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Optimization of citrus peels D-limonene extraction using solvent-free microwave green technology

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ABSTRACT

Attention is presently drawn to the development of a new and green alternative technique for the extraction of essential oil from citrus plant materials. This study was aimed at the extracting essential oil from orange and lemon peels using solvent-free microwave method. This process uses microwave-assisted hydro-diffusion technique to extract essential oil from citrus peels. Response surface methodology was used to investigate the effect of microwave power (200–1,000 W) and extraction time (10–40 min) on the essential oil yield. The oil extracted was characterized using Fourier transform infrared radiation (FTIR) and Gas chromatography–mass spectrometry analysis to determine the functional groups and chemical components present, respectively. The optimum yield of extract from orange and lemon peels were 3.7 and 2.0%, respectively at corresponding power of 1,000 W and time of 10 min. The analysis of variance results showed that the resulting models for both orange and lemon peels were significant and microwave power had greater influence on the extraction processes at both linear and quadratic levels. The FTIR analysis revealed prominent functional groups of alkenes that majorly constitute limonene compound at 1,642 and 1,643 cm^{-1} for orange and lemon peels, respectively. The present process permits fast and efficient extraction, avoids water and solvent consumption, and allows substantial energy savings.

KEYWORDS

Citrus; essential oil; extraction; microwave; optimization; solvent-free

Introduction

Extraction of oil from solid plant matrices is used for the production of important and useful products such as limonene, β-mycene, terpineol, α-pinene among other useful compounds. Several techniques such as conventional (soxhlet extraction, hydrodistillation, maceration) and unconventional (ultrasound, microwave-assisted, pulsed electric field assisted, pressurized liquid, enzyme-assisted, and supercritical) methods have been used for extracting valuable and health beneficial compounds from plant materials (Bousbia et al., 2009; Périno-Issartier et al., 2013). The choice of a method for the extraction of these compounds is related to the chemical composition of the resulting product (Costa et al., 2014; Lopresto et al., 2014). Solvent-assisted extraction techniques has received wide application for a number of decades for the extraction of stereo-chemical types of molecules for specific application (Azmir et al., 2013). Water- and petroleum-based compounds (*n*-hexane, dichloromethane, diethyl ether, and ethyl acetate) are among the commonest solvents used for extraction of plant-based essential oils (Chemat and Esveld, 2013; Périno-Issartier et al., 2013). However, the use of petroleum-based solvents for essential oil extraction is characterized with high energy and time consumption, lower product yield, and generation of hazardous by products (Cheng et al., 2014).

To curb some of these challenges, alternative innovative techniques such as ultrasonic assisted (Cheng et al., 2014), microwave extraction process (Chemat and Esveld, 2013), and controlled pressure drop (Azmir et al., 2013) methods have been recently investigated. Among these methods mentioned above, solvent-free microwave (SFM)-assisted extraction technique has been found to have high potential of overcoming the drawbacks associated with other conventional methods.

The solvent-free microwave extraction (SFME) method is an emerging solvent extraction

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technology which is relatively simple (Li et al., 2012). The process SFM extraction (SFME) consists of the microwave-assisted dry distillation of a fresh plant matrix without addition of water or any organic solvent. It differs from the modified hydro-distillation (HD) which requires large volume of water. The process is characterized with selective heating of the water content of the plant material, which results into the swelling of the plant tissue and eventual burst of gland to release oil (Filly et al., 2014). This process thus frees the essential oil from the plant matrix and further purified through azeotropic distillation (Farhat et al., 2010). The excess water is refluxed back into the extraction vessel to restore the original water to the plant material. This process has been used to extract compounds from plant samples, food, and plastics. It is a process characterized with high extraction rate and reduced solvent consumption (Azmir et al., 2013). The SFME has been found to enhance efficiency of energy transfer, promote effective heating, reduce extraction time, increase extract yield, improve extract quality and at low operation cost (Li et al., 2012).

The preponderances of citrus as the largest grown fruit crop, the healthiest and most consumed beverages globally has been highlighted (Cheong et al., 2012; Farhat et al., 2011). The commonest examples of citrus crops are orange, lime and lemon. Orange accounts for over 60% of the entire citrus crop. It is reported that about 45% of an orange bulk are its peel which is usually discarded as waste (Yeoh et al., 2008). According to Farhat et al. (2011) essential oil from orange peels are predominantly used to add desired aroma to perfumes, cakes, ice creams, carbonated soft drinks, and room air fresheners. Besides citrus fruits have high content of vitamin C, taste, and the peels contains some natural chemicals which are desirable in cosmetics and pharmaceutical industries. d-Limonene is one of such chemicals extracted from citrus peel which has potentials for many applications depending on plant matrices and method of extraction (Veillet et al., 2010; Virot et al., 2008).

