

Improving the Quality of Locally Produced Vegetable Oils in Ghana Using Zeolite ZSM-11

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Authors' contributions

This research work was done in collaboration with all authors. Author MAA designed the study, performed the statistical analysis and wrote the final manuscript. Authors DD and GEF did the literature search and laboratory work. Authors BSN and RZ managed the study. All authors read and approved the final manuscript.

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ABSTRACT

The local vegetable oils (Coconut and Palm Kernel Oils) are usually faced with short shelf-life which has been associated with the presence of some unwanted materials present in them. This study explores the potential of using locally manufactures Zeolites in removing these impurities to improve the shelf life of these oils. The Zeolite used in this study was synthesized using locally available raw materials such as bauxite and kaolin through the hydrothermal process X-ray diffraction studies of the synthesized Zeolite were found to be part of the ZSM-11 Zeolite type group. FTIR analysis of the sample showed a weak vibrational peak at 1642cm^{-1} and a strong band at 987cm^{-1} . Batch process for the removal of these unwanted materials was performed. Percentage reduction of 85.71%, 89.22%, 89.36% and 83.81% was recorded for impurities, moisture content, free fatty acid and peroxide value reduction in Palm Kernel Oil and 85.71%, 83.33%, 93.26%, 90.57% for Coconut Oil. Kinetic studies showed the process favored the Pseudo-first order with R^2 of 0.956 and 0.971 for Coconut and Palm Kernel Oil respectively for impurities The plot of (t / q_e) vs. t exhibits a very high linearity and can be concluded that the adsorption rate of the moisture

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content, impurities, FFA and peroxide value is dependent on the adsorbate used and contact time. The final oils had very good physicochemical parameters with improved qualities. It can be concluded that the synthesized Zeolite was effective for the refining of locally produced Coconut and Palm Kernel Oils and can be accessed for commercial applications.

Keywords: ZSM-11; X-ray diffraction; FTIR analysis; vegetable oils; shelf-life; adsorption; kinetic models.

1. INTRODUCTION

The cultivation of oil crops has expanded massively in the past 25 years, transforming millions of lives in rural communities [1]. Vegetable oils can be categorized into two main forms, edible and non-edible oil [2]. The edible types are directly consumed like production of margarine, canned foods, bakery, confectionery, fried foods, etc and the non-edible types include those used for the production of detergents, paints, special varnishes, fatty acids, pharmaceuticals and cosmetics products, biodiesel production, lubricants and painting [3-5]. They contain traces of monoacylglycerols and diacylglycerols, as well as variable amounts of other components such as phospholipids, free and esterified sterols, triterpene alcohols, tocopherols and tocotrienols, carotenes, chlorophylls and other coloring matters, and hydrocarbons as well as traces of metals, oxidation products, undesirable flavors [6]. They are referred to collectively as the Tropical oils and are typically known to be rich in saturated fats [7]. The vegetable oil processing industry involves the extraction and processing of the oils from a variety of fruits, seeds and nuts and this may include husking, cleaning, crushing, and conditioning after which the oil is boiled at high temperatures and filtered [8]. The crude oils usually contain large amounts of triacylglycerols, solid particles with lipase content, free fatty acids, colored pigments such as chlorophylls, xanthophylls and carotenes, and some odoriferous sapid substances present or produced as a result of deterioration [9]. Since oils and fats are exposed to high temperature, atmospheric oxygen and water content of foodstuff it may result in a series of reactions namely hydrolysis, oxidation and polymerization [10-12]. These reactions produce rancid odors, undesirable flavors, and discoloration that considerably influence the functional, sensory and nutritional quality of oils. The oxidative stability, as well as its shelf life, is affected [13]. Refining processes are therefore needed to improve the quality of the oils but this comes with

the effect of high temperature on the quality of the oil produced. Repeated heating of oils at high temperatures (160–190°C) over a long period of time predisposes the oil to thermal oxidation, hydrolysis and polymerization with a configuration change of fatty acid from cis to trans isomers and accelerates the formation of oxidized and polymerized lipid species in the frying medium [14]. In Ghana [15] the refineries do not buy Coconut and Palm Kernel Oil from the small-scale processors due to its poor quality which is attributed to the high free fatty acid, peroxide value, impurities and moisture content which reduces the shelf life of tropical oils [15]. An alternative to the conventional process of refining, which is very expensive to the local producer is the use of inexpensive material such as Zeolite. They are inorganic porous materials having a highly regular structure of pores and chambers that allow molecules to pass through and cause others to be either excluded or broken down [16]. They can be synthesized and tailored to suit different applications such as the refining of tropical oils. In this research work, Zeolite is synthesized from Kaolin and Bauxite and used to improve the oxidative stability of locally produced Coconut and Palm Kernel vegetable oils and thereby increase their shelf-lives.

2. MATERIALS AND METHODS

2.1 Sampling of Oil

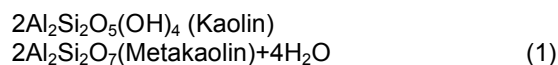
The vegetable oils for this research work are Coconut and Palm kernel oils. They were purchased from a local market in Cape Coast, Central Region of Ghana of stored in airtight polypropylene bottle containers.

