Croatian Journal of Food Science and Technology
journal homepage: www.ptfos.unios.hr/cjfst/

Original scientific paper
DOI: 10.17508/CJFST.2019.11.2.10

Optimization of the operating variables for the extraction of soy oil in a single screw expeller using a central composite design (CCD)

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ARTICLE INFO

Article history:
Received: September 19, 2018
Accepted: June 13, 2019

Keywords:
expeller
optimization
pressure
soybean
toasting time

ABSTRACT

Steam pressure (P) and toasting time (T) were considered under the Central Composite Design (CCD) for the optimization of oil and cake yield, as well as trypsin inhibitor and phosphorus content removal from soybean during the extraction process in the expeller machine. The soy oil was characterized for its acid, iodine, and peroxide values. The obtained optimized experimental condition [Run 11 (2.50 kg/ms^2 and 60.00 min)] resulted in 80%, 13 L/100 kg, 0.266 mg/g, and 0.39 mg/g cake yield, oil yield, trypsin inhibitor (TI), and phosphorus concentrations (PC), respectively. The correlation coefficients (R^2) of the model equation developed for cake yield, oil yield, TI concentrations, and PC were 0.9922, 0.9545, 0.9747, and 0.6771, respectively. The peroxide, acid, and iodine values of the soy oil extracted were 18 meq O_2/kg oil, 1.60 mg KOH/g oil, and 60.40 g/100 g, respectively. The optimisation tools facilitated the efficiency of the expeller in generating better products.

Introduction

Soybean is sought after, uniquely, for its oil and cake, which are of high economic importance in the world (Lui, 2014). Soybean is a vital source of plant protein and food energy in human being and livestock nutrition (Adeyeye et al., 2012). Soy oil is used commonly as the one of the best cooking vegetable oils and as an essential ingredient in several food products in the food industry, due to its high level of unsaturation. Essentially, soy oil contains three fatty acids, such as acids linoleic, linolenic, and arachidonic acid. Its fatty acid composition contains alpha, beta, and gamma tocopherols, which are natural anti-oxidants. The absence of cholesterol in the oil has made it more attractive and it is not easily affected by environmental factors. Soybean contains about 40% of protein, on a dry basis, and this is dispensed majorly in its meal or cake. The soy protein is particularly valuable because of its amino acid, lysine, tryptophan, and thrionine composition, which enriches the protein quality in human and livestock diets (Murphy et al., 2011).

Oils are generally obtained from oilseeds, through various processes, such as hydraulic screw press (double screw extraction and single screw extraction), supercritical fluid extraction, solvent-solvent extraction, and enzyme-assisted extraction (Bair et al., 2013; Araromi et al., 2017). The main methods generally used for oil extraction include batch hydraulic pressing, continuous mechanical pressing (expeller), and solvent extraction. The yield and quality of the extracted oil and the resulting cake vary with the choice of the extraction method; however, cost, health, and environmental impact play important roles. The method employed for oil seed extraction depends on the type of seed, the seed characteristics, and the oil content of the seed (Singh and Bargale,

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2000; Lawson et al., 2010). Seeds such as soybean, with low oil content, are extracted better using solvent. The advantages of solvent extraction over other methods is linked to higher oil yield, larger processing capacity, superior bleaching quality, among others. Care is often given to the extraction processes selected for soy oil extraction and cake production in terms of product quality and the removal of undesirable elements. Thus, oil and cake produced through the solvent extraction method, by most industries, cannot be used conveniently for direct human consumption and livestock feed, because of the presence of trace elements that could be carcinogenic to human health and livestock (Roshan et al., 2012).

Mechanical pressing has been considered more efficient than solvent extraction in oil extraction (Guarienti et al., 2012)**.** Mechanical pressing is fast replacing hydraulic presses, which is due to the initial conditions of the grains, such as moisture content, temperature, and the constructive aspects (design axis and the camera) of the press. The incorporation of other unit operation processes to assist the designed process may further improve the value of phosphorous and the anti-nutritional factor (anti-trypsin) of the cake products which are higher than the recommended standard**.** This implies that a standard method of extraction is required for better cake and oil production.

The twin-screw press developed by Isobe et al. (1992) has been used successfully to recover about 93% oil from untreated dehulled sunflower seeds, while the horizontal expeller used by Akerele and Ejiko (2015) to express oil from groundnut recorded about a 72.94% extraction efficiency. The horizontal screw press developed by Adetola et al. (2014) for palm oil extraction provided a 79.56% oil extraction efficiency. Some studies have verified the effects of some process factors on various expellers for various oilseeds. Tunde-Akintunde et al. (2001) reported the influence of temperature (70-80 °C) and heating time (15-30 min) on the increased oil yield for mechanically expressed soybean oil. High oil yield obtained from palm kernels using a mechanical extraction process with 47.0% and 64% extraction efficiency was reported by Akinoso et al. (2006) and Ajibola (1989).

The focus of optimizing mechanical pressing processes is to increase oil yield from oilseeds. Typical screw press performance is affected by factors such as pressing temperature, restriction diameter, pressing capacity, oil flow, oil yield, and/or residual oil content in the oilcake (Jacobsen and Backer, 1986; Ferchau, 2000). Second-order model (response surface) designs, which employ more than two factor levels to allow fitting of a full quadratic polynomial, can be employed to optimize a pressing process in order to locate the optimum set of experimental conditions of the screw press. In this study, a Central Composite Design (CCD) under the Response Surface Methodology (RSM) was applied to investigate the effects of the pressure of super-heated steam (P) and toaster time (T) on the soybean cake and oil yields using screw press extraction.

