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# RECOVERY OF OIL AND PECTIN FROM CITRUS LIMETTA

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**Abstract-** *Citrus Limetta is the most common fruit available. The present work addresses to the extraction of the oil and the pectin from the fruit peel which is the waste of Citrus Limetta juice processing industry. The research highlighted that these peels are good source of oil and pectin and hence, can become the raw material for food processing industries. Cake remained after the process in future treated for isolation of pectin. Here, we will observe the effect of peel size of Citrus Limetta on the yield of oil. Also, in this research we will see the conditions in which orange oil can be stored. Oil and pectin is used by pharmaceutical industry, food industry etc. and is widely used in manufacturing of soaps, cosmetics, herbal medicines, perfumes etc. This process can be beneficial for industrial extraction of pectin from an economic and environmental point of view.*

***Key Words:*** *oil recovery, pectin extraction, peel size, storage of orange oil, cost estimation.*

## 1. INTRODUCTION

Orange juice is one of the most widely-consumed beverages today. Approximately 50-60%[6] of the processed fruits are transformed into citrus peel, which is composed of peels, seeds and membrane residues. With the increase in production of processed fruit juice, fruit waste generated is increasing enormously. Large amounts of these waste pose the problem of disposal without causing environmental pollution. These wastes can be effectively disposed by manufacturing by-product from them. Orange peel oil has been chosen for extraction[11,13] because it provides a great potential for further commercial form. Peel of citrus fruit has numerous glands that contain oil that is typically recovered as major by-product. Each citrus fruit has its own characteristic set of compounds that comprise the oil and that are responsible for its flavor and aroma to products such as carbonated drinks, ice creams, cakes and perfumes etc.[13]

Recently developed extraction methods like supercritical fluid extraction, microwave assisted extraction and solvent method[18] has been used for oil extraction. The basic parameters influencing the quality of an extract are the solvent used for extraction, the manufacturing process used with the type of equipment employed. In this research, Simple Distillation has been chosen because, this process has many advantages over conventional methods in terms of costing, yielding, and time.

Citrus fruits belong to six genera which are native to the tropical and subtropical regions of Asia, but the major commercial fruits belong to the genus Citrus. The genus Citrus includes several important fruits such as Oranges, Mandarins, Lime, Lemons and Grape fruits. The Citrus essential oils are mixture of volatile compounds and mainly consisted of monoterpene hydrocarbon. Citrus oils are mixture over a hundred compounds that can be approximated into three fractions: terpene hydrocarbons, oxygenated hydrocarbons and non-volatile compounds. The terpene fraction can constitute from 50% to more than 95% of the oil. However, it makes little contribution to the flavor and fragrance of the oil. Oranges are now commonly enjoyed by many and used for other purposes besides general consumption. It can be made into juice or incorporated into food products. It is a fruit with significant history, and oranges will remain a popular choice among consumers as a source of vitamin and to aid in wellbeing maintenance.

Pectin[4] is naturally occurring biopolymer that is finding increasing applications in the pharmaceutical and biotechnology industry. It has been used successfully for many years in the food and beverage industry as thickening agent, a gelling agent, and a colloidal stabilizer. Pectin has been widely studied and published but it difficult to characterize as a model system due to the heterogeneous nature of the polymer. This research will describe the source and production, chemical structure, and general properties of pectin.

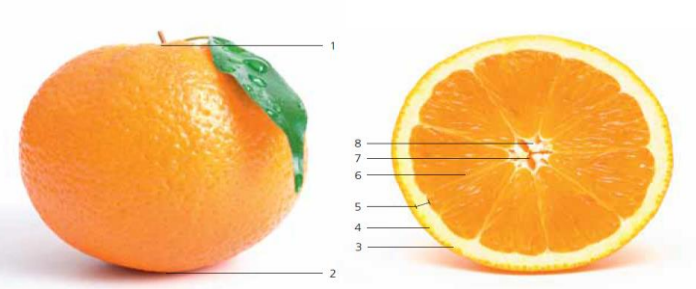


**Fig -1**: Cross-Section of Citrus Limetta

1. Stern end (proximal)
2. Stylar or blossom end (distal)
3. Outer cuticle layer
4. Oil Cells
5. Flavedo
6. Juice Vesicles
7. Fruit axis (half hollow)
8. Seed (partially cut)

## 2. MATERIALS AND METHODS

All the chemicals and reagents used were of analytical grade.

