Multi-Purpose Pilot Unit for Supercritical Fluid Extraction

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Biodiversity of the South American countries has motivated several studies about the technology development of extraction process from vegetable raw material. Supercritical fluid extraction appears as an important unit operation in achieving new volatile and non-volatile natural extracts with application in cosmetic, fragrance, cleaning, and pharmaceutical products, principally. The multi-purpose equipment presents as principal characteristic the operational flexibility with regard to extractor volume, solvent flow rate, temperature range, pressure range, and the co-solvent use, as well as the equipment is designed to operate simultaneously with two extractions in different conditions. This apparatus is equipped with two CO_2 cylinder, two high pressure pumps, one co-solvent high pressure pump, two pre-heaters, three extraction vessels, two variable monitoring software, and one automated control software. Indeed, the equipment advances that are strongly linked to the development of new natural products correspond to auto cleaning system and software of control process (monitoring and storage of the variables). Tests were performed on the equipment with plants native to southern Brazil to obtain extracts volatile and nonvolatile, and some results are presented below: Achyrocline *satureioides* - essential oil and oleoresin to perfumery; *Valeriana glechomifolia* - non volatile extract with antidepressant-like effect, and *Hypericum carinatum* - no volatile extract with antimicrobial and antidepressant activity.

INTRODUCTION

Supercritical extraction is a separation and purification process that has advantages compared with traditional process as steam distillation, organic solvent extraction [1, 2], including non-waste product (carbon dioxide as solvent), a feature which has attracted industrial interest. Then, this research has aim to develop a multi-proposal supercritical extractor in pilot-scale from theoretical conception, equipment assembly, software development, and implementation experiments [3, 4]. The main purpose of the equipment is its operational flexibility: process employed with different raw materials, extraction vessels with various volumes, equipment operation in parallel, extraction with and without co-solvent, and control and storage of process variables in supervisory-control software.

The multi-purpose equipment was designed and constructed with characteristic industrial sensors, temperature sensor, pressure sensor, flowmeter, level sensor, and others, to facilitate the scale up by relatively low cost. The equipment was designed to operate simultaneously with two extractions in different operational conditions (parallel) in a same structure. Moreover, the equipment presents an auto cleaning system which possibility the cleaning after each extraction to avoid contamination of extracts,

characteristic very important when works with different raw-materials, especially for pharmaceutics employments.

MATERIALS AND METHODS

The multi-purpose pilot unit for supercritical fluid extraction was the result of a cooperation project university/industry whose objective was the development of a Brazilian technology of supercritical fluid extraction of the caffeine from *Ilex paraguariensis* [4]. In the Figure 1 is presented the flow chart of the equipment and principal characteristics.

