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OPTIMIZATION OF ESSENTIAL OIL EXTRACTION FROM KINNOW (*CITRUS RETICULATA* BLANCO) PEEL: A RESPONSE SURFACE METHODOLOGY APPROACH INTEGRATING ULTRASONIC AND OHMIC HEATING PRETREATMENTS

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ABSTRACT

Kinnow peel, a rich source of essential oil (EO), holds significant potential for applications in the food and pharmaceutical industries due to its aromatic and bioactive properties. However, the efficient extraction of EO from kinnow peel remains a challenge, necessitating the exploration of innovative extraction techniques. This study aimed to investigate the impact of two pretreatment methods-ultrasonic-assisted extraction (USAE) and Ohmic heating-aided extraction-on the extraction kinetics and chemical composition of kinnow peel EO using hydrodistillation (HD). Ultrasonic extraction parameters (power, time) and Ohmic heating variables (voltage, temperature, and salt concentration) were optimized using response surface methodology to achieve maximum yield. The results indicated that USAE produced higher EO yields (0.8% to 2.40%) compared to Ohmic heating (0.8% to 2.25%). The optimal conditions for USAE were 225.94 W, a solvent ratio of 692.23 mL, and 24.05 minutes, yielding a predicted 2.338% EO with a desirability of 0.961. For Ohmic heating, the best results were achieved with 219.02 V, 1.45% salt concentration, and 75.60°C, yielding 2.302% EO with a desirability of 1.000. These findings highlight that ultrasonic pretreatment provides superior extraction efficiency for essential oil (EO) without altering its natural composition, making it a promising method for industrial applications.

Keywords: Kinnow peel, Essential oil, Extraction techniques, Characterization, Response surface methodology

INTRODUCTION

Citrus reticulata, commonly known as kinnow, is a widely consumed citrus fruit appreciated for its abundant juice, rich flavor, and nutritional value. It is primarily cultivated in northern India, particularly in the states of Punjab, Haryana, and Rajasthan. In recent years, the increasing demand from the food industry for fresh and processed kinnow has led to a significant expansion in its cultivation area due to its excellent quality attributes, especially its tangy taste (**Mahawar *et al.*, 2020**). However, the citrus juice production process generates substantial byproducts, primarily peel, which accounts for 30-40% of the waste, along with pulp, rag (membranes and cores), and seeds (**Grover *et al.*, 2024a**). These byproducts, constituting approximately 45 to 60 % of the total fruit, are often discarded, raising environmental concerns (**Grover *et al.*, 2024b**). The decomposition of such waste material can lead to soil acidity, negatively impact water quality, and release harmful volatile organic compounds (**Gavrilescu, 2021**).

Citrus fruits are not only important for human consumption in fresh or processed forms but also represent a significant source of essential oils (EOs), particularly in their peels (**Grover *et al.*, 2024a**). Extracting EOs from citrus byproducts, such as peel and pomace, is widely recognized as an effective waste management strategy in fruit processing industries. These oils are valuable for their aromatic properties, serving as cost-effective sources of flavors and fragrances in various industries, including cosmetics, food, and beverages (**Bhattacharya *et al.*, 2023**). Citrus EOs are generally regarded as safe (GRAS) by the US Food and Drug Administration (**Tisserand & Young, 2014**) and shows better antimicrobial and antioxidant properties (**Grover *et al.*, 2023**). Several methods are commonly used for extracting EOs from citrus peels, such as water distillation, steam distillation, solvent extraction, and cold pressing. Each of these methods has its advantages and limitations in terms of efficiency, cost, and quality.

To improve the yield and quality of EOs, modern extraction techniques are being developed, including Ohmic heating and ultrasonic-assisted extraction (USAE). Ohmic heating is an advanced food processing technology that involves applying alternating current to generate internal heat within the food material, enabling rapid and uniform heating (**Astráin-Redín *et al.*, 2024**). This method preserves the nutritional and organoleptic properties of the food, as the heating is more controlled than conventional methods relying on external heat transfer. USAE, on the other hand, employs ultrasonic waves to create cavitation bubbles that enhance mass transfer and increase the release rate of essential oils from plant materials. This technique offers the advantage of reduced extraction time while minimizing the risk of thermal degradation, thus preserving the quality and volatile nature of the extracted oils (**Shen *et al.*, 2023**).

The efficiency of EO extraction, as well as the total yield and chemical composition, is highly influenced by the pretreatment methods employed. Improper techniques can lead to the degradation of volatile and sensitive compounds, affecting the quality and flavor of the extracted oils (**Arafat *et al.*, 2020**). Therefore, the present study aims to systematically evaluate the effects of two innovative pretreatment methods, ultrasonic-assisted extraction (USAE) and Ohmic heating, on the yield and composition of kinnow peel EO. Response surface methodology (RSM) was used to optimize the extraction parameters and determine the most effective conditions for each method.

