Approach to Evaluation of Solvent Extraction of Oil from Neem Seed

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Abstract

The effect of agitation and type of impellers and contact time on the solvent extraction of oil from Neem seed was studied in this work. Food grade ethanol was employed as the extraction solvent. 2^2 factorial design optimization technique was applied using Minitab 14 software to investigate the effect of impeller speed and contact time on the percentage yield of oil in agitated solvent extraction of oil from Neem seed. 2 levels for each factor were considered for flat blade turbine impeller (A1) and rushton turbine impeller (A2) at confidence level of 95% (α = .05). The maximum percentage yield was 36.86% and was obtained when impeller type A1 was operated at 84 rpm for 40 minutes contact time at 50^oC extraction temperature and particle size of 0.425 – 0.710mm.The factorial analysis revealed that impeller speed, contact time and their interaction have significant effect on the extraction yield of oil from the Neem seed. The properties of the Neem oil extracted were found to be: specific gravity, 0.9111; pH, 6.5; refractive index, 1.4668; iodine value, 70.21g/g; acid value, 34.33mgKOH/g and Saponification value, 180.95 mgKOH/g. These values compare favourably with literature values.

Keywords : Neem oil, Extraction, Optimization and Minitab 14.

Introduction

Neem tree, which is also known as *Azadrichta indica*, is one of the best known trees in India, which is known for its medicinal properties. Extraction of oil has been of great interest worldwide and this has been as a result of the constant increase in the world population. The present global Neem oil production level cannot cater for all need of the population which includes domestics and industrial uses [1].

Neem oil extract, which is the fatty acid-extract of Neem tree seeds, is the most widely used product of the Neem tree. Neem seeds contain about 25 - 45% oil and provide the major source of Neem chemicals [2]. The average composition of Neem oil is shown in Table 1.

Table 1: Average Composition of Neem Oil

Formula	Fatty acid	Composition range
Linoleic acid	$C_{18}H_{32}O_2$	6-16%
Oleic acid	$C_{18}H_{34}O_2$	25-54%
Palmittic acid	$C_{16}H_{32}O_2$	16-33%
Stearic acid	$C_{18}H_{36}O_2$	9-24%
Linolenic	$C_{18}H_{30}O_2$	ND*
Palmitoleic acid	$C_{16}H_{30}O_2$	ND*

Source [2]

ND* = Not Determined.

Optimization is the use of specific methods to determine the most cost-effective and efficient solution to a problem or design for a process. This technique is one of the major quantitative tools in industrial decision making [3]. The need for optimum operating conditions is very vital to avoid wastages of raw materials, energy, time of extraction etc.

In this study, food grade ethanol was used for the extraction of oil from the Neem seed using agitated pilot solvent extraction plant. The effect of turbine impeller speed (mixing intensity) and contact time on percentage yield of oil from the Neem seed was investigated for 2 different impeller types. Minitab 14 software was used to get the design of experiment (DOE), the results obtained were analyzed and the optimal operating parameters were determined.

The standard properties of Neem oil are shown in Table 2.

Table 2: Standard Properties of Neem Oil

Property	Literature Value	Unit
Odour	Garlic	-
Specific gravity at 30^{o}C	0.908-0.934	-
Refractive index at 30°C	1.4615-1.4705	-
Ph	5.7 – 6.5	-
Iodine value	65 – 80	g/g
Acid Value	40	mg KOH/g
Saponification value	175-205	mg KOH/g

Source: [1, 4 and 5]

A wide variety of problems in the design, construction, operation, and analysis of chemical plants (as well as many other industrial processes) can be resolved by optimization.

Agitation refers to the induced motion of a material in a specified way, usually in a circulatory pattern inside some sort of container. Mixing is the random distribution, into and through one another, of two or more initially separated phases. Mixing is applied to processes to reduce the degree of non-uniformity, or gradient of a property in a system such as concentration, viscosity, temperature and so on. Mixing is achieved by moving material from one region to another to enhance mass and heat transfers [6].

