Supercritical Fluid Extraction of St. John's Wort (*Hypericum perforatum* L.) and Marigold (*Calendula officinalis* L.)

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Recently more attention has been paid to the healthy way of life, mainly to the importance of healthy nutrition intake. That means the general use of natural products, food supplements and different natural medical preparations at homes like natural antidepressant (St. John's wort) and anti-inflammatory (marigold) preparations.

The goal of this work was to extract valuable components and natural agents from the St. John's wort (*Hypericum perforatum* L.) and marigold (*Calendula officinalis* L.). In addition to develop food supplements and herbal products from the extracts.

The Soxhlet extraction and supercritical fluid extraction were carried out in laboratory and pilot scales. Three different solvents (ethanol, *n*-hexane, carbon dioxide) were applied in the experiments, all of them are permitted extraction solvents in the production of food and food ingredients in European Union (88/344/EEC).

(1) CO_2 tank

(2) coolers

(3) pump

(4) heat exchangers

(5) extractor

(6) separators

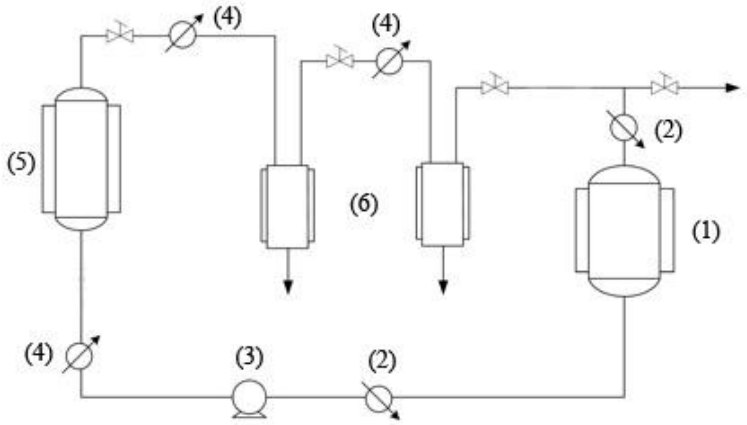

Figure 1.: The equipment of supercritical fluid extraction in pilot plant scale.

The operating method of the supercritical fluid extraction unit depicted in Figure 1. is the following. The carbon dioxide is supplied from the tank (1) through a cooler (2) by a pump (3) and the heat exchanger (4) provides the supercritical conditions. The soluble compounds of herbs are dissolved by the supercritical carbon dioxide (sc-CO_2) in the extractor (5). After the pressure reduction the dissolved components are precipitated in the separators (6).

The effects of the operational parameters (pressure and temperature) on the extraction yield and the recoveries of the biologically active minor compounds were studied using a 3^2 full factorial experimental design. The pressure and temperature were varied over the ranges

of 100-450 bar and 40-60 °C, respectively. The evaluation of the effects were performed by Pareto diagrams and fitted response surfaces using Statistica software (StatSoft Inc., Tusla, OK, USA). The yield and recoveries were compared to those obtained with *n*-hexane and ethanol (96% v/v).

The obtained extracts were analysed by HPLC analytical methods.

The extracts of the studied herbs were applied in product development experiments.

The obtained yields changed between 1.35-44.05 g kg^{-1} in case of St. John's wort according to the solvent power of the supercritical fluid. The extraction yields of the supercritical fluid extraction are comparable to the yield of the extract, obtained with *n*-hexane according to their similar solubility parameters. The yield of the ethanolic extract was almost six times higher than the yield obtained with sc-CO_2 in case of St. John's wort. The evaluation of the extraction yield values obtained by a full factorial design revealed the phenomena of the crossover pressure and indicated that the pressure possesses more significant effects on the yield between 100 and 300 bar than above 300 bar. According to the Pareto chart, the linear and quadratic terms of extraction pressure, the interactions of the temperature, the linear and quadratic terms of extraction pressure, and the interactions of the quadratic terms of temperature and extraction pressure as well had significant effects on the extraction yield values at the studied 5 % significance level.

The recovery of hyperforin from St. John's wort was (g minor component kg^{-1} dried raw material): 0.09-9.56. The statistical evaluation showed that the linear and quadratic terms of the extraction parameters and their interactions had significant effects on the recovery of hyperforin, although above 300 bar the effects of both the pressure and temperature decreased on the yield of hyperforin. In addition the best extract was achieved at 450 bar and 60°C.

In case of marigold the obtained extraction yields changed between 1.69-124.36 g kg^{-1}. The higher results are similar to the values obtained by *n*-hexane in laboratory scale Soxhlet extraction. The extraction yield obtained with ethanol was almost four times higher, than those obtained with sc-CO_2. The recovery of total faradiol-esther from marigold was 0.14-12.75 g kg^{-1}. The linear and quadratic terms of the extraction pressure has significant effect on the recoveries of total faradiol-esther. The fitted response surface (Figure 2.) shows that the best extract can be achieved at 450 bar and 60°C.

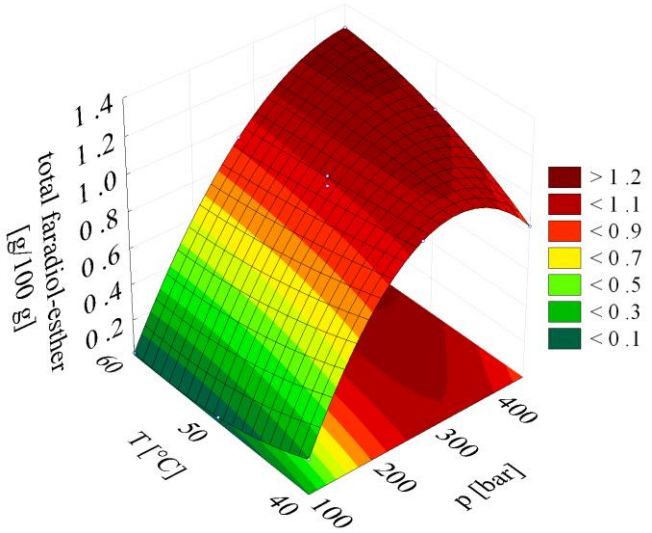

Figure 2.: Fitted response surface for total faradiol-esther of marigold.

The obtained extracts are useful as the ingredients of different organic products or cosmetics in the form as a raw extract or with some additives like cyclodextrins.

ACKNOWLEDGEMENTS

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