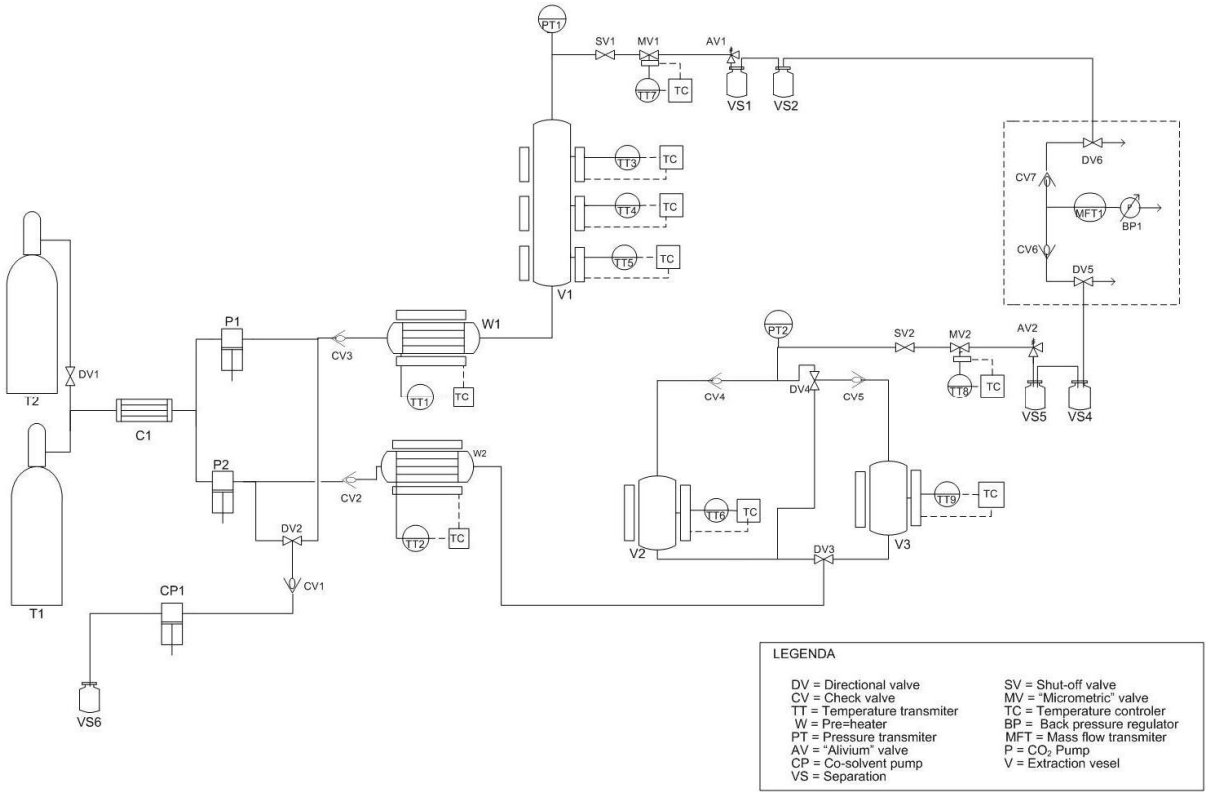


Figure 1: Flowchart of the supercritical extraction pilot scale equipment

The equipment developed is constituted of three extractions vessels: 1000mL (V1), 500mL (V2) and 100mL (V3); two high pressure pumps for carbon-dioxide (P1, P2), one co-solvent pump (CP1); two storage cylinders of CO_2 (T1,T2); two pre-heaters (W1,W2); one condenser (C1); one system to measure the flow of CO_2, two separation vessels, and one automated control system.

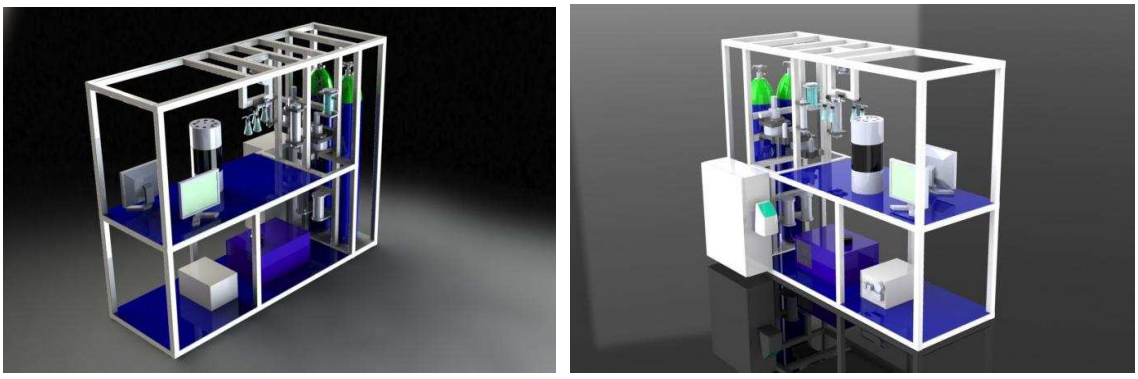
The main characteristic of the supercritical extractor is that the system was developed to work with two extractions simultaneously, with different process conditions in a same structure. This assembles guarantees saving around 30% of investment against two equipments with same characteristics. This fact is possible due to some equipment parts are multiplexed: like co-solvent pump, control system, and the mass flow measurement system of CO_2 as well as the equipment becomes more compact.

As shown in the flowchart of the Figure 1, the carbon-dioxide that is stored in one of both cylinders (T1, T2) flows through condenser (C1) to ensure the solvent in liquid state. This liquid is pressurized in one of both pumps (P1, P2). The supercritical fluid can be introduced in the extractor vessels V1 (1000 mL), V2 (100 mL) or V3 (500 mL). According to the positioning of directional valves DV3 and DV4 the vessels (V2, V3) can work either in parallel or in series. The depressurization occurs in micrometric valves MV1 or MV2 and the extracts are collected in the separation vessels SV1/SV2 or SV3, SV4.

