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Extraction of Essential Oil from Onion (*Allium Cepa*) using Hybrid Fluid Mixture (Ethanol and CO_2) as Solvent

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Abstract

Essential oils contains the essence of the plant's fragrance, characteristic of which it is derived. To develop of this study, the extraction was carried out in a laboratory scale unit, for temperature and pressure previously defined, having as solvent the carbon dioxide and ethanol mixture. The onion sample used as raw material was previously chopped and dried and the essential oil was extracted. The essential oil produced was purified and the yield determined by the relation between weigh of essential oil produced and initial onion mass inside the extractor tank. In all cases, the proportion of CO_2 flowrate used in the mixture, have impacts on the essential oil recovery and the products obtained were characterized in term of density, acid index and refraction index. With the result from this research, we can conclude that the best performance of extraction of essential oil was obtained with high flowrate of CO_2 in the mixture, that result in high extraction yield.

Keywords: Essential Oil, Supercritical Solvents, Conventional Solvents, CO_2, Extraction Processes.

1.Introduction

For Reddy [1] and Tongnuanchan and Benjakul [2], the natural essential oils is present in structure of cell plants, mainly in the flowers, leaves, bark, roots, fruits that can be separated using hydro-distillation or extraction with solvent. Essential oils are a combination of low molecular mass chemical compounds, which contain: alcohols, polyphenols, terpenoids, carbonyl compounds, aliphatic substances, and they have distinct aromas and possess biological properties.

For Cavalcanti [3] and Sharmeen et al. [4] the essential oils are applied as additives in cosmetic, pharmaceutical, food, textile and perfumery industries when incorporate the functional characteristics in these products. For other hand these oils incorporate vegetable energy and organic compounds in many industrial products mainly in the foods to preserve flavorings content and fragrances, vitamin supplements, chemical standards and among others.

Commonly, the extraction processes use of an extractor and a conventional solvent or supercritical fluid, or both, known as a hybrid mixture, where the proportion can be adjusted to improve the extraction yield [5].

The type of extraction used in these processes have good relation with the quality of products, in term of the purity degree of essential oil produced and the recovery capacity. The concepts of mass transference in the interface between the solid-liquid or solid-gas phases can be used to measure the efficiency of those process.

Dos Santos et. al [5] state that the extraction process based on knowledge of mass transference allows identified the procedures to enhance the quality and quantity of the products. In this way, Shakir [6] states that with the increase of the extraction temperature, increase the interaction between the phases in the system, which results in the increase the mass transference from solid to liquid phase.

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Oligae, Sean and Androoz [7] and Danlami et. al. [8] observed that the solvent extraction involves the contact between the solvent and the solid material, allowing the transference of solute from solid to solvent phase, based on the solubility principle. For this, extraction occurs due existence of concentration differences between the phases involved in this process. Meanwhile, the concentration of solute in the solid phase decreases due the transference this component to the solvent phase, until the establishment of the equilibrium between the phases involved in this process, characterized by the absence of mass transfer (Figure 1).

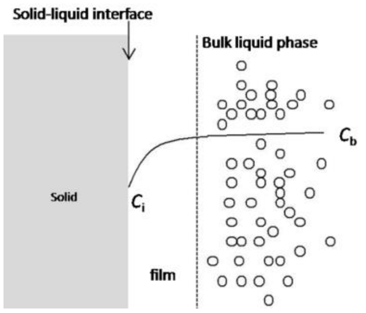

Figure 1. Mass transference between solid-liquid phases

Filippis [10] developed study using CO_2 as supercritical solvent and the results obtained showed good performance of production of essential oil, in term of technology and economic. The study developed by the author involved more than 90 types of plant and showed the importance of operational variables on the capacity of production. For the author, the technology and operational conditions used in each study showed the best relation for each type of essential oil produced, which is associated with the content of each compound.

Between the technologies more explored in development of science, associated with the extraction of natural oil, stands out the hydro-distillation, based on direct contact between solid and vapor phase, when occur the mass transference of essential oil to vapor phase, as showed in Figure 1.

Thus, to increase of extraction yield, the temperature, pressure, vapor flow rate and particle size of the solids should be controlled in the operational unity. In this way, one of more adequate strategy is based on optimization operational conditions to increases the extraction efficiency and yield. After this step, the essential oil produced should be purified to answer the international standards of quality [11].

