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*12TH EURO FED LIPIDS CONGRESS: OILS, FATS AND LIPIDS: FROM LIPIDOMICS TO INDUSTRIAL INNOVATION*

Dossier

# Recovery of oils from press cakes by $CO_2$-based technology

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**Abstract –** In a context of a strong demand for vegetable oils, the development of new “green" processes is essential to provide methods that avoid the use of organic solvents, work at relatively low temperatures and preserve the quality of the final products. The supercritical fluid extraction (SFE) process meets these objectives. In this study, five meals (hemp, flax, sesame, poppy and walnut) were subjected to SFE at 45 °C and 250 bar in the presence of ethanol as co-solvent. Using this method, between 77 and 98% of the lipids present in the meal could be extracted. Except for poppy, 50% of the lipids present in the cake were recovered as a separate oily liquid phase. SFE was not selective toward certain types of triglycerides: the oils obtained exhibited the same fatty acid profile than the total lipid extracts from the cakes. In contrast, the composition in minor lipid components was dependent on the type of meal investigated. For example, hemp lipid extract was particularly rich in chlorophyll.

**Keywords:** Supercritical fluid extraction / press cake / oil recovery / oil quality

**Résumé – Technologie à base de $CO_2$ pour l'extraction d'huiles de tourteaux de pressage.** Dans un contexte de forte demande en huiles végétales, le développement de nouveaux procédés « verts » est indispensable pour proposer des procédés évitant l'utilisation de solvants organiques, travaillant à relativement basse températures et préservant les qualités nutritionnelles et organoleptiques des produits finaux. Le procédé d'extraction par fluide supercritique (EFS) répond à ces objectifs. Dans cette étude, cinq tourteaux végétaux (chanvre, lin, sésame, pavot et noix) ont été soumis au procédé d'EFS à 45 °C et 250 bar en présence d'éthanol comme co-solvant. Ce procédé a permis d'extraire entre 77 et 98% des lipides présents dans les tourteaux. À l'exception du pavot, 50% des lipides présents dans les tourteaux se retrouvent sous la forme d'une phase liquide huileuse indépendante. L'EFS n'est pas sélective vis-à-vis de triglycérides particuliers. En effet, l'huile extraite a le même profil en acides gras que les lipides totaux du tourteau. Néanmoins, selon la nature du tourteau considéré, la composition en composés lipidiques mineurs est très variable. Par exemple, la quantité de chlorophylle extraite est très importante dans le cas du tourteau de chanvre.

**Mots clés :** Extraction par fluide supercritique / tourteau de pressage / extraction d'huile / qualité de l'huile

## 1 Introduction

Oil demand is in constant increase for years due to nutritional and industrial issues. For nutritional aspects, recommendations concerning the omega 3/omega 6 balance are responsible for new developments in specialty oils and for a global increase of the oil volume demand. In the same time, the use of oil for biodiesel production is also increasing. By still containing 10 to 30% of oil, press cakes are valuable sources of oils that, by regards of environmental and human health concerns, could be recovered by eco-efficient processes. So far, large scale oil facilities process is based on a pressing step followed by a solvent extraction using hexane to exhaust the cake (Fine, 2013). As an alternative and eco-efficient approach, supercritical fluid extraction (SFE) can be envisaged.

The recovery of oil from oilseeds by SFE is a solid-fluid extraction process that uses mostly carbon dioxide as extracting fluid. Indeed, oil solubility in supercritical carbon dioxide ($scCO_2$) is relatively high (0.1 to 200 g oil/kg $CO_2$) depending on the fluid density (*i.e.* pressure and temperature) (del Valle *et al.*, 2012). $ScCO_2$ is an apolar fluid so ethanol is often used as a cosolvent to modify its polarity and to favor co-extraction of more polar compounds. SFE of oilseeds has been extensively studied on various matrices over the past decades (Eggers, 1996). However, SFE applied on press cakes is more confidential and the consequences of pressing on SFE performances still have to be evaluated.

