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Experimental Evaluation of Fractional Distillation for Essential Oil Extraction from Citrus and Cedar Residues for potential applications in fragrance formulations

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Abstract. This research paper the extraction of essential oils from orange peels (Citrus sinensis), lemon peels (Citrus limon), and cedar sawdust through fractional distillation as a valorization strategy for agro-industrial waste derived from citrus consumption and lignocellulosic residues generated by the furniture manufacturing industry. To this end, the peels and sawdust were collected, conditioned, and distilled in a conventional fractional distillation system using 500 g of raw material per batch. The results showed extraction yields of 2.1% for orange peel, 0.86% for cedar sawdust, and 0.24% for lemon peel, with orange peel exhibiting the highest extraction efficiency. The essential oils obtained displayed yellow coloration, characteristic citrus and woody aromas, and immiscibility in water. These properties suggest their potential application in the formulation of fragrances and cosmetic products.
Keywords: fractional distillation, essential oils, citrus residues, cedar, D-limonene, agroindustrial and waste valorization

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1 Introduction

Essential oils, also known as essences, are mixtures of volatile organic compounds, primarily terpenes and their derivatives, found in a wide variety of plant species. These oils provide plants with their characteristic aromas and flavors, and are extracted using several techniques, among which distillation is the most common. Due to their aromatic and biological properties, essential oils are widely used in the pharmaceutical and cosmetic industries (Pedrini & Lucheroni, 2025; Cevallos et al., n.d.).

During the processing of citrus fruits, industries leave behind peels, seeds, and pulp, materials often dismissed yet rich in volatile compounds and can be used for the extraction of essential oils. These by-products, especially from lemon (Citrus lemon) and orange (Citrus sinensis), carry notable levels of limonene along with various aromatic components. Meanwhile, handling cedar trees yields leftover wood matter like sawdust and shavings. Such timber outputs are classified as lignocellulosic biomass, typically seen as discard but far from inert. Within them lie sesquiterpenes and distinct woody aroma molecules worth capturing. Though overlooked, both residues offer potential through essential oil recovery. Their value emerges not despite being residuals, but because of their chemical makeup (Solano-Rentería et al., 2026).

The accumulation of these residues can lead to environmental problems related to the proliferation of microorganisms, the generation of unpleasant smells, and an increased organic load in soil and water, Nevertheless, citrus peels contain bioactive compounds of great value, especially essential oils rich in D-limenene, a monoterpene commonly used in the food, cosmetic, pharmaceutical, and energy industries due to its aromatic, antioxidant, and antimicrobial properties. Likewise, cedar residues contain essential oils rich in sesquiterpenic compounds such as cedrol and α-cedrene, which are used in the production of perfumes, cosmetic products, insect repellents and formulations with antimicrobial and antifungal activity (Solano-Rentería et al., 2026; Campo & Ávila, 2010).

One widely applied technique for isolating and cleaning liquid blends is fractional distillation. Separation happens because substances in the mixture turn into vapor at different temperatures. Compounds that boil at lower heat emerge earlier, while others appear later as temperature increases gradually. Rising vapors grow richer in lighter elements as they move up the tower. This shift allows clearer portions to be captured over time (Silvestre et al., 2019).

This method is based on differences in volatility, where mass transfer occurs through contact between a vapor phase and a liquid phase at the same temperature and pressure. Mass transfer takes place simultaneously through vaporization and

condensation. The overall effect is an increase in the concentration of the more volatile component in the vapor phase and of the less volatile component in the liquid phase. Only after most of the more volatile liquid has been removed from the mixture—depending on the thermodynamic properties of the system and the degree of separation—does the less volatile liquid undergo evaporation and condensation processes (Silvestre et al., 2019).
Although numerous studies have reported the extraction of essential oils from citrus residues, most have focused on optimizing extraction conditions for a single raw material or identifying the chemical composition of the recovered oils. Comparative evaluations involving citrus residues and lignocellulosic biomass processed under the same experimental conditions using fractional distillation remain limited. Such comparisons are valuable because they enable the selection of raw materials with greater extraction potential prior to more advanced studies involving process optimization or chemical characterization.
Therefore, the objective of the present study was to comparatively evaluate the extraction performance of orange peels (Citrus sinensis), lemon peels (Citrus limon), and cedar sawdust using laboratory-scale fractional distillation under identical operating conditions. The comparison was based on extraction yield, recovered oil volume, density, extraction time, and specific energy consumption. Since the chemical composition of the extracted oils was not determined experimentally, references to compounds such as limonene, cedrol, and α-cedrene are based exclusively on previous literature and should not be interpreted as direct evidence of their presence in the oils obtained in this study.
Unlike previous studies that focused on individual feedstocks, this work provides a comparative evaluation of citrus and lignocellulosic residues under identical fractional distillation conditions using extraction yield and specific energy consumption as performance indicators.

