



Extraction of D-Limonene from Sweet Orange peels using Simple Distillation

P. Akhila Swathanthra¹, M. Murali Naik ²

Department of Chemical Engineering, SVUCE

Author for Correspondence: muralinaik117@gmail.com

Abstract

D-Limonene is a compound that can be acquired from the rinds of the citrus family like oranges, limes, and mandarins. It is quite possibly the most well-known terpene present in nature. It has a few applications and a wide scope of advantages. Numerous medical service suppliers have supported the possible advantages of D-limonene guaranteeing that it can forestall or treat some ailments, for example, Bronchitis, Cancer, Diabetes, Gall stones, etc. It is likewise utilized in enterprises to make hand sanitizers, fragrances, plant pesticides, and synthetic solvents.

In the present study D-Limonene is extracted using the peel of oranges. The peel of oranges is boiled in water and the oil produced (D-limonene) is distilled in steam at a temperature just below 100 °C, well below its normal boiling point which is 176°C. The immiscible oil can then be separated from water by simple techniques. Direct extraction by heating would result in decomposition whereas steam distillation does not destroy the chemicals involved. This study of extracting D-limonene can be made using different solvents such as methanol, hexane, and distilled water at various temperatures and times of heating which gives us varied results from which the optimum process is concluded.

Keywords: D-Limonene, Sweet Orange Peels, Simple Distillation setup.

1. Introduction

D-Limonene is an essential oil that is available in high concentrations in the peels of citrus fruits. Which has several uses and applications in many disciplines including cosmetics, pharmaceuticals, and cleaning solutions. And it can also be used as a Biofuel because of its flammability [1,7]. Extraction of this highly useful essential oil from the peels of citrus fruits can be done in many methods such as (1) Cold Pressing (2) Solvent Extraction (3) Simple Distillation. Super Critical CO₂ extraction [2,3]. In the current investigation, we used Simple Distillation set for the process of extraction of D-Limonene using orange peels [11,14]. Limonene is a popular additive in foods, cosmetics, cleaning products, and natural insect repellents[13]. For example, it's used in foods like sodas, desserts, and candies to provide a lemony flavor. Due to its strong aroma, limonene is utilized as a botanical insecticide [6,9]. It's an active ingredient in multiple pesticide products. Other household products containing this compound include soaps, shampoos, lotions, perfumes, laundry detergents, and air fresheners [10]. Limonene has been studied for its potential anti-inflammatory, antioxidant, anticancer, and heart-disease-fighting properties.

One line that fits best to our motivation in initiating this project is BRINGING THE BEST OUT OF WASTE". Every year, a whopping 3.8 million tons of orange peels are dumped as waste. D-Limonene is an essential oil that can be extracted from orange peel wastes, which costs around Rs.500/- to Rs.700/- per ounce in the current market. Extracting D-Limonene from such discarded orange peels gives us two benefits which are extracting essential oil and reducing the orange peel waste. [12,15]

2. Material

1 kg of Orange fruits were obtained from local market in Ananthapuramu, after peeling the oranges, we obtained 230 grams of orange peels, the solvents used in the present study are distilled water and methanol. All chemicals were used of analytical reagent grade only.



Fig.1 Orange peels

3. Methodology

Initially the oranges were obtained from the local market and peeled using a kitchen peeler. The peels were carefully examined and weighed on an electronic scale. A simple distillation apparatus is set up containing a distillation flask, thermometer, condenser, heater, and collecting flask. The sample of orange peels along with the solvent was added into the distillation flask and were heated, simultaneously the condenser was turned on.

Two solvents were used to obtain varied results and to identify the optimum. The essential oil along with the solvent is collected in the collecting flask, the presence of d-limonene was known by the formation of a separate layer on top of the solvent and later on, the oil can be separated by using a separating funnel or a syringe. This process was repeated for various temperatures and times of heating.