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**Table 1.** Press cake compositions (different letters are for statistically different lipid content (Tukey HSD test at 95% confidence interval)).

| | Hemp | Linseed | Poppy | Sesame | Walnut |
|---|---|---|---|---|---|
| Water content* (% db) | 7.3 (⩽12) | 8 ± 1 (⩽12) | 9.2 (⩽12) | 7.6 (⩽10) | 5.4 (⩽10) |
| Lipid content* (%db) | 11 ± 2$^{c}$ (⩽12) | 11 ± 1$^{a,c}$ (⩽12) | 12 ± 1$^{a,c}$ (⩽12) | 23 ± 2$^{b}$ (⩽25) | 8 ± 1$^{a}$ (⩽45) |
| Ash (%)** | ⩽15 | ⩽10 | ⩽15 | ⩽10 | ⩽10 |
| Proteins (%wb)** | 25–40 | 15–45 | 30–45 | 30–50 | 20–40 |
| Cellulose (%wb)** | 20–35 | ⩽10 | 5–15 | ⩽10 | ⩽10 |

* Values in parentheses are from supplier's datasheet. ** Values are from supplier's datasheet.

In this work, SFE was investigated as a technique to recover residual edible oils from various press cakes presenting different fatty acid profiles. Hempseed oil is a linoleic oil, typically composed of 56% of linoleic acid (18:2 n-6), 22% of $\alpha$-linolenic acid (18:3 n-3), and 9% of oleic acid (18:1) (Callaway, 2004). Walnut oil can be classified in the same category with 56–65% of 18:2, 14–21% of 18:1, 9–15% of 18:3 and 6–8% of palmitic acid (16:0) (Morin, 1996a). Poppy seed oil is also a linoleic oil with 69–77% of 18:2, 13–18% of 18:1, 9–11% of 16:0 and less than 3.5% of other fatty acids (Morin, 1996b). Linseed oil (also call flaxseed oil) is a linolenic oil and is typically composed of 59% of 18:3, 15% of 18:2, 18% of 18:1 other fatty acids accounting for less than 10% of the total fatty acids (Przybylski, 2005). Sesame oil is an oleic-linoleic oil containing 39–47% of 18:2, 37–42% of 18:1, 8–11% of 16:0 and 4–6% of stearic acid (18:0) (Morin, 1996b). These seeds present different fatty acid profiles but have in common a high degree of unsaturation.

Supercritical extraction has already been investigated for these five seeds and nut, and the studies generally focused on the effect of process parameters (namely pressure, temperature and flow rate) on extraction yield (on sesame (Corso *et al.*, 2010; Döker *et al.*, 2010); on hemp (Da Porto *et al.*, 2012a, 2012b); on poppy (Bozan and Temelli, 2003); on walnut (Oliveira *et al.*, 2002; Salgin and Salgin, 2006)). For instance, on linseed, SFE optimization (Bozan and Temelli, 2002; Ivanov, 2012; Jiao, 2008; Özkal, 2009; Rombaut, 2013) showed that an increase of temperature and pressure increased the oil yield. Depending on extraction conditions, up to 82% of oil could be extracted with neat $CO_2$. In Ivanov's work (2012), ethanol was used as cosolvent, but it was directly added to the load prior to $CO_2$ extraction. The higher the quantity of ethanol, the higher was the oil yield (up to 99%). Whilst SFE is mainly investigated on raw seeds, SFE on press cake is more rarely reported. Dealing with walnut press cake, Martinez *et al.* (2008) recovered the oil with yield higher than 90% for SFE carried out at 40 MPa and temperatures higher than 50 °C.

The oil quality is generally assessed by calorimetry, peroxide value (for oxidative status characterization) or absorbance at specific wavelengths (for minor compound determination). Whatever the seeds, the oil recovered by supercritical technology was generally of a lower quality than cold pressed oils. For example, Martinez *et al.* (2008) first pressed walnuts and then performed SFE on the press cake. The two oils produced were analyzed by same methods. SFE oils presented higher acid value (0.4% oleic acid *vs.* 0.1% for cold press oils) and lower oxidative stability (0.3 to 0.8 h *vs.* 3 h for cold press oils; measured with the Rancimat method). So SFE oils were more degraded and less stable than cold pressing oils. However, SFE oils contained higher amounts of carotenoids (9 µg/g of oil *vs.* 2 µg/g for cold press oil).

