



Effect of Bleaching on the Physico-chemical Properties of Two Selected Vegetable Oils Using Locally Sourced Materials as Adsorbent

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Authors' contributions

This work was carried out in collaboration among all authors. Author JOA designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors AEO and OOA managed the analyses of the study. Author JOA managed the literature searches. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/CJAST/2019/v36i430244

Editor(s):

(1) Dr. Aleksey Aleksandrovich Hlopitskiy, Professor, Department of Technology Inorganic Substances, Ukrainian State University of Chemical Technology, Ukraine.

Reviewers:

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Complete Peer review History: <http://www.sdiarticle3.com/review-history/44390>

Original Research Article

Received 02 September 2018

Accepted 05 November 2018

Published 19 July 2019

ABSTRACT

The quality and stability of palm kernel oil (PKO) and cottonseed oil (CTSO) as affected by bleaching using modified oyster shell powder (OSP) and periwinkle shell powder (PSP) were evaluated by analysing their physicochemical properties. The two adsorbents potential was improved on by modification using 5 M HCl and H₂SO₄. The determinations of the parameters were carried out by titration, while the refractive index was determined using a refractometer. The results indicate that the saponification values (SV) of PKO and CTSO increase after bleaching. However, the result further shows that the peroxide values (PV) of bleached PKO using OSP and PSP modified with 5 M HCl decreased (17.49±0.01 and 20.04±0.08 respectively) and also the iodine values decreased (3.15±0.13 and 3.41±0.02 respectively).

The same decrease in PV and IV values is observed in PKO bleached using 5 M H₂SO₄. The values of PV for OSP and PSP are 4.99±0.005 and 12.77±0.77 respectively while the IV values for OSP and PSP are 1.85±0.01 and 12.53±0.09 respectively. Also, 5 M HCl and H₂SO₄ modified OSP

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and PSP reduced the IV content in CTSO. (5 M HCl OSP and PSP are; 3.15 ± 0.13 and 3.41 ± 0.02 respectively while 5 M H_2SO_4 OSP and PSP values are; 3.33 ± 0.04 and 3.13 ± 0.06 respectively). The study revealed that the bleaching through the use of OSP and PSP influenced the components and properties of the PKO and CTSO, improved on their qualities and therefore, the adsorbents can serve as alternatives to the costly adsorbents available in the markets.

Keywords: Bleaching; degumming; oyster and periwinkle shells powder.

1. INTRODUCTION

Vegetable oils and fats are lipid substances that are extracted from plants. Fats and oils are composed of free fatty acids, mono- and diglycerides, unsaponifiable lipids, colour pigments which include carotenoids, chlorophyll, gossypol and related compounds [1,2]. Crude vegetable oils contain other objectionable substances which affect the quality of the oils. The objectionable substance which affect the quality of the oils. The objectionable substances or impurities in vegetable oils may be biogenic i.e. synthesis by plant themselves but can also be impurities are taken up by the plants from their environment [3].

Crude vegetable oils that are edible contain some extraneous materials such as proteinaceous matter which reduces smoke point while others cause objectionable colour development [4].

Vegetable oils are an essential part of the human diets and their production through extraction techniques have increased immensely as a result of higher demand and consumption due to increase in the world population [5]. Bleaching requires the use of adsorbents singly or in combination for the removal of coloured pigments and impurities or contaminants from oil to give it a desirable quality [6].

Refining is carried out to improve shelf life, quality, and nutritive values of the resultant oils [6,7]. Vegetable oils most especially are widely utilised by food industries as a result of their nutritional properties [8]. Physical and chemical refining are the most common methods available for refining of vegetable oils. Both processes are able to produce refined, bleached, and deodorized vegetable oils of desirable quality and stability suitable for edible purposes [9]. During refining, various kind of minor constituents which may include dirt, moisture, gums (phosphatides), waxes, colour pigments, pesticides residues, odoriferous materials, trace metals (Cu, Fe), free fatty acids. In order to

become suitable for a human users and consumption, crude vegetable oils are refined to get rid of the unwanted components to produce a stable finished product within desired colour and a pleasant taste with the least possible damages on the desirable components and with the least possible loss of oil [10]. Generally, refined oil is clear, odourless, and resistant to rancidity.