The extraction of d-limonene from citrus peels have been reportedly performed using several approaches ranging from traditional soxhlet method (Lopresto et al., 2014), microwave steam diffusion mechanism (Farhat et al., 2011), solvent extraction method (Cheong et al., 2012), optimization of conventional and nonconventional extraction methods (Li et al., 2012) and several other methods as found in literatures (Azmir et al., 2013). To the best of our knowledge, the application of the emerging green solvent-free microwave extraction technology for the recovery of d-limonene from citrus crops is seldom found in literature.

The aim of the present research is to study the optimization of solvent-free microwave technology for the extraction of essential oil from orange and lemon peels. The effect of essential operation factors such as microwave power and time were studied using the response surface methodology (RSM).

Materials and methods

Materials

The fresh orange and lemon peels were obtained locally from Ultra-modern market in Minna, Nigeria. A house hold microwave oven was modified and used for this research.

Design of experiment

A two-factor user defined (UD) combined with the RSM was applied to determine the best combination of process variables for the essential oil extraction from orange and lemon peels. The independent variables studied were the microwave irradiation power (X_1: 200–1,000 W) and extraction time (X_2: 10–40 min), while the response variable was the yield of oil extracted. The ranges of the two factors were selected based on findings from previous researchers (Costa et al., 2014). Seventeen random experiments were generated by the software to fit the full linear equation model given:

$$Y = \beta_o + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k} \beta_{ij} X_i X_j \quad (1)$$

where Y represents the response variable (yield of extracted oil), β_o, β_i, β_{ii}, and β_{ij} are the regression coefficients of variables for intercept, linearity, square, and interaction, respectively. X_i and X_j

are the levels of the independent coded variables. The actual variables were transferred to a range between −1 and 1 for the evaluation of the factors.

The data obtained from SFME tests were analyzed statistically with a response surface analysis procedure (Design-Expert 7.0.0 trial, state-ease, Inc., Minneapolis, MN, USA). Analysis of variance was performed to calculate and simulate the optimal conditions for the SFME of orange and lemon peels essential oil.

Essential oil recovery

The house-hold microwave was perforated at the top to enhance connection of pipes. The inlet pipe was connected to a conical flask (reactor) placed inside the microwave oven while the other end (outlet pipe) was connected to a condenser. The condenser was used to condense vapor (steam plus oil) from the sample in the reactor to liquid. Connected to the condenser was a pipe used to convey the hydrosol to a reagent bottle which was used for further separation of the content into two distinct layers (water and oil). Schematic diagram of the experimental set up is shown in Figure 1.

About 150 g each of orange and lemon peel samples were weighed out and transferred into different 100 mL round bottom flasks without any solvent added. At different times, each of the round bottom flasks were placed in a modified domestic microwave oven (Euro smart 2000) in 1,000 W power output with adjustable value of 200 W interval. The microwave power and time process parameters were varied as reproduced by the experimental design matrix RSM software. The essential oil yield was maximized by optimizing the experimental SFME variables.

The oil extracted was separated using a separating funnel and dried under anhydrous sodium sulfate, then stored in a freezer until further analysis was performed. The yield was gravimetrically determined as a percentage ratio of the mass of the extracted essential oil to the mass of the fresh orange and lemon peels introduced initially to the extraction vessel as expressed in Equation (2)

$$\text{Extraction yield of essential oil}(\%) = \frac{\text{Mass of extracted essential oil}}{\text{Mass of dried material}} \times 100 \quad (2)$$

Characterization of the extract

Functional groups present in the essential oils were determined by Fourier transform infrared radiation (FTIR). Two thin (0.1–0.5 mm) film potassium bromide discs were made from same crystals. A drop of the extracted oil was fixed on the surface of one of the discs and the second one was placed on top to spread the oil into a thin film. The spectrum of the embedded oil was taken by placing the discs in an FTIR machine (FTIR-8400S). Tetrachloromethane was used to keep the apparatus used clean.

