extracted from fruit peels, flowers, leaves, stems, roots and seeds. These oils are often used for their flavor and their therapeutic or odiferous properties, in wide selection products such as foods, pharmaceuticals, medicines, perfumery industry and cosmetics. The main objectives of this research is to extract essential oil from orange (*citrus sinensis*) peels by two methods: hydro distillation and solvent extraction, to study the effect of extraction conditions (solvent type, extraction time, and temperature) on oil yield and compare the results of the two methods, the resulting essential oil was analyzed its physical and chemical properties. From the results obtained, it was observed that the orange peels could give the maximum yields of essential oil to be 3 % and 2 % when hydro distillation and solvent extraction were employed, respectively, and that was an indication that the highest yield of essential oil was given by hydro distillation among the methods considered. The physical and chemical properties values obtained from the characterization of the oil revealed that it could be used in different process industries for production of other valuable products.

KEYWORDS: Essential oil, Hydro distillation, Effect of temperature.

INTRODUCTION
Essential oils are used in a wide variety of consumer goods such as detergents, soaps, toilet products, cosmetics, pharmaceuticals, perfumes, confectionery food products, soft drinks, distilled alcoholic beverages (hard drinks) and insecticides. The world production and consumption of essential oils and perfumes are increasing very fast. Production technology is an essential element to improve the overall yield and quality of essential oil. The traditional technologies pertaining to essential oil processing are of great significance and are still being used in many parts of the globe. Water distillation, water and steam distillation, steam distillation, cohobation, maceration and enfleurage are the most traditional and commonly used methods. Maceration is adaptable when oil yield from distillation is poor. Distillation methods are good for powdered almonds, Rose petals and rose blossoms, whereas solvent extraction is suitable for expensive, delicate and thermally unstable materials like jasmine, tuberose, and hyacinth. Essential oils are generally derived from one or more plant parts, such as flowers (e.g. rose, jasmine, carnation, clove, mimosa, rosemary, lavander), leaves (e.g. mint, Ocimum spp., lemongrass, jamrosa), leaves and stems (e.g. geranium, patchouli, petitgrain, verbena, cinnamon), bark (e.g. cinnamon, cassia, canella), wood (e.g. cedar, sandal, pine), roots (e.g. angelica, sassafras, vetiver, saussurea, valerian), seeds (e.g fennel, coriander, caraway, dill, nutmeg), fruits (bergamot, orange, lemon, juniper), rhizomes (e.g. ginger, calamus, curcuma, orris) and gums or oleoresin exudations (e.g. balsam of Peru, Myroxylon balsamum, storax, myrrh, benzoin).[3] Citrus fruits belong to six genera (Fortunella, Eremocitrus, Clymendia, Poncirus, Microcitrus, and Citrus), which are native to the tropical and sub-tropical regions of Asia, but the major commercial fruits belong to genus Citrus. The genus citrus includes several important fruits such as oranges, mandarins, lime, lemons and grape fruits. Essential oils are mixtures of over a hundred compounds that can be approximated into three fractions: terpene hydrocarbons, oxygenated compounds and nonvolatile compounds. The terpene fraction can constitute from 50 to more than 95% of the oil.[4] Essential oil from citrus is a large type of natural flavours and fragrances which is popularly used in food industries, daily chemical products and health care field. Orange peel oil has been chosen for extraction because it provides a great potential for further commercial form. Peel of citrus fruit

has numerous glands that contain oil that is typically recovered as major by product. Each citrus fruit has its own characteristic set of compounds that comprise the oil and that are responsible for its flavor and aroma to products such ascarbonated drinks, ice creams, cakes, air-fresheners and perfumes. Recently developed extraction methods like supercritical fluid extraction, microwave assisted extraction and Sox let method has been used for oil extraction, know a day they also use solvent extraction, water distillation and steam distillation methods. In this research, solvent extraction, steam distillation, and water distillation methods use to extract oil from orange peel and compare those terms of costing, yielding and time and reproducingbetter natural aroma of orange essential oil.

