



Characterization of Natural Ca-Bentonite Clays from Ebenebe in Anambra State, Nigeria, Impregnated with $\text{Ca}(\text{NO}_3)_2$, $\text{Zn}(\text{NO}_3)_2$ and KOH for use as Catalysts for Viscosity Reduction in Transesterification Reaction of Castor Oil

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ABSTRACT: Natural Ca-Bentonite from Ebenebe in Anambra State, Nigeria, was successfully impregnated with $\text{Ca}(\text{NO}_3)_2$, $\text{Zn}(\text{NO}_3)_2$ and KOH and further calcined under different conditions of temperature and time using standard methods. Both the natural and impregnated bentonite clays were characterized using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Fluorescence Spectroscopy (XRF) and Scanning Electron Microscopy (SEM) and then applied as catalysts in transesterification reaction of refined castor oil. Viscosity reduction test was used to ascertain the best catalyst for the transesterification. FTIR spectra of the activated clay catalysts show the formation of new functional groups, as the intensity of the Al-O-H stretching group (3633cm^{-1}) of the natural clay increased with slightly higher wavenumbers of 3640cm^{-1} , 3644cm^{-1} , 3644cm^{-1} , 3644cm^{-1} , 3640cm^{-1} and 3644cm^{-1} for CaB_2 , CaB_3 , ZnB_2 , ZnB_3 , KB_{400} , and KB_{700} catalysts respectively. XRF result identified the natural bentonite clay as Ca-bentonite clay with Ca-oxide having the most dominant component of 76.71%. XRF chemical compositions of the six activated Ca-bentonite clay catalysts when compared to the natural Ca-bentonite clay confirmed successful impregnation. The SEM images of all the prepared catalysts showed that impregnation and calcination changed the surface morphology of the natural clay. Finally, transesterification reactions of refined castor oil samples were done with 3% w/w natural and impregnated Ca-bentonite catalysts, methanol to oil ratio of 6:1, 2 hours reaction time and 65°C reaction temperature. Results obtained showed a descending order of catalyst viscosity reduction ability to be $\text{KB}_{700} > \text{CaB}_3 > \text{CaB}_2 > \text{ZnB}_3 > \text{NCB} > \text{ZnB}_2 > \text{KB}_{400}$.

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Clay minerals are small fragments of hydrous layer silicates. These important minerals with specific characteristics include montmorillonite, kaolinite, illite, vermiculite and chlorite (Ejikeme *et al.*, 2013). Bentonite clay is a versatile class of smectite natural mineral, formed from alteration of siliceous glass – rich volcanic rocks and composed mainly of montmorillonite. It consists of 2:1 layer (Hwu *et al.*, 2001), between which are exchangeable hydrated ions. A layer is composed of an octahedral sheet surrounded by two tetrahedral sheets (T.O.T) (Jahan *et al.*, 2012). The basic unit of the octahedral layer is an octahedron which consists mainly Al^{3+} or Mg^{2+} surrounded by six oxygen atoms or hydroxyl groups. The tetrahedral layer has a tetrahedron as its basic unit and it contains one Si^{4+} at the centre with four oxygen atoms or hydroxyl groups at the corners. Bentonite was first used as a term to describe a particular highly colloidal,

plastic clay, found near Fort Benton at the cretaceous beds of Wyoming, USA (Ujeneza, 2012; Kutlic *et al.*, 2012). Bentonite clays are classified as Sodium (Na) -, Calcium (Ca) -, Potassium (K) - or Aluminium (Al) - bentonites depending on the dominant element (Ali *et al.*, 2016). Ca – bentonite (a non-swelling type bentonite) and Na – bentonite (a swelling type bentonite) possess some physical properties that make them valuable to various industries (Olugbenga *et al.*, 2013). Clay minerals and deposits generally vary in nature. Abdullahi *et al.*, 2017, reports that samples from the same deposit tend to differ in the nature of exchangeable cations they contain and in the degree of chemical substitution within the smectite structure. Abdullahi *et al.*, 2017, further reports that investigations by the Nigerian mining co-operation established the existence of bentonitic clay reserves of over 700 million tonnes in different parts of Nigeria.