Materials and methods

Materials and reagents

The soybean sample used in this experiment was bought from the open market, Benue State, Nigeria. The seed samples were identified and authenticated at the Forestry Research Institute of Nigeria (FRIN) Jericho, Ibadan, Nigeria. The reagents used include isopropyl alcohol (IPA 99%), a phenolphthalein indicator, an acetic acid-chloroform solution, a potassium iodide solution (KI Saturated), a starch indicator solution, the Wijis solution, cyclohexane, glacial acetic acid, hydrochloric acid, a soluble starch solution, and potassium dichromate ($K_2Cr_2O_7$). Other reagents were sodium thiosulphate ($Na_2S_2O_3.5H_2O$), sodium hydroxide (NaOH), HCl, acetic acid (30% acetic acid and 70% distilled H_2O), Tris Buffer (1.21 g of Tris-hydroxylmethyl amino methane and 0.59 g of $CaCl_2.2H_2O$), BAPA Substrate, trypsin solution, ammonium molybdate, antimony potassium tartrate, distilled water, and potassium hydrogen phosphate (KH_2PO_4). All the reagents were of analytical grade.

Sample preparation

The soybean sample was air-dried and winnowed to remove any adhering chaff. Further sorting, through hand picking, was carried out to remove heavy foreign materials. The seeds were then sun-dried for moisture content reduction, thereby preventing spoilage.

Central Composite Design (CCD) optimization of the expeller process

The expression was carried out with an existing expeller machine in Songhai Delta, Amukpe Sapele. The soybeans were pre-crushed to expose the surface area using a pre-crusher machine. Process factors, such as the pressure of super-heated steam (P) and toaster time (T), were optimized using a CCD under the Response Surface Methodology (RSM) of the Design of Experiment (DOE) software (7.0.1). The factor levels considered are listed in Table 1 and the thirteen proposed experimental runs were conducted in triplicate. The pre-crushed soybeans (100 kg) were fed into the toaster for toasting using varied steam

pressures from a steam boiler. The cake and oil yield was recorded as Y1 and Y2, at specified pressure and time of toasting. Other responses were phosphorus concentration (Y3) and trypsin inhibitor (Y4), which were used to establish the quality of the residual cake and extracted oil, respectively.

Table 1. Factor Level Selected for the Design

Factors	Units	Levels	
		Low	High
Pressure (P)	kg/ms^2	1.0	2.50
Toaster time (T)	Min	30.00	60.00

Determination of oil and cake yields

The oil yield was determined according to the method of Man et al. (2012). The oil yield was determined as the amount of oil obtained from the mass (100 kg) of soybean fed into the equipment. The cake yield was determined by recovering all the cake in the equipment, allowing it to cool to room temperature, and then weighing. The percentage cake yield was determined by expressing the amount of cake recovered with respect to the mass of soybean fed into the equipment.

Determination of the trypsin inhibitor

The trypsin inhibitor is attributed to anti-nutritional factors that occur in soybeans and it exhibits resistance to destruction by heat. Thus, it is often used as an indicator of the effectiveness of the heat pre-treatment on soybean or its products. The trypsin inhibitor deters the activities of enzymes, such as pepsin and trypsin, which are responsible for protein digestion. Soy products that are targeted for the production of animal or human food are required to be free of trypsin. The classic method used by Kakade et al. (1974) with some modifications suggested by Korol and Przegalińska (1994) was employed in the determination of trypsin inhibitor activity (TIA) of the soybean cake obtained in this study. The process is based on the hydrolysis of the benzoyl-DL-arginine-*p*-nitroanalide hydrochloride (BAPA) substrate. The reaction led to the generation of yellow colour for the *p*-nitroanilin formed, and this is measured with a spectrophotometer at maximum absorbance of 410 nm, which is proportional to its concentration. The calibration curve was plotted for the relationship between the absorbance (at 410 nm) of the solution and trypsin concentration, under optimal conditions for the enzyme. The trypsin inhibitor activity, evaluated based on the relationship developed from the calibration curve, is expressed as the quantity of trypsin with its activity inhibited. The trypsin inhibitor activity unit – TIU is expressed per milligram of dry matter of soybean. The TIA (mg/g) was converted to TIU/mg using

$$\text{TIU/mg} = \text{TIA} \cdot 1.9 \quad (1)$$

Determination of phosphorus in the soy oil sample

The phosphorus content in the soy oil samples was determined using ASTM D515-88. The oil sample (5 mL) was pipetted into a 25 mL volumetric flask and distilled water was added to bring the volume to approximately 15 mL. Then. 8 mL of a potassium hydrogen phosphate (KH_2PO_4) reagent was added to the sample and made up to a volume with distilled water and then the content was mixed thoroughly. Then the absorbance values in a colorimeter 660 nm were determined using a spectrophotometer, after shaking the content in a flask for 30 min. The standard calibration curve was prepared by plotting the absorbance values against concentrations (ppm). The concentration of phosphorus content in the soy oil sample was calculated using:

$$\text{Conc. P (mg/L)} = [\text{AC Conc (mg/L)} \cdot \text{DF} \cdot \text{EV(L)})/ (\text{Vol of water (L)}] \quad (2)$$

Where DF is the dilution factor, AC is the Conc. (mg/L) and EV is the final volume (L).

Characterization of the oil produced

The extracted oil was characterized for specified oil quality parameters such as acid value, iodine value, and peroxide value.

