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Preliminary Assessment on the Effect of Bentonite and Ibeshe Clay on Bleaching of Rubber (Hevea Brasiliensis) Seed Oil

Oyinlola R. Obanla, Joseph D. Udonne, Olayinka O. Ajani, Augustine O. Ayeni, Farouk U. Mohammed

Abstract: Rubber seed though not common known is very rich in its oil yield and over time its importance is becoming more prominent. Bleaching in the natural sense is relative to the removal of impurities from the oil or material. In this research work, Rubber seed oil was bleached using bentonite clay and Ibeshe clay at 0.5M, 1M and 2M concentrations of Hydrochloric acid. Physiochemical properties as well as spectroscopic analysis such as FT-IR and XRD analysis were carried out and aided in obtaining the bleaching efficiency of both clays. The FT-IR results displayed a visible change in the oil after it was bleached with Ibeshe clay but still retained most its functional group when bleached with bentonite clay. At 2M concentration of the acid, the oil bleached with bentonite showed 53% while with Ibeshe clay it remained at 16%. This summarizes that Ibeshe clay has little to no effect on bleaching performance.

Keywords: Rubber seed oil, bleaching, bentonite clay, Ibeshe clay, Hydrochloric acid, spectroscopic analysis

I. INTRODUCTION

A large percentage of the human populace have reportedly known rubber seed for its main product which is rubber derived in form of latex as an economic product [12]. Other products of rubber tree are seed and wood which are often overlooked. The plant seed is most abundant and for a long time has resulted to waste because of the limited awareness of this product. Rubber seeds are abundant and little attention had been paid to it. The oil present in this seed is found to contain a large percent of long chain poly-unsaturated fatty acids especially Alpha-linolenic Acid (ALA). Alpha-linolenic acid is one of the important elements of omega-3 fatty acids which play important roles in human metabolism [8]. For compression engines, rubber seed oil has become a promising alternative as it can be refined to form biodiesel [9]. In latex foam, it is a foaming agent which in the synthesis of alkyd resin is used for paints and coating [2], it is also used in soap production [11], [6].

Abdullah and Salimon, 2009 report that the yearly produce of this seed is about 42,980 metric tons. Rubber seed oils have high free fatty acid content when compared to other oils, i.e. it is more unsaturated than others [3].

The principle of adsorption is based on bleaching, which is the removal of pigments and other minor impurities present in the oils being used. Bleaching is done basically for commercial and health purposes (in cases of vegetable oils). In bleaching process, soap traces, phospholipids with some portion of iron and copper are adsorbed. The peroxides present are degraded and further removed [13]. The process is carried out at temperature between 90^{o}C -150^{o}C to ensure maximum efficiency and to properly expel the residual H or OH- ions present in the clay mineral. The use of bleached rubber seed oil for soap production improves its quality; it also makes the oil lighter which is beneficial for use as an improved form of biodiesel or lubricant [12]. Bleaching of different oils (vegetable and non-edible) are mostly carried out using bentonite clay [18], [10]. This research study compares properties of rubber seed oil (RSO) bleached with Ibeshe clay and bentonite clay obtained from different geographical locations in Nigeria (Lagos and Ogun state respectively).Bleaching clay greatly compose of the less stable and less completely weathered clay minerals. They are characterized by high content of loosely held water and by partial solubility in dilute ordinary acids, such as hydrochloric acid or sulphuric acid. The minerals present in the bleaching earth are such that must have strong attraction and great adsorption content to remove the impurities such as bleaching of oils with carbonized seed hulls reported by El-Hamidi and Zaher, 2016.

II. EXPERIMENTAL

A. Materials

The bentonite clay was obtained from Ogun state while the Ibeshe clay was obtained from Ibeshe in Lagos state, Nigeria. All chemicals and solvents used in this study were of analytical grade obtained from Chemical Engineering laboratory, Covenant University, Ota, Ogun state, NigeriThe Bentonite and Ibeshe clay were crushed separately into powder and passed through a 106 µm mesh sieve to obtain powdered sample. Rubber seed used in this study was obtained from Benin City, Edo state, Nigeria. The outer seed shells were manually removed, the seeds were pulverized into small particle sizes and oven dried for 1 hour 30 minutes at 110 °C. The seeds were removed from the oven to cool and further pulverized into fine particle sizes.

