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Optimizing the extraction process of sesame seed's oil using response surface method on the industrial scale

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ABSTRACT

In the present study, some chemical parameters of remained oil contents of the pressed cakes obtained of local sesame seed's oil extraction and also from different stages of extraction process were determined. To measure these parameters of extracted oil, three diverse heating temperatures (75, 90 and 105 °C) with three states of moisture contents (4.5%, 5.5% and 6.5%) for output seeds were designed. Analysis's results of obtained data by response surface method demonstrated that an increase in given temperature, increases oil content in pressed cakes and also insoluble fine partial content of the final extracted oil, while decreases protein's content in the meals. Increasing moisture content of output seeds of heating cabinet, indicated decrease in oil content of the meals. According to the results of optimizing process, it can be stated, heating process by temperature 75 °C and moisture content of output seeds in confine of 6.3–6.5%, are high yielded combined parameters that may result oils with high quality and byproducts with the minimum remained oil content.

1. Introduction

Sesame (*Sesamum indicum* L.) from Pedaliaceae, is a very common oil seed crop being cultivated in the tropical and high temperate regions of the world (Biabani and Pakniyat, 2008). This oil is main product of India, Sudan, China and Burma (60% of produced sesame oil in the world) (ElKhier et al., 2008). That is the most traditional edible oil crops which has been using in mankind cases and has widely been cultivated in Asia and Africa (Ali et al., 2007). This healthful product, with annual production of 760,000 million tons in 2003, had 12th rank as largest vegetable oil products used in the world, which is more higher in quantity than olive and safflower oils products (ElKhier et al., 2008). The most significant property of sesame oil is resistance to oxidative deterioration (Manley et al., 1974). Sesame seed oil has been using in a huge spectrum for cooking, bakery process and producing margarine. Also sesame seeds are used mainly for preparing tehina (sesame paste product similar to peanut butter) and Halva (a most public traditional food which is available on Iranian dishes) (Abou-Gharbia et al., 1997; Sankar et al., 2005). The analysis of sesame's composition cited that this worthy product is a very rich source of oil (44–58%), protein (18–25%) and carbohydrates (~13.5%) (Kamal-Eldin and Appelqvist, 1994; Mohammed et al., 2011). Sesame seeds due to involving oil with high quantity and quality are named queen of the oil seeds for mankind usages (Brar, 2008).

The current mode of sesame seed oil extraction at the traditional level is briefly, (1) pounding the seeds in a mortar and (2) pouring hot water into the mortar causing the oil to float on the surface, from where it is skimmed off. This method of extraction is, however, very slow and low yielded (Majdi et al., 2007). Also oil can be produced by most natural methods, chemical materials or additives which are not used commonly. The finest oil extraction mode of sesame seeds is mechanical cold press (at a low temperature as such lower than 45 °C), which filtration of the fluid will improve the quality. Free fatty acid contents and peroxide values are common parameters for determination of the oils quality (ElKhier et al., 2008). The conventional process for sesame oil extraction includes: (1) cleaning, (2) dehulling, (3) roasting, (4) grinding and (5) oil extraction, respectively (Fukuda and

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Namiki, 1988). Roasting is a critical stage which influences color, composition, and organoleptic qualities of the extracted oils as well as oxidative stability (Yen and Shyu, 1989). In one study, observed results demonstrated that with increasing temperature given to canola seeds, non-triglyceride component contents and acidity of the oil were increased (Prior et al., 1991). Other similar study cited that antioxidative activities of defatted sesame meals, increased with an enhance in temperature of roasting (Jeong et al., 2004). Scientists expressed that increasing in stirring temperature, leads to oil with high acidified contents (Boselli et al., 2009). Enhancing in temperature of roasting and moisture content of seeds, diminish oil contents of the meals (Iezadikha et al., 2011). Zhang et al. applied RSM for optimization of Zanthoxylum Bungeanum seed oil transesterification to biodiesel interactions by using canon as a catalyst (Zhang et al., 2010). Optimization of transesterification variables for biodiesel production from cottonseed oil using RSM has also been reported by (Fan et al., 2011). Due to the deficiency of matters in the field of hot-press method for extraction oil of sesame seeds in industrial scale, the main purpose of this investigation is optimization of given temperatures and moisture contents of output seeds of oven, to obtain oil and meal with high quality.