Cottonseed is among the most unsaturated edible oils. Crude cottonseed oils unsuitable for use in most food application without refining because of its dark colour, high free fatty acid content and objectionable flavour and colour [11]. An important nutritional benefit derived from cotton seed oil is its high level of antioxidants such as tocopherols and numerous minor components which include gossypol, phospholipid, and hydrocarbons [12]. Cotton seed oil is free of cholesterol as in other oils extracted from plants. Linoleic acid is the major polyunsaturated fatty acid present in cottonseed oil with three times as much unsaturated as saturated fatty acid. Cottonseed oil is a healthy vegetable oil and one of the dew oils advised for reducing saturated fat intake.

Palm kernel oil is edible plant oil derived from the kernel of the palm oil *elais guinensis*. Palm kernel oil is more unsaturated and can be hydrogenated to a wider range of products which could be used either alone or in blends with other oil for industrial purposes. Lauric oil in palm kernel oil is very important in soap making, most palm kernel oil is now used for the manufacture of short chain fatty acids, fatty alcohols, methyl esters, fatty amines, for use in detergents cosmetics and many other cosmetic products but less consideration is given it for other purpose [13]. Generally, refining processes modify the physical and chemical properties and constituents of these oils to the extent that it could be harmful to human health hence the the objective of this research work is to evaluate the effect of refining on the physicochemical characteristics of cottonseed and palm kernel oils.

2. MATERIALS AND METHODS

2.1 Materials

Two different adsorbent samples (oyster shell and periwinkle shell powders labelled as OSP and PSP respectively) was selected for the bleaching process. The adsorbent samples (oyster and periwinkle shells) were purchased in Warri, Delta State while cotton seed oil (CTSO) was bought from a local producer in Gusau, Zamfara State and palm kernel oil (PKO) was purchased in Ado Ekiti, Ekiti State, Nigeria. All the chemicals used for the study were of analytical grade.

2.2 Methods

2.2.1 Adsorbents modification

Each of the adsorbent samples was treated with 5 M HCl. The reagent (100 mL) was added to 250 g of each of the sample, heated in a water bath at 80°C for 30 min. the resulting mixture was filtered and washed severally with distilled water in order to remove the residual acid reagent. The residue was dried in an electric oven at 105°C for 2 hr. the same procedure was repeated using 5 M H₂SO₄.

2.2.2 Bleaching studies

2.2.2.1 Degumming of oils

The gumming is to remove gum (organo-phosphorous compounds) from the oils before bleaching so as to have a quality product after bleaching.

The oil (cottonseed) was heated at an initial temperature of 60°C before the addition of a food grade acid (phosphoric acid). The acid, 0.1% of oil weight with the acid concentration approximately 85% was added and mixed thoroughly for 15 min to decompose the non-hydratable phosphatides present in the oil as well as to coagulate the phosphatides making them insoluble and thus easily adsorbed during bleaching. The same procedure was repeated for palm kernel oil.

2.2.2.2 Bleaching of oils

The bleaching experiments were performed by batch method on a thermostatically controlled hot plate with stirrer and contact thermometer. The

degummed cottonseed oil (50 g) was treated with 2.5 g of activated oyster shell powder at 90°C for 30 min. the hot mixture was filtered using filter paper. The absorbance was measured by weighing 2.5 g of the filtrate and transferred into 25 mL standard flask and made up to the mark with n-hexane (w/v). The bleaching efficiency of the oyster shell powder was determined by measuring the colour of the bleached cottonseed oil using UV-Visible spectrophotometer (model 752) at 390 nm. The same procedure was repeated using periwinkle shell powder and palm kernel oil samples. The percentage bleaching efficiency for each adsorbent sample was calculated using equation 1:

$$\% \text{ bleaching efficiency} = \frac{A_0 - A_t}{A_0} \times 100 \quad (1)$$

Where; A₀ and A_t are the absorbance of the unbleached and bleached oil samples.

2.2.3 Analysis of oil

2.2.3.1 Determination of free fatty acid (FFA)

The amount of free fatty acids was determined by simple titration using [14]. Few drops of the oil were transferred into a 250 mL conical flask and 50 mL freshly neutralised hot ethanol and 1 mL phenolphthalein indicator was added. The mixture was boiled for about five minutes and titrate while hot against 0.1 N NaOH to the first permanent pink colour after 35 sec.