B. Extraction of Oil from Rubber Seed

Oil was extracted from pulverized rubber seeds using solvent extraction method as reported by Obanla et al. (2018).

C. Activation of bentonite and Ibeshe clay and bleaching of rubber seed oil

Bentonite and Ibeshe clay was activated adopting Usman et al., (2012) method. The crude clay sample was sun dried at 35 °C and reduced to particle sizes of 106µm. HCl of concentrations 0.5 M, 1.0 M and 1.5 M and used. A mixture of 250 ml of varied HCl concentrations and 50 g of clay sample was stirred at 110 °C for 30 mins. The resulting slurry was washed and filtered until a neutral pH was observed. The activated clay was then oven dried for 3 hours at 110 °C and kept air tight. The bleaching experiment was performed in a batch process. A known quantity of rubber seed oil (RSO) obtained by Obanla et al. (2018) was mixed with 3 g of the activated clay samples and stirred for 30 minutes at 130 °C. The process was repeated using bentonite and Ibeshe clay activated at different HCl concentrations.

Properties	RSO	RSO bleached with Ibeshe clay	RSO bleached with Bentonite clay
Density (kg/m^3)	0.828	-	-
Viscosity	26.7	-	-
Free Fatty Acids (FFA)	7.72	9.25	6.26
Iodine Value	65.53	78.87	69.60
Acid Value	15.43	18.5	12.51
Colour	Dark brown	Reddish brown	Light brown

III. RESULTS AND DISCUSSION

The FFA and acid values of the crude rubber seed oil was higher than the FFA and acid values of bentonite clay bleached RSO while it was lower than the FFA and acid values of Ibeshe bleached RSO (Table 1). This implies that RSO losses a little of its acidity when bleached with Ibeshe clay while it becomes more acidic when bleached with bentonite clay. A highly acidic oil is not edible but it's useful for other purposes such as paint industries and biodiesel production process (as long as the acid value is reduced by esterification process) [16]. The acid value of the oil shows the degree of unsaturation of the oil [5]. Before and after bleaching it is seen that the iodine value changes and thus confirms that the triglyceride molecules break down during bleaching thus increasing the number of free fatty acids The iodine value was observed to be high; this shows that the number of unsaturated free fatty acid present in the oil is high. Based on studies by [17]. The high iodine value suggests that the oil is a semi-drying oil, which highlight its potential as a raw material in lubrication.However, the crude RSO extracted was dark brown this was similar to that reported by Aigbodion et al. (2005). This implies that this oil is suitable in cases where its consideration doesn't span from bright colours. The efficacy of the Ibeshe clay as a bleaching agent was demonstrated in the reddish brown colour observed in Table 1 while a lighter brown colour was observed using bentonite clay. This proves that both Ibeshe and Bentonite clay are good bleaching agent in its decolorization ability.

A. Effect of concentration on bleached RSO with both clays

Figure 1 shows that Bentonite clay demonstrates a higher efficiency of bleaching RSO than Ibeshe clay in all concentrations of HCl used, this implies that the higher the concentration of the acid the more refined the oil becomes

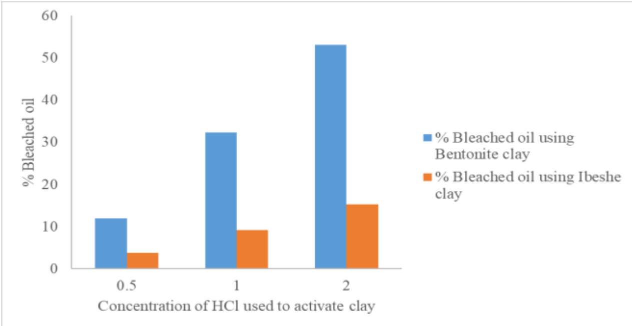

Fig 1: Effect of concentration of the RSO bleached with Bentonite and Ibeshe clay

