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Extraction and Characterization of Essential oil of Sweet Lime (*Citrus Limetta Risso*) peel using Microwave-assisted Hydrodistillation

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Abstract

In this study, essential oil of Sweet lime peel, which is a waste in juice industry, was extracted using microwave assisted hydrodistillation and characterization of essential oil has been performed using GC-MS. Extraction efficiencies of extraction process was investigated by varying three factors, time of extraction, microwave power, particle size and dryness period. The results showed that all the concerned factors have a positive effect on the yield. Oil yield is proportional to extraction time and microwave power. Higher yield can be obtained with smaller particle size and dried sample. The essential oil of sweet lime peel is mainly comprised of d-limonene (on average 78.30%). oil is mixture bioactive isomers. microwave assisted hydrodistillation is successfully applied in sweet lime peel processing.

Keywords: Sweet lime, essential oil, Microwave extraction, characterization.

Introduction

The Sweet lime (*Citrus limetta risso*), commonly known as mousambi Fruit in India, is a native plant of Asia, which is best cultivated in India, China, southern Japan, Vietnam, Malaysia, Indonesia and Thailand. The mousambi fruit is commonly eaten fresh or made as juice which is rich source of vitamin C and instant energy[1]. Citrus juices are the most common among the fruit juices around the world and constitute a major portion of the food industry[2]. Sweet lime peel is source of flavonoids, pectin and essential oil[3]. The Citrus peel oils have a strong and desirable aroma with refreshing effect. They have been used as flavoring in foods, beverages and pharmaceutical products. They also have been used as fragrance in perfumes, cosmetic and aromatherapy. Moreover, citrus essential oils have been recognized as safe due to their wide spectrum of biological activities such as antimicrobial, antioxidant anti-inflammatory and anxiolytic[4,5,6].

In essential oil industry, Steam distillation is the primary method to extract the essential oil from herbs, spices, medicinal and aromatic plants for commercial product[7], many other methods can be used for extraction of essential oils from sweet lime peel, e.g. hydro-distillation (HD)[8], solvent extraction[9]. Since last decades, many other new techniques have been developed with greater advantages over traditional methods e.g. supercritical fluid extraction, ultrasonic and microwave extraction[10]. Today, microwave extraction is well known to selective and volumetric heating of target, less extraction time, high product quality[11]. This technology finds application from analytical laboratory systems to industrial extractor and various other fields of chemical industry like; chemical synthesis[12,13], drying[14] and organic extraction[15].

In this present work, extraction of Sweet lime peel essential oil is done by microwave-assisted hydrodistillation (MAHD) method. Effects of process parameters like, extraction time, power input of microwave and different drying durationon extraction efficiencies was investigated on percentage yield of sweet lime peel oil. Essential oil was characterized using GC-MS analysis.

Material and Methods

Raw materials: Sweet lime peels were collected from juice centers, located in different place of Gwalior city. The peels were cut in halves, green color and 2-4 mm in thickness. They were assumed fresh at the time of collection. Double distilled water was used as solvent throughout the experimentation. Anhydrous sodium sulphate was purchased from Merk lab.

Experimental setup: Microwave-assisted hydro-distillation (MAHD) was performed at atmospheric pressure with a microwave frequency of 2.54 G Hz using a household microwave oven which was modified to facilitate the hydro distillation as shown in figure-1. This was a multimode microwave reactor with a maximum delivered output power of 800 W, and input power of 1200W, having the voltage supply of 230 volt and with total capacity 18.5 liters. Three pieces of glassware were fabricated by J-Sil, Agra, Riser, Condenser and separating funnel to fit the microwave modifications and to facilitate the oil extraction process.

Figure-1
MAHD reactor setup

Extraction of essential oil: In MAHD process, 100g sample of fresh/dried peels of Sweet lime was charged in the still flask and the 500 ml of water was added and the apparatus was assembled as shown in the figure-1. The oil extracted from the sample by microwave radiation energy was cooled down and the condensate was channeled out to a separating funnel. The condensate was then allowed to settle for a few minutes, before the oil was collected. Next, the oil collected was weighed to calculate the yield. The same steps were repeated for each of the samples with different power, dryness and time periods. The extraction time was 120 min.

