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Optimization of oil extraction from coconut using response surface methodology

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ABSTRACT

In this study, oil extracted from coconut fruits was optimized using Response Surface Methodology (RSM). Effects of roasting temperature and roasting time on the yield and oil quality were investigated. Thirteen experimental runs applying a central composite design combined with RSM was employed. The parameters measured were oil yield, free fatty acid, colour, refractive index, specific gravity and pH. Statistical analysis with response surface regression showed that the oil yield, free fatty acid, colour, refractive index, specific gravity and pH of coconut oil were significantly ($p<0.05$) affected with roasting temperature and time. Based on response surface, optimum conditions were roasting temperature of 110 ^{o}C and roasting time of 17 min. Analysis of variance indicating that the model was adequate for representing the experimental data. The treatments resulted in oil yield ranging from (31.28 to 42.37%), free fatty acid (1.86 to 5.34%), colour (0.46 to 0.66 abs), refractive index (1.47 to 1.51), specific gravity (0.98 to 1.04) and pH (6.28 to 6.94). Oil extracted from coconut was successfully optimized using RSM. The regression models obtained has provided a basis for selecting optimum process variables for the recovery of oil using solvent extraction.

Keywords: coconut fruit, extraction, response surface methodology, oil yield, optimization.

INTRODUCTION

One of the most important and enduring source of oil is coconut palm. This fact is attributed to the ability of coconut (*Cocos nucifera L.)* to withstand abnormal climatic condition and its consistency in production and processing. Hence, it is the oilseed that has the highest productivity rate and investment return [1]. Coconut can be obtained in two forms based on their moisture content namely; wet coconut and dry coconut or copra; the oil can be obtained from both. However, copra is commonly used for oil extraction and the oil is used for food and cosmetic purposes. Rotaries and expellers are used for crushing the dry coconuts (known as copra) for recovery of oil. The fruits (coconut) are collected from the coconut palm and the palm kernels are later removed by crushing the palm fruits. The kernels can be consumed in the unprocessed form or in the roasted form; but most of them are used for the industrial production of vegetable oil. The production of coconut oil and its by-products, raw and fried cake, is an important source of income for women in Nigeria. The oil is used in the preparation of food and further processed as an ingredient in soap industries in a process known as saponification [2]. Some of the waste products are also dried to yield biomass that is deployed as fuel for cooking.

Oil from coconut kernel is very rich in glycerides of lower chain fatty acids. The oil is low in iodine value and this make coconut oil to be highly stable towards atmospheric oxidation. Also, it has a high saponification value, high saturated fatty acids content and it is a liquid at room temperatures of 27 °C. Various fractions of coconut oil are used as drugs [3]. The nutritional analysis of oils obtained from oilseeds shows that, it provides the calories, vitamins and essential fatty acids in the human diet in an easily digested form with low sterol. This has been the main reason why the rate of vegetable oil consumption is on the increase when compared with animal fat [4]. Apart from the nutritional benefit and industrial uses (cosmetics and soap making) of the edible vegetable oils, the relatively oil-free residues of the extracted seed are valuable by-products with many uses which includes; livestock feed, fertilizer and the manufacture of adhesives.

Separation of oil from oilseeds is an essential processing operation and the processing method employed in separating the oil from the seeds has a direct effect on the quality and quantity of protein and oil obtained from the oilseeds [5]. There are three fundamental approaches for the processing of oil from the palm kernels. These are: extraction method, mechanical oil expression, and the combination of the two methods. In this study, coconut oil was separated from the coconut fruits using extraction method. Extraction method involves bringing the extraction agent (solvent), an aqueous, supercritical CO_2; in contact with the oilseeds in order to dissolve the oil present in the seed. This produces a mixture of the dissolved oil (solute) and the extraction solvent. The mixture is separated by evaporation or distillation to recover the plain oil. The method is most popular in North America, it is found to be highly efficient with over 98% oil recovery. A single extractor can handle very large capacity up to 4000 tonnes per day [4]. Most of the oilseeds and nut are pre-treated by application of pressure and heat so as to liquefy the oil in the plant cells and facilitate its release during extraction [6].Standard conventional methods such as grinding, roasting, de-hulling, flaking, cooking, and steaming are applied on the seeds for preparing them for oil extraction. These operations are usually carried out to break apart the oilseeds constituents in order to facilitate the release of oil during extraction [7].