The results were determined using equation 2.

$$\% \text{ FFA (as palmitic acid)} = 25.6 \times N \times V/W \quad (2)$$

Where N = normality of NaOH solution, V = volume of NaOH used, w = weight of oil.

2.2.3.2 Determination of peroxide value

Peroxide value was determined according to [14].

The oil sample (5 g) was weighed into 250 mL stoppered conical flask. Acetic acid chloroform solvent mixture (30 mL) was added and swirled to dissolve. Saturated potassium iodide (KI) (0.5 mL) was added, the mixture was allowed to stand in dark for one minute with occasional shaking, and 30 mL of distilled water was added. The mixture was titrated slowly with 0.1 N sodium thiosulphate solutions and shaken vigorously until yellow colour almost disappears.

About 0.5 mL starch solution was added as an indicator and the titration continued with vigorously shaking until blue colour disappeared.

Peroxide value is expressed as milliequivalent of peroxide oxygen per Kg sample (meq/kg) using equation 3.

$$\text{Peroxide value} = \text{titre} \times N \times 100 / \text{weight of sample} \quad (3)$$

Where Titre = volume (mL) of sodium thiosulphate used; N = Normality of sodium thiosulphate solution.

2.2.3.3 Determination of iodine value (IV)

The iodine value of an oil or fat is the number of grams of iodine absorbed by 100 g of the fat or oil, by using Wij's solution. The iodine value is a measure of the amount of unsaturation (number of double bonds) in fat or oil. Iodine value was determined according to Pantzaris and Mohammed [14].

The oil sample (0.2 g) was accurately weighed into a 250 mL conical flask with glass stopper, to which 25 mL of carbon tetrachloride had been added. The content was well mixed. Wij's solution (25 mL) was pipetted and added to the mixture, swirled to mix properly and flask was kept in dark for half an hour. Potassium iodide solution (15 mL) was added followed by boiled and cooled water. The mixture was titrated with standardized sodium thiosulphate solution and starch were used as an indicator. The titration was continued until the blue colour formed disappeared with thorough shaking and the iodine value was determined according to equation 4.

Blank determination was also carried out in the same procedure as test sample but without oil.

$$\text{Iodine value} = 12.69(B - S) N/V \quad (4)$$

Where B = Volume in mL of standard sodium thiosulphate solution required for the blank; S = Volume in mL of standard sodium thiosulphate solution required for the sample; N = Normality of the standard sodium thiosulphate solution; W = Weight in gram of the sample.

2.2.3.4 Determination of saponification value (SV)

AOAC method, 920.160 [14] was used for the determination of saponification values of the oils. The oil was weighed (2.0 g) into a 250 mL

Erlenmeyer flask. Ethanol potassium hydroxide solution (25 mL) was pipetted and added to the flask. A blank determination was carried out along with the sample. The resulting mixture was refluxed for one hour. The resulting solution was titrated against 0.5 N hydrochloric acid. Phenolphthalein was used as indicator (about 1.0 mL). The end point was obtained when the pink colour changed into colourless. Saponification value was calculated using the expression as in equation 5.

$$\text{Saponification value} = 56.1(B - S) N/W \quad (5)$$

Where B = Volume in ml of standard hydrochloric acid required for the blank; S = Volume in ml of standard hydrochloric acid required for the sample; N = Normality of the standard hydrochloric acid; and W = Weight in gram of the oil taken for the test.

2.2.3.5 Refractive index (RI)

The refractive index is widely used in quality control to check for purity of materials and to follow hydrogenation and isomerization. The refractive indices were measured by refractometer at constant pressure.

3. RESULTS AND DISCUSSION

The physicochemical parameters of treated and untreated cottonseed and palm kernel oils are reported in Tables 1 to 4. The saponification value indicates the ability of the oil to be used in soap making. It was observed that the oils treated with oyster shell and periwinkle shell powders have high saponification values compared to the values observed in untreated oil samples as this suggested that the oils are normal triglycerides and very useful in production of liquid soap and shampoo in industries. It is an indication of index of high average molecular weight of triacylglycerol in the oils. The high saponification values showed the presence of a greater number of ester bonds which suggest that the fat molecules were intact [15].

Peroxide value is used as an indicator of determination of oils; it is a measure of reactive oxygen content of a fat in terms of milliequivalents per 1000 g of fat. The peroxide values of untreated and treated cottonseed oil (CTSO) (5M HCl modified oyster and periwinkle shell powders) were 19.94±0.09 and 20.53±0.74 respectively and the values of CTSO (5 M H₂SO₄ modified oyster and periwinkle shell powders) were 50.62±0.87 and 50.51±0.71 respectively.

