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RESEARCH ARTICLE

Optimization of Crude Fish Oil Extraction Using Mechanical Screw Expeller Validated by Response Surface Methodology

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ABSTRACT

Fish has been identified as a source of essential oil, but there is sparse literature on the effect of processing factors on the characteristics of oil extracted from any fish species. Optimization of oil extraction with the aid of expeller was achieved by applying response surface methodology. The variables were choke clearance (1–3 mm), screw clearance (1–3 mm) and screw speed (50–70 rpm), while the responses were oil yields. Data obtained were analyzed at $P < 0.05$. Choke clearance, screw clearance and screw speed conditions significantly influenced all the responses at $P < 0.05$. The optimum oil extraction condition was 1 mm choke clearance, 1mm screw clearance and 70 rpm *screw speed*, which gave 22.422 % oil yield.

Keywords: Cost, benefit, packaging liners, plastic and wooden crates, postharvest losses quality

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INTRODUCTION

Tomato Fish is one of the most popular food items for human consumption throughout the world. Fish oil is the lipid fraction extracted from fish and fish by-products. Fish oils are excellent sources of long chain polyunsaturated fatty acids (omega-3 fatty acids), which are mainly composed of cis-5,8,11,14,17- eicosapentaenoic acid (EPA) and cis-4,7,10,13,16,19-docosahexaenoic acid (DHA). Omega-3 fatty acids have gained much more importance because of its health benefits. The functional and biological properties of omega-3 fatty acids include: prevention of atherosclerosis, protection against arrhythmias, reduced blood pressure, beneficial to diabetic patients, protection against manic depressive illness, reduced symptoms in asthma patients, protection against chronic obstructive pulmonary diseases, alleviate symptoms of cystic fibrosis, improving survival of cancer patients, reduction in cardiovascular disease and improved learning ability. At least two servings of fish products every week has been recommended by the American Heart Association to reduce the effect of cardiovascular diseases (Khoddami et al., 2009; Khoddami et al., 2012).

Fish oil is best obtained through extraction, a process that refers to an operation in which one or more components of a liquid or a solid phase substances are transferred to another liquid phase. To understand the principles of fishmeal and oil production, it is necessary to consider the raw material as composed of three major fractions: solids (fat-free dry matter), oil and water. The purpose of the process is to separate these fractions from each other as completely as possible, with the least

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possible expense and under conditions that render the best possible products. The extraction of oil from fish is accomplished through a unit operation of mechanical engineering known as expelling (extruding) process.

The selection of equipment for extraction process depends on controlling and influencing factors, which are responsible for limiting the rate of extraction. A number of variables need to be taken into account to optimize oil production using screw presses. For quality preservation, temperature is an important parameter. The friction inside the barrel generates heat which is passed on to the oil. For oil recovery and energy consumption, pressure is more interesting to monitor. There are four important factors to be considered as follows:

Speed: Higher screw speed means more throughput and higher residual oil content in the press cake since less time is available for the oil to drain from the solids. At higher speed the viscosity thus remains lower resulting in less pressure build-up. This again causes the residual oil content to be relatively high (Williams, 2005).

Restriction size: When the restriction size is reduced the pressure required to overcome the restriction increases. A resulting decrease in oil content causes increased viscosity of the paste and further pressure rise.

Moisture content: An optimal moisture level for oil expression is expected to exist. In case of rapeseed it is a moisture level close to 7 % (Bargale and Singh, 2000). For flaxseed the optimal moisture content is expected to be around 6 % (Zheng et al., 2005).

Cooking: Cooking causes increased cell wall rupturing thereby facilitating the outflow of oil. The oil point pressure decreases while pressure build-up increases due to increased viscosity in turn drastically increasing oil recovery.

Mathematical optimization is the selection of a best element (with regard to some criterion) from some set of available alternatives. More generally, optimization includes finding "best available" values of some objective function given a defined domain (or input), including a variety of different types of objective functions and different types of domains. Optimization problems are often multi-modal; that is, they possess multiple good solutions. They could all be globally good (same cost function value) or there could be a mix of globally good and locally good solutions. Obtaining all (or at least some of) the multiple solutions is the goal of a multi-modal optimizer (Battiti et al., 2008).