Response surface methodology (RSM) has been used by different authors for optimization of a process. RSM relates product properties by using regression equations that describe interrelations between input variables and product properties [8]. RSM can be used to reduce the number of experimental runs without affecting the accuracy of results while determining the interactive effect of different variables on the responses [8]. It is different from the procedure that involves the isolation of test variables and changing one parameter at a time [9]. RSM is an essential tool for designing, formulating, developing, and analyzing new scientific studies and product models. These models can then be used to calculate all combinations of variables and their effects within the test range. RSM has been widely used for food processing; some reported cases include; optimization of oil extraction from cocoa beans [10], neem [11] and groundnut kernel [12]. In this study, coconut oil was extracted from coconut fruits using hexane as solvent and the objective of this work was to determine the effect of roasting temperature and time on the yield and quality of coconut oil using RSM. This was targeted towards obtaining optimal benefits in terms of yields and quality of the refined coconut oil. Hence, focus on optimization of oil extraction from coconut fruit can enhance economic status of the seed.

EXPERIMENTAL SECTION

Preparation of materials

Sample of the coconut fruits was obtained from 'Oja-Oba' market in Surulere Local Government, Ogbomoso. The experiments were carried out in Food Science and Engineering Department Laboratory of Ladoke Akintola University of Technology, Ogbomoso, Oyo State, Nigeria. The variety of coconut used in this experiment was West Africa Tall Green, which is commonly found in our locality. It is marked by its tall trunk and medium sized nuts and its fruit is always green colour which turns brown at maturity. The sample was opened and the juice was squeezed out of it, and the meat was grated and ground using a vegetable slicer. Roasting temperatures were achieved by reported method [13]. The initial temperatures of the prepared samples were raised to equilibrium with roasting temperature. This was achieved by wrapping them in polythene bags and placed in oven at desired roasting temperature level. For each treatment combination, 100 g of ground sample were finely spread in a petri-dish and placed in a preset oven at different combinations of temperatures (60, 70, 80, 100 and 120 ^oC) and time (5, 10, 15, 20 and 30 min).

Experimental design

Response surface methodology was used to optimize the processing conditions. A five-level, two-variable design was adopted [9]. The two independent variables in this experiment were roasting temperature (X_1) and roasting time (X_2). Five levels of each of three independent variables were chosen for study, which were -1.41, -1, 0, +1 and +1.41. The experimental design pattern of the two independent variables is summarized in Table 1. A total of 13 level combinations were generated for the two independent variables. A roasting temperature of 80 ^oC and a roasting time of 15 min were chosen as the center points in the process optimization. The center point was repeated five times for the two-variable design (Table 2). Data analysis was done by using Design Expert Version 9.0.1.0 (Stat Ease Minneapolis, USA) software packaged to generate regression equations and analysis of variance was determined at $p<0.05$. The suitability of the adopted models was checked using coefficients of determination (R^2) and lack of fit test.

Table 1: Independent variables used in optimization study

Variables		Levels				
Codes	Actual	-1.41	-1.00	0.00	+1.00	+ 1.41
Roasting temperature (^oC)	X_1	60	70	80	100	120
Roasting time (min)	X_2	5	10	15	20	30

Oil yield

AOAC method [14] was used for oil extraction. Fifty grams of fried sample was packed in a Whatman's filter paper and placed in the Soxhlet extractor with N-hexane as the extracting agent (solvent). The process was left for six continuous hours of extraction, then the solvent was recovered by simple distillation and the residual oil was dried in an oven at a temperature of 65 ± 2°C for one hour. The obtained sample was cooled in desiccators before being weighed. Roasting, cooling and weighing processes were repeated until a constant dry weight was obtained. Oil yield in percentage was calculated using Equation 1.

$$\text{Percentage oil yield} = \frac{\text{weight of extracted oil}}{\text{weight of seed samples}} \times 100 \qquad (1)$$

Oil Quality Determination

The oil extracted was analyzed for the FFA, colour, refractive index, specific gravity and pH using [14]. Specific gravity was determined using empty pycnometer bottle that was weighed, then filled up with water and reweighed. The water was poured out and the bottle was dried. The bottle is filled up with the oil and the weight was measured. The specific gravity was then calculated using Equation 2.

$$Specific\ gravity = \frac{wt\ of\ bottle\ and\ oil\ sample - wt\ of\ empty\ bottle}{wt\ of\ bottle\ and\ water - wt\ of\ empty\ bottle} \qquad (2)$$

