Physico-Chemical Characterizations of Ethiopian Kaolin for Industrial Applications: Case Study WDP Propoxur Formulations

Tadele Assefa Aragaw(✉) and Feleke Kuraz

Faculty of Chemical and Food Engineering, Bahir Dar Institute of Technology,
Bahir Dar University, Bahir Dar, Ethiopia
taaaad82@gmail.com, felek2004@gmail.com

Abstract. This research aims to investigate chemical, physical, mineralogical and optical properties of Ethiopian kaolin with mechanical, wet beneficiation, chemical modification and thermal treatment (calculations) for their appropriateness of industrial application. The effect of beneficiation and calculation methods of treatment on the structure of kaolin was studied using complete chemical analysis (AAS), X-ray diffraction, Fourier transforms infrared spectrometer analysis (FTIR) and Thermal analysis comparatively with the reference kaolin were studied. Complete silicate analysis showed that the Fe_2O_3 and TiO_2 were reduced via beneficiation and beneficiation with surfactant from 1.908% to 0.201% and 0.87% to 0.056%, respectively. Physico-chemical characterizations, thermal analysis effect of calculations results show that improvement to a very high grade with a chemical composition close to that of ideal kaolin. Beneficiation with a surfactant, followed by calcination at 850 °C, showed a further decrease in Fe_2O_3 and TiO_2 impurities. XRD results revealed that all characteristics reflection of kaolin and quartz were showed as major peaks upon calculations and as well as the reference kaolin. The basic properties for industrial grade specification significantly increased after a certain treatment in comparison with reference (imported) kaolin.

Keywords: Kaolin · Calcinations · Beneficiation · Surfactant · WDP propoxur

1 Introduction

Kaolinite is hydrous aluminum silicate member belonging to the dioctahedral1:1 kaolin mineral group. Indeed, kaolinite is the most widespread phase amongst the other kaolin polymorphs, namely halloysite, dickite and nacrite [1].

The theoretical structural formula of kaolin minerals is $Al_2Si_2O_5$ (OH) 4 that means 46.54% SiO_2, 39.50% Al_2O_3 and 13.96% loss on ignition as structurally bonded hydroxyls. Raw kaolin, however, shows some contents of other elements, due to the presence of the cited associated mineral impurities [2, 3]. Kaolinite normally appears as stacked pseudo hexagonal platelets, <2 µm in size, with a common booklet like shape. Each kaolinite layer is considered as a strong dipole, where the siloxane surface is

F. A. Zimale et al. (Eds.): ICAST 2018, LNICST 274, pp. 122–134, 2019.
https://doi.org/10.1007/978-3-030-15357-1_10

hydrophobic and dominated by negative charges, while the aluminum surface exhibits positive charges and is hydrophilic. Thus, the individual layers of kaolinite are strongly bonded by hydrogen and dipolar interactions [4].

Industrial usage of kaolin minerals exhibits physicochemical and mineralogical characteristics which make them very useful in many different applications [5–7]. These characteristics is important for the utilization of kaolinite in a varied field of industrial applications [2, 7]. The applications depend on the physical, chemical, mechanical, and rheological properties of kaolin minerals that act as an active component or as excipient by controlling the efficiency of the dosage forms, and/or improving the drug bioavailability [3, 8–14]. Amorphous calcined kaolin is the water-soluble, more unstable and dissolves more quickly than its crystalline form. Consequently, an amorphizing agent (dispersant) is necessary to help this process and to stabilize amorphous active ingredient in the solid dosage form [15].

The most important physical tests and parameters recommended to qualify kaolin grades as diluents in solid dosage forms include bulk and tapped density of powder, powder fineness, moisture or loss on drying, hardness, friability, disintegration time, dissolution or active ingredient release profile [16, 17].

Currently; Ethiopia imports the China kaolin for Water Dispersible Powder (WDP) propoxur formulation even though there is kaolin deposit here in Ethiopia. This is because Ethiopian kaolin expected to be high in impurity prior to iron compounds as the information gathered from the Ethiopian geological survey. This is the problem not to use for different industrial application including for propoxur WDP formulation as a carrier. To confirm this problem, the raw Ethiopian kaolin has undergone a certain treatment phase and characterization to form the amorphous alumina-silicate kaolin. Thus, investigating physic-chemical characterizations of the domestic kaolin together with different treatment methods (beneficiation, is aimed at removing impurities like feldspar, iron oxide, etc. and thermal (calcination) make it suitable for different industrial application grade is an important issue specifically propoxur WDP formulation.