2.2 Sampling and Preparation of Kaolin and Bauxite

2.2.1 Sampling and pretreatment of kaolin

The kaolin was obtained from the Northern region of Ghana and was ground into powder form to achieve an even particle size with a mesh

of sieve size 0.75 μm under dry conditions. It was then calcined at 600°C in a Nabertherm furnace for 2 hours into metakaolin [17] where the hydroxyl which was bonded to the kaolinite structure was released during the calcination and thus making it very reactive. [18]. The reaction is given as [19]

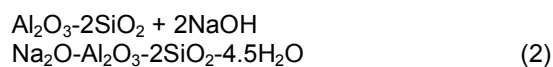


2.2.2 Sampling and pretreatment of bauxite

Bauxite was obtained from Awaso in the Western Region of Ghana, crushed and sieved to obtain an even particle size with a mesh of sieve size 0.75 μm and stored. It was soaked in distilled water to remove organic matter and impurities for three days [20]. The washed bauxite was sun-dried for two days until it was fully dried and pulverized into its powdery form.

2.3 Synthesis of Zeolite

Considering previous processes for the synthesis of zeolite, the method adopted for this was the Alkaline Fusion prior hydrothermal treatment [19,21,22] where 100 g of metakaolin, 20 g bauxite, and 25 g NaOH were weighed into a crucible and fused in a Nabertherm furnace at 600°C for 2 hours [22]. The fused product was allowed to cool to room temperature, ground and 2M NaOH solution was added. The mixture was stirred continuously for an hour to obtain homogenous slurry and then transferred into a stainless steel Teflon Autoclave and placed in a Medcenter Ecocell electric oven at 100°C for 6 hours without stirring or agitation for the zeolite crystals to precipitate [19]. It was allowed to cool to room temperature, filtered, washed with distilled water and dried overnight at 100°C in the oven. The dried synthesized zeolite was then characterized.



2.4 X-Ray Diffraction Analysis of the Synthesized Zeolite

Characterization of the Zeolite crystals was done using the PANalytical Empyrean Powder X-ray Diffractometer of model 000000011136412 which was used to collect data using Bragg-Brentano geometry and a slit configuration of a degree fixed divergence slit of 0. 0.4354°. It was equipped with a CuK α radiation source

($\lambda=1.5406\text{\AA}$, K-Alpha1 [\AA] =1.54060, K-Alpha2 [\AA] =1.54443 and K-Beta [\AA] =1.39225) and operated at 40 mA and 40kV). For phase identification, scans were taken from $2\theta = 5.0150$ to 69.9650 with a step size of 1.000 [19]. The diffraction patterns were identified by comparing it with reference data.

2.5 Fourier Transform Infra-Red Analysis of the Synthesized Zeolite

The functional groups on the Zeolite were determined using the Mattson FTIR spectrometer which was equipped with a ZnSe crystal plate with a Mercury Cadmium Telluride A (MCTA) detector and KBr as a beam splitter. The samples were made thin enough for the infrared light to transmit through. An air background spectrum was collected at the start of the sample collection (Asiedu, 2016). The measurements were done using 100 scans at 4 cm^{-1} resolution, units of $\log(1/R)$ (absorbance), over the mid-IR region of 1200 - 400 cm^{-1} .

2.6 Oil Quality Analysis

The procedure used for the determination of the oil quality was based on that used by [23].

2.6.1 Determination of percentage impurity

This analysis was performed using the procedure adopted by [24] in which crucibles used were washed and then lined with filter paper, washed with hexane, and dried at 100°C for 50 minutes and allowed to cool and weighed (W1). A beaker was weighed (W2) and oil was added and reweighed (W3). Hexane was added to the oils and the flasks swirled and heated to homogenize the mixtures and then poured into the crucibles and allowed to drain. The flask was rinsed with hexane and poured into the crucibles. The crucibles were removed after all the solutions had drained and dried at 105°C for 30 minutes. They were cooled and reweighed (W4). Impurity in the oil was expressed as a percentage from the formula below.

$$\% \text{ Impurity} = \frac{(W4-W1(\text{dry weight of oil}))}{(W3-W2(\text{fresh weight of oil}))} \times 100 \quad (3)$$

Where

- W1 = Weight of crucible + filter paper,
- W2 = Weight of flask
- W3 = Weight of flask + oil
- W4 = Dry weight of crucible + oil

2.6.2 Determination of moisture content

Using the procedure adopted by [24], McCartney bottles were washed with water and oven-dried for 30 minutes and were weighed to the nearest 0.001 g (W1) and 2 g of oil added and reweighed (W2). The samples were dried at 105°C for 4hr and cooled in a desiccator and re-weighed (W3). All samples were replicated three times. The moisture content of the oils was determined by the formula below;

$$\% \text{ MC} = (W2 - W3 \text{ (moisture loss)}) / (W2 - W1 \text{ (fresh weight)}) \times 100 \quad (4)$$

Where:

W1 = Weight of McCartney bottle,
 W2 = Weight of McCartney bottle + oil
 W3 = Dry Weight of McCartney bottle + oil

The same procedure was used by [25] in their recent research work on the properties affecting the cracking of palm nut.

