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EFFECT OF PROCESS VARIABLES ON THE HYDROGENATION OF REFINED PALM OIL

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Abstract— The aim of this research is to analyze the effects of some process variables such as temperature, pressure, and agitation on the hydrogenation of refined palm oil so as to obtain a good yield of modified fat. This was done in a small-scale laboratory. Iodine's test for unsaturation was used to test the saturation levels of the palm oil, the concentration of the oil was measured, and then the experiment was carried out using nickel catalyst to hydrogenate the oil. The variables were analyzed separately keeping the other two constant. Design software like Mat lab version 2013 was used to design the experiment and analytical software like Microsoft excel was used to run the R^2 Adjusted R^2 and ANOVA analysis. Twenty runs of experiments was carried out with the different variables. The result obtained for the modified fat for temperatures 40-80°C and the optimal yield was 95% for 80°C. Pressure of 1-5atm has an optimal yield of 90% for 5atm and for agitation 100-500rpm the optimal yield was 95% for 500rpm. In conclusion increase in the variables increases the yield of the hydrogenated fat.

Keywords— Temperature, pressure, agitation, ANOVA, nickel catalyst.

I. INTRODUCTION

Palm oil is an edible vegetable oil derived from the mesocarp (reddish pulp) of the fruit of the oil palms, primarily the African oil palm *Elaeis Guineensis* and to a lesser extent from the American oil palm and the maripa palm *Attalea Maripa*. Palm oil is naturally reddish in colour because of a high β -carotene content. It is not to be confused with palm kernel oil derived from the kernel of the same fruit or coconut oil derived from the kernel of the coconut palm (*Cocos nucifera*). The differences are in colour (raw palm kernel oil lacks carotenoids and is not red), and in saturated fat content: Palm mesocarp oil is 49 percent saturated, while palm kernel oil and coconut oil are 81 percent and 86 percent saturated fats, respectively.

However, crude red palm oil that has been refined, bleached and deodorized, a common commodity called RBD palm oil, does not contain carotenoids.

Along with coconut oil, palm oil is one of the few highly saturated vegetable fats and is semisolid at room temperature. Palm oil is a common cooking ingredient in the tropical belt of Africa, Southeast Asia and parts of Brazil. Its use in other parts of the world is widespread because of its lower cost and the high

oxidative stability (saturation) of the refined product when used for frying. One source reported that humans consumed an average 17 pounds (7.7kg) of palm oil per person in 2015 (Raghu, 2017).

Hydrogenation converts liquid vegetable oils into solid or semi-solid fats, such as those present in margarine. Changing the degree of saturation of the fat changes some properties, such as the melting range, which is why liquid oils become semi-solid. Solid or Semi-solid fats are preferred for baking because the way the Fat mixes with flour produces a more desirable texture in the baked product.

Because partially hydrogenated vegetable oils are cheaper than animal fats, are available in a wide range of consistencies, and have other desirable characteristics (such as increased oxidative stability and longer shelf life), they are the predominant fats used as shortening in most commercial baked goods.

A side effect of incomplete hydrogenation having implication for human health is the isomerization of some of the remaining unsaturated carbon bonds, resulting in the trans isomers, which have been implicated in circulatory diseases including heart disease. The conversion from cis to trans bonds is favoured because the trans configuration has lower energy than the natural cis one. At equilibrium, the 'trans/cis isomer ratio is about 2:1. Many countries and regions have introduced mandatory labelling of trans fats on food products and appealed to the industry for voluntary reductions.

II. PALM OIL

Palm oil is an important and versatile raw material for both food and non-food industries (Gan, 2012). Recently, palm oil has become the second most consumed oil all over the world with a competitive price compared to other edible oils (Bazlul, 2010). The oil palm is as old as creation. Every part of the tree is useful economically and for domestic purposes (Obahiagbon, 2012). The oil palm is grown commercially in Africa, South America, South Asia and the South Pacific, and on a small scale in other tropical areas. Until recent centuries the palm has been confirmed to West and Central Africa where it existed in a wild, semi-wild, and cultivated state. In Africa it remained a domestic plant, supplying a need for oil and vitamin A in the diet, and it was not until the end of the eighteenth and the beginning of the nineteenth the entry of palm oil into the world oils and fats trade (Shahidi, 2015).



Palm oil is one of the 17 major oils and fats produced and traded in the world today. Within the span of four decades, palm oil has emerged as the fastest growing oil in the world. In fact, palm oil is projected to be the world's largest oil produced, although it is currently occupying second position after soybean oil (Bahruddin et al, 2013). Most palm oil is currently produced in Southeast Asia, even though the oil palm is originally an Africa crop, which was introduced to Southeast Asia in the 19th century. The two largest producers are Malaysia and Indonesia, who together account for roughly 85% of the world palm oil production.