B. Fourier Transform Infra-Red (FTIR) spectroscopy analysis

FT-IR analysis of crude RSO

Figure 2 shows the FT-IR analysis for the crude RSO before bleaching. This peaks show a strong broad appearance and a symmetric and asymmetric N-H stretching group. Because of the presence of two bands in this group, its compound name is aliphatic primary amine. Between the range of 1500 cm^{-}1 – 1750 cm^{-}1 a medium appearance was observed and this was between the ranges of double bonds such as a C=C, C=N or C=O double bond. In relation to the frequency chart, it shows that within the range of 1500 cm^{-}1 – 1750cm^{-}1 the functional group present is the C=C, which represents the Cyclic Alkene group.

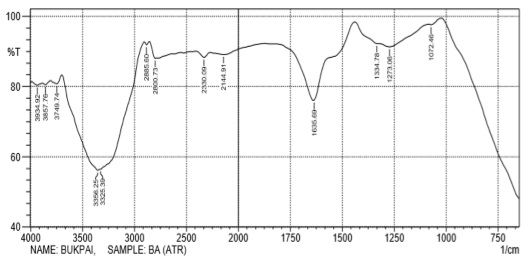

Fig 2: FT-IR spectra of crude R

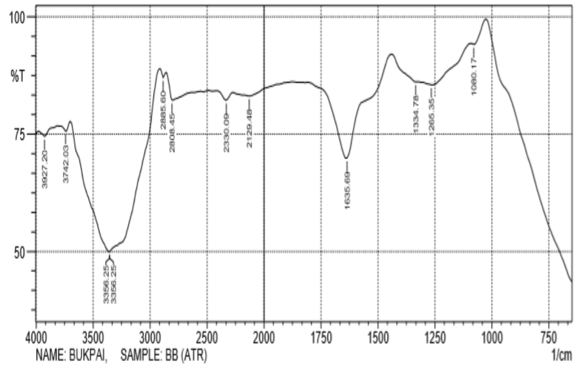

Fig 3: FT-IR spectra of the RSO bleached with bentonite clay activated with 0.5M HCl

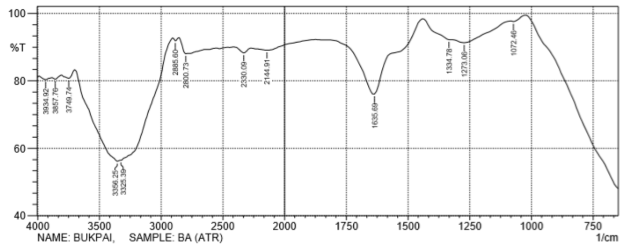

Fig 4: FT-IR spectra of the RSO bleached with bentonite clay activated at 1M HCl

SO extracted

FT-IR analysis of RSO bleached with Bentonite clay activated with 0.5 M, 1 M and 2M HCl

Figure 3 shows the FT-IR spectra of RSO after bleached with Bentonite clay activated with 0.5 M HCl. The figure shows two prominent bands between the ranges of 3000 $cm^{-}1$ – 3500 $cm^{-}1$ this was similar to peaks observed in the crude rubber seed oil reported by Nian-yian et al. (2014).

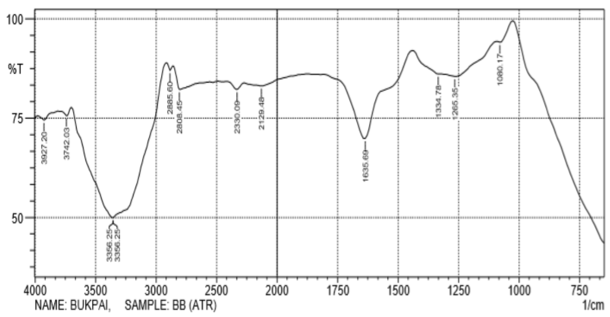

Fig 3: FT-IR spectra of the RSO bleached with bentonite clay activated with 0.5M HCl

Figure 4 and 5 shows similar trend in the FT-IR spectra as the prominent band seen at 3363.97 $cm^{-}1$ shows a strong broad appearance attributed to N-H stretching referred to as secondary amine. The peak with a weak absorbance seen at 2800.73 $cm^{-}1$ indicate the presence of an alkane with a C-C single bond. The band at 1635.60 $cm^{-}1$ shows a narrow medium appearance within the region of C=C double bond as the functional group with the compound name as cyclic alkene which is similar to previous study.

