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Statistical model for solvent oil extraction from soybean (*Glycine max* (L.))

*Shittu S.K., Mari H.H. and Dangora N.D.

Department of Agricultural and Environmental Engineering, Faculty of Engineering, Bayero University P.M.B 3011 Kano, Nigeria.

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Abstract

Oil extraction process using hexane is common among large-scale oil industries in Nigeria. Various factors play vital roles in the quantity and quality of oil extracted from oil-bearing crops using this method. This study therefore aimed at determining the effect of flake moisture content, flake thickness and solvent temperature on oil extraction of soybean using hexane solvent in soxhlet apparatus. A 3 x 3 x 3 factorial experiment in a complete randomized design involving flake moisture content, flake thickness and solvent temperature each at three levels in three replications was adopted. The experiments were done at flake moisture contents of 4.5, 7 and 9% (wb), flake thicknesses of 0.4, 0.7 and 0.9 mm, and solvent temperatures of 60, 65 and 70°C. ANOVA using SAS software procedure was employed to determine the effect of variables on the oil extraction and a regression model for predicting oil yield on the basis of the three factors was developed using *XLSTAT* Microsoft Excel. The results showed that combination of 4.5% flake moisture content, 0.4 mm flake thickness and 70°C solvent temperature gave the highest oil yield (19.37%), whereas 9% flake moisture content, 0.9 mm flake thickness and 60°C solvent temperature gave the lowest oil yield (14.57%). All evaluated factors have a significant effect on oil yield at 1% level. The interaction between the factors did not have a significant effect on oil yield. Model verification results gave mean square error, root mean square error, bias and accuracy of 0.084, 0.290, 0.173 and 0.030 respectively. High value of the coefficient of determination R^2 (0.966) obtained indicated that there is a good agreement between the observed and the predicted values.

1. Introduction

Oil-bearing agricultural products are numerous and are of great economic importance to mankind. The oil contents in these products can be categorized as the edible and non-edible oils. Most oils are embedded in their parent materials and there is a need to extract them out before usage. Oil extraction is the process of recovering oil from oil-bearing agricultural products through manual, mechanical, chemical or mechano-chemical extraction. Sorin-Stefan *et al.* (2007) stated that oil products industry produces edible and non-edible oils with about two-thirds of total oil products being the edible oils used directly in foods or in the manufacture of margarine, mayonnaise, bakery and pastry products, and as cooking fats. The remaining one-third of the total volume of produced oil is the technical oils, used in the production of various non-edible products, such as detergents, paint, glycerin, fatty acids, varnish, pharmaceuticals or cosmetics. Some common oil-seed crops include cotton, castor, groundnut, sunflower, sesame and soybean seeds.

In the past, soybean is grown primarily for its protein and secondary for oil but nowadays it is one of the most commonly used raw materials by large-scale vegetable oil industries in Nigeria. A survey on the types of seeds commonly used for oil extraction by industries in Kano Nigeria, revealed that: soybean seed 47%, groundnut seed 29%, sesame seed 14% and cottonseed 14% (Mari, 2017). Oil extraction from soybean can be done by mechanical or solvent method or combination of both. Lawson *et al*. (2010) revealed that soybean seed with low oil content are better extracted using the solvent method because the method is characterized by higher oil yield, larger processing capacity and lower refining losses. The solvent oil extraction process is more popular and it is preferred over mechanical expulsion because of its high-quality oil with minimal refining (Ramanujan, 2006). Generally, the demand for vegetable oil is on the increasing trend (Parcell *et al*., 2018). One of the ways adequate oil extraction could be achieved is by

*Corresponding author.
Email: *sarafadeenshittu@gmail.com; skshittu.age@buk.edu.ng*

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specifying the best factors combination for optimum oil extraction. Factors affecting oil extraction have been studied by some researchers.

Mwithiga and Moriasi (2007) study the yield characteristics during mechanical oil extraction of preheated and ground soybeans. The study showed that the initial heating of material before the mechanical expression is very important. It was found that the duration of the application and the temperature of the soybean has a linear relationship with the oil yield for temperature falling below 75°C. Sayyar *et al*. (2009) carried out extraction of oil from Jatropha seeds using organic solvent. The study revealed the effects of five operating parameters on the oil extraction. The factors considered include type of solvents, temperature, solvent to solid ratio, processing time and particle size of the meal. Optimum conditions were found using hexane as solvent at 8 h reaction time, temperature of 68°C, coarse particle size (0.5-0.75 mm) and solvent to solid ratio of 6:1. Abdulkarim and Saady (2010) studied the factors affecting the extraction process of oil-bearing flakes of sunflower, cotton and soybean seeds. It was concluded that the best condition for extraction of sunflower cotton and soybean seeds were moisture content of 9-11%, flake thickness of 2-3 mm, solvent ratio of 1:1 by weight temperature of the extraction of 60-65°C for pure hexane and the optimum extraction time of 5 hrs. A study conducted by Lawson *et al*. (2010) on the parameters affecting the solvent extraction of soybean oil. It revealed that at a particle size of 2 mm, flake thickness of 0.25 mm, moisture content of 12-13%, temperature of 69°C and the extraction time between 3.5 and 4.5 hrs gave the maximum oil yield. Yusuf *et al*. (2014) study the effects of heating temperature and seed condition on the yield and quality of mechanically expressed groundnut oil. The study revealed that the oil yield increases with increase in heating temperature but tends to decrease as the temperature increase from 90-100°C.