2 Methodology

2.1 Materials and Chemicals

Kaolin (bombowuha kaolin) used in this study was supplied from Ethiopian petroleum, minerals and biofuel corporation, Addis Ababa. The reference (China) kaolin was supplied from Adamitulu pesticide factory sharing company. All chemicals, reagents, and material that were used are of analytical grade and were purchased from Addis Ababa, Ethiopia.

2.2 Beneficiation

For the separation of impurities from kaolin, produce kaolin products to meet the needs of various industrial applications; wet treatment (beneficiation) process is an important step according to the procedures reported by [18]. Ground kaolin were dispersed in water and surfactants (1% of dried kaolin) were added. The powdered sample was soaked in deionized water for 24 h (one day). The layers were separated by decantation. The fine clay slurry has been dried at 60 °C and sieved about less than 75 µm so as to the fine grits for further analysis and characterization.

2.3 Thermal Treatment (Calcinations)

The beneficiated kaolin was activated through calcination in a muffled furnace from 550–850 °C for 3 h to form reactive state metakaolin. The heating processes derive off water from the mineral kaolin and collapse the material structure, resulting in an amorphous alumina-silicate ($Al_2O_3.2SiO_2$).

2.4 Thermal Analysis

Thermogravimeter (model ATAT2012) was used for the thermal studies. The TG/DSC measurements were carried out from room temperature up to 1000 °C with air atmosphere in a flow rate approximately 15 mL/min.

Instrument temperature was calibrated by heating indium (melting point = 156.6 °C) as a standard at 10, 15 and 20 °C/min [19].

2.5 Mineralogy Study with XRD

Qualitative and quantitative characterizations of the phases and the number of phases that is present in were determined by Rigaku MiniFlex 300/600) with Ni-filtered Cu Ka radiation at different calcinations temperature and as well the reference (China) kaolin having D/teX Ultra 1D silicon strip detector and Standard Sample Holder. Scan axis = 2theta/theta, continuous scanning measurement mode, wave length 1 of 1.54059, with a speed of 0.12 s/step.

2.6 Chemical Analysis

There are different methods accessible for the determinations of elemental analysis techniques for clay minerals (kaolin). In this study notably lithium metaborate fusion, HF attack, gravimetric and AAS analytical methods, from the geological survey of Ethiopia to determine oxide compositions. Digestion of samples with hydrofluoric and nitric acids (total decomposition) and fusion with lithium metaborate at 950 °C was employed [20]. Analytical Procedures were used as the method reported by [21] and elemental concentrations in milligram per liter in the solution have been converted into weight percentage oxide composition as shown in Eq. 1.

$$\text{Elements}(\text{wt\%}) = \frac{\text{mg/l*}(\text{dilution factor}) * (0.01)}{\text{grams of sample}} \tag{1}$$

2.7 Fourier Transformation Infrared (FTIR) Analysis

The FTIR spectra were used for qualitative characterization of surface functional groups of the kaolin. The samples were mixed with KBr and then ground, desorbed and pressed to obtain IR transparent pellets. The percent transmittance was recorded using a PerkinElmer frontier spectrophotometer. The spectra were collected within a scanning range of 400–4000 cm^{-1}.

2.8 Performance Analysis of Formulated WDP Propoxur

Dissolution Rate: The dissolution rate of the propoxur formulations in water medium was done according to USP XXIII paddle method [22]. The stirring speed was 80 rpm and the temperature was maintained ambient. Aliquots (10 ml) were withdrawn at the 0–180 min with 20-min interval and analyzed for the amount of active ingredient dissolved with spectrophotometry at 410 nm using a UV–visible spectrophotometer (PerkinElmer lambda 35) as the Eq. 2.

$$\%\text{propoxur released } = \frac{\text{Amount of propoxur released (mg)} * 100}{\text{dose (mg)}} \tag{2}$$

Where, df = dilution factor; Vd = volume of dilution medium

Dispersion Stability: Dispersion stability was carried out by accurately weighing 0.5 g of the sample, which was added to 200 ml of distilled water and was stirred on an orbital shaker for about 20 min at ambient temperature and allowed to stand in cylinder. 30–180 min with 30 min' interval and residue settled at the bottom was separated by decanting the solution, which was dried on an aluminum foil and weighed, from which percentage yield of complex dispersed in water was calculated, which indicates its dispersion stability [23]. The Dispersion stability is determined by calculating the % yield of dispersion as shown in Eq. 3.

