

EFFECT OF BLEACHING ON THE PHYSICO-CHEMICAL PROPERTIES OF TWO SELECTED VEGETABLE OILS USING LOCALLY SOURCED MATERIALS AS ADSORBENT

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 analyzing their physico-chemical 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 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 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 H₂SO₄ 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 di-glycerides, 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 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].

29 Vegetable oils are essential part of the human diets and their production through extraction
30 techniques has increased immensely as a result of higher demand and consumption due to
31 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]

32 Refining is carried out to improve shelf life, quality, and nutritive values of the resultant oils
33 [6, 7]. Vegetable oils most especially are widely utilized by food industries as a result of their
34 nutritional properties [8]. Physical and chemical refining are the most common methods
35 available for refining of vegetable oils. Both processes are able to produce refined, bleached,
36 and deodourized vegetable oils of desirable quality and stability suitable for edible purposes
37 [9]. During refining, various kind of minor constituents which may include dirt, moisture,
38 gums (phosphatides), waxes, colour pigments, pesticides residues, odouriferous materials,
39 trace metals (Cu, Fe), free fatty acids. In order to become suitable for human use and
40 consumption, crude vegetable oils are refined to get rid of the unwanted components to
41 produce a stable finished product within desired colour and a pleasant taste with the least
42 possible damages on the desirable components and with the least possible loss of oil [10].
43 Generally, refined oil is clear, odourless, and resistant to rancidity.

44 Cottonseed is among the most unsaturated edible oils. Crude cottonseed oils unsuitable for
45 use in most food application without refining because of its dark colour, high free fatty acid
46 content and objectionable flavor and colour [11]. An important nutritional benefit derived
47 from cotton seed oil is its high level of antioxidants such as tocopherols and numerous minor
48 components which include gossypol, phospholipid, and hydrocarbons [12]. Cotton seed oil is
49 free of cholesterol as in other oils extracted from plants. Linoleic acid is the major
50 polyunsaturated fatty acid present in cottonseed oil with three times as much unsaturated as

- 51 saturated fatty acid. Cottonseed oil is a healthy vegetable oil and one of the dew oils advised
- 52 for reducing saturated fat intake.

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

63 **2. MATERIALS AND METHODS**

64 **2.1 Materials**

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

71 **2.2 Methods**

72 **2.2.1 Adsorbents modification**

73 Each of the adsorbent samples was treated with 5 M HCl. The reagent (100 mL) was added to
74 250 g of each of the sample, heated in a water bath at 80 °C for 30 min. the resulting mixture
75 was filtered and washed severally with distilled water in order to remove the residual acid

76 reagent. The residue was dried in an electric oven at 105 °C for 2hr. the same procedure was
77 repeated using 5 M H₂SO₄

78 **2.2.2 BLEACHING STUDIES**

79 **2.2.3 Degumming of oils**

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

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

87 **2.2.4 Bleaching of oils**

88 The bleaching experiments were performed by batch method on a thermostatically controlled
89 hot plate with stirrer and contact thermometer. The degummed cottonseed oil (50 g) was
90 treated with 2.5 g of activated oyster shell powder at 90 °C for 30 min. the hot mixture was
91 filtered using filter paper. The absorbance was measured by weighing 2.5 g of the filtrate and
92 transferred into 25 mL standard flask and made up to the mark with n-hexane (w/v). The
93 bleaching efficiency of the oyster shell powder was determined by measuring the colour of
94 the bleached cottonseed oil using UV-Visible spectrophotometer (model 752) at 390 nm. The
95 same procedure was repeated using periwinkle shell powder and palm kernel oil samples. The
96 percentage bleaching efficiency for each adsorbent sample was calculated using equation 1

$$96 \quad \% \text{ bleaching efficiency} = \frac{A_0 - A_t}{A_0} \times 100 \quad (1)$$

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

99 2.2.5 ANALYSIS OF OIL**100 2.2.6 Determination of free fatty acid (FFA)**

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

106 The results were determined using equation **2**.

$$107 \text{ \% FFA (as palmitic acid) = } 25.6 \times N \times V/W \quad (2)$$

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

109 2.2.7 Determination of Peroxide Value

110 Peroxide value was determined according to [14].

111 The oil sample (5 g) was weighed into 250 mL stoppered conical flask. Acetic acid
112 chloroform solvent mixture (30 mL) was added and swirled to dissolve. Saturated potassium
113 iodide (KI) (0.5 mL) was added, the mixture was allowed to stand in dark for one minute
114 with occasional shaking, and 30 mL of distilled water was added. The mixture was titrated
115 slowly with 0.1 N sodium thiosulphate solutions and shaken vigorously until yellow colour
116 almost disappears. About 0.5 mL starch solution was added as indicator and the titration
117 continued with vigorously shaking until blue colour disappeared.