Some of the methods reported in modeling and optimization of oil recovery from oil bearing materials using software packages are linear regression (Young-Kyoo and Jeong 1995); linear program for optimization (Akinoso et al., 2006); response surface (Sivakumaran and Goodrum, 1987), discrete element (Raji and Favier 2004) and polynomial regression (Owolarafe et al. 2008). Concepts of fluid flow equations have also been reported to be applicable to model oil expression (Hamzat and Clarke 1993). Such theories include Tersaghi's equation for the consolidation of saturated oil to express the behavior of oil seed cake, Hagen Poiseulle equation for flow of fluids in pipe to express flow of oils through the pores of cell wall and Darcy's Law for fluid flow through porous media to express the flow of oil through the inter-kernel voids.

Response Surface Methodology (RSM) is a collection of mathematical and statistical techniques useful for the modeling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize this response. It differs from the procedure that involves the isolation of test variables and changing one variable at a time (Montgomery, 2005). The RSM is important in designing, formulating, developing, and analyzing new scientific studying and products. It is also efficient in the improvement of existing studies and products. The most common applications of RSM are in industrial, biological, and clinical science, social science, food science, and physical and engineering sciences. RSM tests several variables at a time, uses special experimental designs to cut costs, and measures several effects by objective tests. The most important difference is that a computer takes the experimental results and calculates models, using Taylor second order equations, which define relationships between variables and responses (Tripathi and Mishra, 2009; Akinoso and Raji, 2011). The relationships are quantitative, cover the entire experimental range tested, and include interactions if present. The models can then be used to calculate any and all combinations of variables and their effects within the test range. Thus, the thrust of this work is to study the effect of choke clearance, screw clearance and screw speed on the quantity of oil extracted from fish using Response Surface Methodology (RSM). This methodology reduces the number of experimental runs needed to provide sufficient information for experimentally acceptable results (Montgomery, 2005).

Justification of the study

Fish oil is usually obtained by various extraction techniques. Several studies on methods and conditions for fish oil extraction have been conducted in the past including solvent extraction, wet rendering, dry (steam) rendering and wet pressing methods. Extraction and purification of the lipids by conventional methods, such as hexane extraction, vacuum distillation, or conventional crystallization have the disadvantages of requiring high temperature processing which results in decomposition or degradation of the thermally labile compounds and/or employing toxic solvents having adverse health effects (Maqsood *et al.*, 2012). Therefore, various research efforts are currently focusing on developments in the field of oil extraction and purification technologies. The demands on these processing technologies for extracting and purifying the fish oil are that they are eco-friendly and able to provide high oil yields and to minimize the loss of nutrients and provide a high quality oil (Maqsood *et al.*, 2012). Although the emphasis has been on the marketability of the free fatty acid related health benefits of fish oil, it is known that fish body oil and fish liver oil contain other interesting components, such as vitamin A and D. With improved separation techniques and more gentle processing methods, these oils might play an even more important role in the pharmaceutical and food industry in the near future (Adeniyi, 2006). Many works have been done on fish oil production but literature has shown that little or no work has been done in terms of mechanical process of fish oil extraction

MATERIALS AND METHODS

Experimental Materials

For this research work, oily fish species, specifically, Atlantic mackery (*Scomber scombrus*) were obtained fresh from the market in Makurdi.

Experimental Design

I-Optimal design of response surface methodology was used as described by Montgomery (2005). Choke clearance, screw clearance and screw speed were the variables, while the oil yield was the response. Three levels of choke clearance, screw clearance and screw speed were used and 18 samples were generated (Table 1). Choke clearance were 1, 2 and 3 mm, screw clearance were 1, 2 and 3 mm, and screw speeds were 50, 60 and 70 rpm.

