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COMPARATIVE STUDY ON SOLVENT EXTRACTION OF OIL FROM CITRUS FRUIT PEELS BY STEAM DISTILLATION AND ITS CHARACTERIZATIONS

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***Abstract*—**Citrus oil is an essential oil present within the rind of wall of a citrus fruits. In contrast to most essential oils, it is extracted as a by product of juice extraction by centrifugation, producing cold pressed oil. It is found in all citrus fruits peels. In the present investigation Orange, Sweet lime and Lemon peels are used for the extraction of citrus oil. India along with Argentina, Brazil, China, Mexico and the United States of America accounts for almost half the citrus fruit production in the world. As Orange, Sweet lime and Lemon are most common citrus fruits with a lot of nutritional value, its juice are most consumed for its dietary benefits during the summer. After extraction of juice, the citrus fruits peels are treated as waste and lead to environmental pollution due to improper disposal which can be used for the extraction of citrus oil. This paper focuses on improved steam distillation, where the peels are preheated before subjecting to distillation. The citrus oil composed of around 94.5% D-limonene which has many applications ranging from food flavouring agents to cosmetics**.**

Index Terms— Citrus oil, Citrus fruit peels, Essential oil, Steam distillation, Limonene.

I. INTRODUCTION

India along with Mexico, Argentina and Brazil accounts for almost half the citrus fruits production in the world [1]. Orange, Sweet lime and Lemon are the most commonly utilized citrus fruits in the world due to its pleasants [2] and nutritional values. A sample of citrus fruit is shown in Fig1. Because of the huge consumption of citrus fruits juice throughout the world, a large amount of wet solid waste is produced. These wastes mainly include Orange peels, Sweet lime peels and Lemon peels.

Fig.1 Citrus Fruits

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

Fig.2 Oil Gland in Citrus Fruits Peel

Therefore, instead of throwing out these peels as a solid waste it can be utilized for oil extraction [4]. The composition [5, 6] of the oil extracted from citrus fruits peel varies depending on the species of citrus fruits grown in particular area.

The oil is primarily used in perfumery due to pleasant odour, but it is 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 color less 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 citrus fruits 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 citrus fruits peel can also be used as a green insecticide [8, 9]. According to research from Ibrahim et al. showed that the essential oil [9] extracted from citrus fruits peel is effective against several forms of pests ranging from bacteria and fungi to insects.

The main objective of this work is to study comparatively on extraction of optimum amount of citrus oil from various citrus fruits peel by steam distillation and also to vermi compost the fruits peel residue left over after the oil extraction.

II. MATERIALS AND METHODS

A. Materials/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 citrus fruits Orange, Sweet lime and Lemon peels samples are collected from the local juice vendors, Bangalore. A sample of citrus fruits peel are shown in Fig.3

Fig.3 Citrus Fruits Peels Sample

C. Preparation of Citrus Fruits Peels Sample

The collected sample of citrus fruits peels are 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 being supplied to the distillation unit by temperature controlled basket heater.

At the initial stage, experiment is carried out at a temperature of 88^0C for 60 min. time period. The distillate is collected in a conical flask. This distillate has two layers, one dense layer and the 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

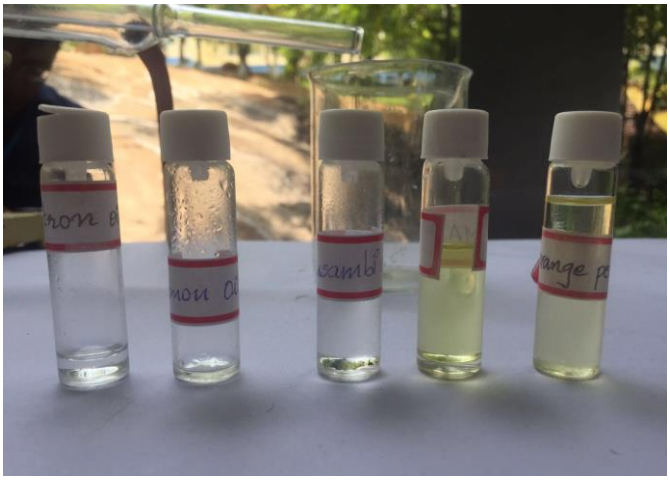
Fig.5 Citrus oil samples

The experiments are continued keeping distillation time and solid to solvent ratio constant varying the temperature of distillation at an interval of 2^0C from 88^0C to 98^0C. 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. This procedure is repeated for Sweet Lime and Lemon Peels.

