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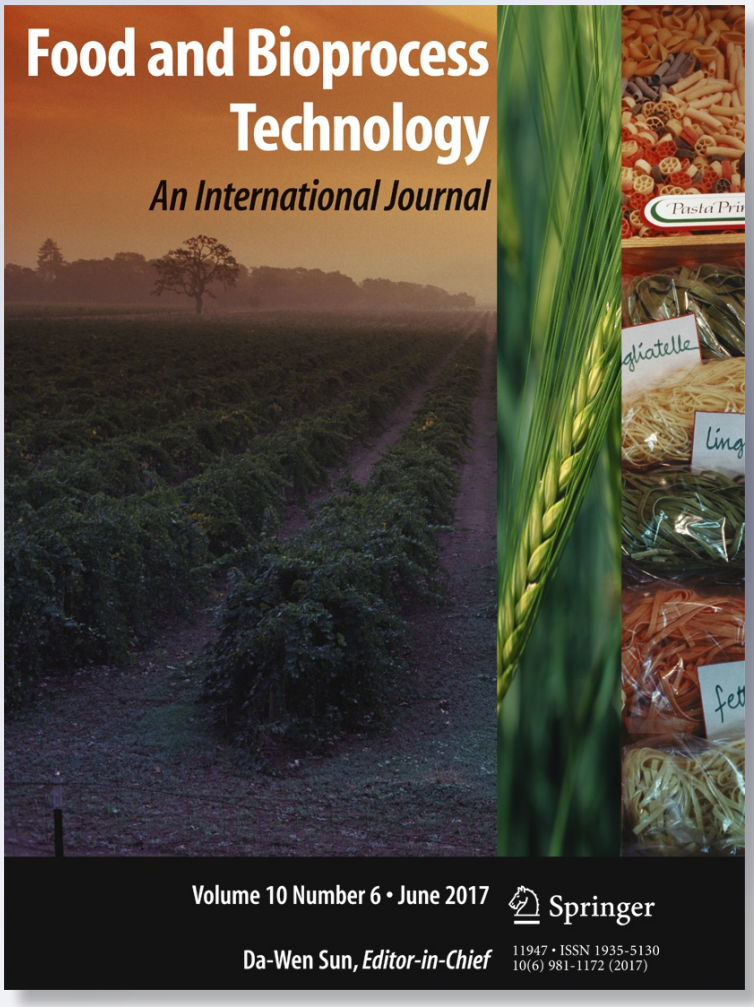

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ORIGINAL PAPER

Optimization of Sesame Oil Extraction by Screw-Pressing at Low Temperature

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Abstract Box-Behnken designs were used to optimize a process for sesame oil extraction by screw-pressing at low temperature (50 °C). Experimental designs included seed moisture content (SMC), pressing speed (PS), and restriction die (RD) as the main processing parameters. Extractions at pilot plant scale showed a peak in oil recovery (OR, 71.1 ± 2.80%) at 12.3% SMC, 4 mm RD, and 20 rpm PS. Theoretical models were scanned against experimental data in order to scale up the proposed oil extraction process to industrial scale. A fitted model for OR showed a maximum predicted value similar to the highest experimental value (74.4 ± 1.23%) under the following conditions: 8.03% SMC, 10 mm RD, and 20 rpm PS. Chemical quality parameters of oils obtained at both pilot and industrial scales were in the ranges stated in Codex (FAO/WHO) standards for non-refined sesame oil.

Keywords Sesame oil · Screw-pressing extraction · Processing parameters · Oil recovery · Chemical quality parameters

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Abbreviations

AAD	Absolute average deviation
Af	Accuracy factor
Bf	Bias factor
FA	Fatty acid
FAME	Fatty acid methyl ester
FC	Fine solid content
FFAC	Free fatty acid content
FID	Flame ionization detector
OR	Oil recovery
OSI	Oxidative stability indexes
PUFA	Poly-unsaturated fatty acids
PV	Peroxide value
PS	Pressing speed
RD	Restriction die
SMC	Seed moisture content
SO	Sesame oil
TLC	Total lignan content
RSC	Radical scavenging capacity
RSM	Response surface methodology
WB	Wet basis

Introduction

Sesame (*Sesamum indicum* L., Pedaliaceae) is a common oilseed crop cultivated in tropical and sub-tropical regions of the world. Sesame seeds have high economical and nutritional value mainly due to their high oil content (45–60%). The world's sesame oil production was about 4,756,000 tons in 2013 (FAOSTAT 2015. FAO Statistics Division); India, Sudan, and China were the major producers. The oil is used mainly for edible purposes, as a salad dressing and to produce shortenings and margarine (Kochhar 2002; Rostami et al. 2014).

Sesame oil (SO) is composed predominantly of mono- and di-unsaturated fatty acids (FA). Oleic and linoleic acids are present at similar amounts (35–50% each), averaging together 90% of the total FA content. Linolenic acid is found at very low concentrations (<1%). As compared to many other edible vegetable oils, SO contains higher amounts of unsaponifiable matter (up to 3% of the total oil content). This includes primarily tocopherols (400–700 mg/kg), free and esterified sterols, and a specific type of lignans—namely sesamin, sesamolin, sesamol, and sesaminol—with important biological properties (Kochhar 2002; Rangkadilok et al. 2010).