Determination of Percentage by Weight of Oil in Fish Sample

Total oil content was determined using methanol/chloroform extraction (Bligh and Dyer, 1959). A representative sample of fish tissue (50 g) was homogenized in a blender for 2 minutes with a mixture of methanol (100 ml) and chloroform (50 ml). Then 50 ml of chloroform was added to the mixture. After blending for additional 30 seconds, distilled water (50 ml) was added. The homogenate was stirred with a glass rod and filtered through a Whatman no.1 filter paper on a Buchner funnel under vacuum suction. 20 ml chloroform was used to rinse the remainder. The filtrate was allowed to settle to separate into the organic and aqueous layers. The chloroform layer containing the lipids was transferred into another beaker and 3 g of anhydrous sodium sulphate was added to remove any remaining water. The mixture was filtered through a Whatman no. 1 filter paper and chloroform was used to rinse the remainder. Finally, a known amount of Butylated hydroxytoluene (BHT) of about 0.02 g was added to the lipid solution as an antioxidant. The solution was then evaporated to a constant weight in a tared 100 ml round-bottom flask with a rotary evaporator at 40 °C. Results were expressed as grams of lipid per kilogram of samples.

$$\text{Fat content (\%)} = \frac{\text{Final weight of flask contents in grams}}{\text{Weight of sample}} \times 100\,\% \quad (1)$$

Fish Oil Extraction

Raw material preparation: Atlantic mackery (*Scomber scombrus*) was used in this experiment. The fish sample was thawed and cut into small pieces using stainless steel knife. Prior to analysis, the internal organs were removed and the fish was washed to remove the residual blood.

Pre-treatment: The material was heated to 90 °C for approximately 15 minutes. This process coagulates the proteins and disrupts the cell membranes thus allowing the leakage out of bound water and oil.

Oil extraction: The oil extraction was done mechanically with an oil expeller press. The expeller powered by a 5hp electric motor was set into operation and known weights (5000 g) of each prepared sample were fed into the machine through the feeding hopper. A continuous helical screw shaft conveyed, crushed, squeezed and pressed the material in order to extract the oil. The oil and water phases (containing water-soluble proteins as well) are separated from the solid phase (press cake). The fluid extracted and the press cake were collected and weighed separately.

Oil clarification: Clarification was done to separate the oil from its entrained impurities. The fluid extracted out of the press is a mixture of fish oil, water, cell debris, and non-oily solids. The fluid was allowed to stand undisturbed to settle by gravity so that the oil, being lighter than water, will separate and rise to the top. The clear oil was decanted into a reception container, sieved and heated to remove moisture in the oil.

Packaging and storage: Clean, dry sealed plastic bottles were used to package and store the oils and kept in a dark box to prevent rancidity.

Method of Evaluation

From the values obtained, the oil yield was calculated. The extraction performance of the machine was evaluated by expressing the oil extracted as a percentage of the total oil content of the fish samples. From the values obtained, oil yield was calculated according to Olaniyan and Oje (2011) as:

$$O_Y = \frac{100\,W_{OE}}{W_{FE}+W_{RC}}\ \% \qquad (2)$$

Where; W_{OE}=Weight of oil extracted, W_{FE}=Weight of fish extract, W_{RC}=Weight of residual cake.

Modeling and Optimization

The choice of variable levels was selected with respect to preliminary trials. The data obtained were subjected to ANOVA and regression model generated. The process was optimized using a commercial statistical package (Design Experts, *version 10*, Stat- Ease, Inc., Minneapolis, USA). *A response surface method with an I-Optimal design was used to optimize the oil yield.* Optimum process parameters were achieved by maximizing oil yield.

RESULTS AND DISCUSSION

Percentage by Weight of Oil in Fish Sample

The oil content was found to be 26.9 % in this study. According to Ackman (1989), fish can be grouped into four categories according to their fat content as lean fish (< 2 %), low (2-4 %), medium (4-8 %) and high fat (> 8 %). In terms of the oil content, the fish species examined can be considered to be in the high fat fish category.