This study contributes to the future research focused on improving essential oil extraction processes. It highlights the potential of using USAE and Ohmic heating as efficient, eco-friendly alternatives to traditional methods. Future studies should further explore the scalability of these techniques for industrial applications and their impact on the sensory and functional properties of EOs in food and cosmetic formulations.

MATERIAL AND METHODS

Materials

Kinnow peel was sourced from local juice vendors in the areas of Hisar and Sirsa, India. Damaged or faulty peels were carefully separated, and the selected ones underwent thorough washing under running water to eliminate any traces of dust, dirt, and microflora. Only the best-suited peels were retained for subsequent processing.

Sample preparation

The peel samples underwent a drying process in a hot air oven (MSW-211, MAC) at temperatures ranging from 30 to 40°C, adjusted according to the quantity being

processed. Following the drying, the peel was ground into a powder using a commercial grinder (Sujata India Pvt. Ltd.). The obtained powder was packed into air tight LDPE pouches and stored at 4±0.2 °C for further use.

Extraction methods

Ultrasonication assisted extraction

Ultrasonic aided hydro-distillation utilized the Ultrasonic Probe Sonicator (Ultrasonic processor BGS-185B) with a maximum output power of 750W. To prepare the solution, 100 g of dried kinnow peel powder were dissolved in distilled water within a 1000 ml beaker. The ultrasonication probe was placed on the beaker, and upon switching it on, a pulse duration of 2 seconds was applied to the sample. Following the ultrasonication treatment with the specified time, power, and solvent as shown in Table 1, the solution as transferred to a conical flask. Subsequently, the flask was positioned in a heating mantle, and the hydrodistillation process was carried out at a temperature of 80 °C for 4 h. Upon extraction of the oil, the knob was opened, and the obtained oil was stored in an Eppendorf tube.
The weight of EO was measured and the percentage yield of oil was calculated. The yield was expressed as follows:

$$\text{Yield (\%)} = \frac{\text{Volume of essential oil extracted}}{\text{Initial weight of dried peel powder taken}} \times 100$$

The extracted EO was preserved at a temperature of 4 ± 0.2 °C until it was required for further analysis.

Table 1 Experimental and predicated extraction efficiencies by ultrasonication assisted hydrodistillation under different conditions

Std	Run	Solvent (mL)	Time (Min)	Power (W)	Actual Yield %	Predicated yield %
1	6	600	10	225	0.9 ±0.07	0.94
2	12	800	10	225	0.8 ± 0.21	0.89
3	10	600	30	225	1.8 ±0.09	1.71
4	11	800	30	225	1.5±0.02	1.46
5	8	600	20	150	1.3±0.02	1.24
6	14	800	20	150	0.9 ±0.03	0.79
7	15	600	20	300	0.9± 0.10	1.01
8	13	800	20	300	1.1 ± 0.1	1.16
9	5	700	10	150	0.8 ± 0.01	0.82
10	2	700	30	150	1.45 ± 0.05	1.55
11	4	700	10	300	1.1 ±0.01	0.95
12	7	700	30	300	1.6 ±0.05	1.57
13	9	700	20	225	2.3 ±0.1	2.27
14	1	700	20	225	2.2 ±0.1	2.27
15	3	700	20	225	2.4 ±0.1	2.27

All values are mean ± standard deviation of triplicate analysis

Ohmic heating assisted extraction

Ohmic heating hydro distillation was conducted using a specially designed device with platinum electrodes, created at the Department of Food Technology of Guru Jambheshwar University of Science and Technology. Hisar, India. In the ohmic heating assembly, 100 g of dried peel powder were dissolved in 700 ml of distilled water, with specific maintenance of ohmic heating temperature, voltage, and salt concentration (Table 2). After the ohmic heating treatment, the subsequent steps in the procedure closely resembled those of the HD method. Throughout the entire process, careful monitoring of processing parameters, including temperature, voltage, and salt concentration, was maintained.