Palm oil, obtained from a tropical plant, *Elaeis guineensis*. The genus *Elaeis* comprises two species, namely *E. guineensis* and *E. oleifera*. Palm oil is a lipid extracted from the fleshy orange red mesocarp of the fruits of the oil palm tree. The oil palm tree has an unbranched stem and belongs to the palm family. The tree, which can grow to a height of 20 to 30 meters, has an economic life span of 25 to 30 years. The female bunch produced can weigh as much as 30-40kg and contain up to 2000 fruits which are black in colour when young, turning to orange-red when ripe (Kalyana et al 2012). Malaysia has been the leading exporter of palm oil, reflecting the country's large production and refining capacities, its small domestic market for palm oil. In much of Asia, palm oil is popular because of its relatively low price vis-à-vis soybean oil, its main competitor, reinforced by the modest freight costs from Southeast Asian suppliers. Also. The temperatures in the tropical/subtropical regions are warm enough to allow refined bleached deodorized (RBD) palm olein to be used as household oil, without fear of clouding. Today, palm oil is the most widely used vegetable oil in the world. Palm oil and palm kernel oils accounted for 33% of total vegetable oil production, soybeans are the next largest source of vegetable oil 27%, followed by rapeseed (the basis of canola oil) 15% in 2011. In addition, Palm oil was not always the most widely used vegetable oil in the world. In 1961, only 1.5million tonnes was produced, compared to 3million tonnes of soybean oil. In 2006, however, palm oil production surpassed soybean oil production for the first time, and by 2011, world production of palm oil amounted to 48.6million tonnes, compared to 41.6million tonnes of soybean oil.

Table 1.1: World Supply and Distribution (Thousand Metric Tons)

Producti on	2010/11	2010/12	2010/13	2010/14	2010/15	2010/16
Indonesia	23,600	26,200	28,500	30,500	33,000	33,000
Malaysia	18,211	18,202	19,321	20,161	21,250	21,250
Thailand	1,832	1,892	2,135	2,150	22,250	2,250
Colombia	753	945	1,140	1,042	1,070	1,070
Nigeria	850	850	910	930	930	930
Other	3,590	4,022	4,138	4,276	4,293	4,293

Table 2.2: Major physical properties of palm oil

Property	Mean(215 samples)	Range
Apparent density at 50°C(g/ml)	0.889	0.888-0.889
Refractive index at 50°C	1.455	1.455-1.456
Solid fat content		
5°C	60.5	50.7-68.0
10°C	49.6	40.0-55.2
15°C	34.7	27.2-39.7
20°C	22.5	14.7-27.9
25°C	13.5	6.5-18.5
30°C	9.2	4.5-14.1
35°C	6.6	1.8-11.7
40°C	4.0	0.0-7.5
45°C	0.7	
Slip melting point (°C)(+)	34.2	31.1-37.6

Table 2.3 physicochemical properties of palm oil and its fractions

	Melting point (°C)	34.2	21.6
Melting Point (°C)	34.2	21.6	44.5-56.2
Relatively density (50°C/water at 25°C)	0.89-0.92	0.91-0.92	0.88-0.92
Refraction Index (η)	1.46	1.47	1.45
Moisture and impurities (%)	0.1	0.1	0-0.15
Iodine Value	47-55.83	55.0-61.54	21.6-49.4
Saponificatio n Value (mg KOH/g)	196-208.2	189-198.0	193-206
Unsaponifiabl e matter (%)	0.01-0.5	0.001-0.5	0.1-1.0



III. CHEMICAL PROPERTIES OF PALM OIL

Chemical Properties of Palm Oil Palm oil consists of mostly glyceridic materials with some non-glyceridic materials in trace amount. TAG is the most abundant glyceridic component in palm oil which comprises of triesters of high aliphatic acids or fatty acids, while monoacylglycerol (MAG) and diacylglycerol (DAG) are the minor glyceridic components in palm oil. The chemical structures of partial acylglycerols (MAG and DAG) and TAG were shown in Figure 1.1