2. Materials and methods

Sesame seeds have bought from Afghanistan for oil extraction operations and chemical experiments, then were transferred to Khorasan cotton and oil seeds factory in north east of Iran.

2.1. Oil extraction mode

Sesame seeds, were stored into Kendo shape silos in dark and room temperature for a while after receiving and extra particles and impurities such as dust, sands, stones, spoiled seeds, small weed seeds and other extra materials were separated by mechanical sieves and were kept by extraction (Ohlson, 1976). Afterward, seeds were transferred into the roasting cabinet and temperature and humidity of the roasting chamber's perimeter were set on 75 °C, 90 °C, 105 °C and 5.5%, 6% and 6.5%, respectively. Roasted seeds were then placed into the screw press apparatus and pressed up to liberate their oil contents. Output pressed cakes were transferred to the next stage for extraction by solvent. After extraction, solvent was separated from micelle (mixture of oil–solvent and meals) by debugger equipment (desolventize-toast).

2.2. Moisture content

Moisture content of the oil at 105 ± 1 °C was determined according to the Eq. (1).

$$\text{Percentage of moisture content} = \frac{W_1 - W_2}{M} \times 100 \quad (1)$$

where W_1 is the initial weight of the vessel with sample before drying, W_2 represents the weight of the vessel and sample after drying and M is the pure sample weight (AOAC, 2008).

2.3. Determination of fat content

Soxhlet apparatus and *n*-hexane (as solvent) were used for oil extraction. Initially, sample with specific weight was put in the thimble and then both were placed inside the apparatus, round bottom flask containing a specific volume of *n*-hexane was fixed, afterward a condenser was tightly connected to the bottom end. The whole set up was heated up by the temperature of 70 °C and oil has been extracted. Additional solvent in the extracted oil was separated by distillation (AOAC, 2008).

2.4. Determination of protein content

Total nitrogen content has been determined by the Kjeldahl assay. Then, protein content was calculated by multiplying obtained total nitrogen content explained previously by 6.25 (GF) (Khalid et al., 2003).

2.5. Determination of insoluble fine partial content

To measure the amount of insoluble fine partial content, 10 ml of the oil was transferred into a eppendorf tube and was centrifuged with 4000 rpm speed for 10 min. Afterward, the percentage of the insoluble fine partial contents was expressed by Eq. (2) (AOAC, 2008).

$$\text{Fine partial percentage} = \text{Mass of the sediment} \times 10 \quad (2)$$

2.6. Experimental design for optimization

RSM (Response Surface Method) was used to optimize the conditions of oil extraction for sesame seeds. A face-centered cube design (FCD) consisting of 13 experimental runs, including three replications at the center point, was selected to evaluate the combined trace of the dependent variables (remained oil, protein and moisture contents in meal, oil insoluble fine partials in obtained oil and oil content in the output cakes). The domain and the center point values of the three independent variables were adjusted on the base of obtained results of moisture contents in the preliminary test for output seeds of toaster (4.5–6.5%) and temperature (75–105 ± 1 °C) before extraction procedure. The tests were performed in random order to diminish the side effects of unexplained variability in the observed responses due to systematic errors. The independent variables involve temperature (x_1, ±1 °C), moisture content of output seeds after roasting and before extraction (x_2, %), while the response (dependent) variables were determined, remained oil (y_1 and y_2, respectively), protein and moisture contents in the meals (y_3 and y_4, respectively), insoluble fine partials in the extracted oil and oil contents in the pressed cakes (y_5). The response functions (y_1–y_5) were involved into linear, quadratic, and interactive components. Experimental data were fitted with quadratic regression according to Eq. (3):