Table 2 Experimental and predicated extraction efficiencies by Ohmic assisted hydro distillation under different conditions

Std	Run	Voltage (Volts)	Salt (%)	Temperature (°C)	Actual Yield (%)	Predicated yield (%)
1	1	180	1.00	70	0.8 ± 0.10	0.96
2	10	220	1.00	70	2.00 ±0.02	1.92
3	5	180	2.00	70	1.00 ± 0.11	1.08
4	13	220	2.00	70	2.3 ± 0.1	2.14
5	3	180	1.50	60	1.33 ±0.03	1.21
6	15	220	1.50	60	1.86 ±0.02	1.97
7	8	180	1.50	80	1.22 ±0.03	1.11
8	6	220	1.50	80	2.25±0.04	2.37
9	2	200	1.00	60	1.49 ±0.01	1.46
10	7	200	2.00	60	1.52 ±0.05	1.57
11	9	200	1.00	80	1.6 ±0.03	1.55
12	11	200	2.00	80	1.74 ±0.03	1.78
13	14	200	1.50	70	1.9 ±0.05	1.87
14	4	200	1.50	70	1.8 ±0.05	1.87
15	12	200	1.50	70	1.9 ±0.05	1.87

All values are mean ± standard deviation of triplicate analysis

Characterization of essential oil

Characterization of extracted oil by Fourier-transform infrared (FT-IR) spectroscopy

The EOs from kinnow fruit peel were subjected to FTIR analysis using a Perkin Elmer instrument. The analysis involved preparing a KBr powder by uniformly crushing it in a crystal pestle and mortar. Subsequently, the fine powder was compressed into a consistent thin pellet using a hydraulic pellet press. To perform the infrared spectroscopy, the EOs sample was directly placed on the surface of a pair of rectangular NaCl plates at room temperature. Measurements were conducted in the infrared region spanning from 4000 to 400 cm^{-1}. Each EO underwent two scans at a speed of 3 cm/s, and an air spectrum was used as a reference during the analysis. This analytical method allows for the identification and characterization of chemical components present in the kinnow fruit peel EOs based on their unique infrared absorption patterns.

Characterization of extracted oil using Gas Chromatography-Mass Spectrometry (GC-MS)

The extracted essential oil (EO) was dissolved in HPLC grade methanol at a ratio of 1:99 and subsequently analyzed using GC-MS instrument (Thermo Fisher Scientific in the USA) which included a GC-Trace 1300, MS-TQS Duo, and Autosampler-TriPlus RSH, coupled with a DB-5MS column (Length- 40 m, ID-0.15µ, Film Thickness-0.15µ). Fore line pressure temperature was set at 210 °C, Transfer line temperature at 200 °C, and the Ionization mode was Electron Impact (EI) at 70eV. Helium served as the carrier gas, with an injector temperature of 200 °C and an injection volume of 1ul. During the GC analysis, the temperature conditions ranged from 50 °C (held for 5 minutes) to 210 °C (held for 15 minutes) at a ramp rate of 5 °C/min. The mass range selected for characterization of analytes ranged from 45 to 450. The obtained results were compiled by comparing the spectra with the inbuilt NIST library, facilitating the identification of components present in the EOs.

Statistical analysis

The Response Surface Methodology (RSM) design aimed to investigate the impact of various factors on EO yields, considering parameters such as material-to-water ratio, extraction temperature, and extraction time for hydro distillation. Additionally, the study explored the effects of ultrasound power, material-to-water ratio, and extraction time on yields (Table 1). For Ohmic heating, the variables studied included voltage, temperature, and salt concentration (Table 3). The initial parameter settings were determined based on early experimental findings. Design Expert version 7.0.0 software was employed for ANOVA (Analysis of Variance) analysis, coefficient calculations, and graphical representation (Tables 2 and 4). To assess the model's reliability in predicting outcomes, both anticipated and actual yields were analyzed. This comprehensive approach allowed for a thorough examination of the factors influencing EO extraction and provided insights into the effectiveness of the applied methodologies.

RESULTS AND DISCUSSION

Ultrasonication assisted extraction method

The ultrasonication-assisted hydro distillation process yielded EO ranging from 0.8% to 2.40% from dried kinnow peel powder. In contrast, **Sandhu *et al.* (2021)** achieved a minimum yield of 3.90% from citrus waste using specific extraction parameters. **Heydari *et al.* (2021)** investigated bitter orange peel EO, obtaining yields between 0.4% and 1.0%, while the present study reported higher amounts. **Yu *et al.* (2021)** observed similar results with Kamquat peel. **Sharma and Tripathi (2008)** reported a maximum essential oil (EO) yield of 1.8% from the epicarp of Indian sweet orange (*Citrus sinensis Osbeck*) using hydro-distillation, while **Hosni *et al.* (2010)** found a peak yield of 1.89% from Tunisian orange peels. Variations in extraction yield are attributed to fruit characteristics and extraction procedures. The results, obtained through Box Benken's method, are presented in Tables 3, displaying experimental findings and predictions from Design Expert version 7.0.0. ANOVA analysis of the quadratic model, including EO extraction power (A), extraction time (B), solvent (C), interaction terms (AB, AC, BC), and second-order terms (A2, B2, C2). The significant F-value of 15.51 for the model indicates its significance; whereas the lack of fit (LOF) was not significant for quadratic model described by the equation 1.