Sesame oil is usually extracted by means of mechanical screw-pressing. The conventional screw-pressing processes used for SO extraction generally include a previous seed roasting step. Although seed roasting can increase oil recovery, it may have deleterious effects on oil quality. Yoshida (1994) and Yoshida and Takagi (1997) have reported increments in both hydrolytic and oxidative degradation parameters when roasting temperature increases from 120 to 250 °C. Besides seed roasting, other seed pre-treatments, such as cracking and moistening, and processing conditions, such as pressing speed and restriction die, are important as well (Martínez et al. 2012, 2013; Singh et al. 2012; Savoire et al. 2013). The seed moisture content at the time of pressing seems to be a key parameter for optimum oil recovery, as reported in previous studies with various oilseeds (Singh and Bargale 1990, 2000; Singh et al. 2002; Martínez et al. 2008, Martínez et al. 2012, Martínez et al. 2013). Singh and Bargale (1990, 2000) have observed peaks in oil recovery (OR) at 7% (WB) moisture content for water-soaked linseed, and at 7.5% for rapeseed, in the moisture content range of 5–12% (WB). Singh et al. (2002) have found increments in OR from both cooked and uncooked crambe seeds as moisture content decreases in the range of 9.2–3.6% (WB). By using screw-pressing at pilot plant scale, optimum oil recoveries from walnut, chia, and almond seeds were obtained at 7.5, 10, and 8% (WB) moisture contents, respectively (Martínez et al. 2008, 2012, 2013). In each case, the oil extraction performance was also dependent on the pressing temperature and other processing conditions.

It seems clear that in oil pressing-extraction processes, several factors may significantly influence the extraction efficiency and the chemical quality of the oils. To overcome the difficulty that supposes to plan an experimental design involving many factors, the response surface methodology is often applied. This method has been successfully used to optimize extraction conditions of various seed oils (Akinoso and Raji 2011; Martínez et al. 2012; Martínez and Maestri 2015). In spite of SO gaining commercial importance, it is surprising that the processes for oil extraction are sometimes inadequate to meet the physico-chemical and organoleptic properties required for use in food products (Kochhar 2002).

The present study was aimed to develop a process for SO extraction by screw-pressing at low temperature. Experimental designs including different levels of seed moisture content, pressing speed, and restriction die were used to optimize OR and quality at both pilot plant and industrial scales. Theoretical models were scanned against experimental data in order to scale up the proposed oil extraction process to industrial scale.

Materials and Methods

Seed Material

Whole (hulled) sesame seeds (*Sesamum indicum* L.) were obtained from commercial plantations located at Salta province, Argentina. Seeds were cleaned using an automated screen (EJR 2000 Zonytest®), packed in polypropylene bags, and stored at 5 °C until further use. In order to achieve the moisture content levels proposed in each experimental design, seeds were hydrated according to the methodology proposed by Singh and Bargale (2000). Briefly, seeds were sprinkled with fresh water, packed in air-tight containers, and stored for 48 h for equilibration. The containers were shaken at regular intervals to distribute moisture uniformly throughout the sample. Finally, seed samples were kept in a vacuum oven at 25 °C until the desired moisture level was reached. The latter was calculated after oven drying of 10-g aliquots of each seed sample at 60 °C for 72 h.

Oil Extraction

Oil extractions at both pilot plant and industrial scales were carried out by screw-pressing at 50 °C. Temperature was constantly monitored with a digital thermometer (TES Thermometer 1307 Type K) inserted into the restriction die.

Pilot Plant Scale

Oil extraction was carried out in a single step with a Komet screw press (Model CA 59G; IBG Monforts, Germany). The effective and total lengths, and the internal diameter of the press barrel were 3.1, 7.0, and 3.5 cm, respectively. The length and diameter of the screw were 15.0 and 3.0 cm, respectively. The experimental design included three levels of each of the following parameters: seed moisture content (SMC 7, 12, and

17% w.b.); pressing speed (PS 20, 40, and 60 rpm); restriction die (RD 4, 5, and 6 mm).

Industrial Scale

A Komet screw press (Model DD 85—PK1 and PK2; IBG Monforts, Germany) was used. The effective and total lengths and the internal diameter of the press barrel were 6.7, 14.7, and 5.6 cm, respectively. The length and diameter of the screw were 19.5 and 5.3 cm, respectively. The experimental design included SMC (range 8–14% w.b.), PS (range 20–40 rpm), and RD (range 10–14 mm) as processing parameters.

Oil Recovery

The OR was calculated considering the initial oil content in the incoming material (sesame seeds) and the residual oil content in the pressed cake. It was expressed as grams of extracted oil per gram of oil present in the incoming material × 100 (g/100 g oil). Oil contents from both sesame seeds and pressed cake were determined by extraction (10 h) using Soxhlet devices, with *n*-hexane as solvent. The solvent was removed using a rotary vacuum evaporator at 40 °C. The oil content was gravimetrically determined and expressed as weight percent on a dry basis (AOCS 2009).

Fine Solid Content in Oil

Fine solid contents (FC) in the extracted oils were calculated according to Martínez et al. (2012). Briefly, oil samples obtained from the screw-pressing treatments were centrifuged at 11,000×*g* for 30 min. The precipitated solids were recovered, washed with *n*-hexane, dried, and weighed. FC was expressed as grams of solid per 100 g of extract (oil + solid).

Oil Analyses

Chemical Quality Parameters

Free fatty acid content (FFAC), peroxide value (PV), and specific extinction coefficients (K_{232} and K_{270}) were quantified according to standard methods of AOCS (2009).