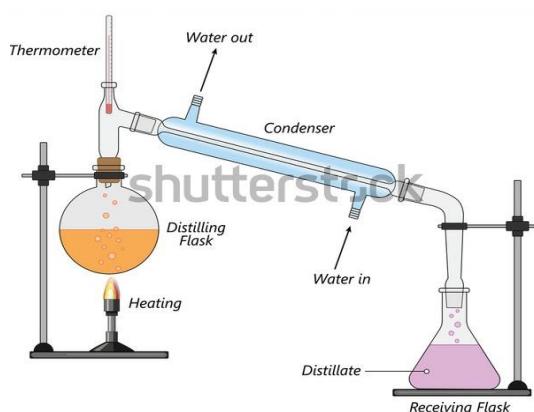


Fig. 2 Simple distillation for extraction of D-Limonene

Experimental Procedure:

Toward the beginning, the lemons are stripped off and the remaining strips are treated with solvents like water, methanol, hexane, and so forth, The blend is refined in basic refining contraption. The fumes created from the interaction contain D-limonene and are consolidated in a condenser. The Limonene from the condensate is isolated utilizing an isolating pipe.

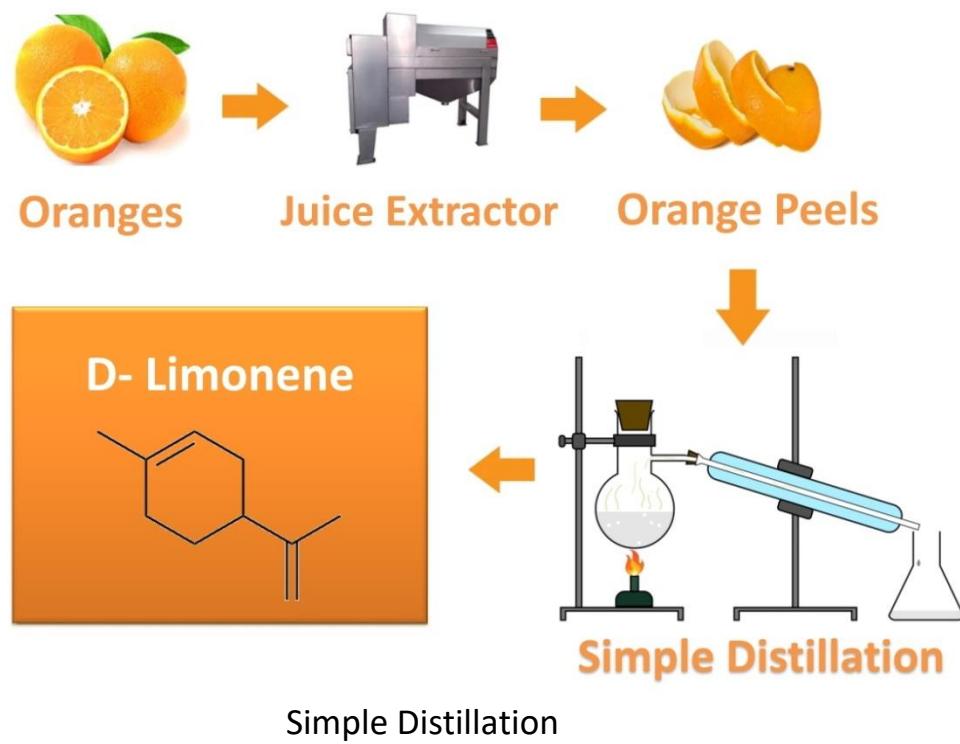


Fig. 3 Experimental Procedure

Table 1: Experimental data for extraction of d-limonene

| Raw Material | Solvent | Weight of Orange Peels (g) | Volume of Solvent (ml) | Heating Time (min) | Temperature (°C) | volume of Essential oil (ml) | volume of solvent in distillate (ml) |
|--------------|-----------------|----------------------------|------------------------|--------------------|------------------|------------------------------|--------------------------------------|
| Orange Peels | Distilled water | 230 | 100 | 30 | 100 | 1.6 | 42 |
| | | 230 | 100 | 50 | 98 | 1.8 | 48 |
| | | 230 | 100 | 60 | 101 | 2.2 | 58 |
| Orange Peels | Methanol | 230 | 100 | 30 | 65 | <0.1 | 93 |
| | | 230 | 100 | 50 | 67 | <0.1 | 94 |
| | | 230 | 100 | 60 | 68 | <0.1 | 97 |

4. Results

Based on the above table we can say that the yield of essential oil increases when time of heating increases and we have observed more volume of oil is extracted while using distilled water as a solvent when compared with methanol.