The aim of this study was to evaluate SFE performance at recovering residual oil when applied to five press cakes that exhibited different lipid contents and matrix characteristics. The process efficiency was evaluated in terms of oil yield and oil characteristics.

## 2 Materials and methods

### 2.1 Raw material

Press cakes were supplied by Bioplanète (Bram, France). Cakes came from organic seeds of poppy (*Papaver somniferum* L. var. nigrum), linseed (*Linum usitatissimum* L.), hemp (*Cannabis sativa* L.) and sesame (*Sesamum indicum*). Walnut (*Juglans regia*) cake was also used. The characteristics of cakes are given in Table 1.

Total lipid content was determined by chloroform/methanol extraction according to Folch method (Folch, 1957). Water content was assessed using a thermobalance (Sartorius) at 105 °C until mass variation became lower than 1 mg. Other characteristics were given by the supplier.

### 2.2 Supercritical fluid extraction

The scheme of the set-up is provided in Figure 1. The equipment is mainly composed of a 0.49 L cylindrical vessel fed by two high pressure pumps (Gilson). Liquid $CO_2$ (Air Liquide, France) was first cooled to –0.3 °C before the pump then heated to 45 °C by an electrical heater before being introduced in the vessel. The vessel that contained the matrix was thermally controlled by an electrical mantle.

Supercritical fluid extraction was carried out at 45 °C and 25 MPa using $CO_2$ (~15 g min$^{-1}$) and ethanol (~7 %wt) as cosolvent. The vessel was first loaded with 35 g of milled press cake then heated to 45 °C. Pure $CO_2$ was introduced until a pressure of 25 MPa and the vessel was isolated at this pressure for 35 min. The two pumps were then activated and the $CO_2$ + 7% wt ethanol mixture flew through the matrix bed. Extracts were collected at atmospheric pressure after the fluid depressurization. The first two fractions, called F0 and F1, were

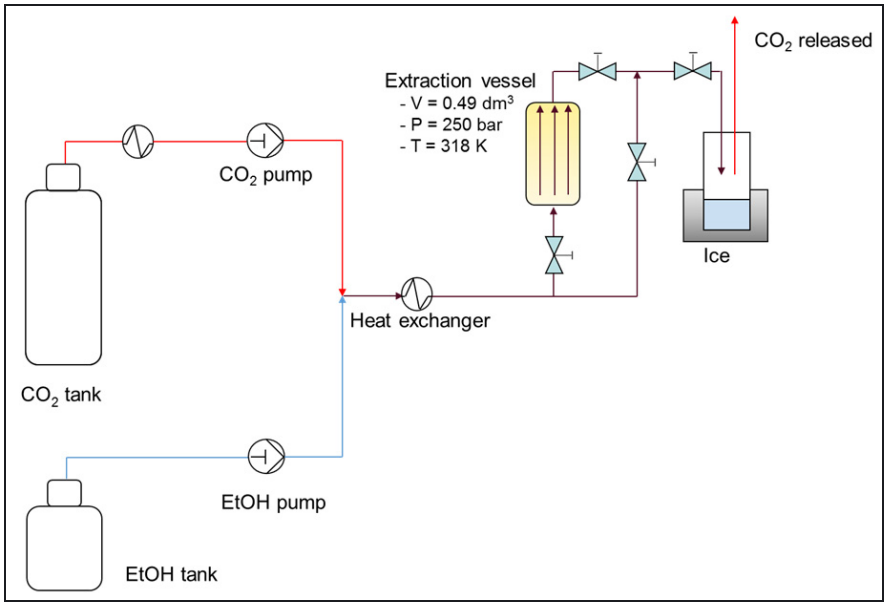


**Fig. 1.** Experimental SFE setup.

each collected after 60 min of flow. After these 120 min, extraction was continued with 27% wt ethanol to exhaust the cake in more polar lipids. At the end, pure $CO_2$ flew over the matrix bed for one hour to remove ethanol. In this work we focused on the oily phase, so only the F0 fraction was characterized.

