

SPECTROSCOPIC STUDY OF SEA BUCKTHORN EXTRACTS

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Abstract

The application of sea buckthorn oil is to incorporate the oil into foodstuffs such as milk, yoghurt, cheese, butter, juice and snacks which represents new opportunities for food manufacturers, food supplements and nutraceuticals providing nutritional supports. The FTIR spectroscopy is a powerful technique for assessing food production and studied materials provides fundamental information on the behavior of the spectral metabolites and bio product.

The extracts were studied from two varieties of sea buckthorn oil Pitesti I and II. Oil obtained from peel and seeds by the Soxhlet extraction with hexane solvent and CO₂ supercritical was analyzed by FTIR spectroscopy. The concentration of fatty acids in oil extracted from seeds and peels was similar in both extraction techniques.

Keywords: sea buckthorn oil, FTIR-ATR, spectral study, supercritical CO₂ extraction

1. INTRODUCTION

Sea buckthorn seed and pulp oils are considered the most valuable components of the berries comprising a unique fatty acid composition, fat-soluble vitamins, and plant sterols (Yang and Kallio, 2002). The seed oil, defined as being highly unsaturated, comprises two essential fatty acids, α -linolenic (C18:3n-3) (the parent substance of omega-3 fatty acid which helps to prevent chronic diseases such as heart disease and arthritis) and linoleic (C18:2n-6) acids (omega-6 fatty acid; unsaturated fatty acid considered essential to the human diet). The contributions of α -linolenic and linoleic acids are commonly at the 20 to 35 and 30 to 40% range, respectively (Yang and Kallio, 2002). Sea buckthorn oil can sometimes be very different, revealing different ratios of saturated/unsaturated (Cenkowski et al., 2006)

In this study, we used two varieties of sea buckthorn: Pitesti I and Pitesti II.

Pitesti I Buckthorn berries are very large variety (0.55 g), round and orange. Sea buckthorn berries are medium - variety Pitesti II (0.3-0.4 g), elongated oval shape and orange.

2. MATERIALS AND METHODS

The study used a JASCO 6300 FTIR spectrometer in the 400-4000 cm⁻¹ with TGS detector, Cosine apodization function, SpectraManager II software. Spectra were taken at room temperature was carried out by ATR (attenuated total reflection), using a diamond ATR crystal, with accumulation 100. The samples were measured in the diamond crystal, without the need for sample preparation in advance. Between measurements, the ATR crystal was cleaned with isopropyl alcohol. The instrument had a spectral resolution of 4 cm⁻¹, which were used in all spectra determinations.

The absorption spectra were recorded at the range of 460-320nm, in a Lambda 25 PerkinElmer spectrometer, with quartz cells, at room temperature

Soxhlet extraction. Soxhlet extractor was used for Sea buckthorn oil extraction. Oil was extracted from 50 g of Sea buckthorn peel and seeds using 100 ml hexane solvent under reflux. The reaction time was 8h. After the extraction had been accomplished, the extracts were left overnight so to allow separation of the oil phase from aqueous phase. Then, a vacuum distillation system at constant temperature was used to recover the oil from the extracting solvent using a rotary evaporator (Rotavapor, Heidolph) and a vacuum pump (Buchi V-700). The rotary evaporator was used for the efficient removal of n-hexane from samples by evaporation at 45°C.

Supercritical Fluid Extraction [SFE]. The Supercritical carbon dioxide extraction system and components were acquired from JASCO (Japan Spectroscopic Co.). Supercritical fluid extractor included the following: 250x 20 mm extraction vessel, column oven temperature (JASCO CO-2060), high-pressure pump (JASCO-PU-2080-CO₂), automated back pressure regulator (JASCO BP-2080). The experiments were carried out at 60°C and pressures of 35 MPa. Solvent mass flow rate was kept at 1.8 ml/min. At this flow rate it can be assumed that equilibrium concentration for the solvent and solute is achieved. Samples of 6 g of dried Sea buckthorn peel and seeds were placed in the extractor. The extracts were collected in one tube throughout the 60min.

