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Evaluation Of Hydrodistillation For The Extraction Of Orange Peel Essential Oil: Impact Of Parameters On Yield And Quality

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Abstract

Objective: To evaluate hydrodistillation as a method for extracting essential oil from orange peel (*Citrus sinensis*) and to investigate the impact of key process parameters — temperature, extraction time, peel size, and water-to-peel ratio on the yield and quality of the extracted oil.

Study Design: Experimental laboratory study.

Place of Study: Department of Chemistry, The Superior University, Lahore, and Government College University, Lahore, Pakistan.

Methodology: Fresh *Citrus sinensis* peels (50 g) were subjected to hydrodistillation using a Clevenger-type apparatus. Extraction was performed at two temperatures (85°C and 100°C) over 200 minutes. The distillate was separated using n-hexane. Oil yield was calculated gravimetrically. Physical characterization (colour, odour, viscosity, solubility) was performed by sensory evaluation. Chemical composition was analyzed by UV-Visible spectroscopy (200–700 nm).

Results: At 85°C, the distillate yield was 12 mL/50 g (24%) after 200 minutes; no distillate was collected in the initial 80 minutes. At 100°C, the distillate yield was 15 mL/50 g (30%), with extraction commencing immediately. The essential oil yield after n-hexane separation was 5.5 mL/50 g (11%). The extracted oil was pale yellow in colour, with a fresh-to-tangy odour, low viscosity, and was soluble in organic solvents but insoluble in water. UV-Vis spectroscopy identified absorption peaks at 203, 208, 210, 234, 268, 278, 340, and 371 nm, corresponding to limonene, β-pinene, α-pinene, linalool, citral, coumarins, geranial, and carotenoids respectively.

Conclusion: Hydrodistillation successfully extracted essential oil from orange peel with 11% yield. Temperature (100°C), extraction time (200 minutes), fine peel grinding, and optimal water-to-peel ratio were identified as critical parameters for maximizing yield. The extracted oil exhibited a limonene-rich profile consistent with literature, confirming hydrodistillation as a viable, cost-effective, and eco-friendly method for orange peel essential oil extraction.

Introduction

Oil is defined as the chemical substance which exists as a liquid at atmospheric temperature and having the characteristics of both hydrophobic water repelling and lipophilic fat loving. Oil is slippery and insoluble in water but soluble in the organic solvents. The oils are characterized with high carbon and hydrogen content. The oils are flammable and surface active [1]. The oils are of many types, which are based on the origin from which they are extracted. Oil may be extracted from animals, vegetables and fruits. The oil are termed as Essential oils (volatile) or Fixed oils (nonvolatile) oils can be classified into three main classes [2]. The oil which are separated from the petroleum and natural gas are known as mineral oils. These are pure, clear and colorless oily liquids. Mineral oils are used in medicines. It is also used as a solvent, insulator and lubricator. For example: Diesel oil, Kerosene oil etc. are mineral oils [3].

The oils which are obtained from the plants are known as essential oils. The essential oils are characterized with pleasant odor and taste. The essential oils contain volatile compounds and also known as volatile oils. These are mainly extracted from the plant parts mainly leaves, flowers, and seeds. Some essential oils are extracted. Jasmine Oil, orange peel essential oil, Lemon peel essential oils [4]. The oils which are extracted from both animals and plants are known as fixed oils. The fixed oils contain nonvolatile compounds and named as carrier oils. These don't show evaporation. The fixed oils are acyl glycerol mainly containing unsaturated fatty acids. Fixed oils can be rancid with time and these are both non-organic and organic in nature. Cotton seed oil, Linseed oil and almond oil are fixed oils [5].

Essential oils has the characteristics taste and odor of the plant which it has been extracted. The essential oils are extracted from different sources differ in their composition. The essential oil is known as essential because it is characterized with the essence of plant fragrance; from which it has been extracted [6]. The freshness, quality and uniqueness of Essential oil are major properties which increase its value. Essential oil is very sensitive to heat, oxygen and moisture. The processes that cause decrease in value of essential oil are the formation of oxygenated terpenes, chemical transformations and polymerization [7].

Essential oils comprise over a hundred substances divided into three categories: terpene hydrocarbons, oxygenated compounds, and nonvolatile components. Terpene function can make up 50-95% of the oil [8]. Terpenes are the primary components of essential oils, which are hydrocarbons of the monoterpenes. The second significant group of compounds found in essential oils are oxygenated derivatives such as alcohol, ketones, esters, and phenols. Minor constituents and trace elements of essential oils are sulfur-containing compounds, and flavonoids, which all contribute to the overall activity of the oil. These minor constituents and trace elements are present in traces but significantly may contribute to the therapeutic benefits of the oil. The versatile role of essential oil in a plant is to attract pollinators or repel harmful insects. Additionally, essential oils may also play a role as intermediate compounds in the plant's metabolic process [9].