### 2.3 Extract characterization

Collected extracts were centrifuged for phase separation. Each phase was then weighted and its volume measured. Ethanol was evaporated under a flux of nitrogen for mass balance assessment. The lipid content of the extracted matrix (hereafter called residue) was determined by the Folch method as well (Folch, 1957).

Extraction yield was calculated as the global extracted mass divided by press cake amount introduced in the SFE vessel. Lipid extraction yield was expressed as the ratio of the mass of total lipid extracted (calculated by difference between press cake lipid content and residue lipid content) to the lipid mass in the press cake. Oil yield was calculated as the ratio of free oil recovered from the oily phase to the total lipid mass of press cake.

Fatty acid profiles were determined by gas chromatography (GC) analysis after transesterification. Total lipid fractions were transmethylated in presence of boron trifluoride-methanol complex (Morisson, 1964). Fatty acid methyl esters were separated on a BPX 70 capillary column (60 m long, 0.25 µm film, 0.25 mm i.d., SGE (Melbourne, Australia)), using hydrogen as carrier gas and a split ratio between 1:50 and 1:60. The GC system consisted in a gas chromatograph (GC 2010 plus, Shimadzu, Kyoto, Japan) equipped with a flame ionization detector maintained at 280 °C. The injector was at 250 °C. The column temperature was programmed from 150 °C to 200 °C (1.3 °C/min) held for 30 min, then from 200 °C to 250 °C (20 °C/min) held for 30 min. Data

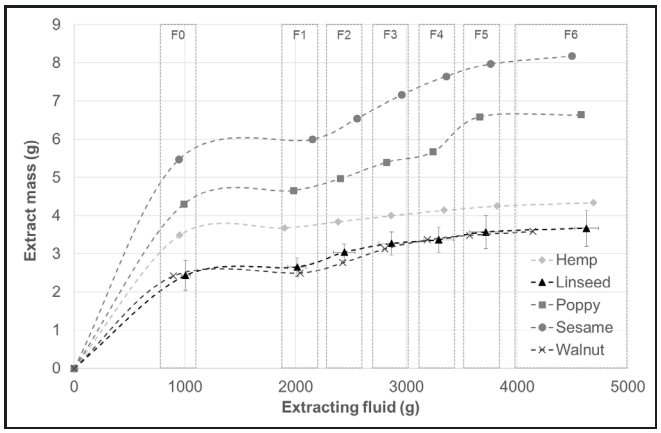


**Fig. 2.** Extraction kinetic.

was collected and integrated by a GC solution v2.4 integration system (Shimadzu). Fatty acids from Sigma were used as standards for calibration.

Absorbance and spectra (300–900 nm) of oily phases were recorded on Hitachi U2800 spectrometer after dilution with hexane and using quartz cells. The carotenoid content was determined as β-carotene equivalent from absorbance at 453 nm using β-carotene diluted in groundnut oil for calibration (0–5 mg/kg) (Lichtenthaler, 2001).

Oxidative status of the oily phases was assessed by peroxide value determination (ISO3960 adapted method).

## 3 Results and discussion

### 3.1 Extraction kinetic

The overall extraction kinetic of the five cakes is given in Figure 2. The first part of the curve, up to 2 kg of fluid, corresponded to F0 and F1 fractions recovered using $CO_2$ + 7%

