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COMPARISON OF SAFFLOWER OIL EXTRACTION KINETICS UNDER TWO CHARACTERISTIC MOISTURE CONDITIONS: STATISTICAL ANALYSIS OF NON-LINEAR MODEL PARAMETERS

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Abstract - In this study the kinetics of oil extraction from partially dehulled safflower seeds under two moisture conditions (7 and 9% dry basis) was investigated. The extraction assays were performed using a stirred batch system, thermostated at 50 °C, using *n*-hexane as solvent. The data obtained were fitted to a modified diffusion model in order to represent the extraction kinetics. The model took into account a washing and a diffusive step. Fitting parameters were compared statistically for both moisture conditions. The oil yield increased with the extraction time in both cases, although the oil was released at different rates. A comparison of the parameters showed that both the portion extracted in the washing phase and the effective diffusion coefficient were moisture-dependent. The effective diffusivities were 2.81 10^{-12} and 8.06 10^{-13} m^2 s^{-1} for moisture contents of 7% and 9%, respectively.
Keywords: Effective-diffusion coefficients; Extraction kinetics; Safflower.

INTRODUCTION

Safflower (*Carthamus tinctorius* L.), like sunflower (*Helianthus annuus* L.), belongs to the *Compositae* family. It is an annual herbaceous plant native to Egypt, East Asia and the western coasts of North America. This species, cultivated for more than 2000 years for its dyeing properties, has sparked the interest of many countries because of its adaptability to different environmental conditions and, more specifically, for the quality of its seed oil (Giayetto *et al*., 1999).

Safflower seed consists of a fibrous and callous hull, two cotyledons and an embryo. It is similar in appearance to sunflower seed, but smaller. Its hull proportion varies widely from 18 to 59% of the seed weight in different cultivars. This value is negatively correlated with the oil and protein content of the seed (Baümler *et al*., 2004; Ekin, 2005; Vosoughkia *et al*., 2011). The lipid content of the dehulled seed is in the range of 36 to 43%. Safflower oil presents a higher refractive index, specific weight, density, iodine value and linoleic acid content than other common edible oils. It is an oil with reduced acidity, often preferred in northern countries due to its light taste. Safflower oil, rich in linoleic acid, is good for the skin and the intestinal mucosa; a favorable action in the prevention and treatment of rheumatic disorders

*To whom correspondence should be addressed is also ascribed to it (Dajue and Mündel, 1996). Traditionally it is used as massage oil for muscle aches and injuries. From an industrial point of view, it is a drying or semi-drying oil, used in the manufacture of paints and other surface coatings. Because it is transparent, it was used in white and/or bright paints so that they do not turn yellow in the course of time (Lu *et al*., 2004).

Safflower oil is obtained by pressing and conventional solvent extraction (Han *et al.,* 2009). Although kinetic assays have been carried out for decades in order to analyze the behaviors of different oilseeds (Fernández *et al*., 2012), few studies have been reported in the literature on the kinetic behavior of safflower seed oil extraction (Hu *et al*., 2012; Han *et al*., 2009), and none was found on the oil extraction kinetics of dehulled or partially dehulled safflower. The principle of solvent extraction is simple and is based on the fact that a component tends to partition between two phases depending on the equilibrium determined by the conditions and nature of the component and of both phases (Bockisch, 1998). The speed of this process is low due to the resistance of the structure to the diffusion of oil (Geankopolis, 2006). The extraction kinetics are influenced by several factors, including the intrinsic diffusion capacity of the solvent and solutes (which is mainly determined by the viscosity of each component), size, shape and internal structure of the seed particles. However, it is well known that the complex chemical nature of raw materials generates problems associated with processing time and extraction efficiency. In order to achieve an efficient extraction, an adequate amount of clean solvent is necessary to maintain the oil concentration in the miscella low enough to dissolve and separate the greatest amount possible of oil in each extraction step, and attain good contact and penetration (Brueske, 1996). In addition, both the knowledge of the seed oil content and how it can be obtained efficiently are of great interest, since the economic contribution to the oilseed trade is based on these data.

