Modeling of the Extraction of Oil from Neem Seed using Minitab 14 Software

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Abstract
2^2 factorial design 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^{o}C 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. The model equations for using A1 and A2 are $Y= 20.9100 + 0.02500X_1 + 0.01838X_2 + 0.00371 X_1 X_2$ and $Y = 17.5734 + 0.00234X_1 + 0.00898X_2 + 0.0038 X_1 X_2$ respectively.
Keywords : Neem oil, Extraction, Modeling and Minitab 14.

1.0 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 Neem oil produced 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.

The term model, as used in this paper, is referred to the ensemble of equations which describe and interrelate the variables or parameters of the extraction process using a designed and constructed agitated pilot solvent extraction plant.

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), analyzed the result and obtained the model equations.

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°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]

Two general categories of models exist:

(i) Those based on physical theory. Mathematical models based on physical and chemical laws (e.g., mass and energy balances, thermodynamics, chemical reaction kinetics) are frequently employed in optimization applications. These models are conceptually attractive because a general model for any system size can be developed even before the system is constructed.

(ii) Those based on strictly empirical descriptions. Empirical models are usually only relevant for restricted ranges of operation and scale-up.

Typical relations for empirical models might be

$$Y = a_0 + a_1 X_1 + a_2 X_2 + \ldots \quad (1)$$

Linear in the variables and coefficients

$$Y = a_0 + a_{11} X_1^2 + a_{12} X_1 X_2 + \ldots \quad (2)$$

Linear in the coefficients, nonlinear in the variables (X_1, X_2).

Where y = response variable, a = coefficient constant, X = operating variable.

When the model is linear in the coefficients, they can be estimated by a procedure called linear regression. If the model is nonlinear in the coefficients, estimating them is referred to as nonlinear regression. In either case, the simplest adequate model (with the fewest number of coefficients) should be used [3].

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 used 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:

$$Y_{ijf} = \gamma + \beta_i X_1 + \beta_j X_2 + \beta_{ij} (X_1 X_2) + \epsilon_{ijf} \quad (3)$$

i = 1,2 …….., a

j = 1,2 …….., b

f = 1,2 ……., 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.

METHODOLOGY

Design of Experiment (DOE)

A 2^2 factorial design was adopted with two-variables two-level DOE using Minitab 14 computer soft ware. The run-by-run experimental design were 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:

(b) Turbine impeller speed : 37 and 84 rpm

(c) 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,V_2 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 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 dried-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 Tables 5 and 6.

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

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 $\alpha = .05$) 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 $\alpha = .05$, 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 A1 investigating the effect of impeller speed and contact time is: $Y= 20.9100 + 0.02500X_1 + 0.01838X_2 + 0.00371 X_1 X_2$ ……………………..……..…(4)
The model equation for using impeller A2 investigating the effect of impeller speed and contact time is: $Y = 17.5734 + 0.00234X_1 + 0.00898X_2 + 0.0038 X_1 X_2$………………………..…..…(5)
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. From the surface plots, the maximum yield can be obtained when the impeller speed and contact time are operated at their high levels.