Table 2: The experimental design and obtained values of the responses

S/N	Coded		Actual		Responses					
	x_1	x_2	X_1	X_2	OY (%)	FFA (%)	CO (abs)	RI	SG	pH
1	-1.0	-1.0	80.0	5.0	31.75	3.12	5.58	1.50	0.99	6.57
2	1.0	-1.0	100.0	10.0	33.85	4.29	6.13	1.47	0.98	6.48
3	-1.0	1.0	60.0	15.0	35.44	2.67	6.72	1.49	0.97	6.94
4	1.0	1.0	70.0	10.0	36.14	1.86	6.55	1.50	0.99	6.58
5	0.0	1.4	120.0	15.0	42.37	5.34	4.82	1.51	1.04	6.69
6	0.0	-1.4	120.0	30.0	41.86	3.25	4.68	1.51	1.00	6.94
7	1.4	0.0	70.0	20.0	34.86	3.26	6.08	1.47	0.97	6.52
8	-1.4	0.0	80.0	30.0	31.28	4.69	5.38	1.49	0.99	6.28
9	0.0	0.0	80.0	15.0	33.85	4.28	6.12	1.45	0.98	6.48
10	0.0	0.0	80.0	15.0	33.74	4.21	6.13	1.48	0.97	6.46
11	0.0	0.0	80.0	15.0	33.85	4.28	6.17	1.48	0.98	6.45
12	0.0	0.0	80.0	15.0	33.62	4.26	6.15	1.44	0.97	6.43
13	0.0	0.0	80.0	15.0	33.55	4.22	6.12	1.46	0.96	6.48

where, X_1 is roasting temperature; X_2 is roasting time; OY is oil yield; FFA is free fatty acid; CO is oil colour; RI is refractive index; SG is specific gravity; pH is acidity of the oil.

Optimization

The optimal conditions of roasting temperature and time were obtained using a commercial statistical package (Design-Expert, Stat ease Inc., Minneapolis, USA). The optimization process was designed at finding the levels of roasting temperature and roasting time, which could maximize oil yield, minimize the free fatty acid while oil colour, specific gravity, refractive index and pH were kept within the range.

RESULTS AND DISCUSSION

Coconut oil quality

Oil yield: Table 2 shows the average values of three replicates for all the experimental runs. The oil yield extracted from 100 g of the coconut fruit ranged from 31.28 – 42.37%.Statistical result revealed that there was significant effect ($p<0.05$) of roasting temperature and time on response. This indicates variation of roasting temperature and time effect on oil yield. Figure 1 shows the response surface plot of interaction. The maximum percentage oil recovered was higher than 33.76% reported for un-roasted coconut by Saibaba *et al*. [15].

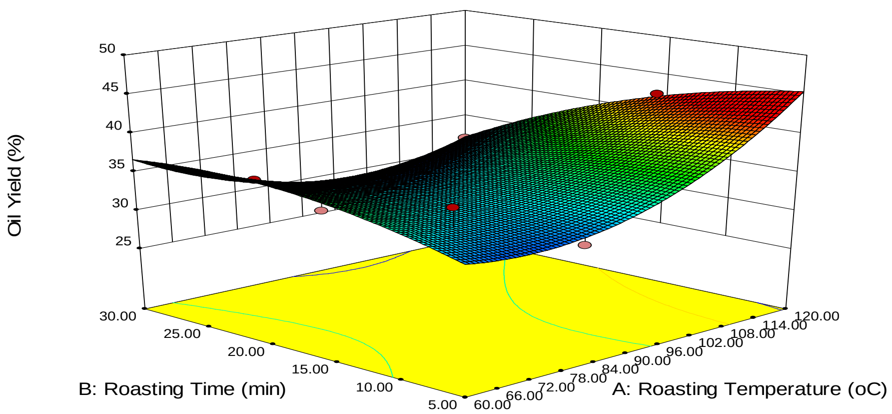

Figure 1: Plot of roasting temperature and time against oil yield

The result obtained agrees with findings that heating determine the percentage of oil yield from coconut [16]. Increase in oil recovered can be attributed to fractionating of intact oil bodies and rupturing of cellular structure as a result of roasting. Roasting of oilseeds breakdown oil cells, coagulation of the protein, adjust moisture contents of the meal to optimal value for extraction and reduce oil viscosity. All these effects allow easy flow of oils [17].