2.6.3 Determination of percentage free fatty acid

In determining the free fatty acid (FFA) content of the oil, a clean dry beaker was weighed to the nearest (W1) and pre-heated oil was added and reweighed (W2) as described by [24] Some amount of ethanol (50 ml) was added to the oil to completely free the fatty acids and the ethanol-oil mixture was titrated with 0.1N NaOH using phenolphthalein indicator. The volume (V) of NaOH required to produce the first permanent pink color was recorded and the free fatty acid content of the oil was determined from the formula:

$$\text{FFA \% (as lauric)} = (T \text{ (ml)} \times \text{normality of NaOH}) / (\text{Weight of sample taken (g)}) \times 0.02 \text{ mg (5)}$$

Where:

FFA (%) = Percentage Free Fatty Acid
 T = Titre

The free fatty acid of Coconut oil was also determined by [26] using the AOCS method Ca 5a-40(1993 which is similar to the method above

2.6.4 Determination of peroxide value

Based on the procedure used by various researchers, 5.0 g of the oil was dissolved in 30

ml acetic acid and 20 ml of chloroform was added. 0.5 ml potassium iodide and 30 ml of distilled water were then added to the mixture. The solution was then titrated with 0.1N standardized sodium thiosulfate using starch indicator until the solution turns from blue-black to colorless.

$$\text{PV meq/kg} = ((S - B) \times N) / (\text{Weight of sample taken (g)}) \times 1000 \quad (6)$$

Where:

PV = Peroxide Value
 S = Sample titration (ml)
 B = Blank titration
 N = Normality of Sodium thiosulphate

This method is similar to that used by [27] in their research on edible oils.

2.7 Adsorption Studies

The Zeolite was used as adsorbate in a batch study with the under room temperature. 100 ml of the vegetable oils were measured and transferred into separate beakers containing 5 g of the zeolite. They were stirred using a laboratory stirrer. The adsorption study was conducted for 5 hours and hourly sampling was done within the period [8]. It was then filtered and transferred into polypropylene containers prior to chemical analysis.

2.8 Oil Quality Tests

The quality of the oils was determined by chemical tests such as peroxide value, impurities, free fatty acid and moisture content [8]. The analysis was conducted at the Ghana Nut Company limited Research Laboratory and the results were compared with standards.

2.8.1 Determination of percentage removal / reduction

The percentage removal of materials from the oil was calculated using the formulae

$$\text{R\%} = ((C_0 - C_t) / C_0) \times 100 \quad (7)$$

Where:

R% = Percentage Removal / Reduction
 C₀ = Initial Concentration (mg/L)
 C_t = Final Concentration (mg/L)

2.9 Kinetic Studies

2.9.1 Determination of pseudo-second order reaction

The Pseudo-First Order Reaction will be determined using the formulae;

$$\log \log (Cq_e - q_t) = \log q_e - \left(\frac{K_1}{2.303} \right) t \quad (8)$$

Determination of Pseudo-Second Order Reaction (HO's Model) [28]

$$\left(\frac{t}{q_t} \right) = \frac{1}{k_2} q_e^2 + \frac{1}{q_e} t \quad (9)$$

Where:

q_e = amount of the adsorbent adsorbed at equilibrium (mg/g)

q_t = amount of the adsorbent adsorbed at a time, t (mg/g)

K_2 = rate constant of second-order adsorption (g/mg/min)

t = time (min)

3. RESULTS AND DISCUSSION

Analysis of Fig. 1 shows that the dominant peaks of the data were observed at $2\theta = 8.09^\circ$, 18.06° , 19.78° , 21.56° , 16.56° , 26.96° , and 50.91° respectively. The main product obtained from the synthesis was Zeolite Socony Mobil-11, commonly known as ZSM-11, coexisting with quartz phase based on the International Zeolite Association standards [29] and research works from [19,30]. These phases were the dominant phases which were observed at different peak levels. The quartz phases were designated as blue which were still dominant at $2\theta = 21.56^\circ$,

21.56° , 26.96° and 50.91° . The ZSM-11 phase was also designated as green was dominant at 8.09° . The unknown phase which is usually considered as interferences or impurities in pure zeolite was designated as red. Therefore the synthesized zeolite was found to be ZSM-11 and quartz. The data were also compared with the ZSM-11 data from the International Zeolite Association Structure Commission and were observed to be similar based on their peak and intensity levels [29].

From the FTIR graph in Fig. 2, it is observed that a very weak vibrational peak at 1642 cm^{-1} can be assigned to the symmetric vibrational mode of surface O-H groups in the zeolite. A very strong band at 987 cm^{-1} is as a result of asymmetric vibrations of the internal T-O bond with a corresponding symmetric vibration recorded as a weak peak at 694 cm^{-1} . Similarly, the O-T-O bending vibration was observed as a very strong peak at 449 cm^{-1} .

3.1 Percentage Removal of Impurities

The percentage reduction of impurities with time is shown in Fig. 3 and depicts a gradual decrease in the values thereby explaining the effectiveness of the adsorbent in impurities removal. It was reduced from an initial value of 0.35 to 0.14 after five hours representing a maximum of 85.71% after 4 hours for Coconut Oil and 0.24 to 0.07 for Palm Kernel Oil with a maximum of 83.33% also after 4 hours.