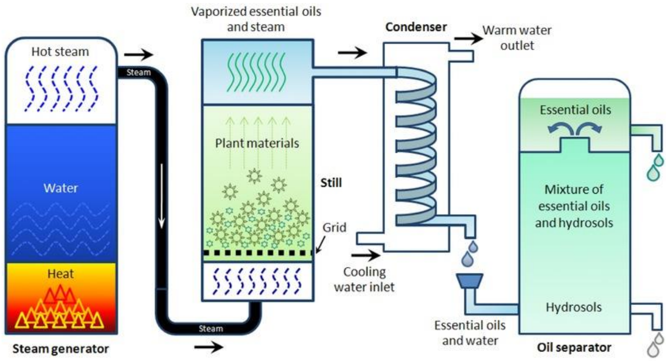

Figure 1. Description of hydro-distillation process [2]

The oil extraction principles are applied in industrial processes. For this case, the particles are reduced to the appropriate size, based on principles of particle size analysis, to extraction of essential oil. Thus, the particles are placed in the extractor tank, ensuring sufficient porosity for steam runoff. Following that, the solvent is placed in the evaporator, when it receives the thermal energy needed to produce the steam that cross the particles present in the extractor tank, when the essential oil is transferred to the vapor phase. Those processes are based on the mass transference principles and result in the essential oil production.

The application of extraction processes in industrial scale involves of the mass, heat and movement transference phenomena, necessary to understand those type of operations. For this the difference of ebullition temperature between the essential oil and solvents should be explore to removal the solvent, using evaporation conceptions.

The conventional procedures used to produce the essential oil from plant materials show as main limitation the instability of this substance due the high temperature applied in the extraction processes. For this, many research have been developed to use the supercritical fluid (SCF) as alternative to traditional methods [5, 12, 13, 14].

Numerous authors have been published the supercritical extraction model and show the relevance of those processes when compared with that from conventional [5, 15, 16], and a broad discussion of modelling aspects is presented elsewhere [8, 9, 14, 17, 18, 19, 20, 36, 37].

For Dos Santos et al. [5], the EO extraction with supercritical fluids (SCF) has been studied and applied by different industrial segments, especially in the food, pharmaceutical and cosmetic industries [22, 23, 25, 27].

Supercritical fluid extraction, generally hybrid, involve the use of supercritical fluid and a co-solvent, to intensification of bubbling in the solid mass, to increase the extraction yield (Figure 2).

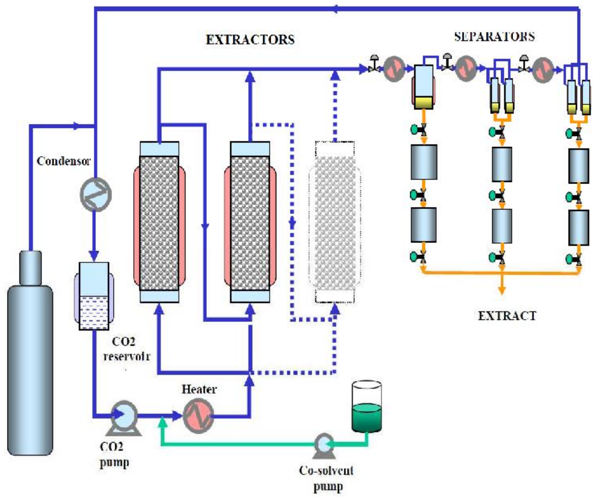

Figure 2. System used in supercritical extraction [24].

With the conditions expected above, should be intensify the mass transfer rate and improve the performance operation to guarantee the applicability of supercritical fluid as an alternative to improve the extraction in industrial processes (Huang et al [26], Silva et al. [27] and Michielin et al. [28]).

With these considerations, was proposed here the extraction of essential oil from natural plants, using hybrid solvents (conventional solvent (ethanol) and a supercritical fluid (CO_2)). The best operational conditions used in this study involve the temperature, pressure and solvent flow rate. Was evaluated too the ratio between masses of plants and solvent to determine the best operational conditions with capacity to increase the mass transference and yield of the essential oil production process.

