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ALKALI LEACHING OF OIL PALM FEMALE INFLORESCENCE ASH: APPLICATION OF FACTORIAL DESIGN

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Abstract

This research work involved the use of 2 levels full factorial design technique to investigate the alkali leaching of oil palm female inflorescence ash. A 2^4 full factorial design was used to study effect of four factors: leaching Time, Temperature, Stirring Speed and solute/solvent volume ratio at two levels each. The statistical analysis showed that only solute/solvent volume ratio and its interaction with temperature had effect on the alkalinity. A linear model was developed to predict the process and subsequently was validated with residual plots. The optimization of the model at desirability of 0.951 predicted alkalinity of 10.79.

INTRODUCTION

Oil palm (Elaeis guineensis) 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[1]. Oil palm produces separate male and female inflorescences on the same palm in an alternating cycle of variable duration depending on genetic factors, age, and particularly environmental conditions, with the production of male inflorescences generally favored by water stress[2]. Occasionally mixed sex inflorescences are produced at the transition between the male and female cycle[3]. Mature inflorescences are visible typically 32 – 36 months after seed germination[4].

The female inflorescence reaches a length of 35cm or more when fully developed, and each of its rachillae bears 5 – 30 floral triads. The triads are arranged spirally around the rachilla axis, and each is subtended by a spiny floral traid bract[5].

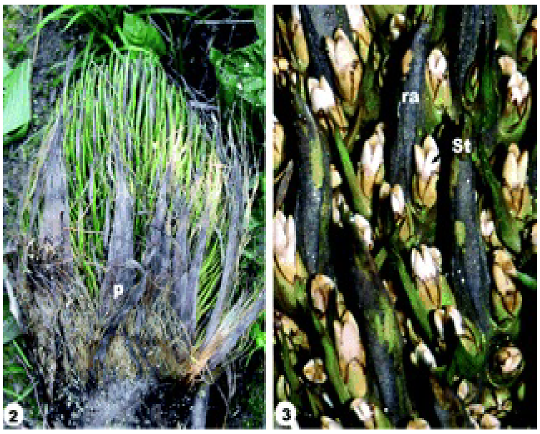


Figure 1. The macroscopic view of mature female inflorescence of oil palm (2) after opening of the peduncular bract and prophyll (3) female rachillae carrying pistillate flowers at maturity with exposed stigmas[1].

Due to increase in world population and demands for chemical products, there has been much pressure on the industry for chemical raw materials, consequent upon that, renewal raw material have to augment to meet the worldwide increasing demand. Some of the chemical raw materials are fossil based materials that are expensive to explore and impart negative effect on the environment due to greenhouse effect.

According to the Lead Market Initiative of European Commission (2007), products based on renewable raw materials are seen as one of the most promising future markets[6]. The use of renewable raw material as a feedstock for the production of materials, chemicals and other biobased products can save fossil resources and reduce negative impacts on the environment. It can also support the agricultural and forestall sector, and lead to innovations in, for example, biomaterials or biobased chemicals[6].

Ash from female inflorescence of palm oil tree is a renewable raw material that is readily available and cheap. Locally, the ash is often referred to as “potash” because people believed that it has high potassium content. Study on the distribution of absorbed nutrient among the different plant component revealed that potassium is the highest nutrient on a fresh fruit bunch with 3.71kgK per ton of bunch with 257.4kgK as 302.8kgK_2O[7]. The use of alkali from the Ash as a chemical raw material has not gained an international recognition, locally, people use it in the production of soap and also in cooking. In cooking, when added, can speed up the rate in which hard foods can soften.

The aim of this experiment was to investigate the effect of some process factors (individuals and interaction) on the leaching of alkali from the ash, to obtain a mathematical representation that can predict the leaching process and validate it. To equally optimizes the process. The process factors considered were: leaching temperature, leaching time, stirring speed and sample/solvent volume ratio.

MATERIALS AND METHODS

The mature female inflorescence of palm oil tree was obtained after removing the ripe fruits from the bunch. It was obtained from Olo, in Ezeagu LGA of Enugu State of Nigeria. The ash was obtained after burning the empty bunch without fuel.

