Supercritical antisolvent (sas) co-precipitation of ethyl cellulose and rosemary essential oil in SC-CO₂

Pocheville, A., Garay, I., Hernando, I.

Gaiker Technology Centre, Parque Tecnológico Edif. 202, 48170, Zamudio, Spain pocheville@gaiker.es

Essential oils, which are widely known for their antimicrobial, antifungal and/or antioxidant properties, are aromatic, volatile and labile liquids obtained from plants or vegetables. The food industry is considering consumers claim for substitution of chemical additives (preservatives, antioxidants or flavours) with natural extracts/essential oils in food products and packaging coatings [1]. Because of essential oils instability, a great researching effort is being made for improving their thermal and chemical stability and for facilitating their handling when processing. Looking for solutions, different ways of microencapsulation have been described [2], but the impregnation of these compounds in polymeric matrices is another way of incorporating these active compounds in packaging devices.

The objective of this work has been the co-precipitation of the coating material *Ethyl Cellulose (EC)* and the active compound *Rosemary Essential Oil (RO)* using the Supercritical Antisolvent (SAS) precipitation technique with supercritical carbon dioxide (SC-CO₂), in order to obtain active microparticles. EC is a hydrophobic material used in different fields such as medicine, cosmetics, food products and pharmacology. It has been widely used for controlled delivery systems, sustained release, tablet drug packaging material, long-acting formulations, taste masking or coatings [2]. RO is characterized for its variety of properties, and it is commonly used in food industry, cosmetics, herbal care, aromatherapy and pharmacology.

Supercritical fluids technology, which is considered a green technology, has been studied for different applications in the last decades. In SAS precipitation, SC-CO₂ acts as the antisolvent, so that, when a solution containing the compounds of interest reaches the supercritical medium, the SC-CO₂ dissolves the solvent and the compounds of interest co-precipitate in form of microparticles. A supercritical fluid equipment Thar R100 System with a SAS-50 vessel has been used for the co-precipitation of EC and RO, and different parameters have been studied: pressure, EC:RO rate, flow rate (QCO₂:Qsolution) or solution concentration. The morphology and size of the obtained precipitates were analyzed by scanning electron microscopy (SEM) and the RO impregnation rate was guantified by extraction and further GC-FID analysis. EC agglomerated microparticles (1-4µm) have been precipitated with a very low RO impregnation rate (%ROimpregnation<0.5%). According to the results, the flow rate (QCO₂:Qsolution) is essential for microparticles formation, and particle size increases when a higher Qsolution is used. No noticeable changes in particle size have been observed when increasing operation pressure. Although the obtained RO impregnation rate has been very low, Supercritical Antisolvent (SAS) precipitation using SC-CO₂ is an alternative technique for nano-microparticles precipitation or compounds micronization, with application in different fields (e.g. food industry, cosmetics, pharmacology or materials).

References

- [1] European Patent Application EP 1 657 181 A1, (2006).
- [2] B. Voncina, et al., Textile and Polymer Journal, Vol. 1, No. 1 (2009) 13-19.
- [3] Ana Rita C. Duarte, et al., International Journal of Pharmaceutics, 308 (2006) 168-174.