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Adulteration of Crude Palm Oil with Red Dye from the Leaf Sheath of Sorghum Bicolor

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Abstract
The present work was undertaken to investigate the possibility of adulterating CPO with natural potash (lake salt) and red dye from the leaf sheath of *sorghum bicolour*. Concentrations of potash and red dye ranging from 0.01-0.1% and 0.1-1.0% respectively were prepared in water; and then added to fresh CPO at oil/adulterant ratios ranging from 10:1-10:10, to obtain adulterated CPO with known concentrations and adulterant ratios. Adulterated samples were examined visually for appearance, and evaluated for quality indices and sensory attributes. Values of quality indices were similar for both the adulterated and unadulterated samples, while the acceptability of taste and mouthfeel were clearly higher for the unadulterated sample. The use of potash changed the characteristic orange red color of CPO to yellowish red even at levels below 0.01%; and also resulted in product with increased consistency. CPO can be successfully adulterated with 0.25% red dye from the leaf sheath of *sorghum bicolor* at a maximum oil/dye ratio of 1:1. The adulterated oil can remain unidentified by consumers for at least 30 days. A simple, rapid and effective test by means of which a consumer can easily detect the presence of red dye in CPO was also developed.
Keywords: Crude palm oil; Adulteration; Red dye; Natural potash; Sorghum bicolor.

1. Introduction

The oil Palm (*Elaeis guinensis*) is West Africa's most important oil producing plant. The fruit produces two distinct types of oil: orange-red crude palm oil which is extracted from the mesocarp and brownish yellow crude palm kernel oil extracted from the seeds (kernel). The former consists of mainly palmitic and oleic acids and the latter of mainly lauric acid. Both oils are important in the world trade. Crude palm oil (CPO) is the richest natural source of carotenoids and tocotrienols. The carotenoids (500–700 ppm) are responsible for the characteristic deep orange-red color (Gunstone, 2005), while its semi-solid consistency at tropical room temperature is mainly due to the presence of triacylglycerols of palmitic and oleic acids (Gee, 2007).

In recent years there has been rising production (supply) and consumption (demand) of palm oil in Nigeria, with demand growing faster than the supply. As a result, the trend has been that of increasing domestic consumption unequally matched by a rather slow growth in production. This widening gap between demand and production has also been accompanied by increasing reports (from the media) of adulteration. Adulteration of fats and oils is an old problem and has been the subject of many studies (Imai et al., 1974; Rossell et al., 1985; Aparicio and Aparicio-Ruiz, 2000). Often cheaper oils have been sold in place of, or mixed with, more expensive oils.

There is a wide spread speculation in Nigeria that crude palm oil is being adulterated. The adulteration is believed to be practiced by producers in order to increase the quantity of CPO, for the sole purpose of profit maximization. Unfortunately, the adulteration practice is normally done without considering its possible effect on the quality of palm oil and the health of consumers. The adulterants reportedly used include carrot, papaya, natural potash and red dye; with potash and red dye being the preferred and most widely used adulterants due to their abundance and low cost (Authors' communication with local retailers). Natural potash, also called lake salt and locally known as "kanwa" is a mineral consisting of chlorides, sulphates and carbonates of Na, Ca, and K, as well as some micronutrients (Aribido, 2001). Kanwa is also a food condiment used locally as tenderizer for tough cuts of meat; as thickener for increasing the viscosity of soup; and as emulsifying agent between oil and water. Red dye on the other hand is the aqueous extract (evaporated to dryness) of the leaf sheath of *sorghum bicolor* (Ogwumike, 2002; Akande et al., 2010). The plant is known as "Karan dafi" in Northern Nigeria where it thrives, and the dye extracted is commonly used in coloring leather red, coloring of clothes, calabashes and as a body pigment.

One major problem associated with the use of adulterants is that these compounds have not undergone stringent studies and the level of threat they may pose to human health when consumed, not well established. For crude palm oil, adulteration could lead to loss of quality and nutritive properties, loss of organoleptic attributes and overall degradation of the oil. No literature was found on adulteration of CPO with non-oil product such as potash and red dye of natural origin. The objective of this work was to investigate the possibility of adulterating CPO with potash and red dye from leaf sheath of *sorghum bicolor*. Effort was also made to develop a simple, rapid and effective test for identifying adulterated crude palm oil.