Yield (Y)= +2.27-0.075*A+0.34*B+0.038*C-0.050*A*B+0.15*A*C-0.025*B*-0.60*A^2-0.42*B^2-0.62*C^2 .....Equation – 1

Table 3 Statistical analysis on the variation of extraction yield **(**ANOVA, Partial sum of squares - Type III)

Source	Sum of squares	dF	Mean Square	F-value	P-value	Comment
Model	4.03	9	0.45	15.51	0.0038	Significant
A-Power	0.045	1	0.045	1.56	0.2669	SD=0.17
B-Time	0.91	1	0.91	31.60	0.0025	Mean=1.39
C-Solvent	0.011	1	0.011	0.39	0.5596	CV (%) =12.19
AB	1.000E-002	1	1.000E-002	0.35	0.5815	R^2=0.9654
AC	0.090	1	0.090	3.12	0.1375	AP – 10.669
BC	2.500E-003	1	2.500E-003	0.087	0.7802	Pred R^2=0.6007
A^2	1.13	1	1.31	45.46	0.0011	Adj R^2=0.9032
B^2	0.65	1	0.65	22.68	0.0050	
C^2	1.42	1	1.42	49.36	0.0009	
Residual	0.14	5	0.029			
Lack of fit	0.098	3	0.033	1.39	0.4438	Not significant
Pure error	0.047	2	0.23			
Core total	4.17	14				

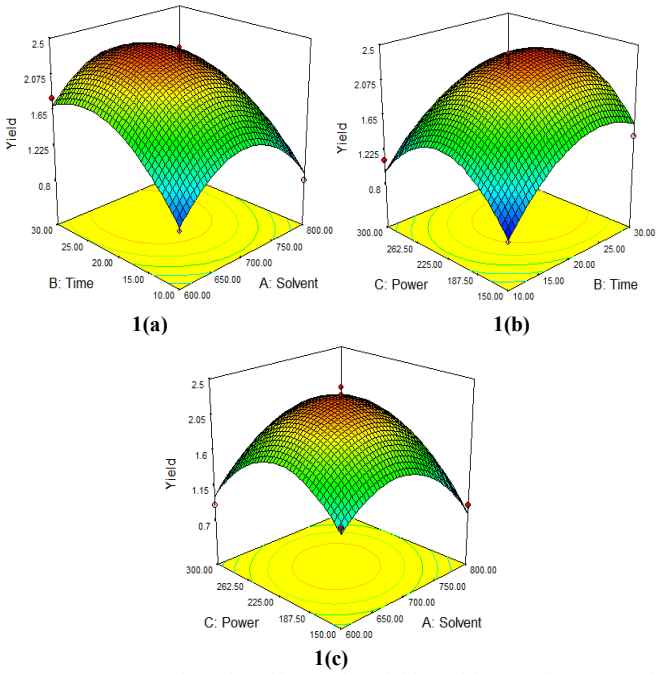
Influence of various factors on the yield of EO

Figure 1 illustrates the relationship between EO yield and three key process factors. A consistent trend was observed across all factors, wherein increasing the levels of power, extraction time, and material-to-water ratio led to a rise in the extracted oil yield up to a central value. Beyond this point, however, the yield began to decrease. Similarly, the extraction yield showed a continuous increase with rising solvent quantities up to the central value, after which an increase in solvent quantity led to a decline in yield.

In the case of ultrasonication power, the EO yield consistently increased from 150W to 225W, reaching the center value, and subsequently decreased when the power was further increased to 300W. A similar pattern emerged for ultrasonication treatment time, where an increase from 10 minutes to 20 minutes resulted in a higher yield, but continuous increments beyond 20 minutes led to a decline in extraction yield. This decline in yield with prolonged ultrasonic duration and higher power aligns with findings from prior research (**Goula, 2013**).

The observed pattern may be attributed to the prolonged ultrasonic time and higher power causing the rupture of more cell walls, leading to the suspension of impurities like cytosol and insoluble materials in the extract. This, in turn, lowers the permeability of the solvent into cell structures and hinders the transfer of dispersed oil out of the solid structure, as noted in previous studies (**Tian *et al.*, 2013**).