Acid value

The acid value of the soy oil was determined according to the standard (ISO 660, 1996). About 25 mL of denatured alcohol (mixture of 1/1(VN) of 95% ethanol and diethylether) was neutralized with a fresh solution of 0.1 M KOH in the presence of 3 drops of phenolphthalein. This mixture was added to the soy oil sample (0.5 mL) in a 250 mL conical flask, which was swirled carefully before adding 3 drops of indicator. The mixture was titrated against a 0.1 mg/L solution of ethanoic potassium hydroxide solution until a permanent pink colour was attained. The acid value was evaluated from Eq. 3

$$\text{Acid Value} = (56.1 \cdot \text{V} \cdot \text{N})/\text{W} \quad (3)$$

Where, V = volume of standard potassium hydroxide in mL, N = normality of the potassium hydroxide solution, and W = weight of the sample in g.
The acidity is frequently expressed as a free fatty acid, which is calculated as:

$$\text{Free fatty acid} = (28.2 \cdot V \cdot N)/W \text{ percent by weight} \quad (4)$$

Peroxide value

The peroxide value (PV) is defined as the degree of lipid oxidation in foods. The PV of the obtained soy oil was determined according to the AOAC method 965.33 with slight modifications suggested by Krishnan et al. (2015). The oil sample (5 g) was weighed into a 250 mL stoppered conical flask where acetic acid-chloroform mixture (30 mL) and saturated potassium iodide solution (0.5 mL), were added later. The mixture was kept in the dark for 1 min before adding water (30 mL) to it. The mixture was then titrated against a 0.1 N sodium thiosulphate solution, while shaking vigorously until the yellow colour is almost gone, in order to liberate the iodine. A starch solution (0.5 mL) was added as the indicator, and shaken vigorously until the blue colour disappeared, thus indicating the release all I_2 from $CHCl_3$ layer. The peroxide value expressed as a milli equivalent of peroxide oxygen per kg of sample (meq/kg) was evaluated from Eq. 5:

$$\text{Peroxide value} = (\text{Titre} \cdot N \cdot 100)/(m) \quad (5)$$

Where, Titre = mL of sodium thiosulphate used, N = normality of sodium thiosulphate solution, and m = weight of the sample

Iodine value

The iodine value (IV) was determined according to the standard ISO 3961, 1996, based on the Wijjs method. The oil sample (0.2 g) was dispensed into a round neck bottle, mixed with chloroform (5 mL) and the Wijis reagent (8 mL) (9 mL of iodine trichloride and 10 g of iodine in chloroform (300 mL)/acetic (700 mL) solution). The bottle was stoppered and shaken, gently, then placed in the dark for 1 hr, after which 7 mL of KI (100 g/L) and 75 mL of distilled water were added and titrated against a 0.05 M sodium thiosulphate solution using starch as the indicator. A blank test was carried out simultaneously without the oil, under the same conditions. The IV was quantified according to Eq. 6:

$$\text{I.V.} = ((\text{Blank} - \text{sample}) \cdot 0.01269)/w \cdot 100 \quad (6)$$

Statistical analysis

The experimental data obtained for the study was analysed using one-way analyses of variance (ANOVA) and multiple linear regression analysis embedded in the Design-Expert software (7.1.0.) (State-Ease, Minneapolis, MN). The statistical significances were evaluated by determining the F-value at 95% ($p<0.05$) and the fitness of the polynomial model developed was evaluated based on the coefficient of determination (R).

Results and Discussion

Response from the experimental data

The maximum cake yield of 95% was obtained during the experimental Run 1 (2.50 kg/ms^2 and 15 min), Run 4 (1.00 kg/ms^2 and 15 min), Run 6 (0.69 kg/ms^2 and 37.50 min), and Run 9 (1.75 kg/ms^2 and 5.68 min) (Table 2). The minimum cake yield of 80% was obtained during the experimental Run 7 (1.75 kg/ms^2 and 69.32 min) and Run 11 (2.50 kg/ms^2 and 60 min). The maximum oil yield of 13 L/100 kg was obtained during the experimental Run 7 (1.75 kg/ms^2 and 69.32 min) and Run 11 (2.50 kg/ms^2 and 60 min). The minimum oil yield of 3 L/100 kg was obtained during the experimental Run 1 (2.50 kg/ms^2 and 15 min), Run 2 (1.75 kg/ms^2 and 37.50 min), Run 4 (1.00 kg/ms^2 and 15 min), Run 6 (0.69 kg/ms^2 and 37.50 min), and Run 9 (1.75 kg/ms^2 and 5.68 min). The maximum and minimum trypsin inhibitor concentrations of 0.299 and 0.239 mg/g were obtained during the experimental Run 6 (0.69 kg/ms^2 and 37.50 min) and Run 5 (1.00 kg/ms^2 and 60 min), respectively. The maximum phosphorus concentration (1.95 mg/g) was obtained during Run 5 (1.00 kg/ms^2 and 60.00 min), while the minimum concentration (0.28 mg/g) was obtained during the experimental Run 4 (1.00 kg/ms^2 and 15 min).
The desired condition for this experiment is low cake yield, high oil yield, low trypsin inhibitor concentration, and low phosphorus concentration. Thus, a compromise is needed among the available options, since all the desired conditions were not achieved during the same experimental run (Jokić et al., 2010; Salman, 2014; Araromi et al., 2017). However, preference was given to the trypsin inhibitor and phosphorus concentrations which are attributed, invariably, to the quality of the soy oil extracted. Consequently, Run 11 (2.50 kg/ kg/ms^2 and 60.00 min), where the relatively lowest cake yield (80%), highest oil yield (13 L/100 kg), relatively lowest trypsin inhibitor (0.266 mg/g) and phosphorus concentrations (0.39 mg/g) were achieved, was selected.

It is usually desirable to completely remove the amount of phospholipids present in soy oil or to reduce it to very low concentrations, because of their emulsifying properties and the possibility of causing losses of neutral oil during refining (Skevin et al., 2012). The amount of phospholipids in soy oil is evaluated indirectly by phosphorus content of the oil, and the reduction of phosphorus content in the oil is usually accomplished through degumming or bleaching (Wang, 2002; Skevin et al., 2012).