Free fatty acid (FFA): The FFA value of the oil ranged between 5.34 and 1.86% (Table 1). Heat treatments effect on FFA was significant ($p<0.05$). Response surface plot of the relationship is shown as Figure 2. It was observed that with decrease in roasting temperature and time, there was decrease in FFA. FFA is formed due to the hydrolysis of triglycerides and is responsible for off flavour development during storage. In appropriate conditions such as high temperature, moisture and presence of active lipase are responsible for the formation of FFA in fat containing raw materials or oils [6]. Moisture content and lipase activity in the oilseed could be controlled by thermal process such as heating [4]. Increase in FFA content of the oil with increase in roasting temperature and time may be due to thermal oxidative decomposition of oil during roasting.

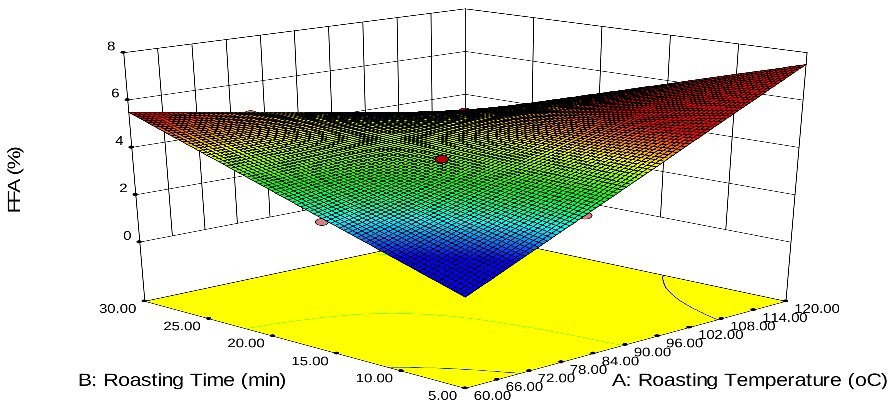

Figure 2: Response plot of roasting temperature and time against free fatty acid

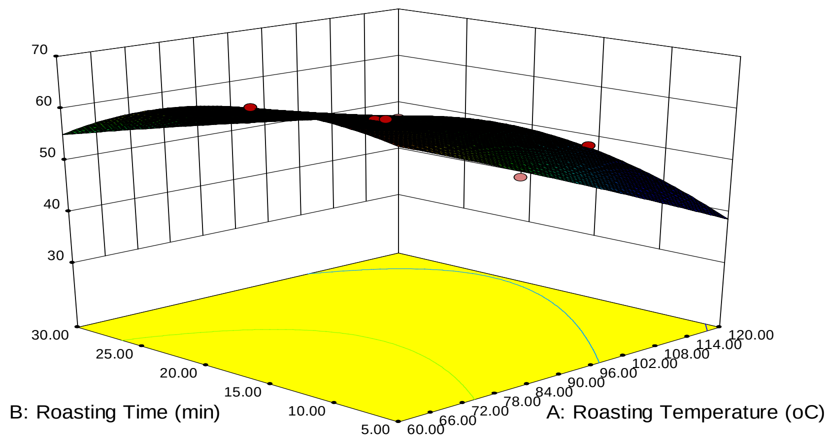

Figure 3: Response surface plot of roasting temperature and time against colour

Colour: Colour intensity of the oils was between the values of 0.47 and 0.67. The oil colour was significantly ($p<0.05$) dependent on heat treatments. The first attribute that determine consumer's preference for any food is colour [18]. Thus, the colour intensity may be an indicator for the presence of carotenoids. Response surface plot is shown in Figure3. Colour intensity increased with roasting temperature and time. Hafez *et al*. [19] also reported increase in colour intensity of roasted soybeans and this is similar to response plot shown in Figure 3. The increase in absorbance with an increase in roasting temperature and time could be attributed to colour formation by both non enzymic browning reaction and phospholipids degradation during roasting process [20].

Refractive index: The value of refractive index ranged between 1.47 and 1.51 %. Heat treatment method significantly ($p<0.05$) affected the oil refractive index. Increase in refractive index of the oil was observed with increase in both roasting temperature and time (Figure 4). The refractive index is a quality factor that allows a rapid sorting of oils suspected of adulteration; its value shows the level of oil purity. The value of refractive index obtained for coconut oil in this study is indicated that the oil has a high degree of purity. Refractive index is a function of roasting condition [21].