According to Etelka et al. [31] the presence of impurities destroy the sensory characteristics of the oil which is related to the quality. This implies that using the Zeolite, which was very effective in reducing the impurities level of the vegetable oils could help improve its quality.

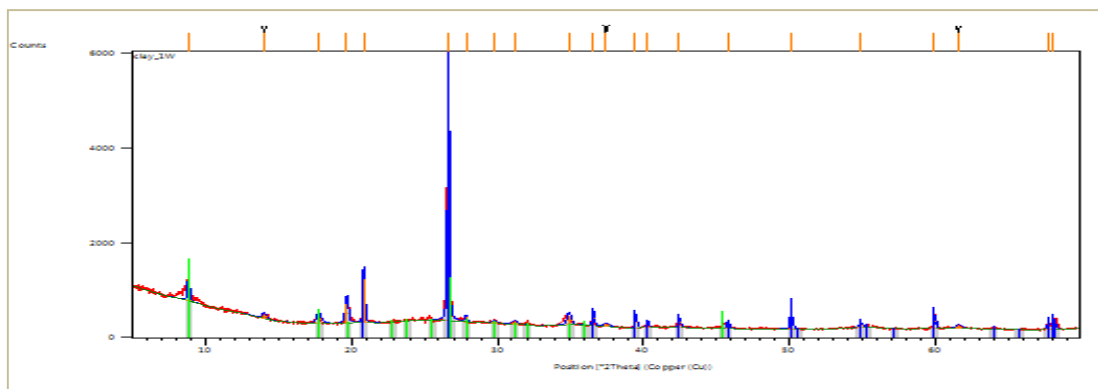


Fig. 1. X-ray diffraction pattern of Zeolite synthesized from Kaolin and Bauxite

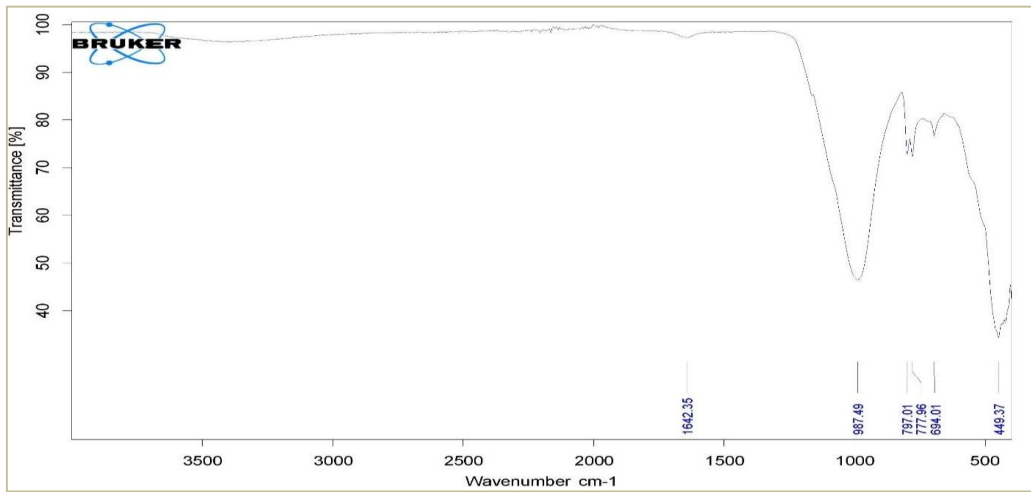


Fig. 2. FTIR Spectra of Zeolite synthesized from Kaolin and Bauxite

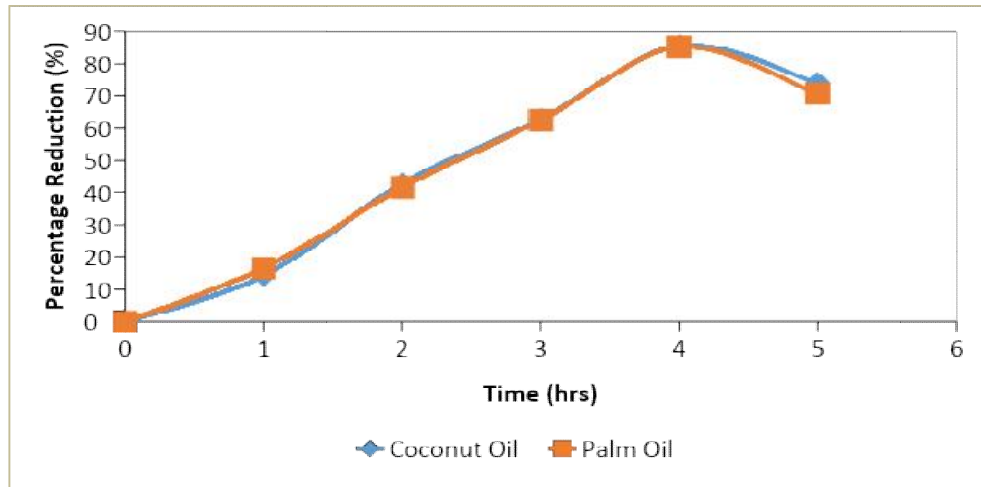


Fig. 3. Graphs of Percentage reduction of impurities against time for Coconut and Palm Kernel Oil

3.2 Percentage Reduction of Moisture

The moisture content of any oil is known to be the cause of hydrolytic and microbial rancidity in vegetable oils [32-34]. The values of the adsorption studies for the reduction of moisture in the oils can be seen in Fig. 4. The initial moisture content for Coconut oil was 0.30 g as compared to 2.32 g for the Palm kernel oil and these values decreased to 0.07 and 0.3 after five hours of treatment with Zeolite.