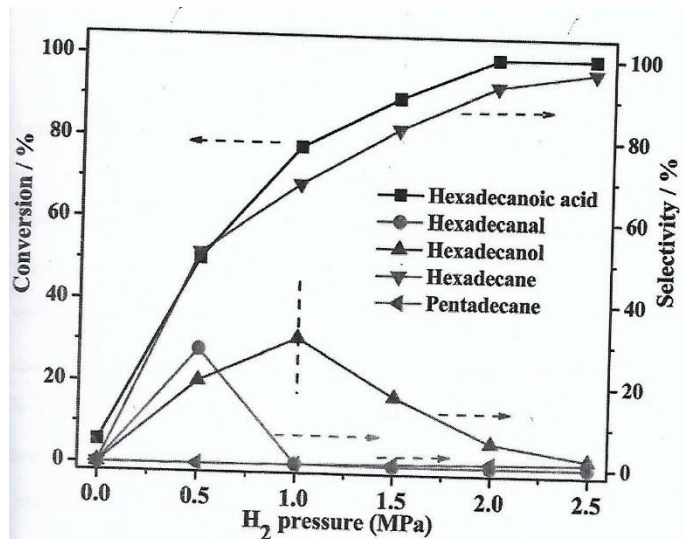
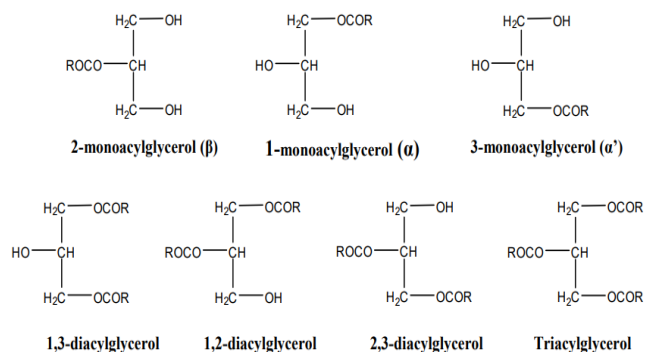


Fig 2.2 Effect of hydrogen pressure on palm using different solvents

Table 2.4: Effect of pressure on the hydrogenation of soy oil.

Entry	Pressure (bar)	Time (hrs)	Conversion (%)	Selectivity (%)
1	10	72	65	100
2	20	72	100	98
3	50	72	100	65

Temperature Relationship with Conversion

Sample	Temperature	Content (Mole/15kg)	Conversion (%)	Selectivity C18:0/TFA
32	99	$F_i = 2.43$ $F_0 = 3.45$ $D = 1.02$	5.16	1.48
22-A	130	$F_i = 2.43$ $F_0 = 5.54$ $D = 3.11$	17.70	0.48
27-A	137	$F_i = 2.43$ $F_0 = 6.38$ $D = 3.95$	23.37	0.77
29-A	149	$F_i = 2.43$ $F_0 = 8.12$ $D = 5.69$	32.03	0.78

IV. IODINE VALUE

The iodine value (or iodine absorption value or iodine number or iodine index) in chemistry is the mass of iodine in grams that is consumed by 100 grams of a chemical substances. Iodine numbers are often used to determine the amount of unsaturation fatty acids. This unsaturation is in the form of double bonds, which react with iodine compounds. The higher the iodine number, the more C=C bonds are present in the fat. This particular analysis is an example of iodometry. A solution of iodine is yellow/brown in colour. When this is added to a solution to be tested, however, any chemical group (usually in this test C=C double bonds) that react with iodine effectively reduce the strength, or magnitude of the colour (by taking iodine out of solution). Thus, the amount of iodine required to make a solution retain the characteristics yellow/brown colour can effectively be used to determine the amount of iodine sensitive groups present in the solution.

The chemical reaction associated with this method of analysis involves formation of the diiodo alkane (R and R' symbolize alkyl or other organic groups).

The precursor alkene (RCH=CHR') is colourless and so is the organ iodine product (RCHI=CHIR').

In a typical procedure, the fatty acid is treated with an excess of the Hanus or Wijs solution, which are, respectively, solutions of iodine monobromide (IBr) and iodine monochloride (ICI) in glacial acetic acid. Unreacted iodine monobromide (or monochloride) is then allowed to react with potassium iodide, converting it to iodine, whose concentration can be determined by titration with thiosulfate. Table 2.4 shows the iodine value of different oils.