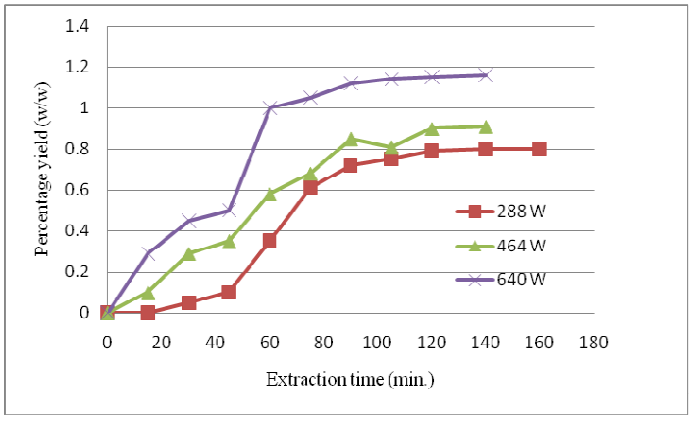
Light yellow colored, apparently colorless oil, with a somewhat lemon like and pleasant odor, was obtained.The extracted oil was separated from distillate and dried over the minimum amount of anhydrous sodium sulfate to remove traces of moisture[16]. The percentage oil yield is expressed as follows:

$$\text{Oil yield} = \frac{\text{mass of extracted oil}}{\text{mass of sample}} \times 100 \quad (1)$$

Characterization of essential oil: Chemical characterization of the essential oil was performed on a SHIMADZU gas chromatograph-mass spectrometer (Model QP 2010 Plus, Shimadzu Corporation, Japan) using a capillary column Rtx-5 MS (30 meter X 0.25 mm id. X 0.25 micrometer Film thickness). The flame ionization detector (FID) was maintained at 320 °C, and the injector temperature was 250 °C. The oven temperature was programmed to increase from 50 to 280 °C at a rate of 3 °C/min. The carrier gas was hydrogen at a flow rate of 1.21 ml/min with a split ratio of 100. Identification of compounds was based on comparisons with mass spectra from the literature (NIST, US National Institute of Standards and Technology).

Results and Discussion

Effect of microwave power level and extraction time on essential oil yield with fresh sample: Effects of microwave power and extraction time on essential oil yield, using the fresh sample is shown in figure 2. Fresh samples of same weight, 100g. each were extracted with three microwave power levels 288, 464 and 640 W for maximum of 150min and with 500 ml of distill water. During the experiment, the water is continuously refluxed in the flask to avoid the burning of sample.



Figure–2
Variation of oil yield with Fresh sample at different power

levels

It can be seen from the figure-2, that oil yield increase with both higher power levels and extraction time. Yield of oil increase rapidly up to 80 minutes and then slowed down. At Medium low power level maximum yield was 0.87% in 140 minutes, at Medium power level it was 0.91% in 120 minutes and at Medium high power level, highest yield 1.16 % resulted in 120 minutes. It can be concluded that microwave power is directly proportional to extraction which is in agreement with the previously published literature[17].

It was also seen thatmicrowave power is inversely proportional to total time for the extraction (figure-2 and figure-3). According to Chen and Spiro[18] at higher power levels, the microwaves (i.e., the radiations) can be absorbed by water more intensively and as a result the disruption rate of cellular texture and release of essential oils also increase. Using scanning electron microscopy, Lucchesi *et al.* reported that cells undergone MAE resulted in faster rupture than those undergone the conventional extraction[19].

Effect of microwave power level and extraction time on essential oil yield with dried sample: Effects of microwave power and extraction time on essential oil yield, using the fresh sample is shown in figure-3. Dried samples of same weight, 50g each were extracted with two microwave power levels 464 and 640 W for maximum of 140min and with 1000 ml of distill water. During the experiment, the water is continuously refluxed in the flask to avoid the burning of sample.

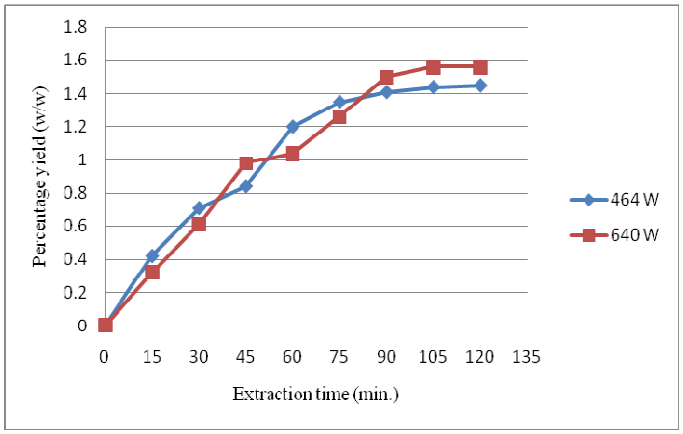
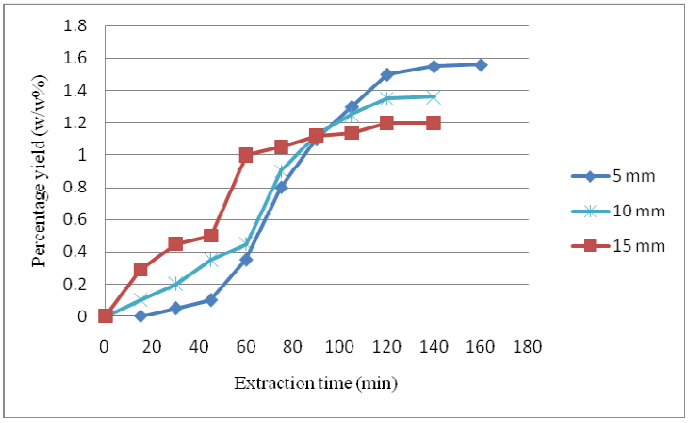