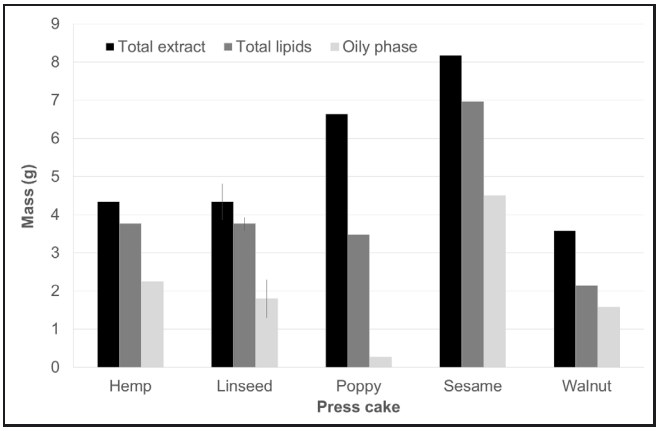


**Fig. 3.** Extracted masses.

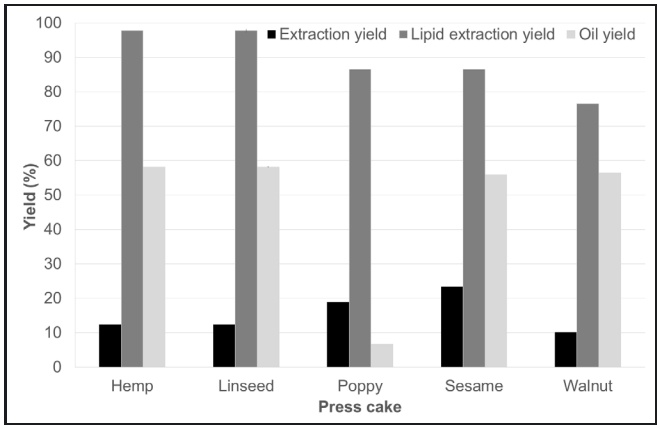


**Fig. 4.** Extraction yields.

ethanol. Most of the extracted amount came from F0 since F1 contributed only for few milligrams. This F0 fraction represented 65 to 67% of the total extract for all press cakes except hemp for which it represented 80%. The extraction kinetics exhibited then a second increase after 2 kg of fluid that corresponded to the increase of ethanol to 27 %wt. This higher content of ethanol allowed for extracting more polar lipids like for instance phospholipids and ensured thus that the extraction is effective for all classes of lipids (Catchpole *et al.*, 2009).

### 3.2 Extraction efficiency

The total extracted mass was highly dependent on the raw material nature and varied from 3.6 g for walnut to 8.2 g for sesame (Fig. 3). The mass could be correlated to the initial lipid content of the press cake (Tab. 1). As observed when comparing sesame and walnut, the higher the lipid content, the higher was the extracted mass. Hemp, linseed and walnut, that are similar in terms of lipid contents (8 to 11%), resulted in intermediate and similar extracted amounts. Only poppy showed a different behavior with an extracted mass fifty percent higher than hemp despite similar lipid content. For the five press cakes, the extracted material was mainly composed of lipids, in a content that varied from 50 to 97%. Hemp, linseed and sesame extracts contained more than 85% of lipids whereas extracts were composed of 52 and 60% of lipids for poppy and walnut, respectively.

Walnut exhibited a different behavior that could be due to a different cell structure owing to the matrix nature (walnut is not an oleaginous seed but a nut) (Morin, 1996b). Poppy's behavior was also quite surprising but its particular behavior could not be related to a particular chemical composition of the oil or the structure of the seed.

Excepted for poppy, all F0 fractions were biphasic with an oily phase at bottom and an ethanolic supernatant on top of it. The biphasic nature of F0 could be explained by the low solubility of vegetable oil in ethanol at ambient temperature (Johnson and Lusas, 1983). Supercritical $CO_2$ is a very good solvent of triglycerides (Sahena *et al.*, 2009) so neutral lipids were indeed extracted. But, after decompression, when the fluid mixture splits in gaseous $CO_2$ and liquid ethanol, the collected volume of ethanol was not high enough to dissolve the oil so a biphasic extract was recovered. With again exception of poppy, more than half of the extracted lipids were recovered as a separate oily phase. The mass of this oil was highly dependent of the raw material and was well correlated to the press cake initial lipid content. For poppy, only 7% of extracted lipids were in a separate phase that was, moreover, solid at ambient temperature.

