Effects of Process Parameters on the Yield of Alkali from Oil Palm Male Inflorescence (Ash).

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ABSTRACT

This study investigated the effects of process parameters on the yield of alkali from oil palm male inflorescence ash. The dried stalk of the oil palm inflorescence bunches were size reduced and burned in an incinerator in the presence of air. A measured quantity of the ash was dissolved in known quantity of distilled water in a 500ml glass reactor to obtain the alkali. Different stirring speeds, temperature, concentration and time were considered during the experiment for the production of the alkaline solution. Finally the concentrations of the different alkaline samples were determined from the experimental runs. The optimum parameter values were found to be a time of 50mins, a stirring speed of 500rpm, a temperature of 50^0C, and a concentration of 60g/l of distilled water. An optimum yield of 11.74% alkalinity was recorded. Standard statistical software design expert (8.0.2) was applied in the plotting of various graphs and generation of the ANOVA table from which a linear model was developed. From the model equation it was observed that parameter B which is the stirring speed gave the highest effect and also of highest significance. These facts were subsequently validated.

(Keywords: ANOVA, ash, factorial design, male inflorescences, leaching)

INTRODUCTION

Oil palm (*E. guineesis*), a native of forested portions of Western and Central Africa, particularly along rivers, has been spread across Africa by human migrations or intergroup exchange (Anon, 1986). It is a single-stemmed palm, which bears, like the majority of palm species, a single vegetative shoot apical meristem maintained throughout the lifetime of the plant and localized at the center of the leaf crown (Adam et al, 2005). Africans probably took palm oil to Madagascar in the 10^{th} century, the same time it is thought to have been established on the southern coast of Kenya-Pemba and Zanzibar (Harley, 1988).

It's difficult to determine the natural habitat of the oil palm because, it does not grow in primeval forest, it flourishes in habitats where forests have been cleared. Normally, palm oil tree occurs in forests and along rivers and streams, both in the native range in West African and in some introduced areas. It is a succession favored by slash and burn, and its gene pool has expanded as farmers clear land and create more open habitat for the germination of its seeds (Anon, 1986).

Male and female flowers are borne on the same plant but open at different times, so that cross-pollination is necessary. A male inflorescence contains 700 to 1200 flowers and may yield 80g of pollen over a 5-day period. The major local raw material for alkali production is the palm inflorescence which is of two types, the male and female inflorescence.

Alkali has been of prime importance as raw material both for industrial, laboratory and domestic purposes. Ash from male and female inflorescence of palm oil tree is a renewable raw material that is readily available and cheap. Locally, the ash is often referred as "potash" because people believed that it has high potassium content, European commission (ETC/SCP report, 2010). Local people use it in the production of soap and also in cooking. The alkali reacts with oil to produce very soft soap – the native soap.

For this research the production of alkali from local material, the male palm inflorescence is investigated. The production process involves some basic unit operation processes such as size reduction, drying, incineration, leaching and filtration. The key process that produces the alkali is leaching where the K^+ contain in the ash is converted to the alkali (KOH). This leaching process is affected by a number of factors which include time, temperature, concentration (ash-to-water ratio) and level of agitation (stirring) (Mba, 2005). The individual and combined interactive effects of these factors on the yield of alkali were considered in this work.

In the study of the interactive effects of the factors, a two level-four factor factorial design was employed and the results were optimized. The study requires 16 experiments with four center points. Time, stirring speed, temperature and concentration are the independent variables selected to optimize the conditions for the yield of alkali (Montgomery, 1978).

ANOVA (Analysis of variance) is used in the analysis of experimental data. Half normal plot and Pareto chart were also used to select the statistical significant effects in the model. A Pareto chart is of use in quality control. It is a bar chart that lists the defects/derivations that are observed in descending order. By the use of such charts, areas of concern can be identified and efforts made to correct the defects that account for the largest percentage defects or derivations (Murray and Larry, 2008). The 16 experiments were carried out and the data obtained were statistically analyzed using design Expert (8.0.2) version to find a suitable model for the highest alkalinity yield from the four independent variables.

