ISSN: 0975-8585

Research Journal of Pharmaceutical, Biological and Chemical Sciences

Extraction of Lemon Oil from Lemon Peel using Solvent Extraction Method – Optimization by using RSM.

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ABSTRACT

The lemon peels are treated as waste materials after extraction of juice and lead to environmental pollution due to improper disposal. It may create environmental problems for local communities due to the presence of biomaterials in lemon peel. Since there is a great need for development of new and environmental friendly design processing techniques could be turned into an asset. Hence this work focuses on extraction of lemon oil from lemon peel waste using solvent extraction method, where the Lemon peels are preheated before extraction. The preheating enhances the oil yield. The effect of temperature, time, and solid to solvent ratio are also studied to obtain optimum temperature, time and solid to solvent ratio to produce maximum volume of oil. The lemon oil composed of around 95% D-Limonene which has many applications ranging from food flavoring agents to cosmetics. Design expert Software is used to optimize the parameters and the new model has been proposed for the extraction of citrus oil.

Keywords: lemon oil; D-Limonene; RSM; optimization

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ISSN: 0975-8585

INTRODUCTION

Because of the huge consumption of lemon throughout the world, a large amount of wet solid waste is produced. This waste mainly includes lemon peels. The peels contain numerous oil bearing glands that enclose significant amounts of citrus oil. The oil is primarily used in perfumery due to pleasant odour [1], but it also seen that they are used in food products such as sweets, beverages, and cakes. It is one of the most commonly used essential oils in food industries. Lemon oil is considered to be the powerful antimicrobial agent of all the essential oils. It acts as an antioxidant and protect cell against free radical damage. Lemon oil is used to stimulate lymph drainage, to rejuvenate energy, purify skin and act as a bug repellent. It acts as a natural disinfectant [2].

Limonene

The limonene present in the oil is a cyclo-terpene and is a colorless hydrocarbon that exists in the liquid state at normal temperature and pressure, and has the ability to solubilize the fats. Limonene is a naturally occurring chemical which is used in many food products, soaps and perfumes for its lemon-like flavor and odor. [3]

Design of Experiment - Statistical Method

Design of experiment is a systematic method to determine the factors affecting a process and the output of that process. This information is needed to manage process inputs in order to optimize output.

Response Surface Methodology (RSM) is a statistical method that uses quantitative data from appropriate experiments to determine regression model equations and operating conditions. RSM is a collection of mathematical and statistical techniques for modeling and analysis of problems in which a response of interest is influenced by several variables. [4]

METHODS AND MATERIALS

The lemon peels samples are collected from the local juice vendors. The collected sample of lemon peels is cleaned and pith is manually separated from the outer colored part of the peels. Because the majority of the oil is present in oil sac of the peels. The separated peel is then preheated at a temperature of 45˚C for two hours.

100g of pre-heated orange and lemon peels sample is taken in a round bottom flask, and then 200ml of water is added to the preheated peel. Heat is supplied by temperature controlled basket heater. At the initial stage, experiment is carried out at a temperature of 80˚C for 60 min. time period as shown in Fig.1.The distillate is collected in a conical flask. This is then separated using a separating funnel. The less dense upper layer is the citrus oil. This oil is then stored in a vials as shown in Fig.2.

Fig 1: Solvent Extraction Apparatus

ISSN: 0975-8585

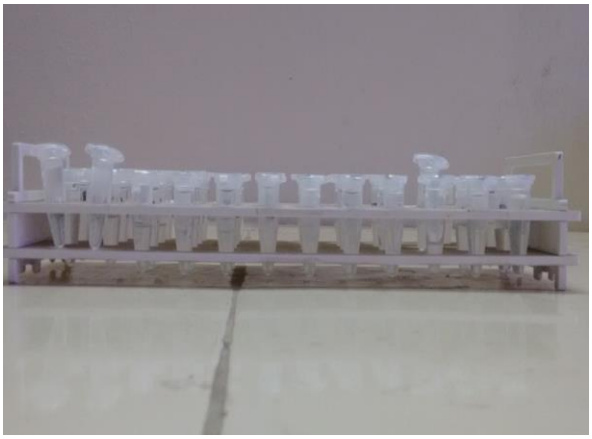

Fig 2: Oil Stored in Vials

The experiments are continued keeping distillation time and solid to solvent ratio constant varying the temperature of distillation at an interval of 2˚C from 80˚C to 92˚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 next phase, experiments are carried out keeping distillation time and temperature constant by varying the solid to solvent ratio from 50g/80ml to 50g/120ml. This gives the optimum condition for extraction of citrus oil from orange peels by distillation. Finally the experiment is carried out at optimum temperature, time and solid to solvent ratio to obtain the optimum volume of oil. [5]

Conformation Test for Limonene

Citrus oil is extracted from lemon peels has taken for Bromine test to confirm the presence of limonene in citrus oil. Because citrus oil contain 95% Limonene.

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 color of the Bromine – water gets changes from red brown to pale yellow as shown in Fig.3. 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.