$$\%\text{Yield of dispersion } = \frac{\text{P}}{\text{P0}} * 100 \tag{3}$$

Where P = weight of sample dispersed in water and
P0 = weight of original sample

Wettability: About 2 g of the sample was weighed and pour uniformly and quickly in the beaker containing 100 ml of hard water (342 ppm) and start the stopwatch simultaneously [24]. The time (seconds) was noted when the whole material is completely submerged in the water.

3 Results and Discussions

3.1 Differential Scanning Calorimeter (DSC) Analysis

Ethiopian kaolin, where the samples are taken, is poorly ordered kaolin as the dehydroxylation peak shows 514 °C. The thermal analysis gives information on weight loss; recrystallization, decomposition and phase transformation reveal the thermal behavior of the kaolinite structure [25]. A large release of moisture and volatile matters has seen concurrent with the weight loss and endothermic in the TG/DSC data. In kaolin composition as can be seen in Fig. 1, DSC/TG signatures a strong case for the presence of lattice energy. Information about interactions between mineral components is obtained from DSC value where the thermal effects at temperatures up to 514 °C have clearly observed. Beneficiated kaolin showed two endothermic and exothermic peaks at 309 and 940 °C, and 85 and 514 °C respectively Fig. 1. The DSC curve of kaolin showed a broad characteristic melting endotherm with a maximum at 940 °C. Finally, the recrystallization and transformation of dehydrated substance to mullite, cristobalite, and quartz were observed at the endothermic peaks at 940 °C is characteristic for metakaolin dissociation and formation of spinel. That means a higher degree of structural order produces a higher endothermic peak while a smaller particle size leads to a lower endothermic peak [26].

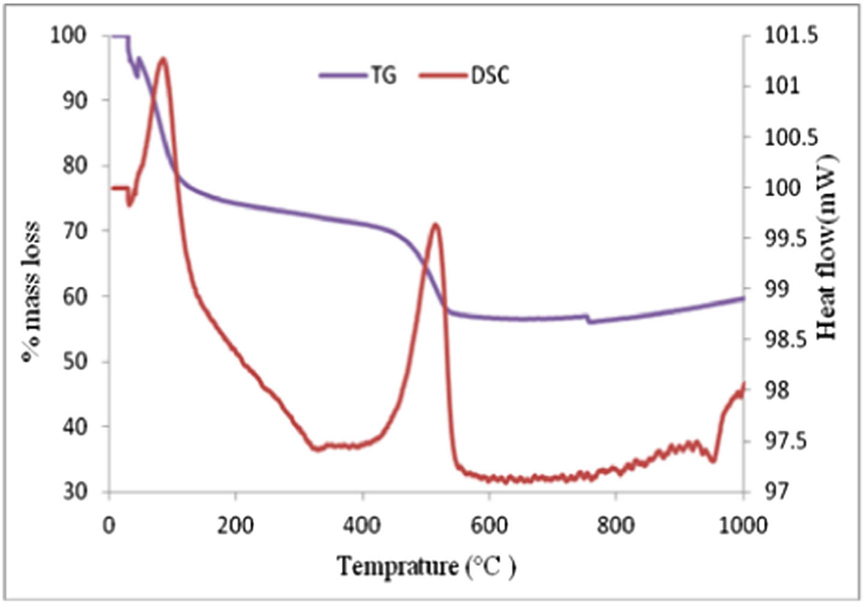

Fig. 1. TG–DSC signals of beneficiated Ethiopian kaolin.