Fatty Acid Composition

Fatty acid composition was analyzed by gas chromatography according to procedures reported earlier (Maestri et al. 2015). Briefly, oil samples (0.5 g) were subjected to alkaline saponification by reflux (45 min) using 30 mL 1 N KOH in methanol. Unsaponifiable matter was extracted with *n*-hexane (3 × 30 mL). The fatty acids were converted to methyl esters (FAME) by reflux (45 min) using 50 mL 1 N H_2SO_4 in methanol and analyzed by gas chromatography (Perkin-Elmer, Shelton, CT, USA) using a fused silica capillary column (30 m × 0.25 mm i.d. × 0.25 μm film thickness) CP Wax 52 CB (Varian, Walnut Creek, CA, USA); carrier gas N_2 at 1 mL/min; split ratio 100:1; column temperature programmed from 180 °C (5 min) to 220 °C at 2 °C/min; injector and detector temperatures at 250 °C, FID. The identification of FAME was carried out by comparison of their retention times with those of reference compounds (Sigma-Aldrich, St. Louis, MO, USA).

Total Lignan Content

The total lignan content (TLC) was measured according to Bhatnagar et al. (2015). From each treatment, triplicate oil samples (0.01 g each) were dissolved in 10 mL of hexane/chloroform (7:3, *v*/*v*). After 1 min of vigorous shaking, the absorbance (288 nm) of the mixture was determined. TLC was expressed as sesamol equivalents (g/kg oil), using the following equation:

$$\mathrm{TLC} = [(A/W) \times (100/230.1)] \quad (1)$$

where *A* is the absorbance of the sample, *W* is the weight of the sample in grams, and 230.1 is the specific extinction coefficient for sesamol.

Radical Scavenging Capacity

The radical scavenging capacity (RSC) of the extracted oils was measured according to Maestri et al. (2015). A 100-mg oil aliquot was dissolved in 1 mL toluene. Each oil/toluene solution was vortexed (20 s, ambient temperature) with 3.9 mL toluene solution of the free stable DPPH (2,2-diphenyl-1-picrylhydrazyl) radical (DPPH•) at a concentration of 10^{-4} mol/L. Against a blank of pure toluene, the absorption at 515 nm was measured in 1-cm quartz cells using an UV-visible spectrophotometer (Perkin-Elmer Lambda 25, Shelton, CT, USA). RSC toward DPPH• was estimated by means of the following equation:

$$\mathrm{DPPH}\bullet_r = 1\left[\frac{\text{absorbance of control} - \text{absorbance of test sample}}{\text{absorbance of control}} \times 100\right] \quad (2)$$

where $\mathrm{DPPH}\bullet_r$ expresses the amount of the radical that remains in the medium after antioxidants present in the oil sample are depleted (Espín et al. 2000).

Oxidative Stability

Oxidative stability indexes (OSI) were determined by the Rancimat (Metrohm, Herisau, Switzerland) method (Cd 12b-92 AOCS 2009) using 3-g oil aliquots. Air flow rate was set at 20 L/h, and temperature of the heating block was maintained at

110 °C. Results corresponded to the break points in the plotted curves and were expressed as induction time (in hours).

Experimental Design and Response Surface Analysis

Response surface methodology was used to model and optimize the oil extraction conditions from sesame seeds. Experiments were planned applying a Box-Behnken design (Montgomery 2005). Three different levels were used for each of the following processing parameters: seed moisture (X_1), restriction die (X_2), and pressing speed (X_3). The evaluated responses were OR (Y_1), FC (Y_2), PV (Y_3), FFAC (Y_4), K_{232} (Y_5), K_{270} (Y_6), TLC (Y_7), OSI (Y_8), and RSC (Y_9). Quadratic polynomials were fitted to express the responses (Y_n) as a function of factors (Eq. 3), where Y is the response, β_0 is the constant term, β_i represents the coefficients of the linear parameters, X_i represents the factors, β_{ii} represents the coefficients of the quadratic parameter, β_{ij} represents the coefficients of the interaction parameters, and ε is the random error.

$$Y = \beta_0 + \sum_{i=1}^{K} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{j=1}^{k} \beta_{ij} X_i X_j + \varepsilon \qquad (3)$$

Results were analyzed by a multiple regression method. The experimental results were applied to obtain the regression models. Quality of the model fitness was evaluated by ANOVA. The fit of the model to the experimental data was given by the coefficient of determination, R^2, which explains the extent of the variance in a modeled variable that can be explained with the model. Only models with high coefficient of determination were included in this study. All determinations were performed at least in duplicate, randomly, and replicas of the central point were done to allow estimation of pure error as square sums. Two different designs were carried out: pilot-scale experiments, as preliminary assays to analyze the effect of levels of processing parameters (X_1, X_2, X_3) on the evaluated responses, and industrial-scale experiments, to define the adequate extraction conditions. Statistical analysis of data was performed using Statgraphic Plus software (v5.1, USA). For validation of the model, the response variable (OR, Y_1) was measured, in triplicate, under optimal extraction conditions, and the absolute average deviation (AAD), the bias factor (Bf), and the accuracy factor (Af) were calculated according to Desobgo et al. (2015). Finally, the percentage error between the predicted and the experimental values were calculated according to Barrera et al. (2016).

