PROCESS OPTIMIZATION OF OIL EXPRESSION FROM WATERMELON (*CITRULLUS LANATUS*) SEEDS

Busari, R. A., Fadeyibi, A., Adebayo, K. R., Olapipupo, R. F., Muhammed, F. A. and [2]Balogun, A. L

[1]Food, and Agricultural Engineering Department, Kwara State University Malete, Nigeria
[2]Department of Agricultural and Bio-Environmental Engineering, Kwara State Polytechnic,Ilorin, Nigeria
Corresponding e-mail: rasheed.busari@kwasu.edu.ng

ABSTRACT

Watermelon fruit contains large quantities of seeds and these seeds are under-utilized. It contains reasonable amount of oil (22%) which if expressed will serve as vegetable oil for human consumption or biodiesel for powering agricultural machines and boost the income of the farmers. The main objective of this research work is to study the interaction effect of operating parameters on the mechanical oil expression from the seed. The variables considered include applied pressure (85.00, 90.00, 95.00, 100.00 and 105.00 kN/mm^2), roasting temperature (70.00, 80.00, 90.00, 100 and 110 °C) and moisture content (6.00, 8.00, 10.00, 12.00 and 14.00 %). A total sum of 20 batch experiments were carried out and the maximum oil yield was 13.49% (at corresponding moisture content of 6.00%, roasting temperature of 85.00^{o}C and applied pressure of 105.00 kN/mm^2 respectively) and minimum oil yield obtained was 9.41%.(at corresponding moisture content of 14.00%, roasting temperature 80 °C and applied pressure of 85.00 kN/mm^2 respectively). While the optimum oil yield of 12.42% was obtained from the expression at corresponding moisture content of 6.00%, roasting temperature of 80.00^{o}C, and applied pressure of 105.00 kN/mm^2. The result showed that the three independent variables had significant effect on oil yield and regression model equation was developed to predict the oil yield from watermelon seeds at known variables.

Keywords: Watermelon seeds, Optimization, Roasting temperature, Moisture content, Applied pressure.

1. Introduction

The seeds of watermelon are underutilized in Nigeria. The juice from watermelon fruits is consumed on daily basis, while rind and seeds are major solid wastes (Poornima *et al.,* 2019). Watermelon fruit contains large quantities of seeds and the oil content 22% as reported by Poornima *et al.,* (2019). Figures 1, 2 and 3 show typical watermelon fruit, watermelon pulp and seeds embedded in it and dry watermelon seeds, respectively. The demand for energy and vegetable oil are increasing at a high rate due to increasing world population. Vegetable oils are very important ingredient for both domestic cooking (for feeding, bakery, margarine, and food industry) and non-food industry (production of paints, detergents, fatty acid, special varnishes, and cosmetics products (Koocheki *et al*., 2007). Presently, oil can be expressed using different methods such as solvent oil extraction, traditional or local methods and mechanical extraction methods (screw or hydraulic press). Although existing chemical methods of oil extraction (solvent) is characterised with high yield of oil recovery and also associated with several demerit such as high cost, retainment of solvent in oil, skill labour is required and not easily accessible to local farmers (Liauw *et al*., 2008). To overcome fore mentioned problems the best method of oil expression is mechanical expression method, as this method can be easily accessible to local farmers and affordable. The percentage oil recovery from oil-bearing seeds dependent largely on the quality of the oilseeds and method of oil expression. At the same time, the expression process needs to be well managed in order to get optimum oil recovery from the oilseeds. Also, there are several parameters that can be manipulated during expression process to increased oil yield. These variables that can be manipulated include the seeds moisture content, the size of particles, applied pressure, roasting durations and roasting temperature (Olaoye and Busari, 2017). The effect of these variables has been studied by a good number of researchers (Baryeh, 2001; Southwell and Haris, 1992; Ajibola *et al.,* 1990). Also, Bamgboye and Adejumo (2011) reported that particles size, roasting temperature, roasting durations, moisture content, operating pressure and methods of oil expression affects the yield of oil during extraction. For expression process, maximum oil recovery and less residual cake is expected. Therefore, it is necessary to control the above-mentioned variables.