3.2 Mineralogy with XRD

X-ray diffraction (XRD) result reveals structural defects in kaolin because of variability in the peak positions and modulation of their intensities in kaolin XRD patterns. XRD identification of order/disorder is challenging because of overlapping peaks and interferences in kaolin [27, 28]. The degree of kaolinite XRD patterns exhibits broadband between 2theta = 21.03 and 28.12 showed diffuse halo peak which is a characteristic amorphous phase present metakaolins [29]. At those peaks of kaolin was observed in all calcination temperatures with an intensity which increases the rate of calcination. This corresponds with the variation of loss on ignition in metakaolin. The diffraction pattern shown in Fig. 2 give kaolin and quartz as the major minerals in kaolin as well as reference (China) kaolin. The Pattern illustrates the beneficiated kaolin at different calcination temperature and reference kaolin. It was clear that the kaolin is mainly composed of kaolinite mineral as indicated from peaks existing at 2θ values are 21.03, 24.11, 25.133, 27.95, 27.91 and 53.29 has been reported in previous studies [30]. This indicates that the kaolin used in this research is ideal kaolin with anorthic (triclinic) lattice structure. High proportion of quartz mineral was detected from peaks existing at 2θ values 26.88 both in the reference and domestic kaolin and small proportions 39.92 and 50.75 [31] from the reference kaolin but not the domestic kaolin and also it is clearly shown by the patter, as the temperature increases the intensity increases together with amorphousness increase by decreasing the quartz peak at 2θ values 26.88. Therefore, thermally activate kaolin produces structural changes promoting its reactivity. Figure 2 shows a small proportion of titanium oxide (anatase) 2θ values 24.4–25.28 both in reference and Ethiopian kaolin.

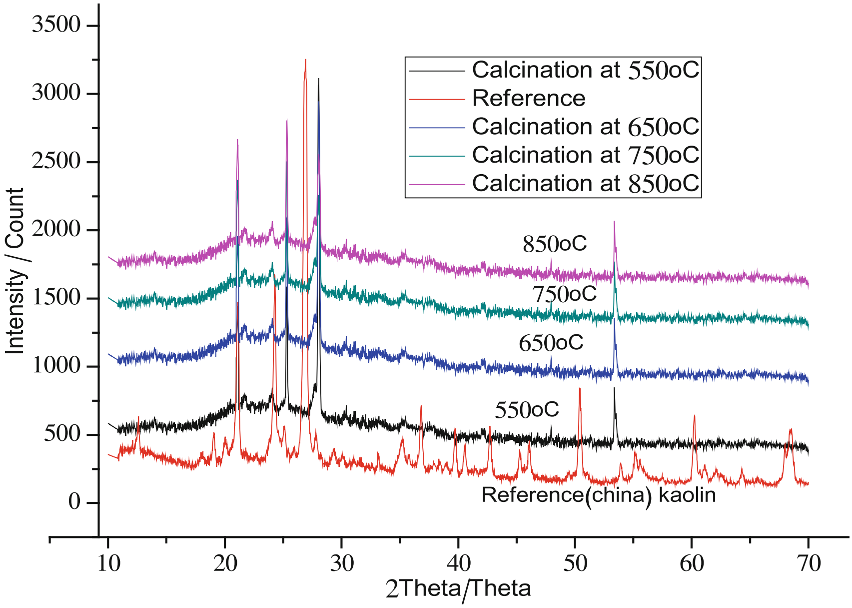

Fig. 2. X-ray diffraction patterns of kaolin at different calcination temprature (550 °C, 650 °C, 750 °C and 850 °C) and reference (china) kaolin

3.3 Chemical Composition

The chemical composition of kaolin was determined by AAS as summarized in Table 1. The common chemical composition analysis detected is the basic components of the kaolin mineral aluminum oxide, silicon oxide and (LoI). It can be seen that the treated kaolin shows better improves the quality of the raw kaolin to almost pure kaolin. It is unluckily that the composition of Fe_2O_3 decreases from 1.908 to 0.201 in Calcined kaolin with a surfactant which is even lower than the china kaolin. This made known a slight decrease in TiO_2 and Fe_2O_3 contents in the kaolin with a simple calcination temperature but further reduction in both TiO_2 and Fe_2O_3 was obtained after beneficiation with a surfactant. It can be seen that anatase (titanium oxide) is concentrated in the fine end of the kaolin particle size distribution [32]. Characteristically, kaolin with less than one percent of iron oxide is considered to be the best quality for a variety of applications. Thus, most important is the increase of the Al_2O_3 content from 32.71 to 39.80% and the SiO_2 increase from 44.8 to 51.37% with drops of LOI from 15.9 to 1.9 due to simple moisture removal and hydroxyl removal of kaolin to produce metakaolin, which is the most active form of kaolin. The major oxide composition analysis tells us a substantial reduction from the kaolin to beneficiation with a surfactant. This confirms that the chemical composition of the treated kaolin with different treatment mechanisms having 51.37% SiO_2 and 39.80% Al_2O_3 which is high-quality grade kaolin for industrial application [33].