Distilled water used for the leaching was obtained from PYMOTECH research center and laboratory Enugu, Enugu State Nigeria.

Leaching of alkali

This was carried out in a 500ml glass reactor immersed in a temperature controlling water bath, equipped with a thermostat and motorized stirrer.

A known quantity of the ash was placed in the glass reactor with some solvents; this 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 whatman fitter 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 on table 1 below. The solvent used for the leaching was distilled water.

The condition of the experiment was strictly based on the design matrix on table 2.

Experimental design

Experimental design is an excellent tool for studying the individual and interaction effects of all parameters simultaneously.

There are several important problems with the conventional approach of changing one or two variables in a run. It may take several rounds of experiments to find the optimum point. In cases where variables must be changed in large steps, the optimum may not be found at all [8]. An experiment is called factorial experiment if the treatments consist of all possible combinations of several levels of factors. It reveals the effect of interaction of process variables and improves process optimization. The design layout for the full factorial design with both actual response values and values predicted by the model is shown on table 2 below

RESULTS AND DISCUSSION

The variables considered on this experiment were Temperature (A), Time (B), Stirring Speed (C) and Sample/Solvent Volume Ratio (D). The experimental runs were randomized to satisfy the statistical requirement of independences of observations. Randomization acts as insurance against the effects of lurking time – related variables[10.]

The half normal probability plot was used to select the statistically significant effects that were included in the model.

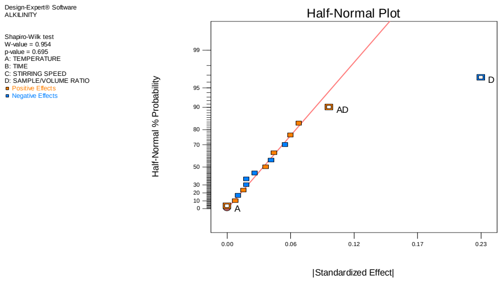


Figure 1 Half normal probability plot

From the Figure I, only the single effect of D which was the sample/solvent volume ratio and its interaction effect with A which was temperature that were significant.

Pareto chart is an additional graphic used to display the t-values of the effects. 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.

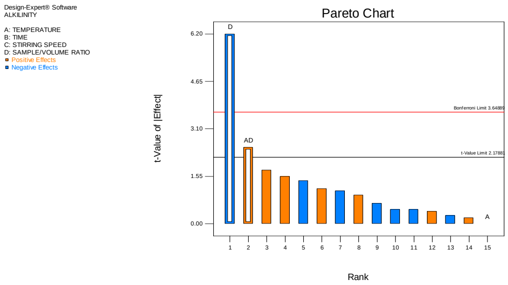


Figure 2 Pareto chart of the selected effects

From the chart, it was confirmed that only solute/solvent volume ratio and its interaction with temperature were statistically significant because they lied above t-value limit of 2.17881.

Analysis of variance (ANOVA)

To protect against spurious outcomes, it is absolutely vital that one verify the conclusions drawn from the half normal plots and Pareto chart by doing Analysis of Variance (ANOVA) and associated diagnostics of residual error[10].

ANOVA is a statistical method that partitions the total variations into its component parts each of which is associated with different source of variation[9].

The ANOVA result is shown on table 3 below. The model F – value of 14.86 implied the model was significant. There is only a 0.02% chance that a "model F – value" this large could occur due to noise.

Values of "Prob>F" less than 0.0500 indicated model terms were significant. In this case solute/solvent volume ratio (D) and its interaction with temperature (AD) were significant model terms. The single effect of temperature (A) was included to support hierarchy.

Values greater than 0.1000 indicated the model terms were not significant.

“Adeq precision” measures the signal to noise ratio. A ratio greater than 4 is desirable. A ratio of 8.687 indicated an adequate signal. The model can be used to navigate the design space.

Model equation

The model is a mathematical equation used to predict a given response. The final equation in terms of coded factor is

Alkalinity = +10.63 – 0.12D +0.046AD (1)

The final equation in terms of Actual factors is an alternative predictive model that expresses the factors in their original units of measure.