$$y = b_0 + b_1x_1 + b_2x_2 + b_{11}x_1^2 + b_{22}x_2^2 + b_{12}x_1x_2 \quad (3)$$

where y is the predicted response, b_0 is the intercept; b_1 and b_2 are linear coefficients; b_{11} and b_{22} are squared coefficients; b_{12} is the interaction coefficient; and x_1 and x_2 are the coded levels of variables (X_1 and X_2). The Design-Expert 6.0 (Stat-Ease Inc., Minneapolis, MN, USA) was used to determine the analysis of variance (ANOVA) and the coefficient of determination (R^2 and adjusted R^2) to evaluate the quality of fitness of the model.

3. Results and discussions

3.1. Remained oil content of the pressed cake

The obtained results of oil content of the pressed cakes with constant increase in temperature and moisture content of output seeds of roaster, have obviously demonstrated that an increase in the moisture content of output seeds from 4.5% to 6.5%, can diminish the extracted oil content of the seeds (Fig. 1). Unlikely an increase in temperature showed an enhance in extracted oil of the seeds. Increasing the roasting temperature from 75 to 105 °C and moisture content from 4.5 to 6.5% can result to an increase in the oil content of pressed cakes due to an increase in the temperature with increase of the moisture can be attributed to the destruction of cell walls of the seeds and creating a substance like a sticky plastic material

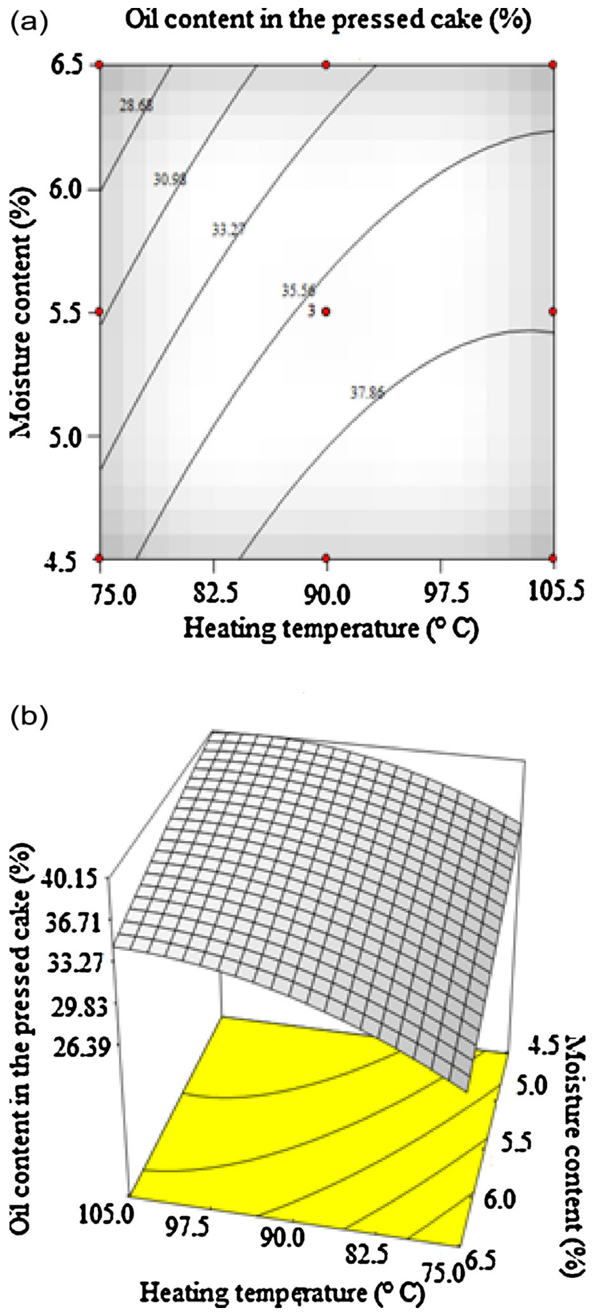

Fig. 1. (a) Linear design variation of oil content in the pressed cakes in diverse temperatures (°C) and moisture content of output seeds (%). (b) Response surface diagram of the oil content of the pressed cake (%).

