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Temperature-dependent diffusion coefficient of oil from different sunflower seeds during extraction with hexane

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ABSTRACT

Oil extraction from confectionery, oilseed and wild sunflower seeds with *n*-hexane was investigated by laboratory tests carried out in a stirred batch extractor at several temperatures (40, 50 and 60 °C). The rates of extraction were determined from ground sunflower seeds (particle sizes between 0.420 and 1.000 mm). The oil yield in the extract increased with higher contact time and extraction temperature in all the cases. Equilibrium constants at 50 °C for different solvent–ground seed ratios are reported. A mathematical model of oil extraction from seeds of sunflowers, based on a modified diffusive process in spherical geometry of particles, was proposed. The analysis of significance of the coefficient of fitting regression models showed significant differences between temperatures for each genotype and between genotypes at each temperature. The resulting diffusion coefficient ranged from 1.34×10^{-12} to 1.87×10^{-12} m²/s for confectionery, 2.06×10^{-12} to 5.03×10^{-12} m²/s for oilseed, and 9.06×10^{-13} to 1.18×10^{-12} m²/s for wild sunflower. The temperature dependence of the diffusion coefficient was represented by an Arrhenius-type equation for each sunflower seed studied. Activation energy values of 13.74, 33.95 and 11.32 kJ/mol were obtained for confectionery, oilseed and wild sunflower, respectively.

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1. Introduction

In recent decades scientific and technological advances have turned their focus to raw materials of vegetable origin considering that their application will allow not only to cope with the food demand, but that they may also replace some of the raw materials derived from non-renewable hydrocarbons. Oilseed species can be considered the suppliers of industrial raw materials for a variety of oleochemicals used in the manufacture of biodegradable plastics, pharmaceuticals, detergents and lubricants, among other products (Perez et al., 2004; Perez and Nolasco, 2010).

Even though the oilseed industry is consolidated and the technological changes are not radical, the improvement objectives are focused on finding the appropriate processing conditions to obtain the greatest yield with a good product quality. Solvent extraction is the most widely used method to separate the oil from the seed. Although this extraction principle is simple, the phenomenon is influenced by type and composition of seed, pretreatments that affect the cellular structure, size and geometry of particles, temperature and solvent, among others. Oilseed processing plants require appropriate designs that deal with the physical characteristics of each particular grain. Diffusivity is an important transport property

for predicting the mass transfer coefficients, and it is useful for designing mass transfer equipment.

In the 1950s, the first mathematical models to predict oil extraction based on experimental data mainly for soybeans, cottonseed and peanuts were reported (Karnofsky, 1949; Coats and Karnofsky, 1950). Different approaches have been presented to explain the oil content remaining in the solid as a function of flake thicknesses and extraction time (Boucher et al., 1942; Fan et al., 1948; Coats and Wingard, 1950). The literature reports that the limiting extraction stage is due to the chemical composition of the residual oil, mainly to the increment of less soluble components such as phosphatides and non-glycerides (Coats and Karnofsky, 1950; Coats and Wingard, 1950).

The most widely accepted model to explain the extraction process considers two main mechanisms: a washing process of the oil on the grain surface, and a diffusion process, which can take place in one or two phases, depending on the proportion of broken and intact cells that remain after pre-extraction treatments (Patricelli et al., 1979; So and Macdonald, 1986). This model has been applied in the study of oil extraction from dehulled sunflower, rapeseed and olive cake. The same approach was recently used by Carrin and Crapiste (2008) to develop a two-dimensional nonsteady-state mathematical model for vegetable oil–solvent extraction in a DeSmet type extractor. These previous studies refer to the oil extraction from different oilseeds, but there is a lack of information about the effect of seed genotypes on extraction kinetics and the