Research objectives

General objectives

To extracted essential oil from orange peel using different methods and effect of solvents, time, temperature to maximize yield

Specific 0bjectives

- To extract essential oil from orange peel samples.
- To determine the optimum time and temperature to extract orange peel essential oil for the highest yield.
- To conduct the physicochemical characterization of essential oil produced.
- To compare the best extraction methods depend on yield.

MATERIALS AND METHODS

Chemicals

Analytical grade chemicals that were used in the study include N-hexane, petroleum, methanol, acetone, hydrochloric acid, potassium hydroxide, nitric acid, sodium hydroxide, distilled water, and phenolphthalein indicator.

Material/Instruments

All glassware's used in the extraction of essential oil were washed with dilute nitric acid and rinsed in distilled water, dried in hot oven prior to use, to avoid any unwanted reaction during the extraction process. The materials/instruments used for this work were round bottom flask, Basket heater, thermometer, measuring cylinder, separating funnel, electronics balance, grinder, Knife.

Collection and identification of orange peel

Orange peel was collected from Anand Nagar market Bhopal. Orange peel was botanically identified using the standard morphological characteristic features. Orange peels were collected in plastic bags and transported to the laboratory of chemistry for extraction and characterization of oil.

Preparation of orange peel powder

The collect sample of orange peels is cleaned and pith is manually separated from the outer colored part of the peels. That is because of the reason that the majority of the oil in oil sac present in them. After the orange peels were obtained, they were washed, cleaned with water and chopped, placed on blotting paper, spread out and dried under shade at room temperature for 5 days. The shade dried orange peels was grinded to give consistent and fine powder (Figure 1) using electrical grinder. The powdered orange peels was then stored in well labeled airtight container at ambient temperature and protected from sunlight for further use.

Figure 1: Prepared powder sample.

Extraction of essential oil by hydro distillation

Water or hydro distillation is one of the oldest and easiest methods. It consists of distillation flask, Basket heater, horizontal condenser and a conical flask. About 150 g of the orange peel powder was weighed using digital weighing balance and then transferred into a round bottom flask with large amount of water added to cover the peels. The flask was connected to the still column which was connected to the condenser. Heat is supplied to the distillation unit by temperature controlled basket heater. The experiment is carried out at different Temperature and. time period. The distillate is collected in a conical flask. This distillate has two layers, one dense layer and other less dense layer. This is then separated usinga separating funnel.[5]

Solvent extraction

Essential oil extraction from orange peels was done using the Soxhlet method. The orange peels were pureed using a blender. A round bottom flask was washed, oven dried, and cooled in a desiccator. To carry out this procedure, the ground peels were sieved using a standard 0.6 mm particle size sieve. A dried mass of 10 g of the orange peel powder was weighed, and the weight recorded. The weighed sample was dropped in the Soxhlet extractor apparatus. The extraction was carried out using normal hexane, methanol, and petroleum as the extraction solvent. In the Soxhlet apparatus, the solvent in the round bottom flask was heated from the heating mantle to become evaporated and got condensed down through the sample where it was able to extract the oil along, thereby, giving a mixture of oil and solvent, which was later separated.[5]

Separation of essential oil

When the mixture is poured into a separatory funnel, the oil and water separate into two distinct layers. Since water is denser than oil, it is collected at the bottom of the funnel. After this the funnel tap is opened and the liquid at the bottom of the funnel is transferred into a container. The oil and solvent (n- hexane, methanol….) mixture was separated using rotary evaporator and the separation was carried out by principle of rotary evaporator.

Calculation of oil yield

The yield of the oil extracted using each of the two methods of extraction was calculated using equation.

% yield = Weight of oil extracted/ Weight of sample used x100

Characterization of the essential oil

The essential oil extracted from orange peels at the optimum processing conditions (the highest yield) was characterized by determining the physicochemical properties. Outlined below are the tests carried out on the oil [6].

Physical characteristics of extracted essential oil

Sensory analysis of the essential oil

Sensory analysis was carried out on the oil to determine its physical properties. This involved sense of sight, smell and touch.

Determination of solubility of the essential oil in water

A few drops of the oil was added to a test tube containing little amount of water. The test tube was stirred thoroughly with a stirring rod. Two separate phases were observed. The insolubility of the oil in water was inferred from that operation.

