DEVELOPMENT OF A NEW AND ROBUST ULTRASONIC DEVICE FOR SUPERCRITICAL EXTRACTION PROCESSES

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One of the difficulties in the supercritical fluid extraction is to find a way to accelerate the extraction itself and to minimise extraction time for a certain extraction yield. Since high pressures are normally used in supercritical extraction processes, mechanical stirring is difficult to be applied. Important extraction volume losses as well as complex mechanical devices are implied to use mechanical stirrers at supercritical conditions. As an alternative, ultrasounds have been considered since high ultrasonic intensities allow accelerating other processes and as a result, enhancing their performance.

Taking into account previous achievements, the aims of this work were to develop a new self-controlled prototype, robust enough in order to to fulfil industrial requirements to produce it commercially. For this purpose, a new configuration device has been tested under supercritical conditions and applied to supercritical extractions.

INTRODUCTION

Due to the increasingly stringent environmental regulations, supercritical fluid extraction (SFE) using CO_2 has gained wide acceptance in recent years as an alternative to conventional solvent extraction in many analytical and industrial processes. SFE presents important advantages (non-toxic, recyclable, cheap, relatively inert and non-flammable) over traditional techniques [1]. However, one of the main difficulties when applying a supercritical fluid as solvent for extraction is usually slow kinetics of the process. The solubilities of many substances in supercritical fluids are usually lower than in fluids used for conventional extraction processes; therefore the mass transfer rate is smaller [2]. A classical way to accelerate separation or extraction processes is to apply a mechanical agitation system. Another possibility is the use of high-intensity ultrasounds.

High-intensity ultrasounds are elastic waves with frequencies higher than 20 kHz and intensities over 1 W/cm^2. Ultrasonic radiation represents an efficient way of producing deep agitation, enhancing mass transfer processes, because of some mechanisms (microstirring, compressions and decompressions in the material, heating, and cavitation) [3, 4]. In the case of SFE, the characteristics of the equipment do not easily allow the use of mechanical stirrers; therefore, ultrasonic waves could be an interesting alternative for this purpose.

A previous work of this research group pointed out the feasibility of integrating an ultrasonic field inside a supercritical extractor without losing a significant volume fraction. This pioneer method in the extractive processes enables accelerating mass transfer and then, improving supercritical extraction times.

This extraction process gave rise to a non-antecedent patent [5] and up to now, only a system for nano/microparticle generation has been published [6]. This system has nothing to do with the extraction purposes and there is no any commercially available device to enhance the supercritical extraction processes with ultrasounds.

The aim of this work is to develop a new self-controlled prototype, robust enough to fulfil industrial requirements to produce it commercially. For this purpose, a new configuration device has been tested under supercritical conditions and applied to supercritical extractions.

MATERIALS AND METHODS

Raw material

Almonds (*Prunus amygdalus*) from a variety known as "Marcona comuna" were used in this investigation. The raw almonds were blanded, pealed, dried and finally grounded before the extraction process. The particle size was measured using different sieves and was estimated in a range of 3-4 mm.

Equipment description

The experiments were carried out in a SCE pilot plant (**ainia**, Valencia). The supercritical pilot equipment mainly included a high pressure vessel (5L), two separation units (a cyclone and a decanter), a diaphragm pump and different sensors for temperature, pressure and flow rate.

RESULTS AND DISCUSSION

The ultrasonic equipment consisted of a power ultrasound generator, an impedance adapter, a transducer and a control computer. In order to study the effect of the acoustic waves in supercritical fluid extraction, an experimental system based on the integration of a power ultrasonic transducer inside the extractor, inserted on the upper lid of the vessel, was designed and developed, in a similar way than expressed in patented process but focusing on non-previously covered aspects. A scheme of the equipment as described in already achieved patent is shown in figure 1 [7].

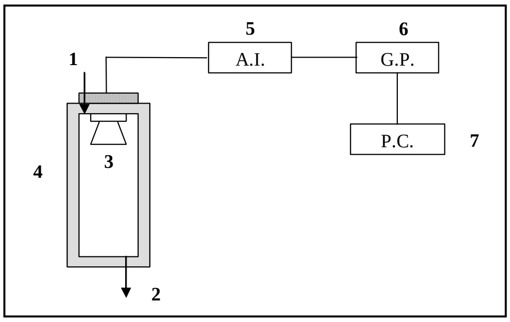

Figure 1. Experimental set-up scheme.