MATERIALS AND METHODS

The mature male palm inflorescence of oil palm tree was obtained after removing the fruits from the bunch. It was obtained from Abor village in Udi Local Government Area of Enugu State of Nigeria. The ash was obtained after burning the empty bunch in open air. Distilled water was used for the leaching of alkali (Echegi et al., 2012).

Leaching of Alkali

A known quantity of the ash was dissolved in a known quantity of distilled water. The reactor was placed in a thermostat water bath at a known temperature and stirred with a motorized stirrer at a known speed for a predetermined time interval. At the end of the experiment, the solution was filtered using what man filter paper to separate the residue from the filtrate. The level of alkalinity of the filtrate was determined using pH meter model 3510 Jenway England. The factors and levels used for the full factorial design were shown in Table 1.

Table 1: Factors and Levels for Two-Level Full Factorial Design.

S/N	Factors	Low levels (-)	High levels (+)
1	Temperature (^{0}C)	20	50
2	Time (Min)	40	50
3	Stirring speed (rpm)	500	750
4	Concentration (g/l)	20	60

In this work, two level four factor factorial design was employed in the optimization study, requiring 16 experiments with four center point. The time, temperature, stirring speed and concentration were the independent variables selected to optimize the conditions for the yield of alkali. The results of the independent investigations with the four factors and at two levels (low and high) were subjected to factorial design of the experiment with a view to exposing the individual and interactive effects on the yield of alkali (Montgomery, 1978). The 16 experiments were carried out and data were statistically analyzed by design-Expert (8.0.2) version to find a suitable model for the highest alkalinity yield from four variables.

RESULTS AND DISCUSSION

The variables considered in this experiment were time (A), stirring speed (B), Temperature (C), and concentration. (D). The experimental runs were randomized to satisfy the statistical requirement of independence of observations. Randomization acts as insurance against the effects of lurking time-related variables (Yosra and Nawaz, 2001). In full factorial experiment, all main factors and their interactions are compared with one another (Mark et al., 2007). The layout for the full factorial design with response values is shown in Table 2.

Table 2: Experimental Design Matrix (Response Valves).

Std order	Run order	A Time mins	B Stirring speed (RPM)	C Temp. (^0C)	D Concen-tration (g/l)	Alkalinity
2	1	50.00	500.00	40.00	20.00	11.51
18	2	35.00	625.00	45.00	40.00	11.5
5	3	20.00	500.00	50.00	20.00	11.44
3	4	20.00	750.00	40.00	20.00	11.74
8	5	50.00	750.00	50.00	20.00	11.71
12	6	50.00	750.00	40.00	60.00	11.2
17	7	35.00	625.00	45.00	40.00	11.2
9	8	20.00	500.00	40.00	60.00	10.65
15	9	20.00	750.00	50.00	60.00	11.3
14	10	50.00	500.00	50.00	60.00	10.56
20	11	35.00	625.00	45.00	40.00	10.65
7	12	20.00	750.00	50.00	20.00	11.54
10	13	50.00	500.00	40.00	60.00	10.84
4	14	50.00	750.00	40.00	20.00	10.84
16	15	50.00	750.00	50.00	60.00	10.46
6	16	50.00	500.00	50.00	20.00	11.06
1	17	20.00	500.00	40.00	20.00	11.17
11	18	20.00	750.00	40.00	60.00	11.02
19	19	35.00	635.00	45.00	40.00	10.61
13	20	20.00	500.00	50.00	60.00	11.72

From Table 2, the response or yield that is obtained by inputting the several values of the independent variables in the Design expert software to generate the alkalinity of the various experiments.

One Factor Effect

From the statistical analysis done, it showed that all the single factor effects studied had significant effect on the alkalinity both in the positive and negative direction. Effects B and C had a positive effect as can be seen from the plot while effect D had a negative effect as can be deuced from the plot.

Figure 1A shows a positive graph, as the initial concentration is increasing the alkalinity increases.

Figure 1B shows a positive graph, as the temperature is increasing the alkalinity increases.

Figure 1C shows a negative graph, as the stirring speed is increasing the alkalinity decreases.