2 Material and Methods

A modified version of the methodology described by Solano-Rentería et al. (2026) was used in this study. The original protocol was adapted to the laboratory facilities available for this research while maintaining the same extraction principle. The main modifications consisted of the use of a conventional laboratory-scale fractional distillation system and the evaluation of three different agro-industrial and lignocellulosic residues under identical operating conditions to allow a direct comparison of their extraction performance.
Figure 1 illustrates the flow chart of the experimental procedure used for the extraction of essential oils from citrus and lignocellulosic residues by means of fractional distillation. The diagram outlines the main stages of the process, beginning with the collection and preparation of the raw materials and ending with the recovery of the essential oil.

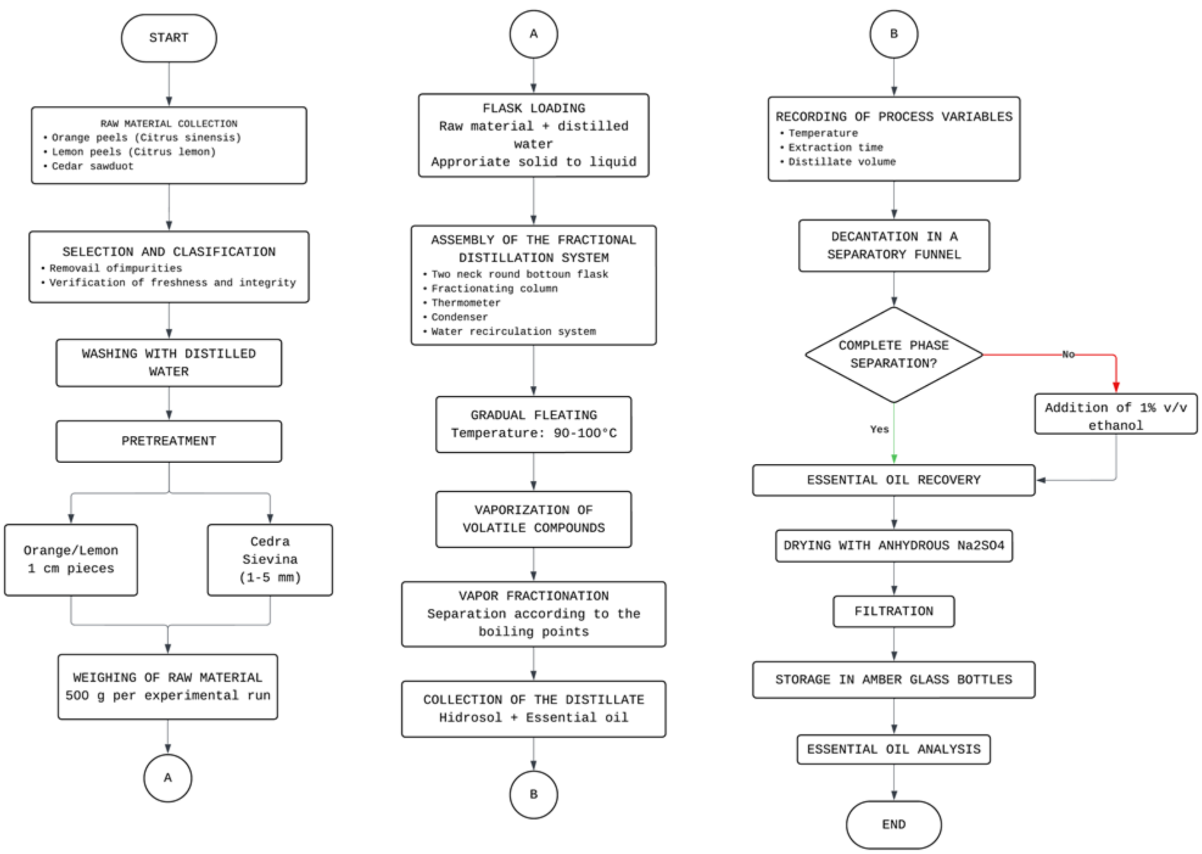

Fig. 1. Flowchart for the production of essential oil.