The efficiency of an extraction process is classically evaluated by yields. Here, three yields have been defined corresponding to the three levels of extract characteristics. The global extraction yield is the total extracted mass divided by the press cake amount; the lipid extraction yield is the mass of extracted lipids divided by the mass of lipids in the loaded press cake whereas the oil yield only considers the oily phase divided by the initial amount of lipids in the loaded press cake.

The global extraction yields were comprised between 10 and 23% (Fig. 4), the highest values being for sesame and poppy (23 and 19%, respectively) and the lowest for walnut (10%). The lipid extraction yield indicates the performance of the technique to remove the initial amounts of lipids in cakes. Lipid extraction yields were relatively high varying from 98 to 76% for hemp and walnut, respectively. Linseed, poppy and sesame exhibited lipid yield in the range of 87–92%. These yields were higher than the majority of those reported for lipid extraction by neat $CO_2$ from same seeds (Bozan and Temelli, 2002, 2003; Corso *et al.*, 2010; Da Porto *et al.*, 2012a, 2012b). However, on walnut press cake, Martinez *et al.* (2008) obtained an oil yield of 92% that is higher than the 76% obtained herein.

Regarding specifically the oil phase, yields were in the same range for hemp, sesame and walnut press cakes (~56%) and in lesser extend for linseed (45%), but it was far smaller for poppy (7%). As already mentioned, the F0 poppy fraction was really different from the other F0 since lipids did not form a separate oily phase.

### 3.3 Composition of F0 fraction

F0 fractions were characterized in terms of fatty acid profile and peroxide values for the oily phase, whereas the carotenoids were quantified on both the oily and the ethanol phases. Results of characterization are overviewed in Table 2.

**Table 2.** Fatty acid profiles, and minor compounds composition.

| | | Hemp | | Linseed | | Poppy | | Sesame | | Walnut | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Press cake | SFE F0 oily phase | Press cake | SFE F0 oily phase | Press cake | SFE F0 oily phase | Press cake | SFE F0 oily phase | Press cake | SFE F0 oily phase |
| Fatty acids (%) | 16:0 | 8.2 | 7.1 | 7.1 | 6.4 | 10.2 | 9.7 | 8.3 | 8.4 | 8.2 | 6.8 |
| | 18:0 | 3.4 | 2.8 | 3.6 | 3.4 | 2.2 | 2.2 | 5.8 | 6.3 | 3.1 | 2.5 |
| | 18:1 | 15.1 | 15.8 | 18.7 | 18.7 | 13.9 | 14.3 | 38.6 | 41.4 | 15.7 | 16.1 |
| | 18:2 | 55.4 | 56.9 | 22.1 | 20.9 | 72.6 | 72.7 | 44.5 | 41.8 | 61.0 | 62.5 |
| | 18:3 | 14.9 | 14.2 | 46.8 | 49.2 | 0.6 | 0.6 | 0.4 | 0 | 10.3 | 11.6 |
| | Others | 3.0 | 3.3 | 1.6 | 1.4 | 0.4 | 0.5 | 2.5 | 2.1 | 1.7 | 0.6 |
| | | Oily phase | | Oily phase | | Oily phase | | Oily phase | | Oily phase | |
| Carotenoids (mg eq. $\beta$-caroten/ kg oil) | | 231 | | 22 | | 0 | | 0 | | 9 | |
| Chlorophyll | | Presence | | Absence | | Absence | | Presence | | Presence | |
| Peroxide value (meq. $O_2$/kg oil) | | >15 | | <15 | | n.d. | | >15 | | <15 | |
| | | Ethanolic phase | | Ethanolic phase | | Ethanolic phase | | Ethanolic phase | | Ethanolic phase | |
| Carotenoids (mg eq. $\beta$-caroten/kg dry extract) | | 1810 | | 45 | | 3 | | 1 | | 12 | |
| Chlorophyll | | Presence | | Absence | | Absence | | Absence | | Absence | |