The moisture reduction process can also be graphically represented in Fig. 4. The initial value of 0.3 g was reduced to 0.05 g after 4 hours representing 83.33% for Coconut Oil and 2.32 g

was reduced to 0.25 g representing a percentage reduction of 89.22% for Palm Kernel Oil. This means the adsorption process was very effective within the first hour for Palm Kernel oil.

3.3 Percentage Reduction of Free Fatty Acid (FFA)

There was a decrease in the values of the free fatty acid from an initial value of 2.82 to 0.19 after 4 hours representing a 93.25% reduction for Coconut Oil and 5.64 to 0.6 for Palm kernel oil representing 89.36% after 4 hours.

The percentage reduction of the free fatty acid content with time on treatment with the

synthesized Zeolite can also be seen in Fig. 5 were the gradual increment was observed to reach a maximum after 4 hours.

3.4 Percentage Reduction of Peroxide Value

Peroxide serves as a useful indicator of the extent of oxidation of lipids, fats, and oils and also shows the degree of peroxidation and measures the number of total peroxides in the substance [35]. It is widely used as a measurement of unwanted reactions in foodstuffs and oils as well as in biological samples where

such reactions are implicated in physiological processes related to the modification of macromolecules causing the initiation of degenerative diseases [36] cancer [37] and aging [38].

The percentage reduction of peroxide value in meq/kg had initial values of 1.59 and 11.2 for Coconut and Palm Kernel Oils and was reduced to 0.3 and 1.8 respectively. The maximum percentage reduction was observed to be 90.57% and 86.61% after 4 hours for Coconut and Palm Kernel Oil respectively as shown in Fig. 6.

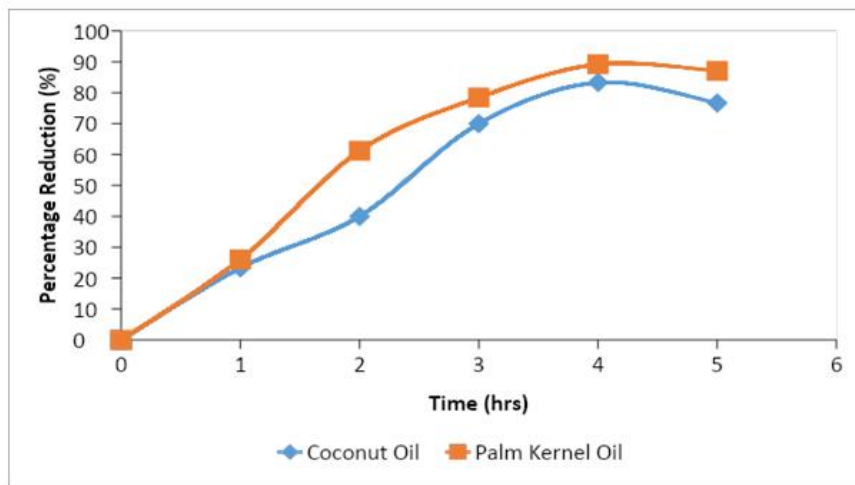


Fig. 4. Graph of percentage reduction of moisture against time for coconut and palm kernel oils

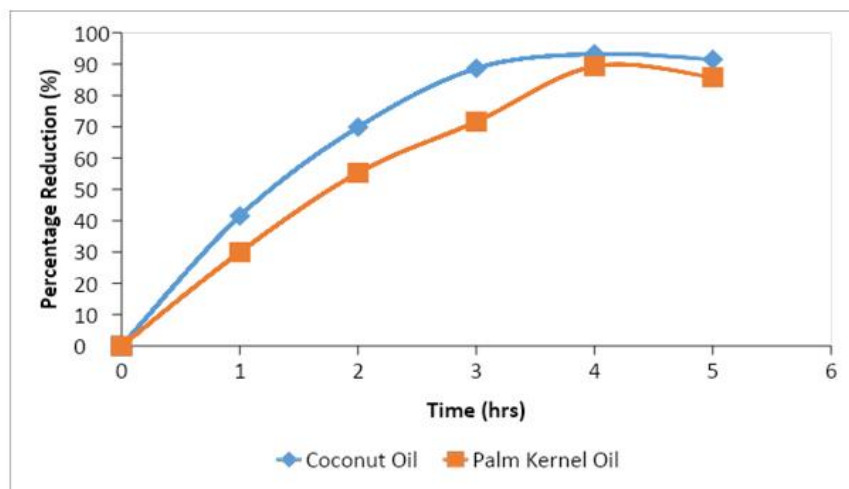


Fig. 5. Graph of Percentage Reduction of Free Fatty Acid against time for Coconut and Palm Kernel Oil