Calculations of vaporization efficiency

Mass of oil collected in distillate $M_1 = V_1 * S.G_1$

$$= 1.6 * 0.85$$

$$M_1 = 1.36 \text{ g}$$

Mass of water collected in distilled product $M_2 = V_2 * S.G_2$

$$= 42 * 2.16$$

$$M_2 = 90.72 \text{ g}$$

Table 2. Equilibrium Vapour Pressure data of Limonene

| S.No | Vapour Pressure P_A (mm Hg) | Temperature (°C) |
|------|----------------------------------|------------------|
| 1 | 1 | 14 |
| 2 | 10 | 53.8 |
| 3 | 40 | 84.3 |
| 4 | 100 | 108.3 |
| 5 | 400 | 151.4 |

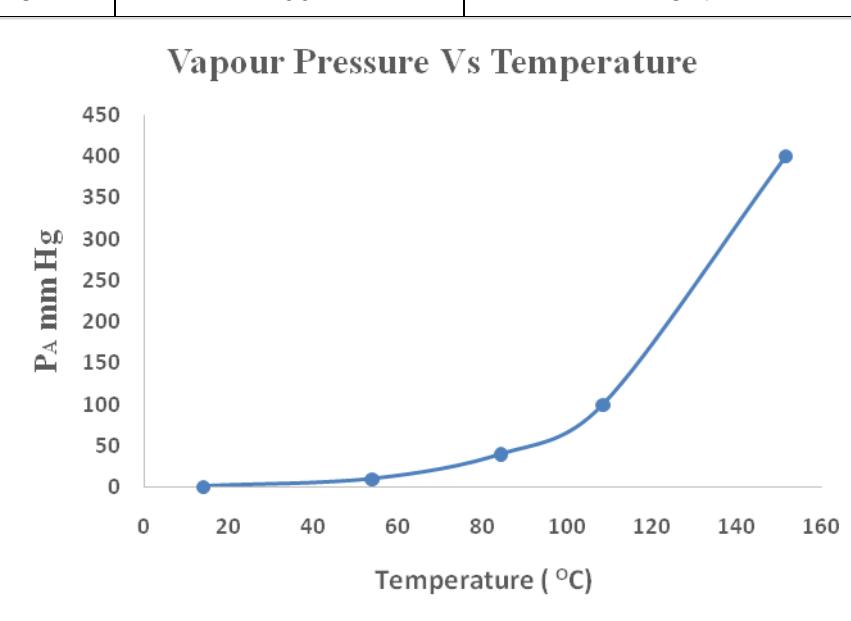


Fig.2 Variation of vapor pressure with temperature

$\eta_{vap} = (A/B) \text{ distillate (from experiment)} / (A/B) \text{ distillate (from graph)}$

Experimental (A/B) = $(M_1/M_2) = (1.36/90.72)$

$$= 0.0149$$

Theoretical weight ratio (A/B) = $[P_A/(P_t - P_A)] * [M.\text{wt}(A)/M.\text{wt}(B)]$