Gas chromatography–mass spectrometry (GC–MS) was used to analyze the chemical compounds that constitute the oil extracted. The essential oil was toted out on a Hewlett-Packard gas chromatograph Model 5890, paired to a Hewlett-Packard MS model5989B, assembled with

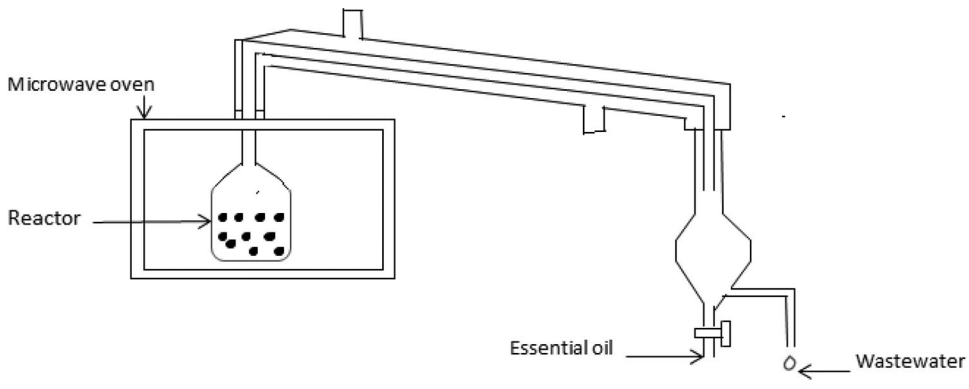

Figure 1. Solvent free microwave extraction set up.

an HP1 MS column (12 m × 0.2 mm, 0.33 µm); programming from 60 (5 min) to 300°C at 5°C min^{-1}, 5 min hold; the carrier gas was helium at 1.0 ml min^{-1} flow rate; injection in split mode was (60:1); injector and detector temperatures were 225 and 300°C, respectively. The electron impact mass spectrometry (EIMS) mode was at 70 eV; electron multiplier was 2,500 V; ion source temperature was 180°C; mass spectral data were acquired in the scan mode in the *m*/*z* range 33–450 (Périno-Issartier et al., 2013). The essential oils constituents were classified by correlating their mass fragmentation patterns with those of the accessible reference. Identification was also established by electronic Wiley and NIST mass spectral data base. The retention indices (RI) of the volatile oils constituents were determined relative to the retention times of series of hydrocarbons.

Results and discussion

Statistical analysis and model fitting

Multiple regression analysis was used to perform the model fitting of the extraction process. The second-order polynomial quadratic model was used to describe and express the yield of both orange and lemon peels essential oil as a function of the two independent variables (microwave power and extraction or irradiation time) as depicted in Equation (3) (orange peels oil) and Equation (4) (lemon peels oil). Equations (3) and (4) were developed to show the relationship between the essential oil extraction yield as dependent variable with the significant independent terms (Li et al., 2012). The effect of variables such as time and microwave power on the response were tested for significance by analysis of variance (ANOVA). The ANOVA for both orange and lemon peels extraction processes are presented in Table 1.

$$Y_{\text{orange}} = 1.86 + 1.65X_1 + 0.65X_2 - 0.72X_1X_2 \quad (3)$$

$$Y_{\text{lemon}} = 2.56 + 0.17X_1 + 0.27X_2 - 1.06X_1^2 \quad (4)$$

The significance of each coefficient was determined by *F*-value and the associated probability *P*-value such that large magnitude of *F*-value and a threshold *P*-value ($P > 0.05$) meant a higher significance of the corresponding coefficient. The two parameters both had influence on the extraction of essential oil using the two precursors (orange and lemon peels) as revealed by the *P*-values obtained from ANOVA shown in Table 1. The ANOVA of both oils extraction process revealed that microwave power (*F*-values of 20.50–97.16) had greater influence on the oil yield than the extraction time (*F*-values of 6.87–5.272) due to its larger *F*-values with corresponding threshold *P*-values ($P < 0.0001$). The significance of each coefficient was determined by large magnitude of *F*-value and smaller value of Prob. > *F* less than 0.05 connoting higher fitness of the corresponding coefficient (Hameed et al., 2009). The result revealed that an appropriate microwave power is important to ensure adequate and quick extraction of the essential oil.

Table 1. Experimental design and responses of the orange and lemon peels essential oil extraction.