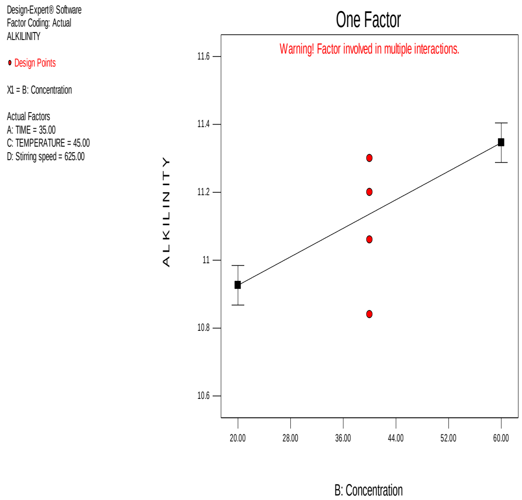

Figure 1a: One Factor (Concentration).

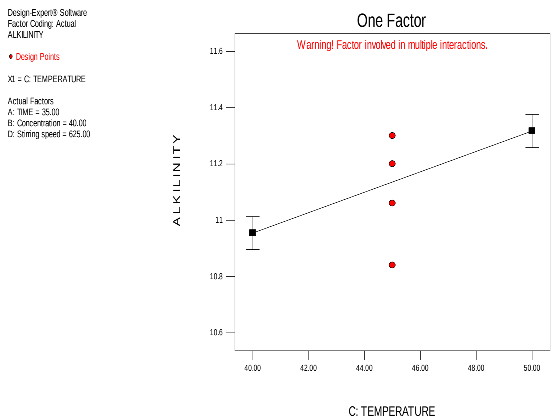

Figure 1b: One Factor (Temperature).

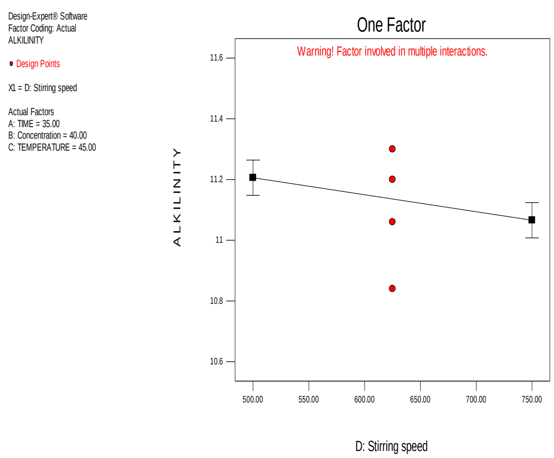

Figure 1c: One Factor (Stirring Speed).

Interaction Effects

Interactions occur when the effect of one factor depends on the level of the other. From Figure 2a below, it is cleared that when the concentration was high (red line), the line angle progresses positively in the linear direction which indicates a strong positive effect as time of leaching increases. When concentration was low (black line), the line angle steps downward, indicating a strong negative effect due to increase in leaching time. The contour plots and the 3D dimensional surface plots equally gave the same interactions as that of the interaction effects.

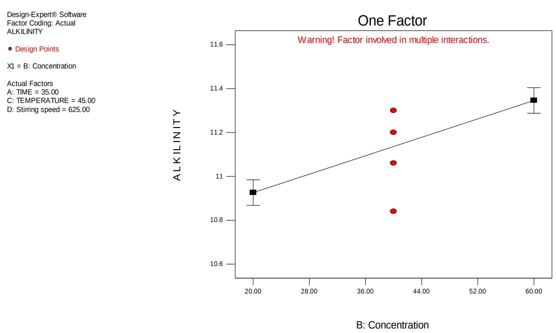

Figure 2a: (a) Interaction effect of time vs concentration

Figure 2A above shows a positive graph, as the concentration is increasing the alkalinity increases.

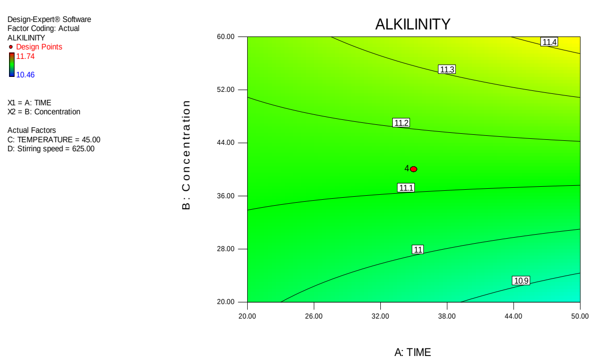

Figure 2b: (b) Contour Plot of Time vs Concentration.