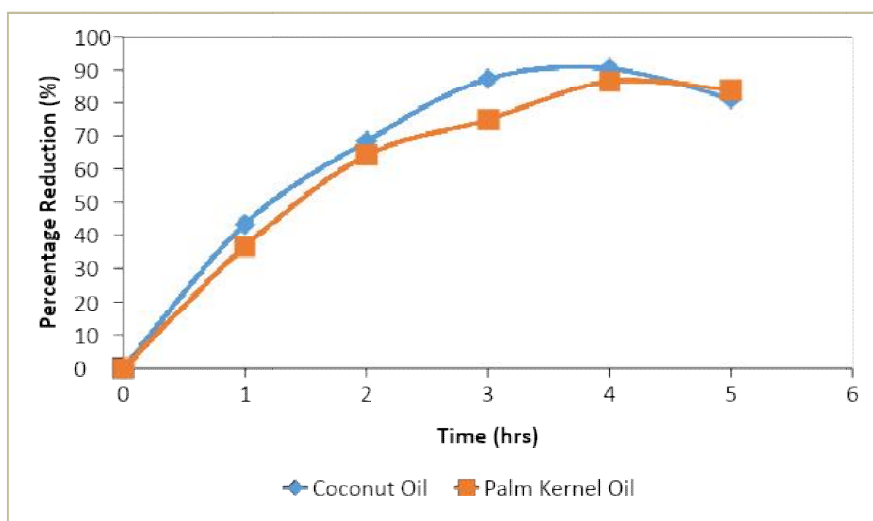


Fig. 6. Graph of percentage reduction of peroxide value against time for coconut and palm kernel oil

3.5 Kinetic Studies

The kinetics of the adsorption process was investigated using the pseudo-first and second-order kinetic models. The graphical representations of the first and second-order pseudo kinetic models are presented in Figs. 7 and 8 respectively.

3.5.1 Pseudo-first order Kinetics

The graphs presented below show the Pseudo-First order kinetics for the reduction of impurities.

It can be seen from Table 1 and Table 2 that the reduction of impurities in the oil process favors the first-order process as shown in Fig. 7 with both having their R^2 values 0.956 and 0.971 for Coconut and Palm Oil respectively although Palm oil shows a higher value and their K_1 was also 0.008 and 0.006 and q_e 0.294 and 0.206.

The adsorption kinetics does not favor the second-order as shown in Fig. 8 with an R^2 value of 0.0061 and 0.0739 for Coconut and Palm oil respectively and their constants were 6.81×10^{-6} and 4.38×10^{-5} with 11.547 and 3.799 as their q_e .

The moisture content, free fatty acid and peroxide value in the oil were also compared to the standard to determine the time that it will take of the treatment process to reach the standard value.

Fig. 9 showed a great reduction for the first hour. From the graph, it can be seen that after three (3) hours of treatment both oils have improved their moisture content and it could be better after that time of treatment.

The reduction in the free fatty acid content was also investigated and compared with standard as shown in Fig. 10.

Table 1. First order kinetics data

Sample	Impurities		
	R^2	k_1	q_e
Coconut	0.956	0.008	0.294
Palm	0.971	0.006	0.206

Table 2. Second order kinetics data

Sample	Impurities			
	R^2	K_2	q_e	h
Coconut	0.0061	6.81×10^{-6}	11.547	0.0009
Palm	0.0739	4.38×10^{-5}	3.799	0.0006