Results and Discussion

An experimental design comprising 16 treatments was carried out (4 central points) for pilot-scale extraction using the described factors and levels (Table 1). Figure 1 shows the main significant effects for OR. The three factors analyzed (SMC,

Table 1 Box-Behnken design at pilot plant scale: effects of process variables on oil recovery and quality parameters

	Factors[a]			Yield and quality parameters								
Assay	X_1	X_2	X_3	OR	FC	PV	FFAC	K_{232}	K_{270}	TLC	OSI	RSC
1	7	6	40	40.9 ± 0.4	19.9 ± 0.2	ND	0.78 ± 0.08	2.95 ± 0.38	0.61 ± 0.03	7.21 ± 0.02	9.56 ± 0.01	44.4 ± 0.8
2	7	5	20	46.5 ± 0.5	11.1 ± 0.0	ND	0.67 ± 0.01	3.33 ± 0.49	0.63 ± 0.03	6.22 ± 0.33	9.62 ± 0.21	44.4 ± 0.1
3	7	4	40	40.6 ± 0.3	22.0 ± 0.2	ND	0.82 ± 0.03	2.82 ± 0.02	0.63 ± 0.01	6.71 ± 0.20	9.63 ± 0.15	44.0 ± 0.8
4	7	5	60	33.3 ± 0.3	26.1 ± 0.1	ND	0.79 ± 0.03	2.77 ± 0.10	0.60 ± 0.01	7.02 ± 0.22	10.05 ± 0.02	43.6 ± 0.4
5	17	5	20	47.0 ± 0.4	4.7 ± 0.2	ND	0.82 ± 0.05	3.02 ± 0.41	0.72 ± 0.05	7.58 ± 0.03	10.31 ± 0.16	41.6 ± 2.8
6	17	5	60	43.6 ± 0.5	6.6 ± 0.0	ND	0.79 ± 0.13	2.67 ± 0.01	0.64 ± 0.01	7.43 ± 0.49	9.88 ± 0.03	40.5 ± 1.2
7[b]	12	5	40	58.7 ± 0.4	12.2 ± 0.0	ND	0.87 ± 0.08	2.96 ± 0.28	0.62 ± 0.01	7.29 ± 0.41	9.98 ± 0.08	41.9 ± 2.1
8[b]	12	5	40	61.2 ± 0.5	13.4 ± 0.1	ND	0.84 ± 0.07	2.96 ± 0.44	0.64 ± 0.04	7.56 ± 0.03	10.38 ± 0.15	41.0 ± 2.9
9[b]	12	5	40	57.7 ± 0.5	13.1 ± 0.0	ND	0.82 ± 0.07	2.97 ± 0.38	0.65 ± 0.02	8.08 ± 0.41	10.38 ± 0.23	39.6 ± 2.6
10[b]	12	5	40	59.4 ± 0.5	13.8 ± 0.0	ND	0.68 ± 0.01	3.08 ± 0.16	0.67 ± 0.02	7.91 ± 0.11	10.04 ± 0.18	40.6 ± 3.8
11	17	4	40	50.5 ± 0.4	4.7 ± 0.1	ND	0.98 ± 0.09	2.77 ± 0.01	0.72 ± 0.01	7.54 ± 0.66	9.42 ± 0.17	42.9 ± 3.7
12	17	6	40	44.6 ± 0.6	7.8 ± 0.1	ND	0.73 ± 0.04	2.68 ± 0.06	0.66 ± 0.02	7.79 ± 0.03	9.78 ± 0.05	38.1 ± 1.5
13	12	4	60	63.7 ± 0.4	10.3 ± 0.1	ND	0.72 ± 0.01	2.82 ± 0.17	0.68 ± 0.04	7.99 ± 0.64	10.11 ± 0.01	37.2 ± 2.1
14	12	6	60	54.1 ± 0.3	17.3 ± 0.1	ND	0.68 ± 0.02	2.73 ± 0.01	0.65 ± 0.01	7.78 ± 0.13	10.34 ± 0.13	39.4 ± 0.9
15	12	6	20	59.1 ± 0.4	10.3 ± 0.3	ND	0.63 ± 0.05	2.65 ± 0.08	0.63 ± 0.01	7.49 ± 0.29	10.1 ± 0.14	38.6 ± 0.9
16	12	4	20	74.2 ± 0.4	4.74 ± 0.0	ND	0.74 ± 0.01	2.71 ± 0.01	0.65 ± 0.01	7.57 ± 0.22	9.76 ± 0.52	36.4 ± 2.1

[a] X_1 seed moisture content (%, WB), X_2 restriction die (mm), X_3 pressing speed (rpm), *OR* oil recovery (g/100 g oil), *FC* fine solid content in oil (g solids/100 g extract), *PV* peroxide value (meq/kg oil), *FFAC* free fatty acid content (mg KOH/g oil), *TLC* total lignan content (g/kg oil), *OSI* oxidative stability index (hours), *RSC* radical scavenging capacity. Values are expressed as arithmetic mean ± standard deviation (n = 2). *ND* not detected