2. Materials and Method

In this study, an experimental extractor system was built, that involves the ethanol and supercritical fluid (CO_2) as a hybrid fluid mixture to extract essential oil from onion. The interaction between solid and vapor phases was evaluated in the extractor tank and was related with the flow rate of ethanol-CO_2 mixture. In this process, the CO_2 was bubbled into ethanol inside in distillation balloon, where the mixture was heated with thermal energy from heating blanket.

The effect of onion mass transfer efficiency was also investigated.

The onion sample used was chopped and dried with controlled circulating air speed and the air was heated using electrical resistance, in order to improve the efficiency extraction. The results are represented in form of graphs that showing the mass loss over the time.

2.1.Experimental Procedure

2.1.1.Raw Material Preparation

In this study the onion (*Allium Cepa*) was used as the raw material, which was chopped into cubes, with surface area, approximately, of 16 mm2, an adequate size for essential oil extraction. The chopped onion was dried, with the control of temperature, residence time and loss of water mass over time. The onion drying was performed considering a total mass loss of 50%, average dry air flow velocity of 1.7 m/s, operating temperature of 40ºC ($\pm$1.2) and average onion particle size of 4 mm. On the other hand, the extraction time used in the process was 240 minutes.

2.1.2. Extraction Procedure

Dos Santos et al. [5] built an experimental apparatus that was adjusted and used in this study and that enabled the acquisition of data presented in this scientific work (Figure 3).

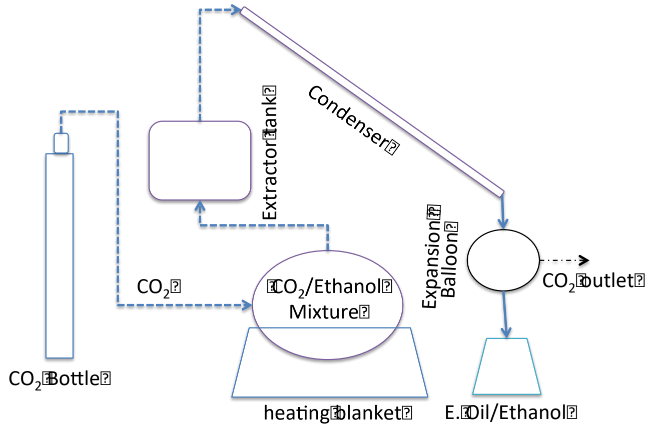

Figure 3. Experimental apparatus used for essential oil extraction [5].

The apparatus built by Dos Santos et al [5] was used and comprise three sections: a) section of mixture and heating of conventional and supercritical fluid; b) Section of extraction of essential oil; c) section of recovery the essential oil.

The experiments were performed using the apparatus proposed by Dos Santos et al. [5], Figure 3, applying the following sequency:

a) placed the raw material in the extraction tank;
b) Cooling the water in the condensation system;
c) Inside the ethanol in the balloon;
d) Heated the ethanol in the balloon;
e) Recirculate the cooling water in the condenser system;
f) Oppen the bottle of the CO_2 valve and flowing of supercritical fluid to bubble into the ethanol;
g) Flowing the mixture fluid to contact with the solid phase;
h) Mass transference from solid to mixture fluid is registered;
i) Recover the ethanol and essential oil in the expansion tank.

The procedure described herein was used to carry out the essential oil extraction using onion as the raw material, as well as the effect of operation parameters on the quality of the process was evaluated (Table 1).

Table 1. Operational conditions used to carry out the experiments.

Nº	Parameters	Exp. 1	Exp. 2	Exp. 3
1	Initial onion mass (g)	255.8	614.97	184.73
2	Pressure (mm Hg)	760.0	760.0	760.0
3	Temperature (°C)	63.4	62.6	55.4
4	CO_2 flow rate (l/min)	0.5	1.44	3.60
6	Ethanol mass in balloon (g)	480.0	481	478
7	Size of raw material particles (mm)	4.0	4.0	4.0

The performance of each experiment was evaluated based on Equation 1.

$$\eta\ (\%) = \frac{Mass\ of\ oil\ extracted\ (g)}{Initial\ mass\ of\ biomass\ in\ the\ extrator\ (g)} * 100 \quad (1)$$

2.1.3.Essential Oil Purification

The essential oil produced from extraction process, describe herein, was purified using vacuum simple distillation that involves heating the liquid mixture to the boiling point and immediately condensing the resulting vapors, and filtration to remove contaminants, supported by technical norms.