Orange (Citrus sinensis) and lemon (Citrus limon) peels were collected from local establishments dedicated to the sale of fresh juices and fruits in the city of Pachuca, Hidalgo, Mexico. Cedar sawdust was obtained from a local carpentry workshop located in the same city.

The plant material was selected according to freshness and physical integrity criteria, with damaged material, impurities, and foreign matter being removed prior to processing. Subsequently, the peels were washed with distilled water and cut into fragments approximately 1 cm in size in order to increase the contact surface area and promote the release of volatile compounds. The cedar sawdust was sieved to obtain particles ranging from 1 to 5 mm in size.

Oil extraction took place through standard fractional distillation (see Figure 2). The apparatus included a two-neck round-bottom flask sitting on a heater, alongside a fractionating column. A thermometer monitored temperature changes throughout the process. The vapor then moved into a glass condenser cooled by circulating water. Connections between parts relied on compatible glass fittings. The collected liquid ended up in a measuring cylinder after cooling. Because of this layout, repeated trials became possible without reassembling everything each time. Separation of the desired oil improved due to gradual boiling point differences inside the column. Each run maintained consistency thanks to stable heat control and steady flow rates.

a) b) c)

Fig. 2. Experimental setup for fractional distillation: (a) Orange residues, (b) cedar sawdust, and (c) lemon residues.

For each experimental run, 500 g of raw material were used. Independent runs were conducted using orange peel, lemon peel, and cedar sawdust. Each sample was placed in the distillation flask together with distilled water at a solid-to-liquid ratio sufficient to ensure uniform heating and prevent carbonization of the plant material. The mixture was gradually heated until reaching a temperature range of 90–100 °C. Temperature was continuously monitored to maintain stable operating conditions throughout the distillation process.

During the extraction process, volatile aromatic compounds were vaporized and transported through the fractionating column, where partial separation of the components occurred according to their respective boiling points. The condensed vapors were continuously collected in graduated containers, and the operating temperature, extraction time, and volume of distillate obtained were recorded. Each experimental run lasted approximately 2 h.

The resulting distillate consisted of a mixture of hydrosol and essential oil. This mixture was transferred to a separatory funnel and allowed to settle to promote phase separation. When necessary, 1% (v/v) ethanol was added to facilitate the recovery of the oily fraction. The recovered organic phase was dried using anhydrous sodium sulfate to remove residual moisture. Finally, the essential oil was filtered and stored in amber glass bottles at room temperature until further analysis.

3 Results and discussion

Table 1 presents the experimental results obtained from the extraction of essential oils by fractional distillation using different raw materials.

Table 1. Experimental results obtained from the extraction of essential

	Raw material (gr)	Essential oil (gr)	Yield (%)	Density 20°C (g/ml)	Volume (ml)	Constant Temperature (°C)	Theoretical oil (gr)	Oil efficiency
Lemon	500	1.2	0.24	0.857	1.0284	95	1.6	75

Orange	500	10.5	2.1	0.84	8.82	95	11.7	89.743
Cedar	500	4.3	0.86	0.931	4.0033	95	5.2	82.6923

The extraction yield was calculated as the percentage ratio between the mass of essential oil recovered and the initial mass of fresh raw material. Orange peel exhibited the highest extraction yield (2.10%), followed by cedar sawdust (0.86%) and lemon lemon peel, further confirming its potential as a promising raw material for the valorization of agro-industrial residues.

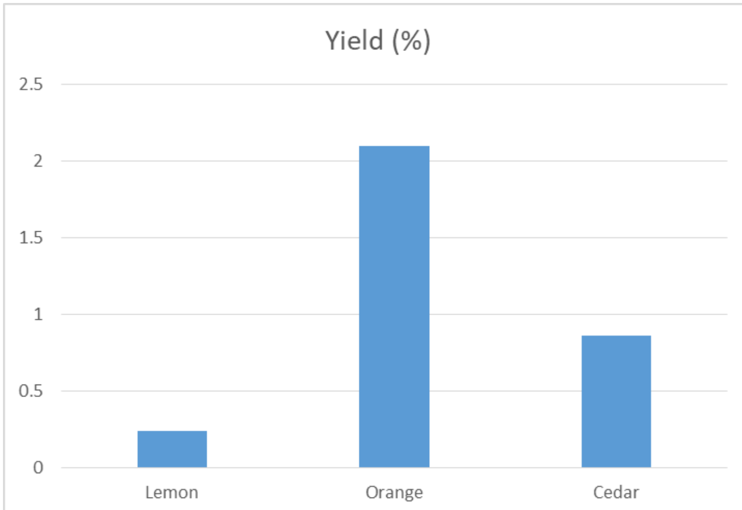

Fig. 3. Essential oil yield obtained from each raw material.