Citrus essential oil is a wide variety of natural flavors, and perfumes that are widely utilized in the food industry, nutrition supplements, pharmaceuticals, cosmetics, and aromatherapy [10]. The essential oil is extracted from plants through different conventional and classical techniques including steam distillation, hydro distillation, solvent extraction, supercritical carbon dioxide fluid extraction, and cold pressing [11]. The six genera of citrus fruits- Fortunella, Eremocitrus, Clymendia, Poncirus, Microcitrus, and Citrus- are indigenous to tropical and subtropical regions of Asia [12, 13]. The annual world production of citrus fruit is approximately 110 million tons.

In 2023, oranges (Citrus sinensis) hold the title of the most extensively grown citrus fruit worldwide, with a total production of 47.4 million tons. Several processed food products, including jam and juice, are generated from around one-third of the oranges produced, resulting in a large quantity of waste. Approximately half of the total weight of oranges is attributed to waste [14], which mainly includes the flavedo, albedo, pulp, and seed. Within the citrus industry, managing solid waste is effectively recognized as a significant waste [15]. Orange waste contains a high concentration of fermentable carbohydrates and moisture, as well as antibacterial characteristics, which make standard landfilling troublesome [16].

In light of the increasing emphasis on sustainable waste management and the valorization of agro-industrial by-products, the present study investigates the potential of orange peel waste as a valuable source of essential oils through hydrodistillation. The research aims to promote sustainable waste utilization by assessing the essential oil content of orange peels and evaluating their extraction yield. Furthermore, the study seeks to develop and apply an environmentally friendly extraction approach that minimizes chemical usage and energy consumption while maximizing resource recovery. By converting orange peel waste into a commercially valuable product, this research contributes to the circular economy concept and highlights the potential of agricultural waste as a renewable raw material for the production of high-value essential oils.

METHODOLOGY

Raw Materials

Citrus sinensis (Orange Peel) was purchased from local market. Wash and dried orange peel and the peels were manually extracted. The quality was ensured to be free from mold or contaminants. The moisture content was 10-20%.

Chemical and Equipment

Round bottom flask, aluminum foil, measuring cylinder, tripod stand, analytical balance, separating funnel, glass vials, glass stirrer, graduated pipette and beaker were used during this experiment.

Sample Preparation

Peels underwent thorough washing and air-drying for several hours to reduce surface moisture. They were then cut into small pieces and ground using a food processor to produce fine particles, increasing surface area available for oil release during extraction.

Hydro distillation Procedure

Hydrodistillation was carried out following the method of Giwa et al. (2018) [17]. Fifty grams (50 g) of finely ground orange peel were placed in a round-bottom flask with 200 mL of distilled water. The flask was connected to a Clevenger-type apparatus and a water-cooled condenser; the cooling system utilized polyethylene glycol as a cooling agent and was set to maintain a condenser temperature of 10–22°C. Heating was applied at two experimental temperatures: 85°C and 100°C. Each extraction was maintained for up to 200 minutes, with distillate volume recorded at regular intervals (every 30 minutes). After hydrodistillation, the distillate (a water-oil mixture) was collected in vials and stored protected from light and oxidation until further processing.

Oil Separation and Yield Calculation

Fifteen millilitres (15 mL) of n-hexane were added to the distillate in a separating funnel. The mixture was vigorously shaken and allowed to settle for 10–15 minutes, enabling separation of the oil (upper, less dense) and water (lower, denser) layers due to density differences and preferential solubility of the essential oil in n-hexane. The oil layer was collected. This procedure was repeated twice to ensure complete extraction. The oil yield was calculated using the following formula:

% Yield = (Weight of oil extracted / Weight of sample used) × 100

Physical Characterization

Sensory evaluation was used to assess colour, odour, and viscosity. Solubility was tested in water and organic solvents (n-hexane, ethanol).

Chemical Composition Analysis

UV-Visible spectrophotometry was employed to characterize the chemical components of the extracted essential oil. The absorbance spectrum was recorded over a wavelength range of 200–700 nm. Absorption peaks were matched to known chromophore groups and reported compounds of orange peel essential oil from the literature.

Quality Parameters

Extraction factors such as temperature, and extraction time influenced the yield and quality of orange peel essential oil. Optimizing these parameters ensured maximum oil recovery while maintaining the integrity of important components like limonene. Increasing power and duration i likely to increase yield, but it may also destroy sensitive chemicals during the process.

RESULTS

The extraction of essential oil from orange peels was conducted using hydrodistillation. Water distillation was performed at a temperature of 85°C. The results, illustrated in Figure 1, show that no distillate was extracted during the initial 80 minutes. However, beyond this point, the distillate yield increased significantly with time. Notably, when the extraction time reached 200 minutes, the distillate yield was 12 mL per 50 grams of orange peel.