When there are several factors in an experiment, a factorial design should be used. By factorial experiment we mean that in each complete trial or run, all possible combinations of the levels of the factors are investigated. When the objective is factor screening, it is usually best to keep the number of factor level low; most often two (2) levels are used. These levels are '+' and '-' called 'high' and 'low' respectively. The effect of a factor is defined as the change in response produced by a change in the level of the factor, and is the difference between the average response at the high level and the average response at the low level. If the calculated effect is five (5), it means that changing from high level to low level caused an average response increase of 5 units. Consider the two factors in this work namely: impeller speed and contact time denoted as A and B respectively, with 'a' levels of factor A and 'b' levels of factor B. If the experiment is replicated n times, the observation from a two-factor factorial experiment may be described by the model:

$$Y_{ijf} = \gamma + \beta_i X_1 + \beta_j X_2 + \beta_{ij} (X_1 X_2) + \epsilon_{ijf} \quad \ldots\ldots (1)$$

$i = 1,2 \ldots\ldots, a$

$j = 1,2 \ldots\ldots, b$

$f = 1,2 \ldots\ldots, n$

Where Y_{ijf} = response; that is percentage Yield of oil from the Neem seed,

γ = overall mean effect that is the average effect of all the two factors: Impeller speed and Contact time on the yield,

β_i = effect of the ith level of factor A, that is the effect of Impeller speed on the yield,
β_j = effect of the jth level of factor B, that is the effect of Contact time on the yield,
β_{ij} = effect of the interaction between Impeller speed (A) and Contact time (B) on the yield,
and ϵ_{ijf} = error component, that is generated due to effects of A and B [7].
X_1 = variable representing factor A (impeller speed)
X_2= variable representing factor B (contact time)
$X_1 X_2$ = variable representing the interaction between factors A and B.

Materials and Methods

Design of Experiment (DOE)

A 2^2 factorial design was adopted with two-variable two-level DOE using Minitab 14 computer soft ware. The run-by-run experimental design was shown in Tables 3 and 4 for impellers A1 and A2 respectively. The runs were replicated twice giving a total of 8 runs (4 x 2) to minimize error for each impeller type. The two factors and their levels considered are:

(a) Turbine impeller speed: 37 and 84 rpm
(b) Contact time: 20 and 40 minutes.

Table 3: DOE for the Extraction of Oil from Neem Seed Kernel for Impeller A1(Flat Blade Turbine Impeller) .

Run Order	Impeller Speed (rpm)	Contact Time (min)
1	84	20
2	37	20
3	37	20
4	37	40
5	37	40
6	84	40
7	84	40
8	84	20

Table 4: DOE for the Extraction of Oil from Neem Seed Kernel for Impeller A2 (Rushton Turbine Impeller) .

Run Order	Impeller Speed (rpm)	Contact Time (min)
1	84	20
2	84	40
3	37	40
4	84	20
5	37	20
6	37	40
7	37	20
8	84	40

Solvent Extraction

The extraction of oil was done using food grade ethanol as solvent in a pilot solvent extraction plant. The pilot plant is mainly made up of extractor, evaporator and condensate receiver.
Impeller was used for agitation in the extractor.