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Activated clays have been used as catalysts for various reactions of industrial interests (Foletto *et al.*, 2011, Didi *et al.*, 2009). Reports on instrumental analysis of natural and activated clay samples for use as catalysts are available (Soetaredjo *et al.*, 2011, Ali *et al.*, 2016). Soetaredjo *et al.*, 2011, impregnated bentonite with KOH, calcined and characterized it with Fourier Transform Infrared Spectroscopy. It served as a catalyst for transesterification of palm oil and gave favourable results. Different plant oils such as castor oil, various grades of shea butter (Ofoegbu- Chibuzo *et al.*, 2022), palm oil, waste vegetable oil, jatropha oil, etc, can be utilized for transesterification reactions. Castor oil, an inedible oil, is a promising feedstock in transesterification reaction and thus, biodiesel production. Castor seed thrives very well on marginal land and though the oil has high viscosity, it has some advantages over other vegetable oils. Castor oil has more oxygen content and this enables more complete combustion (Conceicao,ao *et al.*, 2007). It also does not contain sulphur, thus, there is zero emission of oxides of sulphur (Conceicao,ao *et al.*, 2007). Again, castor oil has higher cetane number which depicts better quality of ignition (Ismail *et al.*, 2016). Castor oil majorly contains hydroxylated fatty acids of ricinoleic acid, unlike other vegetable oils, and this makes it a better lubricity enhancer (Ramezani *et al.*, 2010). Castor biodiesel is also proved to be the best fuel for cold weather as it has a very low cloud point and pour point (Ismail *et al.*, 2016, Forero *et al.*, 2004). It is however, usually blended with Petro diesel, because of its associated high viscosity and density. Transesterification performance varies inversely with viscosity, thus, castor oil with its high viscosity can be transesterified and viscosity reduction used to verify the transesterification performance of the natural and impregnated catalysts. Enweremadu and Alamu, 2010, reported that viscosity is the basic parameter which reflects the extent of the transesterification reaction, as it directly relates to the methyl ester content. Thus, a significant change in viscosity with reaction indicates the removal of more of the heavy glycerine, which in turn signifies more complete reaction. In this study, natural Ca-Bentonite clay samples from Ebenebe in Anambra State, Nigeria, were impregnated with $\text{Ca}(\text{NO}_3)_2$, $\text{Zn}(\text{NO}_3)_2$ and KOH for use as catalysts for viscosity reduction in transesterification reaction of castor oil and characterized.

MATERIALS AND METHODS

Sample collection and preparation: Pulverized natural Ca-Bentonite, sourced from Ebenebe in Anambra State, Nigeria, was obtained through Mandid Nigeria Limited. The samples were rinsed with distilled water, filtered, and oven dried at 105°C for 24 hours to remove

water. The dried bentonite was crushed and sieved to obtain powdered bentonite.

Refined castor oil was obtained from Ayrton Saunders Co Ltd, Liverpool, UK. Analytical grade reagents were obtained.

Catalysts Impregnation: The catalyst impregnation was done following the method described by Ngobiri *et al.*, 2020 as modified. 10g of natural calcium-bentonite (NCB) was first dispersed in 200ml of distilled water and stirred with magnetic stirrer for 10 minutes. At 65°C, a pre-dissolved amount of $\text{Ca}(\text{NO}_3)_2$ (2.5 g in 10 ml), $\text{Zn}(\text{NO}_3)_2$ (2.5 g in 10 ml) and KOH (2.5 g in 10 ml) was added separately to three separate samples of bentonite suspension. The reaction mixtures were stirred to a point of near dryness at the same temperature. The impregnated bentonite samples were dried in a laboratory oven for 24 hours at 110°C. The freshly dried clay samples were grinded into powder using a pestle and mortar and then stored in separate air tight containers for calcination.

Catalysts Calcination: $\text{Ca}(\text{NO}_3)_2/\text{Ca}$ -bentonite catalyst was divided into sample A and sample B. Sample A was calcined in a tubular muffle furnace at a nitrate decomposition temperature of 650°C (Olivares, 2012) for 2 hours to obtain CaO/Ca -bentonite catalyst sample A (CaB_2). Similarly, sample B was calcined at 650°C for 3 hours to obtain CaO/Ca -bentonite catalyst sample B (CaB_3). $\text{Zn}(\text{NO}_3)_2/\text{Ca}$ -bentonite catalyst was divided into sample C and sample D. Sample C was calcined at 650°C for 2 hours to obtain ZnO/Ca -bentonite catalyst sample C (ZnB_2) while $\text{Zn}(\text{NO}_3)_2/\text{Ca}$ -Bentonite catalyst sample D was calcined at 650°C for 3 hours to obtain ZnO_3/Ca -bentonite catalyst sample D (ZnB_3). KOH/ Ca -bentonite catalyst was divided into sample E and sample F. Sample E was calcined at a temperature of 400°C for 5 hours (Soetaredjo *et al.*, 2011) to obtain $\text{K}_2\text{O}/\text{Ca}$ -bentonite catalyst sample E (KB_{400}) and KOH/ Ca -bentonite catalyst sample F was calcined at a temperature of 700°C for 2 hours to obtain $\text{K}_2\text{O}/\text{Ca}$ -bentonite catalyst sample F (KB_{700}).