3. RESULTS AND DISCUSSIONS

The yield of dry matter (seeds, peel) after separation of the juice and drying was 11.85 % for the variety Pitești I and 10 % for the variety Pitești II. The yield of the oil, extracted with n-hexane in a Soxhlet equipment was: 12.5 % for the variety Pitești I and 12 % for the variety Pitești II. Sea buckthorn oil contains various carotenoids such as lycopene or beta-carotene. Analyzing the content of carotenoids assigned bands intensities can be obtained quantitative assessment on sea buckthorn oils.

FTIR spectroscopy has grown in recent years, the characterization of agricultural products, foodstuffs. The FTIR spectroscopy becomes a powerful technique for assessing food production in processed food tasting so studied materials provides fundamental information on the spectral behavior of metabolites and bioproduct. ATR-FTIR spectroscopy is a rapid and non-destructive technique that has been used for investigated covalent bond vibration in sea buckthorn oil.

In FTIR spectra of sea buckthorn some of the most significant bands are shown in Figures 1-3 and in Table 1.

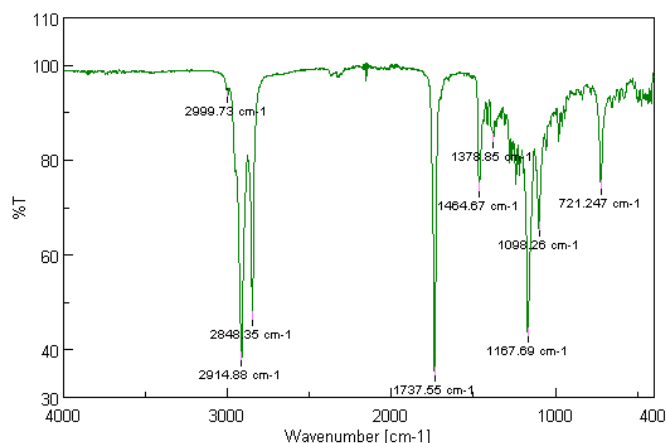


Figure 1. ATR-FTIR spectra of sea buckthorn variety oil-Pitești I

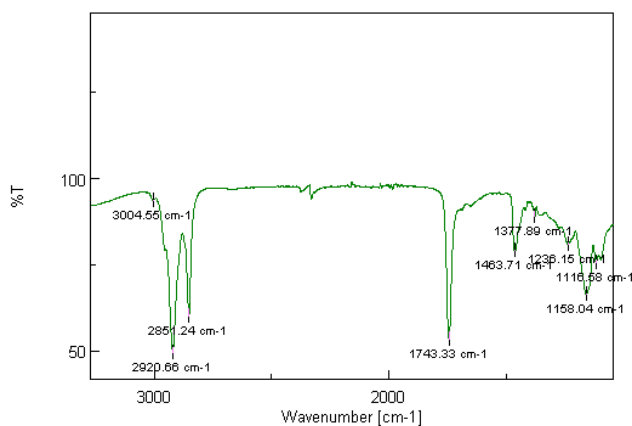


Figure 2. ATR-FTIR spectra of sea buckthorn variety oil-Pitesti II

In the IR spectra the most prominent vibrations observed in sea buckthorn in variety Pitesti I and II were the carbonyl group vibration, at $1737,55\text{ cm}^{-1}$ respectively $1743,33\text{ cm}^{-1}$, (strong) for C=O.