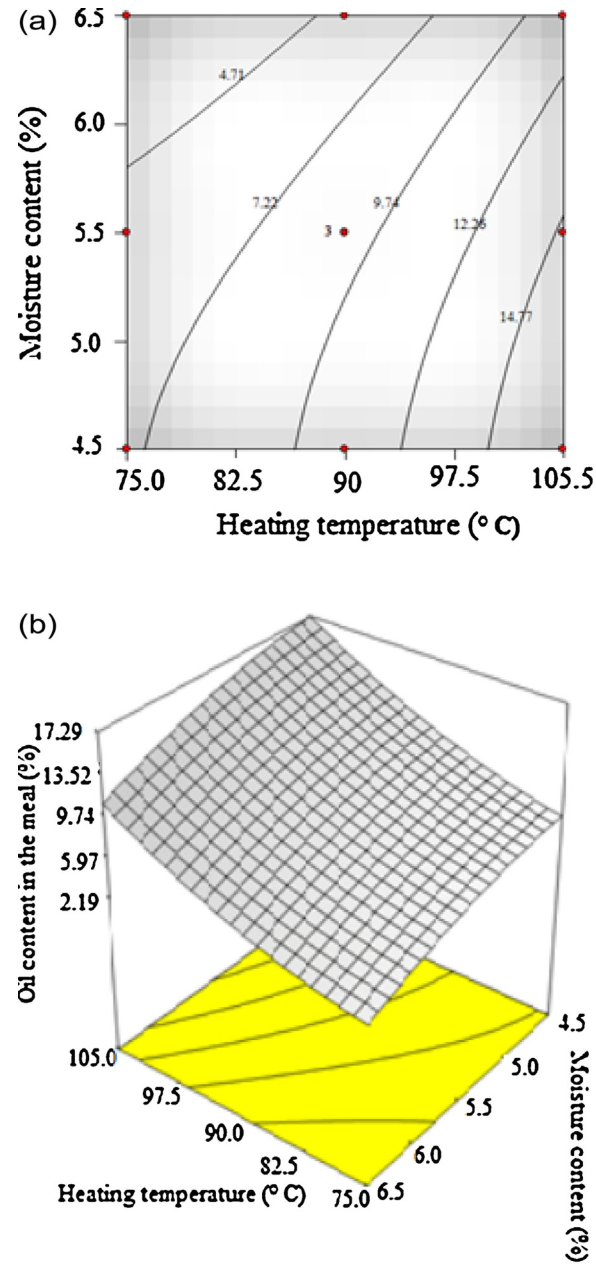

Fig. 2. (a) Linear design variation of oil content of the meal in diverse temperatures (±1 °C)(%)and moisture content of the output seeds (%). (b) Response surface diagram of the oil content of the meal (%).

that can diminish the performance of mechanical press and as a result diminish extraction efficiency. The minimum oil content of the pressed cake has been observed on temperature 75 °C and moisture 6.5% that was presented in Fig. 1 as well. So variation in the moisture content and temperature as nonlinear and linear parameters have shown a critical role on the efficiency of oil extraction of sesame seeds (Tables 1 and 2).

3.2. Variations of oil content in the meal

Fig. 2 indicates response surface of variations of oil in the meal concerning to the variations in the moisture content of output seeds and degree of roasting temperature. The results have demonstrated that the mutual trace of cooking temperature and moisture content of output seeds do not demonstrate significant effect on the oil content of the meals ($P > 0.05$). The most significant effect on the final model of oil content of meals has been related to the variations in the cooking temperature ($p < 0.05$). Obtained results have indicated that a decrease in the temperature with high moisture content of the output seeds, diminishes oil contents in the meals, progressively, and a decrease in the temperature in low moisture content in the output seeds, diminishes the oil content of the meals linearly. An increase in temperature, because of decreasing of solvent viscosity and increasing of kinetic energy may increase the diffusion rate of the solvent into seed's cells and therefore enhances the rate of oil extraction. High temperature can decompose cell walls and so the target compounds are more available, as a result of that, this procedure enhances the extraction rate and diminishes the oil contents in the meals (Kahyaoglu and Kaya, 2006; Pan et al., 2000). An

Table 1
Model selection for dependent (response) variables.