Characterization: FTIR Spectrophotometer (Shimadzu 8400S) was used for qualitative analysis (functional groups) of raw bentonite and impregnated bentonite samples. XRF Spectrophotometer (Bruker Penguian model) was used for qualitative and quantitative analysis (chemical compositions) of natural bentonite and activated bentonite catalyst samples. Scanning Electron Microscopy (Sputta Coater model QISOIRES) was used to study the surface morphology of the natural and prepared catalysts.

Catalysts Transesterification Run: The sample catalysts (NCB, CaB₂, CaB₃, ZnB₂, ZnB₃, KB₄₀₀, & KB₇₀₀) were used for the transesterification of refined castor oil. This was carried out in a 250ml glass reactor equipped with a reflux condenser and magnetic hot plate stirrer set at 65°C. 3w/w% catalyst loading of the castor oil was added to a known volume of methanol and heated up to 65°C. The castor oil was heated up to 65°C and subsequently added to the reaction mixture of methanol to oil mole ratio of 6:1 under vigorous stirring. The transesterification reaction was carried out for two hours and the solution was thereafter centrifuged at 2500 rpm for 15 minutes in order to separate out the biodiesel from the catalyst and glycerol. After separation, 20ml of warm distilled water was gently poured and the mixture was gently stirred to avoid foam formation (Van, 2004). It was then left over night (12hrs) in a separation funnel to settle into water impurities phase and biodiesel phase. The biodiesel layer was then heated up to 105°C for 1hour to evaporate any remaining water molecule. After drying, the weight of biodiesel was taken while the biodiesel was poured into an air-tight container and stored in a dark cupboard for further analysis.

Science & Technology Co. Ltd was used to obtain the kinematic viscosities of the various transesterified samples at 40⁰C using ASTM D445-19 standard method.

RESULTS AND DISCUSSION

Table 1 summarizes the important functional groups assignment of NCB, as given in the FTIR spectrum of NCB. From the table, Al/Mg-O-H stretching (inter-octahedral) appeared at 3633 cm⁻¹, H-O-H stretching at 3450 cm⁻¹, Al-OH bending deformation (Al-O bending vibrations) at 918 cm⁻¹, Si-O stretching vibrations or (Si-O bending vibration) at 1119 cm⁻¹, and OH deformation vibration of water (bending vibration of groups in adsorbed water molecules) at 1689 cm⁻¹. According to previous report by Ritz *et al.*, 2010, the first four assignments in Table 1 identify NCB as bentonite clay. Table 2, presents the important functional groups assignment from the FTIR spectra for the natural Ca-bentonite catalyst, NCB, for the CaO/Ca-Bentonite catalyst samples A and B (CaB₂, CaB₃), for the ZnO/Ca-bentonite catalyst samples C and D (ZnB₂, ZnB₃) as well as for K₂O /Ca-bentonite catalyst samples E and F (KB₄₀₀, KB₇₀₀).

Kinematic Viscosity Test: HK-265 kinematic viscosity apparatus from Liaoning Huake Petroleum Apparatus

Table 1: FTIR Assignments of natural Ca-bentonite clay (NCB)

Wavelength (cm ⁻¹)	Assignment
3633	Al/Mg-O-H stretching (inter-octahedral)
3450	H-O-H stretching vibration
918	Al-OH bending deformation (Al-O bending vibrations)
1119	Si-O stretching vibrations or (Si-O bending vibration)
1689	OH deformation vibration of water (bending vibration of groups in adsorbed water molecules)

Table 2: Major FTIR Assignments of natural and activated Ca-Bentonite clay samples

