Determination of the liquid-solid relation for the hydrodistillation process of *Cymbopogon citratus* (DC.) Stapf

Paz-Estacio, Wiliams (E-mail: agi2017071@uea.edu.ec)\(^a\), Radice, Matteo (E-mail: mradice@uea.edu.ec)\(^a\), Scalvenzi, Laura, (E-mail: lscalvenzi@uea.edu.ec)\(^a\), Guardado-Yordi, Estela, (E-mail: e.guardadoy@uea.edu.ec)\(^a,b\), Diéguez-Santana, Karel, (E-mail: karel.dieguez.santana@gmail.com)\(^a\) and Pérez-Martínez, Amaury, (E-mail: amperez@uea.edu.ec)\(^a,b\)

\(^a\) Universidad Estatal Amazónica

\(^b\) Universidad de Camagüey

**Graphical Abstract**

**Abstract**

Essential oils are mixtures of secondary metabolites which can be used as additives for food, cosmetic and pharmaceutical industry. Hydro-distillation technique is one of the most common extraction process but optimization studies are quite uncommon. The present study performed a general mass balance concerning the hydro-distillation of *Cymbopogon citratus* oil in order to determine the input and output currents. the following solute (leaves)-solvent (water) relationships (g/g) were defined: 50-200; 100-200; 150-200; 200-200; 250-
200; 300-200. The greatest amount of oil and the lowest decrease in yield was performed with the following solute/solvent ratio: 200/200 (g/g), obtaining an amount of 1.7 mg of oil and a yield of 1 mg oil/leaves. The present study suggests increasing solute/solvent ratio investigation in order to optimize hydro-distillation process.

**Keywords:** hydrodistillation, *Cymbopogon citratus* oil, solute/solvent ratio.

---

**Introduction**

*Cymbopogon citratus,* which common name is “hierba luisa”, is a plant belonging to the Poaceae family, well known in the Andean region of South America [1]. This plant is widely used by indigenous communities in Ecuador, in the form of decoction or infusion as an antispasmodic, hypotensive, antipyretic, anti-catarrhal and tranquilizer remedy [2]. Moreover, *C. citratus* has been reported also a useful herbal source for functional food and beverages, due to its antioxidant, anti-inflammatory and antibacterial activity [3]. Emphases should be taken on the use of such a unique herb for various food and pharmaceutical purposes.

Essential oils are mixtures of secondary metabolites obtained by various techniques such as steam distillation, hydrodistillation, extraction with supercritical CO₂ from plants belonging to different botanical families [4]. The use of essential oils is wide, due to the high content on compounds showing antimicrobial properties against several species of insects, bacteria, yeasts and fungi. Essential oils are also used in food coatings, cosmetics and medicines [5]. Essential oil extraction is usually carried out by hydrodistillation and steam distillation. On hydrodistillation, plant raw material is submerged in water at its boiling point, volatile components are evaporated, condensed and separated [6]. The aim of present study was to determine the solute relation for the hydrodistillation process, considering a given capacity.

**Materials and Methods**

**Plant material**

Fresh leaves of *C. citratus* plants were collected in Pastaza province (Puyo, 10 de Agosto parish). Damaged leaves were discarded and standardized raw material was washed with potable water, in order to remove dust and external agents.

**Essential oil extraction**

The essential oil was extracted from fresh leaves of *C. citratus*, using a Clevenger type glass distiller, using the hydro-distillation technique (Figure 1) [7].
Once the plant raw material was washed, it was chopped in order to obtain a greater contact surface area. The experimental installation was assembled (Figure 1) according to the following parts: a distillation equipment, tripod, mesh, 1000 mL balloon, heat exchanger, double nut clamps, support and a Bunsen burner, which was the source of heat. The balloon was filled with water and raw material. The hydrodistillation began and the first drop was obtained after 40 minutes. The distillate was collected in a glass flask in order to avoid a change on the perfume. This distillation procedure was repeated several times and allows to obtain two products: the essential oil and the hydrolate. Immiscible liquid separation was carried out with a separator funnel and essential oils were stored in tubes and labeled. Finally, for each one of the solute/solvent ratio treatment the following fractions were weighed: residue from the distillation process, the essential oil and the hydrolate. Yield was determined for each one of the mentioned fractions, as reported by Scalvenzi at al. [8].

General mass balance was performed respectively to determine the input and output currents. Additionally, graphs were prepared to visually show the distribution of data, and thus summarize the information obtained. These graphs were made considering the volume of essential oil and the yield, depending on the solute/solvent ratio. The yield being the mass of the essential oil obtained per gram of C. citratus; and the solute / solvent ratio equal to the C. citratus / water ratio.

The essential oil yield was determined on fresh leaves and was the result of six separate hydrodistillations. After extraction, essential oils were stored in tubes and labeled and visual comparisons of the different volumes were made. During the stages of the distillation, condensation and separation process, the following solute (leaves)-solvent (water) relationships were defined: 50-200; 100-200; 150-200; 200-200; 250-200; 300-200.