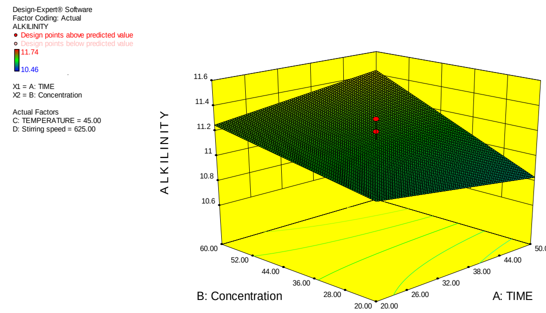

Figure 2c: (c) 3D Surface Plot of Time vs. Concentration.

Figure 3a, shows the interaction effect between time and temperature. It can be observed that when temperature was low at 40^0C (black line), the line angle steeply downwards indicating a strong negative effect as the reaction time increases. But when the temperature was high at 50^0C (red line) these was a visible positive effect as the line angle moved in a linear direction as the time of reaction increases. The contour plots and the 3D dimensional surface plots equally gave the same interactions as that of the interaction effects.

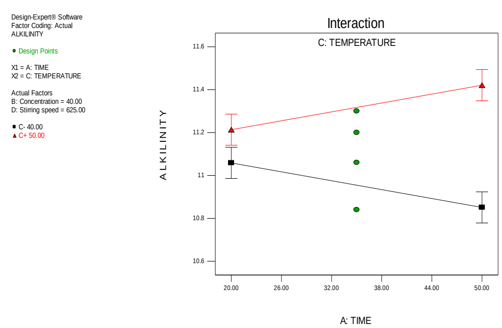

Figure 3a: (a) Interaction Effect of Time vs Temperature.

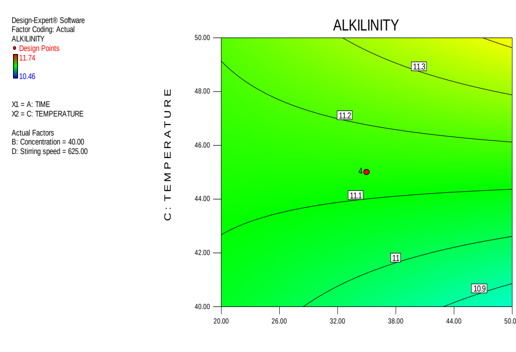

Figure 3b: (b) Contour Plot of Time vs Temperature.

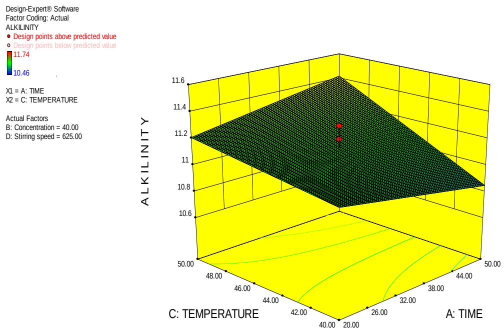

Figure 3c: (c) 3D Plots of Time vs Temperature.

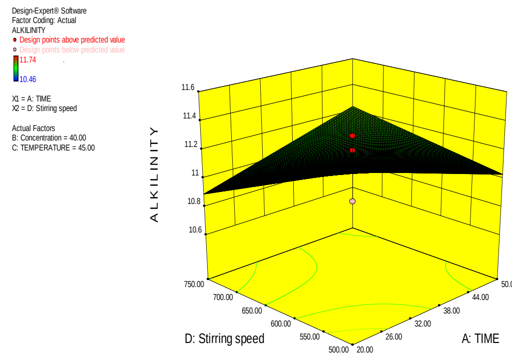

Figure 4c: (c) 3D Plots of Time vs Stirring Speed.

Figure 4a below, showed the interaction between time and stirring speed. It can be deduced from the above interaction plot that when the stirring speed was high at 750rpm (red line), there was a strong positive effect on the yield of alkali as the reaction time increases, but when the stirring speed was low at 500rpm (black line) the line angle steeply downward as can be observed as the time of reaction increased. The contour plots and the 3D dimensional surface plots equally gave the same interaction as that of the interaction effects.