Bentonite	Wave length(cm ⁻¹)	Assignment
NCB	3633	Al-O-H stretching (inter-octahedral)
CaB ₂	3640	Al-O-Ca
	1331	NO ₂ ⁻
	1354	NO ₃ ⁻
CaB ₃	3644	Al-O-Ca
ZnB ₂	3644	Al-O-Zn
ZnB ₃	3644	Al-O-Zn
KB ₄₀₀	3640	Al-O-K
KB ₇₀₀	3644	Al-O-K

The activation of NCB with Ca(NO₃)₂ affected the structure of the bentonite by changing the intensity of Al-O-H stretching group and absorption band wavelength from 3633cm⁻¹ to 3640cm⁻¹ and 3644cm⁻¹ for CaB₂ and CaB₃ respectively. This indicates the presence of a new functional group Al-O-Ca and is in line with previous reports by Soetaredjo *et al.*, 2011 and Ali *et al.*, 2016. A medium peak at 1354cm⁻¹ indicates the presence of NO₃⁻ ion and the peak at 1331cm⁻¹ shows the presence of NO₂⁻ ion (Urresta-

Aragon *et al.*, 2012) for CaB₂ spectrum only. Thus, implying that 2 hours calcination time used for CaB₂ did not completely decompose the Ca (NO₃)₂ to CaO. Conversely, the NO₃⁻ ion peak and NO₂⁻ ion peak did not appear in the spectrum for CaB₃, which means that complete decomposition of Ca(NO₃)₂ to CaO at 3 hours calcination was achieved.

Similarly, activation of NCB with Zn(NO₃)₂ affected the structure of the bentonite by changing the intensity

of Al-O-H stretching group and absorption band wavelength from 3633cm⁻¹ to 3644cm⁻¹ and 3644cm⁻¹ for ZnB₂ and ZnB₃ respectively. This indicates the presence of a new functional group Al-O-Zn and corresponds with previous reports by Soetaredjo *et al.*, 2011; Ali *et al.*, 2016. The absence of NO₃⁻ and NO₂⁻ peaks in both spectra shows that the calcination temperatures of 650°C at 2hours and 3hours respectively resulted to the complete decomposition of Zn(NO₃)₂ to ZnO. (Urresta-Aragon *et al.*, 2012).

For the activation of NCB with KOH, the intensity of Al-O-H stretching group increased moderately at calcination temperature of 400°C and greatly at calcination temperature of 700°C. Also, a shift of absorption band of this group from 3633cm⁻¹ to higher wave lengths of 3640cm⁻¹ and 3644cm⁻¹ for calcination temperatures of 400°C and 700°C respectively, indicates the presence of a new functional group (Al-O-K group) in the catalysts being investigated.

Table 3: Chemical Composition of Natural and Impregnated Bentonite Samples

	CaO	SiO	Fe ₂ O ₃	SO ₃	Al ₂ O ₃	K ₂ O	SrO	TiO ₂	Cl	P ₂ O ₅	ZnO
NCB	76.71	11.27	4.15	3.06	2.84	0.75	0.63	0.28	0.09	0.06	BDL
CaB ₂	77.87	9.64	3.95	2.51	2.61	0.62	0.61	0.28	0.11	BDL	BDL
CaB ₃	80.05	9.71	3.65	1.97	2.56	0.56	0.53	0.26	0.11	BDL	BDL
ZnB ₂	70.91	8.61	3.77	2.20	3.43	0.63	0.57	0.27	0.09	BDL	8.55
ZnB ₃	70.69	9.11	3.74	2.16	3.59	0.49	0.55	0.22	0.10	BDL	8.33
KB ₄₀₀	64.52	8.84	3.39	1.03	1.82	19.49	0.53	0.19	0.09	BDL	BDL
KB ₇₀₀	67.56	5.66	3.53	1.54	1.07	19.56	0.54	0.29	0.12	BDL	BDL