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

$$120 \text{ Peroxide value = titre } \times N \times 100 / \text{ weight of sample}$$

121 where Titre = volume (mL) of sodium thiosulphate used; N = Normality of sodium
122 thiosulphate solution

123 **2.2.8 Determination of iodine value (IV)**

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

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

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

$$137 \text{ Iodine value} = 12.69(B - S) N/V \quad 4$$

138
139 Where B = Volume in mL of standard sodium thiosulphate solution required for the blank; S
140 = Volume in mL of standard sodium thiosulphate solution required for the sample; N =
141 Normality of the standard sodium thiosulphate solution; W = Weight in gram of the sample.

142 **2.2.9 Determination of saponification value (SV)**

142 AOAC method, 920.160 [14] was used for the determination of saponification values of the
143 oils. The oil was weighed (2.0 g) into a 250 mL Erlenmeyer flask. Ethanol potassium
144 hydroxide solution (25 mL) was pipetted and added to the flask. A blank determination was
145 carried out along with the sample. The resulting mixture was refluxed for one hour. The
146 resulting solution was titrated against 0.5 N hydrochloric acid. Phenolphthalein was used as
147 indicator (about 1.0 mL). The end point was obtained when the pink colour changed into
148 colourless. Saponification value was calculated using the expression as in equation 5

$$149 \quad \text{Saponification value} = 56.1(B - S) N/W \quad 5$$

150
151 Where B = Volume in ml of standard hydrochloric acid required for the blank; S = Volume in
152 ml of standard hydrochloric acid required for the sample; N = Normality of the standard
153 hydrochloric acid; and W = Weight in gram of the oil taken for the test.

154 **2.2.9.1 Refractive index (RI)**

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

158 **3.0 RESULTS AND DISCUSSION**

159 The physicochemical parameters of treated and untreated cottonseed and palm kernel oils are
160 reported in tables 1 to 4. The saponification value indicates the ability of the oil to be used in
161 soap making. It was observed that the oils treated with oyster shell and periwinkle shell
162 powders have high saponification values compared to the values observed in untreated oil
163 samples as this suggested that the oils are normal triglycerides and very useful in production

164 of liquid soap and shampoo in industries. It is an indication of index of high average
 165 molecular weight of triacylglycerol in the oils. The high saponification values showed the
 166 presence of greater number of ester bonds which suggest that the fat molecules were intact
 167 [15].

168 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(gI 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

169

170 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(gI 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

171

172 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(gI 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

173

174

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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(gI 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

177 • Each value presented in table 1 to 4 represents mean ± SD of two replicates.

178 Peroxide value is used as an indicator of determination of oils; it is a measure of reactive
179 oxygen content of a fat in terms of milliequivalents per 1000 g of fat. The peroxide values of
180 untreated and treated cottonseed oil (CTSO) (5M HCl modified oyster and periwinkle shell
181 powders) were 19.94±0.09 and 20.53±0.74 respectively and the values of CTSO (5 M H₂SO₄
182 modified oyster and periwinkle shell powders) were 50.62±0.87 and 50.51±0.71 respectively.
183 The increase observed in the value is as a result of catalytic effect of the treated adsorbent
184 samples on the decomposition of the oils peroxides which is attributed to lipid oxidation. [16]
185 has reported an increase in peroxide value during bleaching of refined cottonseed oil using
186 acid activated ashes of apricot stones, and the results were higher when tonsil clay was used
187 for the bleaching process. However, there was reduction in the peroxide values of PKO
188 treated (5M HCl modified oyster and periwinkle shell powders, 17.49±0.01 and 20.04±0.08
189 respectively and also 5 M H₂SO₄ modified oyster and periwinkle shell powders with peroxide
190 values of 4.99±0.005 and 12.77±0.77 respectively). [17] and [18] have reported that breaking
191 down of hydro-peroxides primary oxidation products on the adsorbent surfaces promotes
192 reduction in oxidation level of vegetable oils during bleaching process. Iodine value is a
193 measurement of the unsaturation of fats and oils. The value could be used to quantify the
194 amount of double bonds present in the oil which reflects the susceptibility of oil to oxidation.