Both solvent and material exhibited a quadratic effect on yield. Increasing the solvent up to the center value positively influenced extraction yield, but beyond that point, as the solvent-solid ratio increased, the yield began to decrease. The decrease in oil extraction could be attributed to the occurrence of bubbling at higher solid-to-solvent ratios, as observed in previous research (**Sikdar *et al.*, 2016**). Another potential factor is that an increase in solvent levels beyond 700ml might lead to a decline in yield, possibly due to excessive water causing the dissolution or emulsification of EO (**Tran *et al.*, 2019**). Moreover, larger water volumes can result in extended distillation durations and higher operational costs. In a related study on the composition of EO from citrus peel, **Li *et al.* (2022)** identified similar trends in ultrasound time and solvent-solid ratio during ultrasonication treatment.



1(a) 1(b)

1(c)

Figure 1 Response surface plot of interaction yield % with extraction power, time and solvent ratio in UHHD

Optimum conditions value according to desirability function

Utilizing the surface response graphs and considering the data similarities presented earlier, optimization was conducted to determine the most favorable extraction conditions for kinnow EO. The optimal parameters identified were a power of 225.94W, a solvent ratio of 692.23 mL, and extraction duration of 24.05 minutes. The predicted oil yield under these optimum conditions was 2.338%, with

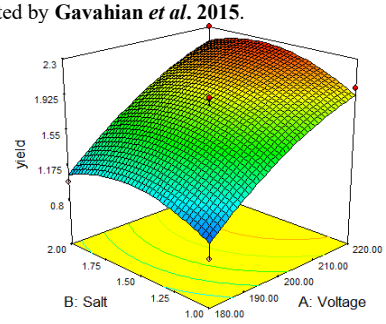
a desirability score of 0.961. Upon experimentation, the actual yield at the optimized conditions was found to be 2.4%.

Ohmic heating assisted extraction method

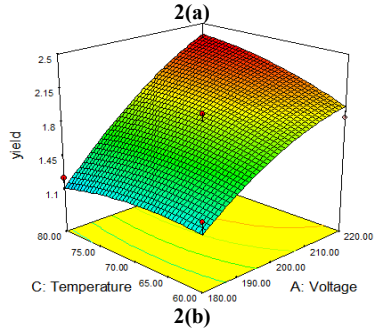
The ohmic heating-assisted hydro distillation process yielded EO from dried kinnow peel powder with a maximum and minimum yield of 2.25% and 0.8%, respectively.

Effect of Ohmic heating voltage, salt concentration and temperature on the yield

Figure 2 illustrates that increases in voltage gradient, processing temperature, and salt concentration generally enhance EO yield in the ohmic heating-assisted hydrodistillation (OHHD) process. The OHHD technique consistently showed a much higher EO yield compared to the traditional hydrodistillation (HD) method. This higher EO yield is attributed to the more intense and efficient utilization of energy in the ohmic heating process, leading to the disruption of samples within the system, as noted by **Gavahian *et al*. 2015**.



2(a)



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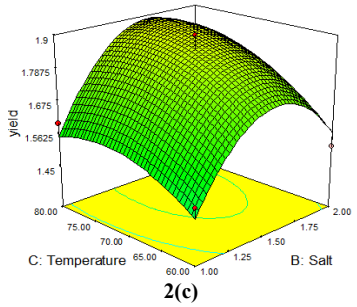


2(c)

Figure 2 Response surface plot of interaction yield % with extraction temperature, voltage and salt concentration in OHHD

Electroporation, a non-thermal effect of ohmic heating, induces permeability in the cell walls and membranes of samples, simplifying EO extraction compared to the HD method. The extraction yield demonstrated only slight changes when salt concentration varied from 1% to 1.5% to 2.0%. Additionally, an increase in temperature from 60 to 80°C, coupled with an increase in voltage, led to a rise in extraction yield. This can be attributed to the strengthened electric field, resulting in enhanced ion mobility in the peel powder and increased collisions between molecules, as suggested by **Al-Hilphy *et al.* 2020**.

HD involves the transfer of heat from the outside to the inside, whereas in OHHD, heat is transferred from the inside to the outside, reducing heat loss. This difference is attributed to the unique characteristic of OHHD. Joule's law is employed in OHHD to convert electrical energy into thermal energy. **Moongngarm *et al.* (2022)** conducted a study demonstrating that applying electric field strength of 200 V/cm to Stevia leaves with a moisture content of 40% resulted in the highest temperature increase. This highlights the effectiveness of ohmic heating in generating thermal energy within the sample.