Table 1. Physico-chemical properties of PKO bleached with 5M HCl OSP and PSP

Parameters	OSP		PSP	
	Unbleached	Bleached	Unbleached	Bleached
SV(mg KOH/g)	251.61±0.78	284.32±0.56	251.61±0.78	321.3±0.59
PV(meq/Kg)	24.99±0.01	17.49±0.01	24.99±0.01	20.04±0.08
IV(gl ₂ /100 g)	4.26±0.01	0.88±0.01	4.26±0.01	1.93±0.01
FFA(mEq/L)	11.12±0.26	10.59±0.01	11.12±0.26	10.21±0.02
AV(mg/KOH)	22.60±0.01	21.03±0.25	22.60±0.01	20.49±0.06
RI	1.458±0.00	1.461±0.00	1.458±0.00	1.456±0.00

Table 2. Physico-chemical properties of CTSO bleached with 5M HCl OSP and PSP

Parameters	OSP		PSP	
	Unbleached	Bleached	Unbleached	Bleached
SV(mg KOH/g)	190.32±0.60	231.73±1.54	190.32±0.60	281.67±1.00
PV(meq/Kg)	0.78±0.03	19.94±0.09	0.78±0.03	20.53±0.74
IV(gl ₂ /100 g)	11.41±0.01	3.15±0.13	11.41±0.01	3.41±0.02
FFA(mEq/L)	0.71±0.01	1.71±0.03	0.71±0.01	1.28±0.01
AV(mg/KOH)	1.42±0.00	3.37±0.01	1.42±0.00	2.55±0.01
RI	1.469±0.00	1.470±0.00	1.469±0.00	1.472±0.00

Table 3. Physico-chemical properties of PKO bleached with 5 M H₂SO₄ OSP and PSP

Parameters	OSP		PSP	
	Unbleached	Bleached	Unbleached	Bleached
SV(mg KOH/g)	251.61±0.78	349.82±49.7	251.61±0.78	246.71.3±0.32
PV(meq/Kg)	24.99±0.01	4.99±0.005	24.99±0.01	12.77±0.77
IV(gl ₂ /100 g)	4.26±0.01	1.85±0.01	4.26±0.01	2.53±0.09
FFA(mEq/L)	11.12±0.26	11.18±0.48	11.12±0.26	9.31±0.49
AV(mg/KOH)	22.60±0.01	21.88±0.48	22.60±0.01	21.52±0.12
RI	1.458±0.00	1.459±0.001	1.458±0.00	1.453±0.00

Table 4. Physico-chemical properties of CTSO bleached with 5 M H₂SO₄ OSP and PSP

Parameters	OSP		PSP	
	Unbleached	Bleached	Unbleached	Bleached
SV(mg KOH/g)	190.32±0.60	207.09±1.31	190.32±0.60	308.32±0.60
PV(meq/Kg)	0.78±0.03	50.62±0.87	0.78±0.03	50.51±0.71
IV(gl ₂ /100 g)	11.41±0.01	3.33±0.04	11.41±0.01	3.13±0.06
FFA(mEq/L)	0.71±0.01	1.48±0.11	0.71±0.01	2.17±0.13
AV(mg/KOH)	1.42±0.00	2.86±0.05	1.42±0.00	4.10±0.59
RI	1.469±0.00	1.478±0.01	1.469±0.00	1.464±0.01

Each value presented in Tables 1 to 4 represents mean ± SD of two replicates

The increase observed in the value is as a result of catalytic effect of the treated adsorbent samples on the decomposition of the oils peroxides which is attributed to lipid oxidation. Denniston et al. [16] has reported an increase in peroxide value during bleaching of refined cottonseed oil using acid activated ashes of apricot stones, and the results were higher when tonsil clay was used for the bleaching process. However, there was a reduction in the peroxide values of PKO treated (5M HCl modified oyster

and periwinkle shell powders, 17.49±0.01 and 20.04±0.08 respectively and also 5 M H₂SO₄ modified oyster and periwinkle shell powders with peroxide values of 4.99±0.005 and 12.77±0.77 respectively). Aly and Girgis [17] and Young [18] have reported that breaking down of hydro-peroxides primary oxidation products on the adsorbent surfaces promotes reduction in oxidation level of vegetable oils during bleaching process. Iodine value is a measurement of the unsaturation of fats and oils. The value could be

used to quantify the amount of double bonds present in the oil which reflects the susceptibility of oil to oxidation.