Run	Microwave power (W)	Extraction (min)	%Yield of orange	%Yield of lemon
1	200	10	0	0
2	1,000	10	3.7	2.0
3	200	40	0	0
4	1,000	40	3.6	2.3
5	600	40	2	1.8
6	200	25	0	0
7	1,000	25	2.2	2.0
8	600	17.5	1.9	1.8
9	400	32.5	1.7	1.62
10	800	25	1.7	1.6
11	400	17.5	1.4	1.25
12	800	32.5	3.9	2.5
13	600	25	1.4	1.2
14	400	25	1.4	1.2
15	800	17.5	2.5	2.1
16	600	10	1.0	0.9
17	600	32.5	2.3	2.0

The models were modified by removing the insignificant terms (interaction of microwave power and time X_1X_2; quadratic effect of extraction time X_2^2) as specified by their *P*-values which were between 0.2882 and 0.542 (Table 1). The significant terms of the two models [Equations (3) and (4)] were X_1 (*P*-value of 0.0001), X_2 (*P*-value < 0.043), and X_1^2 (*P*-value < 0.02) connoting the microwave power, extraction time, and quadratic effect of the microwave power, respectively.

The optimum microwave power, extraction time and the models' correlation coefficients for extraction of the essential oil were 1,000 W, 10 min, R^2 0.813 for orange peels precursor, and 1,000 W, 10 min, and 0.928 for lemon peels precursor, respectively. The correlation coefficient R^2 (0.813 and

0.928) of the two model Equations (3) and (4) signified that the extraction data could be explained above 80–90% by the response surface models (Lopresto et al., 2014). The extraction of the essential oil was validated three times and the result showed a mean percentage error of 1.25 and 0.987% for orange and lemon peels, respectively.

The three-dimensional response surface profiles for orange and lemon peels extraction are presented in Figure 2. Microwave power plays a significant role on oil yield during solvent free extraction. It was observed that for both low (10 min) and high (40 min) extraction time, the increase in microwave power from 200 to 1,000 W led to a corresponding increase in oil yield from 0–3.7 g to 0–2.0 g for the orange and lemon peels, respectively. It is very obvious that the extraction efficiency increased to 2.4 and 1.33% for orange and lemon peels, respectively.

The result shows that as the extraction time increases from 10 to 40 min at a microwave power of 1,000 W, the orange peel oil yield decreased from 3.7 to 3.6 g. This showed a reduction in the extraction efficiency from 2.5 to 2.4% after additional 30 min. The extraction efficiency in this study is high when compared to 1.48% reported for sweet orange peels by Kamaliroosta et al. (2016). In the case of lemon peels, the increase in extraction time from 10 to 40 min for the same microwave power (1,000 W) resulted into an insignificant increase in yield and extraction efficiency. The oil yield only increased from 2.0 to 2.3 g with a corresponding extraction efficiency of 1.33 and 1.53%, respectively. Intuitively, a 0.2% increase in extraction efficiency for additional 30 min as against 1.33% obtained after 10 min is not economical. The finding in this study shows appreciable consistency with the work of Li et al. (2012) who reported the insignificant effect of extraction time increase during microwave extraction of essential oil from *Dryopteris fragrans*. It is important to add that the extraction efficiency of 1.33% obtained for lemon peel extract is appreciably higher than 1% by reported by Boughendjioua and Djeddi (2017).

A bird eye view of the synergetic effect of the two parameters revealed that the simultaneous increase in both parameters (microwave power and extraction time) do not have significant effect on the essential oil recovery, rather lower extraction time (10 min) and higher microwave power (1,000 W) are essentially needed to achieve maximum yield of the oil. The three-dimensional response surface profiles has further supported the ANOVA results in Table 1. Costa et al. (2014) has reported that higher microwave extraction time may promote volatilization and decomposition of active ingredients in the oil.

