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EXTRACTION OF ESSENTIAL OIL FROM ORANGE PEEL (CITRUS SINENSIS) BY STEAM STRIPPING

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ABSTRACT

Aims: In the present work, the objective was to obtain essential oil from the orange peel (citrus sinensis), through the use of simple distillation. Subjects and Methods: For this, combinations of sodium bicarbonate at (0,3%; 0,5 % and 0,7%) by completely random design. The oil and water separation were carried out by decantation. In the obtained oil, the relative density, refractive index, acidity was analyzed and the best treatment was determined by the oil yield. Finally, a chromatographic analysis was performed in the best treatment. Results: Next, the analyzes carried out, of the 4 types of essential oils obtained, the A2 and A3 treatments have a density close to that recommended, in the refractive index the A2 treatment is better than the others, in acidity the A1. However, in performance, which was our experimental response, treatment A3 (5% $NaHCO_3$) was the best with a value of 7,60mL. After chromatographic analysis, 18 separate components were identified, of which 5 were confirmed (α-Pineno, β-Pineno, Limonene, þ-Cimeno and Linalol). In addition, components such as: Ter-Terpinene, Methylanthranilate, Benzaldehyde, Citral, Linalyl Acetate were observed. Conclusion: The concentration of $NaHCO_3$ helps to extract the essential oil from the vegetable tissues of the orange peel.

INTRODUCTION

Essential oils are not chemically pure substances, they are made up of several compounds, until now the yield of essential oil from citrus has reached 0,4 – 0,8%, most of these have their boiling points within a range of 150 –300°C. In addition, it has been shown that both essential oils and extracts from plant matrices have antimicrobial and antioxidant properties, so that essential oils are of great industrial interest, in food and especially in perfumery. [1,2].

Most essential oils are obtained by the steam entrainment method, a fairly simple process since the mixture must be heated until the volatiles evaporate. Vapors pass through a condenser and go into a liquid state.[2] Chemically the oils are formed by terpenes, monoterpenes and sesquiterpenes (hydrocarbons, alcohols, ketones, etc.) that can be acyclic, monocyclic, bicyclic, tricyclic, sometimes also carry derivatives of phenyl propane and rarely coumarins.[3] Isoamyl alcohol, an important structural unit of essential oils, can be obtained from amino acids containing 6 carbon atoms (leucine).[4,5]

According to their origin, essential oils are classified as: Natural; Artificial and Synthetic According to their origin, essential oils are classified as: Natural; Artificial and Synthetic. [6] Essential oils from orange peel are typical volatile or essential, consisting of mixtures of terpenes, sesquiterpenes, higher alcohols, aldehydes, ketones, acids, esters, and camphor or waxes. [7]

Physical and organoleptic properties of essential oils

Generally, essential oils are liquid at room temperature, volatile with air, they are easily alterable, although they do not become rancid like lipids, they have a tendency to polymerize, it is also considered that the density of the essential oil varies from 0,84 to 1,18 g/mL (characteristic responsible for the separation of the oil from the water at the end of the distillation), likewise, an average refractive index of 1,5. [8,9]

Gas chromatography

There are two types of gas chromatography (GC): gas-solid chromatography (GSC) and gas-liquid chromatography (GLC), the latter being the most widely used, and which can simply be called gas chromatography (GC). This is carried out in a gas chromatograph consisting of various components such as the carrier gas, the sample injection system, the column and the detector. [10]

Gas phase chromatography coupled to mass spectrometry (GC-MS), allows a qualitative analysis to be carried out in a single operation, for a sample of the order of 1 μL, together with an indication of the proportions in which components are found. [11]

The orange (Citrus sinensis, Valencian variety) is one of the most popular and healthy fruits in the world. It has a high content of vitamin C. Its flavor, especially of some varieties, is truly superb for its acidity and sweetness. As all citrus fruits contain 40 and 50% juice, approximately 90% of its content is water with 5% sugars. [12,13]

Ecuadorian orange Valencian variety

Ecuador produces varieties of common and Valencian orange, they are exclusively intended for the fresh market, it is known that this citrus contains numerous very important compounds or nutrients with specific characteristics, among which are essential amino acids and nitrogenous compounds, flavonoids, pectic substances, volatile flavor constituents, vitamins C, A, E, B6, thiamine, riboflavin, niacin, folic acids, pantothenic acid, minerals and lipids. [14]

Considering those previously described, the objective of this work was to extract essential oils from Citrus sinensis orange peel by means of steam dragging

MATERIALS AND METHODS

This research work was carried out in the General Laboratory of the Faculty of Agricultural Sciences of the Bolivar State University.

