**International Journal of Technical Research and Applications** e-ISSN: 2320-8163,
www.ijtra.com Volume 4, Issue 3 (May-June, 2016), PP. 341-346

# EXTRACTION OF CITRUS OIL FROM ORANGE (CITRUS SINENSIS) PEELS BY STEAM DISTILLATION AND ITS CHARACTERIZATIONS

**D.C. Sikdar[1], Rohan Menon[2], Karan Duseja[3], Piyush Kumar[4], Priksha Swami[5]**
Dept. of Chemical Engineering,
Dayananda Sagar College of Engineering,
Bangalore 560 078, India
dulalsikdar@gmail.com[1]
menonrohan95@gmail.com[2]
karanduseja100@gmail.com[3]
piyushdehri@gmail.com[4]
swamipriksha@gmail.com[5]

***Abstract*—Citrus oil is an essential oil present within the rind of wall of a citrus fruit. In contrast to most essential oils, it is extracted as a byproduct of juice extraction by centrifugation, producing cold pressed oil. It is found in all citrus fruits like lemon, orange, sweat lime etc. In the present investigation orange (citrus sinensis) peels is used for the extraction of citrus oil. India along with Brazil, China and the United States of America accounts for almost half the orange production in the world. As orange is one of the most common fruits with a lot of nutritional values, it is most consumed for its dietary benefits. After extraction of juice, the orange peels are treated as waste and lead to environmental pollution due to improper disposal can be used for the extraction of citrus oil. This paper focuses on improved steam distillation, where the orange peels are preheated before subjecting to distillation. The citrus oil composed of around 95% D-limonene which has many applications ranging from food flavouring agents to cosmetics.**

*Index Terms*— Citrus oil, Orange peels, essential oil, Steam distillation, Limonene.

## I. INTRODUCTION

India along with Brazil, China and the United States of America account for almost half the orange production in the world [1]. Oranges are some of the most commonly utilized fruits in the world due to its pleasant taste [2]. and nutritional values. A sample of Orange fruits are shown in Fig1. Because of the huge consumption of orange juice throughout the world, a large amount of wet solid waste is produced. This waste mainly includes orange peels.

Fig.1 Orange fruits

The peels [3] contain numerous oil bearing glands that enclose significant amounts of citrus oil. The oil gland present in orange peels is shown in Fig.2.

Fig.2 Oil Gland in Orange Peels

Therefore, instead of throwing out these peels as a solid waste can be utilized for oil extraction [4]. The composition [5, 6] of the oil extracted from orange peels varies depending on the species of citrus fruit used.

The oil is primarily used in perfumery due to pleasant odour, but it also seen that they are used in food products such as sweets, beverages, and cakes. In fact, it is one of the most commonly used essential oils in food industries. They are also used to flavour distasteful drugs in pharmaceutical industries to make them easier to consume. The oil also finds application in extraction processes [7] as an eco-friendly solvent as the major constituent of the oil is limonene. The limonene present in the oil is a cyclo-terpene and is a colourless hydrocarbon that exists in the liquid state at normal temperature and pressure, and has the ability to solubilize the fats. Studies have already been conducted to extract the essential oil from the peels of orange through steam distillation. However, not much research has been done to identify the optimal condition for extraction of citrus oil through steam distillation. The oil extracted from orange peels can also be used as a green insecticide [8, 9]. According to research from Ibrahim et al. showed that the essential [9] oil extracted from orange peels is effective against several forms of pests ranging from bacteria and fungi to insects.

The main objective of this work is to extract the optimum amount of citrus oil from orange peels by steam distillation and also to vermicompost the orange peels residue left over after the oil extraction.

## II. Materials and Methods

### *A. Material/Instruments Used*

The materials/instruments used for this work were round bottom flask, Basket heater, distillation unit, thermometer, measuring cylinder, conical flask, separating funnel, Gas Chromatograph.

### *B. Sample Collection*

The orange peels samples are collected from the local juice vendors, Bangalore. A sample of orange peel is shown in Fig.3


Fig.3 Orange Peels Sample

### *C. Preparation of Orange Peels Sample*

The collected sample of orange peels is cleaned and pith is manually separated from the outer coloured part of the peels. That is because of the reason that the majority of the oil in oil sac present in them. This is then preheated at a temperature of 45 $^0$C and kept for two hours.

### *D. Extraction of Oil by Steam Distillation*

The Distillation set up is arranged as shown in Fig.4.It consists of distillation flask, Basket heater, horizontal condenser and a conical flask.