Table 2.6: Iodine Value of Fat and Oils

Oil/Fat	Iodine Value
Palm Oil	44-51
Butter	26-40



Olive Oil	80-88
Castor Oil	82-90
Coconut Oil	7-10
Palm Kernel Oil	16-19
Cocoa butter	35-40

V. MATERIAL AND METHOD

The following under listed materials were used to carry out the experiment

- a. Funnel
- b. Pipet
- c. Conical Flask
- d. Measuring Cylinder
- e. Beaker Stirrer
- f. Water Bath
- g. Syringe
- h. Balloon
- i. Electronic weighing balance
- j. Spatula
- k. Magnetic Stirrer
- l. Burette
- m. Water Bath
- n. Filter paper
- o. Thermometer
- p. Conical Vial
- q. Clamp
- r. Matlab Version 2014
- s. Microsoft Excel
- t. Hydrogen gas
- u. Nickel catalyst
- v. Iodine
- w. Refined Palm oil
- x. Isopropanol
- y. Hydrogen gas

VI. METHODS

Quantitative Micro scale Hydrogenation of Palm Oil

A 250ml beaker was filled with water and heated on a water bath to maintain a steady temperature of 40°C. 0.10g of refined palm oil was added to the vial, 3.0ml of isopropanol(solvent), and a spin vane, the concentration of the oil was measured. A small amount of 10% nickel catalyst (approximately 15mg) was added to the conical vial and the vial was secured to the take-off adapter. The vial was then clamped above (not in) the water bath. A 20ml syringe fitted with a small gauge needle was used to remove air from the system. The septum on the take-off adapter was pierced and enough air was removed from the system to draw water up into the graduated pipette to the 0.0ml mark (pipette mostly filled with water). The syringe was then

removed. The constancy of the water level shows that the system was leak proof. The system was then charged with hydrogen using a gas cylinder with different pressure gauges and filled with hydrogen secured to a hose set at 1atm. The needle of the balloon assembly was pushed through the septum on the take-off adapter and the stopcock was opened. The flow of gas was used to push all the water out of the pipette and was continued for an additional 20seconds to ensure excess hydrogen for the reaction was in the system.

The Hose connected to the pressure gauged was removed. Using the syringe some hydrogen was removed from the system to bring the water level up to the 22.0ml mark (pipette nearly empty of water). The conical vial was lowered into the water bath and the stirrer was turned on to a constant speed of 300rpm. The water level in the pipette was measured every five minutes while the reaction agitation and pressure was maintained. When the uptake of hydrogen was slowed to less than 0.2ml per five-minute period (usually within two hours) a final reading was made and the conical vial is removed from the water bath. The product was kept in an appropriately labelled container and measured. The experiment was repeated at the temperatures of 50°C, 60°C, 70°C, 80°C while the agitation and pressure was maintained at 300rpm and 1atm respectively. After the temperature has been experimented on the pressures 5atm, 10atm, 15atm, 20atm, 30atm was experimented with a constant temperature and agitation of 25°C and 300rpm. After temperature and the pressure have been experimented, the agitation was then experimented at irrespective of the variable used and the data from the measurement of the oil and fat was used to get the yield.

VII. ANALYTIC TECHNIQUES AND DETERMINATION OF YIELD

This is an expression of how much fat was produced in the hydrogenation process relative to the amount that could be theoretically produced in a standard experiment. It is obtained using equation 3.1

$$\text{Yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100 \quad 3.1$$

$$\text{Actual yield} = \frac{\text{Concentration of fat produced}}{\text{Initial concentration of oil}} \quad 3.2$$

VIII. RESULTS AND DISCUSSION

Qualitative Analysis

The qualitative analysis of the effects of some variables on the hydrogenation of refined palm oil extract was performed using the standard methods. The results are presented in figure 4.1 to 4.3