Experiment management

The previously cleaned orange peel was cut into small segments (0,5 cm), next left to macerate for 24 hours with different percentages of NaHCO3 diluted in water as established in the statistical design (Table 1).

Table 1. Description of the experimental design

TREATMENTS	CODE	DESCRIPTION
1	A_1	Pure water
2	A_2	Sodium bicarbonate 0,3%
3	A_3	Sodium bicarbonate 0,5%
4	A_4	Sodium bicarbonate 0,7%

This method allowed the softening of the vegetable tissue of the orange peel, facilitating the extraction of the essential oil by steam distillation.

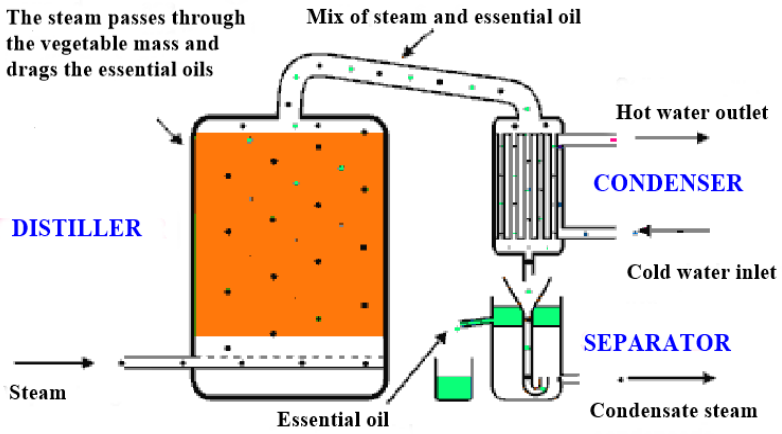

Figure 1. Process of obtaining essential oils by steam entrainment

For distillation, 25% of water was added in relation to the weight of the orange peel. The oil and water separation were carried out by decantation. Finally, the oil obtained was packed in amber containers at 25°C.

Relative density of the oil obtained

The relative density was performed after extracting the essential oil and using a pycnometer previously cleaned with a sulfochromic mixture and rinsed with water.

Refractive index

The sample was placed in the clean and dry prisms of a refractometer (DR-101 BRIX of the IVYMEN brand), for the reading, the temperature was stabilized at 20°C.

Acidity

To the sample in grams, dry, melted and filtered, contained in a 300 mL flask, was added ethyl alcohol, previously neutralized, if the dissolution of the free fatty acids is not complete in the cold, it was gently heated by means of a steam bath at reflux until

complete dissolution, then the phenolphthalein titration was carried out, as established by Arévalo-Martín [14]

The best treatment was determined by the yield of the oil obtained

ANALYSIS IN THE BEST TREATMENT

Chromatographic analysis

The qualitative and quantitative determination of the essential oil was carried out by gas chromatography, using a gas chromatograph equipped with a VARIAN flame ionization detector-Mod. 2.860, the column length was 12 feet, diameter 1/8 inches with Chromosorb support w 60/80 DMCS, the stationary phase was Carbowax 20M, at 15. This analysis was performed in the Instrumental Analysis Laboratory of the National Polytechnic School (Quito).

Statistical analysis

A completely randomized design (CRD) was applied, the treatments applied were previously detailed in Table 1. The experimental size was 1 kg with 3 repetitions.