The model most used for representing the extraction of oilseed by solvent is a modification of Fick`s Second Law, which takes into account more than one mechanism: washing and diffusion and, in some cases, this second stage is subdivided in two, taking into account unhindered and hindered diffusion (Fernández *et al*., 2012). Some studies found that first-order kinetics represented this process using *Sterculia foetida* seeds and algaes as raw material (Sivakumar *et al*., 2012; Suganya and Renganathan, 2012). The industrial process of safflower oil extraction in some plants of Argentina, similarly to what happens with sunflower, begins with the dehulling of the seeds. Thus, the volume of material circulating in the extraction system decreases. Fiber content is also reduced by this process, thus making the flour suitable for the diet of monogastric animals (Baümler *et al*., 2002). Farran *et al*. (2010) studied the nutritional quality of meals obtained from seeds passed once or twice through the dehulling equipment. Reduction of hull content improved the protein and metabolizable energy contents, conferring to the meal characteristics similar to soybean meal.

The present analysis provides information considered of interest from a scientific and an industrial point of view, to determine the extraction kinetics of partially dehulled safflower seeds by analyzing the effect of moisture content. The aim of this work was to study the oil solvent extraction kinetics at two characteristic moisture levels using a modified diffusion model, and to analyze statistically the non-linear model fitting parameters.

MATERIALS AND METHODS

Preparation and Characterization of Raw Materials

Safflower seeds grown in Salta (25 ° and 65.5 ° LO LS), Argentina, were kindly provided by an oilseed company. Seeds were cleaned and manually selected to remove all foreign matter and broken or immature grains. Then the seeds were packed in hermetic bottles and stored at room temperature until further use. Moisture (Method 1.121, IUPAC, 1992), oil (Method 1.122, IUPAC, 1992), cellulose and lignin content (Van Soest *et al*., 1991) were determined according to standard methods. Measurements were replicated twice.

The hull:kernel ratio was determined by manually dehulling 10-g seed samples. To remove the hull from the kernel, two longitudinal cuts were made in the hull using a blade. Moisture content of the fractions was determined and the ratio was obtained on a dry basis (% d.b.). The mechanical dehulling of the grains (measured on a clean 10-g sample) was performed using a pilot impact dehuller. The dehuller unit was based on a combination of impact and shear forces acting on the grains. Rotor speed was controlled with a variable frequency drive and calibrated with a tachometer at 3300 rpm. The "dehulling ability" is the relationship between the percentage of mechanically extracted hull and the total hull content, both expressed as a weight percentage on a dry basis, and “fines” refer to the material not retained on the 2-mm sieve after the dehulling process in a pilot equipment. Baümler *et al*. (2002) determined that the best operating conditions to maximize the dehulling ability of safflower seeds (with a percentage of fines not higher than 10%) could be obtained if the system was operated at the above-mentioned speed, with a double pass through the dehuller and a grain moisture content of about 9% (d.b.). This moisture content was close to the optimum levels found by de Figueiredo *et al*. (2013) when analyzing the dehulling ability of six American safflower cultivars (from 8.4 to 8.9% d.b.). In the present work, a fraction of the sample with 9% d.b. moisture content was conditioned at 7% d.b. final moisture content. This value was selected mainly for two reasons: it is the value used by oil extraction plants, and it is the standard value used for the transportation and safe storage of safflower seeds (Bockisch, 1998).

Partially dehulled seeds (45% d.b.) were ground in a coffee grinder (Moulinex) and screened to a particle size in the 0.420-1.000 mm range for the different tests. The particle size and size distribution were characterized using a Horiba LA-910 laser-scattering particle size analyzer (HORIBA, Japan). The particle size was calculated by the analyzer based on the average value of the particles' geometrical length measured through different orientations of scattering light incidence. Circularity was used as the shape descriptor and calculated using a combination of ImageJ® and digital microphotograph. Photographs of the meals were taken using a digital camera (Nikon D3100, 14 Mega pixels resolution) connected to a desktop magnifier (15X).

Rate of Oil Extraction

Samples of the meal, approximately 5.0 ± 0.1 g, were submitted to extraction in 250-mL Pyrex flasks with a hemispherical base, immersed in a temperature controlled water bath at 50 ± 0.1 ºC, and magnetically stirred (200 rpm) to maintain in suspension the particles of the ground seeds.

Technical grade *n*-hexane was used at a 1:10 meal-to-solvent ratio (wt/vol) (Pérez *et al*., 2011). The ground sample and solvent were heated separately using a water bath to the extraction temperature before each test, and the temperature was checked with a thermocouple. Then the solvent was put into the recipient containing the sample.

Assays were carried out at different times (from 600 to 64800 s) until equilibrium was reached. The amount of oil obtained at 64800 s was identified as M_∞ (mass of solute that diffuses at infinite time) because this time was considered to be long enough to attain the equilibrium state (Pérez *et al*., 2011). When the established time had elapsed, miscella was separated from the meal by filtration through Whatman No. 42 filter paper, collected, and the solvent evaporated in an R-3000 Büchi vacuum rotary evaporator (Switzerland), not exceeding 55 ºC. Residual hexane was removed in a nitrogen stream to constant weight. The amount of oil was measured gravimetrically using a Sartorius balance (Model: PB211D, precision: 0.1 mg).