Generally, factors that affect solvents oil extraction are solvent temperature, flake moisture content, flake thickness, solvent to flake ratio and extraction time. These factors could be used to generate models equations which describe the physical/chemical process of oil extraction. Simulation and Modelling are of great importance in engineering because they provide information fast and safe cost; finding important input parameters with changing simulation inputs; can be used with new design and policies before implementation and can be used to investigate a wide verity of what if questions about real-world system (Banks *et al*., 2010).

An informal study carried out in Kano cluster areas of oil extraction indicated that most of the companies carry out their oil extraction at different flake moisture contents, flake thicknesses and solvent temperatures using their own discretion. This led to low oil yield in some oil companies. Hence, determination of the best combination of the above factors through modeling is therefore imperative. The objectives of this study are therefore to: (i) determine the effect of flake moisture content, flake thickness and solvent temperature on solvent oil extraction of soybean using hexane in a Soxhlet apparatus. (ii) use the obtained data from experiments to develop a statistical model using multiple linear regression analysis and (iii) validate the developed model using the coefficient of determination.

2. Materials and methods

The materials used for the experiment were: soybean seeds, hexane and distilled water. The equipment used includes Soxhlet apparatus, flaking machine, desiccators, digital thermometer, glass beakers, sensitive weighing balance (model 88 ADAM), Ohaus moisture analyzer (model ATS60) and Mitutoyo digital micrometer screw gauge (model PS65).

2.1 Samples preparation and experimental procedure

Samples were conditioned to desired moisture contents of 4.5%, 7% and 9% (wb) using the method described by Lawson *et al*. (2010). The method involves adding calculated amount of distilled water and applying Equation (1).

$$Q = \frac{w_1(m_d - m_i)}{(100 - m_d)} \quad (1)$$

Where, w_1 = initial mass of the sample (g); m_i = initial moisture content of the sample % (wb); m_d = final (desired) moisture content of sample % (wb); and Q = mass of water to be added (g)

Figure 1. Soxhlet apparatus

Conditioned soybean seeds samples were fed into a flaking machine to achieve flakes thicknesses of 0.4, 0.7 and 0.9 mm. After flaking, samples thicknesses were checked using the digital micrometer screw gauge. Flakes of various thicknesses and moisture contents were weighed 10 g and wrapped in filter paper and placed in the thimble of the extractor of the Soxhlet apparatus

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(Figure 1). Extraction flask of the apparatus was filled with hexane up to 150 mL and heated to temperature of 60°C. The experiment was allowed to run for 3.5 hrs Lawson *et al*. (2010). Extracted oil was removed and emptied into a beaker to remove traces of solvent and moisture. The oil was placed in desiccators to cool. After cooling, the contents of the beaker was weighed and the percentage of the oil yield was calculated using Equation (2) (Lawson *et al*., 2010). The experiment was repeated at hexane temperatures of 65 and 70°C.

$$Y_o = \frac{W_O}{W_S} \times 100\% \quad (2)$$

Where, Y_0 = oil yield (%); W_0 = weight of extracted oil (g); and W_s = weight of sample (g)

2.2 Experimental design and procedure

Factorial experiment in a completely randomized design was adopted. The factors considered were three levels of flake moisture content, three levels of flake thickness and three levels of solvent temperature. All the treatments were replicated three (3) times totaling eighty -one experiments. Analysis of variance (ANOVA) using SAS software procedure was employed to determine the effect of the variables on the oil yield.

2.3 Model development

A multiple regression model for predicting oil yield on the basis of the three factors (flake moisture content, flake thickness and solvent temperature) was developed using *XLSTAT* Microsoft Excel.

