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Effect of Washing Red Palm Oil and Low Grade Tallow Fat with Hot Water on Oil Bleachability and Bleached Oil Colour and Odour Stability

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ABSTRACT

Technical Palm Oil (TPO) and Special Palm Oil are the two grades of Palm Oil classified by the Nigerian market. SPO is a superior grade to TPO. The two grades differ from one another based on their free fatty acid (FFA), moisture, unsaponifiable matter and impurity contents. The following are the standard specifications: SPO (FFA 5% max, moisture 0.5% max, unsaponifiable matter 0.10%; impurity 0.10% max); and TPO (FFA 15% max, moisture 1.5% max, unsaponifiable matter 0.25%; impurity 0.25% max). SPO contains a higher Total Fatty Matter (TFM ~ 95.7%) content than TPO (TFM ~ 88%). SPO also contains a higher saponification value (198) than TPO (197). Both grades of oil share the same Lovibond scale ($5^1/_4$ inch cell) average colour characteristics (25R, 15Y, 10B, 1N). In this study, SPO and TPO were subjected to chemical bleaching using Fuller's earth and mineral acid under thermal conditions without pre-washing with hot water to improve their colour and odour for consumer acceptability as edible oils for deep frying. The average colour of the bleached oil was 1.5R, 10Y, 0B, 0N. The bleached oil colour and odour reversed spontaneously to 4.5R, 11Y, 0B, 0N within 4hr, and this reversion produced an objectionable odour and colour. The edible oil produced was unacceptable to consumers. Bar soap produced with it gave poor and inconsistent colours and odours accompanied by low consumer acceptability and loss of sales. Likewise when low grade tallow fat was bleached without washing with hot water, the bleached oil colour reversed from 1R 7Y 0B 0N within 4hr to 3R 8Y 0B 0N. The oil reversion was accompanied by animal fat odour which transferred into finished soap products. However, when these oils or fat were pre-treated by washing with hot water at 80ºC for 30 minutes and the impurity ran off, the bleaching cost was reduced by 50% and the bleached oil colours were lower (0.8R, 4Y, 0B, 0N) and stable for both palm oils and tallow fat. No extraneous odour was associated with the bleached oils. No colour or odour reversion was observed after 4 days, and edible oil and bar soap produced received high consumer acceptability.

INTRODUCTION

In Nigeria, palm oil and other vegetable oils are produced from their respective fruits and seeds by traditional extraction methods. In other parts of the world, crude fats and oils are produced by rendering, expression, or solvent extraction. These oils and fats contain variable amounts of nonglyceride impurities (Goh *et al* 1985; Abidi, 2003). The impurities in high-grade animal fats and certain vegetable oils, such as palm oil, palm kernel oil and coconut oil, consist principally of free fatty acids, proteinaceous and albuminous substances, as well as colouring matter, metal ions and insoluble matter (Goh *et al* 1985; Abidi, 2003).

Bailey (1948) reported the presence of fatty acids, glycerides, phosphatides, sterols, tocopherols, hydrocarbons, pigments (gossypol and chlorophyll), sterol glucosides, and protein fragments, as well as resinous and mucilaginous material in crude palm oil and other vegetable oils, while Rossi *et al* (2001) reported extensively on the tocopherol contents and type in palm oil.

Not all impurities in crude oils are undesirable. The sterols are colourless and heat stable and, for all practical purposes, inert; hence they pass unnoticed unless present in unusually large amounts (Goh *et al* 1985; Abidi, 2003). Tocopherols perform the important function of protecting the oil from oxidation. For this reason, they are classified as highly desirable constituents of most oil and fat products (Rossi *et al*, 2001; Andersen, 1962). Most of the other impurities are objectionable, and they render the oil dark-coloured, cause it to foam or smoke, and are precipitated when the oil is heated in subsequent processing operations (Andersen, 1962; De Greyt *et al*, 1999).