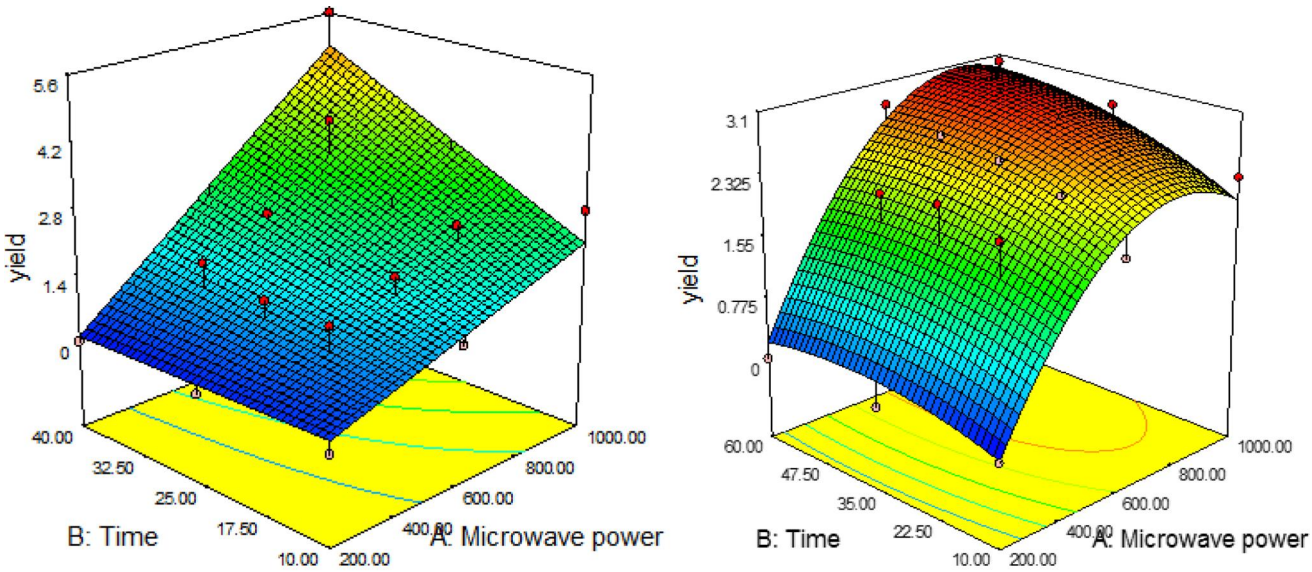

Figure 2. The three-dimensional response surface plots of (a) orange (b) lemon peels.

Table 2. Analysis of variance.

Source	Sum of squares	DF	Mean square	*F*-value	*P*-value
Orange peel					
Model	25.86	5	8.62	18.87	<0.0001
X_1 (power)	20.50	1	20.50	44.88	<0.0001
X_2 (time)	3.14	1	3.14	6.87	0.0212
X_1X_2	2.22	1	2.22	2.87	0.459
X_1^2	4.24	1	4.24	16.54	0.0117
X_2^2	0.21	1	0.21	1.097	0.542
Lemon peel					
Model	15.000	5	3.000	28.229	<0.0001
X_1 (power)	10.325	1	10.32	97.155	<0.0001
X_2 (time)	0.560	1	0.560	5.272	0.0423
X_1X_2	0.134	1	0.132	1.245	0.2882
X_1^2	3.127	1	3.127	29.426	0.0002
X_2^2	0.1137	1	0.114	1.0698	0.3232

Fourier transform infrared analysis of orange and lemon peels oils

The essential oils extracted from orange and lemon peels were both composed of numerous functional groups as revealed by the FTIR analysis (figures not shown). Prominent peaks attributed to limonene (a *mono*-terpene) were detected at different identifiable band positions outside the finger print regions that are consistent with the building blocks of limonene. Some of such functional groups are the C-H stretch of aromatics found at 809.18 and 3067.88 cm^{-1} peaks. The C-H stretch of alkane functional groups at 2923.22–2933.83 cm^{-1} peaks. Aromatics amines C-N stretch peaks at 1277.88 and 1275.95 cm^{-1}, aliphatic amines C-N stretch at 1130.32 and 1142.86 cm^{-1}, alkenes C=C stretch at 1642.44 and 1643.41 cm^{-1} for orange and lemon oils, respectively. The functional groups identified in the extracted oils are in conformity with the structure of limonene reported by Ezejiofor et al. (2011).

The chemical components of the essential oil extracted under optimal solvent-free microwave extraction condition from orange and lemon peels were characterized using GC–MS, the analysis results are presented in Table 2.