Fig.4 Distillation unit

100g of pre-treated orange peels sample is taken in a distillation flask. To that 200ml of water is added. Heat is supplied to the distillation unit by temperature controlled basket heater.

At the initial stage, experiment is carried out at a Temperature of $88^0$C for 60 min. time period. The distillate is collected in a conical flask. This distillate has two layers, one dense layer and other less dense layer. This is then separated using a separating funnel. The less dense upper layer is the citrus oil. This oil is then stored in a glass bottles shown in Fig.5

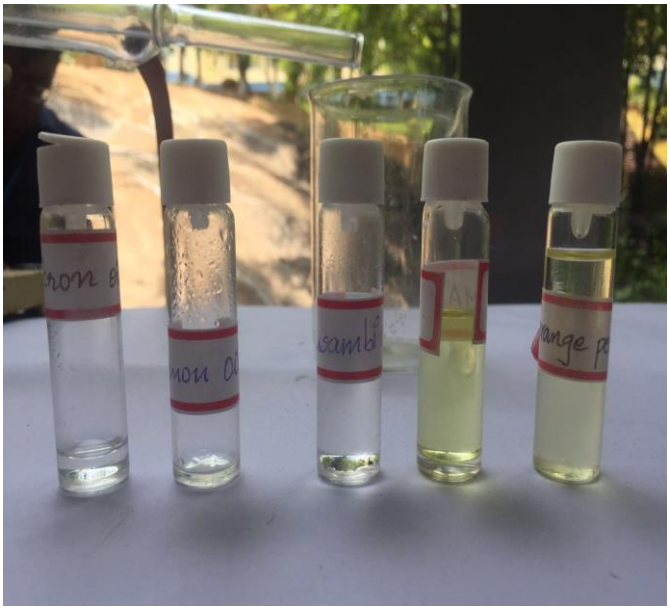

Fig.5 Citrus oil samples

The experiments are continued keeping distillation time and solid to solvent ratio constant varying the temperature of distillation at an interval of $2^0$C from $88^0$C to $98^0$C. This gives the optimum distillation temperature.

In the next phase, experiments are carried out keeping the distillation temperature and solid to solvent ratio constant by varying the distillation time at an interval of 15 min. from 15min. to 75 min. time period. This gives the optimum distillation time.

In the final phase, experiments are carried out keeping distillation time and temperature constant by varying the solid to solvent ratio from 100g/160ml to 100g/240ml. This gives the optimum condition for extraction of citrus oil from orange peels by distillation.

The residue of the orange peels after oil extraction is vermicomposed to improve the NPK value of the soil. The characteristic of citrus oil is determined by standard method of Gas Chromatography.

## III. Results and Discussions

### *A. Confirmation test for Limonene*

Citrus oil is extracted from orange peels by cold press method and subjected to conformation test for limonene content in the oil.

Bromine test: A dilute Bromine-water solution is prepared and taken in a test tube. To that citrus oil extracted from orange peels is added. If limonene is present in the oil extracted, the colour of the Bromine - water gets changes from red brown to pale yellow. This is because of the fact that the Bromine present in the Bromine – water solution occupies the space between the two double bonds present in limonene. The conformation test for Limonene is shown in Fig.6.

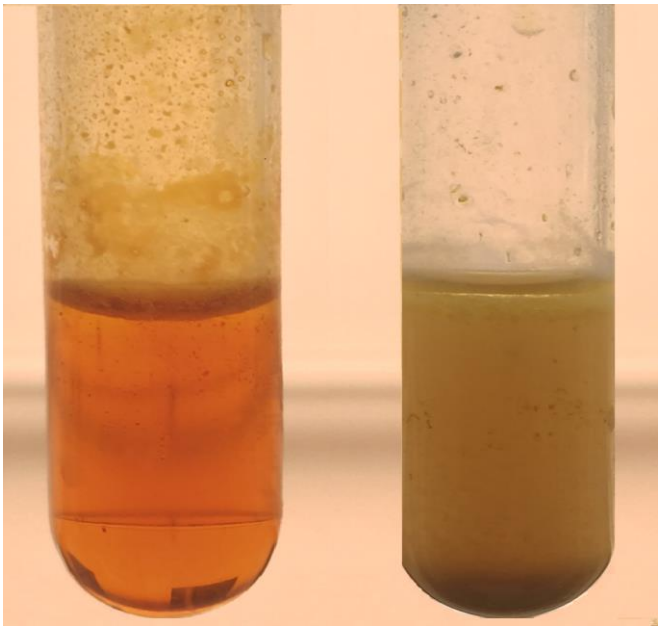

Fig.6 Conformation test for Limonene

### *B. Effect of Temperature on Steam Distillation of Orange Peels for Citrus Oil Extraction*

Initially, the extraction of citrus oil from orange peels by distillation is carried out by changing the temperature at an interval of 20C from 880C to 980C while the other parameters time and solid to solvent ratio kept constant. The results obtained are given in Table 1.