Regarding the fatty acid profile, the oils obtained by SFE have almost the same fatty acid composition than the raw materials. This absence of selectivity of SFE towards specific triglycerides as measured by the fatty acid composition was also observed in literature on various raw materials (Da Porto *et al.*, 2012a; Martinez *et al.*, 2008). Peroxide value determination was performed on all F0 oily fractions except for poppy whose oily fraction was solid and of too low volume to allow measurements. The oil peroxide values were highly affected by press cake species. Depending on the raw material, peroxide value was below or above the limit fixed by the codex alimentarius (Codex Alimentarius, 1981). On that basis, only oils produced from linseed and walnut press cakes were of alimentary grade.

Carotenoids were quantified by spectrophotometry (Fig. 5). In oily phases, carotenoids ranged between 0 and 231 mg/kg oil, and the content was highly dependent on the raw material. The hemp extract exhibited content more than tenth time higher than the other press cake extracts. This high value could be due to carotenoids but it could also came from the presence of chlorophylls that interfered in the quantitation. Presence of chlorophylls was evidenced both by the strong green color of F0 and by the absorbance spectrum (Fig. 5). Although chlorophylls absorb at higher wavelengths than carotenoids (600–650 nm instead of 400–500 nm), their residual absorbance at 453 nm will contribute to the carotenoid content as well. Hemp oil is not specially known for high chlorophyll content. According to Matthäus and Brühl, 2008, chlorophylls might sign immature pressed seeds. Indeed, the chlorophyll content of seeds decreases with maturity and fully mature seed are usually devoid of chlorophyll. Chlorophylls

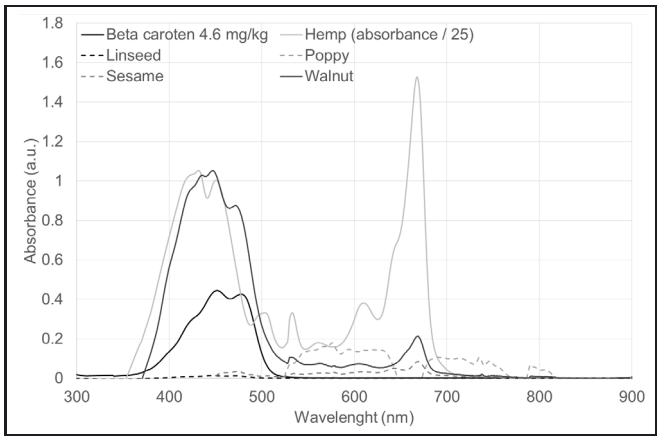


**Fig. 5.** Absorption spectra (for hemp, spectrum is divided by 25).

are lipophilic molecules, so if they are still present in seeds they will be recovered with oil. Regarding carotenoids in the ethanol supernatant, the content ranged from 1 to 45 mg/kg dry extract, with exception of the 1810 mg/kg for hemp. Whatever the cake, carotenoid content of ethanolic phases was higher than that of the oil.

## 4 Conclusions

In this work, valorization of food industry by-products has been studied through the recovery of lipids from press cakes. SFE with ethanol as co-solvent allowed the recovery of 75 to 98% of the lipids and was highly influenced by the raw

material nature. Although the process used ethanol, the oil phase could be easily separated from the supernatant by decantation. The fatty acid profile of the oil was not affected by the extraction process. Hence, the proposed process could be of interest for efficient de-oiling of press cakes. Moreover, carotenoids and chlorophylls were extracted depending on their presence in the raw material. Although these compounds distributed in both oil and ethanol phases, their extraction showed that pigment-enriched oils could be produced by the process. About economic aspects of SFE, recent studies have highlighted the interest of such a technology for natural product extraction. The investment costs are not notably higher than those of classical extraction plants (Perrut, 2000) and the cost of manufacturing are often highly dependent from raw material cost (Pereira, 2010). In fat and oil transformation, SFE is gaining more and more interest over the years (Temelli, 2009). To conclude on economic viability of press cake $CO_2$ extraction, a cost analysis should be considered in the future.

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