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Impurities in oils and fats render these oils unstable after treatment for use as edible oils and in the manufacture of soap, paints, cosmetic products, lubricants and greases (Sonntag *et al,* 1982). Certain impurities, such as phosphatides, proteins, or protein fragments, and gummy or mucilaginous substances, are soluble in the oil only in an anhydrous form and can be precipitated and removed if they are simply hydrated (Sonntag, 1982). Hydration is accompanied by steaming the oil or mixing it with water or a weak aqueous solution. Impurities separate from hydrated oil when allowed to stand for about an hour, and they can then be removed (Goh *et al* 1985; Abidi, 2003).

The standard method of bleaching is by adsorption or treatment of the oil with bleaching earth. The various chemical bleaching methods applied to inedible fats and oils all depend on oxidation of the pigments to colourless or lightly coloured materials (Jamieson, 1943; Patterson, 1993).

Red palm oil, produce excessive smoke when being used as edible oil for deep frying.Therefore, bleaching of palm oil, tallow fat and palm kernel oil is intended to remove those impurities that produce excessive smoke when cooking, and to improve the colour of the oil before use as edible oil, and in soap making, production of paints and for the manufacture of other cosmetic products. The term ''bleaching'' is reserved for the treatment designed solely to reduce the colour of the oil (Jamieson, 1943; Patterson, 1993).

Palm oil exhibits certain characteristic behaviour ascribed to its high content of β-carotene. When it is heated to a high temperature, the original deep colour disappears and solid ''break'' material is precipitated and much of the remaining colour pigment is oxidized. Oxidation has an important effect on the colour of fats and oils. While oxidation bleaches the carotenoid pigments, it develops the colour of other types of colouring material and in some cases apparently even produces coloured compounds of a quinoid nature (De Greyt *et al*, 1999). It has been shown that the partial oxidation of vegetable oils causes an increase in their red-yellow colour, most of which is apparently due to formation of the chroman-5, 6-quinones (Corredoira and Pandolfi, 1996) as described by Golumbic (1942).

In a study of the bleaching of cotton seed and soybean oils, King and Wharton (1949) have produced evidence that oxidation not only develops new pigments but also stabilizes existing pigments against adsorption, with the adsorbent itself strongly catalyzing the oxidation reactions. Traces of iron and some metallic contaminants greatly favour colour development in bleached oils and fats and thereafter causes reversion. Thus when any vegetable oil or fat is pre-treated by hydration, the impurities migrate to the aqueous portion of the mixture and this was removed (Rossi *et al*, 2001; Abidi, 2003). The washed oil bleached with fuller's earth remained stable for over 4 days before use for the manufacture of soap (Rossi *et al*, 2001; Abidi, 2003).

It is well known that carotenoid pigments are readily absorbed. The yellow-red colour of most vegetable oils is reduced without difficulty by treatment of the oil with bleaching earth. Oils from badly damaged seeds, or fruits or animal tissues or from rotten fruits, however, which contain brown pigments evidently derived from decomposed protein and carbohydrates, may be very resistant to bleaching by adsorption and may contribute to reversion (De Greyt *et al*. 1999).

Acid adsorbents have generally been shown to be more efficient bleaching agents than neutral materials, and the effect of acidity in fuller;s earth is particularly marked in bleaching to remove green colour pigments (Rossi *et al*, 2001; Abidi, 2003). It has been shown that in a study of soybean oil bleached with five different earths, that the bleaching efficiency is not determined by ''free'' hydrogen or hydroxyl ion concentration of the earth, as calculated from the pH of an aqueous suspension, but is a function of the ion adsorption capacity, as measured by the difference between the ''free'' ion concentration and the concentration of titrable ion (De Greyt *et al*. 1999).