2. Materials and methods

2.1 Sample preparation

To assure the absence of adulterant of any type, the crude palm oil used in this study was processed in the Lab from freshly harvested ripe palm fruits of Tenera variety. The fruits were supplied by a local palm oil processor, while the potash and red dye used as adulterants were purchased from a local grocery store. CPO was extracted in the Lab using traditional technique (Okogeri and Otika, 2011): Fresh palm fruits were parboiled in a cooking pot (to prevent enzymatic spoilage and to soften fruits mesocarp for easy pounding) and then pounded using wooden pestle and mortar until pulp and nuts were obtained. The nuts (palm kernels) were removed and the pulp manually squeezed to obtain a red viscous fluid (oil, fiber, water, impurities), which was heated for traces of water to evaporate, and finally sieved using metal basket to obtain a clear red palm oil.

2.2 Sample adulteration

Aqueous potash and dye solutions ranging from 0.01-0.1% and 0.1-1.0% respectively were prepared by dissolving 1g of potash or red dye in 100ml distilled water, and then diluting with distilled water to obtain the required concentrations. Exactly 10g of the freshly processed CPO (section 2.1) was weighed into 145 transparent plastic vials of 25ml each and warmed in a water bath set at 45^{o}C to decrease oil's viscosity. Then to 50 of the vials, potash solutions were added while to the remaining 95, dye solutions were added; following the ratios shown in Table 1. (Adulterant concentration of 0.10% and a corresponding Oil/adulterant ratio of 10:1 for instance, indicate that 1ml of 0.1% adulterant solution was added to 10g of fresh red palm oil). The samples were agitated, capped and then stored on the shelves of the laboratory under diffused light and inspected visually (for color and appearance) every 24 hours for 30 days. Fresh red palm oil with no added adulterant served as the control.

During the entire storage period, samples whose appearance differed slightly or completely from that of the control were eliminated from the study, retaining only samples whose color and consistency were comparable to those of the control.

2.3 Analysis of samples

Free fatty acid, peroxide value and Iodine value, were determined according to AOCS Official Methods Ca 5a-40, Cd 8b-90, and Cd 1d-92 respectively (AOCS, 2004).

The fatty acid profiles of the control and adulterated samples were evaluated by GC analysis of methylated samples according to AOCS Official Method Ce 1h-05 [10]. FAMEs were analyzed on a HP 6890 GC system from Hewlett Packard, using a DB-23 capillary column (60m x 0.32mm x 0.25µm film thickness) from Agilent Technologies.

Color analysis of samples was performed using ColorTec-PCM (ColorTec, Clinton, NJ) colorimeter, operating on the CIE (Commission Internationale de l'Eclairage) L*, a*, b* color scheme. The measurement was displayed in CIE L* a* b* (Belbin, 1993). L* value represents lightness-darkness dimension, a* value represents red-green dimension, and b* value represents yellow-blue dimension. Only a* value was used as the CI in this study. The lower the value, the more the color of the oil deviates from redness.

2.4 Sensory Evaluation

Sensory analysis was used to assess the organoleptic quality of adulterated samples. Fifteen trained panelists were selected from final year students of the Department of Food Science and Technology, Ebonyi State University, Abakaliki. The panelists were screened using the triangle test (Nobel, 2006) and selected based on their ability to consistently discriminate between fresh CPO and CPO with added red dye. Adulterated CPO samples were evaluated by rating on a 9-point hedonic scale, where 1 = dislike extremely, 5 = neither like nor dislike, and 9 = like extremely, according to acceptability of color, taste, mouthfeel, and overall acceptability. Sample preparation and evaluation of sensory attributes of samples were carried out based on the recommendations described by Malcolmson (2005).

2.5 Identification of adulteration

Identification of adulterated CPO was studied by subjecting adulterated samples to heating using a hot plate with temperature regulator. Precisely, 60g of CPO samples adulterated with 0.25% dye solution at oil/adulterant ratios of 10:1, 10:2, 10:4, 10:8, and 10:10 were introduced into a 100ml beaker and heated at 100, 160, 170, and 180^{o}C until the color of sample changed from the characteristic orange-red color of CPO to colorless.

2.6 Statistical analysis

Analytical data for unadulterated and adulterated CPO samples were processed using Microsoft Excel 2007.

