

## EMERGING TECHNOLOGIES FOR MARA SEA BUCKTHORN (*Hippophae rhamnoides* L.) BERRIES VALORIFICATION

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### Abstract

*This paper aims to assess the viability of using novel oil extraction methods for Sea Buckthorn (*Hippophae rhamnoides* L.). Supercritical fluid extraction (SCFE) although extensively used for oil extraction in other countries, is not commercially used in Romania at the moment. Cost constraints, as well as the ease of use, more established methods such as solvent extraction and cold pressing have delayed the implementation of such technologies. Three oil sources were investigated: oils extracted from dry berries using SCFE and cold pressing, and oil extracted from druff (residues after juice extraction) using SCFE. The oils have been analyzed using a HPLC unit, and their carotenoid levels were compared. The results show a slight variation in the carotenoid composition in relation to the extraction methods. This suggests that the SCFE method is viable to be used for large scale Sea Buckthorn oil production.*

**Key words:** Sea Buckthorn, supercritical fluid extraction, cold pressing, oil, Romania, carotenoids.

### INTRODUCTION

Recent trends in nutrition are starting to integrate novel foods into diets. Because of their so called “nutraceutical” values, Foods such as Sea Buckthorn are seen as a viable alternative to conventional sources of vitamins, minerals and other essential elements of a healthy lifestyle (Yang and Kallio, 2001; Upadhyay et al., 2011).

Traditional products from the Sea Buckthorn berries include juices, liqueurs, wine, jams, candy, and ice-cream. However, the berry’s unique chemical and nutritional composition has offered economic potential as a health food (Krejcarová, 2015, Suryakumar and Gupta, 2011). Sea Buckthorn oil contains a large amount of fatty acids, liposoluble vitamins and sterols, and are considered the most valuable part of the berries (Cenkowski et al., 2006; Górnaś et al., 2016).

The method used to extract oil from oil-bearing materials is the limiting factor of its quality, as heat or solvents interfere with its purity and chemical composition. Chemical solvent extraction is at the moment the preferred method for obtaining Sea Buckthorn oil. Its very high extraction efficiency is economically viable,

however, unless properly executed using sophisticated equipment, proper hygienic conditions and strict parameter process control, it has the potential to leave traces of residual chemical solvents in the finished product, making it unfit for human consumption (Bargale et al., 1999).

The SCFE method removes these concerns, as it uses CO<sub>2</sub> in its supercritical state (liquid) to extract the oils from the source material (Jose, 2015; Walker et al., 2007). The main advantage of the method is the ease of the solvent’s recovery after extraction, ensuring that the finished product is 100% pure (Fornari, 2016; Mohamed and Mansoori, 2002).

This paper aims to investigate the differences between different extraction methods, seeing that the nutraceutical market is gaining more and more recognition from Romanian consumers, and demand for alternative nutrient sources is due to rise.

### MATERIALS AND METHODS

#### Raw materials

Ripe berries of Mara sea buckthorn (*Hippophae rhamnoides* L.) were collected from the Biofarmnet plantation, Ialomita county, located

in the South region of Romania. Harvesting was done by cutting whole branches off the shrubs in September through October, then quickly freezing them at -38°C. This allows the berries to be removed from the branch with minimal damage by shaking. The berries are sorted and graded by hand then frozen at -18°C. Stored at this temperature, the berries maintain their physico-chemical properties and can safely be kept for up to 6 months without any significant damage such as freezer burn or spoilage.

Whole berries were dried in a discontinuous drier at 40°C for six days. The dried berries were extracted by cold pressing and supercritical carbon dioxide. For higher valorification of raw material, after the juice processing, the residues (draff) were extracted by supercritical carbon dioxide.

#### **Supercritical carbon dioxide extraction (SFE) of sea buckthorn berries**

Extractions were carried out using a pilot-plant sized supercritical carbon dioxide extractor (Natex, Prozesstechnologie GesmbH, Austria, Fabr. no. 10-023/2011) designed with a single cylinder extraction vessel and two separators. The extraction was carried out for 1000 g of raw material.

The extractor basket was filled with ~ 0.300 kg of ground dried sea buckthorn in three batches. During extraction, the solvent (technical CO<sub>2</sub>, 99.99% purity supplied by Messer S.A., Romania) was constantly chilled to remain liquid and able to be recirculated. The solvent was brought to supercritical conditions at 7.30 MPa, and a flow rate of 20 kg/h, as indicated by the data sheets from ABB software (ABB - Mannheim, Germany). The extraction conditions were carried out using Xiang Xu et.al. (2008) experimental parameters (pressure of 27.6 MPa, temperature of 34.51°C and extraction time of 82.0 min). The oil was collected at the end of the process, weighed and analysed using the HPLC method.

