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Optimum Parameters for Bleaching of Crude PKO using Activated Snail Shell

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Abstract- The optimum parameters (temperature, contact time and adsorbent-to-crude PKO ratio) were determined for the bleaching of PKO using activated snail shell. A D-optimal design for twelve experimental runs at three levels, for temperature, contact time and different amounts (masses) of activated pulverized snail shell (adsorbent-to-crude PKO ratio) was used for the study, and the resulting data fit to a quadratic model. The model adequacy and statistical significance of coefficients were also determined. MATLAB 7.0 was used in the optimization and 3-D plots. The results reveal that the effect of temperature and adsorbent-to-crude PKO ratio were significant and non-linear (quadratic), while time has a non-linear (quadratic) but insignificant effect on the extent of bleaching. The optimum values for temperature, time and adsorbent-to-crude PKO ratio are 187.85^{O}C, 45mins and 2.34: 20 (g/ml) respectively.

Keywords- *snail shell, pulverised, bleaching, activated, palm kernel oil.*

I. Introduction

Palm kernel oil (PKO) is black viscous oil extracted from the kernel of oil palm. In its raw form it contains impurities such as organic pigments, oxidation metals, trace metals and traces of soap. For PKO to be used effectively, in most industrial processes, these impurities in it have to be extensively removed, thus making bleaching inevitable (Young, 1982; Macrae, 1993; Campbell et. al, 1999).

Bleaching involves a mass concentration of the colour pigment at the interface between the fluid and the bleaching agent. it is achieved as a result of intermolecular forces between molecules of solid and the substances adsorbed and is readily reversible (Richardson, Harke and Backhurst, 2002).

Adsorption bleaching is the most effective form of bleaching in which various adsorbents like carbon, silica gel, activated alumina and activated clay are used. The bleaching agent should be one that will change the tint of the oil without altering the chemical properties of the oil (Purvis, 1975; Parker 1987; www.fullerseearth.com, 2005).

Several bleaching agents, especially clays, have been studied in recent times for various bleaching temperatures and times. Clays that have been studied include acid, neutral and caustic activated clays (Brophy et. al 2004; Arumughan et. al 2004; Okwara and Osoka, 2006). Clays are non- renewable resources. This work studies the optimal conditions, with respect to bleaching temperatures and contact time (Malek and Farooq, 1997) for the use of snail shell (a renewable resource) as an adsorbent in the bleaching of PKO.

Snail shell is ideal for this process because it contains calcium carbonates (a carbonaceous material), and much of the substances volatilizes on heating, leaving behind a porous structure of carbon that usually contain some hydrogen. This may, then, be activated to further open up the pores and increase total surface area. In addition, it is also passive to crude PKO.

II. METHODOLOGY

A. Apparatus/Reagents

Stainless bowls, Stirrer, roller mill, 79 micrometer sieve and 0.5mol/dm3 dilute HCl, 0.3N NaOH, beam balance, heating furnace, stop watch, beaker, cylinder, filter paper, 85% H_3PO_4, centrifuge, electric oven with thermostat, spectrophotometer and test tubes.

B. PROCEDURE (Pulverization of Snail Shell)

The snail shell was first washed with water and sun dried. It was then manually broken into smaller pieces, washed with 0.5 mol/dm^3 of dilute HCl, to remove other remaining forms of impurities and sun dried, then it was ground into powder in a roller mill and finally sieved using 79 micrometer sieve.

C. PROCEDURE (Thermal Activation of the Snail shell).

60g of the sieved shell powder were heated in an electric furnace up to a temperature of 300^{O}C for 30 minutes such that its colour changed from brown to white. It was then allowed to cool to room temperature.

D. PROCEDURE (Chemical Activation of the Snail shell)

50g of the thermally activated snail shell was reacted with 20ml of 85% H_3PO_4 in a beaker with continuous stirring for 2mins. The reaction was exothermic, and a gas with an offensive smell was given off. The reaction mixture was mixed with 25ml of distilled water to wash away the unreacted

H_3PO_4, then it was filtered and the residue collected allowed drying.

E. PROCEDURE (Bleaching of Crude PKO)

The transmittance of the crude PKO was first determined using spectrophotometer at 400nm to serve as a control. Different masses (1g, 2g, and 3g) of the activated snail shell were weighed out using the beam balance.