Table 1. Chemical composition of raw, reference (china), beneficiated, Beneficiated with surfactant and calcined (850 °C) kaolin

SAMPLES	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	K_2O	Na_2O	TiO_2	LOI
Raw	44.8	32.71	0.434	1.908	1.51	0.79	0.019	0.87	**15.9**
Metakaolin without beneficiation	46.4	37.1	0.289	0.983	1.12	0.427	0.038	0.67	**2.03**
Calcined with beneficiation	47.48	38.80	0.325	0.462	0.27	0.544	0.050	0.52	**2.01**
Calcined and beneficiation with surfactant	51.37	39.80	0.325	0.201	0.17	0.104	0.050	0.056	**1.90**
Reference (China) kaolin	46.6	38.8	0.06	0.51	0.08	0.03	0.12	0.48	**1.93**

3.4 FTIR Analysis

As can be seen from the spectra Fig. 3, the bands placed between 3702.17 cm^{-1} regions corresponds to Si-OH stretching vibration from the raw and reference kaolin and the 3628.54 cm^{-1} bands correspond to the inner layer OH (Al-O-H) stretching the raw and reference kaolin obtained by [28, 34]. But no any peak at those of the calcined kaolin. From this, it can be deduced that there is no OH from the calcined kaolin. The absorption band at 1107 cm^{-1} is assigned to Si-O in-plane stretching vibration. The bands placed at 1080.21 cm^{-1} regions correspond to skeleton Si-O-Si in-plane stretching vibration. The frequency vibration 910 cm^{-1} indicated as OH deformation linked to cationic iron and aluminum. The FTIR spectra depict the predominance of kaolin mineral in the studied sample and did not show any peak for impurity such as smectite. The spectral region between 803.89 cm^{-1} is very sensitive against the crystalline and purity of the kaolin mineral. AL-OH ("gibbsite-like") layer shows at 785.68 cm^{-1} [35]. Si-O-Al bending vibration and Si-O bending vibration shows at 564.78 and 463.8 cm^{-1} respectively. The bending vibrations of water molecules adsorbed to kaolin surface (hygroscopic moisture) are responsible for the bands at 1660 cm^{-1}. As can be seen from Fig. 3, the raw kaolin clearly shows a sharp peak at those bands but the calcined and reference kaolin is an insignificant exhibit. Calcined kaolin has enhanced percent transmittance than the raw kaolin also higher than the reference kaolin. Behind to this is, the structure of kaolin breaks down and the iron-containing minerals are converted to oxides compositions that pass on color with low brightness to the product.

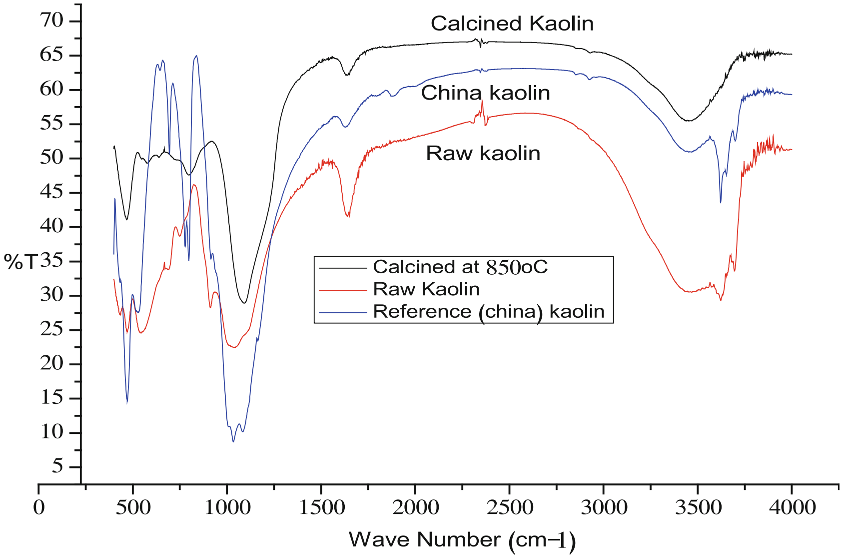

Fig. 3. FTIR- spectra of kaolin (Raw, Calcined and reference (China) kaolin)