The band appearing at $3004,55\text{ cm}^{-1}$ (Pitesti I) and $2999,73\text{ cm}^{-1}$ (Pitesti II) in the spectrum is attributed to the CH stretching of =C-H bonding (Gullen et al., 1997, 1998; Trif et al., 2007). The two intensive bands at $2848,35\text{ cm}^{-1}$ and $2914,88\text{ cm}^{-1}$ (Pitesti I) are assigned to the aliphatic CH₂ asymmetric and symmetric stretching vibration. At $1464,76\text{ cm}^{-1}$, $1463,71\text{ cm}^{-1}$ respectively is observed a band which is assigned to =C-H scissors deformation vibration; the band near 1377 cm^{-1} is assigned to the bending vibration of CH₂ groups. The bands at $1098,26\text{ cm}^{-1}$ and $1167,69\text{ cm}^{-1}$ (Pitesti I), $1158,04\text{ cm}^{-1}$ (Pitesti II) are assigned to the vibration of C-O ester groups and CH₂ group. FTIR spectra of these sea buckthorn oils present very similar spectral shapes due to close chemical structure of fatty constituents.

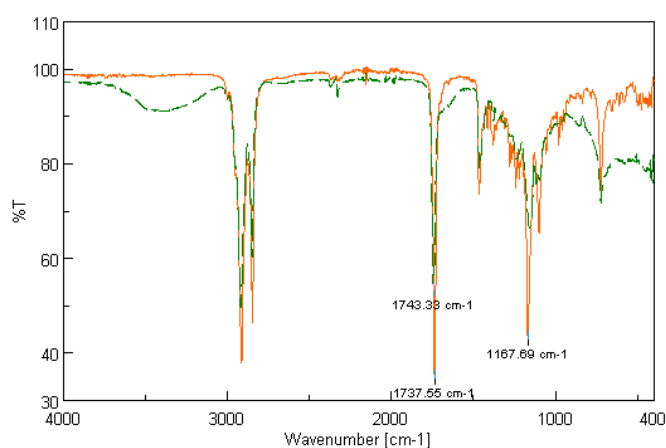


Figure 3. ATR-FTIR spectra of sea buckthorn oils Pitesti I and II (red variety I Pitesti, green variety II)

Compared to the spectral characteristics of vegetable oil, there are more bands in the spectrum at 1220.72, 1054.87, 981.59, 958.45, 926.63 cm^{-1} (variety Pitesti II) assigned carotenoid content (Figure 4). Spectrum sunflower oil was introduced to the comparison of spectral AG. Analyzing the content of carotenoids assigned bands intensities can be obtained qualitative assessment in buckthorn oils (Irudayaraj and Tewari, 2003)

Table 1. Wavenubers (cm^{-1}) of selected bands in FTIR spectra of sea buckthorn oils

Oil/variety	$\nu_{\text{C-H}}$ (cm^{-1})	$\nu_{\text{CH}_2 \text{ asim}}$ (cm^{-1})	$\nu_{\text{CH}_2 \text{ sim}}$ (cm^{-1})	$\nu_{\text{C=O}}$ (cm^{-1})	$\delta_{\text{-CH}}$ (cm^{-1})	δ_{CH_2} (cm^{-1})	$\nu_{\text{C-O asim}}$ (cm^{-1})	$\nu_{\text{C-O asim}}$ (cm^{-1})
Pitesti I	2999,73	2914,88	2848,35	1737,55	1464,76	1378,65	1167,69	1098
Pitesti II	3004,55	2920,66	2851,24	1743,33	1463,71	1377,89	1158,04	1096,58

The bands in the spectral region 981-915 cm^{-1} (trans CH = CH) is attributed to β -carotene. By analyzing the intensities of the bands attributed to the content of carotenoids and marker bands having the bands of 1054 cm^{-1} for in plane vibrations of $-\text{CH}-$ and 959 cm^{-1} for *trans* conjugated alkene $-\text{CH}=\text{CH}-$ out-of-plane deformation mode (M.H. Moh and col, 1999) is clear that sea buckthorn oil variety Pitești-I has the highest content of carotenoids (Figure 4 and Figure 5 from UV spectra)