$$= [1.98/(728-1.98)] * [136/18]$$

$$= 0.02063$$

$$\eta_{vap} = 0.01499/0.02063$$

$$= 0.7267$$

$$\eta_{vap} = 0.7267 * 100$$

$$\eta_{vap} = 72.67 \%$$

Vaporization Efficiency = 72.67%

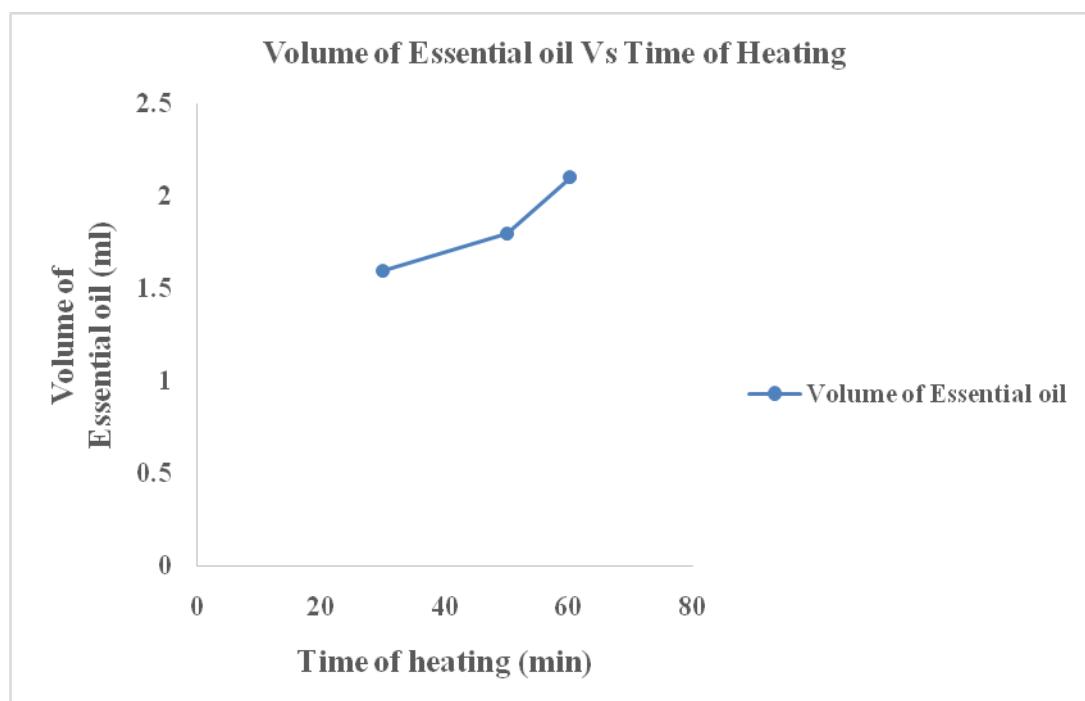


Fig. 3 Volume of Essential Oil increases with increases in time of heating keeping the temperature constant.

5. Conclusions

Volume of Essential oil increases with an increase in temperature while the time of heating was maintained constant. Volume of Essential Oil increases with increases in time of heating while the temperature was kept constant. The volume of solvent collected in the distillate is greater than the volume of oil collected at different temperatures and times of heating.

6. References

1. IOSR Journal of Applied Chemistry (IOSR-JAC) e-ISSN: 2278-5736. Volume 9, Issue 9 Ver. II, PP 16-17, September 2016.
2. Oriental journal of chemistry ISSN: 0970 - 020X, ONLINE ISSN: 2231-5039.
3. Fattah, A., Sh, M., Yehia, H. A., Fouzy, A. S. M., Ramadan, M. M., and Nooh, A. Antifungal efficacy and chemical composition of essential oil from the Egyptian sweet orange peel (*Citrus sinensis*, L). Manuscript Info Abstract. 3(10): 1257–1269, 2015.
4. Global Journal of engineering science and researches 4 pp. 42-48.
5. Aldrich Chemical Co. Aldrich Catalog/Handbook of Fine Chemicals, Milwaukee, WI, p. 766 , 1992.
6. Kodama R., Noda K., Ide H. Studies on the metabolism of *d*-limonene (*p*-mentha-1,8-diene) II. The metabolic fate of *d*-limonene in rabbits. Xenobiotica, 4:85–95, 1974.
7. Extraction and Characterization of D-limonene oil from orange peels using different solvents Ketaki Mirdha and Chittaranjan Routray, School of Applied Sciences, Centurion University of Technology and Management, Odisha, India, May 2020
8. Sun J, D-Limonene: safety and clinical applications, Altern Med Rev, 12(3),259-64 , 2007.
9. Fahlbusch, Karl-Georg; Hammerschmidt, Franz-Josef; Panten, Johannes; Pickenhagen, Wilhelm; Schatkowski, Dietmar; Bauer, Kurt; Garbe, Dorothea; Surburg, Horst, Flavors and Fragrances. Ullmann's Encyclopedia of Industrial Chemistry, P-44, 2003.
10. Sharma, Kavita; Mahato, Neelima; Cho, Moo Hwan; Lee, Yong Rok, Converting citrus wastes into value-added products: Economic and environmentally friendly approaches, Nutrition. 34, 29–46, 2017.
11. Siddiqui, Shahida & Pahmeyer, Maximilian & Assadpour, Elham & Jafari, Seid. Extraction and purification of d-limonene from orange peel wastes: Recent advances. Industrial Crops and Products. 177. 114484. 10.1016/j.indcrop.2021.114484, 2022.
12. Odio, Carlos.. d-Limonene Recovery. 45. 10.1115/CEC1996-4204, 1996.
13. Ferajee, Mona and Kazim Akhtar. "Extraction of Biofuel from Orange Peel and its Characterization." , 2017.
14. Pauline, Mercy Nisha. "Extraction of Orange Oil by Improved Steam Distillation and its Characterization Studies.", 2015.
15. Gavahian, Mohsen et al. "Recent advances in orange oil extraction: an opportunity for the valorisation of orange peel waste a review." International Journal of Food Science & Technology: n. pag, 2018.