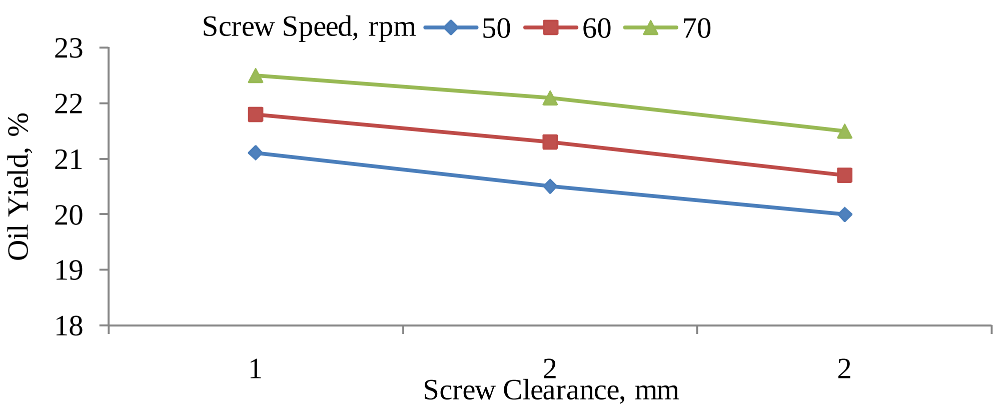
Oil Yield

The oil yield ranged from 17.8 % to 22.5 % as presented in Figure 1. From the results, it is evident that the highest yields were achieved in extraction carried out at all choke clearances and screw clearances with the screw speed of 70 rpm. During whole extraction, the oil yields at 70 rpm were the highest. The effect of screw speed on oil yield revealed that increase in screw speed enabled high oil expression to be achieved. A decrease of oil yield was observed when the choke clearance was increased from 1 mm to 3 mm at all levels of the screw clearance and screw speeds. The screw clearance affected oil yield at the various levels of choke clearance and screw speeds. As shown in Figure 1, when the screw clearance was increased from 1 to 3 mm, at choke clearance of 1 mm and screw speed of 70 rpm, oil yield decreased by only 4.4 %, when the choke clearance was increased from 1 to 3 mm, at screw clearance of 1 mm and screw speed of 70 rpm, oil yield decreased by 13.8 % and when the screw speed was increased from 50 rpm to 70 rpm, at choke clearance of 1 mm and screw clearance of 1 mm, oil yield increased by 6.6 %.

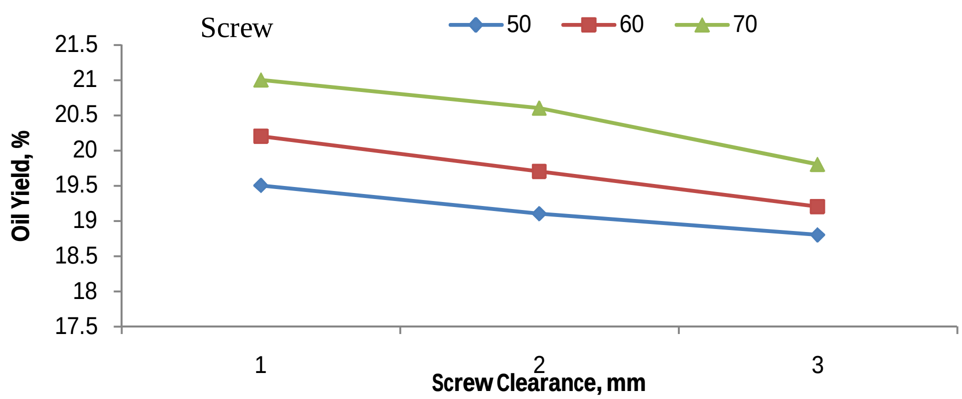
This is in agreement with the statement of Bamgboye and Adejumo, (2007) who reported that a reduction in speed of rotation of the shaft, for example, could reduce the oil yield, increasing the oil content in the cake and solids in the oil. Akinoso *et al*., (2009) while evaluating the effects of compressive stress, feeding rate and speed of shaft screw press on palm kernel oil yield observed same trend of increase in oil yield with increased speed. *Ezeoha and Akubuo, (2017)* while investigating the effects of speed of shaft screw press and choke gap on palm kernel oil yield observed same trend of increase in oil yield with decreased choke gap.