RESULTS AND DISCUSSION

Relative density

The relative density of the obtained essential oil is distributed in two groups, where treatment A2 has a density of 0,80 g/mL, followed by treatment A3 (Table 2). According to the Mexican standard NMX-F-063-1987 [15], the density of essential oil must be 0,84 g/mL), this indicates that the treatment A1 in terms of essential oil is the purest compared to the others. Similar results were obtained in Colombia by Yánez et al. [16], with a density of 0,84 g/mL and 0,8 g/mL by Díaz et al. [17] Density values higher than that informed in our study were obtained in Ecuador by Telenchana, [18], with a mean relative density of 0,88 g/mL and Bernal, [19], with 0,89 g/mL, both with a solubility of 20°C.

Refractive index

Four different groups are observed in the refractive index of the oil, numerically the treatment A1 (0% sodium bicarbonate) and A2 are superior to the others (Table 2). This decrease in refraction in treatments is due to the concentration of sodium bicarbonate, which has a greater number of total solids, which prevents the passage of light, concluding that the lower the NaHCO3 concentration, the higher the refractive index. Similar values of refractive index were obtained by Díaz et al. [17] and Mendoza and Pérez, [20], with a value of 1,47; likewise, Cerón–Salazar and Cardona–Alzate, [21], with 1,45.

Acidity

In the acidity analysis, four different groups were identified, where treatment A1 (0% sodium bicarbonate) had higher acidity (Table 2), close values were obtained by Cerón–Salazar and Cardona–Alzate, [21], with a mean of 0,29; It is important to consider that as the sodium bicarbonate concentration increases, the acidity decreases. NaHCO3 influenced both physically and chemically due to its characteristic alkalinity, allowing stability of the acids in the orange peel. [22]

Table 2. Tukey ordered ranges for physical analysis of essential oil.

Treatments	Relative density	Refractive index	Acidity
A_1	0,84b	1,4721a	0,53a
A_2	0,80a	1,4716b	0,35b
A_3	0,81a	1,4708c	0,28c
A_4	0,83b	1,4703d	0,20d

Performance in essential oil

For this analysis, the volume of oil obtained after distillation was considered.

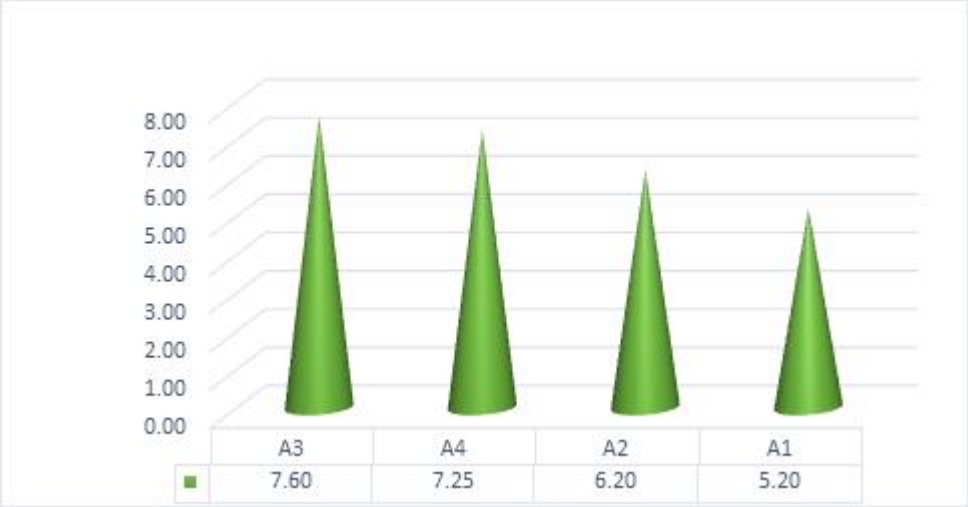

Figure 2. Yield of the essential oil obtained

Statistically, it is appreciated that there is a high significant difference between treatments in the volume (mL) of oil obtained, where treatment A3 (0,5% sodium bicarbonate) was superior to the others, followed by treatment A4 (0,7% sodium bicarbonate) (Figure 2), concluding that, the ideal concentration of $NaHCO_3$ for obtaining the maximum essential oil is 0,5%. Results of 5,73% in performance were obtained by Bernal, [19]; and Mendoza and Pérez, [20], with 1,2%; unlike, Cerón–Salazar and Cardona–Alzate, [21], who obtained 60,32% but through mathematical simulation, in such a way that our results are quite concordant.