American oil and fat bleachers and refiners usually determine the colours of oil (raw, bleached or refined) at 55^oC, by matching in a suitable tintometer a $5^1/_4$ inch column of the oil sample against red and yellow Lovibond colour glasses. The red glasses are standardized by the National Bureau of Standards in terms of the Priest-Gibson N Colour scale. For quality and standardisation purposes, the intensity and shade of colour of the oil or fat is satisfactorily expressed in terms of red units ((De Greyt *et al*. 1999).

MATERIALS AND METHODS

Materials:

Special Palm Oil (SPO) was purchased from Calaro Oil Palm Estate, in Cross River State of Nigeria. Technical Palm Oil (TPO), Low grade tallow (LGT), Fuller's earth and Tetra-oxo-sulphate 6 acid (H_2SO_4) were purchased from the open market in Aba, Abia State of Nigeria. Laboratory and plant scale equipment were used for this study. These included a vacuum pump, Quickfit 1L bleaching flask, laboratory hot plate, anti-bump glass beads, Whatman No 4 filter paper, magnetic bar, electrothermal mantle and a Lovibond tintometer.

Methods:

2kg of SPO was analysed for Moisture, free fatty acid, impurity, SV and total fatty matter (TFM) contents using the AOAC methods of ( ). A weight of 100g was taken from this stock and heated to 55^oC and the colour was taken using a Lovibond Tintometer at $5^1/_4$ inch cell and expressed as R Y B N (where R=Red, Y=Yellow, B=Blue and N=Neutral).

Standard bleaching procedures were employed. 500g of the oil was charged into a 1 litre quickfit flask. 4% Fuller's earth and 0.50% H_2SO_4 were added and mixed using a glass rod. Glass beads were added to serve as anti-bumping agents while a magnetic bar was added to effect mixing. The oil was bleached by heating the contents to 120 ^oC under vacuum (10 milibar) for 60 minutes. The bleached oil was neutralized with 0.15% $CaCO_3$. It was allowed to cool to 65^{o}C and filtered with Whatman No 4 Filter paper into a dry flat bottom flask, under vacuum. The colour of the filtered oil was measured with a Lovibond Tintometer using a $5^1/_4$ inch cell at 55^{o}C and expressed as R Y B N.

The above analysis and treatment was applied to both the Technical palm Oil (TPO) and the low grade tallow fat (LGT) samples. For LGT the bleaching procedures were carried out at a lower temperature of 100 ^{o}C using the same vacuum (10 milibar) and holding for 60 minutes. The bleached oil was neutralized with 0.05% $CaCO_3$. It was allowed to cool to 65^{o}C and filtered under vacuum with Whatman No 4 Filter paper into a dry flat bottom flask. The colour of the filtered oil was measured with a Lovibond Tintometer using a $5^1/_4$ inch cell at 55^{o}C and expressed as R Y B N.

In order to determine the effect of pre-treatment of the oils by washing with hot water before bleaching and to compare reversibility tendencies between untreated and pre-treated oils, 500g of SPO was charged into the 1 litre quickfit flask and mixed with 300g of water. A magnetic bar was introduced to effect mixing. The mixture was heated to 80^{o}C under vacuum (10 milbar) and held at a temperature range of 80-85^{o}C for 30 minutes. The heating was discontinued and the hot mixture was vigorously shaken for 5 minutes for effective washing and poured into a separating funnel to stand for 30 minutes. The washed oil phase was separated from the aqueous phase containing impurities. The phase containing impurities was discarded.

The washed oil phase was treated with 2% Fuller's earth and mixed. Glass beads and a magnetic bar were added to the flask. The oil was bleached at 90^{o}C under vacuum (10 milibar) for 30 minutes without addition of mineral acid. The bleached oil was neutralized with 0.05% $CaCO_3$. It was allowed to cool to 65^{o}C and filtered under vacuum with Whatman No 4 Filter paper into a dry flat bottom flask. The colour of the filtered oil was measured with a Lovibond Tintometer using a $5^1/_4$ inch cell at 55^{o}C.