Determination of density of the oil

The density of the oil extracted was determined by weighing an empty beaker and recording its value. Thereafter, essential oil was poured into the beaker and the weight was taken. The density of the oil was thus calculated using equation.

Density = Weight of oil sample/ Volume of oil sample x100

Determination of specific gravity of orange peel essential oil

All experiments were carried out in triplicates, and average values taken. A clean and dry bottle was weighed using a weighing balance. Distilled water was poured into the bottle and weighed. In the same manner, the same volume of oil was poured into the same bottle and weighed. The specific gravity was calculated as the ratio of weight of oil to that of water[5] as given in equation.

Oil specific gravity = Weight of particular volume of oil extract/ Weight of equal volume of water x100

Chemical characteristics of extracted essential oil

Determination of saponification value of the essential oil

Saponification value, being the weight of potassium hydroxide expressed in milligrams that is required to saponify 1 g of oil was also determined in this work. To carry out this, 2 g of the oil was weighed into a 200-ml conical flask into to which 50 ml of 0.5 M of KOH was added. Then followed by addition of 3 drops of phenolphthalein indicator and it was titrated against 0.5 M HCl until pink color disappeared that observed during phenolphthalein indicator added to the mixture of oil sample and KOH. This procedure was repeated without the oil and the titre value was determined from the blank value.[13] The saponification value was calculated using Equation.

Saponification value = V2−V1/ w x28.1

Where, V1 is the blank titrate value, V2 is the sample titrate value and W is the weight of the sample.

Determination of acid value of orange peel essential oil

To determine the acid value of the extracted oil, 2 g of the oil sample was weighed into a conical flask containing 50 ml of ethanol. Three drops of phenolphthalein indicator were added to the mixture. The resulting mixture was titrated against 0.1 M NaOH[5] and consistently shaken until a dark pink color was observed. The acid value was calculated using Equation 5. The volume of the 0.1 M NaOH was noted.

Acidic value = 56.1x V x N/ weight of sample

Determination of ester value of the oil

Ester value, which is defined as the number of milligrams of potassium hydroxide required to saponify the fatty acid esters in one gram of the oil, was also determined for the oil extracted in this work. It was obtained as the difference between the saponification value and the acid value of the oil[5] as given in Equation.

Ester value= saponification value- acid value

RESULT AND DISCUSSION

The effects of extraction temperature and extraction time on essential oil yield from orange peels were investigated. Volume of essential oil obtained from orange peel was different for different temperatures and at a particular temperature for the different time of heating. From Table 1&2 for the essential oil extraction, in the range of 90-100^{o}C for extraction temperature and 30-180 min, the observed maximum essential oil yield was 2 ml at extraction temperature of 90^{o}C and extraction time of 180 min. Similarly the observed maximum essential oil yield was 4.5 ml at extraction temperature of 100^{o}C and extraction time of 180 min. Increase in extraction temperature and time leads to a corresponding increase in essential oil yield, the oil will eventually break out of the peel matrix and thus be extracted. Hydro distillation extraction method of obtaining essential from orange peels was carried out at a temperature of 90^{o}C in this work, and the results

obtained from this were as shown in Figure 2. It can be seen from the figure that at no oil was obtained from the extraction from the initial time up till about 60 min. From the graph it was observed that volume of Essential Oil obtained from orange peels is almost increasing linearly with time of heating at a particular temperature (90^0C). It was also observed from the Figure 2 that the yield of oil obtained when the extraction time was 180 min was 2 ml/150 g orange peel powder. Carrying out the same water distillation procedure of the essential oil extraction at a temperature of 100^oC gave the results that are shown in Figure 3. From the results given in Figure 3, it was noticed that oil was produced by the extraction at the temperature of 100^oC from the initial time of the process, and, even, the amount of oil given was observed to keep on increasing with increase in extraction time. For this process, which was carried out at 100^o C, the yield of oil obtained when the extraction time was 180 min was obtained to be 4.5 ml / 150 g peel. Analyzing the results given in Figures 2 and 3, it can be seen that temperature is one of the factors affecting extraction process because the amount of oil obtained at the temperature of 90^oC was found to be different from the amount given when the extraction temperature was 100o C. It has also been observed that the high temperature could favor extraction process because the oil given by the extraction process carried out at 100^oC was found to be higher than that given when the temperature was set to 90^oC.