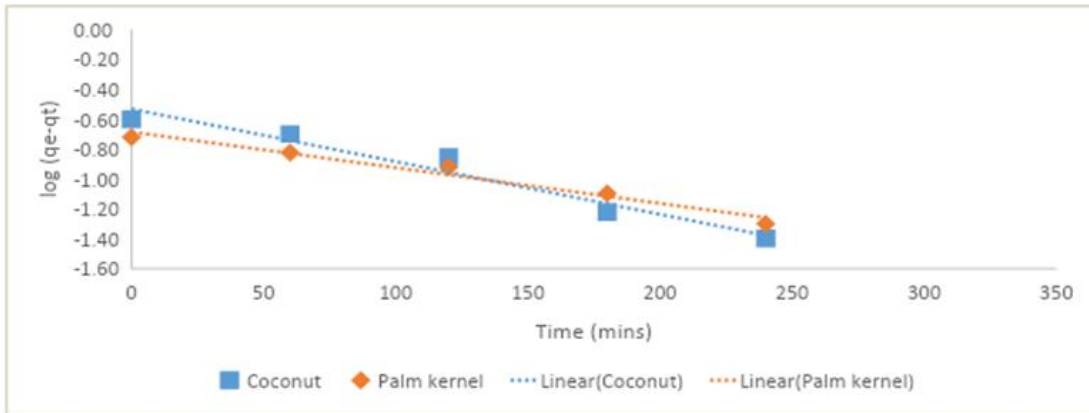


Fig. 7. First order kinetics for the reduction of Impurities from the Oil

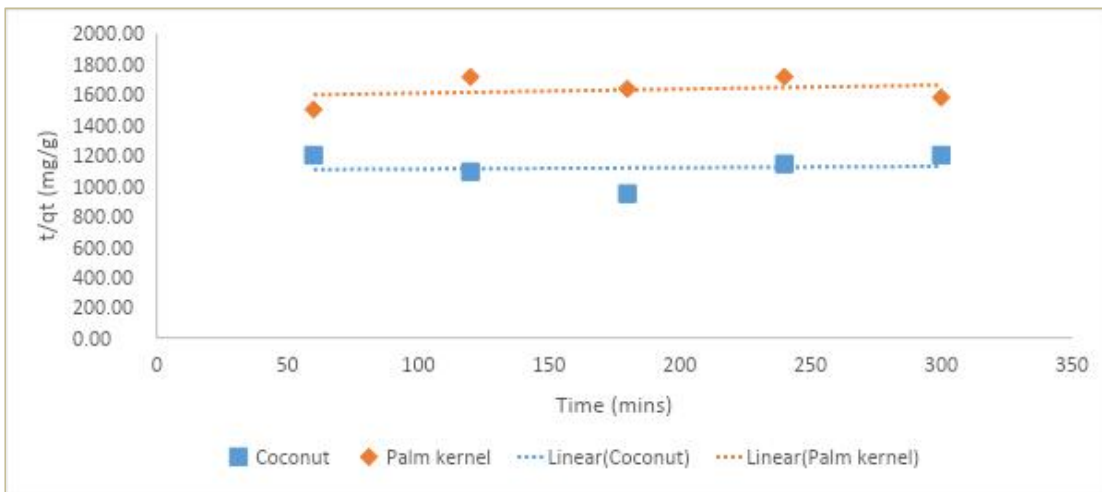


Fig. 8. A second order kinetics for the reduction of Impurities from the Oils

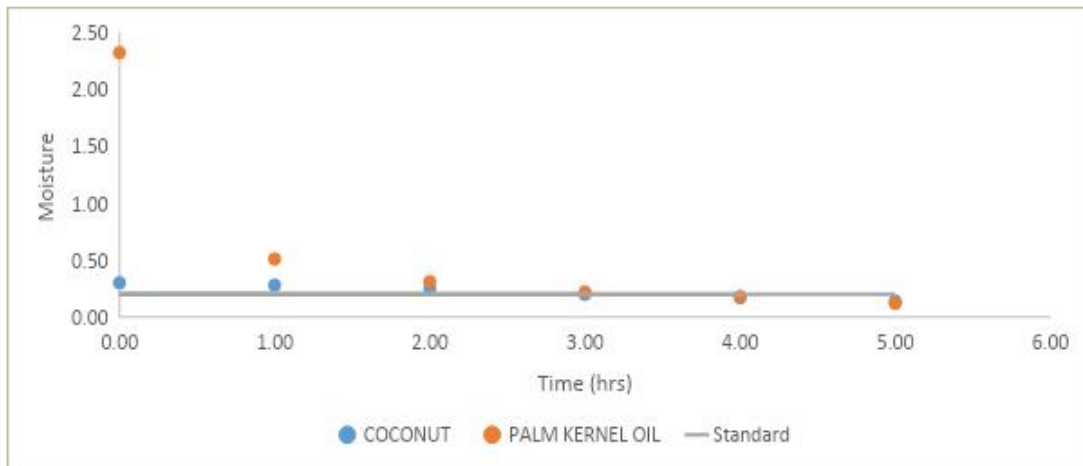


Fig. 9. A graph of reduction of Moisture with Time

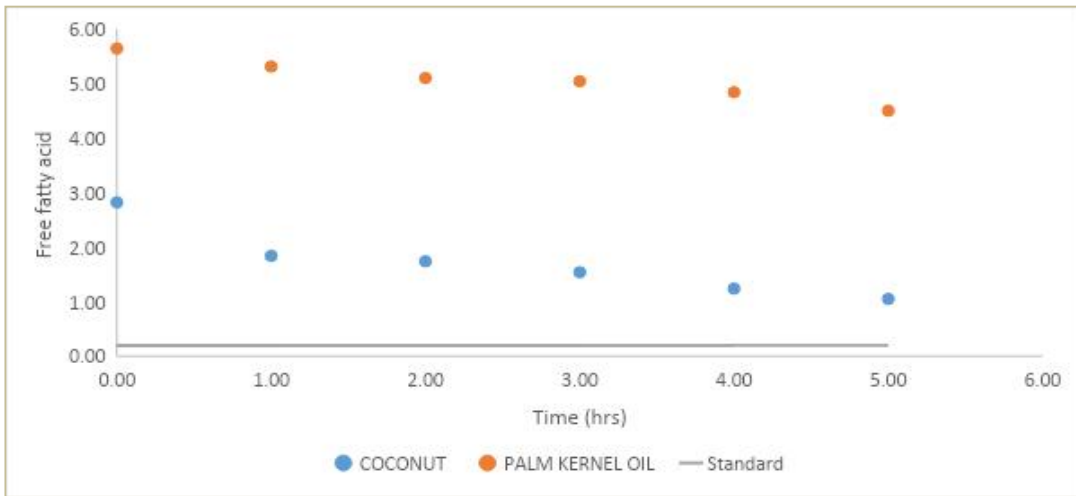


Fig. 10. A graph showing the reduction in free fatty acid content of the oils

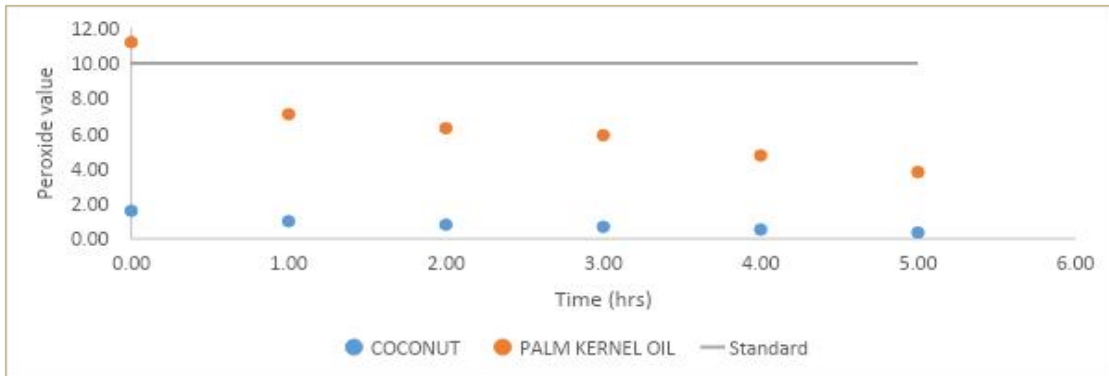


Fig. 11. A graph showing the reduction of peroxide value with time for the oils

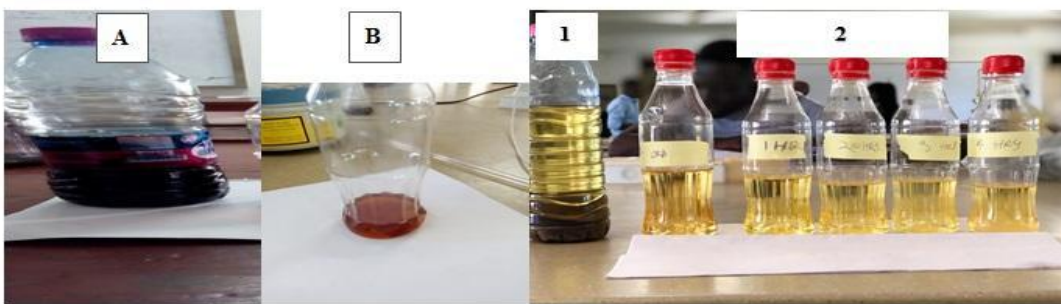


Plate 1. Pictures showing the raw and treated Oil. (A) and (B) represents the unrefined and refined Palm Kernel Oil whereas 1 and 2 represents unrefined and refined Coconut Oil respectively

It was realized that there was a significant reduction but not as expected to the standard value of 0.20. The reduction in the value to meet the standard value had an initial value of 2.82 and 5.64 for Coconut and Palm Kernel Oil respectively and after five (5) hours of treatment

the final values are 0.24 and 0.8. This implies to reach the maximum value; treatment with the Zeolite should go beyond five hours. This was also confirmed by [39] who researched the purification of used palm oil by adsorption. Their results showed that active carbon modified by

perhydrol treatment influenced the quality and the Physico-chemical properties of the used oil.

The effect of the time for the treatment process on the peroxide value was also investigated as shown in Fig. 11. It was high in Palm kernel oil but after an hour of treatment, it fell within the standard value.

4. CONCLUSIONS

