SUPERCRITICAL FLUID EXTRACTION OF VOLATILE COMPONENTS FROM FOOD PRODUCTS.

R. Martínez-Velasco, L. González-Arnáiz, S. Beltrán*, J. Rovira, M. T. Sanz and I. Jaime.

Departamento de Ingeniería Química. Facultad de Ciencias. Universidad de Burgos. Plaza Misael Bañuelos s/n. Universidad de Burgos 09001 Burgos beltran@ubu. es

Abstract

Supercritical fluid extraction (SFE) takes advantage of both the density of the solvent and the vapour pressure of the solutes for dissolving the solutes to be extracted. In this sense, SFE may be considered a unit operation in between distillation and extraction. This consideration is especially important when extracting flavours, aromas and, in general, volatile components present in natural subtracts, since these components have a high vapour pressure and therefore a tendency to migrate to the supercritical phase, being in some cases totally miscible with supercritical carbon dioxide¹.

Different works on SFE and fractionation of essential oils and related products show that SFE can produce a good reproduction of original flavours or fragrances¹. Extraction at mild pressures (from 90 to 100 bar) and temperatures (from 40 to 50 °C) and fractionated separations at, for example, at 0 °C, 90 bar and 15 °C and 20 bar have been suggested as optimum operation conditions^{2,3}

The aim of this work is to obtain different aroma concentrates for its use in the food industry. Some vegetable and shellfish flavours have been obtained. A semi-pilot plant with a 2 L extractor and two separators (1 L and $\frac{1}{2}$ L) connected in series has been used. The solvent used was carbon dioxide. The experimental work was focused to the optimization of the extraction conditions and the recovery of the aromas recycling the solvent. Wet and freeze-dried subtracts were used.

The extracts were analysed by means of GC-MS and compared with extracts obtained by Solid Phase Dynamic Extraction (SPDE) of the whole subtract. Sensorial analyses and the electronic nose indicated the validity of the different extracts obtained for its use as aroma concentrates in the food industry.

As a specific example, onion SFE extracts, extracted at 300 bar and 40 °C and recovered at 20 °C and 90 and 30 bar in separators 1 and 2 respectively, contained 33 compounds, 13 of which were sulphur compounds that are the type of compounds responsible for the onion flavour. The profile of the extracts was similar in both separators, although concentration was lower in the second one. No significant differences were found either in the profiles of the extracts obtained from fresh and freeze-dried onion; therefore, as a global process for industry, processing fresh onion was recommended although a significant amount of water was co-extracted.

Pressure and temperature in the last separator have to be the lowest the possible for the volatile compounds to condensate. However, pressures lower than 20 bar are unfeasible in plants where pressurization takes place with a pump if non-dried subtracts are processed and recycling of the solvent is desired. The reason is that condensation of the carbon dioxide, to avoid pump cavitation, needs temperatures lower than -18 °C when pressure is 20 bar; thus, small quantities of water remaining in the solvent when recycling it, would freeze giving rise to plugs that would impede the CO_2 flow.

- [1] Reverchón E. Journal of Supercritical Fluids. 1997, 10, 1-37
- [2] Kakasy A.Z., Lemberkovics É., Simándi B., Lelik L., Héthelyi É., Antal I., Szöke É. *Flavour and Frangance Journal* **2006**, 21, 598-60
- [3] Reverchon E. Marco I. D. Journal of Supercritical Fluids. 2006, 38, 146-166