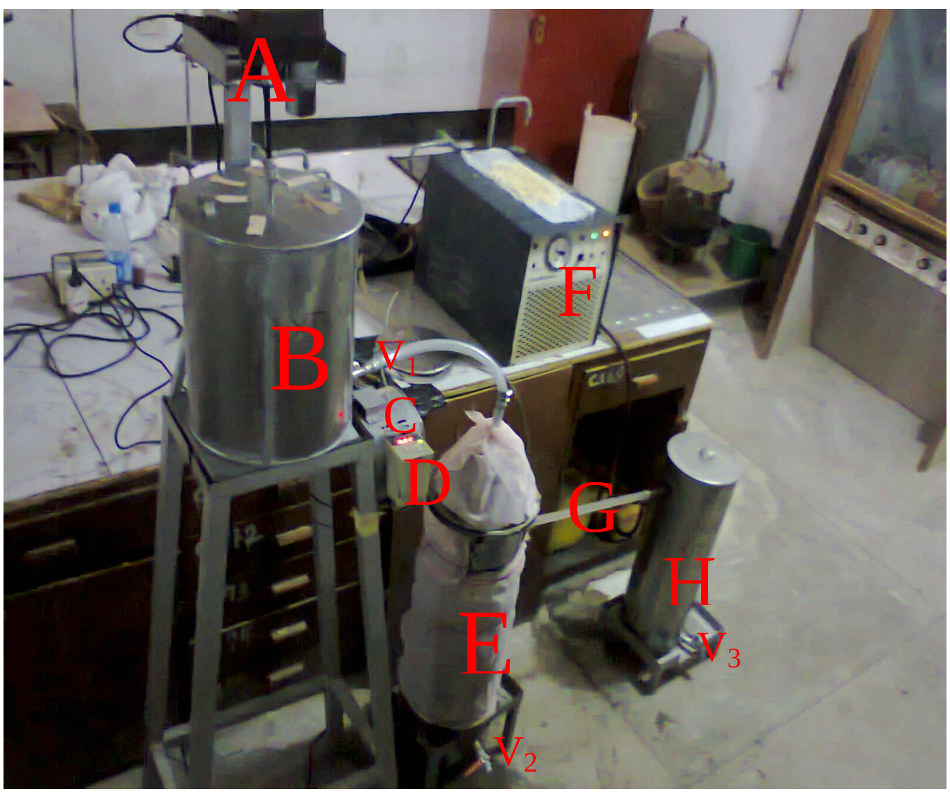
The pilot plant was adequately checked and appropriate valves; V_1, V2 and V_3 were closed. The electrical fittings were equally checked and ascertained to be in good conditions. The chiller was switched on and set to 0^oC and allow to work for 30 minutes to attain stability and cool the condenser; this was done to aid easy condensation of the food grade ethanol vapour to liquid. 21.23 litres of food grade ethanol and 0.3348kg (334.8g) of ground Neem seed kernel of particle sized 0.425 – 0.71mm were charged into the extractor.

The main switch and 50^oC switch were put on. The electric heater for the extractor was switched-on and the XMTD electronic temperature controller manufactured by XY Instrument Ltd, China was set to 50^oC for a period of time to stabilize the system at 50^oC. The stability was noticed by the aid of a temperature sensor placed in the extractor and a click short sharp sound that was heard and the temperature controller light changed from green to red which indicates that the system is stabilized at 50^oC.

Once the stability was attained, the electric motor manufactured by Brook Crompton Doncaster, England was switched-on and regulated at 84 rpm with the aid of a speed control unit using flat blade turbine impeller (A1) which was already mounted on the shaft; mixing and agitation commenced immediately for a period of 20 minutes. The above procedure was repeated based on the guide obtained from Minitab 14 computer software design of experiment (DOE). The DOE are shown in Tables 3 and 4 for impellers A1 and A2 respectively, while impellers A1 and A2 and the pilot solvent extraction plant are shown in Plates 1-3 respectively.