[b] Central points

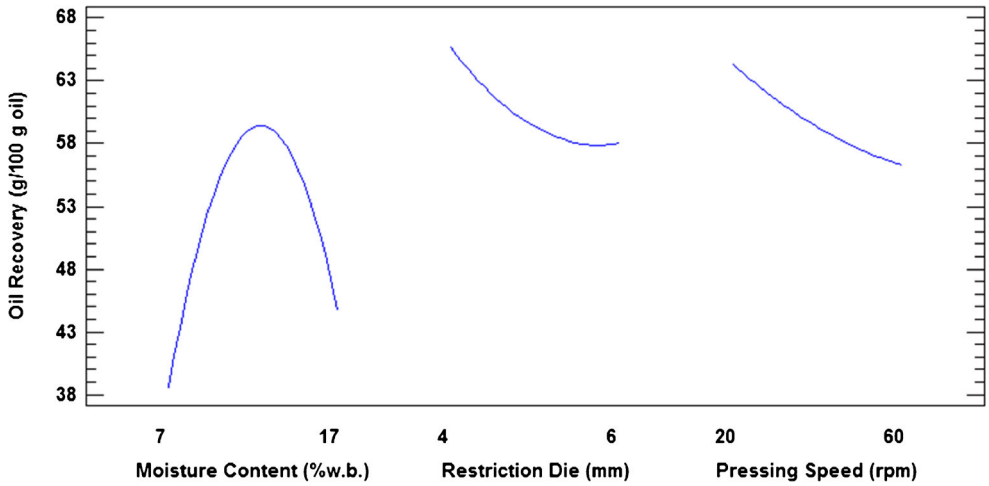

Fig. 1 Main significant effects on oil recovery in pilot-scale extractions

PS, and RD) showed p values lower than the significance level ($p \leq 0.05$) (Table 2). OR and FC in oil were between 33.3 and 74.3%, and between 4.7 and 26.1%, respectively (Table 1). Increasing SMC from 7 to 12% increased OR. Moisture content is a key factor controlling oilseed expression (Savoire et al. 2013). Water addition before pressing causes an expansion and breaking of cell structures, increases plasticity, and contributes to press feeding owing to its effects as barrel lubricant. However, high moisture contents may result in poor oil recoveries because of insufficient friction during pressing. In the present study, the highest OR was obtained by using a combination of 12% SMC, 20 rpm PS, and 4 mm RD. This treatment also showed the lowest value of FC (4.7%). At that moisture content, an increase of both PS and RD produced a significant reduction in OR.

The oils obtained at the various pilot-scale extraction conditions had TLC ranging between 6.22 and 8.08 g/kg oil. The concentration of lignans in sesame oils varies widely depending on seed pre-treatment conditions (mainly roasting temperature) and oil extraction methods. In commercial sesame oils, obtained from roasted seeds, Wu (2007) has reported an average value of 11.5 g/kg of total lignans. On the other hand, Rangkadilok et al. (2010) have found sesamin and sesamolin (the two major lignans in SO) contents varying in the ranges 0.93–2.89 g/kg and 0.30–0.74 g/kg oil, respectively. Results from the present study suggest that SO with high lignan concentration may be obtained at pressing temperatures markedly lower than temperatures usually used for roasting.

In agreement with results by Prescha et al. (2014), the specific extinction coefficients (K_{232} and K_{270} values) were found to be in the ranges 2.65–3.33 and 0.60–0.72, respectively. These values appear to be higher than those generally found in raw, cold-pressed vegetable oils (CODEX-STAN 210—1999). However, we found that SO obtained at the various extraction conditions employed did not register peroxide values (PV). Several studies have demonstrated a strong effect of sesame lignans in suppressing lipid peroxidation owing to

Table 2 Values of regression coefficients calculated for sesame oil recovery at pilot and industrial scale

Independent variable	Regression coefficient	Standard error	Significance level (p)
Pilot scale			
Constant	51.4556	1.4882	
X_1	18.1188	2.1046	0.0279
X_2	−28.0845	2.1046	0.0113
X_3	−1.0392	2.1046	0.0086
X_1^2	−0.7061	2.9765	0.0000
X_2^2	2.5237	2.9765	0.1409
X_3^2	0.0025	2.9765	0.5301
X_1*X_2	−0.3090	2.9765	0.3392
X_1*X_3	0.0245	2.9765	0.1505
X_2*X_3	0.0690	2.9765	0.3896
R^2			96.8
AAD			0.035
Bf			1.015
Af			1.109
Industrial scale			
Constant	499.132	3.2409	
X_1	−12.0166	3.9692	0.0242
X_2	−54.9604	3.9692	0.0194
X_3	−1.2757	3.9692	0.2020
X_1^2	−0.1307	5.8426	0.7037
X_2^2	1.6727	5.8426	0.0706
X_3^2	−0.0090	5.8426	0.7700
X_1*X_2	0.8796	5.6134	0.1188
X_1*X_3	0.0742	5.6134	0.4639
X_2*X_3	0.0591	5.6134	0.6910
R^2			87.2
AAD			0.036
Bf			1.036
Af			1.036

[a] X_1 seed moisture (% w.b.), X_2 restriction die (mm), X_3 pressing speed (rpm), *AAD* absolute average deviation, *Bf* bias factor, *Af* accuracy factor

Table 3 Box-Behnken design at industrial scale: effects of process variables on oil recovery and quality parameters