Models Using Response Surface Methodology (RSM)

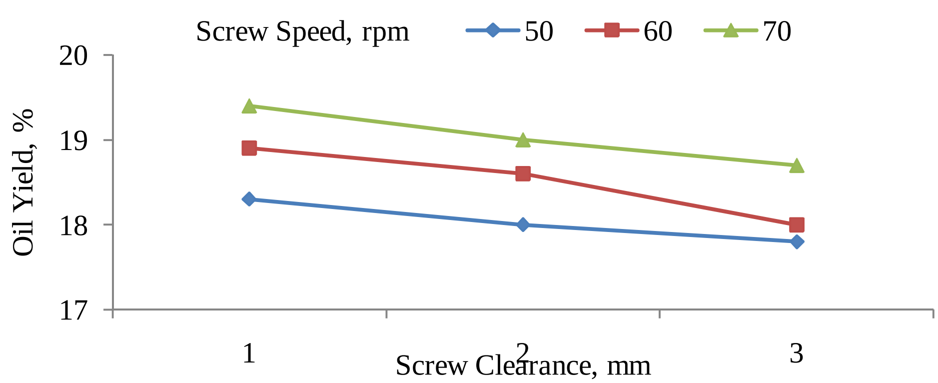
The data for the response variable Y (% oil yield) obtained from the 18 experimental points (Table 1) was used for a statistical analysis to optimize the process variables of choke clearance, screw clearance and screw speed. The oil yields ranged from 18 to 22.5 % with a mean value of 20.1 %. The best-fitting model was determined by multiple regressions with backward elimination. The un-significant factors and interactions were also removed from the model. The estimated regression coefficient of the response surface models for the oil extraction along with the corresponding coefficient of determination values (R^2) and lack of fit test are shown in Table 2. The relationship between the three variables (choke clearance, screw clearance and screw speed) and the important process response (extracted oil) for mechanical oil extraction process was analyzed using RSM. The study utilized RSM to develop a prediction model for optimizing the extraction of oil from fish. Significance model terms are desired to obtain a good output in a particular model.



(a) Choke Clearance of 1 mm



(b) Choke Clearance of 2 mm



(c) Choke Clearance of 3 mm

Figure 1. Mean yields of oil extraction at different levels of independent variables

The experimental conditions and the corresponding response values from the experimental design are presented in Table 1. The independent and dependent values were analyzed to obtain a regression equation that could predict the response within the given range. Using the obtained empirical data, a model equation to predict the effects of choke clearance, screw clearance and screw speed on oil yield was developed. Based on lack of fit test and coefficient of determination R^2, linear model was found to be suitable to express the oil yield (OY). The model equation for the fish oil extraction is as follows:

$$OY = + 19.52292 - 1.41476 X_1 - 0.45833 X_2 + 0.068172 X_3 \quad (3)$$

Where, X_1=choke clearance (mm), X_2=screw clearance (mm) and X_3=screw speed (rpm).

The adequacy of the response surface equations was demonstrated by a comparison between the experimental values and the predicted values based on a response regression analysis (Mirhosseinia *et al*., 2008). Model verification was carried out using combinations of variables at different levels within the experimental range.