3. Results and discussion

3.1 Visual evaluation of adulterated samples

Adulterated samples prepared using dye solutions of concentrations exceeding 1% showed bright redness which was by far brighter than the characteristic orange-red color of the control. Likewise dye concentrations lower than 0.1% had light-red color which was by far lighter than that of the control. Consequently, minimum and maximum concentrations of dye solutions were established as 0.10 and 1.0% respectively. The use of potash solutions at levels from 0.01% and above increased the volume of CPO by up to twofold, but also changed the characteristic orange red color of CPO to yellowish red and also resulted in a product with a more viscous consistency. On the other hand, the use of potash solutions below 0.01% resulted in two immiscible liquid phases. Consequently, potash was considered to be far from an ideal adulterant and eliminated from further study. Interest was therefore focused on red dye which appeared to be a representative adulterant for CPO.

To determine the maximum oil/adulterant ratio, varying amounts of dye solutions were added to CPO (Table 1). It was observed that to get an adulterated sample with no noticeable color or consistency difference from the control, the volume of adulterant must not exceed the weight of the unadulterated CPO. Oil/adulterant ratio of 1:1 was therefore adopted as the maximum adulteration ratio. Thus, dye solutions ranging from 0.10-1.0% were prepared and added to fresh CPO at ratios ranging from 10:1 to 10:10 (oil/dye solution). However, upon the addition of dye solutions to fresh CPO, it was observed that the 0.10, 0.15 and 0.20% solutions resulted in adulterated CPO with two immiscible phases. These samples (total of 15) were therefore pulled out from further studies. It was also observed that while samples prepared from 0.80, 0.85, 0.90, 0.95 and 1.00% dye solutions had orange-red color comparable to that of the control, the samples also had more viscous consistency than the control irrespective of oil/adulterant ratio. These samples (total of 25) were also pulled out from further studies. The remaining 55 adulterated samples prepared using 0.25-0.75% dye solutions had no noticeable color or consistency difference from the control and were subjected to further evaluation.

3.2 Changes during storage

The samples adulterated with 0.25-0.75% solutions at ratios ranging from 10:1-10:10 were stored on the shelves of the lab under diffused light and examined visually for possible changes in color and overall appearance, every 24 hours for 30 days. Color and appearance are usually the first and most obvious characteristics evaluated by any consumer of CPO and changes in these characteristics are usually perceived as indicating poor quality product, irrespective of the reason or effect on overall performance. Visual examination therefore measures exactly what the consumer perceives. The storage condition (transparent plastic vial and diffused light) was chosen to mimic a typical storage conditions in retail stores; while the storage time (30 days) was estimated by the authors to well represent the interval between adulteration, retailing, and consumption.

Based on the observed pattern of changes in color and appearance over time, the adulterated samples were grouped into: A25 (samples adulterated with 0.25% dye solution at 10:1 to 10:10 ratios); B45 (samples adulterated with 0.30-0.45% dye solutions at 10:1 to 10:10 ratios); and C75 (samples adulterated with 0.50-0.75% dye solutions at 10:1 to 10:10 oil/adulterant ratios). The changes in color and appearance of these samples during 30-day storage are summarized as follows:

Day 0-7: There was no noticeable difference between sample A25 and the control; the redness of sample B45 became brighter than that of the control, and from day 3 there were red particles dispersed within sample B45; while for sample C75 there was increase in redness and consistency from day 2.

Day 8-14: For A25 and on day 9, there was slight increase in consistency of the 10:10 ratio sample but barely noticeable. The rest of the samples that make up A25 remained the same as in day 0-7; For B45 and on day 8, samples adulterated at 10:5 to 10:10 ratios became more viscous than the control; while for C75 there was further increase in consistency on day 8 irrespective of level of adulteration, and from day 9, the 10:8, 10:9 and 10:10 ratio samples separated into two distinct fractions.

Day 15-30: No further changes were observed in A25, samples remained the same as in day 8-14; Sample A45 also remained the same as in day 8-14; while from day 15, the fractions observed in C75 became irreversible even upon agitation.

As a consequence of the above visual examination, 0.25% was chosen as the ideal concentration of red dye and 10:1 to 10:10, as the possible and effective oil/adulterant ratios. Under these conditions, CPO can be successfully adulterated and remain undetected visually by consumers for at least 30 days. The ability of red dye to bind oil and water can be attributed to saponins which have been reported to be among the phytochemicals present in the leaf sheath of *sorghum bicolor* [8]. Saponins, a large family of structurally-related compounds of steroid or triterpenoid aglycone (sapogenin) linked to one or more oligosaccharide moieties by glycosidic linkage, possess strong surface active properties conveyed to them by the presence of both polar (sugar) and nonpolar (steroid or triterpene) groups [16]. It is plausible that the emulsifying activity of red dye was most effective at the concentration of 0.25%.