2.4 Methods of model verification

Verification of the model was carried out using Equations 3-6 as given by Wilmott (1981):

$$\text{MSE} = \frac{1}{n}\sum_{i=1}^{n} Vi^2 \quad (3)$$

$$\text{RMSE} = [\frac{1}{n}\sum_{i=1}^{n} Vi^2]^{\frac{1}{2}} \quad (4)$$

$$\text{Bias} = \frac{1}{n}\sum_{i=1}^{n} Vi \quad (5)$$

$$\text{Accuracy} = (\text{bias})^2 = \left(\frac{1}{n}\sum_{i=1}^{n} V_i\right)^2 \quad (6)$$

Where MSE = Mean square error; RMSE = root mean square error; $V_i = P_i – O_i$ at data locations where i = 1, 2, 3…; P_i = predicted value at n = i; O_i = observed values at n = i; and n = number of values in the check data set.

2.5 Method of model validation

To validate the model, two sets of experiments were used. The first experiment was used to create the model and the second experiment was used for validation. The predicted values were obtained from the model equation while the observed values were the second experimental values. In order to validate the model, the predicted values were plotted against the observed values and the coefficient of determination, R^2 was determined to ascertain the agreement between predicted and observed values.

3. Results and discussion

The mean values of the oil yield at different moisture content, flake thickness and temperature are presented in Table 1. The table revealed that the maximum oil yield of 19.37% was obtained at the lowest flake moisture content of 4.5%, lowest flake thickness of 0.4 mm and the highest solvent temperature of 70°C. Also, the minimum oil yield of 14.57% was obtained at the highest flake moisture content of 9%, highest flake thickness of 0.9 mm and the lowest solvent temperature of 60°C. The results show that the higher the moisture content, the lower the oil yields. This agrees with the findings of Lawson *et al*. (2010) and Jacq *et al*. (2006). The reason for these is that hexane is highly insoluble in water, with high moisture content the extraction efficiency of the solvent will be drastically reduced leading to poor oil yield results. The results revealed that the thinner the flake thickness, the higher the oil yields. The reason for this observation is due to the less resistance offered by the thin flakes to the flow of the solvent and there are more surface areas of flakes at lower thickness resulting in more solvent actions on the samples. This observation agrees with the findings of Lawson *et al*. (2010) and Abdulkarim and Saady (2010). The results show that the higher the temperature, the higher the oil yields. But the temperature should not exceed the boiling point of the solvent. The reason for this observation is that at higher temperature of the solvent, there is a breakdown of oil cells leading to decrease in oil viscosity and increasing the number of oil siphons which allow oil to flow more readily through. This trend corroborates with the findings of Ramanujan (2006), Ebewele *et al*. (2010), Momoh *et al*. (2015) and Abdulkarim and Saady (2010).

3.1 Effect of flake moisture content, flake thickness and solvent temperature on oil yield

Results of analysis of variance carried out on the oil yield of soybean during solvent oil extraction are presented in Table 2. The results show that flake moisture content, flake thickness and solvent temperature have a significant effect on oil yield at 1% level. All the interactions were not statistically significant on the oil yield.

The results for further analysis using Duncan multiple range analysis is presented in Table 3. The results revealed that the mean oil yield was found to be highest (17.92%) at the lowest flake moisture content for the given experimental conditions. The results showed

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Table 1. Mean values of oil yield at different flake moisture content, flake thickness and solvent temperature

Moisture content (%)	Flake thickness (mm)	Temperature (°C)	Oil Yield (%)
4.5	0.4	60	18.76
4.5	0.7	60	17.20
4.5	0.9	60	16.40
4.5	0.4	65	19.07
4.5	0.7	65	17.83
4.5	0.9	65	16.87
4.5	0.4	70	19.37
4.5	0.7	70	18.47
4.5	0.9	70	17.33
7	0.4	60	17.97
7	0.7	60	16.30
7	0.9	60	15.40
7	0.4	65	18.65
7	0.7	65	16.95
7	0.9	65	16.08
7	0.4	70	19.33
7	0.7	70	17.60
7	0.9	70	16.77
9	0.4	60	18.10
9	0.7	60	16.00
9	0.9	60	14.57
9	0.4	65	18.43
9	0.7	65	16.20
9	0.9	65	16.08
9	0.4	70	18.77
9	0.7	70	16.40
9	0.9	70	16.57

Table 2. Analysis of variance results for effect of flake moisture content, flake thickness and solvent temperature on oil yield of soybean

Sources of variation	Degree of freedom	Sum of squares	Mean square	Calculated F	Tabular F	
					1%	5%
Replication	2	11.84	5.92	1.52	5.02	3.17
Flake moisture content, *M*	2	21.19	10.59	23.74**	5.02	3.17
Flake thickness, *S*	2	86.09	43.04	96.48**	5.02	3.17
Solvent Temperature, *T*	2	16.41	8.20	18.39**	5.02	3.17
Interactions: $M \times S$	4	2.45	0.61	1.37^{NS}	3.69	2.54
$S \times T$	4	0.89	0.22	0.50^{NS}	3.69	2.54
$M \times T$	4	0.57	0.14	0.32^{NS}	3.69	2.54
$M \times T \times S$	8	1.99	0.25	0.56^{NS}	2.86	2.12
Error	54	24.09	0.45			
Total	80	152.81				