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Nomenclature

A_n, B_n	diffusion model-fitting parameters	R_g	gas constant (kJ/mol K)
a, b	equilibrium model-fitting parameters	t	diffusion time (s)
D_e	effective diffusion coefficients (m ² /s)	T	absolute temperature (K)
D_o	preexponential constant (m ² /s)	ΔE_d	activation energy (kJ/mol)
D_p	average diameter (μm)	C_{om}	oil content per kg of dried oil-free solid at equilibrium (kg oil/kg inert solid)
M_x	mass of substance that diffused at infinite time (kg oil/kg dry defatted meal)	C_{ob}	the miscella oil content (bulk) at equilibrium (kg oil/kg solvent)
M_o	surface oil (kg oil/kg dry defatted meal)		
M_t	mass of substance that diffused at time t and infinite time (kg oil/kg dry defatted meal)		
R	average radius (m)		

applicability of the above mentioned models to represent the process.

In the present work, the influence of kinetic parameters such as temperature and solvent-meal relation in the hexane-extraction of oil from three genotypes of sunflower seeds is investigated. In addition, a diffusive extraction model with a temperature-dependent diffusion coefficient is used to describe simultaneously the experimental behavior of these genotypes.

2. Materials and methods

2.1. Materials

Three groups of tests were performed using confectionery, oil-seed (*Helianthus annuus*) and wild (*Helianthus petiolaris*) sunflower seeds. Seed samples were obtained through the courtesy of INTA (Argentina). The hexane used in oil extraction experiences was reagent grade.

2.2. Preparation and characterization of the samples

Whole seeds were ground in a coffee grinder (Moulinex) and screened to a particle size in the range of 0.420–1.000 mm for the different tests, in accordance with the optimal milling values suggested by the bibliography consulted (Chien et al., 1990; Myint et al., 1996). The particle size and size distribution were characterized using a Horiba LA-910 laser-scattering particle size analyzer (HORIBA, Japan). The particle size is calculated by the analyzer, based on the average value of particle's geometrical lengths measured through different orientation of incidence scattering light.

The initial moisture and oil content of ground seeds was determined by IUPAC (1992) 1.121 and 1.122, respectively. Fatty acids, crude fiber and protein content were determined by AOCS standard methods (1993).

The data obtained were analyzed by performing an analysis of variance, and the comparison of means was performed using Tukey's test ($p \leq 0.05$).

2.3. Equilibrium

The equilibrium content of oil in sunflower meal and n -hexane solvent were obtained in a stirred batch system. It consisted of a Pyrex 150 mL flask immersed in a thermostatically controlled waterbath at 50 °C ± 0.1, stirred by a magnetic bar to maintain in suspension the particles of ground seed.

Samples of the meal, approximately 5.0 ± 0.1 g, were submitted to extraction with different meal-to-solvent ratios (R_{hexane}) 1:5, 1:7.5, 1:10, 1:15 and 1:20 (wt/vol). The meal and the solvent were brought to extraction temperature before every experience. The miscella was withdrawn after 14 h, which was considered enough

time to attain the equilibrium state. The amount of oil in the liquid phase was measured gravimetrically by solvent evaporation. Extractions were replicated twice for each of the experimental conditions.

2.4. Rate of oil extraction

The same device and procedure previously described was used to obtain the extraction data at different times until achieve equilibrium state. A sufficiently high stirring speed was used, 200 rpm ($N_{Re} = 11355$), to assure a negligible external resistance to mass transfer in modeling the extraction kinetic. The tests were carried out at three temperatures (40, 50 and 60 °C) with a meal-to-solvent ratio of 1:10 wt/vol. The results are given as mean values of duplicate samples.

2.5. Mathematic model

A modified model of Fick's Law of diffusion in non-stationary state for spherical particle geometry, suspended in a homogeneous medium of constant concentration without volume restriction, was used. The solution of the Fick's equation is given by:

$$\frac{M_t}{M_\infty} = 1 - \sum_{n=1}^{\infty} A_n \exp(-B_n t) \tag{1}$$

where t is a diffusion time (seconds) and M_t and M_∞ represent the mass of the substance (kg oil/kg dry defatted meal) that diffused at time t (seconds) and infinite time, respectively.