The results were obtained using Box-Behnken's method, and Tables 3 present both experimental findings and predictions generated by Design Expert version 7.0.0. The ANOVA analysis for the quadratic model involved EO extraction variables such as voltage (A), salt concentration (B), temperature (C), as well as interaction terms (AB, AC, BC) and second-order terms (A2, B2, C2), with three independent variables as shown in Table 4. The model's F-value of 10.39 indicates its significance.The "Lack of Fit F-value" of 0.12 suggests that the Lack of Fit is insignificant compared to pure error, implying that the model adequately fits the data and quadratic model described by the equation 2. Additionally, the "Adequate Precision" metric evaluates the signal-to-noise ratio, providing an assessment of the model's reliability and precision.

Final equation in terms of coded factors

Yield = +1.87+0.51*A+0.084*B+0.076*C+0.025*A*B+0.13*A*C+0.027*B*C-0.13*A^2-0.21*B^2-0.07*C^2 .....Equation 2

Table 4 Independent variables of Ohmic heating **(**ANOVA, Partial sum of squares-Type III)

Source	Sum of squares	dF	Mean Square	F-value	P-value	Comment
Model	2.45	9	0.27	10.39	0.0095	SD.= 0.16
A-Voltage	2.06	1	2.06	78.62	0.0003	Mean =1.65
B- Salt	0.056	1	0.056	2.14	0.2033	R^2 =0.9492
C- Temperature	0.047	1	0.047	1.77	0.2403	Pred R^2=0.2234
AB	2.500E-003	1	2.500E-003	0.095	0.7699	A P =10.705
AC	0.063	1	0.063	2.38	0.1832	
BC	3.025E-003	1	3.025E-003	0.12	0.7479	
A^2	0.064	1	0.064	2.46	0.1777	
B^2	0.16	1	0.16	6.19	0.0553	
C^2	0.018	1	0.018	0.68	0.4465	
Residual	0.13	5	0.026			
Lack of fit	0.12	3	0.041	12.44	0.0753	Not significant
Pure error	6.667E-003	2	3.333E-003			
Cor Total	2.58	14				

Optimum conditions value according to desirability function

The optimal extraction conditions for kinnow EO were identified as a voltage of 219.02 V, salt concentration of 1.45%, and temperature of 75.60°C for Ohmic heating-assisted extraction. The predicted yield under these optimized conditions was 2.302%, with a desirability score of 1.000. In practical experimentation, the actual yield at these optimum conditions was found to be 2.2%.

Effect of pretreatment on EO extraction yield

Evidently, the UAHD and OHHD demonstrated improved extraction kinetics compared to HD alone, maximum extraction yield for UAHD, OHHD and HD were 2.40%, 2.25%and 2.10%. This heightened extraction efficiency aligns with the recognized benefits of USAE of EO, as noted by **Chemat *et al.* (2011)**. Both UAHD and OHHD methods yielded higher EO quantities and required less time. This improvement can be attributed to UAHD leveraging the principle of acoustic cavitation, which facilitates the breakdown of plant cell walls. This breakdown promotes solvent penetration, mass transfer, and the rapid release of bioactive compounds from cells to the collector, as discussed by Singla and Sit (2021). In

conclusion, the proposed UAHD method proved more effective in enhancing plant EO output.

The OHHD technique stands out by efficiently converting all input electrical energy into thermal energy. In comparison to traditional methods, OHHD demonstrated lower energy consumption to achieve the same quantity of EO. This characteristic positions OHHD as a "green technology" in the market, attributable to its notable features such as the absence of organic solvents, the elimination of radiation leakage risks, low energy consumption values, and a reduced CO_2 emission rate.

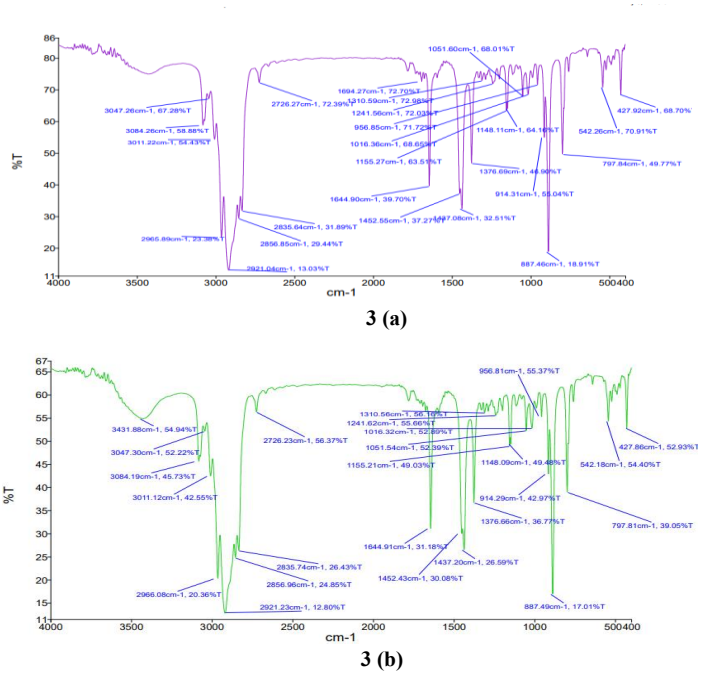
Characterization of extracted essential oil