Plate 1: Impeller A1

Plate 2: Impeller A2



KEY

A = ELECTRIC MOTOR

B = EXTRACTOR

C = SPEED CONTROLLER

D = TEMPERATURE CONTROLLER

E = EVAPORATOR

F = CHILLER

G = CONDENSER

H = CONDENSATE RECEIVER

V_1 = CONTROL VALVE FOR MIXTURE

V_2 = CONTROL VALVE FOR COLLECTING NEEM OIL

V_3 = CONTROL VALVE FOR COLLECTING CONDENSED ETHANOL

Plate 3: Pilot Solvent Extraction Plant for Extracting Neem Oil from Neem Seed

After extraction, the electric heater and electric motor were switched-off and the control valve, V_1 was fully opened. The mixture flow through the reinforce rubber tube and through the inverted funnel for filtration to take place with the aid of a stainless steel filter mesh of size 0.00001m (0.01mm) attached to the cake receiver. The impeller shaft was disconnected from the electric motor and top of the extractor was opened and 0.424 litre of ethanol was introduced for washing to take place through percolation. After washing, the cake receiver was collected via the cake discharge outlet and placed in an oven. The weight of the cake was taken after every one hour until constant weight is achieved.

The control valves V_1, V_2 and V_3 were shut and the temperature sensor was transferred to the evaporator. The 78^oC switch was switched-on and the temperature controller set to 78^oC. The heating was maintained at 78^oC so that evaporation of the food grade ethanol can take place. The vapour ethanol passed through the already cooled condenser and was collected in the ethanol condensate receiver as liquid ethanol. After 4hr 25mins of evaporation, a sample of oil was collected via V_2 and analyzed. The collected Neem oil was dried in an oven for 10 minutes to dry-off any residual food grade ethanol .The main switch was switched-off and V_3 opened to collect the recovered solvent for recycling.

Result and Discussion

The optimum percentage yield of oil from the Neem seed was 36.86% obtained when operating impeller A1 (Flat Blade Turbine Impeller) at 84 rpm for 40 minutes contact time; while for impeller A2 (Rushton Turbine Impeller) under similar operating conditions have the best percentage yield of 31.25%. The difference in percentage yield can be associated with the presence of a disc on Rushton turbine impeller which hindered the upward flow of the mixture there by reducing the rate of leaching of the oil from the Neem seed around that region. The results show that increase in mixing intensity and contact time increases the yield for individual type of the impellers. This is because the higher the agitation of the medium, the faster the rate of oil transfer from the Neem seed to the solvent medium and the longer the contact time, the higher the quantity of oil extracted.

The results obtained from the experiment were shown in Table 5 and 6.

Table 5: Percentage Yield of Oil from Mixer - Extractor for Impeller Types A1 using Food Grade Ethanol as Solvent.3

Run order	Impeller speed (rpm)	Contact time (min)	Cake weight (g)	YIELD (%)
1	84	20	236.64	29.32
2	37	20	253.51	24.28
3	37	20	249.04	25.62
4	37	40	242.70	27.51
5	37	40	238.98	28.62
6	84	40	211.40	36.86
7	84	40	215.68	35.58
8	84	20	234.66	29.91

Table 6: Percentage Yield of Oil from Mixer - Extractor for Impeller Types A2 using Food Grade Ethanol as Solvent.

Run order	Impeller speed (min)	Contact time (min)	Cake weight (g)	YIELD (%)
1	84	20	255.59	23.66
2	84	40	232.60	30.53
3	37	40	254.62	23.95
4	84	20	251.07	25.00
5	37	20	266.68	20.35
6	37	40	256.68	23.33
7	37	20	264.65	20.95
8	84	40	230.19	31.25

Minitab 14 software was used to analyze the results. The analysis was done using confidence level of 95% (i.e α = .05) to determine the effects, coefficients, F and *P* values of the main and interactive factors. If the value in the F column from the estimated effect and coefficient table is greater than the F value obtain from the statistical table, such factor is significant. Using α = .05, if the value in the *P* column of the estimated effects and coefficients table is less than .05, such factor is significant. The estimation of the effect, coefficients and ANOVA were done and the results shown in Tables 7 – 10 for impellers A1 and A2.

Table 7 shows the individual effects, coefficients and *P* values of the main and interactive factor. The impeller speed have the highest individual effect of 6.4100, that is changing from high level
to low level caused an average response increase of 6.4100 units. Impeller speed, contact time and the interaction between the impeller speed and contact time have a P values of .000, .001 and .036 respectively; these factors were all significant because the *P* values were less than .05. Similarly, from Table 8, the impeller speed, contact time and the interaction factor were all significant.