The same treatment was applied to TPO and Low grade tallow oil. The bleaching conditions of the washed oils were the same with those applied to SPO washed with hot water.

The colours of bleached and unbleached oils were studied for 4 days. Colour measurements were taken with a Lovibond Tintometer using a $5^1/_4$ inch cell at 4 hourly intervals.

Statistical Analysis:

All data were expressed as Mean ± SEM. The mean values were taken from the results of three determinations. The data obtained were subjected to analysis of variance (ANOVA). The statistical significances were achieved when $p<0.05$.

RESULTS AND DISCUSSION

Table 1 shows the results of the quality characteristics of the raw, washed and bleached oils that were assessed for bleachability and bleached colours. After washing the raw oils, the moisture content increased slightly for (SPO 0.4 to 0.5%) but decreased significantly ($p<0.05$) for TPO (1.5 to 0.6%), and LGT (1.3 to 0.52%) respectively. After bleaching, the FFA values decreased significantly ($p<0.05$) in all the oils (SPO 1.52 to 1.20%; TPO 9.5 to 9.0% and LGT from 10 to 9.2%) respectively.

These treatments increased the respective %TFM values of the washed oils. Hot water washing and subsequent bleaching eliminated all objectionable impurities in the oils (Table 1). The washing of oil followed by bleaching, significantly ($p<0.05$) improved the overall total fatty matter (%TFM) content and the quality of the major organoleptic (colour and odour) parameters of the oil. Also the washing of the oils, followed by bleaching significantly ($p<0.05$) reduced the colour of the oils in a step-wise manner (SPO 17R to 12 R to 1.0R; TPO 18R to 10R to 1.3R and LGT 12R to 4R to 0.9R) respectively. The saponification values of the oils were not altered by these treatments (Table 1). The bleached oils did not congeal in storage, but remained free flowing and golden bright in colouration. These findings are consistent with the results obtained by De Greyt *et al* (1999).

Table 1: Quality Characteristics of Raw, Washed and Bleached Oils

Parameter Tested	Raw SPO	Washed SPO	Bleached SPO	Raw TPO	Washed TPO	Bleached TPO	Raw LGT	Washed LGT	Bleached LGT
% Moisture	0.4±0.2	0.5±0.1	0.1±0.01	1.5±0.1	1.6±0.1	0.2±0.01	1.2±0.1	1.3±0.2	0.1±0.1
% Free Fatty acid (FFA)	1.52±0.2	1.20±0.1	1.00±0.0	9.5±0.2	9.22±0.2	9.05±0.02	10±0.5	9.9±0.4	9.2±0.2
% Impurity	0.12±0.1	0.02±0.0	0.00±0.0	0.4±0.2	0.05±0.0	0.00±0.0	0.37±0.2	0.01±0.0	0.00±0.0
% Total Fatty Matter (TFM)	93.64±0.2	93.95±0.1	94.54±0.2	85±0.1	85.2±0.2	86.76±0.2	85±0.5	85±0.1	87±0.4
Sap. Value (SV)	198±1	198±2	198±1	197±2	197±1	197±1.0	196±1	196±2	196±2
Colour Redness ($5^1/_4$ inch cell).	17±2.0	12±1.5	1.00±0.0	18±0.5	10±0.3	1.30±0.05	12±0.2	4±0.4	0.9±0.2

Values are mean ± S.E.M of three determinations. Assumption: absolute TFM = 95.60%
SPO is Special Palm Oil; TPO is Technical palm oil; LGT is low Grade Tallow

Table 2 shows the bleached oil colour characteristics and reversion over a period of 4 days (240 min) under ambient conditions. The colour (particularly the redness) was monitored for reversion in both the washed-bleached and unwashed-bleached oils at 55–60°C. The monitoring was carried out initially at 30 minutes interval for 4 hours (table 2), and then daily for 4 days. Hot water washed and bleached oil colours remained relatively stable while the unwashed and bleached oil colour and odour reversed significantly ($p<0.05$) to 5R for SPO, 7R for TPO and 4R for LGT at the end of 4 days in storage at 55–60°C.