**Table1.Effect of Temperature on Steam Distillation of Orange Peels for Citrus Oil Extraction**

| Sn. No. | Temperature (°C) | Oil Extracted (ml) |
|---|---|---|
| 1 | 88 | 0.17 |
| 2 | 90 | 0.26 |
| 3 | 92 | 0.45 |
| 4 | 94 | 1.78 |
| 5 | 96 | 2.40 |
| 6 | 98 | 2.30 |

It is observed from the Table1 that with increase of distillation temperature the oil yield increases and it is maximum at $96^0$C.Further increase in distillation temperature the oil yield decreases that is because of the fact that at higher temperature charring of orange peels takes place at the bottom of the flask. The effect of distillation temperature on oil yield is shown in Fig7.

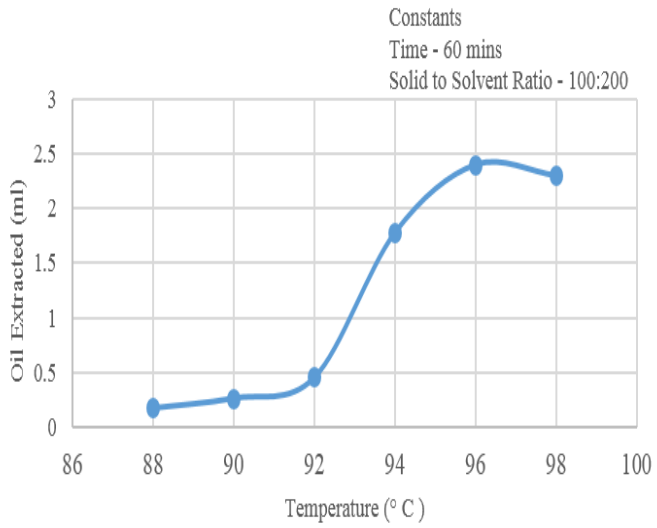


Fig.7.Effect of Temperature on Oil yield

*C. Effect of Time on Steam Distillation of Orange Peels for Citrus Oil Extraction*

In the next phase of experiment, the extraction of citrus oil from orange peels is carried out by changing the time at an interval of 15min. from 15 min. to 75 min. time period, while the other parameters temperature and solid to solvent ratio kept constant. The results obtained are given in Table2.

**Table2.Effect of Time on Steam Distillation of Orange Peels for Citrus Oil Extraction**

| Sn. No. | Time (min.) | Oil Extracted (ml) |
|---|---|---|
| 1 | 15 | 1.00 |
| 2 | 30 | 1.70 |
| 3 | 45 | 2.20 |
| 4 | 60 | 2.40 |
| 5 | 75 | 2.40 |

It is observed from the Table2 that with increase of distillation time the oil yield increases and it is maximum at 60 min. durations. Further increase in time has no effect on oil yield. The effect of distillation time on oil yield is shown in Fig.8

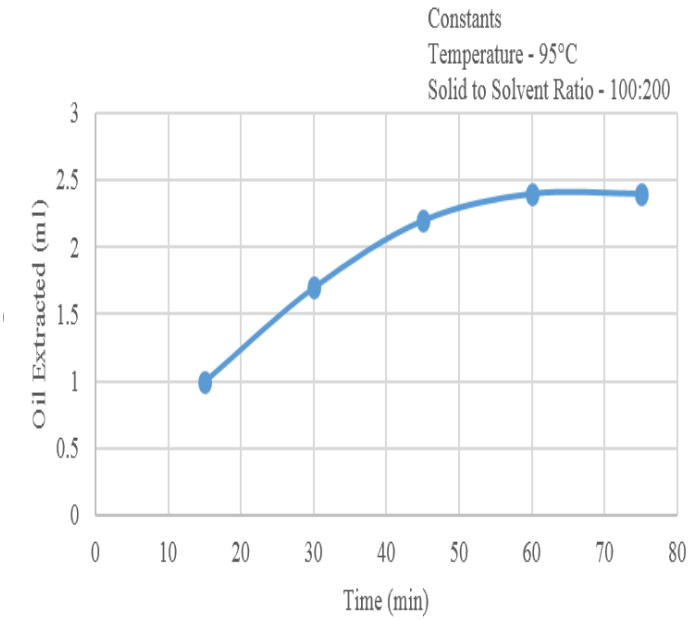


Fig.8 Effect of Time on Oil Yield

*D. Effect of Solid to Solvent ratio on Steam Distillation of Orange Peels for Citrus Oil Extraction*

In the next phase of experiment, the extraction of citrus oil from orange peels is carried out by changing the solid to solvent ratio from 100g/160ml to 100g/240ml, while the other parameters kept constant. The results obtained are given in Table3.