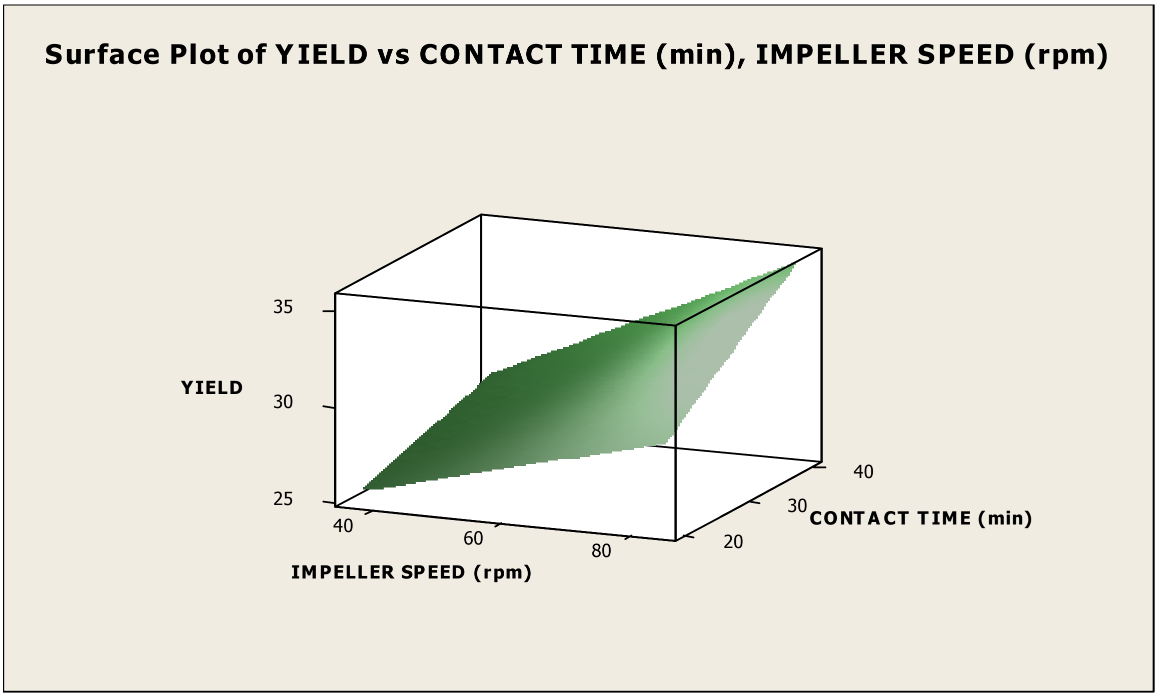

Figure 1: Surface plot of Yield for impeller A1

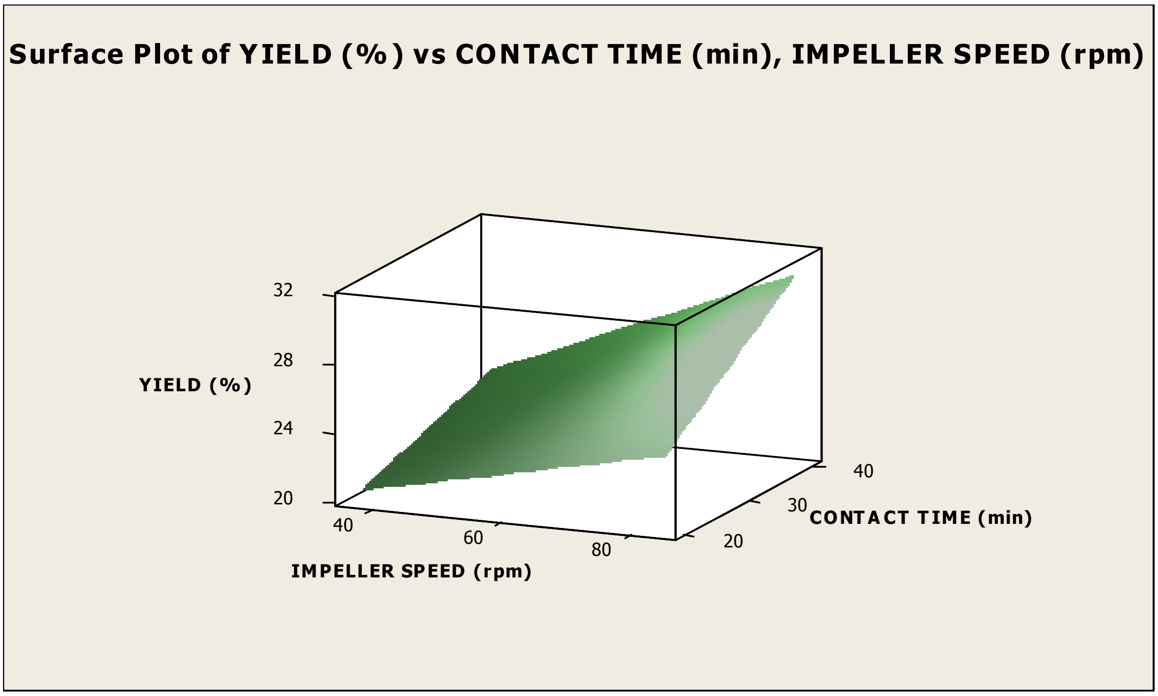

Figure 2: Surface plot of Yield for impeller A2

Model Validation

The experimental and predicted yield values are shown in Tables 12 and 13 for using impellers A1 and A2 respectively.

Table 12: Values for Experimental and Predicted Yields for using impeller A1

Run Order	Impeller Speed (rpm)	Contact Time (min)	Experimental Yield (%)	Predicted Yield (%)
1	37	20	24.28	24.95
2	37	20	25.62	24.95
3	37	40	27.51	28.07
4	37	40	28.62	28.07
5	84	20	29.32	29.61
6	84	20	29.91	29.61
7	84	40	35.58	36.22
8	84	40	36.89	36.22

Table 13: Values for Experimental and Predicted Yields for using impeller A2

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

The model equations 4 and 5 were validated using values within the experimental limits. 45 and 56 rpm were considered for impeller speed, while 25 and 30 minutes were considered for contact time. The validation values are shown in Tables 14 and 15 for using impellers A1 and A2 respectively.

Table 14: Values for Validation of Model using Impeller A1

S/NO	Impeller Speed (rpm)	Contact Time (min)	Yield (%)
1	56	25	27.96
2	45	30	27.59

Table 15: Values for Validation of Model using Impeller A2

S/NO	Impeller Speed (rpm)	Contact Time (min)	Yield (%)
1	56	25	23.25
2	45	30	23.08

The percentage yield of neem oil obtained for validating the model equation 4 in Table 14 fall within the percentage range obtained for predicted yield of 36.22% (84 rpm, 40 mins) and 24.95% (37 rpm, 20 mins) as shown in Table 14. Therefore, the predicted model equation adequately fits the experimental values. From Table 15, the percentage yields obtained for validating the model equation 5 falls within the percentage range obtained for predicted yield of 30.89% (84 rpm, 40 mins) and 20.65% (37 rpm, 20 mins) as shown in Table 15. Therefore, the predicted model equation values adequately fit the experimental values.
The linear relationship 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.