Solvent extraction

The effects of solvent type time on essential oil yield from orange peels were investigated. Solvent extraction method of obtaining essential oil from orange peels was carried out using n-hexane, methanol, and petroleum ether solvent at temperature 60 and 80 degree Celsius for 3 hour in this work, and the results obtained from this were as shown in Table 3. As Table 3 show high amount of essential oil from orange peel was extracted using n-hexane, and methanol and petroleum ether respectively. The results given in Table 3 shows that the variations in the factors chosen as the independent variables of the process were having effects on the yield of oil obtained from the extraction of orange peels. The change in solvent, time and temperature was found to affect the quantity of oil extracted. As result show when the temperature of the experiment work increase quantity of oil extracted also increase and also when the time of the experiment work increase quantity of oil extracted also increase. Therefore oil yield and time also oil yield and temperature are directly proportional to each other.

Characterization of the essential oil

The extracted essential oil was analyzed to determine its physical and chemical properties, and given in Tables 4, and 4 are the results obtained from the analyses for the physical and the chemical properties respectively.

Physical characterization of extracted essential oil

Moreover, the extracted essential oil was analyzed to determine its physical properties, and given in Table 4 are the results obtained from the analyses for the physical properties. As can be seen from the Table-4, the oil was obtained to have virtually yellow colour with tangy smell, and it was observed to be insoluble in water. As can be seen from the Table-4 the specific gravity of the essential oil was 0.843. The specific gravity determines the weight of the essential oil. It is also important in determining the quality and purity of essential oil. Most of the essential oils have specific gravity ranging from 0.696–1.88. Specific gravity values of oils are less than 1 for most of the oils except few containing oxygenated aromatic compounds. The extracted essential oil has a specific density less than 1 which implies that it is lighter than water and consequently will be insoluble in water. The density of the oil was estimated to be 0.86 g/cm3.This is in agreement with the standard value of essential oil from orange peel waste.

Chemical characterization of essential oil from orange peel

The values obtained for the chemical properties of extracted essential oil are given in Table-5. The acid value of the essential oil was 4.2 mg KOH/g. Essential oils are concentrated and contain several volatile aroma compounds which are majorly free fatty acids. Free fatty acids are considered as degrading in oils because they are responsible for oil rancidity. Oils with low acidity are considered as neutralized and safe for making skin care products as high acidity of oils may beharmful for skin.[7] The low acid value of the extracted essential oil indicates that the oil has excellent storage life. The saponification value of the essential oil was 44.96 mg KOH/g. Saponification value is an indicator of the average molecular weight and hence chain length. It is inversely proportional to the molecular weight of the oil.[8] High saponification values of oils are due to the predominantly high proportion of shorter carbon chain lengths of fatty acids.[9] Low molecular weight (short to medium chain) fatty acids have more glyceride molecules per gram of fat than high molecular weight acids. The ester value of orange essential oil was 40.76 mg/g. The experimental values of the orange peel oil acid value, saponification value and ester value are between the standard values.

CONCLUSION

Study on extraction of oil by hydro distillation and solvent extraction methods using different solvents like water, n-hexane, methanol, petroleum ether were done. From the experimental results obtained from the extraction and the characterization of the essential oil from orange peels have shown that the maximum yield of essential oil obtained from the orange peels used in this work were 3 % and 2 % when the methods employed were hydro distillation and solvent extraction respectively, indicating that water distillation was able to give the highest yield of essential oil among the methods considered. Hydro distillation gave the more yield was