	Factors[a]			Yield and quality parameters								
Assay	X_1	X_2	X_3	OR	FC	PV	FFAC	K_{232}	K_{270}	TLC	OSI	RSC
1	14	10	30	43.9 ± 0.4	3.7 ± 0.0	0.18 ± 0.14	1.53 ± 0.02	2.81 ± 0.04	0.75 ± 0.04	7.11 ± 0.21	10.66 ± 0.31	32.1 ± 3.4
2	14	14	30	41.7 ± 0.4	4.0 ± 0.2	0.17 ± 0.09	1.60 ± 0.02	2.82 ± 0.04	0.74 ± 0.04	7.08 ± 0.25	10.89 ± 0.25	31.8 ± 2.9
3	14	12	40	42.3 ± 0.5	5.5 ± 0.0	0.34 ± 0.22	0.99 ± 0.01	2.87 ± 0.08	0.74 ± 0.01	7.38 ± 0.08	10.92 ± 0.15	34.8 ± 2.0
4	14	12	20	45.5 ± 0.4	3.8 ± 0.0	0.47 ± 0.03	0.97 ± 0.02	2.81 ± 0.01	0.74 ± 0.01	7.78 ± 0.11	10.3 ± 0.25	33.3 ± 0.3
5	8	12	40	41.8 ± 0.5	12.9 ± 0.1	ND	0.94 ± 0.01	2.64 ± 0.02	0.61 ± 0.01	6.71 ± 0.36	9.77 ± 0.26	45.7 ± 0.6
6[b]	11	12	30	48.2 ± 0.5	9.5 ± 0.0	ND	1.43 ± 0.03	2.72 ± 0.07	0.68 ± 0.05	7.15 ± 0.16	10.36 ± 0.15	40.5 ± 0.1
7[b]	11	12	30	43.5 ± 0.4	11.7 ± 0.4	0.16 ± 0.01	1.42 ± 0.01	3.76 ± 0.05	1.02 ± 0.01	11.1 ± 0.01	10.36 ± 0.13	40.3 ± 2.7
8[b]	11	12	30	47.7 ± 0.5	9.0 ± 0.0	ND	1.56 ± 0.01	2.69 ± 0.02	0.66 ± 0.01	6.98 ± 0.02	10.32 ± 0.12	38.3 ± 2.4
9	11	14	20	47.5 ± 0.4	9.2 ± 0.0	ND	1.67 ± 0.07	3.76 ± 0.01	1.02 ± 0.01	11.1 ± 0.13	10.79 ± 0.21	40.6 ± 3.2
10	11	10	40	60.8 ± 0.5	9.6 ± 0.1	0.21 ± 0.06	1.38 ± 0.01	3.80 ± 0.02	1.03 ± 0.01	11.1 ± 0.18	10.42 ± 0.25	42.7 ± 0.1
11	8	14	30	52.4 ± 0.5	9.0 ± 0.1	ND	0.82 ± 0.02	2.66 ± 0.10	0.61 ± 0.03	6.71 ± 0.02	9.79 ± 0.14	45.8 ± 1.4
12	11	10	20	64.0 ± 0.6	4.7 ± 0.1	ND	1.22 ± 0.01	2.71 ± 0.03	0.67 ± 0.01	7.33 ± 0.22	10.72 ± 0.12	38.6 ± 0.8
13	11	14	40	45.9 ± 0.5	10.8 ± 0.0	0.15 ± 0.02	1.67 ± 0.07	3.88 ± 0.08	1.05 ± 0.02	11.1 ± 0.19	10.14 ± 0.15	37.8 ± 2.0
14	8	10	30	75.8 ± 0.3	4.8 ± 0.2	0.11 ± 0.01	1.14 ± 0.01	2.68 ± 0.05	0.63 ± 0.02	7.13 ± 0.02	9.63 ± 0.17	38.3 ± 2.5
15	8	12	20	53.9 ± 0.4	7.3 ± 0.0	ND	0.74 ± 0.06	2.62 ± 0.01	0.60 ± 0.01	6.88 ± 0.11	9.87 ± 0.18	40.9 ± 2.4

[a] X_1 seed moisture content (%, WB), X_2 restriction die (mm), X_3 pressing speed (rpm), *OR* oil recovery (g/100 g oil), *FC* fine solid content in oil (g solids/100 g extract), *PV* peroxide value (meq/kg oil), *FFAC* free fatty acid content (mg KOH/g oil), *TLC* total lignan content (g/kg oil), *OSI* oxidative stability index (h), *RSC* radical scavenging capacity. Values are expressed as arithmetic mean ± standard deviation (n = 2). *ND* not detected

[b] Central points

their capacity of hydrogen-atom transfer to lipid alkyl radicals (Shahidi and Naczk 2004; Suja et al. 2004; Lee et al. 2010). In addition, sesame lignans have been found to display a synergistic effect on the antioxidant activity of tocopherols present in SO (Kochhar 2002). So, it is possible that all these antioxidant compounds may react rapidly with conjugated free radicals arising from poly-unsaturated fatty acids to produce stabilized conjugated dienes and trienes, thus breaking lipid chain oxidation reactions and retarding hydroperoxide formation. Other studies evaluating different oil extraction or storage conditions have reported low PV. Yoshida and Takagi (1997) have found PV ranging between 1.73 and 7.07 meq/kg in oil samples obtained from sesame seeds roasted at temperatures between 160 and 250 °C. Prescha et al. (2014) report up to 4.13 meq/kg in oil samples stored during 12 months, at 20 °C, in a 12:12 h light/dark regime. The RSC was found to range between 36.4 and 44.4%. According to Fukuda et al. (1986), Yoshida and Takagi (1997), and Lee et al. (2010), the antiradical activity of SO is mainly attributed to sesamol and tocopherol contents. The different oil extraction conditions did not affect the OSI values which were found to be in the range 9.42–10.38 h. These values were higher than those informed by Kochhar (2002) for commercial refined SO.