3.3 Quality indices

Table 2 shows some quality indices of the fresh CPO alongside those of a representative adulterated sample (A25 at the maximum adulteration ratio of 1:1). FFA which is one of the most important quality parameters in palm oil industry as it indicates the level of deterioration of the oil, recorded values less than 3.5% which has been reported as the average acidity for commercial crude palm oil (Gibon, 2007). This indicates that the CPO produced and used in the present study was obtained from freshly harvested ripe fruits with restricted activity of the endogenous lipase (triacylglycerol acylhydrolase) in oil palm fruits (Henderson and Osborne, 1991), and further suggests that adulteration with red dye reduced the value of FFA (due to dilution effect of the dye solution). Peroxide values followed similar trend. Oil color index was similar for both the adulterated and unadulterated samples, while the iodine values and fatty acid composition were within the reported ranges for CPO (Gee, 2007), for both the adulterated and unadulterated samples. The above findings suggest that measurement of common quality parameters of oil alone, may not be sufficient for identifying CPO adulterated with red dye.

3.4 Sensory attributes

Sample A25 adulterated at the maximum ratio of 1:1 (or 10:10) was further subjected to sensory evaluation and the results are shown in Figure 1. The mean scores for acceptability of color were similar for both adulterated and unadulterated samples (although A25 showed a higher standard deviation as indicated by the error bar in Fig. 1) and are in agreement with the results from color index Table 2) and visual examination (section 3.3). The scores of pleasant odor tilted slightly in favor of A25 but nevertheless were similar for both samples, suggesting that addition of 0.25% dye solution at 1:1 ratio did not mask the characteristic "nutty" odor of crude palm oil. The mean scores for acceptability of taste and mouthfeel; and overall acceptability were clearly higher for the unadulterated CPO, indicating that addition of adulterant reduced the pleasantness of taste and mouthfeel of CPO. The adulterated sample (A25) was also described by the panelists as having a moderate salty taste and an unpleasant aftertaste.

While evaluation of taste and mouthfeel can be helpful in identifying CPO adulterated with red dye, the use of sensory perception by unsuspecting consumers to identify adulteration can be hampered by the eating habit of many CPO consumers. In Nigeria and many West African countries for instance, CPO is consumed either raw (commonly as a sauce for boiled yam) after the addition of salt and spices; or is used as cooking oil during the preparation of variety of dishes. Such habits can mask possible unacceptable taste, mouthfeel and other negative sensory characteristics resulting from adulteration, thereby making the identification of adulterated CPO more challenging from consumer perspective. Considering this additional challenge, efforts were made to develop a test which can be used by consumers to effectively identify crude palm oil adulterated with red dye. Techniques which have been used in the literature to study oil adulteration involve the use of specialized analytical instruments (Imai et al., 1974; Rossell et al., 1985; Aparicio and Aparicio-Ruiz, 2000) whose results can be interpreted only by trained scientists. In this study, primary focus was the consumer.

3.5 Detection test

Considering that the characteristic deep orange-red color of CPO is due to the presence of carotenoids and considering that these compounds are heat sensitive and can be degraded to colorless products at temperatures exceeding 150^{o}C (Gunstone, 2005), CPO and A25 were subjected to heating (section 2.5). At 100^{o}C, both samples gradually became colorless within 3 hours, with CPO being the first to lose color and A25 having red particles dispersed within the mass of now colorless A25. The loss of color in A25 was also accompanied by a substantial decrease in volume which can be attributed to evaporation of water used in the preparation of dye solution (section 2.2). At temperatures of 160, 170, and 180^{o}C, loss of color (from orange-red to colorless) occurred within an average of 4 minutes for both samples, again with the CPO being the first to become colorless, and A25 recording a substantial decrease in volume in addition to having deposits of black particles (burnt red dye) at the bottom of the heating beaker.

Heating a sample of CPO at temperatures exceeding 100^{o}C can therefore be considered a simple, rapid and effective test for identifying CPO adulterated with red dye. Even in the absence of a control sample, the presence of red or burnt particles coupled with substantial reduction of volume can be a strong indication of adulteration.