Adekola (1992) reported that oil yield of coconut seeds was found to increase with increase in roasting temperature levels of 60℃, 90℃ and 120℃. An increase in roasting temperature lower the oil viscosity and improves the flow of oil. Hence, he reported that at 120℃, roasting duration above 15 minutes most especially at roasting duration of 25 minutes, losses of oil yield of about 12.3% was recorded. Southwell and Harris (1992) work on sunflower kernels and revealed that unheated sunflower kernels turnout low oil yield when compared with conditioned and heated samples. Hence, the experiment was carried out to investigate optimum oil recovery in mechanical extraction (hydraulic) under the influence of applied pressure, moisture content and roasting temperature of watermelon seeds.

Fig. 1: Watermelon fruit

Fig. 2: Watermelon pulp and seeds embedded

Fig. 3: Dry watermelon seeds

2. Materials and Methods

The investigation on influence of pressure, roasting temperature, and moisture content on hydraulic expression of oil from watermelon (*Citrullus lanatus*) seeds was carried out in the Food and Agricultural Engineering Department laboratories of Kwara State University Malete, Nigeria. The seeds of watermelon were purchased from Ipata market in Ilorin metropolis.

2.1 Preparation of Sample

The initial moisture content of the seed was 7.5% and further sun dried to 6% which is the safe using Moisture Meter (KT100S; measuring range of 5 -35%). 250g of watermelon seeds sample were conditioned to the varied moisture content levels by adding calculated amount of distilled water. The relationship used for calculated water was presented in eqn. 1 as reported by Poornima *et al*., (2019). Thereafter, the seeds were thoroughly mixed with calculated amount of water and then sealed in different plastic bags. The samples were kept at adequate temperature in a refrigerator for 48 hours to enable the moisture to distribute uniformly in the samples. Before starting of the experiment, the seeds samples were roasted to varied temperature by roasting the samples on portable gas cylinder. Samples were eventually subjected to varied pressure using hydraulic press.

$$W_2 = W_1 \frac{M_1 - M_2}{100 - 1} \qquad 1$$

W_2 = Mass of distilled water added to the sample (kg)
W_1 = Initial mass of the sample (kg)
M_1 and M_2 = initial and desired moisture content of the sample (w.b %).

2.2 Design of experiments

The experiments were carried out by varying roasting temperature, applied pressure and moisture content. The parameters and levels were fixed based on information from the literature and trial experiments as reported by Ajala *et al.,* (2016) and Olaoye and Busari (2017). Table 1 shows the experimental ranges and levels of independent variables for the design experiment. The experimental design contains 20 treatments which were the combinations of roasting temperature, applied pressure and moisture contents using central composite design (CCD) of design expert 6.0.8.; Sample size: 250g of seeds per treatment per replication.

Table 1: Experimental Range and Levels of Independent Variables

Variable Codes	Variables	Unit	Levels and Range				
			-α	-1	0	+1	**+α**
X_1	Applied Pressure	kN	85.00	90.00	95.00	100.00	105.00
X_2	Moisture Content	%	6.00	8.00	10.00	12.00	14.00
X_3	Roasting Temperature	°C	70.00	80.00	90.00	100.00	110.00

2.3 Oil extraction from watermelon seeds

The oil was expressed from watermelon seeds using a hydraulic press (capacity) and developed piston-cylinder rig. The piston-cylinder rig, which was developed is made up of a compression piston, a press cage cylinder, a supporting platform, and an oil collecting pan. The piston serves as the pressing ram and it distsributes pressure received from the hydraulic press evenly on the roasted oilseed sample in the press cage cylinder. The remnant obtained from this hydraulic press can be used for preparing organic manure. The obtained oil was filtered to remove impurities and allowed to be settled and measured, the sample of extracted oil from watermelon seed is presented in fig. 4 while the oil yield was calculated using the relationship in eqn. 2.

$$Y = \frac{W_1 - W_2}{W_1} \, x \, 100 \qquad 2$$

where,
Y = oil yield
W_1 = weight of un-milled watermelon seed.
W_2 = weight of sample cake (after milling)

$$E_e = \frac{WoE}{nWus} \qquad 3$$

where,
Ee = the extraction efficiency (%)
n = the oil content of the seed
WoE = the weight of sample expressed
Wus = the original weight of sample

2.4 Physico-chemical Properties of Watermelon oil Seeds

The physico-chemical properties of expressed oil were examined using recommended standards and methods. The details of the properties examined, and the methods followed for their determination are as follows.