The model proposed takes into account the initial time where rapid non-diffusive phenomenon takes place. In that period, the fresh solvent removes the free surface oil of the meal by washing (M_o). The conditions can be represented as:

$$t = 0 \quad M = 0 \tag{2}$$

$$t = t_o \quad M = M_o \tag{3}$$

$$t = t \quad M = M_t \tag{4}$$

$$t \rightarrow \infty \quad M = M_\infty \tag{5}$$

and the Fick's equation can be solved to obtain, after rewriting:

$$\frac{M_t}{M_\infty} = 1 - \left(1 - \frac{M_o}{M_\infty}\right) \sum_{n=1}^{\infty} A_n \exp[-B_n(t - t_o)] \tag{6}$$

For sufficiently long times Eq. (6) can be simplified to:

$$\frac{M_t}{M_\infty} = 1 - A \exp[-B_1 t] \tag{7}$$

where the pre-exponential coefficient is given by

$$A = \left(1 - \frac{M_o}{M_\infty}\right) A_1 \exp(B_1 t_o) \tag{8}$$

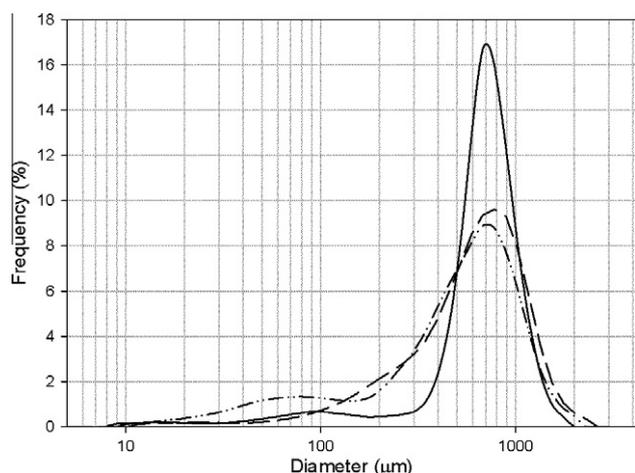


Fig. 1. Particle size distribution of extraction meal: confectionary sunflower (—), oilseed sunflower (---) and wild sunflower (- · - ·).

and the where the coefficients A_1 and B_1 for spherical geometry can be expressed as:

$$A_1 = \frac{6}{\pi^2} \quad (9)$$

$$B_1 = \frac{D_e \cdot \pi^2}{R^2} \quad (10)$$

Variation of the effective diffusion coefficients D_e with temperature was represented for an Arrhenius-type equation, obtaining:

$$D_e = D_0 \exp \left[-\frac{\Delta E_d}{R_g T} \right] \quad (11)$$

where D_0 is a constant (m^2/s^{-1}), ΔE_d the activation energy (kJ/mol), R_g the gas constant (kJ/mol K), and T the absolute temperature (K).

The mathematical model was applied to fit the experimental extraction data for sunflower varieties at different temperatures by using a nonlinear regression (SIGMAPLOT for Windows Version 11, 2008). The fitting regression models for different genotypes 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% (Ratkowsky, 1983).

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

3. Results and discussion

The particle size distribution of the meal samples (Fig. 1) resulted in an average diameter (D_p) of 586 ± 67 , 667 ± 18 and $624 \pm 42 \mu m$ for wild, confectionary and oilseed sunflower, respectively.

The chemical composition of the meal for the three genotypes is presented in Table 1. Tukey's test indicated that the samples showed significant differences in oil content ($p < 0.05$), but not in the fiber content. As for the percentage of protein, only the wild sunflower was statistically different, showing a lower content.