The raw material taken under examination for the extraction of orange oil (d-limonene) and pectin is orange peel[1]. Fresh citrus fruit was purchased from the local market and were harvested in the month of January 2016 to March 2016. The peels were cut into small pieces and dried at 55°C in oven for 48 hours[20].

The present work is divided into following parts[1]:

I. Separation of oil from peels
   a) Using the method of simple distillation
   b) Using ethanol/chloroform as solvent in the method of leaching.

II. Extraction of pectin from oil peels
   a) From fresh peels
   b) From dried cake remained after simple distillation and leaching as in part-I.

### 2.1 Separation of Peels Based on Size

Grind approximately 200 gm of dried orange peel for one second in a mixer. Then keep the grinded material aside. Again take 200 gm dried orange peel and grind in a mixer for 2 seconds. Repeat till 5 seconds. Mix all the grinded material and put it in sieve shaker and run the sieve shaker for approximately 10 minutes. Then, weigh each size of grinded peel left in each sieve tray[18]. I got the results as tabulated below:

**Table -1:** Sieve opening size and weight retained on each sieve

| Sr. No. | Sieve Opening (mm) | Weight Retained (gm) |
|---|---|---|
| 1. | 4.699 | 222.21 |
| 2. | 3.327 | 127.58 |
| 3. | 2.362 | 112.91 |
| 4. | 1.651 | 108.34 |
| 5. | 1.168 | 91.80 |
| 6. | 0.833 | 72.56 |
| 7. | 0.589 | 68.23 |
| 8. | 0.417 | 65.61 |
| PAN | - | 64.97 |
| Total | - | 934.21 |

Remember that earlier I had total 1000 gm or 1 Kg of material but due to handling and errors total weight retained was 934.21 gm.

### 2.2 Separation of Oil from Peels Based on Size

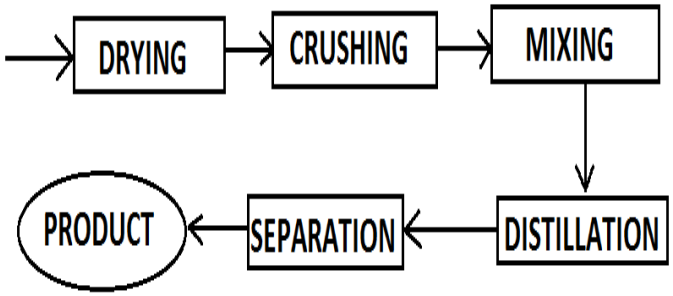


**Fig -2**: Block Diagram for Separation of Oil

a) Simple distillation is employed for removal of essential oil from Orange peel. 50 gm of dried and fined ground orange peel powder from 1st sieve tray is added with 465 ml or 0.465 liters of distilled water which is simple distilled off for approximately one hour. The solid remains of the residue are dried to obtain the dry cake. The distillate resulted into two phases, oil and water. Two phases are separated and orange oil is obtained[15]. For 50 gm of orange powder taken from sieve opening of 4.699 mm, oil obtained is 1.3 ml and 61.22 gm of dried cake is obtained. Similarly, the research was carried out for all the sieve openings with only 50 gm of feed and 465 ml of distilled water.