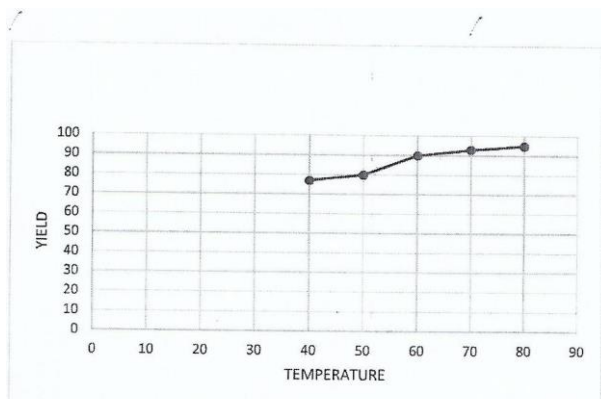


Fig. 4.1 Result of Temperature effects at 1atm and 300rpm

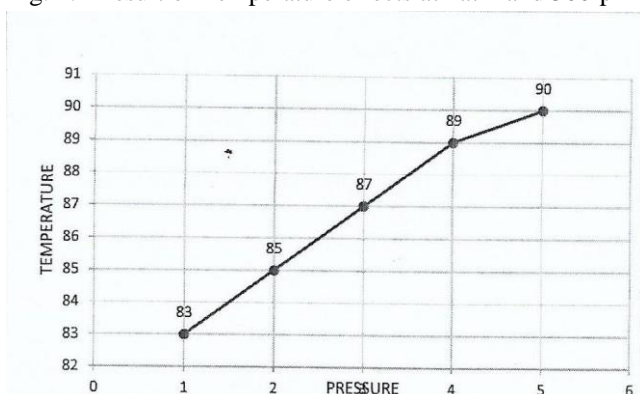


Fig 4.2 result of pressure effects at 25°C and 300rpm

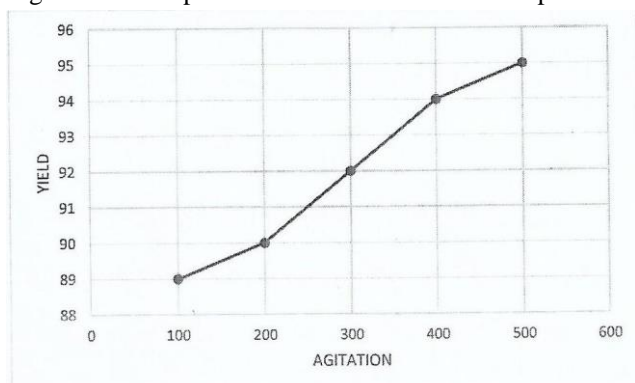


Fig 4.3 Results of agitation effects at 25°C and 1atm

Discussion

Hydrogenation of refined palm oil was monitored by optimizing some process variables which are temperature pressure agitation and catalyst concentration. Table 4.1 shows the model of the performed experiment which was designed using Microsoft Excel. From the table 4.1, there were five experimental runs of three different parameters which are temperature, agitation and pressure using excel spreadsheet for objective of this experiment to be feasible. Since some process parameters were kept constant so as to actually know the optimal variables that are required for high yield in refined palm oil hydrogenation, so

when the other variables were set at their predetermined values, the yield responses were obtained, it was maximum where pressure, temperature, and agitation were 5atm, 80°C and 400rpm respectively.

Table 4.5 & 4.6 shows the fit summary for model selection. As can be seen from the table, quadratic model was suggested. Quadratic model was chosen due to its quality over other models. It has a lowest standard deviation value of 0.4671 compared to other models. This is an indication that the chosen model has data points which tend to be close to the mean (the expected value) R^2 of 0.9715, adjusted R^2 of 0.9589, predicted R^2 of 0.8847 and finally a predicted error sum of squares (PRESS) of 12.69. The good thing about this model is that the predicted R^2 is in reasonable agreement with the adjusted R^2 since there difference is less down 0.002, hence the model fits the data well. And having a low PRESS value of 12.69 compared to other models also indicates a tight fit of the data.

Table 4.7 shows that the F value of the chosen model is 37.91, this implies that the model is significant also, P value less than 0.0001 is also an indication that the model terms are significant. In this case, T, P, AT, AP, TP, A^2 , T^2 , P^2 are all significant models terms. P value greater than 0.05 indicates that the model terms are not significant. Adequate precision in Table 4.8 measures the signal to noise ratio. A ratio greater than 4 is desirable. Having a ratio of 12 indicates an adequate signal. Table 4.9 shows model optimization which searches for a combination of factor levels that simultaneously satisfy the criteria placed on each response and factors.

The yield of hydrogenated fat was increasing with increase in all the variables. This shows that the variables have are direct proportionality to the yield. This is in agreement with the work done by Gunstone et al (2013) for temperature (swern, 2013) for pressure and agitation.

Another important factor is that at higher rates of temperature, pressure and agitation the proportionality may not be direct, for example if the agitation rate exceed 5000rpm it no longer affect the reaction same principle work for other variable too. The activity of the catalyst should not be neglected though it has little to offer in the overall kinetics of the reaction. In general, the elevation of hydrogen pressure and temperature causes the production of highly saturated material, which is normally obtained when large quantities of hydrogen are used (H_2), and a decrease in activation energy, resulting in an increase in the reactivity.

IX. CONCLUSION

From the results of this study, the following conclusion can be drawn:

The hydrogenated fat yield, with reasonable accuracy was obtained within the range of the experimental condition used in this study.

A study of the variables which affect the experiments shows agitation rate to be the most important.



The agitation rate was shown to have significant effects on the yield, not only by itself but also together with other independent variables (Temperature, Pressure and agitation), that is, in interaction with two and three variables. All variables affect the yield in the same way, increase in temperature leads to high yield so as the hydrogen gas pressure and higher agitation.

Although the equation developed by this study is empirical, it is useful. Similar equations could be developed for full-scale hydrogenation equipment. These equations would permit the calculation of the conditions necessary to produce a desired product without experimental hydrogenation in small equipment.

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