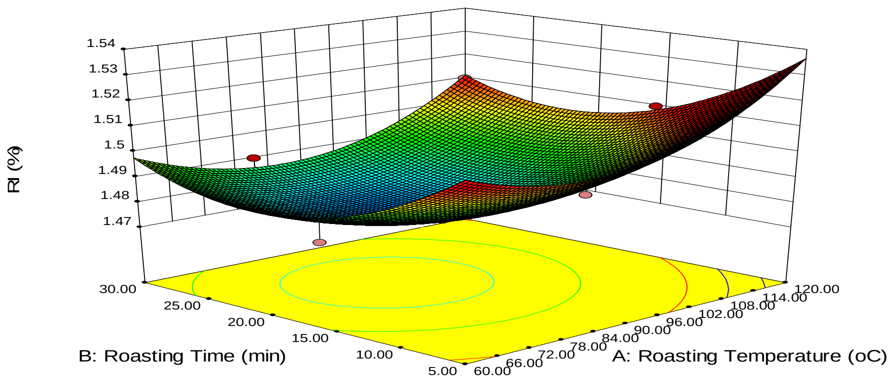

Figure 4: Response surface plot of roasting temperature and time against refractive index

Specific Gravity: The specific gravity of oil ranged from0.96 to 1.04. The data showed significant effect ($p<0.05$) of roasting temperature and time on specific gravity. The range of recorded specific gravity was comparable with 0.92 from literature [22]. Response surface plot of effect of roasting temperature and time is as showed in Figure 5. The specific gravity of oil is a diagnostic index when considering the quality or purity of oil. It is also used for assessing the weight of oil in bulk shipment or oil stored in large tanks, and for the design of piping and tanks for storage.

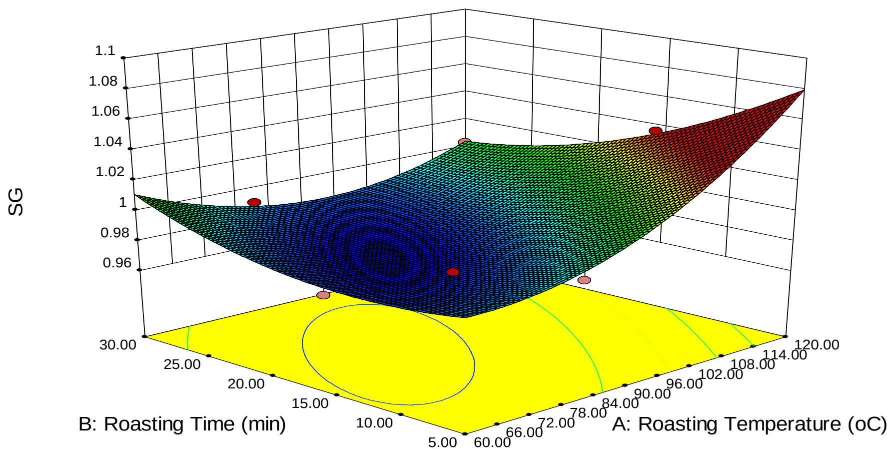

Figure 5: Response surface plot of roasting temperature and time against specific gravity

Oil pH: The pH value of coconut oil ranged between 6.28 and 6.94 (Table 1). The response surface plot for the effect of roasting temperature and time on pH of coconut oil (Figure 6) showed decrease in acidity level of the oil with increase in both roasting temperature and time. Analysis of the data using statistical tools showed significance ($p<0.05$) influence of roasting temperature and time on pH values of the oil. Recorded pH presents a reasonable stability on the oil. Low level of acidity indicates existence of low fatty acid, and improved nutritious value.