**Fig -3**: Set-up of Simple Distillation

**Fig -4**: Dried Orange Peel from 1st Sieve in Distillation Set-up

**Fig -5**: Separating Flask

The results are tabulated below:

**Table -2:** Oil and dried cake for different sizes of sieve

| Sr. No. | Sieve Opening (mm) | Weight Retained (gm) | Oil (ml) | Dried Cake (gm) |
|---|---|---|---|---|
| 1. | 4.699 | 222.21 | 1.3 | 61.22 |
| 2. | 3.327 | 127.58 | 1.5 | 60.42 |
| 3. | 2.362 | 112.91 | 1.6 | 60.27 |
| 4. | 1.651 | 108.34 | 2.4 | 58.12 |
| 5. | 1.168 | 91.80 | 2.8 | 56.40 |
| 6. | 0.833 | 72.56 | 3.3 | 52.97 |
| 7. | 0.589 | 68.23 | 3.5 | 52.41 |
| 8. | 0.417 | 65.61 | 3.8 | 51.32 |
| PAN | - | 64.97 | 4.3 | 49.89 |
| Total | - | 934.21 | - | - |

It can be concluded that as the peel size decreases yield of oil gets on increasing which can be seen clearly from the graph below:

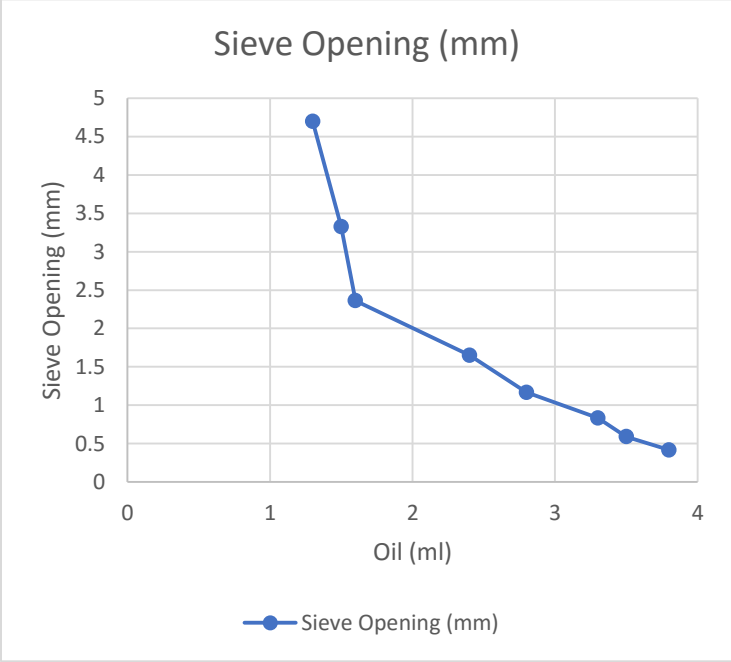


**Chart -1**: Oil obtained v/s Sieve opening

It is clear from the chart that as the sieve opening size decreases amount of oil separated is increased. Hence, we should take minimum size of dried orange peel for the recovery of oil.

b) The method of leaching is also explored for removal of oil from peels. 50 gm of fresh orange peel are extracted with 30 ml of ethanol. After adequate contacting, two phases, solid and liquid are separated. 26.05 gm of wet slurry resulted into 19.73 gm of dry cake[7]. However, oil could not be recovered following this method.

The dried cake obtained in simple distillation is further treated for the separation of pectin.

### 2.3 Extraction of Pectin from Orange Peels

The objective of this part of work is extraction and isolation of pectin from fresh orange peel sample and the dry cake sample left after extraction of oil using simple distillation. The objective includes the manufacturing of pectin from the dried cake left after distillation.