Source		Intercept	Linear	Polynomial	Quadratic	Interactive component	Residue	Total
Oil content in the pressed cake	Sum of squares	13,414.9	138.69	2.20	10.81	0.18	0.02	13,566.77
	Pb>F		<0.001	0.28	<0.001	0.03		
Oil content in the meal	Sum of squares	929.813	182.54	0.59	5.14	0.20	0.49	1118.771
	Pb>F		<0.001	0.43	0.005	0.60		
Protein content in the meal	Sum of squares	6016.92	192.72	1.28	5.73	1.08	0.7	6218.442
	Pb>F		<0.001	0.31	0.0275	0.24		
Moisture content in the meal	Sum of squares	1132.91	18.620	0.09	0.18	0.02	0.025	1151.85
	Pb>F		<0.001	0.14	0.02	0.41		
Insoluble fine partial content	Sum of squares	700.804	255.91	7.91	12.59	0.70	0.323	978.26
	Pb>F		<0.001	0.08	0.002	0.18		

Table 2
Analysis of variance for determined parameters in pressed cakes, meals and extracted oil.

		Oil content in pressed cake		Oil content in the meal		Protein content in the meal		Moisture content in the meal		Insoluble fine partial content in extracted oil	
Source	DF	Sum of squares	Pb>F	Sum of squares	Pb>F	Sum of squares	Pb>F	Sum of squares	Pb>F	Sum of squares	Pb>F
Model	5	151.70	<0.001	188.27	<0.001	199.73	<0.001	18.90	<0.001	276.43	<0.001
A	1	70.84	<0.001	135.69	<0.001	182.23	<0.001	15.04	<0.001	195.32	<0.001
B	S1	67.85	<0.001	46.85	<0.001	10.49	0.003	3.58	<0.001	60.59	<0.001
A^2	1	9.13	<0.001	4.20	0.003	3.85	0.02	0.17	0.01	8.57	0.001
B^2	1	–	–	2.19	0.010	0.24	–	–	–	–	–
–											
AB	1	2.2	0.0007	–	–	–	–	0.09	0.02	7.93	0.002
Residual	5	0.20		0.69		1.78		0.04		1.03	
Lack of fit	3	0.18	0.1441	0.60	0.183	1.34	0.35	0.02	0.60	0.74	0.39
Pure error	2	0.02		0.09		0.45		0.02		0.29	
Cor total	10	151.91		188.96		201.52		18.94		277.46	
R^2			0.999		0.996		0.991		0.998		0.996
Adj-R^2			0.997		0.993		0.982		0.995		0.993
cv			0.58		4.04		2.55		0.92		5.68

increase in the moisture content of the output seeds (from 4.5 to 6.5) in all temperatures has obviously been proved an decrease in the oil contents of the meals.

3.3. Variations of protein content of the meal

Protein and moisture contents are the two main factors which sesame meal quality is based on those two factors which are also the main parameters in commercial field of the obtained meal. However, for most applications the "true protein content versus the available protein" is a critical factor. Protein content of meal is related to the mutual effect of roasting temperature and moisture content of output seeds. Fig. 3 presents as well that an increase in the moisture content of output seeds of 4.5–6.5% can cause a decrease in the protein contents of the meal, but the tensity of the reduction is not significant and this reduction was shown in all of the given temperatures. The variations of temperature from 75 to 105 °C presented a significant trace on the protein content of meal. With a decrease on temperature from 105 to 75 °C protein content of the meal indicated a significant increase. Presented equations in Table 3 obviously represent the relative variations of proteins content of the meals to linear and quadratic parameters for given temperatures and the moisture content of the output seeds.