Table 2. Responses from the Experimental runs Central Composite Design

Runs	Factors		Responses			
	Pressure (kg/ms^2)	Roasting time (min)	Cake yield (%)	Oil yield (L/ 100 kg)	Trypsin inhibitor (mg/g)	Phosphorus concentrations (mg/g)
1	2.50	15.00	95.00	3.00	0.293	1.90
2	1.75	37.50	90.00	3.00	0.291	1.80
3	1.75	37.50	90.00	5.00	0.296	0.37
4	1.00	15.00	95.00	3.00	0.294	0.28
5	1.00	60.00	85.00	12.00	0.239	1.95
6	0.69	37.50	95.00	3.00	0.299	0.29
7	1.75	69.32	80.00	13.00	0.248	1.82
8	1.75	37.50	90.00	5.00	0.291	0.33
9	1.75	5.68	95.00	3.00	0.297	0.39
10	1.75	37.50	90.00	5.00	0.291	0.39
11	2.50	60.00	80.00	13.00	0.266	0.39
12	2.81	37.50	87.00	11.50	0.268	0.39
13	1.75	37.50	90.00	5.00	0.291	0.39

The phosphorus content of the soy oil obtained in this study, at the optimized oil extraction, ranges between 0.28 and 1.95 mg/kg. Wang (2002) suggested that neutralized oil containing 5–10 mg/kg of phosphorus required further processing such as bleaching. Therefore, the phosphorus values (0.28–1.95 mg/kg) of soy oil obtained in this study indicate that the processing mill used for extracting the soy oil is efficient and therefore, further bleaching is not required for the oil.

Designed summary for the experiment

The experimental design used for this study was a Central Composite Design (CCD) under the Response Surface Methodology of the Design Expert software (7.1.0). The factors considered were pressure (kg/ms^2) and soybean roasting time (min), while the responses selected were the oil yield (L/kg), cake yield (%), and trypsin inhibitor concentration (mg/g). The design generated thirteen (13) experimental runs and the quadratic model was chosen as the design model for all the responses, but power transformation was effected for the model developed for oil yield. Some of the outliers were well addressed, at the minimal level, in order to minimize the errors in the data set (Montgomery, 2005). The Degrees of Freedom (DF), Residuals, Lack of Fit, Pure Error, and Corr Total evaluated for the model were 5, 7, 3, 4, and 12, respectively. Minimum DF is recommended and such values ensure a valid 'lack of fit' test (Montgomery, 2005). The standard error analysis of the term of the quadratic model is 0.39, 0.29, 0.33, 0.38, and 0.17 for A, B, AB, A^2, and B^2, respectively, while their R^2 are 0.1817, 0.3382, 0.1817, 0.0169, and 0.3457, respectively. The R^2 values sum up to 1.000, and the high R^2 means that the terms correlated with each other.

Model summary statistics for the responses

The model summary statistics suggested by the software are usually used to select the most suitable model for the selected responses. The standard deviation shows the degree of deviation (error) of the experimental values from the actual values, while the R^2 reflects the efficiency of the experiment. The adjusted R^2 and predicted R^2 are the adjusted and predicted values by the design expert software, respectively, with focus on the model maximizing the adjusted R^2 and the predicted R^2. Suitable models for the optimization of each response were usually selected based on the highest order polynomials, which are not aliased. The cubic models for the selected responses (cake yield (%), oil yield (%), trypsin inhibitor concentration (mg/g), and phosphorus concentrations (mg/g)) had the highest R^2 value (1.000, 0.9987, 0.9955 and 0.7649) with the corresponding lowest standard deviation of 0.000, 55.85, $2.236 \cdot 10^{-3}$ and 2.59 (Table 3).

The choice of the cubic model was negated due to its 'aliased' status, thus the quadratic model was suggested for oil yield (%) and trypsin inhibitor

concentration (mg/g), while two-factor interaction (2FI) was suggested for phosphorus concentrations (mg/g). On the other hand, the choice of the quadratic model was further reinforced based on the large differences between the Predicted Residual Error Sum of Squares (PRESS) of the quadratic. The software suggested linear and quadratic models for the cake yield, but one is to be preferred. The quadratic model was selected with respect to linear based on the lowest standard deviation (0.62) of the data obtained to the mean values (Montgomery, 2005).

Table 3. Model Summary Statistic

Response	Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS
Cake yield	*Linear	1.41	0.9398	0.9264	0.8744	37.04
	2FI	1.20	0.9609	0.9463	0.9016	29.01
	*Quadratic	0.62	0.9922	0.9857	0.8590	41.58
	^Cubic	0.000	1.0000	1.0000	NA	NA
Oil yield	Linear	538.63	0.7241	0.6628	0.5302	$4.447 \cdot 10^6$
	2FI	565.26	0.7299	0.6286	0.3180	$6.454 \cdot 10^6$
	*Quadratic	268.00	0.9545	0.9165	0.6804	$3.025 \cdot 10^6$
	^Cubic	55.85	0.9987	0.9964	0.9630	$3.503 \cdot 10^5$
Trypsin inhibitor	Linear	0.013	0.6477	0.5694	0.2858	$3.169 \cdot 10^{-3}$
	2FI	0.013	0.6918	0.5763	-0.0409	$4.618 \cdot 10^{-3}$
	*Quadratic	$4.328 \cdot 10^{-3}$	0.9747	0.9536	0.7541	$1.091 \cdot 10^{-3}$
	^Cubic	$2.236 \cdot 10^{-3}$	0.9955	0.9876	NA	NA
Phosphorus concentrations (mg/g)	Linear	3.44	0.0629	-0.1454	-0.8948	215.43
	*2FI	2.28	0.6342	0.4971	0.2576	84.41
	Quadratic	2.29	0.7242	0.4943	-0.0722	121.90
	^Cubic	2.59	0.7649	0.3534	NA	NA

*Suggested, ^ Aliased, PRESS (Predicted Residual Error Sum of Squares), NA (Not Applicable)

Regression model equation for the responses

The final model equations were developed in terms of coded (Eq. 7–10) and actual (Eq. 11–14) factors, for the cake yield, oil yield, trypsin inhibitor concentration, and phosphorus concentrations, respectively. The equations also indicate the presence of linear, square, and crossed terms for the two variables investigated.