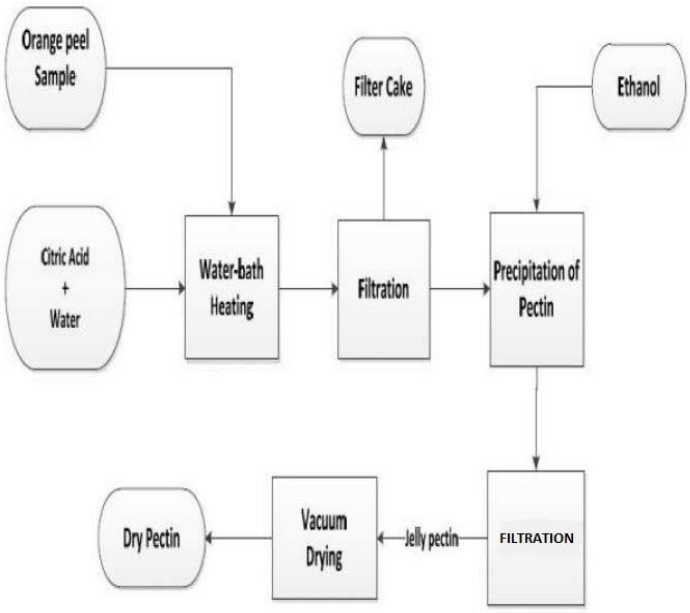


**Fig -6**: Block Diagram for extraction of pectin from orange peel

a) Citric acid in distilled water solutions of desired pH values 1, 1.5, 2, 3, 4 and 5 are prepared. Orange peel samples weighing 10 gm each are dipped into the solution and heated at 80°C for 10 minutes. After cooling the solution, it is filtered using cloth filter and Whitman filter paper under vacuum. Ethanol is added to the filtered solution to facilitate filtration of pectin. The solution is filtered using fine filter cloth or centrifuge at 8000 rpm for 15 minutes at 10°C to separate jelly pectin which is dried under vacuum at 50°C and -100 mmHg gauge pressure for two hours. Dried pectin is thus obtained[13]. Yield percentage of pectin is based on the gram of peel sample taken and is calculated by formula as given below:

$$Y_{pec(\%)}=100\times(p/Bi)$$

Where,

$Y_{pec(\%)}$=extracted pectin yield in percentage

p=amount of dry pectin in grams

Bi=initial amount of orange peel in gram

The maximum yield of pectin is obtained at extraction medium pH of 1.

**Fig -7**: Cloth Filtration

**Fig -8**: Jelly Pectin

## 3. YIELD OF PECTIN

$Y_{pec(\%)}=100\times(p/Bi)$

Where,

$Y_{pec(\%)}$=extracted pectin yield in percentage

p=amount of dry pectin in grams

Bi=initial amount of orange peel in gram

Observation:

Weight of Powder= 10 gm

Weight of dry Pectin=0.31 gm

$Y_{pec(\%)}=100\times(0.31/10)$

Yield (in percentage)=3.1%

**Table -2:** Yield of pectin at different pH values

| Sr. No. | pH of citric acid sol. | Volume of pH solution (ml) | Volume of ethanol sample (ml) | Weight of dry pectin (gm) | Yield (%) |
|---|---|---|---|---|---|
| 1. | 1 | 120 | 50 | 0.31 | 3.1 |
| 2. | 2 | 120 | 50 | 0.19 | 1.9 |
| 3. | 3 | 120 | 50 | 0.30 | 3.0 |
| 4. | 4 | 120 | 50 | 0.26 | 2.6 |

The graph below shows the variation in yield of pectin.