Characterization of EO using FT-IR Spectroscopy

The FTIR spectra of all analysed samples exhibited comparable results concerning the presence of various functional groups. As illustrated in Figure 3 (a & b) and summarized in Table 5, the application of ultrasonic pre-treatment did not significantly alter the composition of essential oil (EO) extracted from citrus peels. This indicates that the fundamental chemical structure of the EO components remained largely unaffected by the ultrasonic treatment.

A distinct and prominent absorption band was observed at 887 cm^{-1}, which corresponds to the out-of-plane bending of the terminal methyl (-CH_3) group in limonene, a major constituent of citrus essential oils. Additionally, intense absorption peaks were detected at 763 cm^{-1} and 757 cm^{-1} in the spectra of bitter orange, sweet orange, and lemon EOs. These bands are characteristic of the ring deformation mode of limonene, aligning with previous findings reported by **Schulz and Baranska (2007)**. Furthermore, the FTIR spectra revealed additional absorption bands at 1694 cm^{-1} and 1644 cm^{-1}, which were attributed to the stretching vibrations of the carbonyl (C=O) functional group. The presence of these bands suggests the occurrence of compounds such as aldehydes, ketones, or esters in the essential oil composition. The spectral region spanning from 1452 cm^{-1} to 1376 cm^{-1} exhibited bending vibrations associated with methyl (-CH_3) groups, further confirming the presence of terpenoid structures.

In addition, absorption bands detected between 1241 cm^{-1} and 1016 cm^{-1} indicated the stretching vibrations of the carbon-oxygen (C-O) bond. This range is characteristic of various oxygen-containing functional groups, including alcohols, ethers, esters, carboxylic acids, and anhydrides, which are commonly found in essential oil constituents. The FTIR spectra of pure lemon EO, which belongs to the same botanical family as mandarin, further supported these observations. The presence of key terpenoid components was confirmed by several distinct absorption bands, including a C-H stretching vibration at approximately 2900 cm^{-1}, a C=O stretching vibration at around 1700 cm^{-1}, a broad O-H stretching band near 3400 cm^{-1}, and a C-O stretching vibration centered at approximately 1100 cm^{-1}. These findings align with the spectral characteristics reported by Elzey (2016), reinforcing the presence of terpenoid compounds in citrus essential oils.



3 (a)

3 (b)

Figure 3 FT-IR spectrum of Kinnow peel oil extracted by UHHD 3 (a) and OHHD 3 (b).

Table 5 Infrared vibrations of UAHD and OHHD extracted essential oil

Link Present	X (cm^{-1})
N-H primary and secondary amines and amide stretch,	3435.68
N-H primary and secondary amines and amide stretch, Alkenes stretch	3084.25
N-H primary and secondary amines and amide stretch, C-H Alkenes stretch	3047.26
N-H primary and secondary amines and amide stretch	3011.17
C-H Alkanes stretch	2965.96
C-H Alkanes stretch	2921.31
C-H Alkanes stretch	2856.91
C-H Alkanes stretch	2835.72
C-H Aldehyde	2726.26
C=O Amide	1694.21
C=C Alkene	1644.89
-CH_3 bend	1452.45
-CH_3 bend	1437.17
-CH_3 bend	1376.67
C-O Alcohols, ether, ester, carboxylic acid, anhydrides	1241.42
C-O Alcohols, ether, ester, carboxylic acid, anhydrides	1155.21
C-O Alcohols, ether, ester, carboxylic acid, anhydrides	1148.12
C-O Alcohols, ether, ester, carboxylic acid, anhydrides	1051.54
C-O Alcohols, ether, ester, carboxylic acid, anhydrides	1016.36
Aromatic out-of-plane bend	956.83
Aromatic out-of-plane bend	914.28
Aromatic out-of-plane bend	887.48
Aromatic out-of-plane bend	797.81

Characterization of EO using GC-MS

The GC-MS analysis results facilitated the identification of various substances present in the EOs extracted from different treatments. Peaks observed in the GC-MS chromatogram (Figure 4) indicated the presence of multiple compounds.