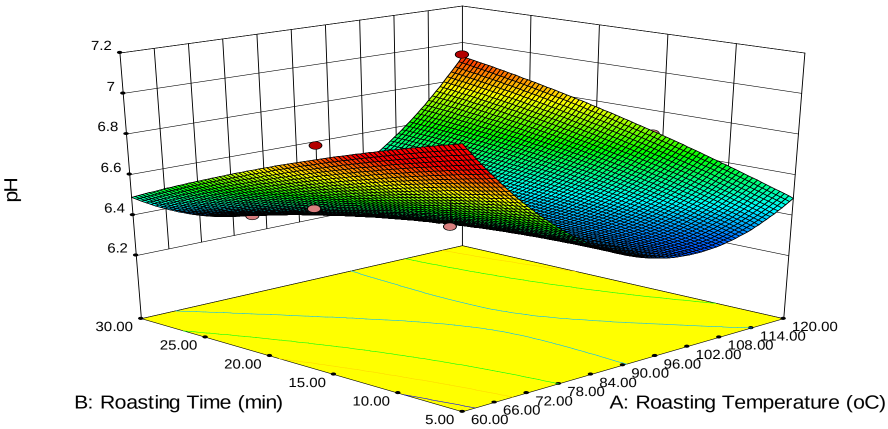

Figure 6: Response surface plot of roasting temperature and time against pH

Models

Empirical models were developed to describe the relationship between roasting conditions and coconut oil quality. The model performance was reasonably good. From the empirical data obtained, standardized equations for predicting the effects of roasting temperature and time on; oil yield, FFA, colour, refractive index, specific gravity and pH of the oil generated are respectively modeled and presented as Equations 4 to 9. As a result of the outcome of the lack of fit test and the coefficient of determination R^2, quadratic model was found to be appropriate for oil yield (OY), refractive index (RI), colour (CO), specific gravity (SG), pH while 2FI was found to be suitable to express the FFA content of the oil, where X_1 is roasting temperature (^oC) and X_2 is roasting time (min).

$$\mathrm{OY} = +33.04 + 1.97X_1 - 2.372X_2 + 4.80X_1^2 - 2.06X_1^2 - 4.33X_1X_2 \quad 3$$
$R^2 = 0.9173$, P – value = 0.0034

$$\mathrm{FFA} = +4.33 + 1.05X_1 + 0.071X_2 - 2.22X_1X_2 \quad 4$$
$R^2 = 0.7607$, P – value = 0.0073

$$\mathrm{CO} = +58.12 - 8.70X_1 - 0.44X_2 - 0.45X_1^2 - 6.02X_1^2 + 4.62X_1X_2 \quad 5$$
$R^2 = 0.9784$, P – value = 0.0001

$$\mathrm{RI} = +1.48 + 9.056\mathrm{E} - 003X_1 - 0.010X_2 + 0.018X_1^2 - 0.019X_1^2 - 2.367\mathrm{E} - 003X_1X_2 \quad 6$$
$R^2 = 0.8985$, P – value = 0.0061

$$\mathrm{SG} = +0.98 + 0.022X_1 - 0.013X_2 + 0.025X_1^2 + 0.016X_1^2 - 0.025X_1X_2 \quad 7$$
$R^2 = 0.9295$, P – value = 0.0021

$$\mathrm{pH} = +6.39 - 0.037X_1 - 0.034X_2 + 0.40X_1^2 - 0.037X_1^2 + 0.25X_1X_2 \quad 8$$
$R^2 = 0.9481$, P – value = 0.0009

Optimum parameters

The coefficients of determination (R^2) for the responses, oil yield, free fatty acid, colour, refractive index, specific gravity and pH were 0.92, 0.76,0.98, 0.90, 0.93 and 0.95, respectively. The coefficients of determination were high for response surfaces and indicated that the fitted models accounted for more than 70% of the variance in the experimental data. That is, the models can be sufficiently used to steer the design space with fewer errors at 5% level of significance. The maximum oil yield (42.37%) obtained in this study is greater than 33.76 % reported for un-roasted coconut by Akpan *et al*. [16].

CONCLUSION

The results of this study suggested that the oil extracted from coconut, when refined, can be used as vegetable oil, since the chemical properties of coconut oil has been shown to fall within the specified range for vegetable oils. Roasting temperature and time combinations influenced both the yield and the quality significantly at 95% confidence level. Models were developed for predicting the effect of roasting temperature and roasting time on the OY, FFA, CO, RI, SG and pH of the oil obtained from coconut fruits by extraction. The models had good fits and four possible optimum solutions were found ranging from 0.50 to 0.64 desirability. The best of the four conditions was roasting at 60 ^{o}C for 16 min which gave 36.59% oil yield, 3.08% free fatty acid, 0.67 abs colour, 1.48 refractive index, 0.98 specific gravity and 6.84 pH. The optimum process condition produced a comparatively high OY with satisfactory colour and good quality storage stability.

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