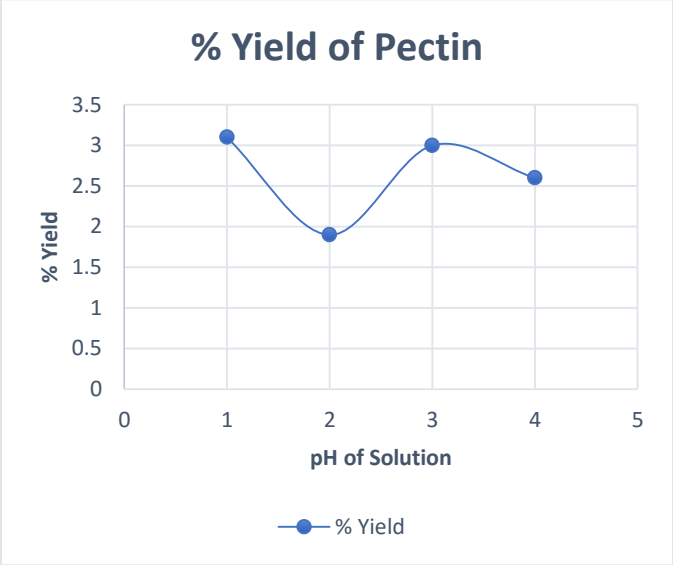


**Chart -2**: pH of solution v/s % Yield of pectin

## 4. STORAGE OF ORANGE OIL AND PECTIN

Orange is an essential oil extracted from the rind of the citrus fruit. Because of its bright color, cheerful scent, orange oil is added to air freshners, soaps and perfumes. Orange oil is also used to flavor beverages and food, hand creams, body lotions, cleaning products and natural deodorants. Orange oil can be combined with other essential oils, such as cinnamon, clove or ginger, to create customized fragrances. Store orange oil properly to ensure that it stays fresh and fragrant[9].

### 4.1 Instructions to Store Orange Oil

- Pour the orange oil into a bottle made of dark glass, such as amber or cobalt blue, to keep out sunlight as sunlight can cause essential oils to deteriorate and loose some of their natural properties. Avoid using plastic bottles, as the orange oil could degrade the material.
- Choose a bottle featuring an orifice reducer, which will allow you to dispense the orange oil a drop at a time.
- Store the orange oil in a wooden box filled with other essential oils if you have a collection.
- Place a single bottle of orange oil in a cool and dry location away from direct sunlight.

### 4.2 Instructions to Store Pectin[10]

- Pectin should always be stored in a cool and dry place, away from direct sunlight.
- The stability of pectin solution and resulting gel was determined by measuring the intrinsic viscosity and gel strength at different storage durations of up to 6 months at 4℃, 25℃ or 40℃[4].

## 5. COST ESTIMATION FOR ORANGE OIL

Cost estimation gives the person a basic idea about the cost of the product. From cost estimation, one can get manufacturing cost of the product and hence, can decide the selling price of the product. But before deciding selling price, keep in mind that the cost of the product should in competition with the market because selling the product at very high cost can put the manufacturer in a huge amount of loss.

BASIS-40 kg of orange powder

- Fixed cost[8]
  1. Cost of land- Rs. 10,00,000/- (for 1000 sq. ft. land)
  2. Cost of shades- Rs.2,50,000/-
  3. Cost of equipment- Rs. 2,00,000/- (Includes all glassware cost)

Total fixed cost= Rs. 14,50,000/-

- Operating Cost[8]
  1. Distilled water-220 L @ Rs. 3 per liter= Rs.660/-
  2. Overhead Charges- Rs. 3000/-

Total operating cost= Rs. 3660/-

Net profit on sales= 25% of operating cost= Rs.915/-

Selling Price of oil=Total operating cost + Net profit on sales

Selling Price of oil= Rs. 3660/- + Rs. 915/-

Selling Price of oil=Rs. 4575/- per liter

## 6. CONCLUSION

Nagpur region is well known in Central Asia as largest orange producing region. It is also known as the California of India, producing excellent quality oranges in a large number. Though it has great production of oranges, the downstream processing and value-added product manufacturing technology is not yet developed. The present work is dedicated for the development of the part of the process technology needed for the extraction of value added products i.e. orange oil and pectin from orange peel, which is the waste of orange juice processing industry. The present work revealed that the maximum amount of orange oil was recovered when dried powder size was minimum and minimum amount of orange oil was recovered when dried powder size was maximum. It is found from the experimentation that the peel source, for extraction of pectin, when taken after extracting orange oil through simple distillation gives higher yield than leaching residue. So, it can be concluded that the process in which orange oil is first extracted using technique of simple distillation followed by acid extraction of pectin is most suitable for industrial production. Also, the maximum yield of pectin was achieved when the pH of citric acid was 1. These results demonstrate the successful extraction of orange oil and pectin, providing potential benefits for industrial extraction of pectin from an economic and environmental point of view.