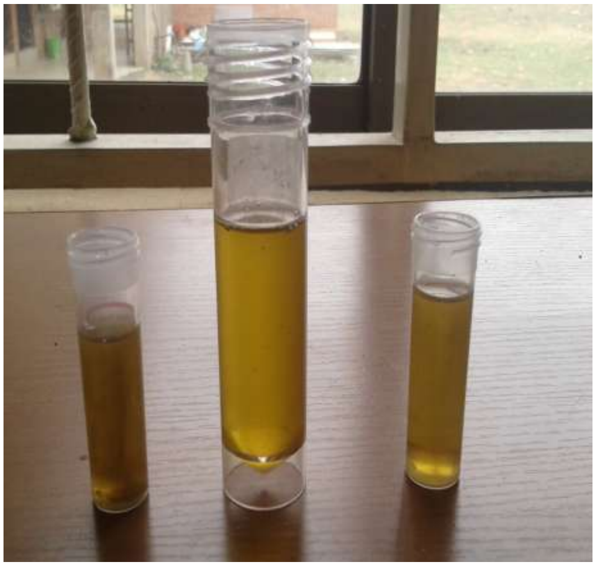

Fig. 4: Sample of Extracted oil from Watermelon Seeds

2.4.1 Determination of Kinematic Viscosity

The samples viscosity was measured following the ASTM D445 standard procedure at 40°C using a calibrated digital Viscometer (NDJ – IB Rotational Viscometer; Shanghai Chamgji) the procedure was highlighted as follows:

i. About 200 ml of watermelon oil was measured into a beaker,
ii. the protecting frame was coupled on the viscometer,
iii. the spindle selected for the sample was coupled into linking screw,
iv. the oil sample was placed directly under the machine spindle,
v. the lifting knob was lowered, and the spindle encounter oil sample till the surface was flushed with the level of the spindle,
vi. suitable speed was selected (30 rpm) from various available speeds,
vii. the machine was run for about 10 min. and the displayed reading was recorded

2.4.2 Determination of Specific Gravity and Density

Both the density and specific gravity of watermelon oil were measured using Density meter (Rudolph Research Analytical: DDM 2911 Automatic Density Meter). The procedures were highlighted as follows:

i. the machine was on; it took some minutes to complete booting,
ii. a syringe was used to inject the watermelon oil sample into the front nozzle port with a lure tip syringe,
iii. the video view was checked to ensure the samples injected into the machine is bubble free,
iv. the scanning camera provided an excellent view of the entire sample cell,
v. the measured values were displayed on the screen of machine.

2.4.3 Determination of pH

The pH value was determined using digital pH meter. The covering cap of the digital meter was removed; the pH electrode glass bulb was cleaned with wet handkerchief and highlighted procedures were followed:

i. the hand-held digital pH meter was switch on
ii. the glass bulb was inserted into the specimen of about 20ml, it was stirred and waited until the displayed reading was stable
iii. the displayed value was recorded for the sample

3. Results and Discussion

The outcome of the mechanical oil expression (hydraulic) from watermelon seeds is presented in Table 2. The percentage oil yield for each batch of the experiments were determined using Eqn. 2. The differences in the percentage oil yield values are indications that the independent parameters had reasonable effect on the oil yield of watermelon (Busari *et al.,* 2019). The predicted oil yield from the software compared well with the yield obtained. The maximum oil yield was 13.49% (at corresponding moisture content of 6.00%, roasting temperature of 85.00ºC and applied pressure of 105.00 kN/mm^2 respectively) and minimum oil yield obtained was 9.41%.(at corresponding moisture content of 14.00%, roasting temperature 80 °C and applied pressure of 85,00 kN/mm^2 respectively). The result showed that the three independent variables were relevant to the watermelon seed oil yield. While the optimum oil yield of 12.42% was obtained from the expression at corresponding moisture content of 6.00%, roasting temperature of 80.00ºC, and applied pressure of 105.00 kN/mm^2.