2.1.4.Characterization of Essential Oil

The essential oil purified was used to determine the physical parameters as acid index, refraction index, viscosity and specific mass.

The procedures used are described in the ASTM D 974 American technical norm and the EN14103 European standard

3.Results and Discussion

The results from the experimental assays, described above, were used to analyze the performance operational of each experiment and the data used to discussion the process performance, supported by data from literature, to better consistence in the scientific and technological development.

3.1.Drying of Raw Material

Table 2 shows the results of drying of raw material (onion) evaluated in this research, whose experiments were conducted based on methodological procedure described above. The results in the table describe the mass loss over time, as loss of water in the structure of the raw material. The experiments were conducted with control of operation parameters, mainly flowrate air and temperature, parameters associated with the contact time between air and raw material, which result of mass transference from solid to gas, based on existence of concentration gradient between the phases involved in those processes.

Table 2. Results obtained for drying of raw material over time

Nº	Time (min)	Mass (g)	Mass loss (%)	Evolution of mass (%)
0	0	1784.8	0	100
1	30	1543.5	11.46	88.53
2	60	1340.2	20.52	79.47
3	90	1161.3	27.72	72.27
4	120	1094.7	34.38	65.61
5	150	965.1	40.82	59.17
6	180	940	45.28	54.72
7	210	917.2	49.32	57.68
8	240	892.4	52.41	47.59

In Table 2, the mass was measured for each 30 min. With this procedure, was registered loss 892.4g of mass, which represent 54.42% of the initial raw material mass. These data were used to design the drying profile presented in Figure 4, which relate the mass losses during the drying time. The drying curve shows the transference mass from the solid to gas phase, on the first hour of drying, that represent 39.15% of the global mass loss.

In the second hour, the mass loss represents 26.45% of the global mass, in the third hour 20.80% and in the last hour 13.5%.

In this process, the mass is transferred by convection from the solid surface to vapor phase and it is proportional to solid area and the transference coefficients. Equation 2 relate the mass transference, related with global coefficient and concentration gradient.

$$N_A = k_c \Delta C_A \quad (2)$$

Where N_A is the flowrate of water (kmol/m^{2}s), k_c is the global coefficient and ΔC_A is the concentration difference between the solid particle and the airflow.

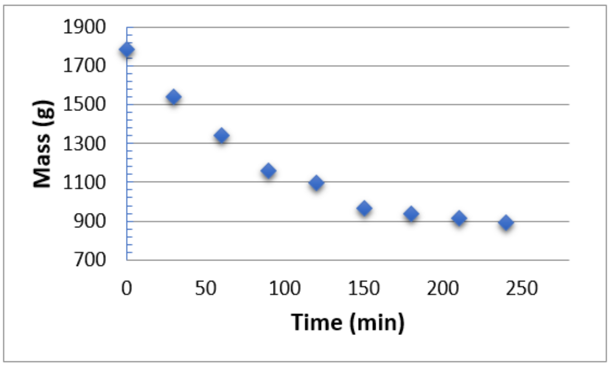

Figure 4. Curve of onion drying

The mass transference rates decrease substantially until relative stability and the reduction is related to the drying time to guarantee the best condition for essential oil extraction.

3.2.Essential Oil Extraction

3.2.1.Experimental Apparatus

The experimental apparatus, proposed by Dos Santos et al [5], was used to carry out the experimental assays applied to produce the essential oil, from dried onion (Figure 3).

3.2.2.Experimental Data Acquisition

In this study the phenomenon as turbulence and effectivity of mass transference were observed and identified limitation that difficulty the use the conventional solvent and that reduce the yield of this process.

For the initial study, was used the Soxhlet apparatus to carry out the experiments. For each assay, was made five solvent discharges, to increase the capacity of extraction of essential oil, from solid onion particles.

Using this procedure, the analyze of extraction results present global yield of the 27.93%. With this limited capacity were tested the insertion of supercritical fluid with conventional solvent to know the impact of this new combination, based on hybrid principles.

Using the same operation conditions and evaluate the performance of extraction process, with mixture of conventional solvent and supercritical fluid, was observed that the presence of supercritical fluid increase the interaction between solid and fluid, that result in increase of the yield, until 60%. Generally, the increase of supercritical fluid flowrate, in the extraction processes, increase the yield, in the nonlinear proportion.

