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# DETERMINATION OF THE FACTORS THAT AFFECT THE QUALITY AND STABILITY OF PHYSICALLY REFINED PALM OIL

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## ABSTRACT

*This work investigated the role of the bleaching stage on the quality and stability of physically refined palm oil. Raw palm oil obtained from Adah palm in Imo State, was characterized before and after refining and used in the investigation. The result showed that colour reduction depends on the conditions of bleaching and the nature of the bleaching clay used. It was established that reduction during deodorization, is mainly due to the decomposition of the carotene pigments at the temperature of deodorization. The stability standard of the refined oil was measured in terms of colour, Free Fatty Acid, Peroxide Value and Anisidine Value, which were compared with those of the American Oil Chemist Society (AOCS).*

**Key words**: *Bleaching, refining, decomposition, characterization, quality, and stability,*

## 1 INTRODUCTION

Vegetable oils are water insoluble substances of plant origin, which consists predominantly of glycerol esters of fatty acids or triglycerides, (Anderson A.J.C, (1998) and (Hoffmann,1989). Demand for refined vegetable oils has increased the world over in the past few years due to increase in world population, rising standard of living and consumer preference. Its needs for consumption and industrial purposes will increase tremendously in the near future. In the energy sector, vegetable oil will provide the source of bio-energy and renewable energy requirements of the world, (Pahl G, 2005).

Vegetable oils find most application as cooking and frying oils as well as in the manufacture of margarine, shortening, baker's fat, soap, grease and lubricants, creams, etcetra, and hence the need to stabilize its quality for these purposes.

Many factors influence the stability of refined oils and have been the subject of much studies. The triglycerides contain approximately 95% of fatty acid and 5% glycerol combined as part of the glyceride molecules and the reactive portion, Okiy, (1978). Type of raw oil, its colour, free fatty acid content, taste, and other physical and chemical properties are such other parameters that need specific attention in order to obtain the much needed quality of the finished product. Refining is carried out with a view to purifying the glycerides and to remove all objectionable properties which make oil unstable, Moor, (1978). Bailey, (1964) asserts that chemical and physical properties of fats and oils are determined by the properties of their component fatty acides. Okiy, also noted that, as the average molecular weight of fatty acid increases, fats progressively have higher melting points and can easily solidity. The raw oil has to be degummed, bleached, and deodorized in order to remove its objectionable properties. The bleaching process is a well known and established operation in the processing of edible oil and a major stage for the stabilization of the final product. This is because, all the oxidation components of raw oil are removed in this stage. Hymore and Ajayi, (1989), Egbuna and Aneke, (2005), had demonstrated that local activated clay can effectively remove carotenoids from palm oil. Mahatta, (2006), asserts that degumming should be carried out before bleaching so as to reduce the tendency of filter media being clogged during bleaching by phosphotide, a gummy substance found especially in palm oil. Proper filtration, according to Soon et al,(1987), should be carried out to reduce entrainment of particles which will

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enter the high temperature of deodorizer to be charred and cause colour reversion and fixation.

The aim of the present investigation is to demonstrate how an effective bleaching stage can lead to good oil quality and stability. It will also show how the quality of the refined palm oil is affected by the conditions under which the bleaching in carried oil.

## 2 EXPERIMENTAL

**Materials/Equipment:** The following materials and equipment were used in the investigation; raw palm oil collected from Adam Plam industry, Imo State, bleaching earth (Activated clay), titration apparatus, a set of sieves, lovibond Tintometer, steam/vacuum apparatus, distillation apparatus and test chemicals. Table 1 shows the physio-chemical properties of the palm oil used in the investigation, while Table 2, shows the conditions of temperature and time used in the experiments.

**Properties:** The properties of the oil that were independently determined include, free fatty acid (FFA) of the crude, bleached and deodorized palm oils. These were done by using the standard AOCS test methods. Phosophorous contents (ppm) of the raw, bleached and deodorized oils, Anicidine and peroxide values (AV and PV respectively), in m.eq/kg, were determined using the same test methods of AOCS, (1985) and (Defense and Tirtiaux,1985).

**Sample:** The bleaching earth was sieved to 70-5 microns and the same sample was used throughout the experiment. Samples of refined oil to be used for the stability test were stored in full, glass bottles at 313K for 28 days. Colour, FFA, PV and AV, were measured at intervals.

**Experimental procedure:**
**Bleaching**: The aim of bleaching was to reduce the carotene pigments so as to minimize the formation of hydro - peroxides during deodorization and storage. The experiment was done with the activated clay. 10% by weight of the clay was added to 100g of the oil sample. The mixture was heated to a constant temperature of $100^0$C with stirring for 30 minutes. The oil was then filtered at the same temperature, and the filtrate characterized. Temperature, time and earth dosage were subsequently varied in the bleaching experiment.