Fig. 2 shows the results of the determinations of residual oil content in the solid and the concentration of oil in miscella (bulk) in equilibrium at 50 °C and various solid-to-solvent ratios (R_{hexane}). At the equilibrium, it was supposed equal oil concentration in both the miscella absorbed in the solid and the one in the bulk. This assumption was reasonable according to the equilibrium results reported by other authors (Patricelli et al., 1979; Majundar et al., 1995). Higher R_{hexane} provided a larger driving force for the

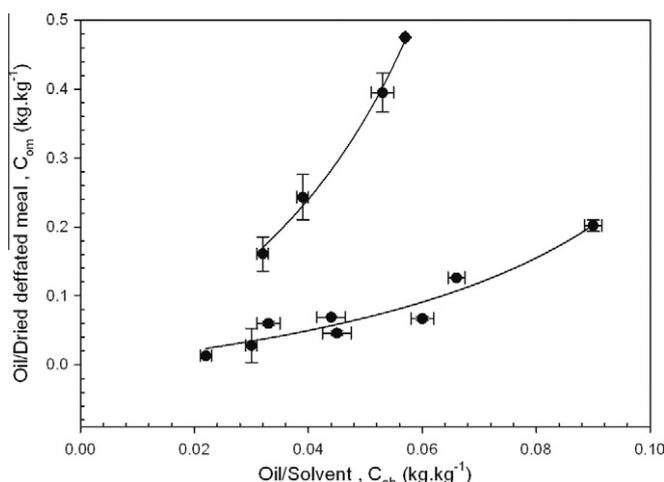


Fig. 2. Relation between the oil retained in the inert solid and the oil content in the miscella at equilibrium (50 °C): confectionary sunflower (●), oilseed sunflower (▲) and wild sunflower (■). Plotted curves were fitted using Eq. (12).

extraction. These results are in agreement with the analysis carried out by Thobani and Diosady (1997) for rapeseed oil extraction. They reported that the driving force increased for R_{hexane} greater than 10, and that the concentration of residual oil decreased in a linear relation.

It can be observed that at a lower solid-to-solvent ratio, the oil content in the miscella retained by the solid was higher. The oilseed sunflower presented a higher retention of residual oil in the solid. It has been reported that the physicochemical composition of the extraction meal with respect to protein and lignocelulosic content could explain the differences in the quantity of miscella retained, since the hexane is physically bound to these fractions (Crossley and Aguilera, 2001). This observation is in accordance with the high percentage of protein that presents the oilseed sunflower (Table 1).

Meal oil content in a dried oil-free basis (C_{om} , kg oil/kg inert solid) versus the miscella oil content (bulk) (C_{ob} , kg oil/kg solvent) at equilibrium were adjusted according to the following relation:

$$C_{om} = \frac{aC_{ob}}{1 + bC_{ob}} \quad (12)$$

where the constant a is the slope of the linear region, and the constant b represents the curvature that is significant at higher concentrations. The values for the three sunflower varieties obtained by nonlinear regression (SIGMAPLOT 11.0) were: 0.6083 and -8.095 for confectionary (r^2 : 0.999), 3.663 and -9.772 for oilseed (r^2 : 0.998), and 1.152 and -6.069 for wild sunflower (r^2 : 0.967). From the comparison of fitting regression models it arise that the equilibrium did not present significant difference among genotypes for wild and confectionary sunflower, so it could be represented by a single equation ($a = 0.916$, $b = -6.5947$, $r^2 = 0.929$; $F = 6.04$, $F_c = 6.49$).

The equilibrium constant or partition constant, defined as the ratio of the residual oil concentration in the solid phase to the oil content in the miscella ($K = C_{om}/C_{ob}$) are in the range 0.59–1.87, 0.95–2.24 and 4.98–8.30 for wild, confectionary and oilseed sunflower, respectively.

Extraction kinetics for confectionary, oilseed and wild sunflower are shown in Figs. 3–5. The experimental data for the three genotypes presented an initial washing zone followed by an asymptotic zone that corresponded to the slow diffusive process. Studies have shown that the fastest extraction rate corresponded to pieces having complete breakage of the cell wall and a layer of

Table 1
Chemical composition of the oil and meal of the three sunflower varieties.