% Yield (distillate, 85°C) = 12 mL / 50 g × 100 = 24%

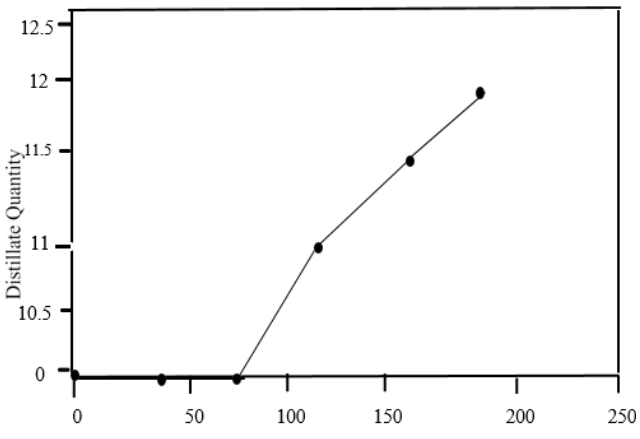

Figure 1 Hydrodistillation extraction process at 85°C

At 100°C, distillate production commenced from the initial time point, with a steady and consistent increase throughout the 200-minute extraction. The distillate yield reached 15 mL per 50 g of peel at 200 minutes (Fig. 2), corresponding to:

% Yield (distillate, 100°C) = 15 mL / 50 g × 100 = 30%

The distillate yield per gram of peel was 0.30 mL/g at 100°C versus 0.24 mL/g at 85°C, confirming that higher temperature improves extraction efficiency.

Essential Oil Yield

Following n-hexane separation of the distillate collected at the optimal temperature (100°C), the essential oil yield was:

% Oil Yield = 5.5 mL / 50 g × 100 = 11%
The oil quantity increased progressively with extraction time (Fig. 3), consistent with the temperature-dependent accumulation pattern observed in the distillate.

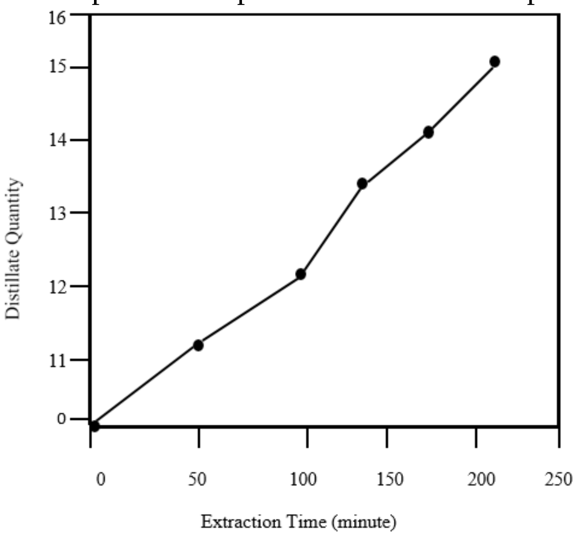

Figure 2 Hydrodistilllation distillate extraction process at 100°C

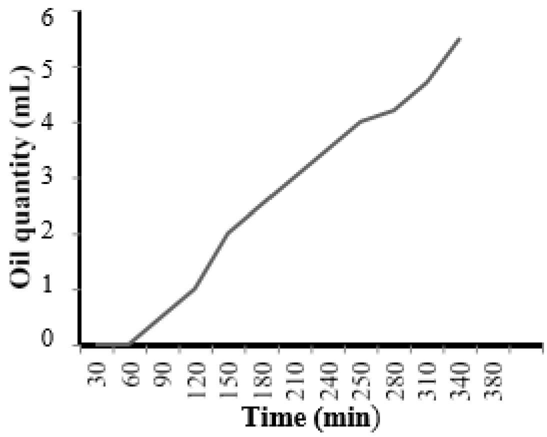

Figure 3 Oil quantity and Time Yield

Characterization of Essential Oil

Following extraction, the oil underwent characterization to confirm its identity and quality. The extraction of essential oil from orange peel using hydro-distillation was characterized to evaluate the efficiency and quality of the process. The characterization involved analyzing the physical and chemical properties of the extracted oil are summarized in table 1. Sensory analysis was conducted to evaluate the physical properties of the oil, employing sensory evaluation techniques that utilize the senses of smell, sight, and taste. This assessment enabled the verification of the extracted liquid as essential oil.