Figure 5a, shows the interaction plot between concentration and stirring speed. It can be observed that when the stirring speed was low and high at rpm of (500 & 750) indicated by the black and red line, they both showed a positive linear movement. Meaning positive effect, until they both converged at a point of concentration 60g/l. the contour plots and the 3D dimensional surface plots equally gave the same interaction as that of the interaction effects.

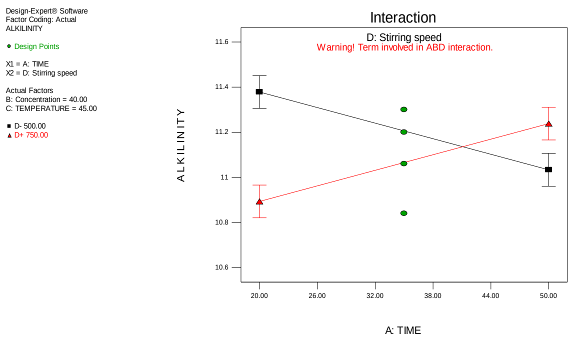

Figure 4a: (a) Interaction Effect of Time vs Stirring Speed.

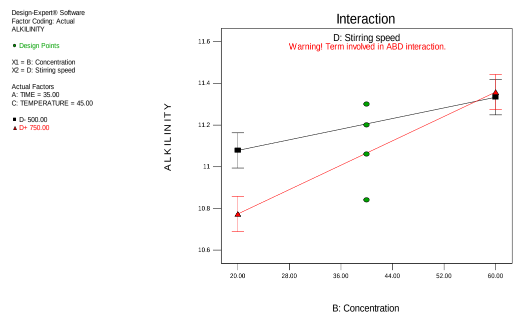

Figure 5a: (a) Interaction Effect of Concentration vs. Stirring Speed.

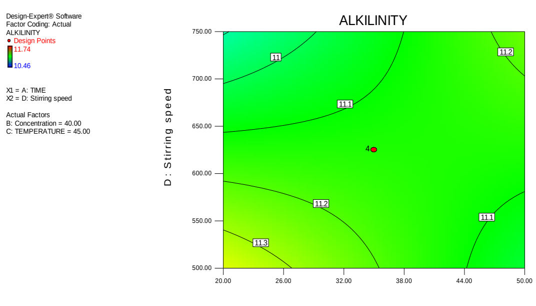

Figure 4b: (b) Contour Plot of Time vs Stirring Speed.

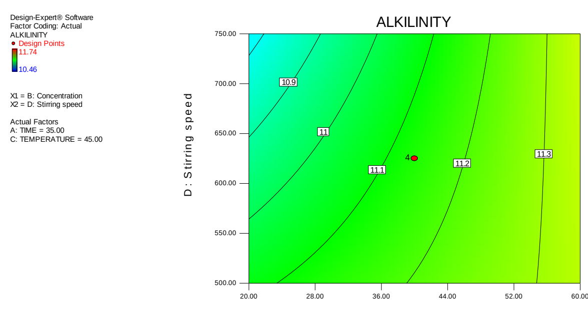

Figure 5b: (b) Contour Plot of Concentration vs Stirring Speed.

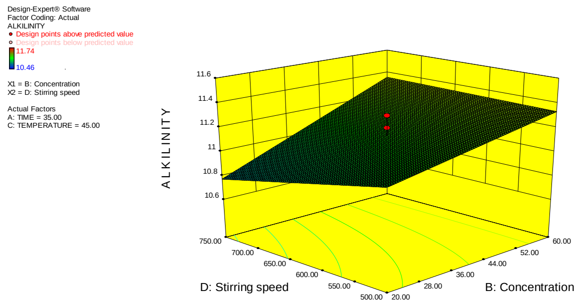

Figure 5c: (c) 3D Plots of Concentration vs Stirring Speed.

It can be observed that when the stirring speed was low and as the temperature increases, there is a slight increase in the yield of alkali as can be observed by the black line of 500rpm. But when the stirring speed was high at 750rmp denoted by the red line, there is a strong positive effect as the temperature of the leaching increases. The contour plots and the BD dimensional surface plots equally gave the same interaction as that of the interaction effects.