**Table.3 Effect of Solid to Solvent Ratio on Steam distillation of Orange Peels for Citrus Oil Extraction**

| S | Solid to Solvent ratio | Oil Extracted |
|---|---|---|

| n. No. | (g/ml) | (ml) |
|---|---|---|
| 1 | 100:160 | 2.10 |
| 2 | 100:180 | 2.20 |
| 3 | 100:200 | 2.40 |
| 4 | 100:220 | 1.85 |
| 5 | 100:240 | 1.25 |

It is observed from the Table3 that with increase of distillation content solid to solvent ratio, the oil yield increases and it is maximum at solid to solvent ratio 100g/200ml. Further increase in the solid to solvent ratio the oil yield decreases that may be due to the facts that at much higher solid to solvent ratio bubbling occurs. The effect of solid to solvent ratio on oil yield is shown in Fig.9.

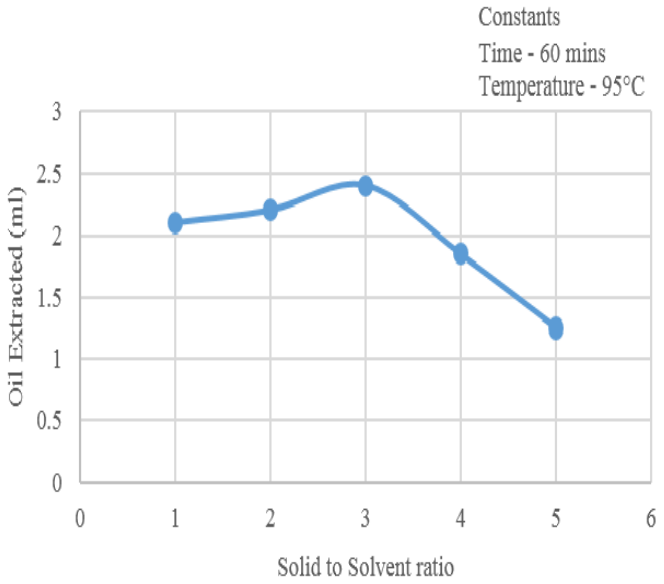


Fig.9 Effect of Solid Solvent Ratio on Oil yield

*E. Characteristics of Citrus Oil Extracted from Orange Peels*

The characteristics of citrus oil extracted from orange peels are determined by standard method of Gas Chromatograph (GC) and the compositions of Alpha-Pinene, Octanol, Beta-Mycrene and D-limonene are reported in Table4.

**Table4 Characteristics of Citrus Oil Extracted from Orange Peels**

| Peak No. | Component | Retention Time (min) | Concentration (%) |
|---|---|---|---|
| 1 | Alpha-Pinene | 5.707 | 1.24 |
| 2 | Octanol | 6.382 | 0.84 |
| 3 | Beta-Mycrene | 6.709 | 3.79 |
| 4 | D-limonene | 7.659 | 94.13 |

The GC analysis of citrus oil extracted from orange peels is shown in Fig.10

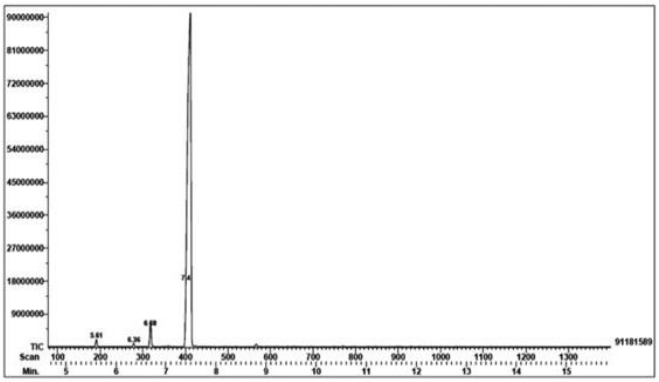

Fig.10.G C Analysis of Citrus Oil

It is found that Limonene is the major component (94.13%) and traces of Alpha-Pinene and Octanol are present in the oil.