Table 2. Extraction times

Time (min)	Lemon Oil (ml)	Orange Oil (ml)	Cedar Oil (ml)
0	0	0	0
5	0	0.6	0.3
10	0.1	1.7	0.6
15	0.2	2.1	1
20	0.3	2.8	1.4
25	0.4	3.7	1.8
30	0.5	4.6	2.1
35	0.6	5.2	2.8
40	0.7	5.9	3.2
45	0.8	6.7	3.6
50	0.9	7.4	3.8
55	1	8.1	4
60	1.0284	8.82	4

Regarding extraction times (Table 2), a progressive increase in the volume of recovered oil was obpeel (0.24%). Likewise, orange peel showed the highest recovery efficiency (89.74%), whereas cedar and lemon reached recovery efficiencies of 82.69% and 75.00%, respectively.

The superior extraction performance observed for orange peel may be associated with the greater abundance of volatile compounds reported for this raw material in previous studies (Solano-Rentería et al., 2026). However, because the chemical composition of the recovered oils was not determined in the present study, this interpretation is based exclusively on published literature and should not be considered direct experimental evidence. Similar trends have been reported for citrus residues, where orange peel generally exhibits higher essential oil yields than lemon peel under comparable extraction conditions.

Figure 3 shows that orange peel produced approximately 2.5 times more essential oil than cedar and nearly nine times more than served as the operating time increased. However, the extraction rate differed among the raw materials evaluated. Orange peel exhibited the most favorable performance, reaching a cumulative oil volume of approximately 8.1 mL after 55 min of operation. Cedar yielded 4.0 mL, whereas lemon produced only 1.0 mL during the same period, as shown in Figure 4.

These results indicate that the availability of volatile compounds in orange peel is considerably greater than that of the other raw materials evaluated. During the first 10 min of operation, a rapid recovery of essential oil was observed for both orange peel and cedar. Thereafter, the extraction rate gradually decreased, suggesting a stage controlled by internal diffusion phenomena and mass transfer from the interior of the plant structures to the vapor phase.

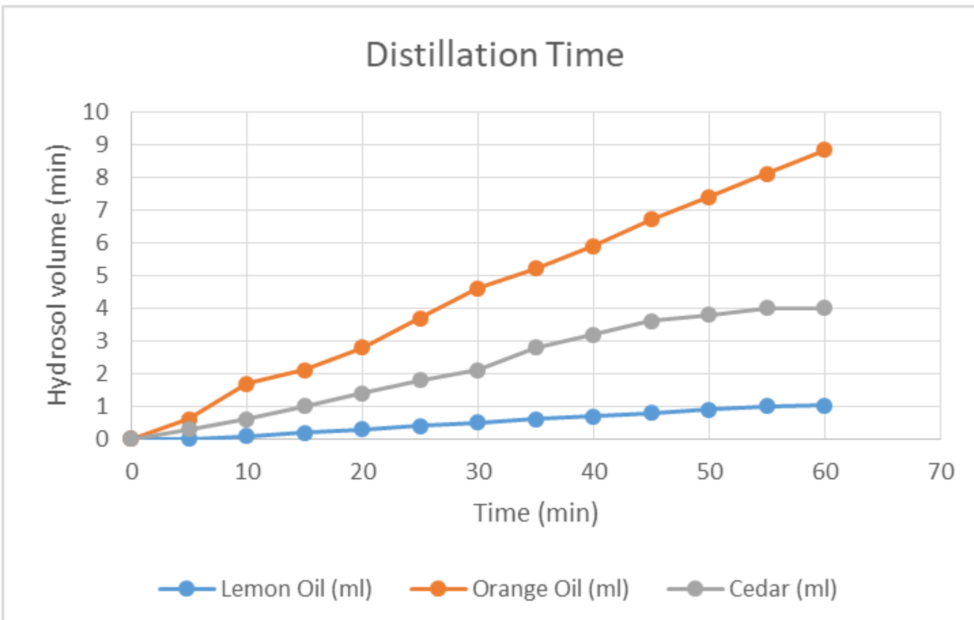

Fig. 4. Distillation time of the materials.

Figure 4 shows that orange peel exhibited an average extraction rate approximately eight times higher than that obtained for lemon peel and nearly twice that observed for cedar. These results suggest that fractional distillation is particularly effective in recovering terpenic compounds from citrus residues, especially those rich in limonene.