3.4. Variations of moisture content of the meal

Low moisture contents of the meal may assist to diminish the microbial attacks, despite high moisture content results to a diluted meal value. The moisture content of meal must be adjusted on the standard confine. An increase in the moisture content of output seeds from 4.5% to 6.5% and roasting temperature of 105 °C did not show significant trace on the moisture content of the meals. An quantized augment of temperature in the low temperatures of the roasting process of the seeds causes more tangible reduction in moisture content of the meal than high temperature. So an increase in temperature and all moisture confines can cause diminishing in the final moisture content of the meal, therefore the minimum moisture content (4.5%) has been indicated in 105 °C. This phenomena is attributed by more leaking of moisture contents of the cells through high temperature. The trend of variations of moisture content of the meal has been presented by means of model 4 from Table 3. The greater trace of the model is attributed to linear parameters and minimum trace is attributed to the quadratic ones of roasting temperature process. Fig. 4 demonstrates the result.

3.5. Insoluble fine partial content of the final extracted oil

The obtained results of insoluble fine partial content of the extracted oil is presented in Fig. 5. Insoluble fine partials contents must be separated to acquire oil with high quality. This process has been performed in the deposit tank, this means that the insoluble fine partial contents are deposited at the bottom and removed. An increase in moisture content of the output seeds and high roasting temperature have presented a decrease in insoluble fine partial content of obtained extracted oil, but an increase of moisture content at low temperature has not indicated salient trace on the insoluble fine partial content of obtained oil. Low temperature has minimum destruction trace on seeds cell, so less insoluble fine partials can leak out cells with oils. Leaking of intracellular materials into the oil at less moisture content of output seeds has been shown more significant role. According to Fig. 5, minimum insoluble fine partial contents has been acquired from sesame seeds which have been roasted at 75 °C and the moisture content of 6.5%. In the model presented in Table 3, the greatest trace on the variations of insoluble fine partial content of the extracted oil is related to linear parameters of roasting temperature and moisture content of output seeds. These results demonstrated that the quadratic parameters of

Table 3
Designed equation models for dependent variable.

Number	Dependent variable %	Equation
1	Oil content in the pressed cake	$y_1 = -18.32 + 1.48x_1 - 4.75x_2 - 0.01x_1^2 + 0.05x_1 \cdot x_2$
2	Oil content in the meal	$y_2 = 1.44 - 0.57x_1 + 9.72x_2 + 0.06x - 1^2 - 0.93x_2^2$
3	Protein content in the meal	$y_3 = -13.55 + 0.83x_1 + 7.58x_2 - 0.005x_1^2$
4	Moisture content in the meal	$y_4 = 15.54 - 0.26x_1 + 3.21x_2 + 0.001x_1^2 - 0.01x_1 \cdot x_2$
5	Insoluble particles of the extracted oil	$y_5 = 31.44 - 0.57x_1 - 2.68x_2 + 0.01x_1^2 - 0.09x_1 \cdot x_2$

moisture content in the output seeds of heating, did not show significant difference on the insoluble fine partial contents of the extracted oils, so they are removed from final offered model ($p > 0.05$).

3.6. Optimizing the process of oil extraction from sesame seeds

The final optimizing process of oil extraction from sesame seeds with concerning to roasting temperature and moisture content of output seeds from heating pot as independent variables (temperature of 75–105 °C and moisture content of 4.5–6.5%, respectively) has been performed by means of response surface method to investigate on minimum remained oil content in the pressed cake, minimum oil content of the meal, reduction of insoluble fine partial content in final extracted oil and maximum protein content of meal. It was resulted heating temperature of 75 °C and adjusted moisture content of output seeds in the confine of 6.3–6.5% can indicate to obtain products with high quality. Suggested parameters for modeling with efficiency alloy of 93% and 96% have cited the reliability of offered selected parameters. The results are presented in Table 4.