Generally, it was observed that the washed and bleached oils were more stable over a longer period before insignificant reversion sets in. On the other hand the unwashed and bleached oil was more unstable, and reversed spontaneously in storage to high Lovibond redness colours within 1 hour. The reversion was attributable to impurities in the oil, some of which are metal ions and free radical species which are agents of oxidative rancidity and damage to the triglyceride structure of the oil.

Table 2: Bleached Oil Colour Reversion with time (min) at Oven temperature of 50oC.

Bleached Oil	Initial Bleached colour (Redness)	After 30 min (R)	After 60 min (R)	After 90 min (R)	After 120 min (R)	After 150 min (R)	After 180 min (R)	After 210 min	After 240 min
Raw SPO	1.3±0.05	1.5±0.02	1.6 ±0.1	2.2 ±0.2	2.5 ±0.4	3.1±0.2	3.5 ±0.3	4.2 ±0.2	4.4 ±0.1
Washed SPO	1.0±0.20	1.1±0.03	1.2 ±0.2	1.3 ±0.0	1.3 ±0.0	1.3±0.1	1.3 ±0.2	1.4 ±0.1	1.4 ±0.2
Raw TPO	1.4±0.03	1.6±0.01	1.8 ±0.2	2.5 ±0.4	3.2 ±0.2	3.6±0.4	4.4 ±0.2	4.6 ±0.3	5±0.2
Washed TPO	1.3±0.05	1.3±0.01	1.3 ±0.0	1.4 ±0.5	1.4 ±0.3	1.4±0.2	1.4 ±0.1	1.5 ±0.1	1.5 ±0.2
Raw LGT	1.4±0.15	1.5±0.05	1.8 ±0.2	2.2 ±0.3	2.4 ±0.3	2.8±0.1	3.2 ±0.2	3.5 ±0.1	3.6 ±0.1
Washed LGT	0.9±0.02	1.0±0.10	1.1 ±0.1	1.2 ±0.1	1.3 ±0.4	1.3±0.2	1.3 ±0.1	1.4 ±0.2	1.4 ±0.1

Values are mean ± S.E.M of three determinations. R is redness of oil

In Nigeria, oils including Red palm oil (RPO), which is classified as either Special Palm Oil (SPO) or Technical palm Oil (TPO) depending on the quality, are bleached to remove impurities, and then used for edible purposes or for the production of soap and other products. The term “bleaching” is reserved for treatment designed solely to reduce the colour of oil. It involves the use of bleaching earth and mineral acids at high temperature under vacuum. However, not all vegetable oils are easily bleached by this conventional means. And even when they are successfully bleached, most edible oils are unstable with respect to objectionable colour and odour. Bleached oils are known to reverse after bleaching to poor colours and odour.

This study proved that hot water washing, removes all impurities including reactive metal species, free radical species, short chain fatty acids and objectionable colours and odours, from oil and the eventual bleaching of the washed oil ensures colour and odour stability over a long period of time.

Palm oil contains significant quantity of impurities including heavy metals, short chain fatty acids, chlorophyll, tocopherols, carotenoids, sterols, albuminous and proteinaceous materials, among others. Bailey (1948), had earlier reported the presence of similar impurities in cotton seed oil, including fatty acids, glycerides, phosphatides, sterols and tocopherols, hydrocarbons, pigments (gossypols and chlorophyll), sterol and protein fragments, as well as resinous and mucilaginous substances.

Rossi *et al*, (2001), reported the presence of tocopherol and its isomers in palm oil. Some of these impurities are objectionable since they render the oil dark-coloured, emit bad odour, cause it to foam or smoke, or are precipitated when the oil is heated during processing. More recently, some of these impurities have been discovered to cause the bleached oil colour and odour to reverse in storage and produce objectionable colours and odour. The main objective of bleaching is to remove such objectionable impurities in the oil with the least possible damage to either the glycerides or the tocopherols or other desirable impurities, and with the least possible loss of oil (Rossi *et al*, 2001).