**Deodorization;** deodorization, which essentially is steam distillation, was aimed at removing odour, colour, FFA and undesirable materials in the oil. This was done at the temperature of 473K and for 60 minutes. At this condition, the β – Carotene pigment bond was broken and the pigments removed with the odoriferous materials, thereby improving the colour and taste of the refined oil. 1 liter of bleached oil was taken into the distillation apparatus and pre-heated to a temperature of 373K. Steam was generated by heating water in a round bottom flask and passed into the oil through a delivery tube. Temperature was then increased to 473K and vacuum was applied by means of the vacuum pump and maintained at 20mmHg absolute. Vapourized moisture, odoriferous matter, FFA, and colour pigments were condensed in the reflux condenser, which used water as a cooling medium. The condensate, which was essentially FFA, was collected in a beaker. The refined oil was then analyzed for FFA, colour, PV and AV and the results are as presented below.

**Characterization of bleached and deodorized oils**: The bleached and deodorized oil samples were subjected to analyses to determine their physical and chemical properties. Among the properties determined, which will be reported here include; colour, FFA, PV and AV.

**Colour pigments:** pigments present in vegetable oils include mainly carotenoids, chlorophyll, and gossypol. The carotene has been found to be an excellent indicator of crude oil quality

**Procedure:** lovibond Tintometer with 1 inch cell was used for the analysis of colour, and the latter read in terms of red colour band that matched the colour of the refined oils.

**Free fatty acid:** 2.8ml of oil of unknown FFA, was measured into a conical flask and diluted with 35ml of ethanol. A drop of phenolphthalein was added. This was titrated against 0.1N sodium hydroxide solution until a permanent pink colour was observed, and the results recorded.

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Percent FFA = (V x M x N)/10W

where, N – Normality of NaOH; V – Volume of NaOH: W – Weight of oil.

**Oxidation products:** When an unsaturated fatty acid chain reacts with air at room temperature, a process known as auto-oxidation, hydro peroxides are formed,(Mahatta, 2006). At high temperature, these peroxides break down to hydrocarbons, aldehydes and ketones. These cleavage products impart odour and flavor to oil and must be removed.

**Peroxide Value:** This is a measure of primary oxidation whose product is hydrocarbons. These hydrocarbons are further oxidized to water, which causes rancidity of the oil on storage.

**Procedure:** 30ml of chloroform – glacial ethanoic acid mixture in the volume ratio of 1:2 was transferred to a conical flask connected to a reflux condenser. The mixture was then heated and the vapour condensed in the lower part of a jacketed tube. When the reflux became steady, about 1.6ml of potassium iodide was added from the top of the condenser. The precipitate of Kl was dissolved by adding 5 drops of water. The mixture was heated for 5 minutes and 2ml of the oil was pipetted into the mixture through the top of the condenser also. The pipette was rinsed with 2ml of chloroform into the boiling mixture, and boiling continued for 5 minutes. 50ml of distilled water was added. 2ml of the sample was then titrated with 0.02N thiosulphate solution, using starch solution as indicator.

PV = (V x N 100)/G

Where V – vol. of thiosulphate used (ml), N- normality of thiosulphate solution, and G – vol. of sample (ml)

**(ii) Anisidine value:** This measures the amount of secondary oxidation product in a sample of oil. Its products are aldehydes and ketones whose oxidation induces higher rancidity effect to the oil.

**Procedure:** The procedure for analysis of AV, is the same as in the PV, except that the temperature at which these cleavage products are formed is higher.

## 3 RESULTS:

We have investigated how the bleaching stage of palm oil refining affects the quality stability of the final product. The ideal conditions under which the clay should be used to optimize oil stability were also established by monitoring their influence on oil colour, FFA content and peroxide values. These conditions include, temperature, clay dosage, and contact time. Their variation necessitated the modification of the bleaching stage so as to achieve the quality standard mentioned earlier. The results of this work are presented in Tables and graphs.

| **Characteristics** | **Crude** |
|---|---|
| Physical coloud | Deep orange red |
| Odour | Slight |
| Taste | Palm fruit taste |
| Sp. Gravity | 0.92 |
| Melting point | $35^0$C |
| Moisture | 1.5% |
| Refractive index | 1.4512 |
| Free fatty acid (FFA) | 4.9% |
| Colour in 1 inch cell | 22.OR |

