Simultaneous extraction and fractionation of oil and fat by means of supercritical carbon dioxide

Annelore Wens, Els D'Hondt, Hannes Sels, Jeroen Geuens
Karel de Grote University of Applied Sciences and Arts, Antwerp, Belgium

Introduction

Oils and fats are important industrial commodities, used in food, nutraceuticals, cosmetics, and biofuels. Their quality, composition (e.g. fatty acid and glyceride profile), and purity profoundly affect possible applications. At the same time, environmental and safety concerns are pushing industries to reduce the use of volatile organic solvents (e.g. hexane) and high-temperature processing. Hence, methods that can simultaneously extract and fractionate oils/fats in a clean, selective, and efficient way are highly relevant [1].

Supercritical carbon dioxide ($scCO_2$) extraction and fractionation offers a promising alternative technology. The supercritical state of CO_2 combines the high diffusivity and low viscosity of gases with the solvating power of liquids at rather mild conditions (critical temperature 31 °C and critical pressure 74 bar). By adjusting pressure and temperature, and by addition of a cosolvent (e.g. ethanol), solvent density and solvation strength can be tuned, thereby making it possible to fractionate different lipid fractions such as free fatty acids, mono-, di– and triglycerides [2].

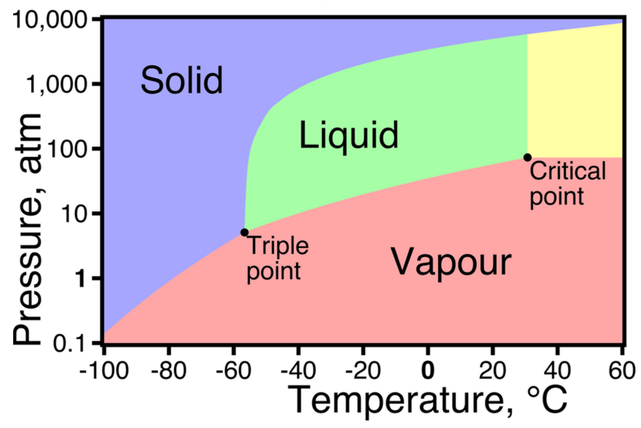


Fig. 1 - Phase diagram CO_2 [3]

Objectives

- Determine optimal conditions for $scCO_2$ fractionation
- Study the influence of the type of fat/oil on $scCO_2$ fractionation

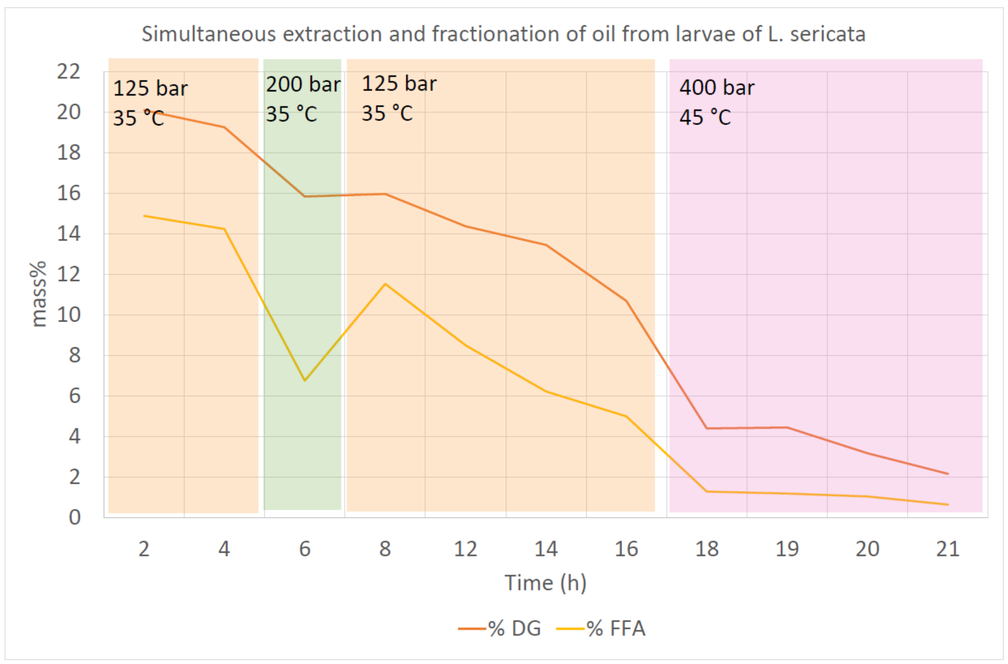
Results & discussion

Experiments were performed on different oils and oil containing resources, such as oilseeds and larvae. By way of example, a selection of results is mentioned here.