**Results and Discussion**

Table 1 shows the results of weight measurements related to the input streams (Water and Leaves) and the output streams (Water + leaves, Oil and Hydrolate). The rest of the currents were determined through mass balances. The 200/300 ratio was the one with the highest amount of essential oil. It can be seen in Table 1 and Figure 2; when the leaves amount increases, the oil amount increases too.
Table 1. Experimental results of the currents weight and mass balance.

| Amount (mg) | Distillation | Condensation | Separation |
|-------------|--------------|--------------|------------|
|             | Input Leaves | Water | Output Steam+Oil | Water+leaves | Input Steam+Oil | Water+Leafes | Input Water+Oil | Hydrolate | Oil |
| 50          | 200          | 146,6 | 103,4 | 146,6 | 146,6 | 145,9 | 0,7 |
| 100         | 200          | 128,4 | 171,6 | 128,4 | 128,4 | 128,4 | 127,4 | 1 |
| 150         | 200          | 129,8 | 220,2 | 129,8 | 129,8 | 129,9 | 128,2 | 1,5 |
| 200         | 200          | 117   | 283   | 117   | 117   | 117   | 115,3 | 1,7 |
| 250         | 200          | 145,3 | 304,7 | 145,3 | 145,3 | 145,3 | 143,3 | 1,9 |
| 300         | 200          | 127,6 | 372,4 | 127,6 | 127,6 | 127,6 | 125,4 | 2,2 |

Observing Figure 2, directly proportional ratio of the essential oil amount can be determined as a function of solute/solvent ratio and inversely proportional ratio of the yield based on the solute/solvent ratio.

The amount of essential oil mass increases simultaneously when there is an increase in the solute/solvent ratio, since by increasing the C. citratus/water ratio there is a greater amount of plant raw material and thus a greater extraction of its essential oil. The yield decreases simultaneously when there is an increase in the solute/solvent ratio, since increasing the C. citratus/water ratio occurs a greater compaction of the bed of C. citratus leaves influencing the process of hydrodistillation, and in turn in the extraction of its essential oil, thus causing a decrease in the yield. The progress of oil quantity and the performance demonstrates that increasing the quantity of product oil to increase the solute is not necessarily a good technological choise, because the yield tends to decrease. In the case of the solute/solvent ratio 0.5 and 0.75, the performance is maintained with the same value corresponding to 1, so it can be suggested that these relationships are fine for this capacity.

![Figure 2. Oil production trends and yields obtained for each of the solute-solvent ratio](image)

**Conclusion**

The increase in the solute/solvent ratio from 50/200 to 300/200 mg fresh leaves/mg water increases the amount of oil produced by 214% and the yield decreased by 47%. This showed that the capacity of the equipment could be variable and not a fixed value as recommended.

The solute/solvent ratio that was obtained with the greatest amount of oil and the lowest decrease in yield was 200/200 with an amount of 1.7 mg and a yield of 1 mg oil/leaves.

**References**
1. Bussmann, R.W.; Sharon, D. Plantas medicinales de los andes y la amazonia - la flora mágica y medicinal del norte del perú. *Ethnobotany Research and Applications* 2018, 15.

2. Rojas, J.; Ronceros, S.; Palacios, O.; Sevilla, C. Efecto anti-trypanosoma cruzi del aceite esencial de *cymbopogon citratus* (dc) stapf (hierba luisa) en ratones balb/c. *Anales de la Facultad de Medicina* 2012, 73.

3. Ranjah, M.A. Lemongrass: A useful ingredient for functional foods. *International Journal of Food and Allied Sciences* 2019, 4, 9.

4. Scalvenzi, L.; Yaguache-Camacho, B.; Cabrera-Martinez, P.; Guerrini, A. Actividad antifúngica *in vitro* de aceites esenciales de *ocotea quixos* (lam.) kosterm. Y *piper aduncum* l. *Bioagro* 2016, 28, 39-46.

5. Barrera, L.; García, L. Actividad antifúngica de aceites esenciales y sus compuestos sobre el crecimiento de *fusarium* sp. Aislado de papaya (*carica papaya*). *Revista Científica UDO Agrícola* 2008, 8, 33-41.

6. Peredo - Luna, H., A.; Palou - García, E.; López - Malo, A. Aceites esenciales: Métodos de extracción. *Temas Selectos de Ingenieria de Alimentos* 2009, 3, 24 - 34.

7. Mohamad, N.; Ramli, N.; Abd-Aziz, S.; Ibrahim, M.F. Comparison of hydro-distillation, hydro-distillation with enzyme-assisted and supercritical fluid for the extraction of essential oil from pineapple peels. *3 Biotech* 2019, 9, 234.

8. Scalvenzi, L.; Yaguache Camacho, B.; Guerrini, A.; Radice, M.; Chiurato, M. Efectos de los aceites esenciales amazónicos de citrus limon y *cymbopogon citratus* sobre el crecimiento de hongos fitopatógenos. *Revista Amazónica Ciencia y Tecnologia* 2016, 5, 206-217.