Extractions were replicated twice for each of the experimental conditions.

Kinetic Model

The experimental data were fitted to a diffusive model proposed by Pérez *et al*. (2011). The model considers the initial washing step, where rapid non-diffusive phenomena take place, according to the following equation

$$\frac{M_t}{M_\infty} = 1 - \left(1 - \frac{M_0}{M_\infty}\right)\sum_{n=1}^{\infty} A_n\, e^{-B_n.(t-t_0)} \quad (1)$$

where t_0 is the period that the washing stage lasts, and M_0 is the amount of oil solute extracted at t_0, M_t and M_∞ represent the mass of the substance (kg oil/kg dry defatted meal) that diffuses at time t (seconds) and infinite time, respectively. For long periods of time, the model can be expressed as shown in Eq. (2).

$$\frac{M_t}{M_\infty} = 1 - Ae^{-B.t} \quad (2)$$

Where the coefficient A is associated with the average value of the oil extracted in the washing step (M_0), and B is associated with the effective diffusivity (D_{eff}). In turn, both the coefficients A and B are influenced by the shape of the meal particle (Pérez *et al*., 2002). When the particles are spherical, the coefficients are given by

$$A = \left(1 - \frac{M_0}{M_\infty}\right)\frac{6}{\pi^2}e^{-B.t_0} \quad (3)$$

and parameter B is associated with the effective diffusivity (D_{eff}, m2/s) (D_{eff})

$$B = \frac{D_{eff}.\pi^2}{R^2} \quad (4)$$

where R (m) is the average particle radius.

Statistical Analysis

The mathematical model was applied to fit the experimental extraction data of safflower at two moisture levels using a nonlinear regression (SIGMAPLOT for Windows Version 11, 2008). The fitted regression models for different moisture levels were compared through their parameters, by using a procedure based on the principle of "extra sum of squares" and "conditional error", with a significance level of 95% (Fernández *et al*., 2012).

The null hypothesis (H_0) and the alternative hypothesis (H_1) were proposed: H_0, the model parameters A and/or B do not depend on moisture (*Global model* if both are consistent with moisture, *common A model* when only B varies with moisture, and *common B model* when only A depends on moisture content). H_1: model parameters A and B depend on moisture content (individual parameter model). With the extra sum of squares of each model, the statistic F_0 was obtained, which allowed a comparison of the models referred to each hypothesis (Fernández *et al*., 2012; Boché and Lavalle, 2004).

The predicted and measured values were compared by regression analysis (Kobayashi and Salam, 2000).

RESULTS AND DISCUSSION

The approximate composition of moisture-free whole safflower seeds used in the experiments is shown in Table 1. Safflower seeds presented an oil content in the range expected for genotypes grown in Argentina (41.4 to 44.9% d.b.; Smith 2002) and comparable to that of an Argentine oilseed sunflower hybrid, whose trading basis is 42% oil content (Table 1). However, hull content (35.9 ± 0.4% d.b.) was higher than typical values observed in this country, 31.9 to 33.2% d.b. (de Figueiredo *et al*., 2013), but similar to those reported in Turkey (Isiğigürel *et al*., 1995). These differences could be attributed to different genotypes. Cultivar improvement has decreased the mean hull content from 45-48% to about 33% (de Figueiredo *et al*., 2013).

Table 1: Safflower seed characteristics.

Determination (% d.b.)	Value
Moisture	9.0 ± 0.38
Oil	43.4 ± 3.6
Lignin	
Whole seed	11.4 ± 0.8
Hull	13.8 ± 1.2
Cellulose	
Whole seed	45.3 ± 2.8
Hull	46.1 ± 2.6
Hull/kernel ratio	0.560 ± 0.007

Lignin content in the whole seed (Table 1) was in the range previously reported for domestic sunflower (8-11%, Pérez *et al*., 2002), whereas cellulose content was higher (14-20%, Pérez *et al*., 2002). However, cellulose content in the hull was lower than that reported by Veldstra *et al*. (1990) for sunflower seeds (60%).

In turn, the oil content of the meal of the partially dehulled seeds (45%) was 40.9 ± 0.4 % d.b. The particle size distribution of the meal samples (Fig. 1) resulted in an average diameter (D_p) of 775 ± 15 µm. Figure 2 shows a sample of partially dehulled safflower meal.