The half normal probability plot was used to select the statistically significant effects that were included in the model. (Mark et al, 2007). The half normal plot shown in Figure 6 shows points that deviate significantly from the normal line. On the other hand they show individual and combined factor effects deviating significantly as B,C, AD, ABD, CD, AB, BD and D.

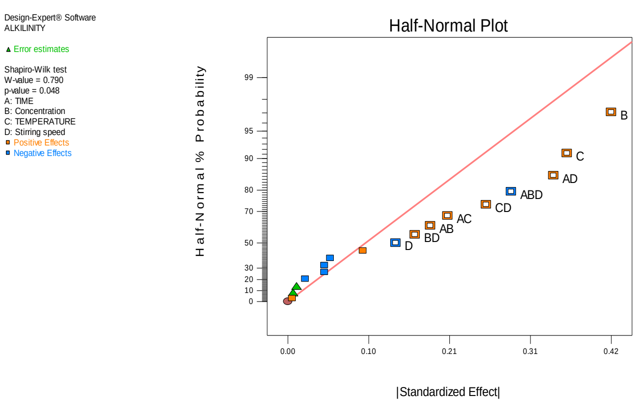

Figure 6: Half Normal Probability Plot.

The same information is given clearer in the Pareto chart of the selected effect in Figure 7. The chart was used to visualize the effects selected by the half normal probability plot. Another use of the Pareto chart is to check for "one more significant effect" that was not obvious on the half normal plot (Murray R.S and Larry J.S, 2008).

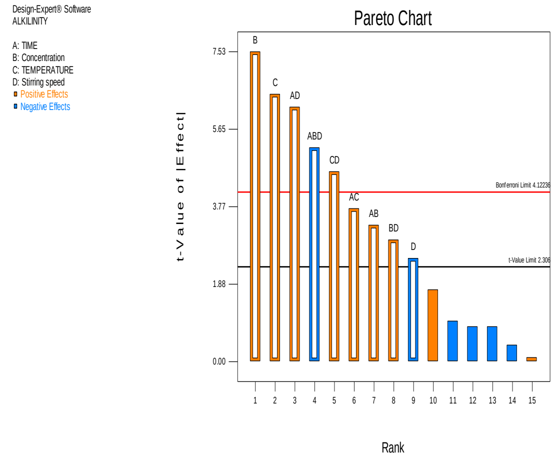

Figure 7: Pareto Chart of the Selected Effects.

ANALYSIS OF VARIANCE (ANOVA)

To protect against spurious outcomes, it is absolutely vital that one verifies the conclusions drawn from the half normal plots and Pareto chart using analysis of variance (ANOVA) and associated diagnostics of residual error (Mark et al., 2007).

The ANOVA result is shown in Table 3 below. The model f-value of 26.46 implied the model was significant. Values of "Prob>f" less than 0.0500 indicate model terms are significant (Yusra and Nawaz, 2001).

In this case B,C,D, AB, AC, AD, BD, CD and ABD are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

Table 3: ANOVA Table.

Source	Sum of squares	df	Mean	f-value	p-value >f
Model	2.81	9	0.31	26.46	< 0.0001
B	0.71	1	0.71	59.91	< 0.0001
C	0.53	1	0.53	44.63	< 0.0001
D	0.078	1	0.078	6.66	0.0297
AB	0.14	1	0.14	11.62	0.0078
AC	0.17	1	0.17	14.62	0.0041
AD	0.48	1	0.48	40.42	0.0001
BD	0.11	1	0.11	9.25	0.0140
CD	0.27	1	0.27	22.52	0.0011
ABD	0.34	1	0.34	28.56	0.0005
Residual	0.11	9	0.012		
Lack of fit	0.077	7	0.011	0.75	0.6762

Model Equation

The model is a mathematical equation used to predict a given response. The final equation in terms of coded factor gives the alkalinity. Y = +11.14 + 0.21B + 0.18C – 0.070D + 0.093AB + 0.10AC + 0.17AD + 0.083BD + 0.13CD – 0.15ABD.