Table 1: The experimental design and obtained values of the responses

Run	Choke clearance, mm	Screw clearance, mm	Screw Speed, rpm	Yield, %
1	2	1	70	21
2	2	3	60	19.2
3	1	3	50	20
4	2	2	70	20.6
5	1	2	60	21.3
6	2	1	50	19.5
7	3	1	60	18.9
8	3	3	70	18.7
9	2	2	70	20.6
10	3	2	70	19
11	1	2	60	21.4
12	2	3	50	18.8
13	2	1	60	20.2
14	1	3	70	21.5
15	1	1	70	22.5
16	1	1	50	21.1
17	3	2	50	18
18	2	3	60	19.5

Table 2: Analysis of variance (ANOVA) of the parameters for the response surface model

Source	Df	Sum of Squares	Mean Square	F- Value	p-value Prob > F	
Model	3	25.21	8.40	553.73	< 0.0001	Significant
A-Choke clearance	1	19.32	19.32	1272.99	< 0.0001	
B-Screw clearance	1	2.52	2.52	166.12	< 0.0001	
C-Screw Speed	1	5.40	5.40	356.04	< 0.0001	
Residual	14	0.21	0.015			
Lack of Fit	11	0.16	0.015	0.89	0.6203	not significant
Pure Error	3	0.050	0.017			
Corrected Total	17	25.42				

R^2: 0.9916, R^2 adj: 0.9899, R^2 pred: 0.9856, Adeq Precision: 80.099 %, CV: 0.61 %

The results of Analysis of Variance (ANOVA) for the RSM are shown in Table 2. The plot of experimental values of extracted oil (%) versus those calculated from Equation 3 indicated a good fit, as presented in Figure 2. Both the predicted vs. actual graph and the normal plot of residual graph shows model points close to the diagonal line, which indicate that the Linear model had a good level of prediction.

Under various conditions, the mean experimental value for oil yield was not significantly different ($p>0.05$) from the predicted value. The high correlation coefficients (0.9916) also confirmed that a close agreement between experimental data and the predicted values calculated using the models had been obtained. The closer the experimental and predicted results, the better they explain the adequacy of the regression equation (Rossa *et al.*, 2011). Colour differences in the fit plotted indicated the level of extracted oil which represents red as the highest extracted oil while narrow down to blue colour was the lowest extracted oil.

The coefficient of determination in the model defined R^2 expresses the degree of The results of analysis of variance (ANOVA) gave a coefficient of determination (R^2) of 0.9916; indicating the adequacy of the applied model. This implies that 99.16 % of the variations could be explained by the fitted model, indicating a reasonable fit of the model to the experimental data. That is, the model can be used to navigate the design space with fewer errors at 5 % level of significance. Joglekar and May (1987) suggested that, for a good fit of a model, R^2 should be at least 0.80. The difference between the Adjusted R-Squared of 0.9899 and Predicted R-Squared of 0.9856 shows the accuracy of prediction of the model and this difference should not be greater than 0.2 (*Stat-Ease,* 2017). For the linear model, the difference between Adjusted R-Squared and Predicted R-Squared is less than 0.2 which makes it an accurate predicting model.