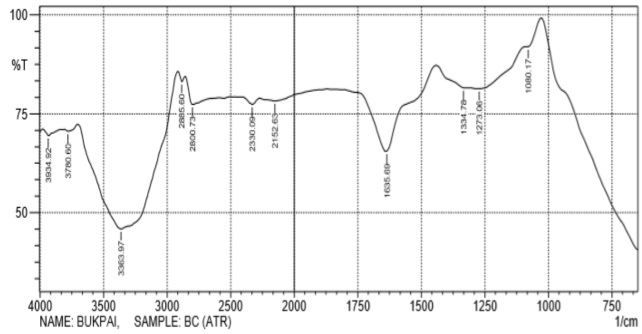

Fig 4: FT-IR spectra of the RSO bleached with bentonite clay activated at 1M HCl

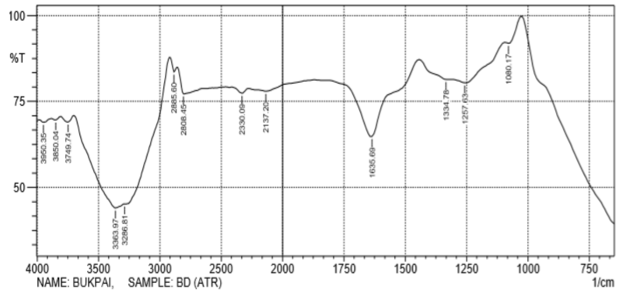

Fig 5: FT-IR spectra of the RSO bleached with bentonite clay activated with 2M HCligures and Tables

From the above, it can be inferred that FT-IR spectra of the crude and bleached RSO indicate that the bleaching does not distort the functional group; this indicates that during the activation stage the acid opened up the pore diameter and surface area between each layer of the clay material thereby enhancing the efficiency of the bleaching process.

FT-IR analysis of RSO bleached with Ibeshe clay activated with 0.5 M, 1 M and 2M HCl

FT-IR spectra of RSO bleached with 0.5 M activated Ibeshe clay was illustrated in Figure 6. The oil bleached at this concentration shows that it had minimal effect on the oil. The spectra was similar to RSO bleached with bentonite clay activated with 0.5 M of HCl. The absorbance band observed at 3356.25 $cm^{-}1$ show a broad medium appearance and an N-H stretching as the functional group identified as a secondary amine. At 1635.60 $cm^{-}1$ absorbance band shows a narrow medium appearance with a C=C double bond identified as cyclic alkene.

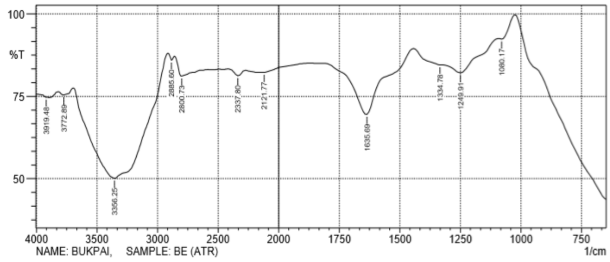

Fig 6: FT-IR spectra of the RSO bleached with Ibeshe clay activated with 0.5M HCl

Figure 7 shows the FT-IR spectra of RSO bleached with Ibeshe clay activated with 1M HCl. A single band was observed at 3417.98 cm^{-1} and it indicate the presence of an N-H stretching functional group classified as a secondary amine. The peak at 2808.45 cm^{-1} shows a weak appearance with a C-C single bond identified as an alkane group. The band seen at 1234.48 cm^{-1} and 1141.90 cm^{-1} represents a C-N stretching functional group. The former band represents a primary amine while the latter represents a secondary amine. The band at 1033.68 cm^{-1} shows a narrow strong appearance with a functional group of S=O identified as sulfoxide.