Table 2: Experimental data for oil yield

Std	Run	A: Moisture Content (%)	B: Pressure kN/mm^2	C: Roasting Temperature (°C)	Oil Yield (%)	Extraction Efficiency (%)
13	1	10.00	95.00	90.00	12.46	56.64
6	2	14.00	85.00	120.00	10.42	47.36
11	3	10.00	90.00	100.00	11.29	51.31
12	4	10.00	100.00	100.00	11.20	50.90
8	5	14.00	105.00	120.00	11.04	50.18
15	6	10.00	95.00	100.00	12.02	54.63
17	7	10.00	95.00	100.00	12.02	54.63
16	8	10.00	95.00	100.00	12.03	54.68
5	9	6.00	85.00	120.00	10.01	45.50
20	10	10.00	95.00	100.00	12.02	54.63
19	11	10.00	95.00	100.00	12.00	54.54
7	12	6.00	105.00	120.00	11.37	51.68
3	13	6.00	105.00	80.00	13.49	61.32
14	14	10.00	95.00	110.00	11.61	52.77
4	15	14.00	105.00	80.00	10.61	48.23
2	16	14.00	85.00	80.00	9.41	4277
18	17	10.00	95.00	100.00	12.00	54.54
9	18	8.00	95.00	100.00	12.02	54.63
1	19	6.00	85.00	80.00	10.10	45.90
10	20	12.00	95.00	100.00	9.96	45.27

3.1 Mathematical Model Equation for Oil Expression

The oil expressions were performed according to the Central Composite Design experimental plan and dependent variable (oil yield) as presented in Table 2. The regression model equation developed from the software for watermelon oil yield with single, quadratic, and interactive variables observed B, C^2, and AC are directly proportional to the oil yield while A, C, A^2, B^2 AB and BC with negatively coefficient indicates an indirect proportionality with the oil yield. The model obtained is given in Equation 2 as

$$Y = 11.77 - 0.531A + 0.7676B - 1406C - 0.3663AB + 0.4562AC - 0.3263BC - 2.06A^2 - 1.04B^2 + 2.12C^2 \quad 4$$

3.2 Statistical analysis of expressed watermelon oil

The regression analysis gives an F-value of 6.27 and p-value of 0.0041 which implies that the model is significant at $p \leq 0.05$. Table 3 shows the ANOVA for the response (oil yield) surface quadratic model. The value of the term A (0.0171), B (0.0021) show that the model are significant at $P \leq 0.05$ while C, A^2, B^2 C^2 AB, AC and BC, are not significant with P values above 0.05. Among all the model terms it was observed that applied pressure has the highest influence in the regression model with F-value of 17 follow by moisture content with F value of 8.16. This implies that applied pressure plays a significant role in the developed model follow by moisture content. The significant of the developed model was also tested with R^2 and Adj. R^2 value. The obtained R^2 and Adj.

R^2 values are 0.8495 and 0.7140 respectively. The fitness of a model is always measured with coefficient of determination and the value should not be less than 0.80 as reported by Akintude, *et al.* (2015). Yuan *et al.* (2008) reported that higher value of the correlation coefficient signified an excellent correlation between the independent parameters. More so, a relatively lower value of the coefficient of variation (CV = 4.78%) indicates a better precision and reliability of the experiments carried out (Yuan *et al.,* 2008).

Table 3: ANOVA for Response Surface Quadratic Model for Watermelon Oil Yields

Source	Sum of Squares	DF	Mean Square	F-value	p-value	
Model	16.62	9	1.85	6.27	0.0041	Significant
A-Moisture Content	2.4	1	2.4	8.16	0.0171	
B-Pressure	5.01	1	5.01	17	0.0021	
C-Roasting Temp	0.168	1	0.168	0.5703	0.4676	
AB	1.07	1	1.07	3.64	0.0854	
AC	1.67	1	1.67	5.65	0.0388	
BC	0.8515	1	0.8515	2.89	0.1199	
A^2	0.7923	1	0.7923	2.69	0.132	
B^2	0.202	1	0.202	0.6858	0.4269	
C^2	0.8384	1	0.8384	2.85	0.1225	
Residual	2.95	10	0.2946			
Lack of Fit	2.95	5	0.5891	3927.03	< 0.0001	Significant
Pure Error	0.0007	5	0.0001			
Cor Total	19.57	19				