Parameter (% d.b.)	Confectionary sunflower	Oilseed sunflower	Wild sunflower
Moisture	7.1 ± 0.3 ^a	7.5 ± 0.2 ^a	6.8 ± 0.2 ^a
Oil	29.2 ± 0.9 ^a	49.9 ± 1 ^b	21.3 ± 0.7 ^c
Crude fiber	21.4 ± 0.8 ^a	23.8 ± 1.0 ^a	24.2 ± 1.3 ^a
Protein	35.8 ± 1.8 ^a	32.8 ± 0.6 ^a	23.0 ± 0.2 ^b
<i>Fatty acid composition</i>			
C _{16:0}	6.3 ± 0.1 ^a	6.4 ± 0.3 ^a	5.2 ± 0.8 ^a
C _{18:0}	3.3 ± 0.3 ^b	2.7 ± 0.2 ^b	1.85 ± 0.1 ^a
C _{18:1}	22.6 ± 1.1 ^{a,b}	19.0 ± 0.2 ^b	27.0 ± 2.3 ^a
C _{18:2}	67.6 ± 1.0 ^a	71.8 ± 0.4 ^a	66.0 ± 3.2 ^a

d.b.: Dry basis. Different letters indicate significant differences ($p \leq 0.05$).

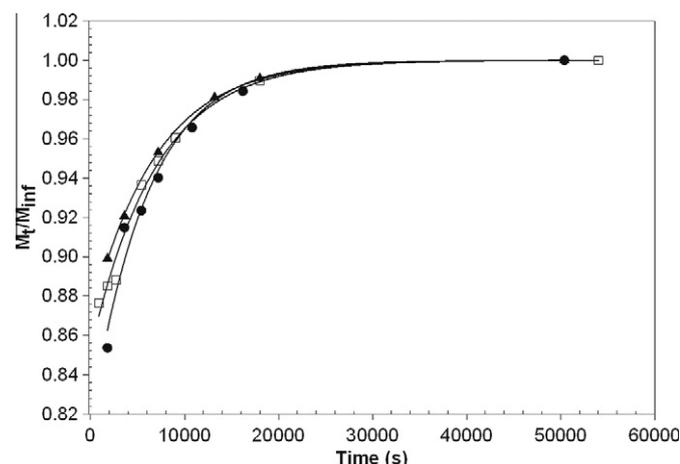


Fig. 3. Experimental and predicted (Eq. (7)) oil extraction kinetic from confectionary sunflower seed at 40 °C (●), 50 °C (□) and 60 °C (▲).

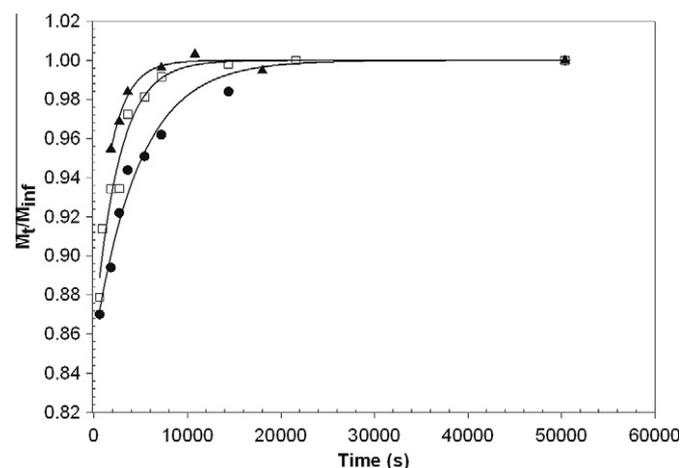


Fig. 4. Experimental and predicted (Eq. (7)) oil extraction kinetic from sunflower oilseed at 40 °C (●), 50 °C (□) and 60 °C (▲).

cytoplasm exposed directly to the solvent, while the slowest rate belonged to intact cells (Crossley and Aguilera, 2001). On the other hand, it is observed that the extraction rate increased with temperature for the three varieties studied, being higher for the oilseed. The change in the yield can be explained by the fact that a temperature rise increases the solubility and diffusion of the oil while decreasing viscosity.