The residue of the citrus fruits peels after oil extraction is vermin composed 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 citrus fruits 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 Bromine-water solution citrus oil extracted from citrus fruits peels is added. If limonene is present in the oil extracted, the colour of the Bromine - water gets changed 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 Citrus Fruits Peels for Oil Extraction

Initially, the extraction of citrus oil from Orange, Sweet lime and Lemon peels by distillation are carried out by changing the temperature at an interval of 2^0C from 88^0C to

98^0C while the other parameters like time and solid to solvent ratio are kept constant. The results obtained are given in Table 1.

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

Sl. No.	Temperature (°C)	Oil Extracted (ml)		
		Orange Peels	Sweet Lime Peels	Lemon Peels
1	88	0.17	0.10	0.10
2	90	0.26	0.25	0.23
3	92	0.45	0.35	0.30
4	94	1.78	1.25	1.15
5	96	2.40	1.75	1.40
6	98	2.30	1.65	1.39

It is observed from the Table1 that with increase of distillation temperature the oil yield of Orange, Sweet lime and Lemon peels increases and it is optimum at 96^0C. Further increase in distillation temperature the oil yield has no effect that is because of the fact that at higher temperature charring of the peels takes place at the bottom of the distillation flask. It is also observed that the oil yield is maximum for Orange peels and minimum for lemon peels. The effect of distillation temperature on oil yield is shown in Fig7.

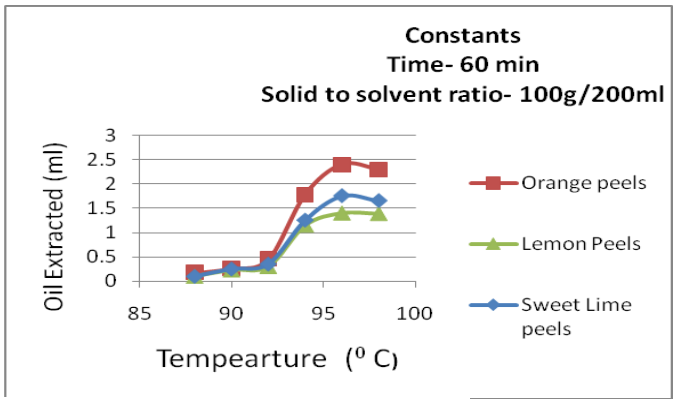

Fig.7.Effect of Temperature on Oil yield

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

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

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

Sl. No.	Time (min)	Oil Extracted (ml)		
		Orange Peels	Sweet Lime Peels	Lemon Peels
1	15	1.00	0.75	0.25
2	30	1.70	1.00	0.60
3	45	2.20	1.35	1.10
4	60	2.40	1.75	1.40
5	75	2.40	1.75	1.40

It is observed from the Table2 that with increase of distillation time the oil yield increases and it is optimum at 60 min. duration. Further increase in time has no effect on oil yield. It is also observed that the oil yield is maximum for Orange peels and minimum for Lemon peels. The effect of distillation time on oil yield is shown in Fig.8

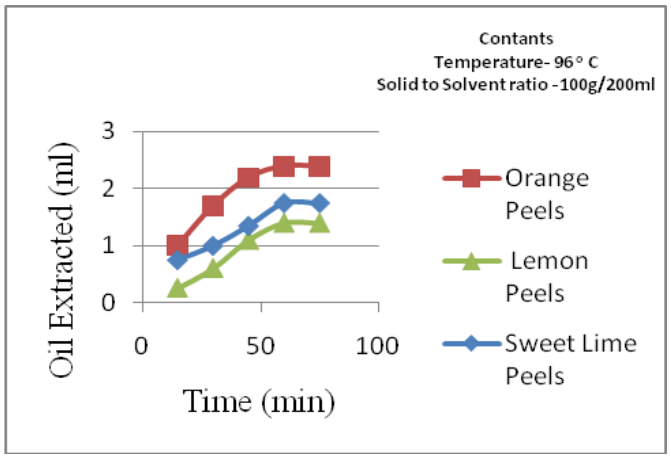

Fig.8 Effect of Time on Oil Yield

D. Effect of Solid to Solvent ratio on Steam Distillation of Citrus Fruits Peel for Oil Extraction

In the next phase of experiment, the extraction of citrus oil from Orange, Sweet lime and Lemon peels by distillation 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 Citrus Fruits Peel for Oil Extraction