Table 1 Physical Properties of Orange Peel Essential Oil

Parameter	Observed Value
Colour	Pale Yellow
Odour	Fresh to Tangy
Viscosity	Low
Solubility	Insoluble in water; soluble in organic solvents

UV-Visible Spectroscopy and Chemical Composition

The chemical composition of the extracted oil was analyzed using various techniques, including UV-Vis spectroscopy. These analyses revealed the presence of various compounds, including limonene, linalool, and citral, which are commonly found in orange peel oil.

UV-Vis spectroscopy is an analytical technique that measures the absorbance and transmittance of light through a sample, offering insights into its composition and properties. The UV-Vis spectrum of the extracted oil exhibits multiple peaks, indicating the presence of various components that absorb light at specific wavelengths.

The absorbance values plotted against wavelength reveal distinct peaks at 203 nm, 208 nm, 210 nm, 234 nm, 268 nm, and 278 nm. Additionally, lower absorbance values are observed at 340 nm and 371 nm. The spectrum was scanned over a range of 200 nm to 700 nm, providing valuable information for identifying and quantifying the components present in the sample.

Table 2 UV-Vis Absorption Peaks and Corresponding Compounds

λ (nm)	Chromophore	Probable Compound
203	Conjugated dienes/alkenes	Limonene
208	Alkenes	β-Pinene
210	Conjugated alkenes	α-Pinene, γ-Terpinene
234	Extended conjugated system	Linalool / Naringin
268	Extended conjugation	Citral / Flavonoids
278	Carbonyl compounds	Coumarins
340	$n \rightarrow \pi^*$ / Charge transfer	Geranial
371	Aromatic compounds	Carotenoids

DISCUSSION

The present study confirmed that hydrodistillation is an effective method for extracting essential oil from orange peel, with temperature and extraction time identified as the principal determinants of yield. The higher distillate yield at 100°C (30%) compared to 85°C (24%) is consistent with established thermodynamic principles: elevated temperature accelerates steam formation and increases the vapour pressure of volatile compounds within the peel matrix, facilitating more efficient and rapid volatilization of the essential oil components [17,18]. The delayed onset of distillate collection at 85°C (no yield within the first 80 minutes) is attributable to the slower rate of steam generation at sub-boiling temperatures, consistent with the findings of Giwa et al. (2018) [17] and Kabuba and Huberts (2009) [18].

The final essential oil yield of 11% (5.5 mL/50 g) after n-hexane separation aligns with values reported in the literature for citrus peel essential oils extracted by hydrodistillation, which typically range from 0.5–3% by weight, though volumetric yields vary depending on peel moisture content and processing conditions [19,20]. The use of n-hexane for phase separation is well-established and ensures complete recovery of the hydrophobic oil fraction from the aqueous distillate [15].

Peel particle size proved critical in this study: reducing peel size to fine particles increased the available surface area for steam contact and oil release, consistent with reports by Golmohammadi et al. (2018) [21]. The water-to-peel ratio (200 mL/50 g = 4:1 v/w) was sufficient for adequate steam generation without excessive dilution of the oil, in agreement with Ngan et al. (2020) [22]. Gentle and consistent heating is necessary to prevent thermal degradation of sensitive monoterpenes such as linalool and citral, which can decompose at excessively high temperatures [23].

UV-Vis spectroscopy confirmed the presence of limonene (principal peak at 203 nm), which is the dominant compound in orange peel essential oil, constituting approximately 90% of the composition [6,7]. Additional peaks at 208 nm (β-pinene), 210 nm (α-pinene, γ-terpinene), 234 nm (linalool/naringin), 268 nm (citral/flavonoids), 278 nm (coumarins), 340 nm (geranial), and 371 nm (carotenoids) confirm a complex, multi-component limonene-rich oil profile consistent with published reports on *Citrus sinensis* essential oil [9,11,24]. The physical properties — pale yellow colour, tangy-fresh odour, low viscosity, and solubility in organic solvents — are all characteristic of authentic citrus peel essential oil [10,19].

Compared to advanced extraction methods such as microwave-assisted extraction, hydrodistillation produces slightly lower yields; however, its simplicity, low capital cost, absence of special instrumentation, and eco-friendly profile make it particularly appropriate for resource-limited settings and for applications in Pakistan's citrus processing industry where orange peel is currently discarded as waste [15,25].

CONCLUSION

Hydrodistillation successfully extracted essential oil from *Citrus sinensis* peel with a yield of 11% under optimized conditions (100°C, 200 minutes, 50 g peel in 200 mL distilled water, finely ground particles). Temperature was confirmed as the most critical process variable, with 100°C producing a 25% higher distillate yield than 85°C. UV-Vis spectroscopy revealed a limonene-dominated chemical profile consistent with high-quality orange peel essential oil. Hydrodistillation represents a viable, cost-effective, eco-friendly, and scalable technique for valorizing orange peel waste into commercially valuable essential oil, with potential applications across the food, pharmaceutical, and cosmetics industries.

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