Extraction system for the industrial use of essential oil of the subtle lemon (Citrus aurantifolia)

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Abstract: The use of essential oils as raw material in formulation of by-products allows the revitalization of industries and regional economic growth. This is the reason why in this work it is described the process to design an extraction system of essential oil from subtle lemon. Main parameters required for the extraction have been considered as well as, the calculation of the obtained yield. Within the system design, distillation tank measurements, condenser, essential oils separator, mechanical part of the equipment, boiler selection and the distribution of the plant are dimensioned. The procedure manuals and equipment operating manuals were prepared for each step of the process of using this oil.

1. Introduction

Citrus fruits have been cultivated for about 4000 years, being fruits the most attractive product for the primitive settlers. Gradually, its global distribution has been accelerated, being taken to Europe by the first travelers due to its captivating appearance of its flowers and properly fruits [1-3]. Genus Citrus family comes from the tropical and subtropical zones of the Asian continent and the Malay Archipelago [4, 5]. The territory that associates its origin is commonly located in Southeast Asia (east of the Philippines), it also includes the south of the Himalayas reaching Indonesia [6, 7]. Specifically, the subtle lemon (Citrus Aurantifolia) is a small tree that reaches 4m height, whose fruit has an edible acid taste and extremely fragrant1. Botanically, it is a hybrid species between C. Médica (citron or French lemon) and C. Aurantium (bitter orange) [8]. In Ecuador, production is mainly centered in provinces such as Manabí, Pichincha, Los Ríos and Loja, where most of the national production is in the coastal region (61.64% of the total) [9]. According to statistics from the Ministry of Agriculture and Livestock (SIAGRO-MAG), in 2005 there were 5180 hectares in the entire country dedicated to the cultivation of citrus fruits in which 34.94% (including all limes and lemons varieties) are found in the province of Manabí, 15.44% in Pichincha (and adjoining areas), 13.32% in Los Ríos and 9.02% in Loja².

The extraction of by-products has become a priority axis in industrial field. This particular case handles citrus fruits as the raw material [10, 11]. In the work of [12] the extraction of pectin and d-lemon from the orange and lemon peel is respectively described. This process is innovative and
environment respectful since it uses only water as dispersion method and microwaves as the power source. Laboratory and semi-industrial scale results are compared, obtaining high performance in the quality of the bioproducts developed. The microwave hydrodiffusion and the oil extraction by gravity of the lemon peel made on a semi-industrial scale is presented in [5]. This allows not only extracting high quality essential oil, but also selectively adjusting the composition of the oil by varying the hydrodiffusion time. For its part in the research of [13] the essential oil of lemon balm (Melissa officinalis L.) is obtained by hydro distillation. Both the performance of the essential oil and its composition showed a significant difference between the strains investigated. These differences were associated with fluctuations in climatic conditions in the research years.

From lemon, several by-products and their derivatives can be extracted, but it is the essential oil that generates the greatest interest in the industrial field [14-16]. Therefore, this project presents the industrial use of subtle lemon essential oil. For this, the following guidelines were taken into account: design of an equipment, a plant, technical parameters considerations, costs for the implementation of the plant and start-up (manuals). This study increases the knowledge about the existence of essential oils, their properties and uses for applications in different industries such as: pharmaceutical, cosmetic and food, promoting information from the catalog of aromatic plants.

This article is organized as follows: the introduction in section 1, in section 2 the methodology is shown. Section 3 presents the extraction equipment design. Results of the experimental tests carried out and the conclusions and future work are described in sections 4 and 5 respectively.

2. Methodology

Obtaining the Raw Material

The raw material was provided by the owners of the farms belonging to the Association of Agribusiness Producers "Reina del Cisne del Cantón Catamayo" (AGROCISCA). It was transported to the Process Engineering laboratories of Loja, Ecuador to be stored and its subsequent use in distillation.

Essential Oil Extraction

To obtain the subtle lemon essential oil, three distillations were carried out with different samples of vegetable raw material in the amounts of 4 Kg, 8 Kg and 12 Kg. Those samples were extracted in a steam distiller operated as shown in Figure 1.

![Figure 1. Essential oil process of extracting.](image)

Extraction Parameters

Within the extraction parameters, the following were considered:

- Accumulated performance.
• Pressure.
• Cooling water flow.
• Steam current flow.
• Condensate current temperature.
• Volumetric flow of condensate water.

Packaging and Storage of Essential Oil
The essential oil obtained from the distillation was placed in hermetically sealed amber jars and labelled following the coding: starting with the initials of the species, date of distillation and the total volume obtained from the distillation. Afterwards, the product was refrigerated at a temperature of -4 °C in order to avoid degeneration by light, since its components are very volatile.