BDL – Below Detection Limit

Table 3 shows the XRF results (chemical compositions) of NCB, CaB₂, CaB₃, ZnB₂, ZnB₃, KB₄₀₀, & KB₇₀₀. From results in Table 3, the calcium oxide in CaB₃ is higher than that of CaB₂, thus, this XRF result confirms the FTIR result, that calcination at 650°C for 3 hours decomposed Ca(NO₃)₂ completely to CaO when compared to calcination at 650°C for 2 hours. On the other hand, ZnB₂ and ZnB₃ samples contain ZnO unlike the raw bentonite and other impregnated bentonite samples showing that these bentonites were successfully impregnated with Zn(NO₃)₂ and the calcination decomposed nitrates to oxides. However, calcination at 650°C for 2 hours gave higher values suggesting that complete decomposition to zinc oxide was achieved within 2 hours. The K₂O compositions of KB₄₀₀ (19.49) and KB₇₀₀ (19.56) are higher than that of natural Ca- bentonite showing that impregnation occurred successfully. However, KB₇₀₀ has a slightly higher composition compared to KB₄₀₀. Scanning electron microscopy (SEM) identifies the surface morphology of a given sample. The SEM analysis of NCB, CaB₂, CaB₃, ZnB₂, ZnB₃, KB₄₀₀, & KB₇₀₀ gave images for the natural and impregnated catalysts, as shown in Figures 1 - 7.

The morphology of the natural Ca- bentonite catalyst, NCB, in Figure 1, showed the surface is heterogenous as it has large, medium and small grains with pores.

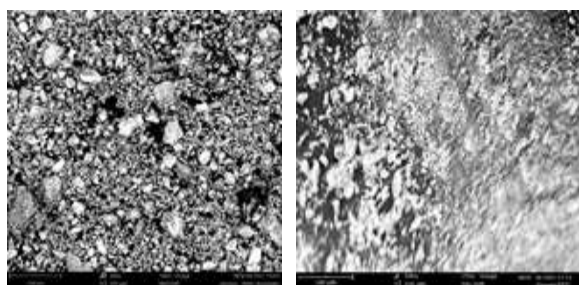


Fig. 1: Morphology of NCB

Fig 2: Morphology of CaB₂

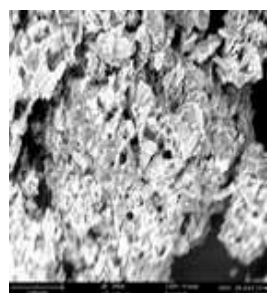


Fig 3: Morphology of CaB₃

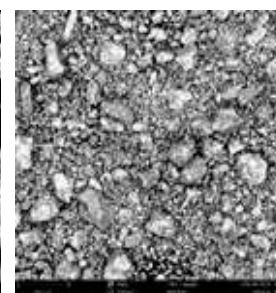


Fig 4: Morphology of ZnB₂

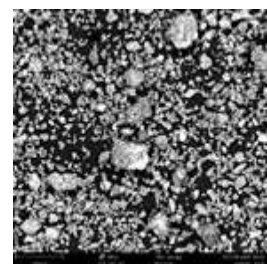


Fig 5: Morphology of ZnB₃

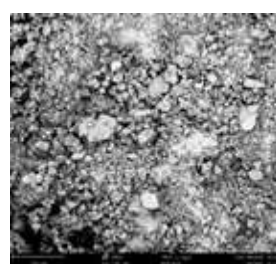


Fig 6: Morphology of KB₄₀₀

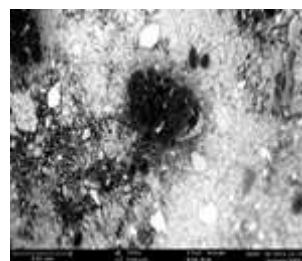


Fig 7: Morphology of KB₇₀₀

The SEM images of CaB₂ and CaB₃ in Figures 2 and 3 respectively, appear very similar, as the natural structure of NCB was destroyed by the impregnation and calcination to give homogenous particles (Naswir et al., 2013, Farias *et al.*, 2015) with smoothed surfaces (Sharma *et al.*, 2021). Thus, they have increased surface area which gives them room for good reactivity. However, CaB₃ morphology has more pores from its smoothed sponge-like surface structure compared to CaB₂ morphology, and this gives CaB₃ catalyst room for more reactivity and thus, more viscosity reduction effect, as given in Table 4, compared to CaB₂ catalyst.