195 The values obtained showed a decrease in iodine values for PKO and CTSO treated with 5 M
196 HCl and 5 M H₂SO₄ oyster shell and periwinkle shell powders. The decrease in the iodine
197 values after treatment with modified shell powder samples suggest that the oils are more
198 saturated with fewer double bonds, which means that there is decrease in the degree of heat
199 treatment given to the oils during refining [19]. It also implies that they are non-drying oil
200 and contain mostly saturated triglyceride molecules. [20] proposed that iodine value above
201 100 makes an oil to be regarded as drying while below 100 is classified as non-drying oil.
202 The FFA of PKO and CTSO showed higher values except in 5 M H₂SO₄ modified PSP in
203 PKO (9.31±0.49), and a little decrease in FFA values in PKO bleached with 5 M HCl OSP
204 and PSP (10.59±0.01, 10.21±0.02 respectively) The increase is as a result of the treatment of
205 the adsorbent powders. The results were in accordance with [21, 22] who reported increase in
206 FFA in bleached oils. The increase is also attributed to the partial hydrolysis of oils that take
207 place during bleaching process which leads to increase in FFA of the bleached oils [23]. The
208 acid modification which creates acidic surfaces that readily split the oil into FFAs [23]. Other
209 factors include residual acidic salts probably from ferric and aluminum salts, the food grade
210 acid used during degumming, possible evaporation of liquid water (moisture) from the
211 adsorbents to the hot oils as this promote the rapid rate of hydrolysis of triglycerides in the
212 bleached oils. It is also suggests the decomposition of the secondary oxidation products
213 which was created as peroxide value. It could also be as a result of poor handling,
214 immaturity, and mold growth [24]. The acid value according to [25] is used to measure the
215 degree to which glyceride in the oil has been decomposed by lipase and other actions such as
216 light and heat. The results obtained from the analysis indicate that the acid values of
217 cottonseed oil were 3.37±0.01, and 2.55±0.01 respectively and in CTSO treated with 5 M
218 H₂SO₄ oyster shell and periwinkle shell powders (2.86±0.05 and 4.10±0.59 respectively).

219 Though, there were increases in the values compared to the untreated CTSO but still fall
220 within the range of accepted level of 4 mg KOH/g of oil according to [26]. However, in PKO
221 treated with 5 M HCl and 5 M H₂SO₄ oyster shell and periwinkle shell powders, little
222 decrease was observed but the values were still much higher than the acceptable level.
223 Higher values is attributed to FFA present in the oil, nevertheless, the higher values is also
224 used to check the level of oxidation deterioration of the oil by enzymatic or chemical
225 oxidation. The acid values can be improved upon by further processing such as dewaxing and
226 deodourization which may enhance its quality for both domestic and individual uses [27].
227 The refractive index is widely used in quality control to check for the purity of materials and
228 to follow hydrogenation and isomerization. It depends on the molecular weight, fatty acid
229 chain length, degree of unsaturation, and conjugation. The refractive index obtained for the
230 oil samples after treatment appeared to be approximately the same, hence bleaching process
231 does not have a significant effect on the refractive index of the oils. The same has been
232 reported by [28], who reported that the refractive index was stable throughout the bleaching
233 process. The slight decrease observed in some of the values obtained is as a result of the
234 continuous removal of impurities during the bleaching process. According to [29], the
235 amount of impurities that are contained in the oil affects the degree of reflection caused by a
236 ray of light during refractive index determination. [30] reported refractive index of 1.47
237 which is close to the values obtained in the study.

238 **3.1 CONCLUSION**

239 The study investigates the effects of bleaching of PKO and CTSO using modified OSP and
240 PSP. The adsorbents performed differently on the constituents in the oil as reflected in their
241 physico-chemical properties values obtained. The increase in the saponification values of
242 both bleached PKO and CTSO showed the presence of greater number of ester bonds,

243 suggesting that the fat molecules were intact and therefore indicate that they are good raw
244 materials for soap making.

245 The peroxide values of PKO bleached with OSP and PSP modified with 5 M HCl and 5 M
246 H₂SO₄ also decreased. There is also a reduction in iodine values of PKO bleached with 5 M
247 HCl and 5 M H₂SO₄ while some of the results show increase in some other parameters
248 indicating further treatment is necessary to improve on the quality of the oil.

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