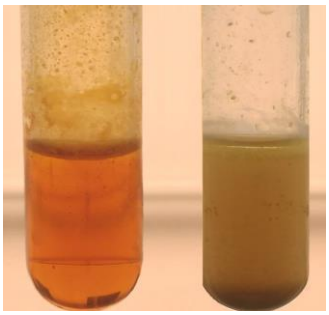

Fig 3: Conformation Test for Limonene

Iodine Test

Iodine test is the characterization test for the presence of any unsaturated compound in a test sample. On addition of iodine, the colorless lemon oil sample extracted by simple distillation turned into brown colour. The brown colour is due to the reaction of iodine with limonene and other unsaturated

compounds like α-pinene, β-pinene, myrcene, limonene and linalool. The presence of limonene and other aromatic compounds in the sample was confirmed. [6]

RESULTS AND DISCUSSIONS

Effect of Temperature on Extraction of Lemon Oil

Initially, the extraction of citrus oil from lemon peels is carried out by changing the temperature at an interval of 2˚C from 80˚C to 92˚C while the other parameters time and solid to solvent ratio kept constant. It is observed from the Fig.4that with increase of temperature the volume of oil increases and it is maximum at 89˚C.Further increase in temperature the volume of oil decreases that is because of the fact that at higher temperature charring of lemon peels takes place at the bottom of the flask.

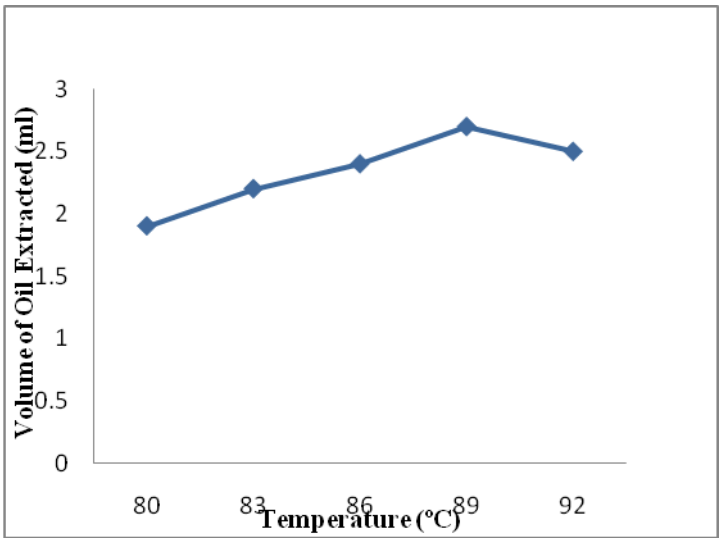

Fig 4: Effect of Temperature on Extraction of Lemon Oil from Lemon Peel

Effect of Time on Extraction of Lemon Oil

The extraction of citrus oil from lemon peels is carried out by changing the time from 15 min. to 75 min with a time interval of 15 min while the other parameters temperature and solid to solvent ratio kept constant. It is observed from the Fig.5that with increase of time the volume of oil increases and it is maximum at 60 min. duration. Further increase in time has no effect on volume of oil.

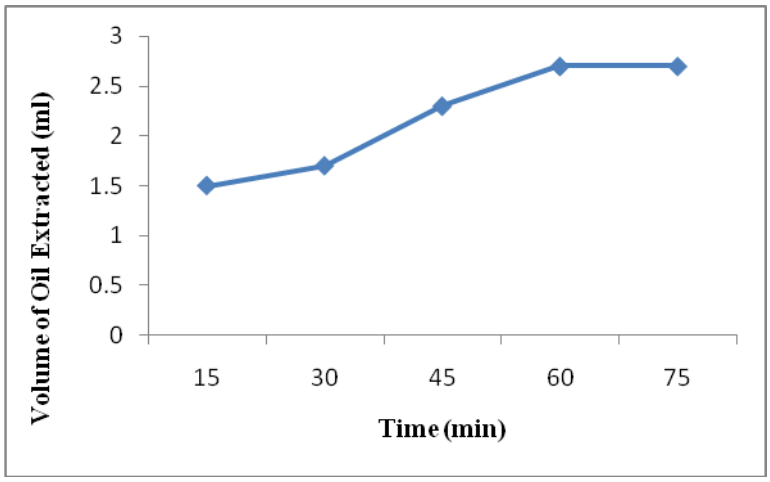

Fig 5: Effect of Time on Extraction of Lemon Oil from Lemon Peel

ISSN: 0975-8585

Effect of Solid to Solvent on Extraction of Lemon Oil

The extraction of citrus oil from lemon peels is carried out by changing the solid to solvent ratio from 50g/80ml to 50g/120ml, while the other parameters kept constant. It is observed from the Fig.6 that with the increase of solid to solvent ratio, the volume of oil increases and it is maximum at solid to solvent ratio 50g/100ml. Further increase in the solid to solvent ratio the volume of oil decreases due to the facts that at much higher solid to solvent ratio bubbling occurs. [7], [8]