Alkalinity= +12.85813 – 1.53750* Sample/Volume ratio + 0.015417* Temperature * Sample/Volume ratio (2)

The uncoded model can be used to generate predicted values but the coefficients cannot be compared. The positive signs in front of the terms indicated synergistic effect, where as negative sign indicated antagonistic effect.

Validation of model

It is necessary to diagnose residuals to validate statistical assumptions. For statistical purposes it’s assumed that residuals are normally distributed and independent with constant variance[10]. Two plots are recommended for checking the statistical assumptions:

- 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[11].

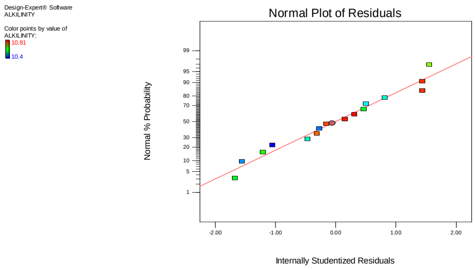


Figure 3: Normal plot of residuals.

From the Figureure above, it can be seen that the deviations from linear were very minor, so it supported the assumption of normality.

Residuals versus predicted level

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

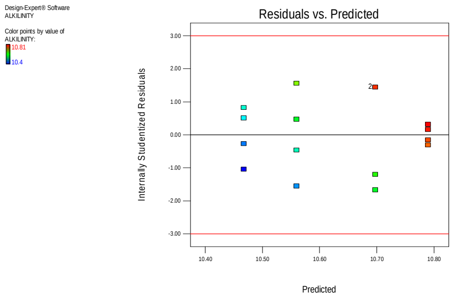


Figure 4: Plot of residual vs. predicted values

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

One factor effects

From the statistical analysis done, it showed that of all the single factor effects studied; only the effect D which was the solute/solvent volume ratio had effect on the alkalinity.

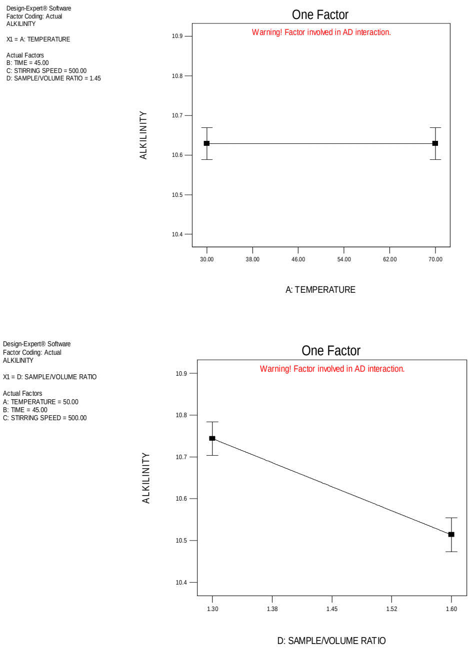


Figure 5: Single effect plot of (a) temperature (b) Sample/ solvent volume ratio

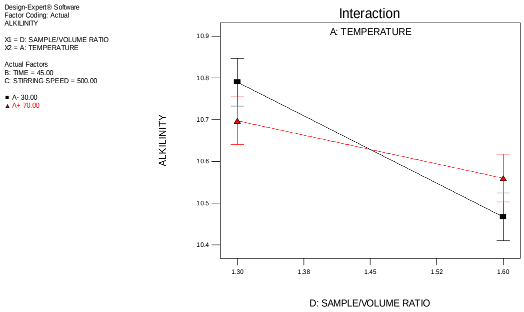
From Figure 5 above, effect D had a negative effect on the alkalinity, which showed that as the quantity of distilled water was increased with the ash content constant, the alkalinity decreased. This meant that as the quantity of the water was increased at the constant value of the ash, it tends to dilute the solution thereby reducing the alkalinity of the solution.

But the other factors had constant alkalinity level at different levels of the factors studied.

Interaction effects

Interactions occur when the effect of one factor depends on the level of the other. From the analysis, it is only the interaction effect of Temperature and sample/volume ratio that was significant.