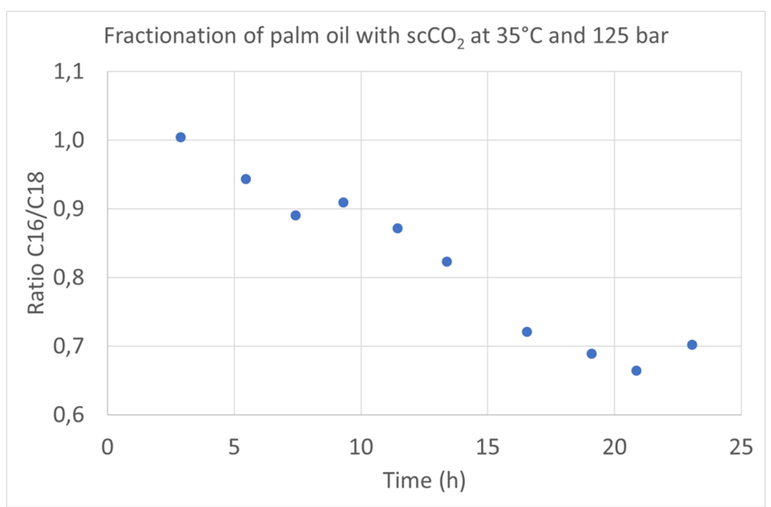
✔ From graph 1, it can be seen that more polar compounds such as free fatty acids (FFA) and diglycerides (DG) can selectively be extracted at lower temperature and pressure.

✔ Graph 2 shows that, even when working at constant temperature and pressure, fractionation between C16 and C18 is feasible. A similar result was obtained when performing extractions on the larvae of Black Soldier fly where a selective extraction of C12 was observed vs. C16 and C18.

✘ However, when experiments were performed on linseed oil containing only 5% C16 vs. 95% C18 and less than 1% of FFA, along with 5% DG, selective extraction of FFA, DG or C16 was not observed. Even in further experiments adding ethanol as cosolvent and working at different temperatures and pressures fractionation was poor to non existing.



Graph 1 - $scCO_2$ extraction and fractionation of oil from L.sericata larvae



Graph 2 - $scCO_2$ fractionation of palm oil (refined and bleached)

Conclusions

Fractionation can only be obtained for oils/fats bearing certain glyceride and/or fatty acid profiles:

- it is not possible to selectively separate fatty acids or glycerides present at low concentrations;
- separation on the level of fatty acid composition is possible but depends on the fatty acid composition of the starting material.

Kinetics need to be improved since complete fractionation is now typically reached after more than 16 h. However, this is mainly due to the nature of the extraction system used in the experiments.

Future work

Experiments will be conducted starting from model systems:

- mixtures of pure FFA or their corresponding methyl esters;
- pure free fatty acids added to a standard oil/triglyceride matrix.

In this way, we hope to get a better understanding on the influence of the glyceride and fatty acid composition on the fractionation.

Fig. 2 - SFT-150 $scCO_2$ extraction system

Materials & methods

All oils and fats used in the experiments were either purchased from local providers or kindly donated by our industrial partners. Extractions were performed in an SFT-150 system (Supercritical Technologies) equipped with a 100 mL extraction vessel. During the dynamic phase of the extraction a constant flow of 20 mL CO_2/min was used. If applicable, ethanol was added as cosolvent by means of a peristaltic pump. Fatty acid, mono-, di- and triglyceride contents were determined by means of GPC analysis on an LC-20A HPLC system (Shimadzu). Fatty acid profiles were determined on a TRACE1300 GC-MS system (Thermo Scientific) after conversion of fatty acids and glycerides to their corresponding methyl esters. For more detail on the materials and methods, you are kindly invited to get in touch with the authors.

References

[1] Dhara et al. (2022) Supercritical carbon dioxide extraction of vegetable oils: retrospective and prospects, *Eur. J. Lipid Sci. Technol.*, *124* (8), 2200006
[2] Rincon et al. (2010) Fractionation of used frying oil by supercritical CO_2 and cosolvents, *Ind. Eng. Chem. Res.*, *49* (5), 2410
[3] https://commons.wikimedia.org/wiki/File:Phase_changes_of_CO2.png

Contact

jeroen.geuens@kdg.be
https://www.kdg.be/en/research/sustainable-industries

KdG Research
Karel de Grote Hogeschool