Table 7: Estimated Effects and Coefficients for Yield (coded units) for Impeller A1

Term	Effect	Coef	SE Coef	P
Constant		29.7125	0.2799	.000
Impeller Speed (rpm)	6.4100	3.2050	0.2799	.000
Contact Time (min)	4.8600	2.4300	0.2799	.001
Impeller Speed (rpm)* Contact Time (min)	1.7450	0.8725	0.2799	.036

Table 8: Estimated Effects and Coefficients for Yield (coded units) for Impeller A2

Term	Effect	Coef	SE Coef	P
Constant		24.8775	0.2186	.000
Impeller Speed (rpm)	5.4650	2.7325	0.2186	.000
Contact Time (min)	4.7750	2.3875	0.2186	.000
Impeller Speed (rpm)* Contact Time (min)	1.7850	0.8925	0.2186	.015

Tables 9 and 10 show the ANOVA tables for testing the significance of factors based on the F and *P* values. The main factors have an F value of $F_{2,4}$ = 103.24 and $F_{2,4}$ = 6.94 from the statistical table. Since 103.24 > 6.94, the main factors are significant. For the 2-way interaction factor with F values of 9.72, it is significant because its F values is greater than $F_{1,4}$ = 7.71 from the statistical table. The significance of the main factors and interaction factor were further confirmed by the *P* value of .000 and .036 respectively, which are less than .05. Similarly, from Table 10, main factors have a significant effect ($F_{2,4}$ = 137.76 > $F_{2,4}$ = 6.94), while the interactive factor have significance effect ($F_{1,4}$= 16.67 > $F_{1,4}$ = 7.71).

Table 9: Analysis of Variance for Yield (coded units) for Impeller A1

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	2	129.415	129.415	64.7077	103.24	.000
2-Way Interactions	1	6.090	6.090	6.0901	9.72	.036
Residual Error	4	2.507	2.507	0.6268		
Total	7	138.013				

Table 10: Analysis of Variance for Yield (coded units) for Impeller A2

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	2	105.334	105.334	52.6669	137.76	.000
2-Way Interactions	1	6.372	6.372	6.3725	16.67	.015
Residual Error	4	1.529	1.529	0.3823		
Total	7	113.235				

Table 11 shows the estimated coefficients of the individual main factors and the interactive factor. The coefficients were used to generate first order regression model equations for the full factorial model using impellers A1 and A2.

Table 11: Estimated Coefficients for Yield (uncoded units) for using Impellers A1 and A2

Impeller	A1	A2
Term	Coefficients	Coefficients
Constant	20.9100	17.5734
Impeller Speed (rpm)	0.02500	0.00234
Contact Time (min)	0.01838	0.00898
Impeller Speed * Contact Time	0.00371	0.00380

The model equation for using impeller A1for the investigation of the effect of impeller speed and contact time is:
$Y= 20.9100 + 0.02500X_1 + 0.01838X_2 + 0.00371 X_1 X_2$ …………………...……… (2)

The model equation for using impeller A2 for the investigation of the effect of impeller speed and contact time is:
$Y = 17.5734 + 0.00234X_1 + 0.00898X_2 + 0.0038 X_1 X_2$…………………………..…… (3)

Where :Y= % yield
X_1 = variable representing factor A (impeller speed)
X_2 = variable representing factor B (contact time)
$X_1 X_2$ = variable representing the interaction between factors A and B.

Surface Plot of Yield Figures 1 and 2 are three – dimensional surface plots, showing the plane of predicted response values generated by the regression model at any point within the experimental region for impeller A1 and A2 respectively. The flat nature of the surface plots show that the regression model equations are first-order model inrespect with X1 and X2 besides the variables interaction. From the surface plots, the maximum yield can be obtained when the impeller speed and contact time are operated at their high levels.