1g of the activated snail shell was transferred to a test tube and 20ml of crude PKO was added. The mixture was transferred to a test tube and 20ml of crude PKO was added. The mixture was well stirred and 0.3 NaOH was added to neutralize the fatty acid present in the oil. The mixture was poured into three different test tubes. The three samples were heated in an electric oven at a constant temperature of $120^{O}C$.

During the heating one of the sample test tubes were removed after 15min of heating, the next was removed after 30 min and the third sample remove after 45min. The samples were then labelled and centrifuged. After centrifugation, the top layer of each sample was taken to the spectrophotometer at 400nm to determine the transmittance. The procedure was repeated for the twelve experimental runs with three replications.

The transmittance was inverted to obtain the opacity (Q) of the sample. The percentage colour reduction was obtained thus (Qc – Q)/Q*100.

Where Qc is the opacity of the control. Control: Transmittance of crude PKO = 0.01(Qc=100).

III. RESULTS AND ANALYSIS

Table1. Percentage Colour Reduction For all Experimental runs.

Decolourisation (%)	Temperature (^{O}C)	Time (mins)	Amount of Adsorbent (g)
88.89	120	45	3
99.01	160	45	2
85.71	120	15	3
75.00	120	45	1
99.04	200	45	1
98.99	160	30	3
75.00	120	15	1
99.22	200	15	3
90.00	200	15	1
99.23	200	45	3
99.22	200	15	2
83.33	120	30	2

The experimental results were fit to the quadratic model below using The Statistical Toolbox of MATLAB 7.0.

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_4x_1x_2 + b_5x_1x_3 + b_6x_2x_3 + b_7x_1^2 + b_8x_2^2 + b_9x_3^2 \quad (1)$$

Model coefficients were obtained and the adequacy of the model was determined using f-test and R2, while regression coefficient was used to determine the significance of the model coefficients and the model accuracy determination was based on the residuals.

From the results of the fit, not all model equation coefficients are significant but the model is adequate for the fit at 90% confidence interval based on the f-test and R2. The residuals also reveal that the model is accurate in its prediction of the responses in comparison to experimental values.

Table 2: Table of t-test and f-test statistics

	Values of coefficients	rcv	rcvsq	F-statistics
b_0	-61.728			SSE = 20.482
b_1	1.4122	0.7879	0.6208	SSR = 979.26
b_2	-0.12497	0.1202	0.0145	DFE = 2
b_3	26.431	0.4443	0.1974	DFR =9
b_4	0.0010439	0.4583	0.2100	F = 10.624
b_5	-0.047469	0.7104	0.5047	P –value = 0.088945
b_6	-0.04875	0.3530	0.1246	R^2= 0.9795
b_7	-0.0035876	0.7670	0.5883	RMSE=1.3065
b_8	0.0035876	0.1191	0.0142	
b_9	-3.267	0.4159	0.1730	

The model equation was optimized using the optimization Toolbox of MATLAB 7.0, to obtain the optimum parameters for bleaching of palm kernel oil using activated snail shell and the results were obtained as:

- Temperature: $187.8494^{O}C$
- Time: 45 minutes
- Adsorbent-to-crude PKO ratio: 2.3448:20 g/ml

Based on the model, 100% bleaching or decolourization of crude Palm kernel oil will be achieved at this optimum condition.

Table 3: Table of Residuals

Measured values	Predicted values	Residuals	Percentage Deviation (%)
88.89	87.256	1.6337	1.8379
99.01	99.440	-0.42975	0.4340
85.71	87.236	-1.5263	1.7808
75.00	76.311	-1.3114	1.7485
99.04	97.406	1.6337	1.6495
98.99	98.56	0.42975	0.4341
75.00	73.366	1.6337	2.1783
99.22	98.231	0.98906	0.9968
90.00	91.956	-1.9560	2.1733
99.23	100.76	-1.5263	1.5381
99.22	98.361	0.8595	0.8663
83.33	83.760	-0.42975	0.5157