Sl. No.	Solid to Solvent Ratio (g/ml)	Oil Extracted (ml)		
		Orange Peels	Sweet Lime Peels	Lemon Peels
1	100:160	2.10	1.15	0.95
2	100:180	2.20	1.40	1.15
3	100:200	2.40	1.75	1.40
4	100:220	1.85	1.65	1.35
5	100:240	1.25	0.90	0.65

It is observed from the Table3 that with increase of distillation content solid to solvent ratio, the oil yield increases and it is optimum at solid to solvent ratio 100g/200ml. Further increase in the solid to solvent ratio the oil yield decreases that is due to the facts that at much higher solid to solvent ratio bubbling occurs which help to carry away the oil vapour during the condensation. It is also observed that the oil yield is maximum for Orange Peel and minimum for Lemon Peel. . The effect of solid to solvent ratio on oil yield is shown in Fig.9.

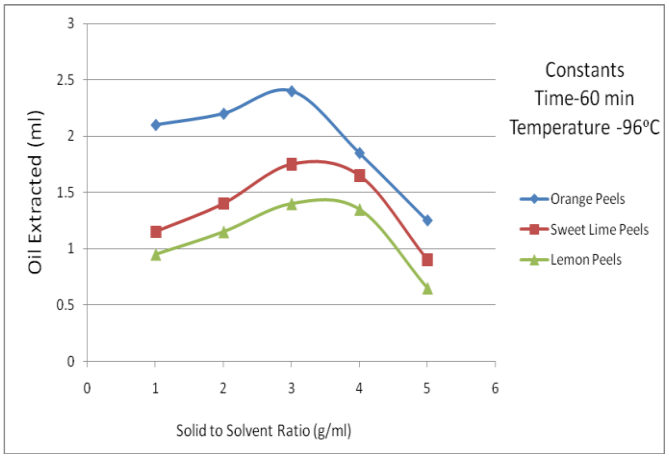

Fig.9 Effect of Solid Solvent Ratio on Oil yield

E. Characteristics of Oil Extracted from Citrus Fruits Peels

The characteristics of oil extracted from citrus fruit 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 Oil Extracted from Citrus Fruits 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 oil extracted from citrus fruits peels is shown in Fig.10

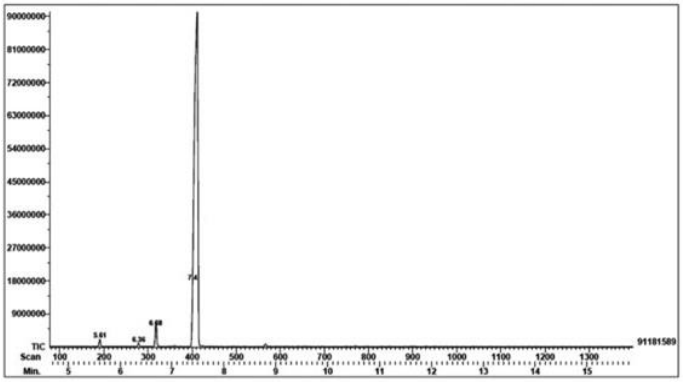

Fig.10.G C Analysis of Citrus Oil

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 Citrus Fruits Peels Residue

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

Table.5 Soil Analysis results

Characteristics	Soil without adding Citrus Fruit Peels Residue	Vermicompost Of Citrus Fruits 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 citrus fruit peels has been improved.

IV. CONCLUSION

The environmental pollution arises due to the citrus fruits peels disposal, which can be overcome by using the same for oil extraction by steam distillation. The optimum amount of citrus oil 2.4 ml/100g of citrus fruits peels can be extracted by steam distillation at the optimum condition of temperature 96^0C, 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 citrus fruits peels improved the NPK value of the soil. Therefore, disposal problem of the residue of citrus fruits peels after citrus oil extraction can be overcome by vermicomposting.

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