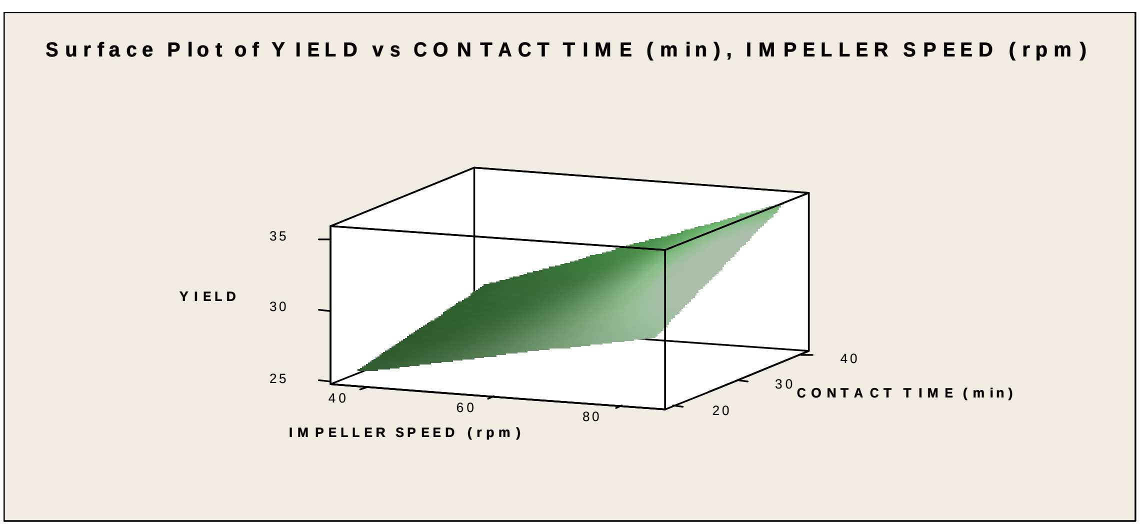


Figure 1: Surface plot of Yield for impeller A1

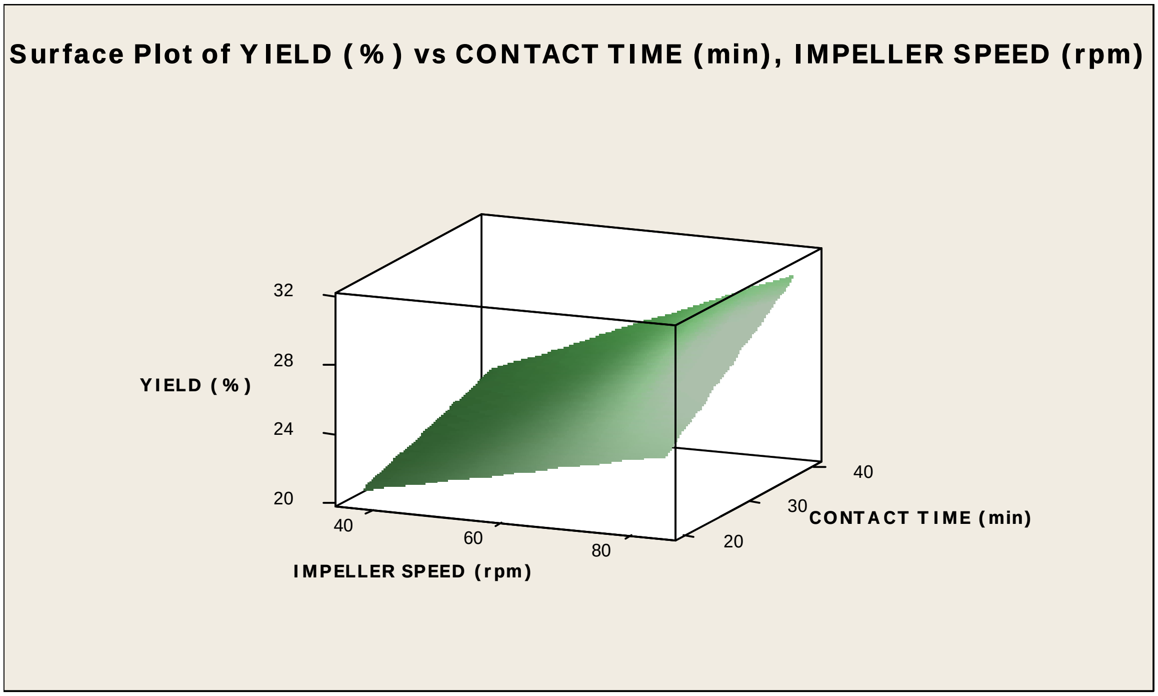


Figure 2: Surface plot of Yield for impeller A2

Diagnostic

The results obtained from the pilot solvent extraction plant was used to validate the model equations.
Tables 12 and 13 show the calculated predicted yield for validating the model equations 2 and 3 for using impellers A1 and A2 respectively.

Table 12: Values for Validation of Model for using impeller A1

Run Order	Impeller Speed (rpm)	Contact Time (min)	Experimental Yield (%)	Predicted Yield (%)
1	84	20	29.32	29.61
2	37	20	24.28	24.95
3	37	20	25.62	24.95
4	37	40	27.51	28.07
5	37	40	28.62	28.07
6	84	40	36.86	36.22
7	84	40	35.58	36.22
8	84	20	29.91	29.61

Table 13: Values for Validation of Model for using impeller A2

Run Order	Impeller Speed (rpm)	Contact Time (min)	Experimental Yield (%)	Predicted Yield (%)
1	84	20	23.66	24.33
2	84	40	30.53	30.89
3	37	40	23.95	23.64
4	84	20	25.00	24.33
5	37	20	20.35	20.65
6	37	40	23.33	23.64
7	37	20	20.95	20.65
8	84	40	31.25	30.89

The linear relationships between the predicted and experimental responses were shown in Figure 3 and 4 when the predicted response was plotted against experimental response. The least square fit line passing through the origin suggests the adequacy of the models.