The qualities of Coconut and Palm Kernel Oil produced locally have experienced some difficulty in terms of acceptability due to high physicochemical parameters and these have affected their sales. This problem necessitated the need to synthesize Zeolite from local raw Kaolin and bauxite and used it as an adsorbent to treat the oil. The synthesized Zeolite was found to be ZSM-11 and its FTIR- analysis showed a very weak vibrational peak at 1642 cm^{-1} which can be assigned to the symmetric vibrational mode of surface O-H groups in the zeolite. It also has a strong band at 987 cm^{-1} is as a result of asymmetric vibrations of the internal T-O bond with a corresponding symmetric vibration recorded as a weak peak at 694 cm^{-1} . Similarly, the O-T-O bending vibration was observed as a very strong peak at 449 cm^{-1} . When applied to the oil the percentage reduction of some of the parameters such as impurities, moisture, free fatty acid, and peroxide values were calculated. It was observed that 85.71%, 89.22%, 89.36%, and 83.81% was recorded for impurities, moisture, free fatty acid and peroxide reduction in Palm Kernel Oil and 85.71%, 83.33%, 93.26%, 90.57% reduction was also recorded Coconut Oil. Kinetic studies showed the process favored the Pseudo-first order with R^2 0.956 and 0.971 for Coconut and Palm Kernel oils for the reduction of impurities. The plot of (t/q_e) vs. t exhibits very high linearity and can be concluded that the adsorption rate of the moisture content, impurities, FFA and peroxide value is dependent on the adsorbate used and contact time.

It can, therefore, be concluded that Zeolite ZSM-11 can be used to improve the quality of locally produced Coconut and Palm Kernel Oils as shown in plate 1 below where A represented the raw unrefined Palm Kernel Oil and B is the refined product. Pictures 1 and 2 represent the raw and refined Coconut oil respectively.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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