From Figureure 6a below, it showed that when temperature was low (black line), the line angle's steeply downward, indicating a strong negative effect due to increase in sample/solvent ratio. The same thing happened when temperature was high (red line), the line angles steeply downward indicating a strong negative effect due to increase in sample/solvent volume ratio.



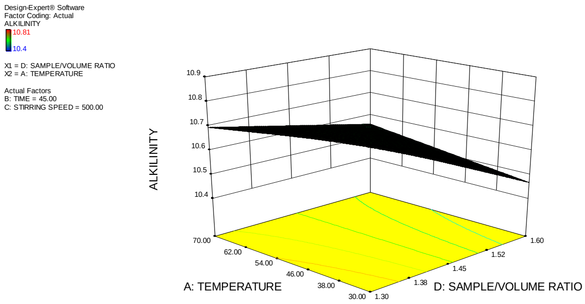


Figure 6: (a) Interaction effect of sample/solvent volume ratio and temperature (b) 3D surface plot

The 3D surface plot equally gave the same interpretation.

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, Temperature, Time, Stirring Speed and Sample/Solvent ratio were 30^{o}C, 45minutes, 500rpm, and 1:30 respectively.

At the desirability of 0.951, the process had the maximum alkalinity of 10.79 at the operating conditions.

CONCLUSIONS

The leaching of alkali from local ash obtained from female inflorescence of palm oil tree was investigated. The effects (Single and interaction) of the leaching variables were studied using 2 level full 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 10.79 at temperature of 30^{o}C, time of 45minutes, stirring speed of 500rpm and 1:30 solute/solvent ratio.

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Table 1

Factors and levels for 2-level full factorial design.

S/No	Factors	Units	Low Levels	High Levels
1	Temperature	^{o}C	30	70
2	Time	Minutes	30	60
3	Stirring Speed	Rpm	250	750
4	Solute/solvent volume ratio	-	1:30	1:60

Table 2

Design Layout with actual and predicted response values

Std order	Run order	Temperature (^oC)	Time (minutes)	Stirring speed (Rpm)	Sample/ solvent ratio	Alkalinity (Actual)	Alkalinity predicted
11	1	30.00	60.00	250.00	1:60	10.50	10.47
6	2	70.00	30.00	750.00	1:30	10.59	10.70
16	3	70.00	60.00	750.00	1:60	10.59	10.56
14	4	70.00	30.00	750.00	1:60	10.60	10.60
2	5	70.00	30.00	250.00	1.30	10.79	10.79
10	6	70.00	30.00	250.00	1:60	10.53	10.79
15	7	30.00	60.00	750.00	1:60	10.45	10.47
7	8	30.00	60.00	750.00	1:30	10.81	10.79
8	9	70.00	60.00	750.00	1:30	10.79	10.70
12	10	70.00	60.00	250.00	1:60	10.64	10.56
5	11	30.00	30.00	250.00	1:30	10.8	10.79
5	11	30.00	30.00	750.00	1:30	10.8	10.79
9	12	30.00	30.00	250.00	1:60	10.40	10.47
13	13	30.00	30.00	750.00	1:60	10.40	10.47
1	14	30.00	30.00	250.00	1:30	10.78	10.78
4	15	70.00	60.00	250.000	1:30	10.62	10.70
3	16	30.00	60.00	250.00	1:30	10.77	10.79

In full factorial multivariate experiment, all main factors and their interactions are compared with one another[9].

Table 3

ANOVA TABLE

Source	Sum of square	df	Mean	F – value	P – value prob > F
Model	0.25	3	0.082	14.86	0.0002 significant
A – Temperature	0.000	1	0.000	0.000	1.0000
D – Sample/Solvent	0.21	1	0.21	38.39	<0.0001
Ratio AD	0.034	1	0.034	6.21	0.0283
Residual	0.066	12	512E-003		
C or Total	0.31	15			

R – Squared = 0.7880, Adj R – Squared 0.7350, Pred R – Square = 06230 adq precision =- 8.687.

The "Pred R – Squared" of 0.623 was in reasonable agreement with the "AdjR – Squared" of 0.7350.

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