Palm oil is the main cooking oil in Nigeria and other West African countries. Apart from its use as edible oil, palm oil is bleached or refined for industrial purposes. The bleached oil is used for the production of laundry and toilet soap bars. During off-season for palm tallow fat is also imported into Nigeria and other West African countries and used for soap production. Some factories in Nigeria produce composite edible oil consisting of blends such as palm oil, palm kernel oil and tallow fat, or palm oil and palm kernel oil, or tallow and palm kernel oil. These oils in their crude form contain appreciable amounts of impurities which elicit objectionable odour and colour.

Vegetable and animal fats are also known to contain heavy metals such as As, Pb, Fe and Hg.

In Nigeria, palm oil is available in commercial quantities during the dry season (September – April) which is the ripening period of the palm fruit. Fresh palm oil is available during this period and at lower price. Manufacturers of edible oil and soap, buy and store the commodity during this period. In storage, the oil degrades due to oxidative rancidity to generate high FFA content with attendant dark colour and objectionable odour. This degradability by oxidative rancidity may be caused by the presence of heavy metal ions and free radical species. Under this condition, the oil is graded as technical palm oil (TPO) on account of its high FFA (>5%), high moisture (>0.5%), high impurity level (>0.01%), high unsaponifiable matter (>0.2%) and low total fatty matter (%TFM) content (<90%), (Table 1).

Such poor quality oil need to be bleached to remove all the agents of reversion before being considered suitable for consumption and use for cooking as well as for soap making. A significant ($p<0.05$) reduction of colour (R1.3, Y 12 B 2 N 0) of oil was obtained on bleaching the oil directly, using 4% bleaching earth and 0.15% mineral acid (H_2SO_4), at 120°C bleaching temperature and under vacuum. The bleached colours obtained were (R 1.3, Y 12, B 2, N 0) on the Lovibond scale at 55°C, using $5^1/_4$ inch cell (Table 1) were unstable. The FFA and bleached oil moisture content were reduced to <1.5% and <0.2% respectively.

When this bleached oil was bottled for sale or stored for later use, the colours reversed significantly ($p<0.05$) to higher values (R >4, Y>15, B>8, N 0), Figure 2), and the FFA increased to >3%. The original odour and colour of the raw oil was restored in the bleached oil after 4 hours in storage under ambient conditions.

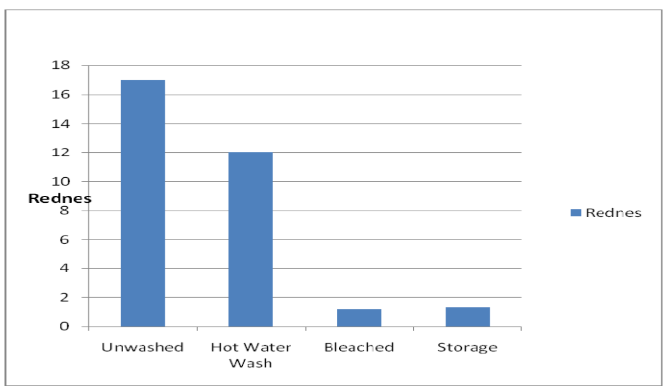

Fig. 1: Effect of Hot Water Washing and bleaching on the Redness of Red Palm Oil (RPO)

However, when the oil was vigorously washed at 80°C under vacuum for 30 minutes, and allowed to stand and cool for 1 h, and the bottom impurity ran-off, the colour of oil improved significantly ($p<0.05$). The washed oil, bleached at lower cost using 2% Fuller's earth, without acid, at 85-90°C under vacuum for 45 minutes and filtered at 80-85°C, further resulted to a significant ($p<0.05$) decrease in the oil colour (Figure 1). Corredoira and Pandolfi (1996) had reported that impurities such as phosphatides, proteins, or protein fragments and gummy or mucilaginous substances which are soluble in most oils only in anhydrous form, could be removed if they are simply hydrated. The team further reported that hydration was accomplished by steaming the oil or mixing it with water or weak aqueous solution.