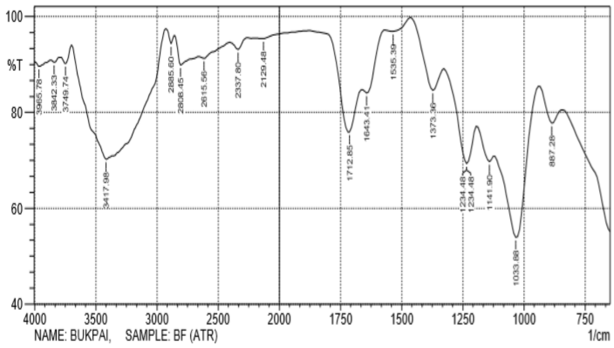

Fig 7: FT-IR spectra of the RSO bleached with Ibeshe clay activated with 1M HCl

Figure 8 shows the FT-IR spectra of RSO bleached using Ibeshe clay activated with 2M HCl. The band peak at 3410.20 cm^{-1}, shows a broad medium appearance indicating the presence of an N-H stretching, representing a secondary amine. The peaks at 1239.26 cm^{-1} and 1165.04 cm^{-1} show a C-N stretching bond. The former representing a secondary amine and the latter indicating a primary amine. At 1034.58 cm-1 band the appearance of an S=O functional group which also indicates the presence of Sulfoxide

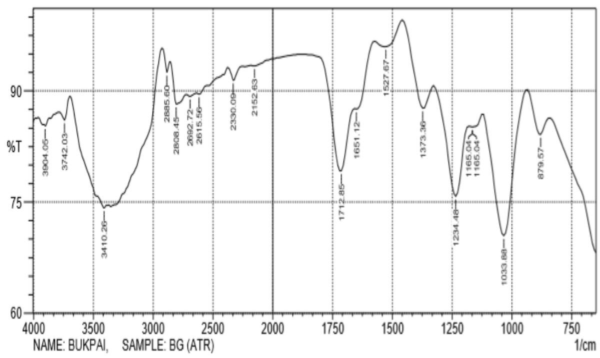

Figure 8: FT-IR spectra of the RSO bleached with Ibeshe clay activated with 2M HCl

From the FT-IR spectra using Ibeshe clay with 0.5 M, 1 M and 2 M obtained above, it can be concluded there was a distortion in the usual arrangement of the oil as regards its functional group and this occur as a result of the destruction of the inner layers of the Ibeshe clay when in contact with the acid. The higher the concentration of the acid the more the structure will be broken down instead of increasing its surface area for adsorption.

C. X-Ray diffraction analysis

XRD analysis showa the minerals present within Bentonite and Ibeshe clay structure. As reported by Usman et al., (2012) XRD analysis for the Ibeshe clay before it was bleached showed that the clay contained minerals such as kaolinite and quartz as its main constituents with trace quantities of Merlinite and Illmenite. Figure 9 and 10 shows the presence of clay minerals (Kaolinite, Quartz, among others) after the bleaching process. Table 2 and 3 displays the pattern list for Ibeshe clay bleached with 2 M and 1 M HCl respectively. The kaolinite and quartz was observed to be higher in Ibeshe clay bleached with 2 M HCl than Bentonite clay bleached with 2 M HCl. This result agrees with previous studies by Usman et al. (2012). Although Usman et al. (2012) claim that due to the presence of kaolinite in the Ibeshe clay it may not be suitable for bleaching. This was confirmed in this study as it demonstrates poor bleaching capacity. Useful insight has thus been obtained as a consequence of low RSO acidity experienced after it was bleached.