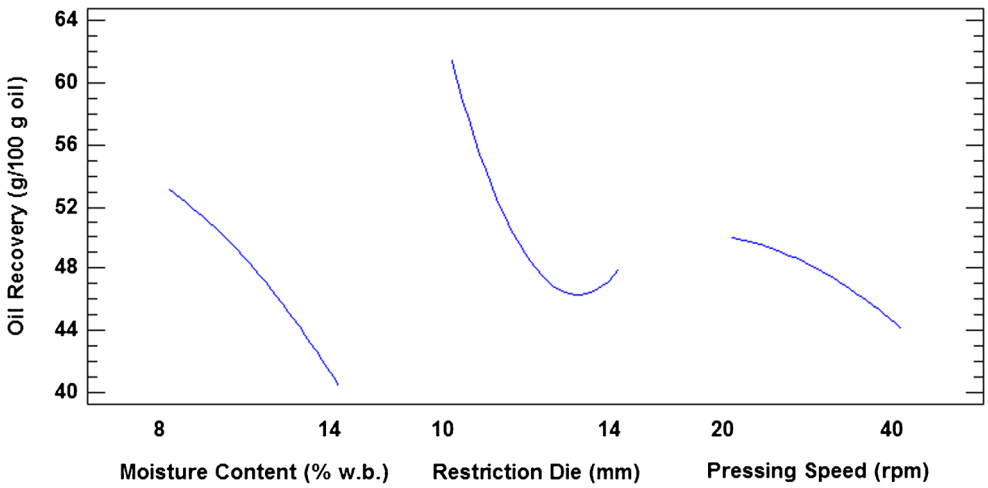

Fig. 2 Main significant effects on oil recovery in the industrial scale test

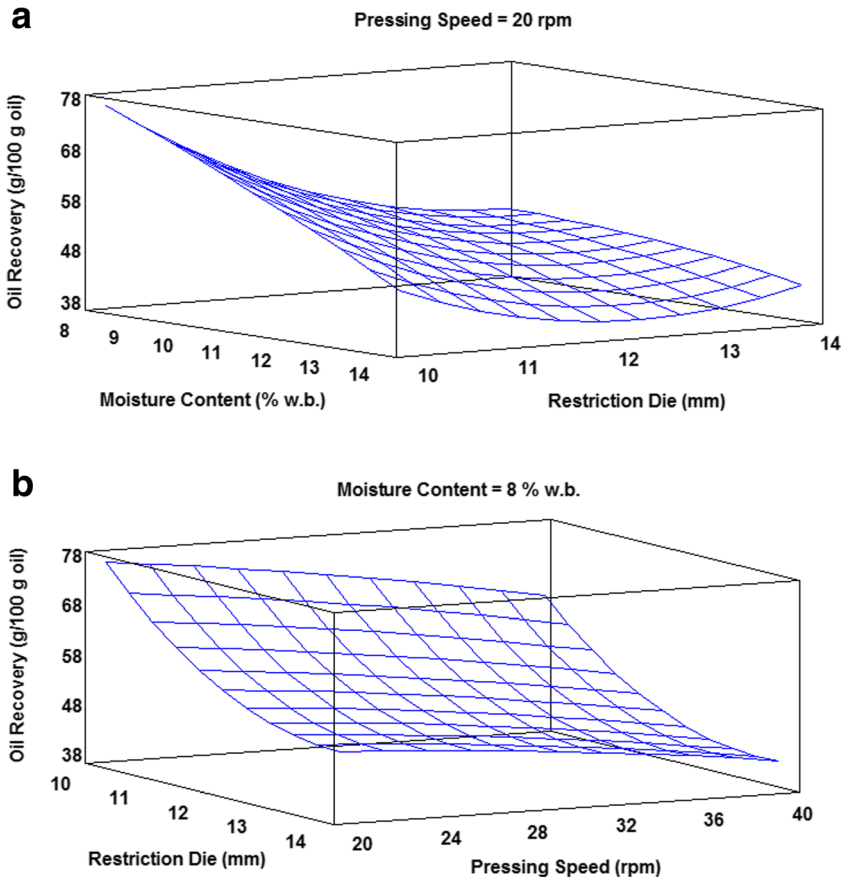

Fig. 3 Effects of seed moisture content and restriction die (**a**) and pressing speed and restriction die (**b**) on oil recovery at industrial-scale test

Oil samples obtained from the different treatments showed similar free fatty acid contents (FFAC) (Table 1). They ranged between 0.63 and 0.98 (mg KOH/g oil); these values are lower than the maximum value stated by Codex Standards (CODEX-STAN 210—1999) for cold-pressed and virgin oils. FFAC showed no significant correlation with SMC. All these data indicate that the processing conditions employed in this work do not produce hydrolytic rancidity.