3.3 Effect of Operating Parameters on percentage oil yield from Watermelon seeds

The interaction effects of moisture content applied pressure, and roasting temperature on the oil expression yield were investigated using the interactive and 3D surface plot of response surface methodology. It was noted from fig. 5 that an increase in oil yield was observed when the applied pressure increases while the same thing was observed as the moisture content increases until the moisture is above 12% before it has negative effect on oil yield. The oil yield was maximum at 11.50 % (Fig 5) when the applied pressure was 100.00 kN/mm^2 and moisture content of 12.46 % while the minimum yield was achieved at 10.20 % when applied pressure was 85.00 kN/mm^2 and the moisture content was 6.00 %. This implied that as applied pressure increases more cell walls were squeeze and resulted into more oil yield from watermelon seeds.

Fig. 6 shows the interaction effects of roasting temperature (X_3) and moisture content (X_2) on watermelon oil yield keeping applied pressure constant. Maximum oil yields were obtained at high roasting temperature of about 100.00 °C and low oil yields were obtained at low roasting temperature (80.00 ºC and temperature above 100.00 °C). Martínez *et al*., (2013); Terigar *et al*., (2011) and Costa *et al*., (2014) investigated that roasting temperature had significant influence on oil yield. At temperature above 100.00 °C, oil yield decreases as the moisture content increases (above 12.00%) while at lower roasting temperature of 80.00 ºC oil yield increases as moisture content increases from 6.00 – 12.00 % and decreases as moisture content increases above 12.00 %. This implied that maximum oil yield would be obtained at moisture content of 6.00 – 12.00 % while extracting at roasting temperature of 100.00 °C.

As presented in Fig. 7, increase in operating pressure caused slight increase in oil yield (85.00 – 105 kN/mm^2) as the roasting temperature was increased from 80 – 120 °C. This is similar to research carried out by Rezzoug *et al.* (2005) they reported that applied pressure is one of the significant variables that have effect on expression yield and the expression yield of the various essential oil compounds. This indicates that higher roasting temperature and applied pressure favour oil yield from watermelon seeds. It can be observed from the analysis of variance (ANOVA) results (figures 2 – 4) that all the variables under consideration had significant effects on oil yield from watermelon seeds.

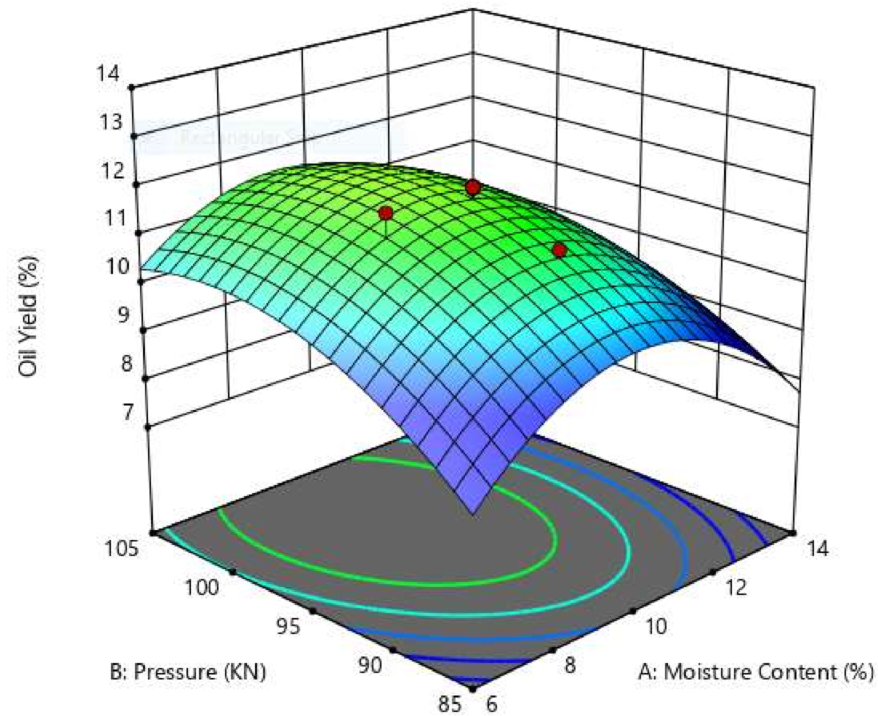

Fig.5: Response Surface Plot on Effect of Applied pressure and Moisture content on oil yield