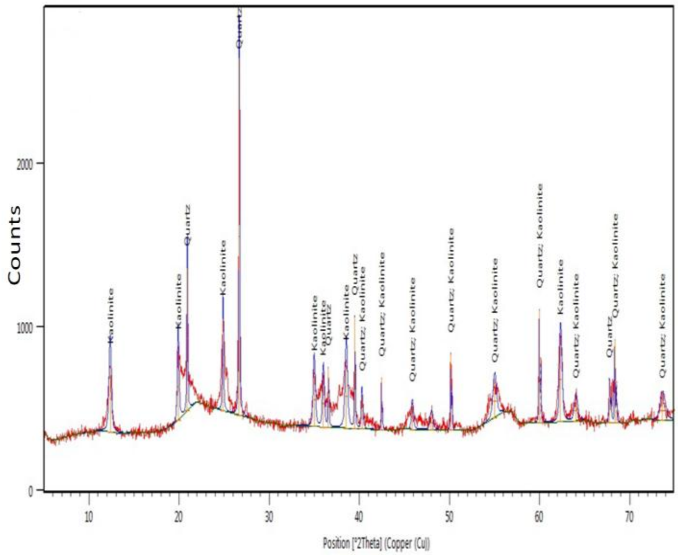

Figure 9: XRD graph of Ibeshe clay bleached with 2M HCl

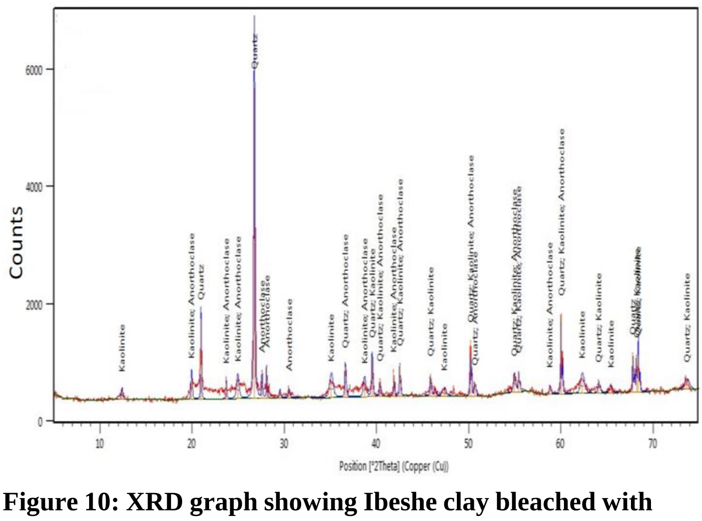

Figure 10: XRD graph showing Ibeshe clay bleached with 1M HCl.

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Table 2: XRD Pattern list for 2M bleached Ibeshe clay

Ref. code	Score	name	Scale factor	formula
96-900-9235	33	Kaolinite	0.283	$Al_2Si_2O_5(OH)_4$
96-901-0146	70	Quartz	0.734	Si_2O_2

Table 3: XRD Pattern list for 1M bleached Ibeshe clay

Ref-code	Score	name	Scale factor	formula
96-900-9235	25	Kaolinite	0.072	$Al_2Si_2O_5(OH)_4$
96-901-0145	67	Quartz	0.734	Si_2O_2
96-900-0838	8	Anorthoclase	0.060	$Na_{1.5}K_{0.5}Al_2Si_6O_{16}$

IV. CONCLUSION

The bentonite and Ibeshe clay were accessed separately based on their bleaching efficiency. It is relative to say that bentonite clay readily refines the properties of the oil compared to Ibeshe clay. From previous studies the chemical composition of $MgO2^+$ and $CaO2^+$ are very small but during the activation stage these ions were exchanged with acid protons to activate the binding site. Thus when activation occurs, these ions are not present, therefore the acid attacks the inner layer instead of the exchange taking place which causes the destruction of layers of the clay.

The RSO is applied in cases where its dark color isn't the main factor considered; an example can be in enhancing of coating surfaces; though its color is improved after bleaching. RSO after much research shows that this vegetable oil is not suitable for edible purposes due to its high iodine valve. Further observation based on the analysis enables us to know that this oil is good as a semi-drying agent and is suited for synthesis of oil vulcanization. Vulcanization here simply implies the addition of sulphur to a rubber-like material or elastomer to improve its quality.

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