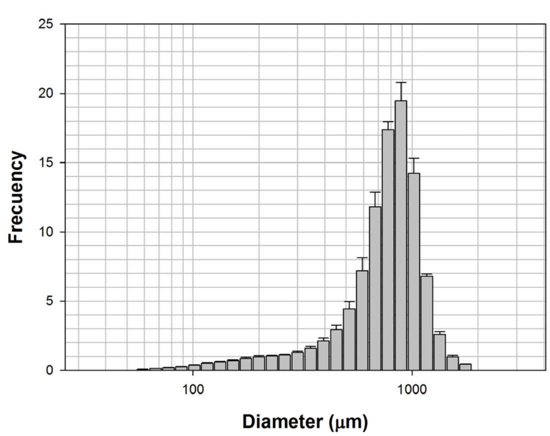

Figure 1: Safflower meal particle size distribution.

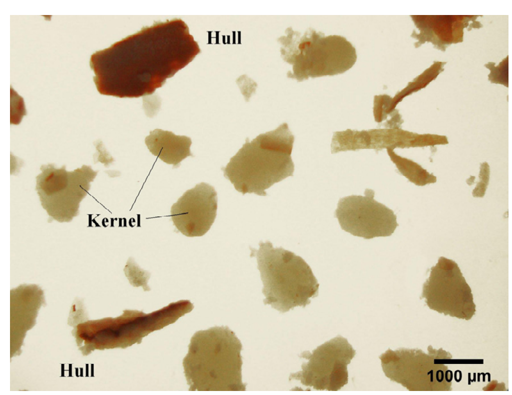

Figure 2: Digital photograph of safflower meal (x 15).

In the microphotograph it is possible to observe the complex and irregular morphology of the material, displaying the differences between the endosperm (light colored material that constitutes the "kernel" or seed core) and the hull (darker color, more rigid material). The endosperm is the cotyledons of the seed and it contains the highest percentage of oil and protein. Its structure is granular, as shown in the images. In turn, the hull consists mainly of cellulose, presenting a rectangular and acircular shape. The analysis of the shape descriptors showed that the particle circularity was 0.89 ± 0.10, and so a spherical geometry for the particle can be assumed.

Fig. 3 shows oil extraction experimental data of M_t/M_∞ at different times for both moisture levels.

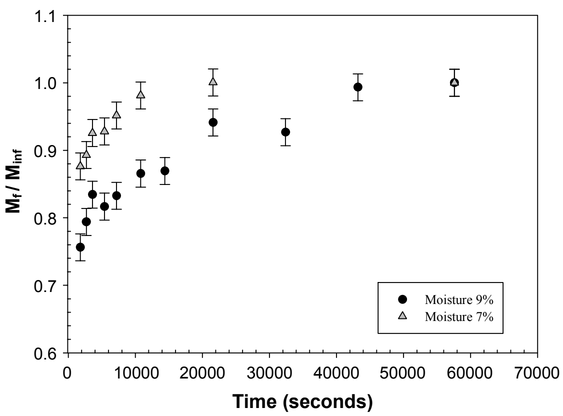

Figure 3: Oil extraction kinetics of partially dehulled safflower seeds at two different moisture levels.

The experimental data for the two moisture levels presented an initial washing zone followed by an asymptotic zone that corresponded to the slow diffusive process. Oil yield increased with extraction time in both assays but the oil was released at different rates depending on their moisture content. The average extracted oil contents were 0.6902 and 0.8602 kg oil / kg dry defatted meal at infinite time for 7% and 9% moisture content, respectively. The variations in the content of lipid extract would indicate that more components are extracted at high-moisture content, including hydratable phospholipids.

When analyzing the first-order kinetics applied by Sivakumar *et al*. (2012) to *Sterculia foetida*, the safflower oil extraction yield data obtained in this work could be represented up to 3600 s of extraction. At that time there was an important amount of oil remaining in the sample (4% and 8.5% at 7 and 9% of moisture content, respectively). Therefore it was necessary to study this process over a wider time range, leading to a non-linear model, in order to meet the industrial requirements (Bockisch, 1998; Anderson, 2006).

Table 2 shows fitted coefficients of the model used to represent oil extraction for all the times studied (from 600 to 64,800 s, until equilibrium was reached): *different A and B* model, *common A* model, *common B* model and *global* model.

The comparison of the *different A and B* model with the *common A*, *common B* and *global* models showed significant differences in all cases ($F_o > F_c$), demonstrating that both parameters A and B depend on the moisture content present in the sample. Thus, in order to represent safflower oil extraction kinetics, the *different A and B* model was selected.