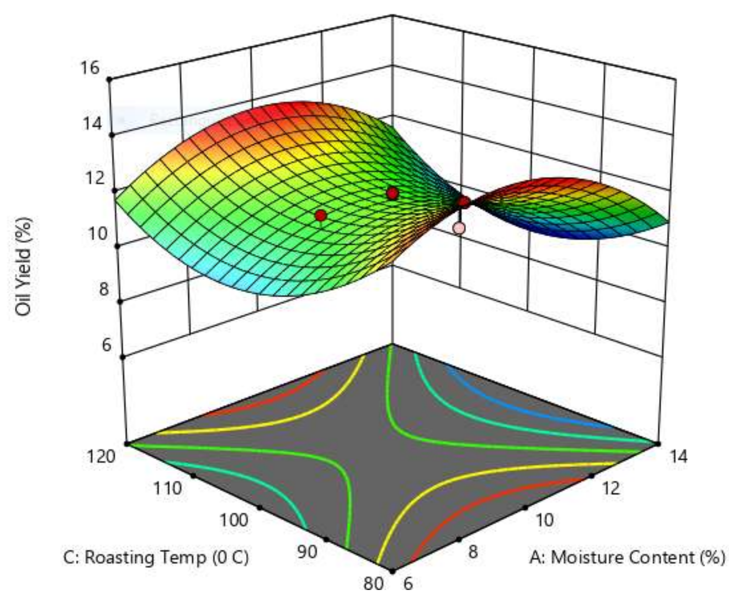

Fig.6: Response surface plot on effect of roasting temperature and moisture content on oil yield

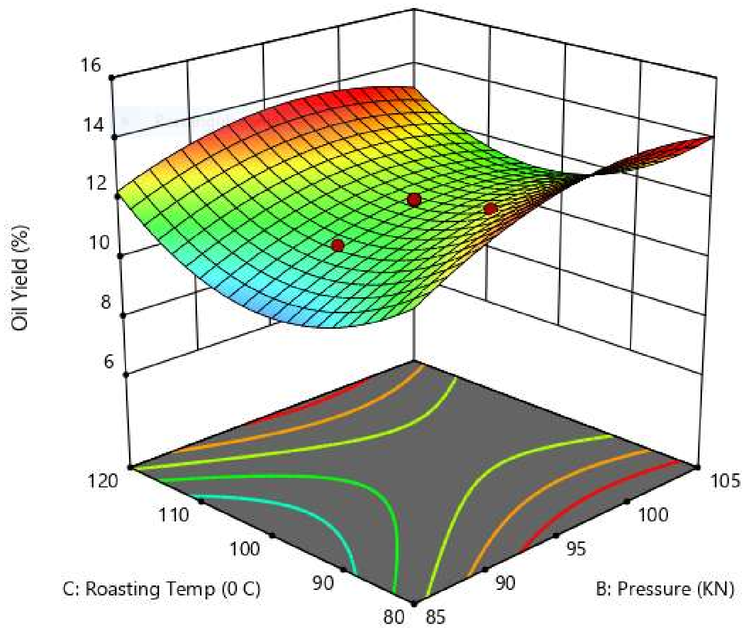

Fig. 7: Response surface plot on effect of roasting temperature and Applied pressure on oil yield

3.4 Process Optimization and Validation

The main objectives of this research work is to determine the optimum process variables at which the oil extraction will be maximum. In the analysis, the target criteria were set, in ranges and response was set to be maximum. The optimum oil yield was validated using the data generated from the laboratory experiment carried out. To validate the accuracy of the model, oil expression was carried out under the suggested optimum conditions. Experimental oil yield was found as 12.40 % while predicted oil yield was 12.51 %. The percentage error was recorded to be 0.89 which confirmed the validity of the model equation developed. Table 4 presents the optimum conditions for oil expression, experimental and predicted value, and percentage error. While the relationship between experimental and predicted values of oil yield from watermelon seeds as shown in Figure 8.