*F. Analysis of Vermicomposting of Orange Peels Residue*

The analysis of vermicomposting of orange peels residue are analyzed by standard method and the NPK values of the soil before adding orange peels residue and after vermicomposting of orange peels residue are given in Table5.

**Table.5 Soil Analysis results**

| Characteristics | Soil without adding Orange peels residue | Vermicompost Of Orange Peels residue |
|---|---|---|
| pH | 7.9 | 7.7 |
| N (ppm) | 5.0 | 8.3 |
| P(ppm) | 10 | 14 |
| K(ppm) | 150 | 210 |

It can be observed from the Table5 that the nutrient content of the soil after vermicomposting of orange peels has been improved.

## IV. Conclusion

The environmental pollution arises due to the orange peels can overcome by using the same for citrus oil extraction by steam distillation. The optimum amount of citrus oil 2.4ml/100g of orange peels can be extracted by steam distillation at the optimum condition of temperature $96^0$C, time 60 min. and solid to solvent ratio 100g/200ml. Gas Chromatography of citrus oil shows that D-limonene, Beta-Mycrene, Alpha-Pinene, and Octanol content in the citrus oil are 94.13, 3.79, 1.24 and 0.84 percent respectively. The analysis of the vermicompost of residue of orange peels improved the NPK value of the soil. Therefore, disposal problem of the residue of orange peels after citrus oil extraction can be overcome by vermicomposting.

## Acknowledgment

We the help from Dr. R. Ravishankar, Dr. Murthy Shekhar, Prof. G.K. Mahadevaraju, Prof. H.N. Pradeep, and Prof. B.S. Thirumalesh, Department of Chemical Engineering, Dayananda Sagar College of Engineering, Bangalore. We would also like to thank Dr. Hemachandra Sagar, Chairman, Dr. Premachandra Sagar, Vice-Chairman, Sri Galiswamy, Secretary, and Dr. C.P.S. Prakash, Principal, Dayananda Sagar College of Engineering, Bangalore for their encouragement and help rendered.

## References

[1] Food and Agriculture Organization of the United Nations, “Citrus Fruit Fresh and Processed annual statistics” 2012.

[2] H. Nguyen, E. M. Campi, W. Roy Jackson and A. F. Patti, Effect of oxidative deterioration on flavor and aroma component of lemon oil, Food Chemistry, Vol.112, no.2, pp.388 to 393, 2009.

[3] M. L. Lota, D De Rocca Serra, F. Tomi and J Casanova, Chemical Variability of peels and leaf essential oils of mandarins from Citrus reticulate Blanco, Bio- chemical systematic and Ecology, Vol.28, no.1, pp.61-78, 2000.

[4] M. Virot, V. Tomao, C. Ginies, F. Visinoni, and F. Chemat, “Green procedure with a green solvent for fats and oils’ determination. Microwave-integrated Soxhlet using limonene followed by microwave Clevenger distillation,” Journal of Chromatography A, vol. 1196-1197, no. 1-2, pp. 147–152, 2008.

[5] R. P. Adams, Identification of Essential Oil components by Gas Chromatography/Quadruple Mass Spectroscopy, Allured publishing Corporation, Carol Stream, III, USA, 2001.

[6] B. Steuer, H. Schulz, and E. Lager, “Classification and analysis ¨ of citrus oils by NIR spectroscopy,” Food Chemistry, vol. 72, no. 1, pp. 113–117, 2001

[7] M. Virot, V. Tomao, C. Ginies, F. Visinoni, and F. Chemat, “Green procedure with a green solvent for fats and oils’ determination. Microwave-integrated Soxhlet using limonene followed by microwave Clevenger distillation,” Journal of Chromatography A, vol. 1196-1197, no. 1-2, pp. 147–152, 2008.

[8] .Mohamed A. Ibrahim, Pirjo Kainulainen, Abbas Aflatuni, Kari Tiilikkala, Jarmo K. Holopainen, “Insecticidal, repellent, antimicrobial activity and phytotoxicity of essential oils: with special reference to limonene and its suitability for control of insect pests,” Agricultural and Food Science in Finald, vol. 10, pp. 243-259,2001.

[9] M. Chutia, P. Deka Bhuyan, M. G. Pathak, T. C. Sarma, and P. Boruah, “Antifungal activity and chemical composition of Citrus reticulata Blanco essential oil against phytopathogens The Scientific World Journal 9 from North East India,” LWT—Food Science and Technology, vol. 42, no. 3, pp. 777–780, 2009