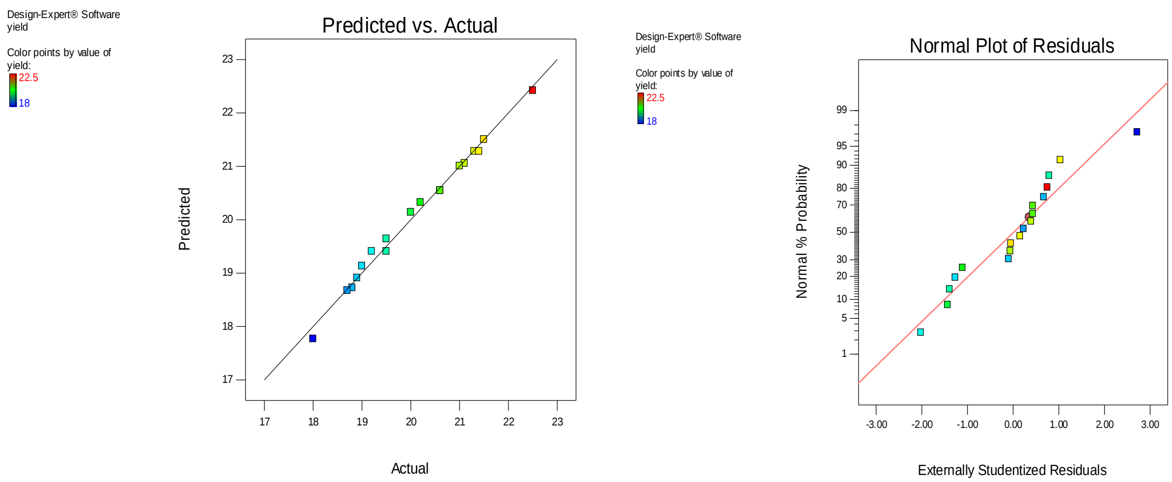


Figure 2. Diagnostic Analyses Graphs for Response Surface Linear Model for Oil Yield

The R^2 and adjusted R^2 were 0.9916 and 0.9899 respectively. These results imply that the response surface model can explain more than 98.99 percent of the variation in the response variables studied. Therefore, the R^2 values of the response models are sufficiently high, to indicate that the response surface model is workable and can be used for estimation of the mean response and the subsequent optimization stages. The lack of fit, which measures the fitness of the model, was found to be non-significant ($p>0.05$), suggesting that the number of experiments were sufficient for determining the effect of independent variables on percentage oil yield (Montgomery; 2001).

The probability (P-value) of the model significance was 0.0001 which is less than 0.05 and the Model F-value was high (553.73); implying that the model is significant. The Coefficient of Variation (CV) indicates the degree of precision with which the experiments are compared (Claver et al., 2010). It is a measure of reproducibility of the model. The CV of the model was obtained as 0.61 %. As a general rule, a model can be considered reasonably reproducible if its CV is not greater than 10 %. Therefore, the developed model could adequately represent the real relationship among the parameters chosen. The desirability lies between 0 and 1; and it represents the closeness of a response to its ideal value. If a response falls within the unacceptable intervals the desirability is 0 and if it falls within the ideal intervals or the response reach as to ideal value, the desirability is 1.

Effects of Independent Variable on Responses

Data obtained were analyzed at $P < 0.05$. The main effects; choke clearance, screw clearance and screw speed conditions significantly influenced all the responses at $P < 0.05$. No interactions were found to be significant at $p<0.05$ to the model. The response surface graph of 70 rpm screw speed is shown in Figure 3, to illustrate the main and interactive effects of the independent variables on the oil extraction. This shows the response surface plot of the effects of choke clearance and screw clearance at 70 rpm screw speed on fish oil extraction. The results indicated that choke clearance displayed a linear effect on the response and the extracted oil decreased with an increase of choke clearance. Screw clearance also displayed a linear effect on the response and the extracted oil decreased with an increase of screw clearance.

Therefore, efficient extraction of oil from oilseeds using the screw press involves a careful establishment of optimal pressing conditions for different oilseeds because the best process parameters are somewhat different for each oil-bearing seed and nut (Deli *et al*., 2011; Aloko *et al*., 2013). Oilseeds variables include moisture content, roasting or heating temperature, particle size, age, variety, maturity, cleanliness, and pretreatments (Bargale, 1997). Screw press variables include diameter of screw shaft, rotational speed, worm pitch, helix angle of worm, flight width, and flight length (Varma, 1998). Olayanju *et al*. (2006) reported that oil yield of beniseed varied with screw press rotational speed. The effect of screw press design parameters, especially compression clearance and speed has great effect on oil expelling efficiency (OEE) and energy consumption (Shankar *et al*., 2012). The most important screw press variables are screw press speed and screw choke gap (Sayasoonthorn *et al*., 2012).

Optimization of Process

Response optimizations were performed to measure the optimum levels of independent variables of choke clearance, screw clearance and screw speed, required to achieve the desired oil yield. The process providing the maximal oil yield involved press operation at low choke clearance, low screw clearance and high screw speed (Figure 3). To determine the exact optimum points for all the independent variables necessary to achieve the optimized condition, a numerical optimization was utilized.