4. Conclusions

Results indicate that measurement of common quality parameters of fats and oils may not be helpful in identifying CPO adulterated with red dye. Evaluation of sensory attributes, particularly taste and mouthfeel, can be considered a reliable approach while subjecting oil sample to heating at temperatures exceeding 100^{o}C appears to be the most simple, rapid and effective method for detecting the presence of red dye in CPO. Even in the absence of a control sample, the dispersion of red particles within the colorless oil or the substantial decrease in volume can be a strong indication of adulteration. Potash was also evaluated as potential adulterant. While this compound showed good oil/water binding power and increased the volume of CPO by up to twofold, it also changed the characteristic orange red color of CPO to yellowish red even at levels below 0.01%; and also resulted in product with increased consistency. Consequently, potash cannot be considered an ideal adulterant for CPO. The present work has demonstrated that CPO can be successfully adulterated with red dye and that the adulterated oil can remain undetected by consumers for a period of 30 days at least. The work has also developed a simple, rapid and effective test by means of which a consumer can easily detect the presence of red dye in CPO.

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Table 1: Adulterant concentrations and ratios

Concentration of Adulterant (%)		
Red dye	Potash	Oil/Adulterant ratio
0.10; 0.15; 0.20; 0.25; 0.30;	0.01; 0.02; 0.03;	10:1; 10:2; 10:4; 10:8; 10:10
0.35; 0.40; 0.45; 0.50; 0.55;	0.04; 0.05; 0.06;	10:1; 10:2; 10:4; 10:8; 10:10
0.60; 0.65; 0.70; 0.75; 0.80;	0.07; 0.08; 0.09;	10:1; 10:2; 10:4; 10:8; 10:10
0.85; 0.90; 0.95; 1.00	0.10.	10:1; 10:2; 10:4; 10:8; 10:10

Table 2: Quality indices of unadulterated and adulterated crude palm oil.

Parameters[a]	Fresh CPO	Adulterated CPO[b]
Free fatty acid [%]	2.28	1.32
Iodine value	51.88	52.56
Peroxide value [meq/kg]	3.53	2.07
Color a* (red)	6.48	6.28
Fatty acid composition [%]		
Lauric (12:0)	0.28	0.30
Myristic (14:0)	1.11	1.18
Palmitic (16:0)	42.48	42.35
Stearic (18:0)	4.18	4.24
Oleic (18:1)	39.18	39.26
Linoleic (18:2)	10.26	10.18
Linolenic (18:3)	0.40	0.43
Arachidic (20:0)	0.35	0.34

[a]Values are mean of duplicate analysis
[b] Sample A25 adulterated at the maximum adulteration ratio of 1:1.

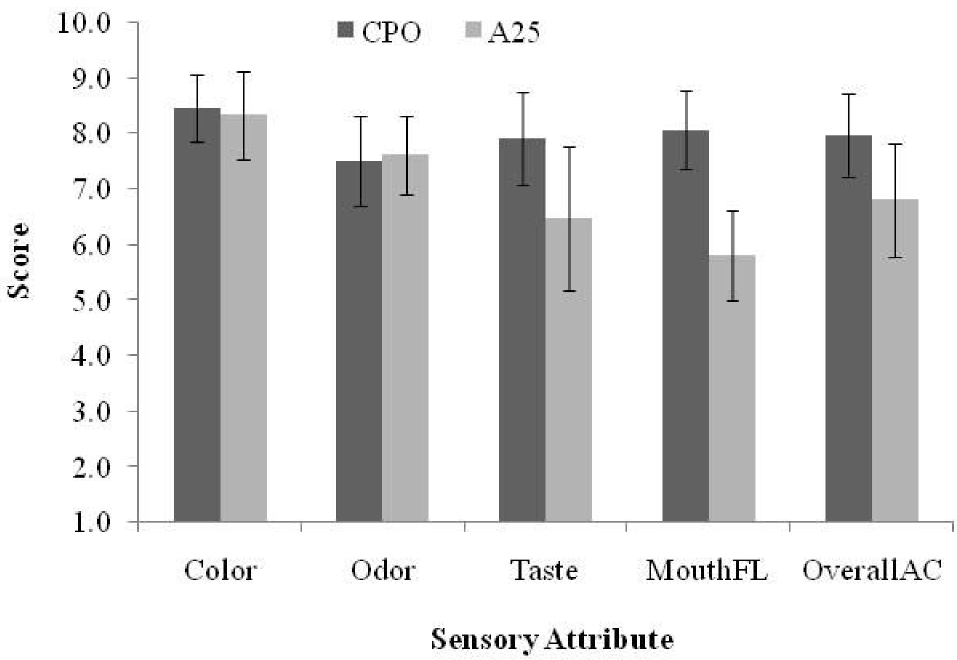

Figure 1: Mean scores (n=15) of sensory evaluation for the unadulterated (CPO) and adulterated (A25) crude palm oil; MouthFL = mouthfeel, OverallAC = overall acceptability.