3. Extraction Equipment Design
For the design of the extraction system there are: the extraction tank, serpentine type condenser, direct fire heater and the auxiliary equipment coupled to the distiller. All of them are identified with letters as shown in Figure 2.

Volume of the Extraction Tank
The volume of the tank was designed according to the capacity of vegetable matter that is needed to obtain 1 Kg of essential oil. Based on this, the free volume within the tank, the volume of water and the volume of vegetal matter were taken into account. The sequence based on the methodology is presented in Figure 3.
Figure 3. Sequence for the volume design of the tank

**Design of the Extraction Tank**

An energy balance within the thermal design of the extractor tank was carried out in order to determine the total heat of the system, which allows to establish the amount of gas (LPG) consumed during the extraction by the direct fire heater. Calculation sequence is presented in Figure 4. For the dimensioning, the distillation of several plant species was considered, whose density will vary, which causes the condensate flow to decrease or increase considerably. Being conscious of the above, the authors propose the design of a serpentine type condenser that was sized to double the calculation obtained (i.e. 1.3 m). This standardized tube is 40½ inch, giving a greater condensate flow cooling. The new length will be 3.1 m consisting of 10 rolling laps, submerged in a tank of 12.5 L capacity with a diameter ratio of 0.20 and height 0.44 m respectively. The tank was implemented of 304 stainless steel.

Figure 4. Sequence for the thermal design of the extractor tank.
Heat Insulation Thickness

To calculate the thermal insulation thickness, a network of thermal resistances corresponding to the layers, internal convention, stainless steel, air and to the external convection was considered. The calculation sequence is presented in Figure 5. The insulation thickness for the extractor tank was 3cm of glass wool, since the extraction tank has 0.2 cm thickness of stainless steel and a length of 70 cm. The diameter/height ratio should be between 1:1 to 1:1.5, so a device with a storage capacity of 100 L and lengths of 60cm and 70cm in diameter ($d$) and height ($h$) respectively is proposed.

Dimensioning the Heater to Direct Fire

For the direct fire heater, the 0.2 cm thick mild steel material was selected. It consists of a support with a diameter and height of 70cm and 40cm respectively, with 4 sup-ports of 0.5 cm diameter each. In addition, the heater contains a regulating key for the gas pass (LPG), with a diameter of 0.4 cm, which connects to a direct burner of 17 cm in diameter.

![Diagram of thermal insulation thickness calculation]

Essential Oil Separator

The essential oil separating equipment is based on the density difference principle, where the condensate water will be evacuated from the bottom, while the oil will be extracted from the top of the equipment. To achieve the aim, the most representative equipment has been selected in the market for the extraction of essential oils: the collection funnel and the Florentine type separator.

Installation and Selection of Auxiliary Instruments

For the selection of auxiliary equipment, it has been considered:

- Selection of pipes.
- Selection of stopcock accessories.
The pipe used to transport steam, which goes from the outlet of the extractor tank cone to the inlet of the condensate tank, is 304 stainless steel. Additionally, a commercial separator was chosen.

**Zoning and Distribution of the Plant**

For the plant zoning, the necessary spaces were taken into account where the activities to obtain and store the essential oil are to be carried out, additional areas were also considered for the correct functioning of the plant. For plant distribution, the relationship between different zones was considered, in such a way that flow of raw material or products does not interfere with the production process, and by the way transport times are reduced. In Figure 6, an architectural plant is dimensioned with rectangular shape with measures of 16.5m x 9m (148.5 m²). Half-height walls are used to separate some areas, maintaining a visual connection between spaces. Additionally, sliding doors to save space in narrow places are used, with large windows to allow the sunlight come inside. Columns for the structure are made of concrete except for the portal, where wooden columns with concrete base are established and cover is made of mostly brick, covered with a paint layer.

At this point, the authors consider the criteria for non-existence of cross-contamination and the spaces occupied by all the equipment. For the plant distribution, the following aspects were taken into account:

- Location of the plant.
- Daily production of essential oil.
- Production process from the entry of raw material until obtaining the final product.
- Dimensioning of distillation equipment, according to the requirements of the process.