All the equations (Eq. 7–14) have positive intercept values of +87.95, + 447.26, +0.28, and +2.20 for cake yield (%), oil yield (%), trypsin inhibitor concentration, (mg/g), and phosphorus concentrations (mg/g). All the positive and negative coefficients indicated positive and negative influences of the independent variables on the selected responses, respectively (Alade et al., 2012). The coefficients -1.81, - 4.36, - 0.83, - 0.31, and - 0.51 obtained for A, B, AB, A^2, and B^2 indicate the negative influence of the two variables (temperature and toasting time) on the extracted cake yield. All the coefficients (361.83, 808.80, 78.17, 350.01, 230.48) obtained for all the model terms A, B, AB, A^2, and B^2, respectively for oil yield were positive, and this indicates that oil yield for the soybean was highly influenced by the two factors (temperature and toasting time) (Araromi et al., 2017). Trypsin inhibitor concentration is positively influenced by pressure ($7.098 \cdot 10^3$), as well as cross terms of pressure and toasting time ($4.667 \cdot 10^3$), while the terms B, A^2, and B^2 influenced the trypsin inhibitor concentration negatively according to their coefficients -0.016, -0.013, and $-3.788 \cdot 10^3$, respectively. The phosphorus concentration was influenced by toasting time (1.26) positively, while it was influenced by pressure (-0.99), as well as cross terms of pressure and toasting time (-3.39) negatively.

Coded equations

$$\text{Cake Yield (\%)} = 87.95 - 1.81A - 4.36B - 0.83AB - 0.31A^2 - 0.51B^2 \quad (7)$$

$$(\text{Oil Yield})^3\ (\%) = 447.26 + 361.83A + 808.80B + 78.17AB + 350.01A^2 + 230.48B^2 \quad (8)$$

$$\text{Inhibitor conc. (mg/g)} = 0.28 + 7.098 \cdot 10^{-3}A - 0.016B + 4.667 \cdot 10^{-3}AB - 0.013A^2 - 3.788 \cdot 10^{-3}B^2 \quad (9)$$

$$(\text{Phosphorus})^3\ (\text{mg/g}) = +2.20 - 0.99A + 1.26B - 3.39AB \quad (10)$$

Actual equations

$$\text{Cake Yield (\%)} = +93.16 + 2.84A + 0.043B - 0.074AB - 0.55A^2 - 2.27 \cdot 10^{-3}B^2 \quad (11)$$

$$(\text{Oil Yield})^3\ (\%) = 1703.73 - 2008.09A - 50.43B + 6.95AB + 622.24A^2 + 1.03B^2 \quad (12)$$

$$\text{Trypsin inhibitor concentration (mg/g)} = 0.246 + 0.071A - 3.04\cdot10^{-4}B + 4.15\cdot10^{-4}AB - 0.02A^2 - 1.68\cdot10^{-5}B^2 \quad (13)$$

$$(\text{Phosphorus})^3\,(\text{mg/g}) = -23.014 + 12.24A + 0.61B - 0.30AB \quad (14)$$

Where A = Pressure and B = Residence Time,

Analysis of variance (ANOVA) for the responses

The model F-value of 152.65 (Table 4) obtained for cake yield implies the model is significant, and there is only a 0.01% chance that a "Model F-value" of this magnitude will occur due to noise. Generally, "Prob > F' less than 0.10 indicates that the model terms are significant while "Prob > F' greater than 0.10 indicates that the model terms are not significant (Montgomery, 2005). Model terms such as A, B, B^2, and AB with "Prob > F' values of <0.0009, <0.0001, <0.0068, and <0.0033, respectively are very significant for the cake yield. The model F-value of 25.15 obtained for the soy oil yield (Table 4) implies the model is significant, and there is only a 0.06% chance that "Model F- value" this large could occur due to noise. The significant model terms for the oil yield are A, B, A^2, and B^2 with 'Prob > F' values of 0.0136, <0.0001, 0.0164, and 0.0027. The 'lack of fit F-value' for oil yield is 58.83, and it implies the lack of fit is significant with only a 3.99% chance of occurrence due to noise. Although a significant lack of fit is not desirable, yet, the model is desirable to be fit (Montgomery, 2005).

The model F-value of 46.17 obtained for trypsin inhibitor concentration (Table 4) implies the model is significant, and there is only a 0.01% chance of occurrence due to noise. All the model terms such as A, B, AB, A^2, and B^2 with "Prob > F' values of 0.0149, < 0.0001, 0.0178, 0.0009, and 0.0024, respectively are very significant for the trypsin inhibitor concentration. The 'lack of fit F-value' for the trypsin inhibitor concentration is 9.24, and it implies the lack of fit is significant with only 3.17% chance of occurrence due to noise. The model F-value of 4.62 obtained for phosphorus concentration (Table 4) implies the model is significant, and there is only a 3.70% chance of occurrence due to noise. Only B and AB, with "Prob > F' values of 0.0725 and 0.0077, are significant model terms for the phosphorus concentration at p<0.10. The 'lack of fit F-value' for the trypsin inhibitor concentration is 0.56, and it implies the lack of fit is significant with only 70.84% chance of occurrence due to noise (Montgomery, 2005).