When a co-solvent is applied, the solvent mixture is performed before the pre-heaters. The pump use to co-solvent pressurization is CP1. The co-solvent system is also used to perform the cleaning by pumping organic liquid solvents through of pipe, extraction vessels, and the micrometric valves in order to avoid the contamination of extracts when working with different raw-materials.

RESULTS

The structural design and equipment disposition as cylinders, cooler, preheaters, pumps, pipes, vessels, valves, sensors, and computers of the pilot-scale supercritical extractor was developed in the Design Software SolidWorks. In the Figure 2 is presented the project of the apparatus.



(a) (b)

Figure 2: Project of the supercritical extraction pilot scale equipment – (a) front view; (b) (rear view)

From the basic design, the structure was assembled. The result of this implementation is possible to observe in the Figure 3 that shows the picture of the Multi-Purpose Pilot Unit for Supercritical Fluid Extraction, equipment installed in the Unit Operations Laboratory, Department of Chemical Engineering at the Catholic University of Rio Grande do Sul [3].

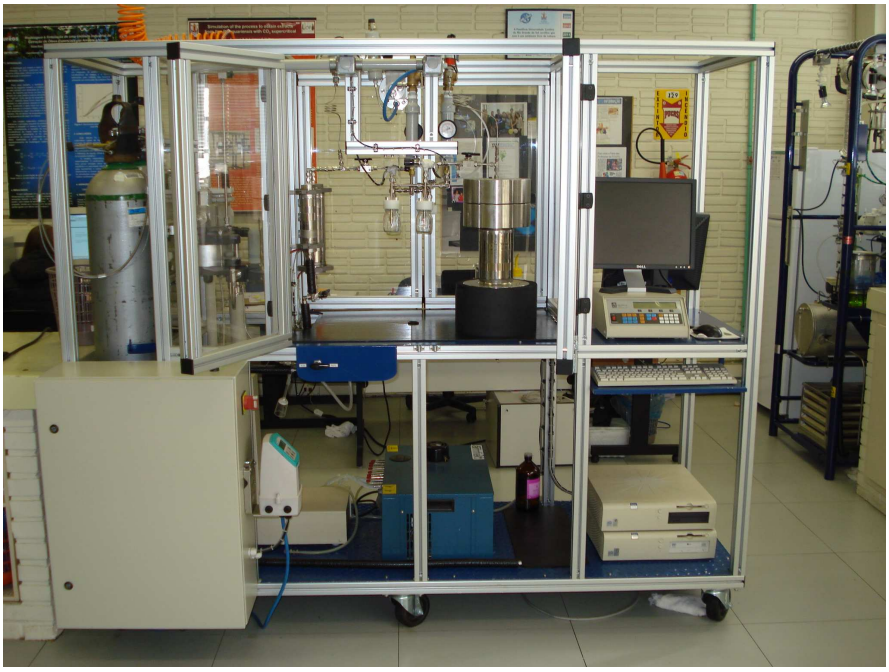


Figure 3: Pilot scale supercritical extraction equipment

Furthermore, an important characteristic of the equipment is that the separation vessel (Figure 4) is easily interchangeable during extraction procedure. This possibility is very important to obtain experimental data for construct the yield curves (yield versus time) and also to obtain different extracts for different extraction time. This information is very relevant to screening of the new products from vegetables matrix and to supercritical process development in industrial scale.

