Integrated recovery of sea buckthorn by supercritical CO_2

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1. Introduction

Sea buckthorn currently attracts the attention of many researchers, mainly for its nutritional and medicinal values. The vitamin C concentration of its fruits is 30 times higher than that of oranges, 25 times higher than that of strawberries. In Europe and Asia, preparations of sea buckthorn oil are interesting because of their richness in fatty acids, vitamins, and sterols. The pulp oils, rich in Omega 7, are highly used in anti-aging and skin regeneration cosmetic creams. However, the seed oils, rich in Omega 3 and Omega 6, are mainly intended for food and the development of natural health products.

CO_2 is the most widely used supercritical fluid, as it has significant advantages. It is **non-toxic, non-pollutant, non-flammable**, widely available at very high degrees of purity (up to 99.99%), and has moderate costs (around $1.60CDN/kg)

The supercritical CO_2 extraction of **functional and bioactive compounds** from sea buckthorn berries is therefore a sustainable recovery method, which benefits from the exceptional development of **green chemistry**.

2. Materials and Methods

This work was carried out on the seeds and pulp of the Leikora cultivar. The effects of the extraction parameters on the oil yield were studied by a factorial experimental design[2]. This experimental design was used both for the seeds and for the pulps of sea buckthorn.

A factorial plan is made, based on the following parameters: pressure (200-500 bars), temperature (40-60°C), CO_2 flow rate (5-20 g/min), extraction time (1-4h), and percentage ethanol (0-5%). The defined optimal operating conditions are 300 bars, 4h, 40°C, and CO_2 at 10g/min. Scale-up was carried out on the pre-commercial fifty-liter unit. The work on this unit meets a dual objective: prepare batches so that our industrial partners can test the market, ii) generate realistic data to develop a technical-economic evaluation and determine a production cost.

3. Results and discussion

The statistical results show that the flow rate does not influence the extraction of seeds or the yield of pulp extraction, while, the temperature has a slight influence on these two factors. Even the flow rate has no influence, a minimum flow rate is still needed to allow effective material transfer. Therefore, results of 5g/min CO_2 flow rate were not good as results of higher flow rates. In the case of a low flow rate, resistance and accumulation in the mass become dominant compared to diffusion between the particles leading to a reduction in extraction yield [1]. The recommended parameters for scaling are 40°C, 10g/min CO_2, 350 bar, and 4 hours of extraction. Extraction yields depend on the nature of the cultivar and the freshness of the raw material. The oils obtained are analyzed by GCMS to determine their fatty acid composition.

Table 1. Comparison of conventional and supercritical extraction yields

Raw material (Leikora)	Conventional extraction parameters						Results
	T (°C)	Solvent	Ratio	Vol. (ml)	Time (h)	Raw material (g)	Yield of oil (%)
Pulps	reflux	hexane	1:10	200	4	20.0	11.6
Seeds	reflux	hexane	1:10	200	3	20.5	4.4
Raw material (Leikora)	**Supercritical extraction parameters**						**Results**
	T (°C)	**CO_2 flow rate (g/min)**	**Pressure (bar)**		**Time (h)**	**Raw material (g)**	**Yield of oil (%m/m)**
Pulps	40	10	350		4	25.0	14.4
Seeds	40	10	350		4	25.0	6.0

The extraction on the pre-commercial unit (fifty-liter volume) was carried out under the scaling conditions of 40°C, 50kg/h CO_2, 300 bars, and 7 hours of extraction for the seeds and 40°C, 80kg/h CO_2, 300 bar and 7 hours for the pulps. The obtained results are very promising, around 12% pulp oils and 8 to 10% seed oil.

4. Conclusion

In addition to juice from sea buckthorn fruits, oils free of the organic solvent can be produced from its pulps, and seeds (residues) thanks to supercritical CO_2 extraction. Oils extracted from sea buckthorn pulp and seeds are rich in omega 3, 6, and 7 oils. The extracted oil from pulp, rich in Omega 7 at 28%, is highly used in the preparation of anti-aging and skin regeneration cosmetic creams. Its seed oils, rich in Omega 3 at 31% and Omega 6 at 33 %, are mainly intended for human food and the development of natural health products. Using the pre-commercial extraction unit provides enough quantity of oil so that our partners could test the market. It also generates realistic data to develop a technical and economic evaluation and determine a production cost.

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