The values obtained showed a decrease in iodine values for PKO and CTSO treated with 5 M HCl and 5 M H₂SO₄ oyster shell and periwinkle shell powders. The decrease in the iodine values after treatment with modified shell powder samples suggest that the oils are more saturated with fewer double bonds, which means that there is decrease in the degree of heat treatment given to the oils during refining [19]. It also implies that they are non-drying oil and contain mostly saturated triglyceride molecules. Kirk and Sawyer [20] proposed that iodine value above 100 makes an oil to be regarded as drying while below 100 is classified as non-drying oil.

The FFA of PKO and CTSO showed higher values except in 5 M H₂SO₄ modified PSP in PKO (9.31±0.49), and a little decrease in FFA values in PKO bleached with 5 M HCl OSP and PSP (10.59±0.01, 10.21±0.02 respectively) The increase is as a result of the treatment of the adsorbent powders. The results were in accordance with [21,22] who reported an increase in FFA in bleached oils. The increase is also attributed to the partial hydrolysis of oils that take place during bleaching process which leads to an increase in FFA of the bleached oils [23]. The acid modification which creates acidic surfaces that readily split the oil into FFAs [23]. Other factors include residual acidic salts probably from ferric and aluminum salts, the food grade acid used during degumming, possible evaporation of liquid water (moisture) from the adsorbents to the hot oils as this promote the rapid rate of hydrolysis of triglycerides in the bleached oils. It is also suggested the decomposition of the secondary oxidation products which was created as peroxide value. It could also be as a result of poor handling, immaturity, and mould growth [24]. The acid value according to [25] is used to measure the degree to which glyceride in the oil has been decomposed by lipase and other actions such as light and heat. The results obtained from the analysis indicate that the acid values of cottonseed oil were 3.37±0.01, and 2.55±0.01 respectively and in CTSO treated with 5 M H₂SO₄ oyster shell and periwinkle shell powders (2.86±0.05 and 4.10±0.59 respectively).

Though, there were increases in the values compared to the untreated CTSO but still fall within the range of acceptable level of 4 mg

KOH/g of oil according to [26]. However, in PKO treated with 5 M HCl and 5 M H₂SO₄ oyster shell and periwinkle shell powders, little decrease was observed but the values were still much higher than the acceptable level.

Higher values are attributed to FFA present in the oil, nevertheless, the higher values are also used to check the level of oxidation deterioration of the oil by enzymatic or chemical oxidation. The acid values can be improved upon by further processing such as dewaxing and deodourisation which may enhance its quality for both domestic and individual uses [27].

The refractive index is widely used in quality control to check for the purity of materials and to follow hydrogenation and isomerisation. It depends on the molecular weight, fatty acid chain length, the degree of unsaturation, and conjugation. The refractive index obtained for the oil samples after treatment appeared to be approximately the same, hence bleaching process does not have a significant effect on the refractive index of the oils. The same has been reported by Oderinde et al. [28], who reported that the refractive index was stable throughout the bleaching process. The slight decrease observed in some of the values obtained is as a result of the continuous removal of impurities during the bleaching process [29]. According to [30], the amount of impurities that are contained in the oil affects the degree of reflection caused by a ray of light during refractive index determination. Ogunsina et al. [31] reported a refractive index of 1.47 which is close to the values obtained in the study.

4. CONCLUSION

The study investigates the effects of bleaching of PKO and CTSO using modified OSP and PSP. The adsorbents performed differently on the constituents in the oil as reflected in their physicochemical properties values obtained. The increase in the saponification values of both bleached PKO and CTSO showed the presence of greater number of ester bonds, suggesting that the fat molecules were intact and therefore indicate that they are good raw materials for soap making.

The peroxide values of PKO bleached with OSP and PSP modified with 5 M HCl and 5 M H₂SO₄ also decreased. There is also a reduction in iodine values of PKO bleached with 5 M HCl and 5 M H₂SO₄ while some of the results show increase in some other parameters indicating

further treatment is necessary to improve on the quality of the oil.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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