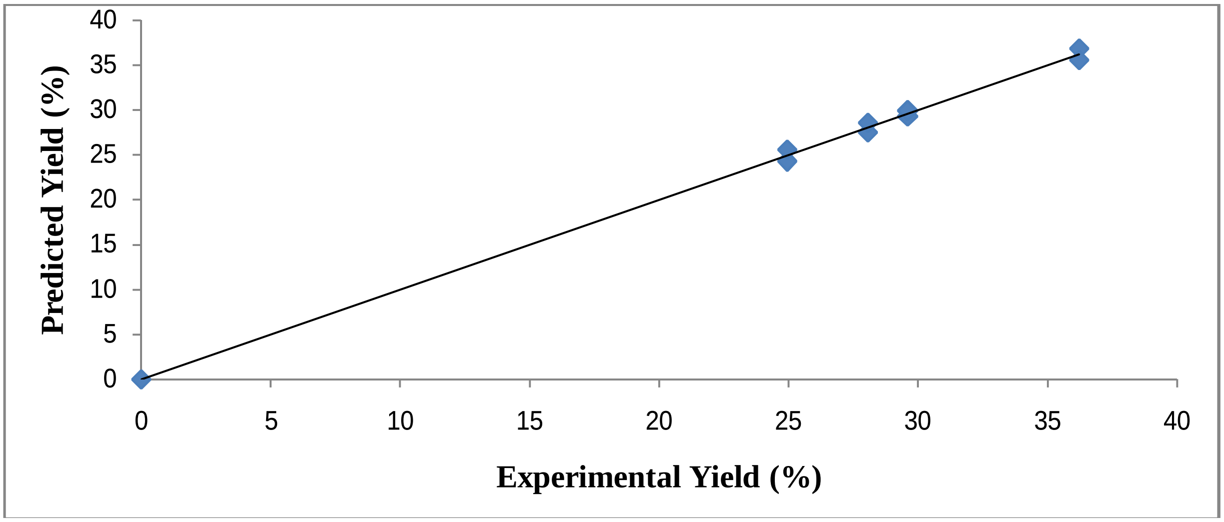


Figure 3 : Predicted Yield Vs Experimental Yield for impeller A1

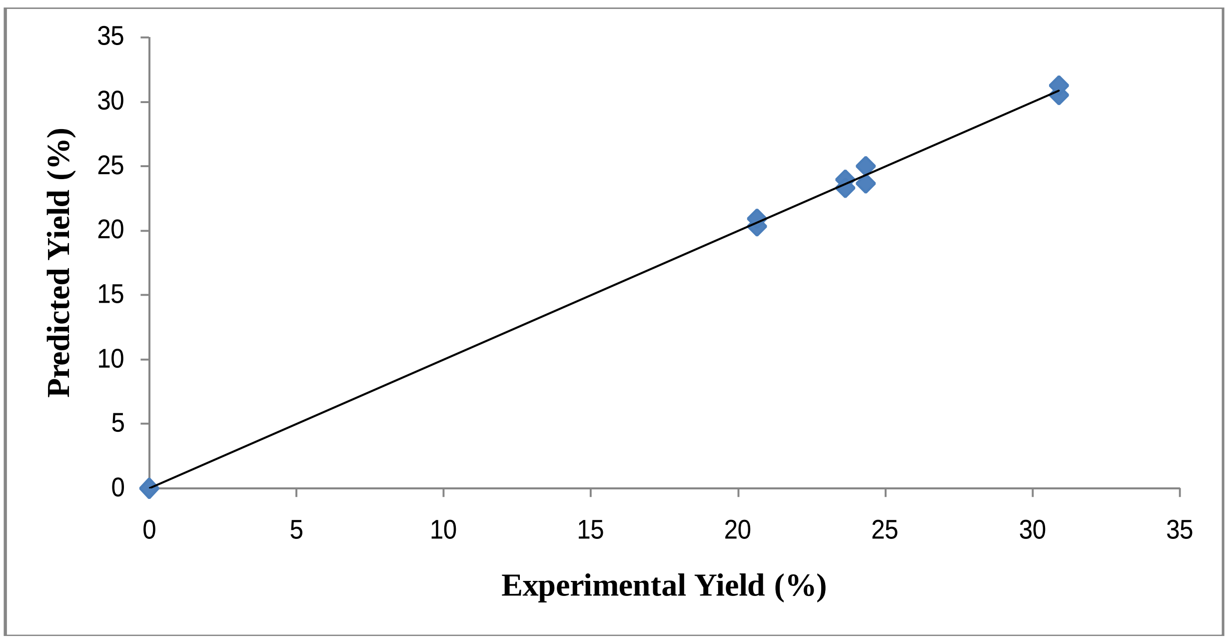


Figure 4: Predicted Yield Vs Experimental Yield for impeller A2

Conclusion

Neem oil was extracted using food grade ethanol as solvent in a pilot agitated solvent extraction plant using the DOE as guide. The effects of agitation, contact time and type of impellers of the agitator that affect the agitated solvent extraction process were evaluated. Using $\alpha = .05$, the main factors : impeller speed (A) and contact time (B) and the impeller speed – contact time interaction (AB) have significant effect on the percentage yield of oil for both impellers A1 and A2. The highest percentage yield was 36.89% within the experimental limit. The results suggest that impeller type A1 should be operated at 84 rpm for a contact time of 40 minutes for optimum yield as seen from the experimental and predicted values. This work provides a basis for selection of extraction equipment and design of agitated solvent extraction of oil from neem seed. The work also provides a window of opportunity for investment in small and medium scale processing of locally available raw materials such as neem seed especially in developing countries to meet the demand of household consumer goods.

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