The bleached oil colour and odour did not reverse while in storage or when bottled for sale. The bleached colours obtained were (R 1.0, Y 10, B 0, N 0) and the average colours after 4hr at ambient conditions were (R 1.3, Y 13, B 0, N 0), (Table 2 and Figure 2). After 7 days, the bleached oil colour and odour did not change to objectionable form. The colour and odour remained characteristic and acceptable to consumers. Washing with steaming reduced significantly the impurities, FFA, odoriferous and colour substances from the oil (Loury, 1943).

When these bleached oils were used for the production of laundry and toilet soap bars, the same trend was observed. The raw oil bleached directly without washing, produced dull coloured soap bars which gave objectionable odour and colour, when compared to standard toilet and laundry soap bars. On the other hand the soap produced with bleached oil previously washed with hot water (80-85^oC), matched the standard bars. The colours were bright, and did not produce objectionable odour and colour. These parameters were generally acceptable to consumers who were selected at random for their individual opinion. These observations and trends were similar to the results obtained with Special palm oil (SPO) and low grade tallow (LGT) treated and bleached with the same procedure.McGuire *et al* (1947), found in laboratory experiments that a crude soybean oil containing beany odour, impurities and 0.019% nitrogen was reduced to 0.00044% nitrogen by one water washing and to 0.00024% and 0.00019% by a second and a third water washing respectively, with the elimination of objectionable colour and odour, as were the results in this study.

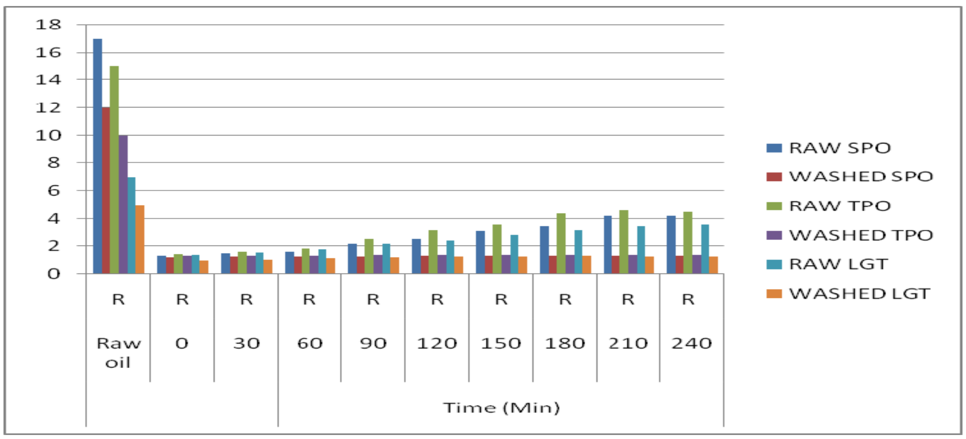

Fig. 2: Bleached oil Colour (redness) reversion progression with time (min)

Conclusion:

It was concluded that hot water washing and bleaching of palm oil, tallow fat and palm kernel oil improved the colour and odour as well as the stability of the bleached. There was increased consumer acceptability of the bleached oil for edible purposes as well as soap produced with it. These results were said to be due to the procedure involving initial vigorous washing with hot water which hydrated and removed all soluble oxidative substances followed by bleaching at low temperature without use of mineral acid. It was further concluded that the reversion of bleached oil was attributable to impurities in the oil, such as metal ions and free radical species which are known agents of oxidative rancidity with the potential to damage the triglyceride structure of the oil molecule.

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