**Technical specifications for the start-up**

The design and dimensioning of the plant were developed with the AutoCAD Software 2016. For the start-up of the extraction plant, a guide was designed that complies with the following aspects:

- Equipment operation manuals.
- Procedure manuals.
4. Experimental Results

The essential oil extraction tests have been carried out by the steam drag method of 4 Kg, 8 Kg and 12 Kg of raw material and the results obtained are shown in Figures 7, 8 and 9 respectively. These quantities of raw material are used since, if the quantity is greater, the distillate flow becomes slower. The union of water and raw material must cover a maximum of 75% of the space, so that the remaining 25% (or more) allows the steam circulation.

Figure 7 shows that 13.2 mL of product is obtained in 60 minutes of essential oil extraction from 4 Kg of vegetable matter, after this time the volume increases 0.36 mL (13.56 mL). Similarly, with an amount of 8 Kg, in 100 minutes, a volume of 41 mL is reached and consequently only 2.63 mL, so that the distillation time of 230 minutes is unnecessary.

Figure 7. Distillation curve of 4 Kg of raw material.

Figure 8. Distillation curve of 8 Kg of raw material.
In Figure 9 the trend is maintained since in 100 minutes of 12 Kg of raw material, 61.75 mL is obtained and then only 2 mL, which causes unnecessary energy expenditure. Finally, Figure 10 shows the results of accumulated performance with a quantity of 12 Kg, where it can be seen a 95.3% performance achieved in a time of 100 minutes. After this time, the energy expenditure is unnecessary. Technical parameters proposed for the extraction of subtle lemon essential oil are presented in Table 1.

| Data                               | Values | Units |
|------------------------------------|--------|-------|
| Atmospheric pressure               | 701    | Hmm   |
| Temperature inside the tank        | 93.5   | °C    |
| Condensate water flow              | 45     | mL/min|
| Cooling water flow                 | 4477   | mL/min|
| Cooling water temperature         | 23     | °C    |
| Water temperature-distillate oil  | 43     | °C    |
| Distillation time                  | 100    | min   |
Calculation of the essential oil performance

The essential oil obtained by distillation is previously separated from the water in a Florentine and is collected in a test tube to measure the volume extracted. Said volume is transformed into units of mass (g) and the performance formula described in (1) is calculated.

\[ R = \frac{m}{p} = 100\% \]  

(1)  

Where:

- \( R \): Performance as percentage.
- \( m \): Amount of obtained oil (g).
- \( p \): Amount of plant material (g).

Investing 10 Kg of raw material, 36.12 g of essential oil was obtained, which produces an efficiency of 0.361% calculated based on (1). The total heat in the heat exchanger results in a value of 5875.01 BTU/h. Starting from an energy balance, results of the internal and external diameters for the dimensioning of the heat exchanger tubes are presented in Table 2. For the tubes area and length, values of internal and external heat coefficients were obtained, which are shown in Table 3.

| Table 2. Diameters of pipes. |
|-----------------------------|
| Outer tube | Inner tube |
| Ft | Ft |
| Internal diameter | 0.02016 | Internal diameter | 0.0016 |
| External diameter | 0.02656 | External diameter | 0.00768 |

| Table 3. Parameters for the dimensioning of the heat exchanger. |
|---------------------------------------------------------------|
| Parameters | Results | Units |
|-----------------------------|----------|--------|
| Global coefficient of heat transfer (U) | 444.519 | BTU/(h) (°F) (ft²) |
| Heat transfer area (Å) | 0.091 | ft² |
| Length of the tubes (L) | 3.7 | ft |

5. Conclusions and Future Work

In this project a 100L tank has been designed, in which information of several plant species is included, according to the production in different stages of the year. In addition, a 3m long coil-type condenser is also designed inside a 12.5 L capacity tank. In order to offer all dimensions required for the extraction process to be carried out, an architectural plant of 150 m² is implemented, divided into 9 sections. All this information is based on the manuals developed for each phase of the distillation process, as well as for the equipment used in the extraction process.

Considering the execution of an industrial production, at least 4 distillers should be placed in operation with an advisable quantity of vegetable raw material of 10 Kg (an adequate portion according to the design which is also easy to use). Since it is pretended to carry out an industrial production and the obtained yield is 0.361%, it should use 276.85 Kg to get 1 Kg of final product. This percentage of performance is low compared to other types of raw material but when making a cost/benefit ratio it is determined that it is profitable for an industrial production. Through the experimental tests it has been shown that 100 min is the optimum distillation time for obtaining subtle lemon essential oil, since performance obtained is approximately 95%, then the energy expenditure is unnecessary considering that volume acquired is minimal.

As future work, the design and implementation of the recirculation system for the cooling fluid is proposed, in order to reduce costs and optimize the extraction process. In addition to the development of a contingency plan as a security measure required throughout the industry.
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