It is necessary to diagnose residuals to validate statistical assumptions. For statistical purpose it is assumed that residuals are normally distributed and independent with constant variance (Mark et al., 2007). The two plots recommended for checking the statistical assumptions are: Normal plot of residuals, Residuals versus predicted level.

Normal Plot of Residuals

The normal plot of residuals is a graphical representation for determining if the data is distributed normally or not (Pokhrel et al., 2006).

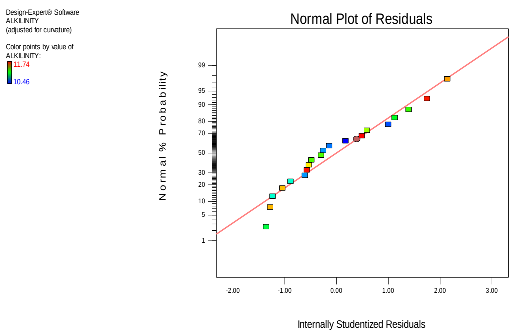

Figure 8: Normal Plot of Residuals.

From Figure 8, it can be seen that the deviations from linear is highly minimal, so it supported the assumption of normality. All the points cluster nearly / evenly above and below the normal line.

Residuals versus Predicted Level

This is a plot of the residuals versus the predicted response values. It tests the assumption of constant variance.

From the Figure 9 it is observed that there was no definite increase in residuals with predicted levels, which supported the underlying statistical assumption of constant variance.

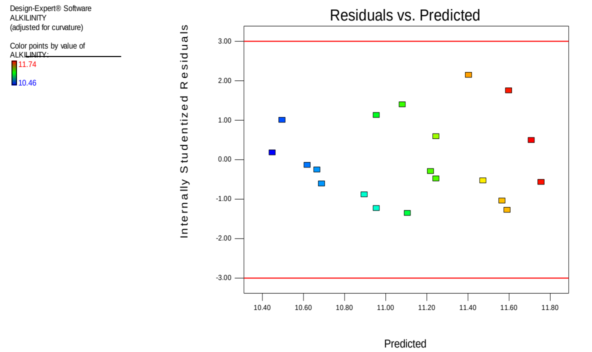

Figure 9: Residuals vs. Predicted Values.

CUBIC INTERACTION EFFECT OF THREE FACTORS

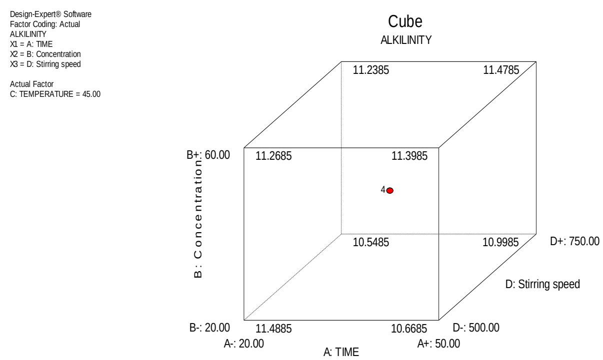

Figure 10: Cubic Interaction Effect.

Figure 10 above, shows that cubic plot of the three factors ABD and that the maximum of the plot is at points A-, B-, D- at the lower front left corner with a value of 11.4885. It is minimum at A-, B-, D+ at the lower back left corner with a value of 10.5485. Where A is time, B is stirring speed and D is concentration.

PROCESS OPTIMIZATION

Once a good model was obtained, it can be optimized. The optimum operating conditions suggested by the DOE model for the four variables namely time, stirring speed, temperature and concentration was at 50mins, 500rpm, 50^{0}C and 60g/l, respectively.

CONCLUSION

The leaching of alkali from local ash obtained from male inflorescence of palm oil tree was investigated. The effects (single and interaction) of the leaching variables were studied using two-level four factor factorial design. The statistical analysis confirmed that the leaching of alkali was enhanced by decrease in solvent volume at the constant quantity of the ash and its interaction with temperature. ANOVA confirmed that the model was significant. The diagnostic plots were used to validate the model developed. Optimizing the model, the optimal alkalinity was 11.74% at temperature of 50^0C, time at 50mins, stirring speed of 500rpm and concentration of 60g/l.

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