Several chemical components were identified but only quantum compositions were analyzed as presented (Table 2) which are similar to the constituents of essential oil from citrus peels and other plants reported by other researchers (Bousbia et al., 2009; Pavela, 2015). Limonene and β-mycene constitutes the highest compositions from the two precursors (orange and lemon peels) oils analyzed. The orange and lemon peels oils extracted had 28.88 and 14.87%, and 44.74 and 19.11% of limonene and β-mycene content, respectively. This result is in agreement with that obtained by Lopresto et al. (2014) and Bourgou et al. (2012) asserting that limonene and β-mycene are the most abundant components in citrus peels.

The percentage composition of limonene shows appreciable consistency with 21–23% reported by Filly et al. (2014) but considerably lower than 58–67% reported by Ferhat et al. (2007). This discrepancy in value observed could be attributed to the chemical and biotype of the plant, the climatic conditions as well as the extractive process (Costa et al., 2014). More so chemical composition may vary in the starting material, being influenced by plant health, growth stage, habitat including climatic, edaphic factors, as well as harvest time (Azmir et al., 2013). These environmental reasons may have contributed to the variation of essential oil yield obtained from this study as compared with other researchers' as presented in Table 3. This research revealed a higher 2 and 3.7% oil yield obtained from lemon and orange precursor respectively compared with Li et al. (2012) and Farhat et al. (2011). However the percentage oil yield obtained from lemon peels reported by

Table 3. Percentage yield of essential oil from different precursors.

Precursor	Extraction method	Extraction time (min)	Yield (%)	Reference
Waste lemon	Soxhlet extraction	240	0.95	Lopresto et al. (2014)
Waste lemon	High pressure-high temperature extraction	30	2.97	Lopresto et al. (2014)
Fresh Rosemary plant	Solvent free microwave extraction	30	0.54	Filly et al. (2014)
Fresh Rosemary plant	Hydrodistillation	90	0.57	Filly et al. (2014)
Orange peels	Microwave steam distillation	12	1.54	Farhat et al. (2011)
Orange peels	Conventional steam distillation	40	1.51	Farhat et al. (2011)
Dryopteris fragans	Solvent-free microwave extraction	34	0.34	Li et al. (2012)
Dryopteris fragans	Hydrodistillation	300	0.29	Li et al. (2012)
Orange peels	Solvent-free microwave extraction	10	3.7	This study
Lemon peels	Solvent-free microwave extraction	10	2.0	This study

Table 4. The GC–MS analysis of orange and lemon peels oils.

	Orange-peels oil	Lemon-peels oil
Components	% Composition	% Composition
α-Pinene	4.64	5.22
β-Mycene	14.87	19.11
Limonene	28.88	44.74
1-Ocatanol	1.61	1.25
Dodecatetrane	0.44	0.35
Dodecateranal	0.48	0.51
Dodecanal	0.34	0.36
Hexadeconoic acid	23.75	1.49
3-Cyclohexen-1-ol	0.62	0.48
Others	24.37	26.49

Lopresto et al. (2014) was higher than that obtained from this research (Table 3).

The chemical constituents of the orange peel oil extracted were determined using GC–MS analysis and the result is presented in Table 4. The result revealed preponderances of oxygenated sesquiterpenes (15.37%) which is considered high (15–20%) in oils obtained by SFME. However, Li et al. (2012) reported that the content of oxygenated sesquiterpenes of the oil obtained by SFME is low (2–3%) for *Cuminum cyminum* but observed high content for zanthozylumungeanums using the microwave assisted extraction. It therefore suggests that obtaining oxygenated sesquiterpenes in essential oil is dependent on the specie or type of precursor rather than the extraction method used. The GC–MS analysis of the oils extracted in this study revealed higher composition of oxygenated components in orange peels oil than the lemon peel oil. This may be responsible for orange peel oil low limonene content when compared with that of the lemon peel oil extracted.

Conclusion

The study has successfully shown the potential of solvent-free microwave extraction method as a green technology for d-limonene extraction from citrus. The study revealed that microwave power had more significant effect on recovery of d-limonene than the extraction time. This was brought to lime light by the significant percentage increase of oil yield observed with corresponding microwave power increase; but increase of extraction time did not transmit to any pronounced oil yield increase. Optimum oil yield from the citrus (orange and lemon) peels were obtained at 1,000 W and 10 min. The FTIR analysis of the sample confirmed the presence of the active ingredient (d-limonene) presence while the GC–MS result revealed the higher oxygenate compounds presence in orange peel oil than that of lemon peel's which may have been responsible for its low oil yield.

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