3.5 Performance Analysis of Formulated WDP Propoxur

Dissolution Rate: The dissolution profiles of the formulated WDP propoxur can be observed that with raw kaolin, calcined kaolin, beneficiated kaolin and beneficiated kaolin with surfactant have shown Fig. 4 us in increasing the solubility of the propoxur technical ingredient. It is seen that the formulation with surfactant gives higher Dissolution of formulated product with kaolin have been observed that the formulations with raw, calcined, beneficiated and beneficiated with surfactant increase its solubility of propoxur technical. As can be seen the formulated WDP propoxur with beneficiated kaolin surfactant gives a higher percentage of the dissolved active ingredient as compared to that of raw, calcined and simple beneficiated kaolin. This is due to the fact that interaction between the chemical modifiers (surfactant) mixed together on the kaolin surface. Dissolutions were compared to modified and without modified kaolin. The dissolution percentage was lower with formulated WDP propoxur in raw kaolin as compared to that of beneficiated kaolin, calcined kaolin and beneficiated kaolin with a surfactant. This excellent percent dissolution is due to the role of surfactant which can form suspensions of the propoxur technical. It is observed that active ingredient showed no significant change in UV absorbance after that room temperature. The values ranged from 65.8% to 95.96% for solutions at room temperature in raw and treated kaolin. This insured solution stability of active propoxur during the period of dissolution testing.

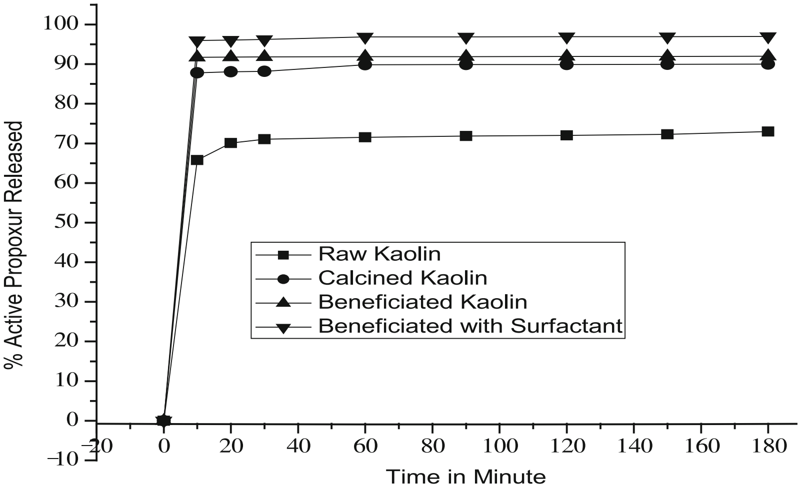

Fig. 4. Dissolution profile studies on formulations of WDP propoxur for raw, calcined, beneficiated and of beneficiated with surfactant.

Dispersion Stability: The dispersion stability expressed as the constancy of the number of particles per unit volume, point out the confrontation to settling and coagulation. In uneven dispersions, the concentrations of particles in the upper dispersion layers decrease because of settling and/or flocculation. As can be seen, Ethiopian raw kaolin has low dispersion stability but the beneficiated with surfactant have high dispersion ability [36].

Dispersion stability depends on the surface area, the particle size distribution, and the solubility of the kaolin mineral in the dispersion medium that is water. If dispersion is stabilized to hinder flocculation and the particle size remains in the colloidal state, below about 0.3 mm, the Brownian motion will prevent settling of particles [36]. The colliding particles become attached to each other unless their surface is modified or some measure has been taken to overcome the attraction between particles. One of the most important functions of a dispersant/surfactant chemical modifier is to hinder the approach of the particles to the close distance where attractive forces dominate and stabilize the dispersion against formulations of WDP propoxur flocculation.

As it can be seen from Fig. 5, the dispersion stability of formulated product with calcined kaolin, beneficiated kaolin and of beneficiated kaolin with surfactant increased sequentially when compared to that of raw Ethiopian kaolin, which indicated that the treatment of kaolin with thermal wet and chemical not only affects the solubility but also the dispersion stability. It can be interpreted from Fig. 5 that the formulated product with beneficiated kaolin and with surfactant show much better dispersion stability that can be attributed to the presence of surfactant molecules in the kaolin structure have the power to form a suspension. Also, the dispersion stability of formulation simple beneficiated with water is slightly comparable to that of the formulation of beneficiated with a surfactant, while that of formulation calcined indicates that the dispersion stability of the formulation which is lower. But have moderate dispersion stability than the raw kaolin.