Regression statistics for the responses

Standard deviation, mean, CV, PRESS, R^2, adj R^2, Pred. R^2, and adequate precession obtained for the cake yield were 0.62, 88.92, 0.70, 41.58, 0.9922, 0.9857, 0.8590, and 37.318, respectively. The Pred. R^2 (0.8590) is in reasonable agreement with the adj R^2 (0.9857). The Standard deviation, mean, CV, PRESS, R^2, adj R^2, Pred. R^2, and adequate precession obtained for the of oil yield were 268.00, 679.41, 39.45, $3.025\cdot10^6$, 0.9545, 0.9165, 0.6804, and 13.025, respectively. The Pred. R^2 (0.6804) is not as close to the adj R^2 (0.9165) as might be expected customarily. This may indicate a large block effect, however, model reduction, response transformation, and outliers were adequately considered, yet this is the best result obtained from the software. The standard deviation, mean, CV, PRESS, R^2, adj R^2, Pred. R^2, and adequate precession obtained for the trypsin inhibitor concentration were $4.328\cdot10^{-3}$, 0.28, 1.54, $1.091\cdot10^6$, 0.9747, 0.9536, 0.7541, and 20.201, respectively. The Pred. R^2 (0.7541) is in reasonable agreement with the adj R^2 (0.9536), standard deviation, mean, CV, PRESS, R^2, adj R^2, Pred. R^2, and adequate precession obtained for the of phosphorus were 2.28, 2.21, 103.08, 84.41, 0.6771, 0.4971, 0.2576, and 6.802, respectively. The Pred. R^2 (0.2576) is not as close to the adj R^2 (0.4971) as might be expected and this case was handled as indicated for oil yield above.

The "Adequate precision", which measures the signal to noise ratio, must be sufficiently greater than 4, so that the model can be used to navigate the design space. The "Adequate precision" values obtained for cake yield, oil yield, trypsin inhibitor and phosphorus concentrations were 37.318, 13.025, 20.201, and 6.802, respectively, indicating the suitability of the developed models (Agarry and Ogunleye, 2012). The coefficients of variance (0.70%, 3.404, and 1.54%) obtained for cake yield, oil yield, trypsin inhibitor, and phosphorus concentrations, respectively, were sufficiently lower than 10 percent. This indicates a high precision and reliability of the experiments (Agarry and Ogunleye, 2012).

Diagnostic case studies for the responses

The residual values indicate the difference between the actual values from the experiment and the predicted values generated by the software for the soy cake yield, oil yield, trypsin inhibitor, and phosphorus concentrations. Negative residual values indicate that the actual value is greater than the predicted value, while positive residual value implies that the predicted value is greater than the actual value. Zero residual values mean that the actual is equivalent to the predicted value on which its comparison is based (Alade et al., 2012).

Table 4. Analysis of Variance (ANOVA) for cake yield, oil yield, trypsin inhibitor concentration, and phosphorus concentration

	Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F
Cake yield	Model	292.62	5	58.52	152.65	< 0.0001*
	A	13.91	1	13.91	36.28	0.0009*
	B	221.12	1	221.12	576.74	< 0.0001*
	AB	6.25	1	6.25	16.30	0.0068*
	A^2	0.40	1	0.40	1.04	0.3465
	B^2	8.54	1	8.54	22.26	0.0033*
	Residual	2.30	6	0.38		
	Lack of Fit	2.30	2	1.15		
	Pure Error	0.000	4	0.000		
	Cor Total	294.92	11			
Oil yield	Model	$9.033 \cdot 10^6$	5	$1.807 \cdot 10^6$	25.15	0.0006*
	A	$8.565 \cdot 10^5$	1	$8.565 \cdot 10^5$	11.92	0.0136*
	B	$7.570 \cdot 10^6$	1	$7.570 \cdot 10^6$	105.40	< 0.0001*
	AB	54990.25	1	54990.25	0.77	0.4152
	A^2	$7.826 \cdot 10^5$	1	$7.826 \cdot 10^5$	10.90	0.0164*
	B^2	$1.721 \cdot 10^6$	1	$1.721 \cdot 10^6$	23.97	0.0027*
	Residual	$4.309 \cdot 10^5$	6	71824.18		
	Lack of Fit	$4.237 \cdot 10^5$	3	$1.412 \cdot 10^5$	58.83	0.0037*
	Pure Error	7203.00	3	2401.00		
	Cor Total	$9.464 \cdot 10^5$	11			
Trypsin inhibitor concentration	Model	$4.325 \cdot 10^{-3}$	5	$8.649 \cdot 10^{-4}$	46.17	0.0001*
	A	$2.134 \cdot 10^{-4}$	1	$2.134 \cdot 10^{-4}$	11.39	0.0149*
	B	$3.126 \cdot 10^{-3}$	1	$3.126 \cdot 10^{-3}$	166.85	< 0.0001*
	AB	$1.960 \cdot 10^{-4}$	1	$1.960 \cdot 10^{-4}$	10.46	0.0178*
	A^2	$7.029 \cdot 10^{-4}$	1	$7.029 \cdot 10^{-4}$	37.52	0.0009*
	B^2	$4.712 \cdot 10^{-4}$	1	$4.712 \cdot 10^{-4}$	25.15	0.0024*
	Residual	$1.124 \cdot 10^{-4}$	6	$1.873 \cdot 10^{-5}$		
	Lack of Fit	$9.240 \cdot 10^{-5}$	2	$4.620 \cdot 10^{-5}$	9.24	0.0317*
	Pure Error	$2.000 \cdot 10^{-5}$	4	$5.000 \cdot 10^{-6}$		
	Cor Total	$4.437 \cdot 10^{-3}$	11			
Phosphorus concentration	Model	72.11	3	24.04	4.62	0.0370*
	A	6.18	1	6.18	1.19	0.3073
	B	22.22	1	22.22	4.27	0.0725*
	AB	64.96	1	64.96	12.50	0.0077*
	Residual	41.59	8	5.20		
	Lack of Fit	14.85	4	3.71	0.56	0.7084
	Pure Error	26.73	4	6.68		
	Cor Total	113.70	11			

A-Pressure, B-Residence Time, * Significant at $p<0.10$

It was observed that runs 5, 6, 8, and 11 had negative residual values of -0.41, -0.88, -0.41, and -0.12 for the cake yield. Other experimental runs had positive residual value and no 'zero' predicted value. The oil yield had negative residual values of -1.92, -1.60, -2.01, -0.92, -0.40, and -0.71 at experimental Runs 1, 2, 6, 7, 8, and 11. Positive residual values were observed at other experimental runs and no 'zero' residual value was observed.