To sum at this point, it can be said that the different treatments employed for sesame oil extractions at pilot plant scale had minimal effects on the chemical quality parameters analyzed, but yield parameters (OR and FC) were largely affected. A quadratic polynomial was fitted to model the oil recovery response at pilot scale. The determination coefficient of the model explained 96.8% of data variability. SMC had a positive linear and a negative quadratic effect on oil recovery. A negative linear effect for both RD and PS was also observed (Table 2). This model indicated a maximum OR at 12.3% SMC, 4 mm RD, and 20 rpm PS. The result demonstrates that the regression equation allowed a successful prediction of oil recovery. The predicted value (72.1%) and experimental value (71.1 ± 2.8%) presented an error of −1.39%, thus suggesting good prediction level of the model.

For industrial-scale extractions, an experimental design of 15 treatments was carried out (3 central points) using the described factors and levels (Table 3). Both SMC and RD, but not PS, showed p values lower than the significance level ($p \leq 0.05$) (Table 2). The values for OR and FC varied between 41.6–75.8% and 3.7–12.9%, respectively (Table 3). The highest OR was obtained at the lowest SMC (8%) (Fig. 2). Similar results were reported for several seed materials including flaked and cooked soybean (moisture content range 7.5–12%), uncooked canola seed (5.7–9.1%), cooked and uncooked crambe seed (3.5–8.4%), flaxseed (6.1–12.1%), and chia seed (10–12%) (Singh et al. 2002, 2012; Martínez et al. 2012). Regarding SO pressing-extraction, Akinoso et al. (2010) obtained the highest OR (50.4%) from seeds (4.6% moisture content) roasted at 124 °C. Recently, Rostami et al. (2014) reported that oil-pressed cakes had minimum retained oil content when sesame seeds (6.5% moisture content) were extracted at 75 °C pressing temperature. A strong decrease in OR was found as RD increased in the range 10–14 mm (Figs. 2 and 3). This may be attributed to insufficient compression of seed material during pressing. On the contrary, PS had a minor effect on OR (Figs. 2 and 3).

Oil extractions at industrial scale showed maximum OR values (75.8%) under the following conditions: 8% SMC, PS 30 rpm, and RD 10 mm. Furthermore, these conditions gave oils with the lowest FC (4.8%) and quality parameters similar to those obtained at pilot plant scale conditions. The

Table 4 Chemical parameters of oils extracted under processing conditions (industrial scale) for maximum oil recovery

Oil parameters		Codex range[a]
PV	0.10 ± 0.01	<10.0
FFAC	1.12 ± 0.01	<4.0
K_{232}	2.71 ± 0.11	NI
K_{270}	0.67 ± 0.02	NI
Iodine value	118	104–120
TLC	7.21 ± 0.55	NI
OSI	9.60 ± 0.87	NI
RSC	38.1 ± 2.2	NI
Main fatty acids (%)		
Palmitic	10.5 ± 1.3	7.2–12.0
Palmitoleic	ND	<0.5
Stearic	4.9 ± 0.2	3.5–6.0
Oleic	41.2 ± 2.2	35.5–50.0
Linoleic	44.2 ± 1.7	35.5–50.0
Linolenic	0.4 ± 0.0	<1.0
Arachidic	ND	<1.0
Behenic	ND	<0.5

PV peroxide value (meq/kg oil), *FFAC* free fatty acid content (mg KOH/g oil), *TLC* total lignan content (g/kg oil), *OSI* oxidative stability index (h, Rancimat, 110 °C), *RSC* radical scavenging capacity, *ND* not detected, *NI* not informed

[a] Codex standards for fats and oils from vegetable sources (CODEX-STAN 210—1999), refined sesame oil

PI, AV, and the relative abundance of the main fatty acids were in concordance with those stated by Codex Standards (CODEX-STAN 210—1999) (Table 4).

A new quadratic polynomial was fitted to model the oil recovery response at industrial scale. The determination coefficient of the model explained 87.2% of data variability. SMC (X_1), RD (X_2), and PS (X_3) had a negative linear effect on OR. In addition, X_1 and X_2 had a negative and positive quadratic effect, respectively (Table 2). The combination of factors that suggested the maximum oil recovery was 8.03% SMC, 10 mm RD, and 20 rpm PS. The result demonstrates that the regression equation allowed a successful prediction of oil recovery. The predicted value (77.1%) and experimental value (74.4 ± 1.2%) presented an error of −3.5%, thus suggesting good prediction level of the model.

The evaluation of real performance of predictive models in complex systems was done according to Desobgo et al. (2015). These authors have proposed the calculation of the AAD, Bf, and Af parameters, which were measures of the relative average deviation of predicted and observed responses. A perfect agreement between observed and predicted responses is related to Bf and Af values of 1 and AAD of 0. The correlation coefficients associated with AAD, Bf, and Af values allowed validation of the models, as shown in Table 2.

Conclusions

Box-Behnken designs were used to optimize a process for sesame oil extraction by screw-pressing at low temperature (50 °C). Experimental designs included seed moisture content (SMC), pressing speed (PS), and restriction die (RD) as the main processing parameters. Extractions at pilot plant scale showed a peak in oil recovery (OR, 71.1 ± 2.8%) at 12.3% SMC, 4 mm RD, and 20 rpm PS. Theoretical models were scanned against experimental data in order to scale up the proposed oil extraction process to industrial scale. A fitted model for oil recovery showed a maximum predicted value similar to the highest experimental value (74.4 ± 1.2%) under the following conditions: 8.03% SMC, 10 mm RD, and 20 rpm PS. Chemical quality parameters of oils obtained at both pilot and industrial scales were in the ranges stated in Codex (FAO/WHO) standards for non-refined sesame oil.

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