achieved at the temperature (100°C) than that of temperature 90^{0}C this implies; when the temperature increase the extracted oil yield was increase, and also the time and the yield of extracted essential oil from orange peel was directly proportional to each other. It was shown by the analysis of variance of time and temperature are very significant factors on the yield of the oil extracted in the process. The physical and chemical properties values obtained from the characterization of the oil revealed that it could be used in different process industries for production of other valuable products such as in pharmaceutical, cosmetic, agricultural and bioactivity. In solvent extraction method, hexane gives slightly better oil yield when compared to methanol and petroleum ether at 60˚ C and 80^{0}C and solvent to solid ratio 6:1 (v/w) for 180 min. Produced waste amounts increase with urbanization, correspondingly consumption growth. Waste recovery is an important and urgent issue. Moreover, since fruit industry grows day by day, generated fruit wastes increase. If the wastes are effectively managed to produce value-added products economic benefits along with reduced environmental problems can be achieved. Orange juice is one of the big juice sectors. From these sectors, large amount of wastes especially orange peels are discarded. However, orange peel wastes can effectively be utilized for the economic production of value-added products such as pectin. Our results revealed that it was possible to produce value-added products from the orange peel wastes such as essential oil.

Table1: Effect of temperature.

Temperature(°C)	Time (min)	Solvent	Oil extracted (ml)	(%) yield
90	180	Water	2	1.33
100	180	Water	4.5	3

Table 2: Effect of time.

Time (min)	Temperature(°C)	Quantity of oil Extracted (ml)	Percentage yield value (%)
30	100	0.7	0.47
60	100	2.1	1.4
90	100	2.5	1.6
120	100	3.3	2.2
150	100	3.8	2.53
180	100	4.5	3

Table 3: Experimental Data and Response obtained from the solvent extraction process.

Solvent	Weight of peels (g)	Volume of solvent (ml)	Temp (°c)	Time (min)	Quantity of oil Extracted (ml)	Percentage yieldvalue (%)
N-Hexane	50	300	60	180	2.3	1.53
	50	300	80	180	3	2
Methanol	50	300	60	180	1.5	1
	50	300	80	180	2	1.33
PetroleumEther	50	300	60	180	1.2	0.8
	50	300	60	180	1.8	1.2

Table 4: Physical properties of essential oil from orange peel

Parameter	Value
Color	Yellow to orange
Odor	Fresh and tangy smell
Solubility	Insoluble in water
Density	0.86
Specific gravity	0.843

Table 5: Chemical properties of essential oil from orange peel

Parameter	Value
Acid value	4.2
Saponification value	44.96
Ester value	40.76

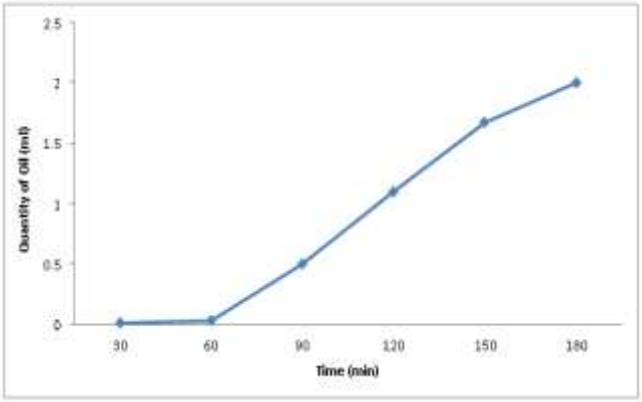

Figure 2: Water distillation extraction process at 90°C.

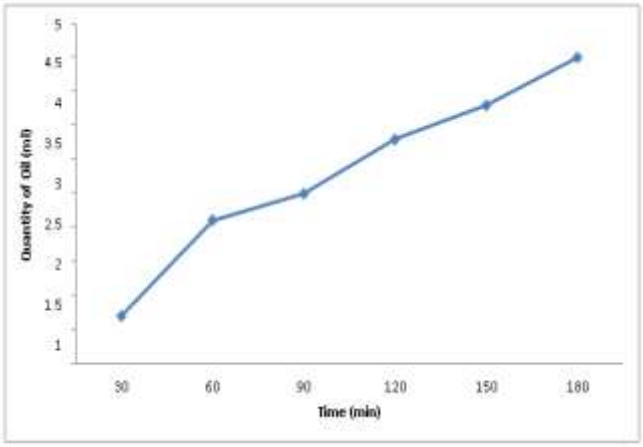

Figure 3: Water distillation extraction process at 100°C.

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