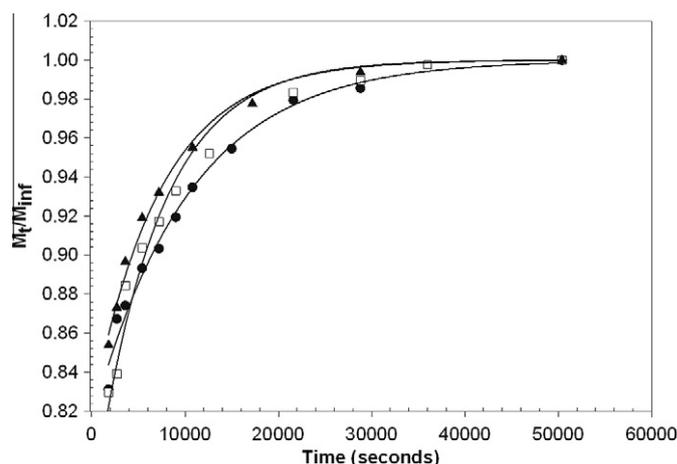


Fig. 5. Experimental and predicted (Eq. (7)) oil extraction kinetic from wild sunflower seed at 40 °C (●), 50 °C (□) and 60 °C (▲).

The experimental kinetic curves were fitted according to the proposed model, Eq. (7); and the obtained coefficients are given in Table 2. The experimental data correlated very well for all samples ($r^2 > 0.97$) and the model is able to represent the observed behavior (Figs. 3–5). The analysis of significance of the fitting regression models showed significant differences between temperatures for each genotype (confectionary, $F = 8.56$, $F_c = 3.18$; oilseed, $F = 31.2$, $F_c = 3.18$; wild sunflower $F = 17.3$, $F_c = 3.11$) and between genotypes at each temperature (T : 40 °C, $F = 62.75$, $F_c = 2.93$; T : 50 °C, $F = 44.29$, $F_c = 2.84$; T : 60 °C, $F = 52.91$, $F_c = 3.11$).

The values for coefficient A diminished as the initial oil content of the samples increased. The oil fraction involved in the initial washing phase, calculated from Eq. (8), represents the 76.6%, 77.7% and 68.6% in average of those extracted at infinite time for confectionary, oilseed and wild sunflower seeds, respectively.

The values for the coefficient B , which includes the diffusion coefficient, increased with temperature and initial oil content. The calculated diffusion coefficient ranged from 1.34×10^{-12} to 1.87×10^{-12} m²/s for confectionary, 2.06×10^{-12} to 5.03×10^{-12} m²/s for oilseed, and 9.06×10^{-13} – 1.18×10^{-12} m²/s for wild sunflower. In turn, there are indications that the diffusion coefficient increased with the decreasing saturation degree of the oil (Anderson et al., 1977). For example, the diffusive coefficient for linoleic acid was 3.6×10^{-11} m²/s, whereas for oleic acid it was 2.6×10^{-11} m²/s (Anderson et al., 1977). As shown in Table 1, wild sunflower had the lowest oil content and the highest percentage of oleic acid, which is in agreement with the values obtained for the diffusion coefficients.

The predictions obtained are in accordance with those reported by other authors for the extraction with hexane, such as 7.0×10^{-13} m²/s for peanut slice with 13% moisture at 25 °C (Fan et al., 1948), and 3.4×10^{-12} m²/s for rapeseed oil at 55 °C (Şaşmaz, 1996).

Fig. 6 shows the effect of temperature on the diffusion coefficient. The confectionary and wild sunflower showed a similar trend while oilseed sunflower had a higher temperature dependence. The corresponding values of activation energy obtained by correlation ($r^2 > 0.922$) were 13.74, 33.95 and 11.32 kJ/mol were obtained for confectionary, oilseed and wild sunflower, respectively.

Simulated results agreed with the data obtained from experimental tests (Fig. 7). In all cases, slopes and intercepts were not significantly different from one and zero, respectively, indicating a good prediction of oil extraction.

Table 2
Model-fitting results of three varieties of sunflower.