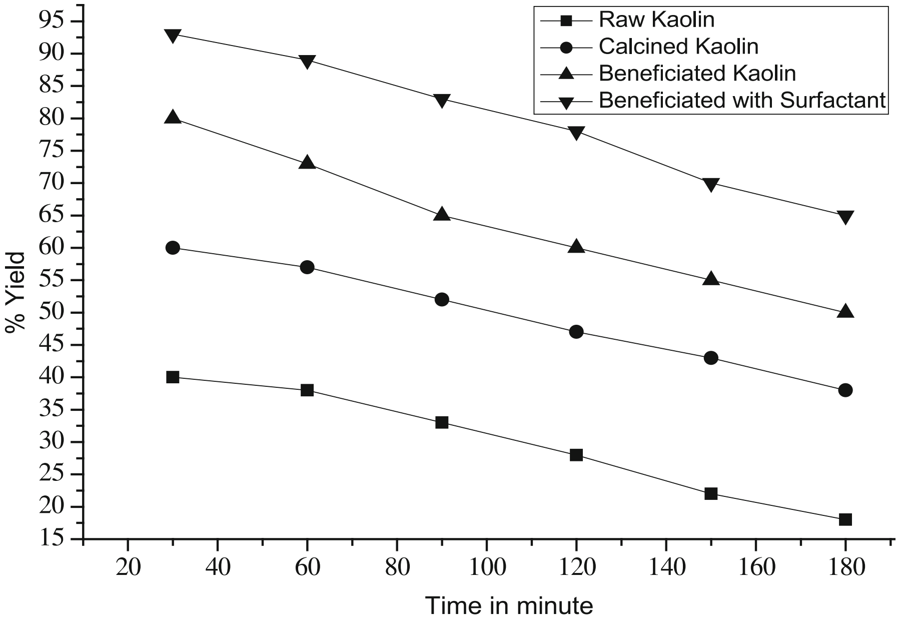

Fig. 5. Dispersion studies on formulations of WDP propoxur for raw, calcined, beneficiated and of beneficiated with surfactant

Wettability: The wetting property studied is immersion wetting. From the results (Table 2) of the wettability test, it is quite evident that the wetting time for formulation of beneficiated with surfactant is lower as compared to that of calcined and beneficiated, which is much lower when compared to raw kaolin, and had resulted due to the decrease in interfacial tension at the solid-liquid interface by the adsorption of the chemical modifier (surfactant) mix together on the kaolin surface causing an enhancement of the wetting property.

Table 2. Wetting time of formulation on raw, calcined, beneficiated and of beneficiated with surfactant kaolin

Formulation	Wetting time (second)
Raw	34.2
Calcined without beneficiation	13.5
Beneficiated with water	10.1
Of beneficiated with surfactant	6.87

Further, it can be made clear that surfactant molecule adsorbed on the surface by Van der Waals attraction get used to the hydrophilic group towards the aqueous solution, thus increasing the hydrophilicity of the absorbent particles and depiction them more wettable with an aqueous solution [37].

4 Conclusion

Ethiopian specifically bombowuha kaolin was successfully modified using beneficiation, chemical modification, and Thermal treatments. The calcination temperature has an ability to increase the brightness and decrease the color values giving an improvement in optical properties. In the present research, a successful beneficiation of Ethiopian kaolin was achieved through a mechanical process of particle separation based on a wet beneficiation method. The beneficiation of the kaolin improves it to a suitable grade for industrial materials. It is demonstrated that chemical modification (with surfactant) can improve the quality of the kaolin further. The characteristics of Ethiopian kaolin clays from western region deposit of the bombowuha led to the following conclusions: the calcination treatment can be adequate starting from 500 °C for the thermal treatments to produce metakaolin as can be seen from TG/DSC analysis. The low weight loss from the thermal analysis conforms as no more organic matter and is poorly ordered kaolin which is suitable to use for the different industrial application.