The qualitative comparison of the extracted EOs involved the identification of various compounds, while the yield value served as an indicator for quantitative comparison. Table 6 demonstrates that similar active compounds were detected in EOs extracted through OHHD and UAHD methods. The predominant compounds in kinnow peel EO were identified as D-Limonene and Carvacrol. The concentration of D-limonene was found slightly higher (76.09%) in the EO obtained through UAHD compared to the EO obtained with OHHD assistance (70.95%). Conversely, the concentration of carvacrol (28.68%) was found higher in the EO obtained through OHHD assistance compared to the EO obtained with UAHD (23.67%). This aligns with findings reported by **Michaelakis *et al.* (2009)** regarding the constituents in EO from citrus species. Similarly, **Gogoi *et al.* (2025)** showed that limonene was the most abundant constituent in EO extracted from orange peel.

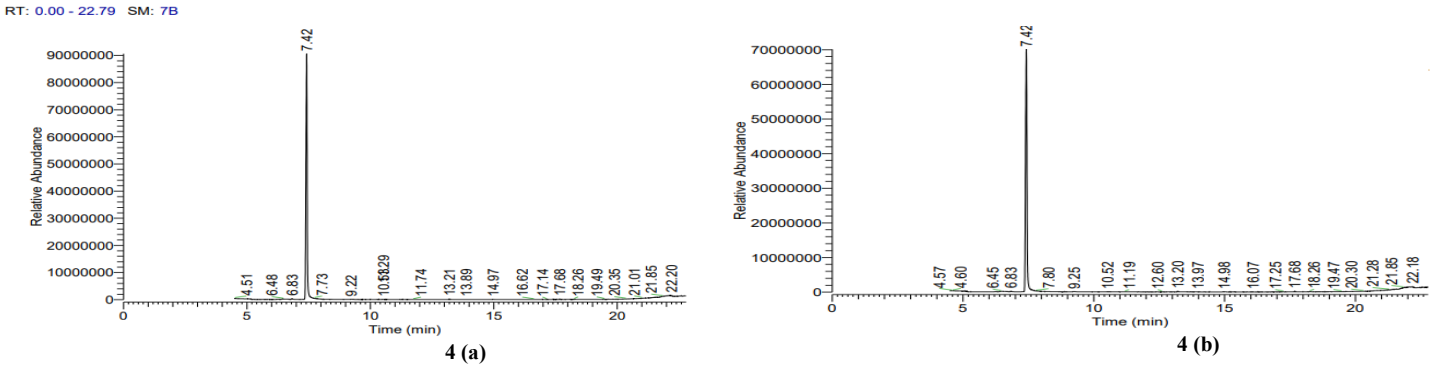


Figure 4 Chromatograph of extracted essential oil by GC MS, 4 (a) UAHD, 4 (b) OHHD

Table 6 Chemical composition of essential oil obtained from kinnow peels by UAHD and OHHD using GC-MS

RT	Compound name	Peak Area (%) UAHD	Peak Area (%) OHHD
7.42	D-Limonene	76.09	70.95
9.23	2,4-Imidazolidinedione	0.09	-
9.23	Boldenone, TMS derivative	0.22	-
13.20	Acetic acid, 3-acetoxy-6-(2-cyanovinyl)-3a,6-dim ethyl-2,3,3a,4,5,5a,6,9,9a,9b-decah ydro-1H-cyclopenta[a]naphthalen-7 -ylmethyl ester	0.22	0.20
13.21	1,3,5-Benzetriol, 3TMS derivative	0.22	-
13.21	1H-Indole-2,3-dione, 5-chloro-1-(trimethylsilyl)-, 3-[O-(trimethylsilyl)oxime]	-	-
13.21	Phosphonoacetic Acid, 3TMS derivative	-	0.24
17.68	Benzoic acid, 4-methyl-2-trimethylsilyloxy-, trimethylsilyl ester	0.18	0.15
17.68	2,4-Dihydroxyacetophenone, 2TMS derivative	0.14	0.15
22.20	Carvacrol, TBDMS derivative	23.67	28.68

RT – Retention time, (-) not detected

CONCLUSION

The current study concludes that the pretreatment methods involving ultrasonic assistance and ohmic heating can serve as modification techniques for achieving faster and more efficient extractions, leading to potential energy and cost savings. Importantly, these techniques did not significantly alter the chemical composition of the extracted substances. In both conventional and novel extraction methods, the yield was influenced by factors such as time, temperature, power, and solute-to-solvent ratio. Notably, solvent concentration played a significant role in the conventional extraction process. The interaction between treatment time and temperature had a discernible impact on the precipitation yield. The use of Response Surface Methodology (RSM) was effective in optimizing extraction conditions. Future studies could explore the detection of ultrasonic extracts in the terahertz range.

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