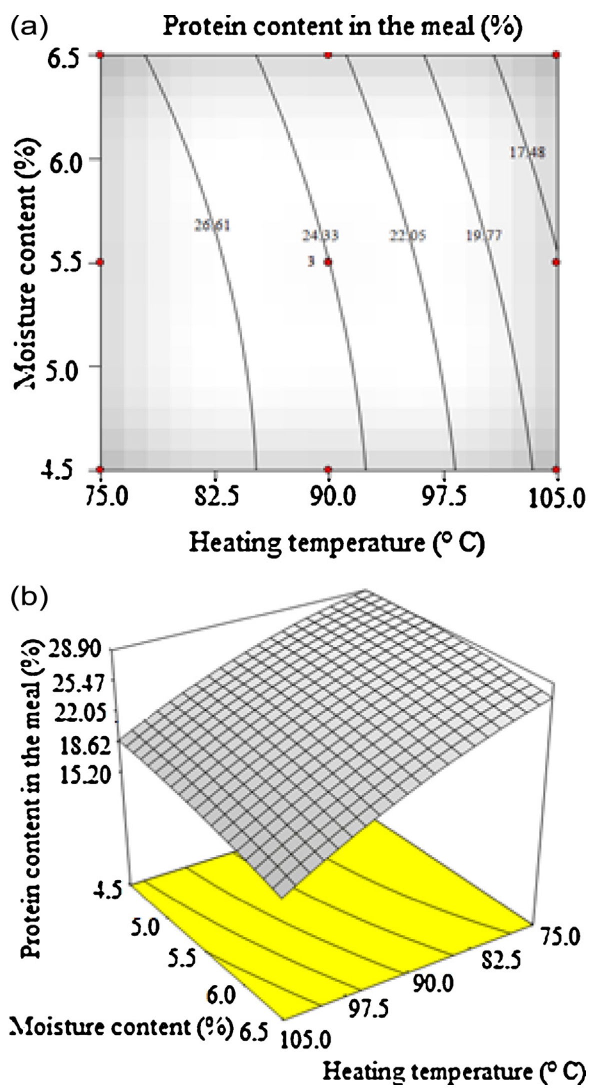

Fig. 3. (a) Linear design variation of protein content of the meal in diverse temperatures (±1 °C) and moisture contents of the output seeds of heating (%). (b) Response surface diagram in the protein content of the meals (%).