Goodness of fit of the selected model was evaluated. F_o^{dc} was 0.95 and 1.02 for the lower and the higher moisture level, respectively. The corresponding critical values F_c^{dc} were 2.82 and 3.44, respectively. Therefore, lack of fit was not significant at a confidence level of 95%, indicating that the selected model is acceptable.

Table 2: Fitted coefficient obtained for the proposed model for safflower oil extraction. Non-linear model comparison.

Description of the proposed fitting	Coefficient	Moisture content (% d.b.)		Fo	Fc
		7	9		
Different A and B	A x 10^2	17.1 ± 1.3	24.1 ± 1.5	---	---
	B x 10^4	1.85 ± 0.21	0.53 ± 0.07		
	Adj.R^2	0.98	0.94		
Common A	A x 10^2	20.05 ± 1.27	20.05 ± 1.27	89.43	3.59
	B x 10^4	2.27 ± 0.13	0.39 ± 0.06		
	Adj. R^2	0.96	0.88		
Common B	A x 10^2	12.01 ± 1.03	28.75 ± 1.89	27.30	3.59
	B x 10^4	0.91 ± 0.13	0.91 ± 0.13		
	Adj. R^2	0.87	0.85		
Global (Common A and B)	A x 10^2	20.05 ± 1.27		83.93	3.68
	B x 10^4	0.91 ± 0.13			
	Adj. R^2	0.97			

The mean value of the coefficient A increased with moisture content. Average values of the oil fraction involved in the washing step were calculated by means of Eq. (2). M_0/M_∞ ratios, expressed as percentages, were 71.87 ± 2.1% and 60.36 ± 2.05% for the moisture levels of 7 and 9% d.b., respectively, thus evidencing that the surface oil content is moisture-dependent. In the sample with higher moisture content it can be observed that the washing step was faster because the presence of more water causes collapse of the cellular structure by dissolving soluble components, making more oil available at this stage. The result obtained for the lower humidity level was similar to that reported for three ground sunflower genotypes studied previously under the same extraction and moisture conditions (Pérez *et al*., 2011).

Coefficient B, corresponding to effective diffusivity, decreased with moisture content. Effective diffusivities, evaluated from Eq. (3), were $2.81\ 10^{-12}\ m^2.s^{-1}$ and $8.06\ 10^{-13}\ m^2.s^{-1}$ at 7 and 9% moisture levels, respectively. The difference in the values obtained could indicate that the water absorbed by the solid prevents the solvent from penetrating the solid matrix due to the polarity difference between them.

Effective diffusivities were close to those obtained previously for ground oilseed sunflower seeds (Pérez *et al*., 2011). A decrease of about 22% in the moisture content of the safflower sample made this coefficient increase by 63%. Fan *et al*. (1948) reported a range of diffusivity values between $3.2\ 10^{-13}$ and $8\ 10^{-13}\ m^2.s^{-1}$ for oil extracted from peanut slices sized to 0.2-0.4 mm, indicating that, for each 1% that the moisture changed, this coefficient varied $0.4\ 10^{-13}\ m^2.s^{-1}$.

CONCLUSIONS

Oil extraction by stirring from partially dehulled safflower seeds was described satisfactorily using a modified diffusive model, obtained from Fick's Second Law, which involved two parameters: A, associated with the fraction extracted during the first washing stage, and B, related to diffusivity. Both were significantly affected by the moisture content of the sample.

The kinetic study of oil extraction from safflower meal showed that the oil was released at different rates depending on the moisture content. The value of the average fraction of oil involved in the washing phase increased with increased moisture content but the diffusive coefficient decreased.

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NOMENCLATURE

A, B	diffusion model-fitting parameters	
d.b.	dry basis	
D_{eff}	effective diffusion coefficients	m^2/s
D_p	average diameter	μm
M	mass of substance that diffused at infinite time	kg oil/kg dry defatted meal
M_o	surface oil	kg oil/kg dry defatted meal
M_t	mass of substance that diffused at time t	kg oil/kg dry defatted meal
M_∞	mass of substance that diffused at infinite time	kg oil/kg dry defatted meal
R	average radius	M
T	extraction time	S
t_0	washing time	S
F_0	contrast statistics for parameter comparison	
F_0^{dc}	contrast statistics for direct comparison	
F_c	critical value of Snedecor's F-distribution for the comparison of the parameters	
F_c^{dc}	critical value of Snedecor's F-distribution for the direct comparison	

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