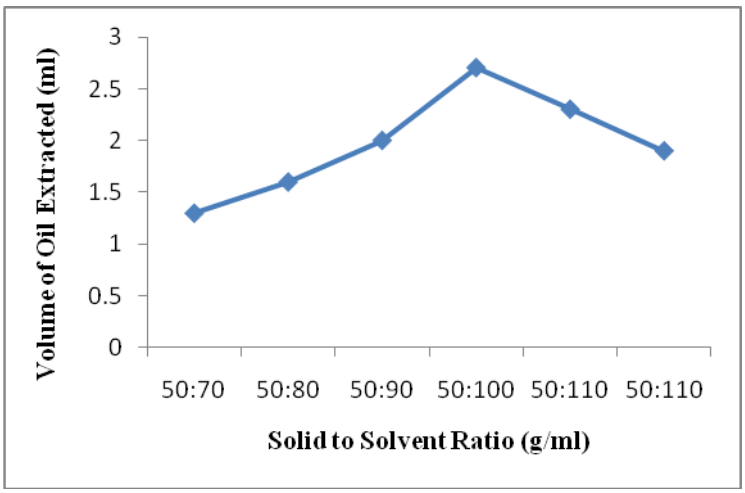

Fig 6: Effect of Solid to Solvent on Extraction of Lemon Oil from Lemon Peel

RSM- Model Developed for Extraction of Lemon Oil

To estimate the volume of oil extracted, empirical models have been developed by using statistical software package design expert. On the basis of experimental data the models have been developed for the determination of oil extracted from lemon by employing RSM based CCD. The final empirical models in terms of temperature and time are represented in Equations (1) and (2).

Final Equation in Terms of Coded Factors:

Oil Extracted=2.03+0.13*A+0.62*B (1)

Final Equation in Terms of Actual Factors:

Oil Extracted =-0.69583+0.020833 *Temperature+0.020833*Time (2)

The response surface graphs for Combined Effect of Time, Temperature on Volume of Oil Extracted shown in Fig.7. From the above graph, it was seen that the above response might be influenced by Temperature and time. It is observed that the increase in temperature results volume oil extracted to increase due to combined effect of time, temperature on volume of Oil Extracted.[9],[10]The deviation between actual and predicted values have been obtained and presented in Fig.8.

ISSN: 0975-8585

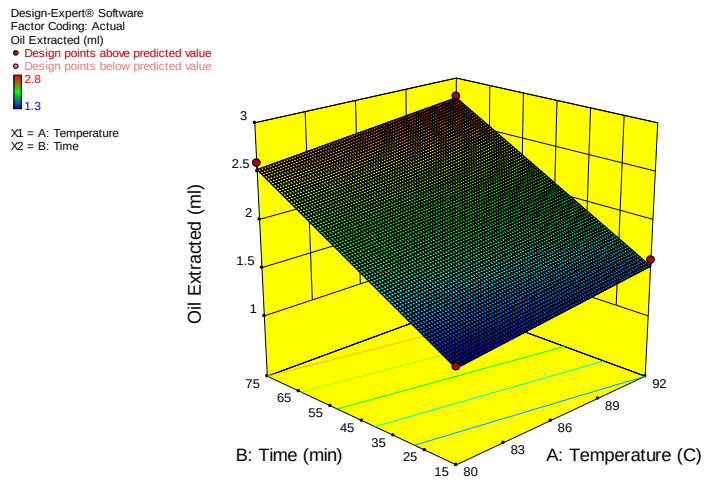

Fig 7: Combined Effect of Time, Temperature on Volume of Oil Extracted

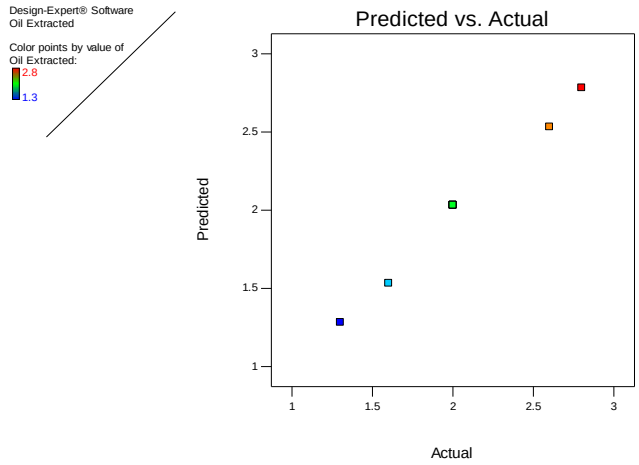

Fig 8: Prediction Graph for Extraction of Lemon Oil

CONCLUSION

The extraction of lemon oil from lemon peel was carried out by solvent extraction method. The experimental study on effect of temperature, time, and solid to solvent ratio was done to obtain optimum temperature, time and solid to solvent ratio to produce maximum volume of oil. The optimum condition for lemon oil extraction is 89ºC, 60 min and 50:100 of solid to solvent ratio. Design of Experiments by using design expert software also was done for the extraction of citrus oil from lemon peel. The new model had been proposed for efficient extraction process.

ACKNOWLEDGMENT

The authors thank the Institute authorities for providing the necessary facilities to carry out the work.

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