#### **Cold pressing extraction of sea buckthorn berries**

Sea buckthorn Mara seeds were cold pressed on site by Biofarmnet SRL. The dried berries were cleaned and sorted. 10 kg of seeds were added in the receiving funnel of the cold

presser. Soon after, 5 kg of oil was obtained. The resulting oil was subjected to a sedimentation period of three days, in order to remove impurities and protein residues.

The oil was then filtered using a 5 micron cloth, then moved into brown glass bottles to protect against oxidation.

Pressing was done using a FARMER 20 cold press with the Farnet Duo screw press attached by FARMET.

#### **Sea buckthorn juice process**

The juice was obtained with a domestic slow juicer (Greenis Slow Juicer, model F-9007) made from BPA free plastics. The juicing strainers are made from GE-Ultem plastic and the super slow 65 rpm speed reduces oxidation with minimal loss of nutrients. The juice was used for jelly production and the residues were dried at 40°C prior to the SFE.

#### **Carotenoids identification by high-performance liquid chromatography (HPLC) analysis**

The sea buckthorn oil samples obtained by supercritical carbon dioxide extraction were analyzed by HPLC in order to identify and quantify the carotenoid levels.

The system used was an HPLC from Thermo Finnigan Surveyor (Finnigan Surveyor LC, Thermo Scientific, SUA), controlled by Xcalibur software system. The carotenoids from each sample were analyzed at 450 nm on a Lichrosorb RP-18 (5 µm) Hibar RT 125-4 column. The elution solvents were 90% acetonitrile (A) and 100% ethyl acetate (B). The injection volume was 20 µL, and the flow rate was maintained at 0.500 mL/min.

The elution profile used was: 0–16 min, isocratic on 15% B; 16–54 min, linear gradient from 15% to 62% B, 54–56 min, isocratic on 62% B; 56–60 min, linear gradient from 62% to 15% B; 60–70 min, isocratic on 15% B (Pop et al., 2014). The quantification of carotenoids was done using a β-carotene calibration curve. The calibration curve for the β-carotene standard was prepared using six different concentrations (0.04–0.1 mg/ml) and dissolving it in ethyl acetate before the analysis. The linear regression factor of the calibration curve for this standard was 0.988.

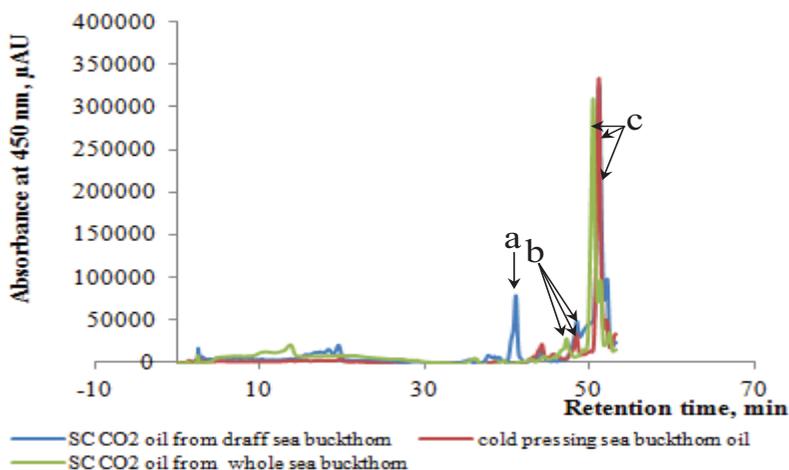


Figure 1. Representative HPLC chromatogram of carotenoids from MARA sea buckthorn oil sample, recorded at 450 nm

## RESULTS AND DISCUSSION

A quantitative HPLC analysis of sea buckthorn oils was used to identify the carotenoids. Figure 1 shows the HPLC chromatogram for all oil samples. Carotenoids identification was made based on their retention time and comparison with literature data.

$\beta$ -criptoxantina (peak a) and lycopene (peak b) were identified in all samples. The quantitative evaluation indicated a variation of carotenoid content in the range 0,028 – 0,33 mg/g, while  $\beta$ -criptoxantina was present only in the sea buckthorn draff oil (0,54 mg/g). Among the carotenoids,  $\beta$ -carotene (peak c) was identified in all oils samples with a small variation in the range (2,65 – 3,07) mg/g.

The carotenoid composition varied largely due to the varieties and extraction methods.

## CONCLUSIONS

After analyzing the results, it has been concluded that further research is needed in order to definitely state that SCFE is a reliable and cost efficient method of extracting Sea Buckthorn oil in Romania. This paper only assessed the feasibility of obtaining oil using such technology; however it has not covered aspects such as costs or market research. It is currently unknown if using such technologies is economically viable in the Romanian market,

as traditional technologies are both established and cost efficient due to their much lower implementation costs.

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