**Application of the integrated supercritical fluid extraction-impregnation process for incorporation of *Melissa officinalis* extract into cotton gauze**

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**Highlights**

* Cotton gauze was impregnated with *M. officinalis* extract using SFE-SSI process.
* Three different process modes were tested.
* FTIR analysis confirmed the presence of *M. officinalis* extract on cotton gauze.

**1. Introduction**

Lemon balm (*Melissa officinalis*) has long been used in herbal medicine due to its numerous biological activities including antioxidant, antimicrobial and antiviral [1].When there is a need to impregnate a solid material with a supercritical extract, merging of the supercritical fluid extraction (SFE) and supercritical solvent impregnation (SSI) into the one, integrated, SFE-SSI process enables energy savings as well as minimization of the extract losses [2-4]. The goal of this study was to obtain cotton gauze functionalized with *Melissa officinalis* extract for potential topical application by the integrated SFE-SSI process. The influence of different processing modes on the obtained impregnation yield was studied.

**2. Methods**

*M. officinalis* folium was milled in a blender and sieved prior the experiments. Integrated SFE-SSI process was performed on laboratory scale unit (High Pressure Extraction Adsorption (HPEA) 500, Eurotechnica, Germany), whereby both, extraction and impregnation, were performed at 10 MPa and 40 °C. Detailed equipment description and possible processing modes are presented elsewhere [3, 4].

**3. Results and discussion**

Three types of processing modes were performed in order to obtain maximal impregnation yield (I). **Mode I:** Batch mode - supercritical fluid was circulated through the system (extractor and adsorber) for 5 h. **Modes II and III:** Circulation of supercritical fluid through the system was performed in cycles (Table 1). Between the cycles, a continuous CO2 flow (10 g/min) through the extractor and adsorber was maintained during 5 minutes.

Introduction of fresh CO2 positively influenced the yield of impregnation (defined as the mass fraction of the extract in the functionalized material). Introduction of fresh CO2 while maintaining the total process time approximately equal, enabled 6.5 times higher impregnation yield when Mode III (2.24%) was applied compared to Mode I (0.34%). The introduction of fresh CO2 positively affected the efficiency of extraction process, leading to the higher amount of the extract available for the SSI. Further increase in contact time led to the decrease of the impregnation yield.

**Table 1.** Impregnation yield of *M. officinalis* extract for different processing modes

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
|  | **No of**  **cycles** | **Cycle duration, h** | **Total contact time** | **I, %** |
| **Mode I** | 1 | 5 | 5 h | 0.34±0.035 |
| **Mode II** | 3 | 2+2+1 | 5 h + 10 min flow | 1.48±0.127 |
| **Mode III** | 5 | 1 | 5 h + 20 min flow | 2.24±0.141 |

The FTIR spectra show the main differences between the impregnated and control cotton gauze in the range 3000–2800 cm−1 and around 1700 cm−1 attributed to C–H and C=O stretching vibrations, respectively, confirming that the balm extract was effectively impregnated into the gauze.



**Figure 1.** FTIR spectra of pure *M. officinalis* extract, control and impregnated cotton gauze.

**4. Conclusions**

Cotton gauze was successfully impregnated with *M. officinalis* extract using the integrated SFE-SSI process at 10 MPa and 40 °C. In order to optimize the process, three different processing modes were tested. The presence of extract on the surface of cotton gauze was confirmed by FTIR analysis.

**Acknowledgement**

This work was financially supported by the Ministry of Education, Science and Technological Development of the Republic of Serbia (Project No III 45001 and III45017), as well as the Higher Education Excellence Program of the Ministry of Human Capacities in the frame of Biotechnology research area of Budapest University of Technology and Economics (BME FIKP-BIO).

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