The computer software package (Design Expert Version 10, Stat Ease Minneapolis, USA) used for the optimization analysis, gave nine possible optimum conditions (Table 3) with desirability ranging from 0.977 to 0.983. The best desirability (0.983) was achieved at choke clearance, screw clearance and screw speed of 1 mm, 1 mm and 70 rpm, respectively**.**

Table 3: Optimization desirability for optimum yield of oil extraction

	Solutions					
Number	Choke clearance, mm	Screw clearance, mm	Screw Speed, rpm	Yield, %	Desirability	
1	1.000	1.000	70.000	22.422	0.983	Selected
2	1.000	1.012	70.000	22.417	0.981	
3	1.000	1.000	69.898	22.415	0.981	
4	1.000	1.000	69.787	22.407	0.979	
5	1.000	1.033	69.997	22.406	0.979	
6	1.012	1.000	70.000	22.405	0.979	
7	1.000	1.045	70.000	22.401	0.978	
8	1.000	1.000	69.622	22.396	0.977	
9	1.000	1.059	70.000	22.395	0.977	

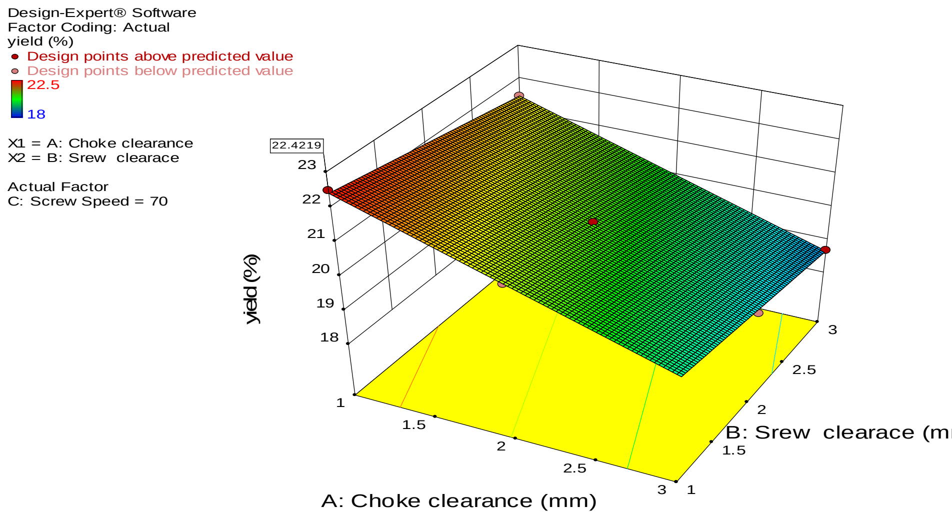


Figure 3: Response plot of Choke clearance and screw clearance against oil yield

From the model, under the optimum conditions, a maximum oil yield of 22.42 % was extracted at level of choke clearance 1 mm, screw clearance 1 mm and screw speed 70 rpm. The optimum oil yield (22.42 %) is lower than the maximum oil yield (22.50 %) obtained in this study. This is expected because of compromises to achieve the conditions. The Diagnostics Analyses graph for Response Surface Linear Model for Oil Yield is shown in Figure 2. The Response Surface graph (Figure 3) indicates highest point of achieving maximum yield by combining the factors.

CONCLUSION

The results obtained from this study showed that fish oil being an essential source of rich healthy oil, can be efficiently extracted by the use of screw press expeller machine. Choke clearance, screw clearance and screw speed influenced oil yield significantly at 95 % confidence level. Models developed showed that fish oil yields were influenced by choke clearance, screw clearance and screw speed. Nine possible optimum solutions were found with desirability ranging from 0.977 *to* 0.983. The best of the nine conditions was 1 mm choke clearance, 1mm screw clearance and 70 rpm screw speed, which gave optimum oil yield of 22.422 %.

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