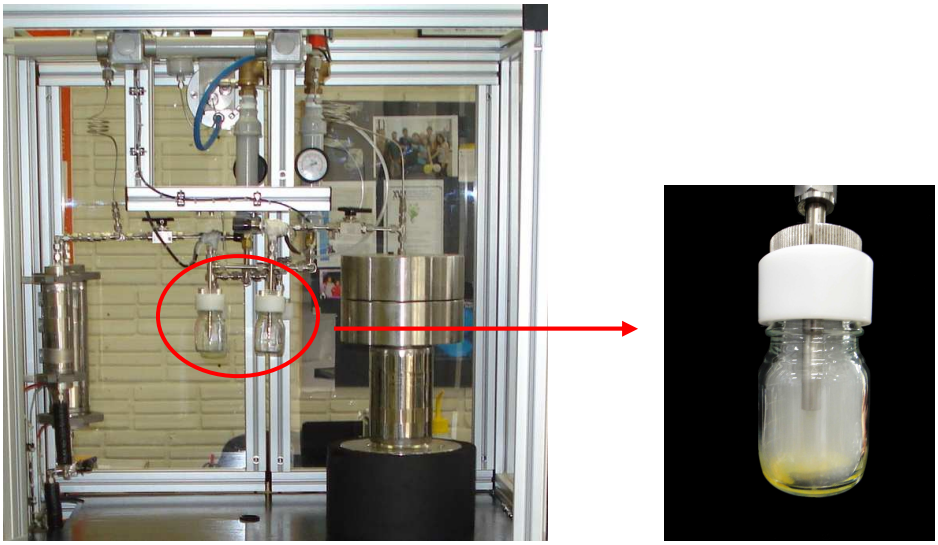


Figure 4: Interchangeable separation vessel

Other relevant advantage of this equipment is the flexibility with regard to capacity of extractor vessels: 100mL, 500mL and 1000mL. This configuration is important because

it is possible to study new products (extracts) in one line - exploratory development, while it is possible to perform a scale up to industrial process in other line with higher capacity.

The main features of the pilot-scale supercritical extraction unit are presented in the Table 1. These properties are related with the objectives of this technologic development, ie, designing an apparatus to extract new products from vegetables matrix and perform the scale up to industrial process.

Table 1: Principal properties of the Multi-Purpose Pilot Unit for Supercritical Fluid Extraction

Properties
• Extraction procedure: serie and parallel
• Parallel extraction: different operating conditions for each line
• Supercritical solvents: carbon dioxide and carbon dioxide + liquid organic co-solvents
• Operational flexibility
• Determination of solvent flow on line (with and without co-solvent)
• Extraction vessel: different volume (100 mL; 500 mL, and 1000 mL); different geometry.
• Separation vessel: easy removal and replacement during the extraction process.
• Automated cleaning system.
• Software of the control, management and security

The equipment has been tested with different vegetable matrices, among them we can highlight: *Achyrocline satureioides* - essential oil and oleoresin to perfumery (Figure 5), *Valeriana glechomifolia* - non volatile extract with antidepressant-like effect [5], and *Hypericum carinatum* - no volatile extract with antimicrobial and antidepressant activity [6].

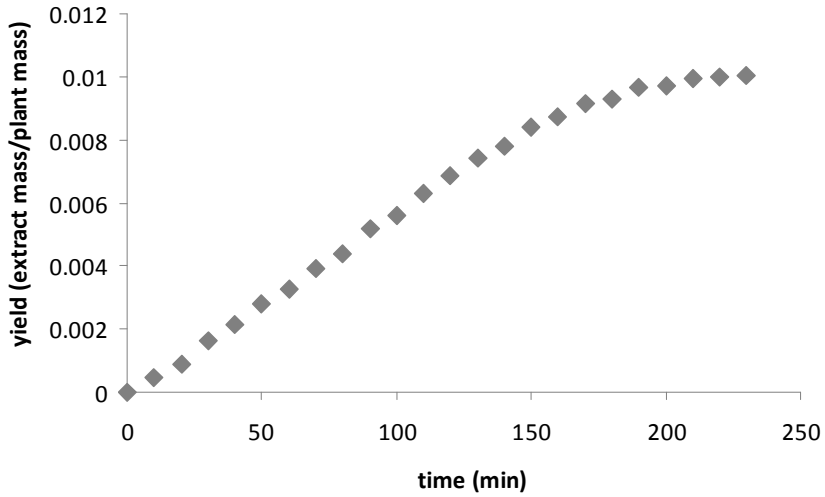


Figure 5: Global yield of the *Achyrocline satureioides* extracts using supercritical CO_2 at 90 bar and 40 ^{o}C:

CONCLUSION

The Multi-Purpose Pilot Unit for Supercritical Fluid Extraction was developed in Brazil to subsidize the sector associated with the natural products from South America biodiversity. In function of diversity of raw materials used, the flexibility of the apparatus will permit technological advances applied to economical sector linked to this type of research.

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