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| | |
|---|---|
| Anisidine valuemeg/kg | 8.0 |
| Peroxide value meg/kg | 6.0 |
| Acid value | 10.0 |

**Table 1. Physio- Chemical properties of the palm oil used in the investigation.**

**Table 2. Laboratory physical refining conditions with 1% bleaching earth.**

| Parameters | **Bleaching** | **Deodorization** |
|---|---|---|
| Temperature ($^0$C) | 95 – 120 | 200 |
| Time (minutes) | 30 | 60 |

**Table 3. Laboratory experimental results compared with the international standard. (Test temperature is 100$^0$C, and bleaching earth dosage is 1%)**

| Parameters | Laboratory experiment | | International standard | |
|---|---|---|---|---|
| | Bleached oil | Deodorized oil | Bleached oil | Deodorized oil |
| Colour in1inch cell | 11.5Red units | 3.4Red units | 10.5Red units | 2.5Red units |
| FFA% | 2.8 | 0.12 | 3.5 | 0.1 |
| PV m.eq/kg | 4.2 | 3.00 | 3.2 | 1.0 |
| AV m.eq/kg | 6.4 | 4.05 | 6.0 | 3.7 |

## 4 DISCUSSION

### Variation of bleaching conditions

**Temperature:** Table 4 presents the bleached oil colour, (Red unit), as a function of temperature. The subsequent deodorization oil colour is shown when deodorized at constant temperature of 473K.

**Table 4. The effect of bleach-temperature on the colour, PV, AV and FFA of physically refined palm oil**

| Temperature $^0$C | Colour in 1 inch cell | | Peroxide value | Anisidine value | Free fatty acid |
|---|---|---|---|---|---|
| | Bleached oil | Deodorized oil 473K | | | |
| 20 | 14.2 | 3.8 | 6.5 | 3.60 | 0.62 |
| 40 | 13.8 | 3.7 | 6.0 | 3.65 | 0.60 |
| 60 | 13.5 | 3.8 | 5.5 | 3.70 | 0.48 |
| 80 | 13.3 | 3.6 | 5.0 | 3.80 | 0.50 |
| 95 | 12.6 | 3.5 | 4.3 | 3.85 | 0.40 |
| 100 | 11.5 | 3.4 | 3.0 | 4.05 | 0.12 |
| 110 | 9.6 | 3.5 | 2.8 | 6.00 | 0.13 |
| 120 | 9.2 | 4.8 | 1.2 | 6.50 | 0.40 |
| 140 | 8.5 | 5.1 | 1.0 | 10.50 | 0.50 |
| 160 | 8.0 | 5.5 | 0.9 | 14.50 | 0.60 |

From the table, the bleached oil colour reduces as bleaching temperatures increases. At a temperature of above 110$^0$C, there is considerable heat effect. The corresponding deodorized temperature is optimized at a bleaching temperature in the range of between 95 and 110$^0$C, with optimum value at 100$^0$C. Table 5, shows the changes in oil colour that occur during storge. The result shows that colour reversion for deodorized oil takes place to give a darker oil when bleached at a temperature above 100$^0$C, and that the reversion occurs at a faster rate. This is shown by the sudden change in colour noticed on the 14$^{th}$ day.

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**Table 5. The effects of colour and PV on the keeping quality of physically refined palm oil, (Bleach temp. constant)**

| Time (days) | Colour (red), 1" cell | Peroxide value of deodorized oil | | |
|---|---|---|---|---|
| | Bleached at $95^0$C | Bleached at $150^0$C | Bleached at $95^0$ | Bleached at $150^0$C |
| 1 | 3.10 | 5.8 | 0.00 | 0.00 |
| 4 | 3.20 | 5.9 | 0.46 | 1.80 |
| 7 | 3.30 | 6.2 | 0.82 | 2.00 |
| 14 | 3.35 | 6.8 | 1.17 | 2.32 |
| 21 | 3.45 | 7.4 | 1.50 | 2.80 |
| 28 | 3.52 | 8.6 | 1.75 | 3.48 |

The peroxide value of the bleached oil is reduced at increasing bleaching temperature, but the rise in Anisidine value suggests that the oxidation products, Ketone and Aldehydes, are not being effectively adsorbed at higher temperatures as shown in table 4. This is reflected on storage as in Table 5. When the PV of deodorized oil rises more rapidly for oil bleached at $150^0$C compared to $95^0$C. The FFA of the bleached oil is gradually reduced up to a temperature of $110^0$C, beyond which, it rises sharply as in Table 4.

**Bleaching clay dosage:** For any given bleaching clay, bleach colour continues to reduce as dosage is increased as shown in Fig 1.