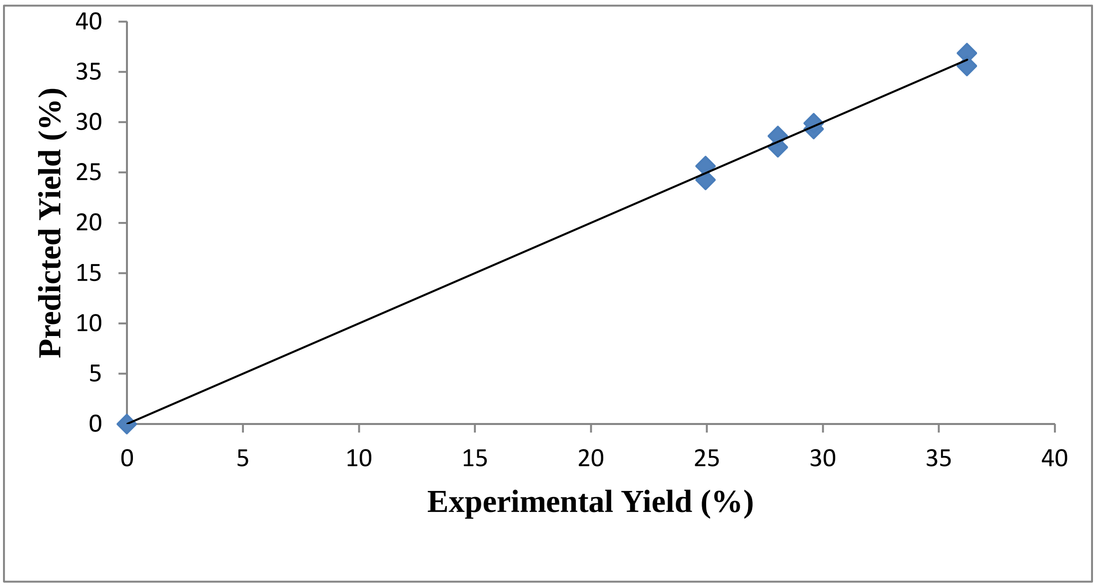

Figure 3 : Predicted Yield Vs Experimental Yield for impeller A1

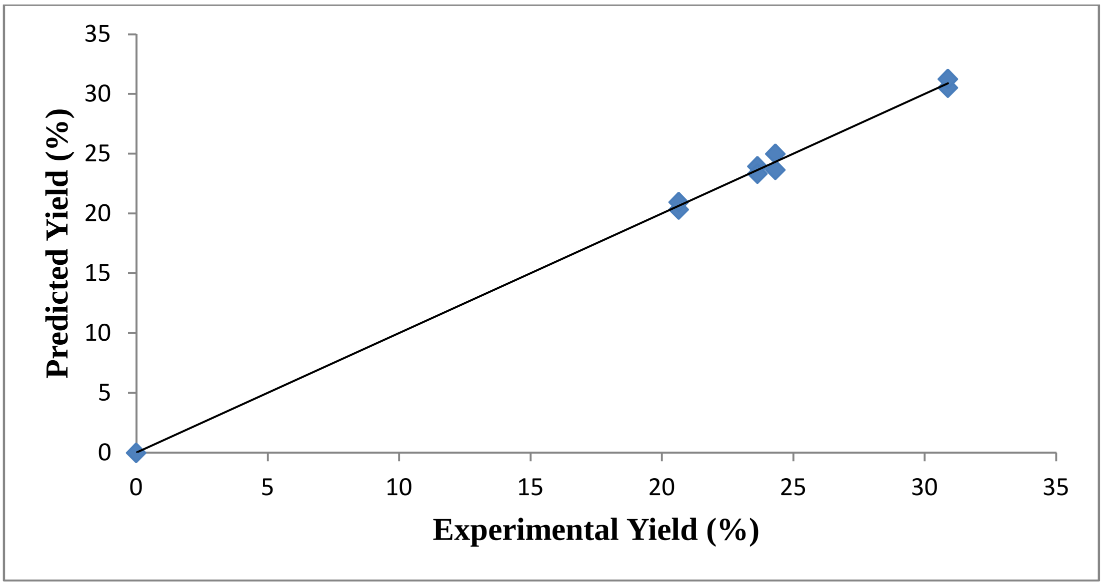

Figure 4: Predicted Yield Vs Experimental Yield for impeller A2

CONCLUSION

Neem oil was extracted using food grade ethanol as solvent in a pilot solvent extraction plant using the DOE as guide. Using alpha (α) = .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 model equations for using A1 and A2 are: $Y= 20.9100 + 0.02500X_1 + 0.01838X_2 + 0.00371 X_1 X_2$ and $Y = 17.5734 + 0.00234X_1 + 0.00898X_2 + 0.0038 X_1 X_2$ respectively.

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