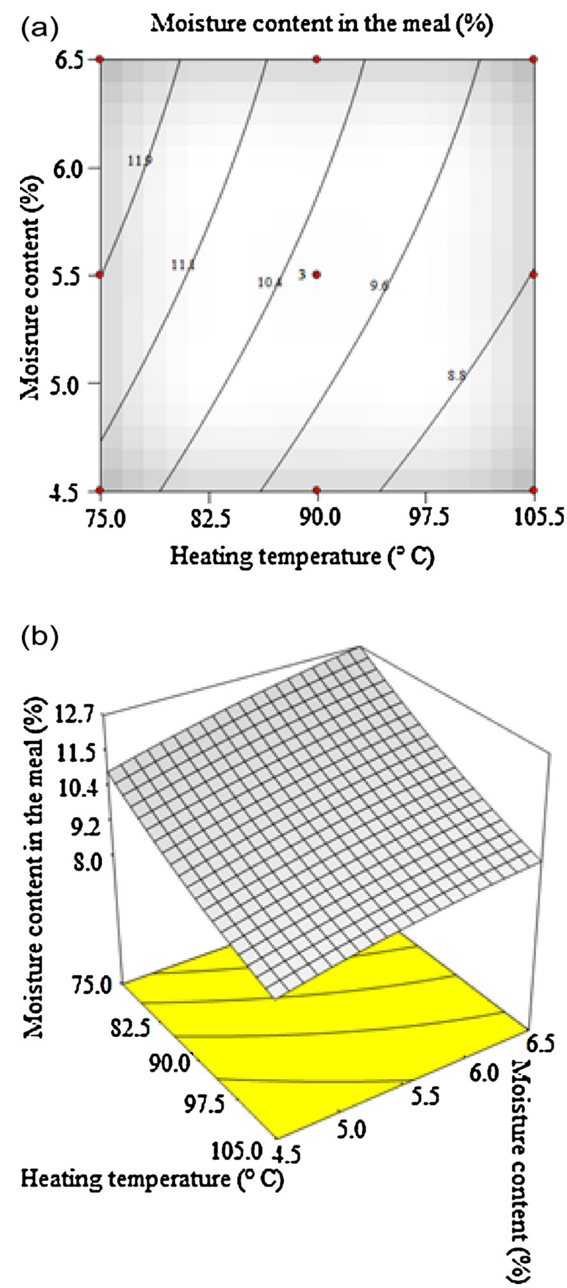

Fig. 4. (a) Linear design variation of moisture content of the meal in diverse temperatures (°C) and moisture contents of output seeds of heating (%). (b) Response surface diagram in moisture content of the meal (%).

Table 4
Optimized conditions.

Number	Temperature (°C)	Moisture content (%)	Oil content in the pressed cake	Oil content in the meal (%)	Protein contents in the meal (%)	Insoluble fine partial content in the extracted oil (%)	Desirability
1	75.0	6.5	26.4	2.2	27.36	1.7	0.963
2	75.0	6.3	27.46	3.13	27.74	1.8	0.936

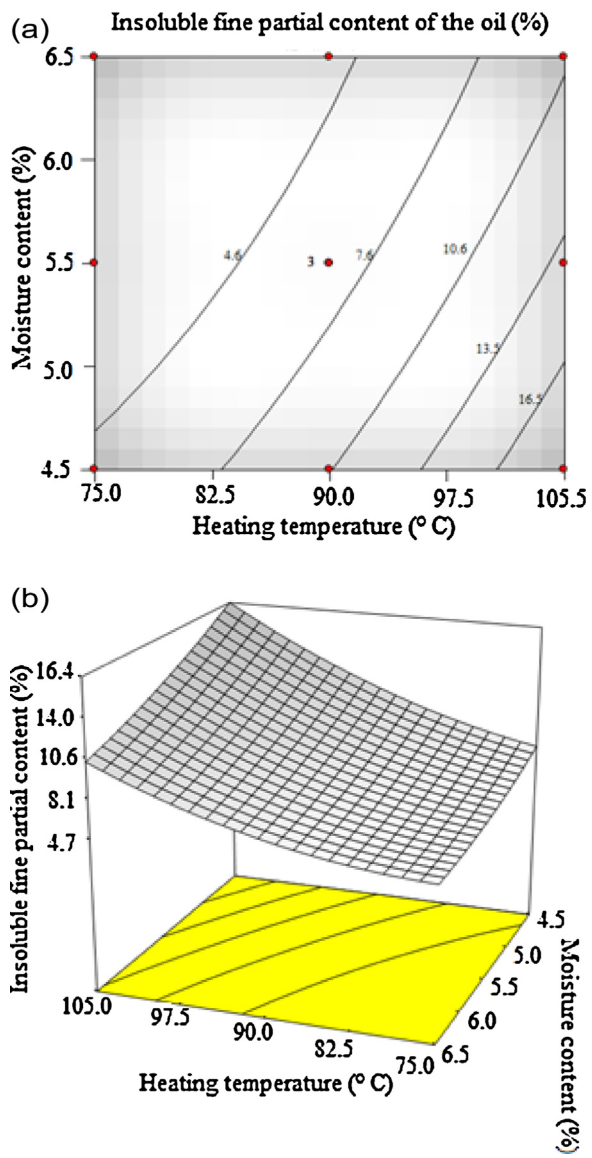

Fig. 5. (a) Linear design variations of insoluble fine partial content of extracted oil in diverse temperatures (°C) and moisture contents of output seeds of heating (%). (b) Response surface diagram for insoluble fine partial content of the extracted oil (%).

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