Table 4: Optimum conditions for oil expression and percentage error

Optimum conditions for oil expression			Experimental Value (%)	Predicted Value (%)	Percentage Error (%)
Moisture Content (%)	Roasting temperature (°C)	Pressure (KN/mm^2)			
6.00	80	105	12.40	12.51	0.89

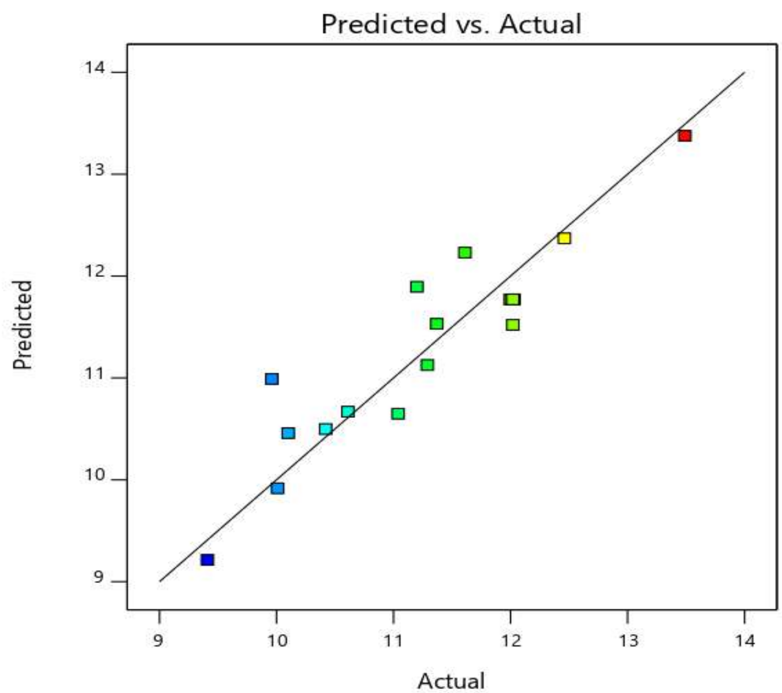

Fig. 8**:** Predicted oil yield Vs Actual oil yield**.**

3.5 Physico-chemical Properties of Expressed Watermelon oil

Physico-chemical properties of expressed watermelon oil such as specific gravity, density, pH, kinematic viscosity, and acid value were examined, and the results were presented in Table 5. Kinematic viscosity of extracted oil was 23.90 mm^2/s. The result obtained was close to work carried out by AL-Harbawy and AL-Mallah (2014) who obtained 24.9 mm^2/s, 24.6 mm^2/s and 23.8 mm^2/s for crude castor oil, refined (cold) and hot, respectively. The acid value of expressed watermelon oil was found to be 2.89 mgKOH/g. Similar trend was observed by Poornima *et al.* (2019) who reported 2.80 mgKOH/g for watermelon oil seeds. The pH value obtained was 6.70. The result obtained for specific gravity was 0.95 which is close to result obtained by Poornima *et al.* (2019). The density of the extracted oil was determined, the result obtained was 955 kg/m^3 whereas, the density of conventional diesel was 824 kg/m^3 and the ASTM standards for biodiesel range from 860 – 900 kg/m^3(Acharya *et al.,* 2016).

Table 5: Physico-chemical Properties of Watermelon Seed Oil.

S/N	Properties	Values
1.	Specific gravity	0.95
2.	Density at 20 °C (g/cm^3)	0.96
3.	Kinematic Viscosity, (mm^2/s)	23.90
4.	Acid value, mgKOH/g	2.89
5.	pH	6.70

4. Conclusion

The results revealed that variables considered had significant effect on the yield of watermelon oil. It has been established that:

i. Increase in applied pressure increases oil yield from the seeds. Pressures above 100 kN/mm^2 do not have significant effect on the oil yield.
ii. The maximum oil yield would be obtained at moisture content of 6.00 – 12.00 % while expression at roasting temperature of 100.00 °C. Roasting temperatures above 100°C do not increase the oil yield of watermelon seeds appreciably.
iii. Higher roasting temperature and applied pressure favour oil yield from watermelon seeds. It can be observed from the analysis of variance (ANOVA) results that all the variables under investigation had significant effects on oil yield from watermelon seeds.

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