Figure-3
Variation of oil yield with dried sample

An experiment with dried sample was resulted in fast recovery of oil from sample. It took only 90 minutes for complete recovery of oil at both the power levels; 464 and 640 W with same effects of microwave power level and extraction time on yield as was with fresh samples. At 464 W power levels yield was 1.41% in 90 minutes and increased very slightly up to 1.45% for next 30 minutes. Therefore 90 minutes was considered as final extraction time. Higher power level resulted in higher oil yield but effect of extraction time was not much signification on power level.

Effect of particle size of sample on oil yield: To observe the effect of size of particle on essential oil yield, a study was conducted with taking three sample sizes; 5mm, 10 mm and 1.5 mm. figure-4 showed the variation in oil yield with changing particle size of sample. From previous experimental run, high microwave power level was selected for this study, as we can see at higher power level most of the oil extracted comparatively in short time with highest oil yield. It was also seen from previous studies that dried sample gave more yield comparatively, therefore, dry sample was chosen for this study to ensure maximum yield of oil.



Figure–4
Variation of oil yield with size of peel sample

It can be seen from figure-4, that the oil yield inversely proportional to the particle size, yield decreases as the particle size increases. Initially, up to 100 minutes larger particle gave the high yields but after minutes yield decrease. The plausible explanation the observed phenomena is that sample with small particle size provides larger direct contact to solvent, which makes easier mass transfer of oil from deep of particle cell to the water solvent. With increasing particle size, added resistance to mass transfer, makes it difficult to transport of oil from oil cells particle to solvent liquid. Similar trends were found in the study of orange peel and mandarin peel oil extraction[20,21].

Chemical composition of essential oil: Table-1 shows the results of GC-MS of sweet lime essential oil. Total of 29 compounds were identified in mass spectrometry. limonene is the main component of the citrus peel oils, which was found to appear in concentrations of 40% to 95% in oranges. In present work, compounds identified from GC-MS showed an amount of d-limonene 78.3%, and other compounds was as follows bergamol (6.21%), β-pinene (5.6%), linalool (5.15%), α-pinene (1.58%),1,8 cineole (0.76%)and rest other compounds shared 2.4 % of total sample. The compounds found in sweet lime peel oil are in agreement with previous reported composition and quantities[22-24].

Table-1
Chemical composition of sweet lime oil

S. no.	Retention Time	Compound	Quantity (%)
1	11.32	d-Limonene	78.3
2	14.59	bergamol	6.21
3	9.14	β-pinene	5.6
4	14.51	linalool	5.15
5	7.54	α-pinene	1.58
6	11.51	1,8 cineole	0.76
7	14.60	α-Terpineol	0.51
8	21.50	Neral	0.28
9	22.31	Geranial	0.21
10	31.72	β-Bisabolol	0.10
11	30.03	β-Bisabolene	0.10
12	9.78	β-Myrcene	0.08
13	10.30	Sabinene	0.08
14	16.95	Citronellal	0.07
15	15.06	α-Terpineol acetate	0.06
16	8.07	Camphene	0.06
17	31.72	α-Bisabolol	0.05
18	31.80	Bicyclogermacerene	0.03
19	20.97	Farnesol	0.03
20	51.07	Terpinen-4-ol	0.03
21	14.59	Trans-nerolidol	0.03
22	53.01	β-Farnesene	0.03
23	10.29	Nonanal	0.01
24	44.30	Phytol	0.01
25	34.07	Hinesol	0.01
26	10.11	α-phellandrene	0.01
27	17.57	Borneol	0.01
28	42.10	Myrcenil acetate	0.01
29	27.09	β-Santalene	0.01

Conclusion

It can be concluded from this study that extraction time is the most dominant factor followed by dryness of sweet lime peel and power input of microwave oven applied, oil yield increase with the extraction time from 10 min to 90 min in each experimental run. Dried sample gave highest yield and can be recommended for industrial practice. Oil is rich in most of antibacterial compounds, having pharmaceutical applications. Moreover, a waste can be converted in to a valuable chemical by processing and microwave assisted extraction is an efficient technique for such process.

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