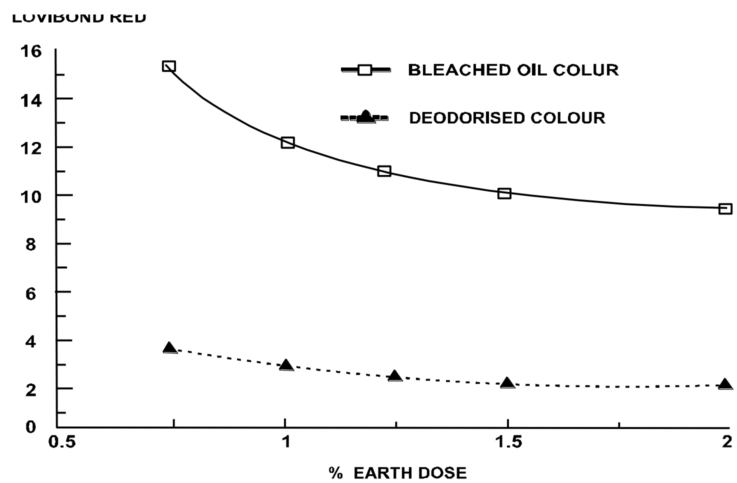


**Figure 1 The effect of earth dosage on the colour of physically refined palm oil.**

The deodorized colour, however, reaches a minimum between 1 and 1.5% clay dosage. There is no necessity to increase bleaching earth beyond 1.5% as this would produce no effect on the colour. The exact level of bleaching earth required will depend on the quality of the oil, and on the adsorptive power of the bleaching earth.

The removal of copper and phosphorous from the oil, is also a function of bleaching earth dosage as shown in figs 2 and 3 respectively. From fig 2, the higher the earth dosage, the increase in the reduction of copper.

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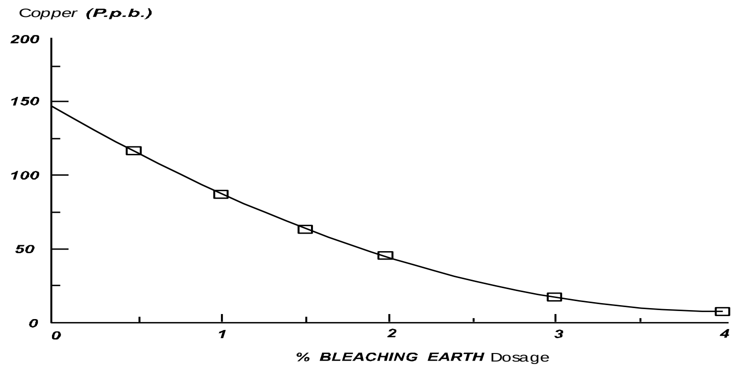


Figure 2 The effect of earth dosage on the copper content of physically refined Palm oil.

However, the reduction is sharp up to a 1% earth dosage for phosphorous, beyond which, it is marginally reduced as shown in fig 3.

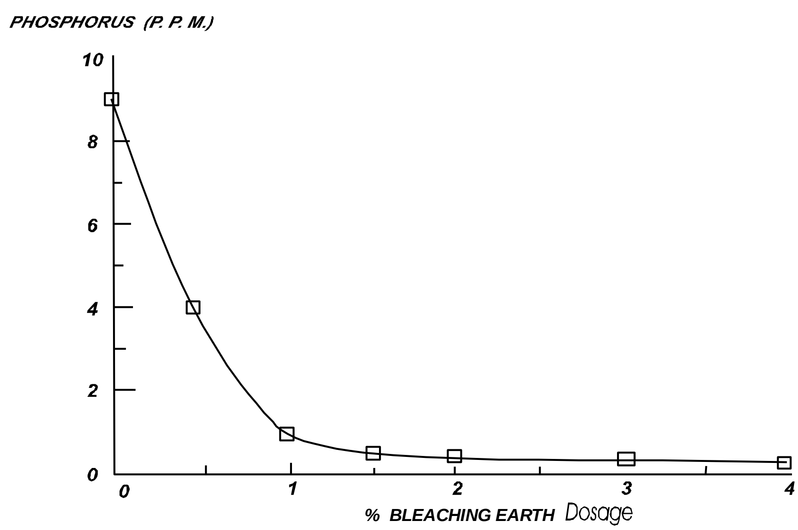


Figure 3 The effect of earth dosage on the phosphorus content of physically refined palm oil.