## REFERENCES

[1]. Shekhar Pandharipande, Harshal Makode," Journal of Engineering Research and Studies" E-ISSN0976-7916, Separation of oil and pectin from orange peel and study of effect of ph of extracting medium on the yield of pectin.
[2]. Jerome V. Diaz, Gordon E. Anthon, And Diane M. Barrett, J. Agric. Food Chem. 2007, 55, 5131-5136, Nonenzymatic Degradation of Citrus Pectin and Pectate during Prolonged Heating: Effects of pH, Temperature, and Degree of Methyl Esterification.
[3]. Manufactured by Douglas Laboratories 600 Boyce Road Pittsburgh, PA 15205 800-245-4440, D.L.Acidophilus + Pectin.
[4]. Pranati srivawscava & Rishabha malviya, Indian journal of natural products & resources vol.2(1), March 2011," Soureses of pectin, extraction &its application in pharmaceutical industry.
[5]. Rajendra Awasthi**,** International Journal of Pharmacy and Pharmaceutical Sciences," Selection of pectin as pharmaceutical excipient on the basis of rheological behavior"
[6]. Ong Hui Fong," Extraction of essential oil from orange peels"**,** January 2012.
[7] Tamara Allafa, Valérie Tomao, Colette Besombes, Farid Chemat," thermal and mechanical intensification of essential oil extraction from orange peel via instant auto vaporization".
[8]. Muhammad Mushtaq Ahmad, International journal of agriculture & biology 1560–8530/2006/08–2–186–190**,** Comparative Physical Examination of Various Citrus Peel Essential Oils.

[9]. Presentee pour l obtention du, Academie D'aix-Marseille, "Hydrodiffusion assistée par micro-ondes.Nouvelle technique d'eco-extraction d'antioxydants".
[10]. Maryam Haghighi and Karamatollah Rezaei, The ScientificWorld Journal Volume 2012, Article ID 967407, 12 pagesdoi:10.1100/2012/967407, "General Analytical Schemes for the Characterization of Pectin-Based Edible Gelled Systems".
[11]. Pranati Srivastava1, and Rishabha Malviya2, the Natural Products Journal, 2011, 1, 65-70 65, "Extraction, Characterization and Evaluation of Orange Peel Waste DerivedPectin as a Pharmaceutical Excipient".
[12]. Luigi Mondello, Alessandro Casilli, Peter Quinto Tranchida, Rosaria Costa, Paola Dugo, and Giovanni Dugo, Journal of Chromatographic Science, Vol. 42, September 2004, "Fast GC for the Analysis of Citrus Oils".
[13]. Tekeste Yeshitla Gebre, International Journal of Scientific & Engineering Research, Volume 3, Issue 10, October-2012 1 ISSN 2229-5518, "Estimation of Solutes in Orange Peel Extract for Pectin Production.
[14]. Chia-Min Lin, Shane-Rong Sheu, Shu-Chen Hsu, Yung-Hsiang Tsai, Food Control 21 (2010) 1710e1715, "Determination of bactericidal efficacy of essential oil extracted from orange peelon the food contact surfaces".
[15]. K. Satish Kumar 10600036, "Extraction of Essential Oil Using Steam Distillation".
[16]. Parth N Patel, Krupa M Patel, Dhaval S Chaudhary, Khushboo G Parmar, Henil A Patel, Chandni D Kansagra and Dhrubo Jyoti Sen, Extraction of Herbal Aroma Oils from Solid Surface
[17]. Health & Safety checked, December 2009, "Extracting limonene from oranges by steam distillation".
[18]. Dr. Stefan Pecoroni,Rudiger Flocke, Wolfgang Gunnewig, "Separators, Decanters and Process Lines from GEA Westfalia Separator for Citrus Processing " Page no. 8.
[19]. Creative Commons Attribution, 28 March 2014.
[20]. E. Haypek, L.H.M Silva, E. Batista, D. S. Marques, M.A.A. Meireles and A.J.A.Meirelles , "Recovery Of Aroma Compounds From Orange Essential Oil", Braz. J. Chem. Eng. vol.17 n.4-7 São Paulo Dec. 2000.