Table 3 shows the yield effect by the CO_2 flow rate in terms of essential oil recovery, in relation of solubility of the oil with increase of concentration of supercritical fluid flowrate.

Table 3. Essential Oil recovery in each experiment

N°	Initial Mass (g)	Mass of Essential Oil Recovered (g)	CO_2 Speed (ml/min)	Extract Yield (%)	Density (g/ml)	Refraction index
Exp 1	255.80	59.22	0.5	23.15	1.132	1.3455
Exp 2	614.97	213.52	1.44	34.70	1.130	1.3535
Exp 3	184.73	118.15	3.60	63.96	1.127	1.3465

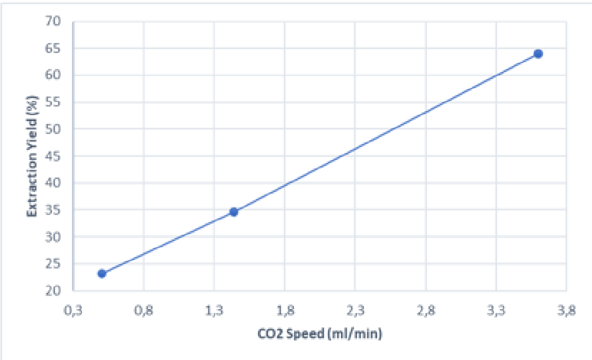

Figure 5. Effect of CO_2 flowrate over extraction yield

The analysis of Figure 4 shows that the increase in the flow of the supercritical fluid in the hybrid mixture with the conventional solvent, promotes the increase of the process performance, in terms of the production capacity of the essential oil. Therefore, process optimization studies must be carried out to identify the ideal operating conditions that can guarantee maximum efficiency

In all of experiments carry out in this study, was used the same operation time. To evaluate the effect of fluid flowrate using supercritical CO_2, Aris et al. [29] and Hassanien [30] concluded that when CO_2 flowrate increase, the extraction yield increase by

3.698%, for every 0.7 ml/min produced. The authors cited above carry out experiments under conditions of low pressure and high temperature, and observed that a rise in the flow rate of 8 ml/min increase extraction yield.

For this case, the solute thus easily moves to the bulk solvent, enhancing the solubilization of the solute in the solvent and increasing the extraction yield (Maran and Priya [31] and Gadkari and Balaraman [32]).

Özkal and Yener [33] observed that when the flowrate of the supercritical fluid increased considerably, increase the mass transference by convection, between solid-CO_2. In this case, this performance can damage the weak parts of the solid particles, leading to freer solute being removed from the solid particle during extraction.

In study of extraction essential oil carried out by Inamuddin and Asiri [34] and Da Silva et. al. [35], using ethanol and CO_2 as solvent, with variate pressures, the author observed that the yield increase with decrease of pressure of CO_2-ethanol system, and the high extraction yield obtained when the experiments was carried out with essay with low pressure.

In the study reported herein, a hybrid system was used, with supercritical fluid and ethanol as the extraction fluid. In this case, the bubbling of the supercritical fluid in a conventional solvent was used to form the mixture, which was applied in the extraction of essential oil.

The essential oil produced in this study was characterized in terms of acid index and refraction index (Table 3). The values obtained for the three experiments were 1.132; 1.130 and 1.127 g/ml, respectively, which are similar of that reported in literature. The acidic index from these experiments, was 3.19 mg KOH/g, while that the data obtained from the literature show an average value of 3.56.

4.Conclusions

We can conclude:

a) The essential oil from extraction using supercritical fluid and conventional solvent provided good yields;
b) The use of CO_2 as supercritical fluid in the extraction processes is the best alternative to improve the extraction performance;
c) Th use of a supercritical fluid to produce essential oil by extraction processes, is more effective than that use of conventional solvent;
d) The design of the proposed equipment that involves the insertion of the supercritical fluid in the conventional solvent to promote a hybrid solvent can be characterized as a technological innovation of this work.

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Compliance with Ethic Requirements: Authors declare that they respect the journal’s ethics requirements. Authors declare that they have no conflict of interest and all procedures involving human or animal subjects (if exists) respect the specific regulation and standards.

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