Peroxide value and AV are reduced by increasing earth dosage, in a similar way as increasing bleaching temperature, Fig 4. However, and as shown in table 4, while PV is significantly reduced at the bleach temperature, AV is sharply increased from a temperature of $110^0$C.

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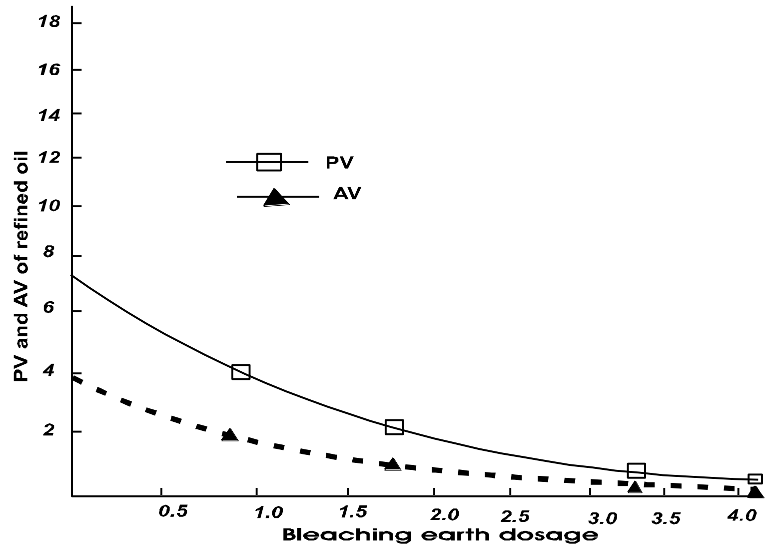


**Fig 4 Reduction of PV and AV at increase earth dosage.**

In view of this, there should be a compromise between increasing earth dosage, which reduces AV, and increasing temperature whose effect is to increase AV. The optimum value is in the range 1-1.5% earth dose, and $100^0$C.

**Bleaching Time:** A contact time of 20-30 minutes at the bleaching temperature is enough to reduce the PV to minimum. Beyond this time, the PV begins to rise, Fig. 5. The reduction in PV is considered more important than the continued fall in bleaching colour which is a heat effect and not a true adsorption.

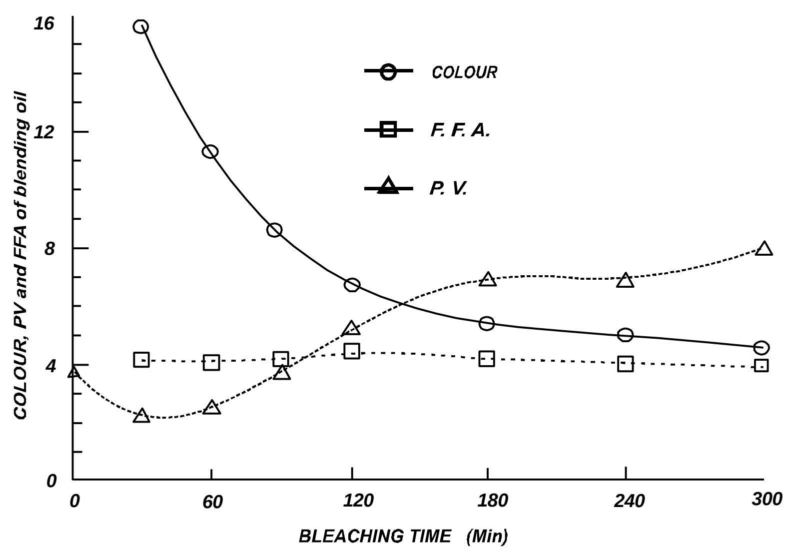


**Figure 5 The effect of extended bleaching times on colour, p.v. And f.f.a. Of bleached palm oil.**

As shown in the figure, FFA does not rise at high contact time when bleaching at $95^0$C. This is an advantage to the refiner. Optimum bleaching time is established to be in the range of 30-60 minutes.

## 5 CONCLUSION

The results obtained in section 3, have demonstrated that the stability and quality of finished palm oil product, can be greatly

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influenced by the bleaching stage. Variation of the bleaching conditions has shown how temperature, clay dosage and time conditions can affect the stability of the final product. The result showed that the bleaching temperature should not be more than 110°C, since the lower bleach colour obtained by increasing temperature does not improve the colour quality of the final product. Increase in clay dosage will improve the quality stability of the final product. However, the exact level required for optimum result, depends on the type of oil and the adsorptive power of the clay. Since an effective bleaching can improve the quality of the final product, an effective removal of the colour and FFA, and the reduction of PV and AV, are necessary in order to stabilized the quality of the physically refined palm oil.

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