

A. Calvo, J. Morancie, Sz. Pländer, E. Székely \*

*Budapest University of Technology and Economics, Department of Chemical and Environmental Process Engineering, Budapest, Hungary*

Supercritical fluids have been widely used to extract target components from varied matrices, at analytical and commercial scales. This is an environmentally friendly alternative to conventional organic solvent extraction. Organic solvent clean products can be obtained, and their natural properties are preserved due to the moderate temperatures used. The most widely used supercritical solvent for separation of natural products is carbon dioxide, typically in the ranges of 10-50 MPa pressures and 35-80 °C temperatures.

The focus of this work is to extract and fractionate bioactive natural products as polyphenols (proanthocyanidins) and fatty acids from the seeds of *Vitis vinifera*<sup>2,3</sup>. Controllable ratios of the major biologically active components offer the possibility of tailor-made product development as food additive or supplement or for cosmetic purposes.

Conventional Soxhlet extraction with 50:50 % ethanol-water mixtures was performed to obtain the raw extract. Cellulose was used as a carrier to turn the leachure of the alcoholic extract into a solid material (mass ratio alcoholic extract:cellulose was 1:1) capable to form a porous bed. Further investigations aim to fractionate this solidified raw extract and compare the possibilities of selective extractions directly from grape seeds, from cold pressed oil and grape seed flour.

Effects of cosolvent concentrations (0-15 % ethanol in carbon dioxide) were studied in detail at constant pressure of 30 MPa. The pressure was varied between 100 and 450 bar in the case of extraction with pure carbon dioxide. The temperature was kept constant at 45°C and the solvent flow was 5-6 kg/h.

Studies on *Vitis vinifera* solidified raw extract revealed slightly increasing extraction yields with increasing ethanol concentrations: 2.53, 4.12 and 7.29% yields at 5, 10 and 15% ethanol concentrations, respectively. Supercritical fluid extraction with pure CO<sub>2</sub> at 450 bar led to 4.38% of extraction yield.

The antioxidant activity was evaluated by using the DPPH\* (2,2-diphenyl-1-picryl-hydrazyl-hydrate) free radical scavenging assay<sup>4</sup>, based on to the concentration where 50% of inhibition of the DPPH\* was achieved (IC<sub>50</sub>). It resulted in 7.04±0.76 µg/ml for the initial grape seed extract. In reference to the supercritical extracts, this factor was achieved at 28.54±2.80, 76.31±2.77 and 197.82±27.61 µg/ml for the extracts obtained with 15, 10 and 5% of ethanol, respectively. There was no relevant antioxidant activity in the supercritical extract with pure CO<sub>2</sub>. In the light of these results it is observed that there is a correlation between the polarity of the extracting solvent and the antioxidant activity of the sample, therefore the interesting substance for these purposes could be the supercritical raffinate.

The research work was supported by Marie Curie DoHipp Programme FP7-PEOPLE – 2012 – ITN ‘Training Programme for the Design of Resource and Energy Efficient Products by High Pressure Processes’. E. Székely thanks the János Bolyai Research Fellowships of the Hungarian Academy of Sciences.

1. L. Wang, C. L. Weller, Trends in Food Science & Technology 17, 2006: 300-312
2. J. M. Ricardo da Silva, J. Rigaud, V. Cheymer, A. Cheminat, M. Moutounet, Phytochemistry 30 (4) 1991: 1259-1264
3. G.K. Jayaprakashia, R.P. Singh, K.K. Sakariah, Journal of Chemistry, 73, 2001: 285-290
4. K. Mishra, H. Ohna, N. K. Chandury, Food Chemistry 130, 2012: 1036-1043
5. Y. Yilmaz, R. Toledo, Trends in Food Science&Technology, 15, 2004: 422-433.

\*Corresponding author: sz-edfi@mail.bme.hu

Keywords: grape seed, Supercritical Fluid Extraction, antioxidants, proanthocyanidins