The residual values observed for trypsin inhibitor were negative at runs 2, 4, 5, 6, 7, 8, 9, and 12 with the values of $-9.988 \cdot 10^{-4}$, $-9.988 \cdot 10^{-4}$, $-4.910 \cdot 10^{-3}$, $-4.698 \cdot 10^{-3}$, $-4.698 \cdot 10^{-3}$, $-8.709 \cdot 10^{-4}$, $2.063 \cdot 10^{-4}$, and $-9.988 \cdot 10^{-4}$, respectively. The phosphorus concentration had negative residual values of -1.52, -1.54, -2.51, -0.54, -1.51, -0.43, and -1.51 at experimental runs 3, 4, 5, 7, 8, 9, and 12, respectively. Other experimental runs had positive residual values with no 'zero' residual value observed. This correlation between the actual values from the experiment and the predicted values was plotted for the soybean cake yield, oil yield, trypsin inhibitor, and phosphorus concentration (Figure 1a-d). The correlation observed from the plots is relatively high, with values of 0.9922, 0.9545, 0.9747, and 0.6771 for the soy cake yield, oil yield, trypsin inhibitor, and phosphorus concentration, respectively.

The interaction of the selected factors (pressure and time) on soy cake yield, oil yield, trypsin inhibitor content, and phosphorus concentration are illustrated in contour plots (not shown). Generally, the contour plots for the responses indicate that the

interaction of pressure and toasting time had an effect on soy cake yield, oil yield, trypsin inhibitor content, and phosphorus concentration. The three-dimensional (3D) representations of these trends are illustrated in Figure 2 for the soy cake yield, oil yield, trypsin inhibitor content, and phosphorus concentration. The contour lines (Figure 2a) are not absolutely parallel, as seen, they slightly curve towards the right end of the figure, and thus the equation can be assumed quadratic. The curve lines in Figure 2b-c indicate a quadratic model, while the asymmetrical curve lines in Figure 2d suggested two-factor interactions.

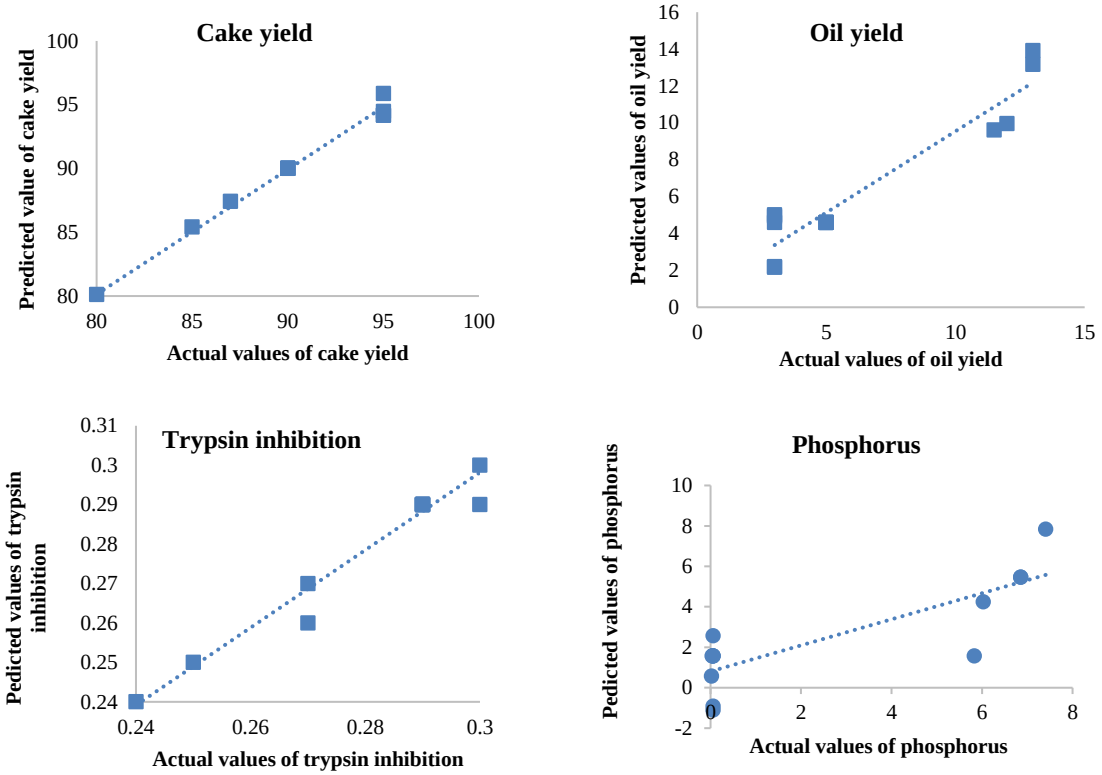

Fig 1. Plot of predicted vs. actual values for cake yield, oil yield, trypsin inhibitor, and phosphorus concentration

Characterization of obtained soy oil

Physicochemical parameters are generally used to evaluate the quality and functionality of a given oil (Farhoosh, et al., 2008). Typical oil must possess good grades of these physicochemical parameters in order to classify them for edible purpose and industrial applications (Jinfeng, et al., 2011). The quality of soy oil extracted in this study was analysed for its peroxide, acid, and iodine values (Table 5).

Table 5. Physicochemical properties of crude and refined oils

Parameters	Units	Soybean oil
Acid value	mg KOH/ g	1.60
Peroxide value	mg KOH/ g	18.00
Iodine value	g/100 g	60.40

Peroxide value

The peroxide value is a measure of the concentration of oil that oxidizes potassium iodide to iodine successfully, and can indicate the extent to which rancidity reactions have occurred in an oil sample during storage due to relative higher oxidation in oils (Hasan et al., 2016). It suggests the quality and stability of oils over time and their increase. The peroxide value of soy oil extracted in this study is 18 meq O_2/kg oil, which is higher than the peroxide values of 1.62, 2.09, 1.72, 1.01, 1.17, and 1.21 meq O_2/kg obtained for Mustafa soybean, Muskan soybean, Pusti soybean, Teer soybean, Fresh soybean, and Rupchanda soybean oils, respectively (Hasan et al., 2016). This variation may be due to the species of the seed samples used for extraction purposes.