Sunflower	Temperature (°C)	Coefficient A ^a		Coefficient B ^a		r ²
		Estimate	%CV	Estimate	%CV	
Confectionary	40	0.1361	6.38	1.19×10^{-4}	8.74	0.9911
	50	0.1516	1.66	1.53×10^{-4}	2.67	0.9988
	60	0.1398	6.20	1.66×10^{-4}	9.97	0.9898
Oilseed	40	0.1452	5.86	2.09×10^{-4}	10.80	0.9752
	50	0.1446	4.38	4.02×10^{-4}	7.96	0.9827
	60	0.1148	11.90	5.10×10^{-4}	10.04	0.9911
Wild	40	0.2100	3.29	1.04×10^{-4}	5.84	0.9895
	50	0.1921	4.14	1.19×10^{-4}	7.34	0.9878
	60	0.1798	3.51	1.35×10^{-4}	6.16	0.9930

^a All coefficients have significance levels less than 0.05.

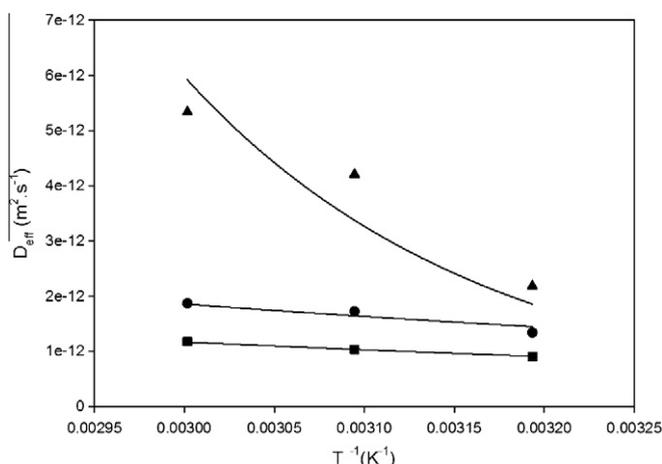


Fig. 6. Temperature-dependent diffusivity of the oil from confectionary sunflower (●), oilseed sunflower (▲) and wild sunflower (■) during *n*-hexane extraction.

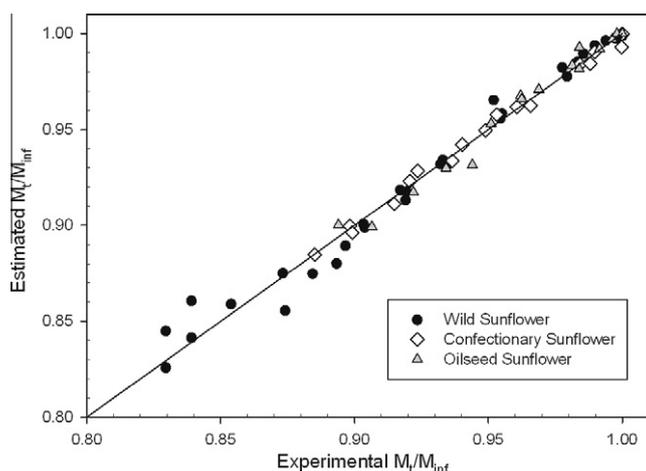


Fig. 7. Linear correlation between M_t/M_{inf} values estimated by model and those experimentally determined. The solid line indicates the 1:1 relationship.

4. Conclusions

Although oil extraction depends hardly of the structure and composition of the solid matrix, no previous studies had been reported on the effect of genotype and meal composition. In this work, we studied the equilibrium and kinetics of oil extraction with hexane from confectionary, oilseed and wild sunflower seeds. A mathematical model of oil extraction from ground sunflower

seed meal, based on an initial washing stage followed by a diffusive stage, was proposed to represent the extraction kinetic. This model is valid for the three analyzed sunflower genotypes, being the model coefficients different according to the genotype and temperature considered. However, it allows estimating the effective diffusion coefficient and its temperature dependence. The diffusion coefficient increased with temperature, initial oil content and degree of unsaturation. Oilseed sunflower showed the highest equilibrium constant, diffusion coefficient and activation energy.

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