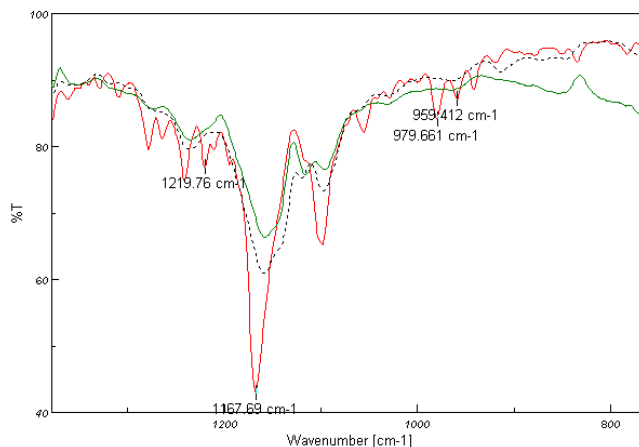


Figure 4. ATR-FTIR spectra of sea buckthorn oils (red-Pitesti I, green-Pitesti II) and compared to sunflower oil (dashed line)

ζ -Carotene (light yellow), which is acyclic with eleven conjugated double bonds, has three well-defined peaks, absorbs at the λ_{max} 378, 400, 425 nm (figure 5).

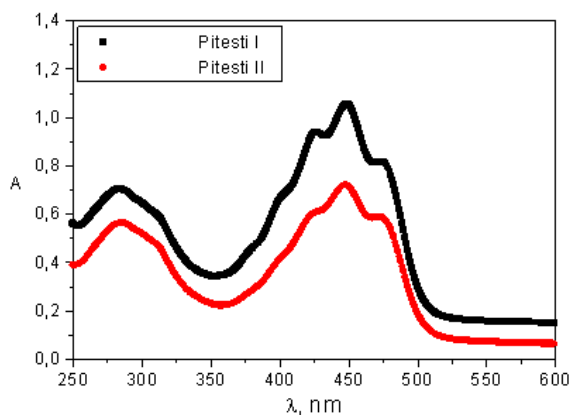


Figure 5. UV spectra for of sea buckthorn oils Pitesti I and II

In figure 6 is presented ATR-FTIR spectra of Pitesti I sea buckthorn oils by supercritical CO₂ extraction. It was observed that there is no difference in the functional groups observed for the two samples.

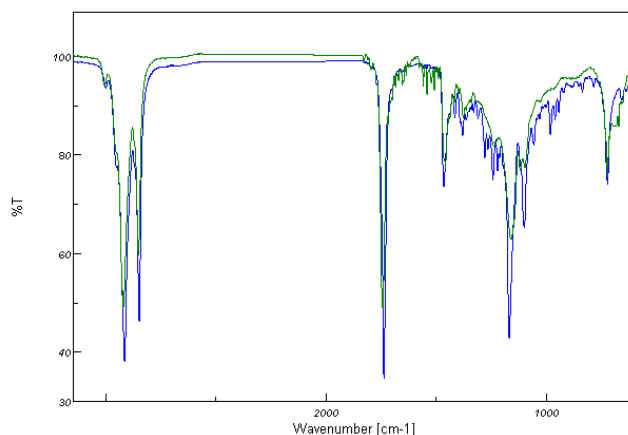


Figure 6. ATR-FTIR spectra of Pitesti I sea buckthorn oils (green- CO₂ supercritical extraction, blue- Soxhlet extraction)

4. CONCLUSIONS

ATR- FTIR spectroscopy is a viable method, rapid and non-destructive analysis to assess the quality and authenticity of vegetable oils. Sea buckthorn oil (Pitesti varieties I and II) include all the spectral characteristics of a vegetable oil. In addition several new bands appear in the spectrum at 1220.72, 1054.87 cm⁻¹ and 981-915 cm⁻¹ are attributed to the carotenoid content of sea buckthorn oil.

The FTIR study revealed that Sea buckthorn oil extracted with Supercritical CO₂ and n-hexane led to the oil-like composition.

5. ACKNOWLEDGEMENTS

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