Determination of some components for gas chromatography

In the chromatographic analysis shown in Figure 3, eighteen separate components were observed, of which 5 were identified, these being: α-Pineno, β-Pineno, Limonene, þ-Cimeno and Linalol. In addition to these components, the presence of 8-Terpinene, Methylanthranilate, Benzaldehyde, Citral, Linalyl Acetate was detected; all of these could not be confirmed.

Table 3: Identification of the components of orange oil by gas chromatography.

# Peak	Sensitivity	Percentage	Compound
1	64×10^{-11}	2,10	----------
2	"""	2,00	----------
3	"""	2,00	----------
4	"""	2,00	----------
5	"""	2,00	----------

6	"""	2,00	----------
7	256 x10^{-11}	89,00	α-Pinene
8	"""	0,07	----------
9	512 x10^{-10}	89,00	β-Pinene
10	"""	89,00	Limonene
11	"""	36,00	----------
12	"""	35,00	----------
13	"""	32,00	----------
14	64 x10^{-11}	52,00	Cimene
15	"""	15,00	----------
16	"""	14,00	----------
17	"""	24,00	Linalol
18	"""	12,00	----------

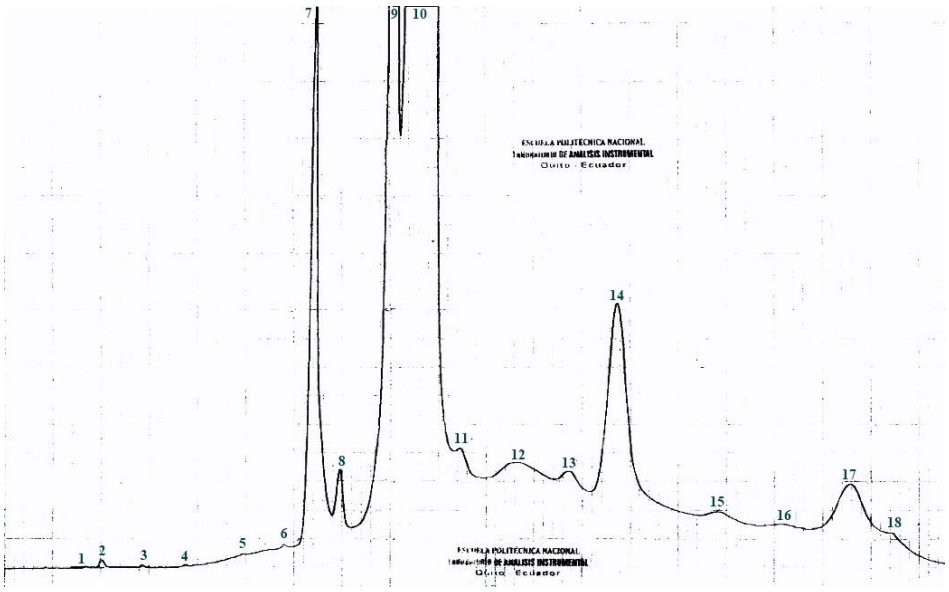

Figure 3. Chromatography in the essential oil of orange peel.

Our country Ecuador does not have standards for essential oil from orange peel, which is why, the work of Soria [23], was taken as a reference, where he extracted essential oils from tangerine and identifying the following: 77, 60%, limonene, 11,20%, p-cymene, less than 10% pinene1, pinene2 and linalol. Comparing our research (Table 3), we found the same components with a slight numerical difference in some of them.

CONCLUSION

It was determined that after steam distillation, the highest volume of oil obtained was from treatment A3 (0,5% Sodium bicarbonate), 48 hours of maceration in sodium bicarbonate, therefore, the concentration of $NaHCO_3$ aids in the extraction of essential oil from the plant tissues of the orange peel.

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