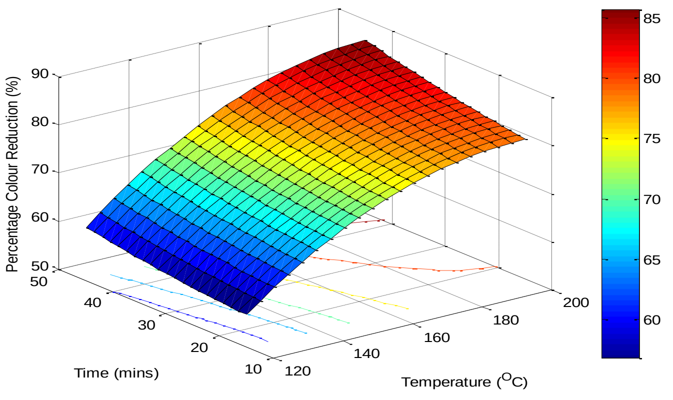

Fig 1: 3-D Plot of Percentage Colour Reduction with Temperature and Time as variables

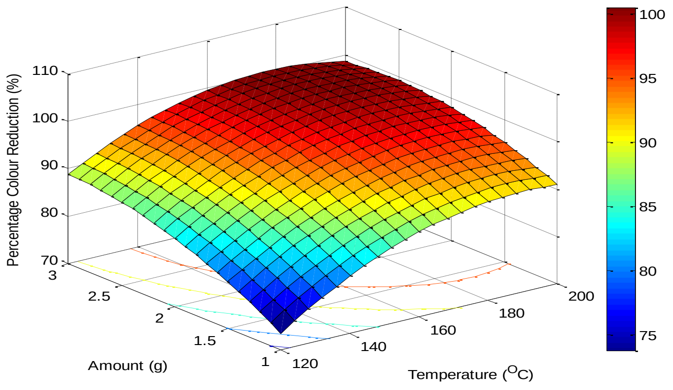

Fig. 2: 3-D Plot of Percentage Colour Reduction with Temperature and Amount as variables

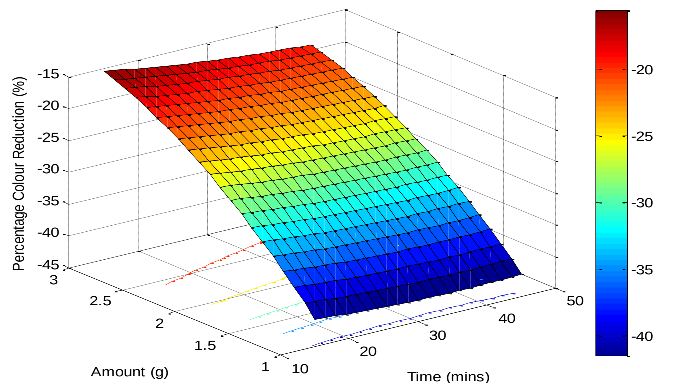

Fig. 3: 3-D Plot of Percentage Colour Reduction with Time and Amount as variables

IV. DISCUSSION

The variables considered in the bleaching process were bleaching temperature and contact time and mass of the activated snail shell (adsorbent-to-crude PKO ratio).

Table 2 shows that temperature is a significant variable, while time is not significant. The linear coefficient for temperature is more significant than its interaction and quadratic coefficients. A similar relationship also exists between the linear and quadratic terms of time and adsorbent-to-crude PKO ratio. The profile of decolourization of crude PKO with time looks linear. This is evident from the 3-D plots of Fig. 1, 2 and 3. The interaction coefficient for temperature and amount of adsorbent (adsorbent-to-crude PKO ratio) is the most significant interaction term, thus, temperature and amount of adsorbent have more significant impact on decolourization of crude PKO than time, within the range of experimental study, and the profile looks quadratic. Percentage colour reduction (decolourization) of crude PKO generally increases with temperature, time and adsorbent-to-crude PKO ratio.

Table 3 shows that the maximum percentage deviation of the predicted response values from the experimental is 2.1783%. This is well below the generally accepted maximum of 5%. Thus the model is accurate.

V. CONCLUSION

Crude PKO can be decolourized using activated snail shell. The functional parameters are temperature, time and adsorbent-to-crude PKO ratio. The decolourizing effect of activated snail can be adequately modeled with a quadratic response surface model on the above three functional parameters and the extent of bleaching increases non-linearly with temperature, time and adsorbent-to-crude PKO ratio

It is recommended to decolorize crude PKO using a ratio of 2.34:20 g/ml of snail shell to crude PKO at a temperature of 187.85^{O}C for 45 min.

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