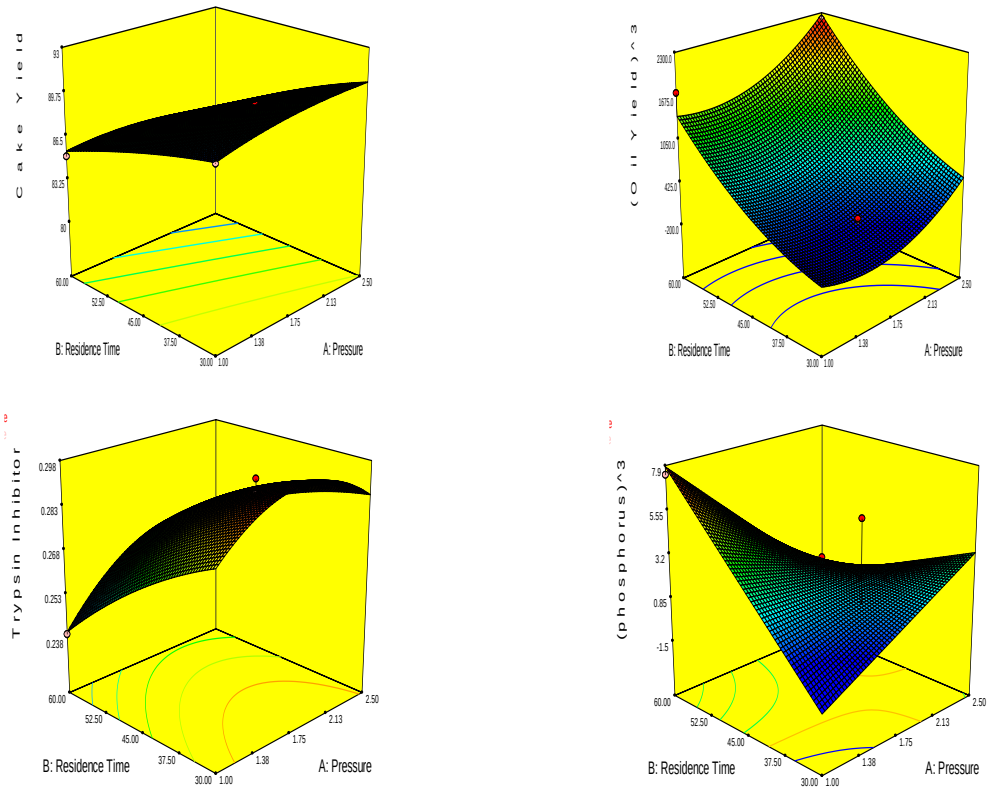

Fig 2. 3D Plot of toasting time and pressure on soy cake yield, oil yield, trypsin inhibitor content, and phosphorus concentration

Acid value

Acid value of oils is an indication of the amount of free fatty acids present in an oil sample and it relates to the purity of the oil. Thus, a high acid value renders an oil sample unfit for cooking purposes due to likely damage to human health (Hasan et al., 2016). It indicates that the triglycerides of the oil are converted into fatty acids and glycerol; this may cause rancidity of the oil (Zahir et al., 2014). The acid value of soy oil extracted in this study is 1.60 mg KOH/g oil, which is higher than the acid values of 0.37, 0.73, 0.56, 0.38, 0.36, and 0.42 mg KOH/g obtained for Mustafa soybean oil, Muskan soybean oil, Pusti soybean oil, and Teer soybean oil, respectively (Hasan et al., 2016). These variations may be attributed to the methods of extraction.

Iodine value

Iodine value measures the degree of unsaturation in a vegetable oil (Ekwu and Nwagu, 2004), and the possibility of oil to go to rancid is dependent on the high level of unsaturation in the oil (AOCS, 2016). The iodine value of soy oil extracted in this study is 60.40 g/100g, which is lower than the iodine values of 85.4, 105.7, 89.8, 90.16, 105.47, and 109.96 g/100 g obtained for Mustafa soybean oil, Muskan soybean oil, Pusti soybean oil, and Teer soybean oil, respectively (Hasan et al., 2016). This relative lower iodine value of soy oil indicates that a high percentage of fatty acids in the soy oil is saturated, so they have a very low C=C double bond, which has a low minimum iodine number (Marinova, et al., 2012). Lower iodine values indicate higher oxidative storage stability of the oil (Hasan et al., 2016).

Conclusions

The Centre Composite Design (CCD) under the Response Surface Methodology of the Design Expert software (7.1.0) has been used successfully for the extraction of oil from soybean in an expeller machine. The pressure (2.50 kg/ms^2) and soybean toasting time (60.00 min) gave the optimum condition for relatively lowest cake yield (80%), highest oil yield (13 L/100 kg), relatively lowest trypsin inhibitor (0.266 mg/g), and relatively lowest phosphorus concentrations (0.39 mg/g). The correlation coefficient (R^2) of the model equation developed for responses (cake yield, oil yield, trypsin inhibitor concentrations, and phosphorus concentration) was high. The quadratic model was

selected for cake yield (%), oil yield (%), and trypsin inhibitor concentration (mg/g), while the 2FI model was selected for the phosphorus concentration (mg/g). The characteristics such as peroxide value, acid value, and iodine value of the extracted soy oil are within the acceptable limits for consumer and industrial uses. The optimisation tools have facilitated the efficiency of the expeller in producing better products.

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