NS= Not significant **= Significant at 1% probability level (Highly significant)

Table 3. Duncan multiple range analysis

Treatment	Mean value	Duncan Grouping
M_1 (4.5%)	17.922	A
M_2 (7%)	17.222	B
M_3 (9%)	16.733	C
S_1 (0.4mm)	18.7077	A
S_2 (0.7mm)	16.9944	B
S_3 (0.9mm)	16.2714	C
T_3 (70°C)	17.8444	A
T_2 (65°C)	17.2944	B
T_1 (60°C)	16.744	C

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that oil yield decrease with increase in the flakes moisture content and the values of oil yields were statistically different at 1% level for all of the flake moisture content levels. This show that controlling the flake moisture content is important during solvent oil extraction. The results from Table 3 using Duncan multiple range analysis show that the mean values of oil yield were found to be highest (18.70 %) at the flake thickness of 0.4 mm. The table showed that oil yield decrease with increase in the flakes thickness and the values of oil yields were statistically different at 1% level for all of the flake thicknesses. It implies that regulating the flake thickness is essential in solvent oil extraction. Duncan multiple range analysis (Table 3) revealed that the mean values of oil yield were highest (17.84 %) at the highest temperature of 70°C. From the table, it is evident that oil yield increased with increase in the solvent temperature and the values of oil yields were statistically different at 1% level for all the temperature levels. It can, therefore, be concluded that correct solvent temperature is necessary for solvent oil extraction.

3.2 Model equation

Regression equation model for predicting soybean oil yield with respect to flake moisture contents, flake thickness and solvent temperature developed using *XLSTAT* of Microsoft Excel can be expressed as:

$$Y = 15.114 - 0.277M - 4.944S + 0.114T \quad (7)$$

Where Y = oil yield (%); M = flake moisture content (% wb); S = flake thickness (mm); and T = solvent temperature (°C)

The model shows that flake thickness (S) have the highest coefficient (absolute value). This implies that the flake thickness has a strong influence on oil yield. From the model equation, it can be deduced that flake moisture content (M) and flake thickness (S) have a negative correlation with the oil yield percentage (Y), while solvent temperature (T) is positively correlated with the oil yield. This implies that oil yield increases with solvent temperature but decreases with flake moisture content and flake thickness. This is in agreement with the results in Table 3.

3.3 Model verification

Mean square error and root mean square error determined were found to be 0.084 and 0.290 respectively. These indicate the magnitude of error in the prediction. Bias and accuracy obtained were 0.173 and 0.030 respectively. The value obtained for bias is positive, indicating over-prediction by the developed model (Fowler *et al.*, 2012). A good estimator should be unbiased with even distribution of under and over-prediction which gives an overall bias of zero (Walther and Moore, 2005).

3.4 Model validation

Substituting different values of flake moisture content, flake thickness and solvent temperature into the model equation values of the oil yield were predicted. Figure 2 presents the plot of predicted against observed values (the value for the second experiment). The plot (Figure 2) has a coefficient of determination R^2 of 0.966. The results indicated that there is a strong agreement between the observed and predicted values.

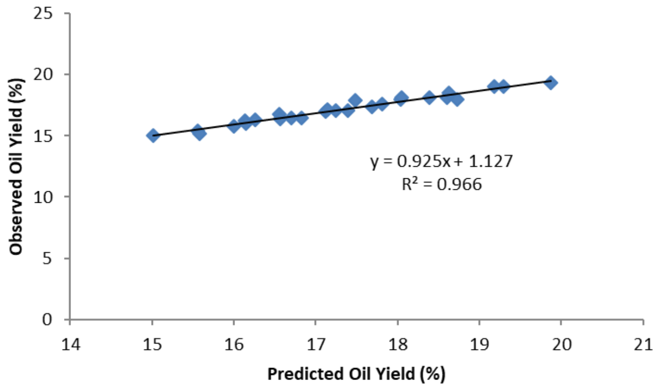

Figure 2. Graph of predicted against observed values

4. Conclusion

Using the data collected from solvent oil extraction from soybean with Soxhlet apparatus, effect of flake moisture content, flake thickness and solvent temperature on oil yield was determined and model equation was developed to predict oil yield at a given combination of the process parameters. All the factors under consideration significantly affect oil yield. The interaction between factors did not have a significant effect on oil yield. Highest oil yield was obtained at the highest solvent temperature, lowest flake thickness and lowest flake moisture content. The flake thickness has a strong influence on oil yield. The validation results of the developed model showed that there is good agreement between the predicted and the observed values. It is therefore recommended that the developed model be optimized to establish the optimum values of the evaluated factors.

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