1. Supercritical fluid entrance/exit. 2. Supercritical fluid entrance/exit 3. Ultrasonic Transducer 4. Supercritical vessel 5. Impedance matching unit 6. Power generator 7. Measurement and control computer

Figure 1: Ultrasonic device used for the SCE assisted by acoustic waves

General scheme was applicable also to new system. The transducer is the part of the equipment that generates acoustic waves. New designed transducer implemented definite advantages versus previous version. New transducer was conceived in order to achieve self-control (automatic control), as it is required for further industrial applications. In this case, the resonant frequency of the traducer used was 20 kHz and the power applied up to 100 W. A software developed in Labview was used to monitor the behaviour of ultrasonic parameters (impedance, frequency and power) during the extraction.

Tests were carried out at different conditions, both with and without ultrasounds. Extraction experiments with ultrasounds exhibited faster kinetic curves and higher extraction yields, as it is showed in figures 2 and 3 as an example. This new ultrasonic device led to notable enhancement both in extraction yields at certain times and required time to achieve a certain extraction yield, higher than those previously reported in the previous patented work. For example, experiments carried out at 32 MPa, 318 K and 10 kg/h 28 MPa gave rise to 40% larger yields when US where applied (figure 2). Even larger differences between extraction curves with and without ultrasounds were achieved, as it follows from figure 3. These experiments were carried out at 28 MPa, 318 K and 12,5 kg/h and extraction yields were improved in about 90%. As a reference, previously built-up device led to improvements of about 20% on extraction yields.

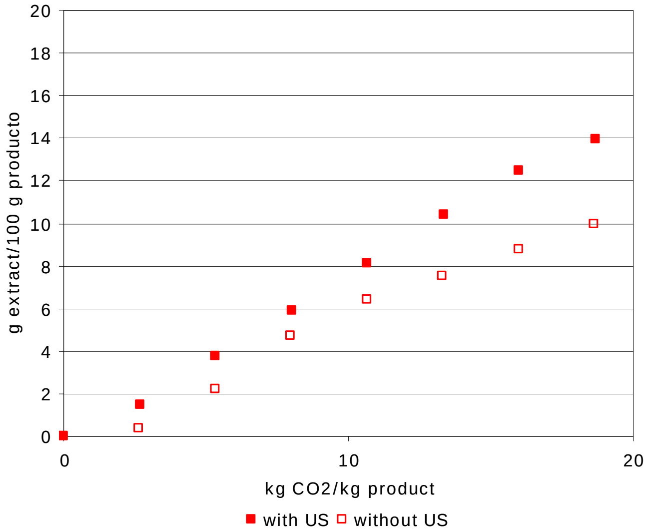

Figure 2: Extraction curves at 32 MPa and 318 K, with and without ultrasounds

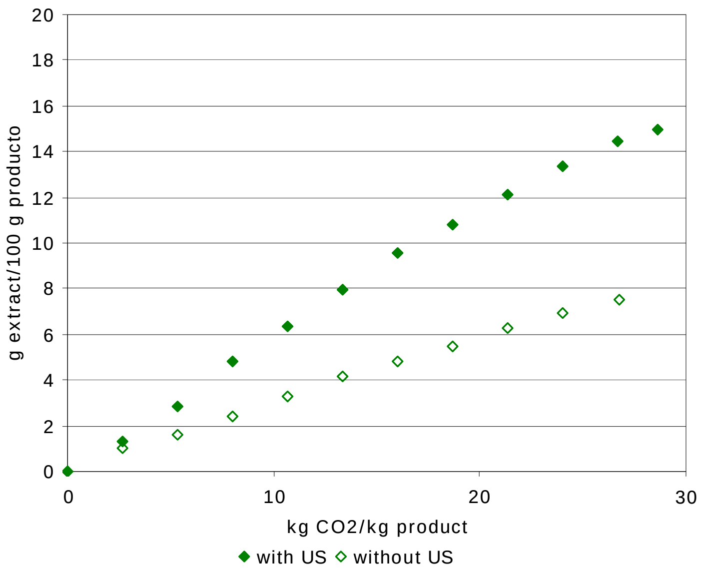

Figure 3: Extraction curves at 28 MPa and 318 K, with and without ultrasounds

Nevertheless, the most important proved fact in this work was related to ultrasonic application system itself. With regard to functional response, new ultrasonic device behaviour was satisfactory. This new device performed according to design targets and solved limitations identified with previously developed device.

CONCLUSIONS

A new system for ultrasonic application in supercritical processes has been designed, build-up and tested. This ultrasonic device performed adequately, without requiring manual correction of parameters. In addition to robust and self-controlling response, the new ultrasonic system also demonstrated to enhance extraction results with respect to experiments carried out without ultrasounds. Whereas previous ultrasonic device led to improvements of about 20% in extraction yields, larger improvements were got with this new system (up to 90%).

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