In addition, after approximately 45 min of operation, the curves began to show a decrease in slope, indicating that a large proportion of the available volatile compounds had already been removed from the system. From an operational perspective, this behavior suggests that excessively extending the distillation time may increase energy consumption without producing significant improvements in the overall extraction yield.

The density of the recovered oils ranged from 0.840 to 0.931 g mL^{-1}. Cedar essential oil exhibited the highest density (0.931 g mL^{-1}), whereas orange and lemon oils presented lower values of 0.840 and 0.857 g mL^{-1}, respectively. These differences may be associated with variations in the overall composition of the extracted volatile fractions reported for these botanical sources. Nevertheless, because no chemical characterization was performed, the observed density differences cannot be directly attributed to specific compounds.

The organoleptic evaluation revealed clear differences in the appearance and aroma of the recovered oils. Lemon oil presented a transparent light-yellow appearance with a fresh citrus aroma, orange oil showed a pale-yellow color and a sweet citrus fragrance, whereas cedar oil exhibited an amber-yellow coloration accompanied by a characteristic woody aroma. All samples were immiscible in water, which is consistent with the hydrophobic nature of essential oils. These observations provide a preliminary qualitative description of the extracted products; however, additional analytical techniques such as GC–MS, FTIR, or NMR are required to determine their chemical composition and confirm the identity of the volatile compounds present.

Preliminary experiments also indicated that fresh citrus peels produced higher extraction yields than peels that had previously been frozen or stored for prolonged periods. Consequently, all subsequent experiments were performed using fresh raw materials to minimize the influence of storage on oil recovery.

The specific energy consumption (SEC) was used as an additional parameter to evaluate the energy performance of the extraction process based on the effective heat input supplied during extraction (kW). Orange peel exhibited the lowest SEC (71 kJ g^{-1}), followed by cedar sawdust (173 kJ g^{-1}) and lemon peel (620 kJ g^{-1}). Since lower SEC values indicate that less energy is required to recover one gram of essential oil, orange peel demonstrated the highest energy performance among the evaluated raw materials. The substantially higher SEC observed for lemon peel resulted primarily from its lower extraction yield rather than from differences in extraction time or operating temperature, which remained essentially constant throughout all experiments.

Although the present study demonstrated the feasibility of recovering essential oils from citrus peels and cedar sawdust by fractional distillation, several limitations should be considered when interpreting the results. The chemical composition of the recovered oils was not experimentally determined using chromatographic or spectroscopic techniques such as GC–MS, FTIR, or NMR. Consequently, references to compounds including D-limonene, cedrol, and α-cedrene are based exclusively on previous literature describing the typical composition of these raw materials and should not be interpreted as direct evidence of their presence in the extracted oils.

In addition, minor experimental uncertainties may have arisen from oil transfer between glassware, incomplete phase separation, retention of oil by the drying agent, evaporation of highly volatile compounds, and possible thermal degradation or oxidation during atmospheric-pressure distillation. These factors may have influenced the extraction yields obtained. Furthermore, the present work represents an exploratory laboratory-scale comparative study; therefore, each raw material was evaluated under a single experimental run, and statistical parameters such as standard deviations and confidence intervals were not determined future research should include replicated experiments with statistical analysis, as well as comprehensive chemical characterization of the recovered oils, to validate their composition and evaluate their potential applications in fragrance and cosmetic formulations.

4 Conclusions

This study demonstrated the feasibility of recovering essential oils from orange peel (*Citrus sinensis*), lemon peel (*Citrus limon*), and cedar sawdust using laboratory-scale fractional distillation under identical operating conditions. Among the evaluated raw materials, orange peel exhibited the highest extraction yield (2.10%) and the lowest specific energy consumption, indicating the most favorable extraction performance, whereas cedar sawdust showed intermediate values and lemon peel the lowest recovery.

The extraction profiles indicated that most of the recoverable oil was obtained within the first 45–60 min of operation, suggesting that extending the distillation time beyond this period may increase energy consumption without substantially improving oil recovery. These findings provide useful information for future optimization of laboratory-scale extraction processes.

The results also demonstrate the potential of citrus and cedar residues as alternative feedstocks for essential oil recovery, contributing to the valorization of agro-industrial by-products within a circular economy framework. However, because this study focused on process performance, the chemical composition of the recovered oils was not experimentally determined. Future research should include replicated experiments with statistical analysis and comprehensive chemical characterization (e.g., GC–MS, FTIR, and NMR) to confirm the composition, quality, and potential applications of the recovered essential oils.

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