From the morphology of ZnB₂ in Figure 4, it can be seen that the natural structure of NCB was not destroyed by impregnation and calcination. ZnB₂ morphology shows more of large particles compared to NCB and ZnB₃ morphologies and this seems to explain its lower viscosity reduction as given in Table 4. The particles are also more compact than the particles of NCB and ZnB₃. The SEM image of ZnB₃ in Figure 5 shows that impregnation and calcination did not destroy the natural structure of NCB. The reduction of particle size from ZnB₂ to ZnB₃ as shown from Figures 4 to 5 occurred as a result of longer calcination time, which is 2hours to 3hours. Again, ZnB₃ morphology showed fewer large particles and more of small particles compared to NCB and ZnB₂. The particles are also clearly not compact. Thus, the SEM images seem to explain the higher viscosity reduction effect, given in Table 4, of ZnB₃ compared to NCB and ZnB₂. The natural structure of NCB grains can still be observed in the SEM image of KB₄₀₀ in Figure 6. However, it seems to have a more compact surface than the SEM image of NCB and thus, the surface area is decreased. The more compact surface could be caused by the aggregation of clay on K₂O on impregnation and calcination at 400°C and possibly, explains the much lower viscosity reduction effect of KB₄₀₀, given in Table 4, at mild reaction conditions. Again, although the XRF results showed relatively good content of K₂O in KB₄₀₀ compared to KB₇₀₀, the FTIR spectra of KB₄₀₀, showed much lower intensity and a lower wavelength. The K₂O seems to be more in the bentonite structure than on the surface, resulting to much lower viscosity reduction effect. An interesting occurrence can be observed from the surface characteristics of KB₇₀₀ in Figure 7. The natural structure of NCB grains has not only transformed to a smooth surface with pores, and thus, very large surface area but also the impregnated K₂O seems to interestingly form nano rod like (Saeed *et al.*, 2021, Sharma et al., 2021) structures on the smooth surface of KB₇₀₀, thus, giving the catalyst a very high reactivity

and thus, high viscosity reduction effect, as given in Table 4, at mild reaction conditions. Helwani et al., 2021, mentioned that calcination of KOH on cement clinker catalyst at 700°C results to high transesterification reactivity. The kinematic viscosity of transesterified castor oil using KB₇₀₀ heterogeneous catalyst (19.9mm/s²) at mild reaction conditions, is comparable to the results obtained by Silva, 2009, using homogenous KOH catalyst (20.3mm/s²) and Keera *et al.*, 2018, using homogenous KOH catalyst (15.4mm/s²).

Table 4: Catalysts Viscosity Reduction Performance

Catalyst	Kinematic Viscosity at 40°C (mm/s ²)	Viscosity reduction (%)
Castor Oil	189.482*	-
NCB	69.050	63.56%
CaB ₂	57.791	69.50%
CaB ₃	42.841	77.39%
ZnB ₂	107.576	43.23%
ZnB ₃	61.188	67.71%
KB ₄₀₀	135.401	28.54%
KB ₇₀₀	19.907	89.49%

*This served as a reference standard

Results of kinematic viscosity of the various transesterified castor oil samples using NCB, CaB₂, CaB₃, ZnB₂, ZnB₃, KB₄₀₀, and KB₇₀₀ catalysts are presented in Table 4. The kinematic viscosity of the end product served as indicator for the effectiveness of the catalyst in completing the ester conversion process (Enweremadu and Alamu, 2010; Brito et al., 2007; Felizardo *et al.*, 2006; Al-Widyan and Al-Shyoukh, 2002; Canakci and van Gerpen, 1999). From the results obtained, there was a significant observed reduction in the kinematic viscosity for both the natural and activated bentonite clays used for transesterification. KB₇₀₀ catalyst gave the highest viscosity reduction of 89.49%, followed by CaB₃ with 77.39%, followed by CaB₂ with 69.50%, followed by ZnB₃ with 67.71%, followed by NCB with 63.56%, then followed by ZnB₂ with 43.23% and lastly by KB₄₀₀ with 28.54%. The activated bentonite catalysts, KB₇₀₀, CaB₃, and CaB₂, whose natural bentonite structures were destroyed by the calcination process to give a smooth and homogenous surface gave the most viscosity reduction (%), an indication that the activation of the bentonite clays proved useful.

Conclusion: Natural Ca-Bentonite (NCB) from Ebenebe in Anambra State, Nigeria, was successfully impregnated with Ca(NO₃)₂, Zn(NO₃)₂ and KOH, and calcined at various time and/or temperatures to produce six different catalysts (CaB₂, CaB₃, ZnB₂, ZnB₃, KB₄₀₀, and KB₇₀₀). KB₇₀₀ proved to be very good for the purpose of transesterification reaction of refined castor oil at mild reaction conditions. Obtained results showed the descending order of catalyst

viscosity reduction ability to be $KB_{700} > CaB_3 > CaB_2 > ZnB_3 > NCB > ZnB_2 > KB_{400}$.

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