The china clays (reference kaolin) and Ethiopian kaolin are highly kaolinitic and have same in physic-chemical and mineralogical properties. Treated Ethiopian kaolin (with mechanical and thermal treatment) is found to have properties closer to that of chain kaolin even by far it is better compared to the reference kaolin based on their particle property and pH value. Impurities in raw Ethiopian kaolin are much higher than the chain kaolin clay and it is the 'iron-containing titania' which impacts the optical, chemical and mineralogical properties. However, the treatment processes (beneficiation and calcination) reduces the iron percentage to a lower level than that the reference kaolin. These titania particles with iron in their structure influence the white shade of clay and make it inferior to the domestic kaolin. Calcination first decreases (up to 700 °C) the brightness and whiteness and increases (up to 1200 °C) giving an overall improvement in optical properties. In the end, it can be concluded that treated Ethiopian kaolin (calcined, beneficiated and with surfactant) significantly increased the kaolin high-grade quality for industrial application.

Acknowledgement. We sincere gratefulness goes to Bahir Dar Institute of Technology for their financial support to do this research. Also, my deep gratitude goes to different company and university, Ethiopian geological survey, Ethiopian petroleum and Biofuel Corporation, Adamitulu pesticide factory and Addis Ababa University. We would like to thank the Faculty of Chemical and Food Engineering, Bahir Dar Institute of Technology staffs for their help and accessing all materials for successful completion.

References

1. Weaver, C.E., Pollard, L.D.: The Chemistry of Clay Minerals, vol. 15, 1st edn, pp. 52–59. Elsevier, Amsterdam (1973)
2. Kogel, J.E., Trivedi, N.C., Barker, J.M.: Industrial Minerals and Rocks: Commodities, Markets and Uses Society for Mining. Metallurgy, and Exploration, Inc, Littleton (2006)
3. Williams, L.B.: Geomimicry: harnessing the antibacterial action of clays. Clay Miner. **52**, 1–24 (2017)
4. Chun-Hui, Z., John, K.: Clays, and clay minerals: geology, properties and uses. Appl. Clay Sci. **74**, 58–65 (2013)
5. Peter, A.C.: Industrial Minerals and Their Uses: A Handbook and Formulary, pp. 125–136. William Andrew, Norwich (1996)
6. Murray, H.H.: Applied Clay Mineralogy, 1st edn. Elsevier, Amsterdam (2007)
7. Chatterjee, K.K.: Uses of Industrial Minerals, Rocks and Freshwater. Nova Science Publishers Inc, New York (2009)
8. Carretero, M.I., Gomes, C.S.F., Tateo, F.: Clays, drugs, and human health. In: (Eds.) 5, Handbook of Clay Science. Elsevier, Amsterdam, pp. 711–764 (2013)
9. Zhu, H., Njuguna, J.: Nanolayered silicates/clay minerals: uses and effects on health: In: Health and Environmental Safety of Nanomaterials. Woodhead Publishing Ltd., Elsevier, Amsterdam, pp. 133–146 (2014)
10. Ghadiri, M., Chrzanowskiab, W., Rohanizadeh, R.: Biomedical applications of cationic clay miner. RSC Adv. **7**, 29467–29481 (2015)
11. Khurana, I.S., Kaur, S., Kaur, H., Khurana, R.K.: Multifaceted role of clay minerals in pharmaceuticals. Future Sci. OA **1**, 3 (2015). https://doi.org/10.4155/fso.15.6
12. Kim, M.H., Choi, G., Elzatahry, A., Vinu, A., Choy, Y.B., Choy, J.: Review of clay drug hybrid material for biomedical applications: administration routes. Clays Clay Miner. **64**(2), 115–130 (2016)
13. Yuan, P., Thill, A., Bergaya, F.: Nanosized Tubular Clay Minerals Halloysite and Imogolite, Developments in Clay Science, vol. 7, 1st edn, pp. 203–215. Elsevier Ltd., Amsterdam (2016)
14. Rautureau, M., Figueiredo Gomes, C.S., Liewig, N., Katouzian-Safadi, M.: Clays and Health. Springer, Cham (2017). https://doi.org/10.1007/978-3-319-42884-0
15. Conley, R.F.: Practical Dispersion: A Guide to Understanding and Formulating Slurries, 1st edn, pp. 78–82. VCH Publishers, New York (1996)
16. Mathur, N., Kumar, R., Tiwari, K., Singh, S., Fatima, N.: Evaluation of quality control parameters on various brands of paracetamol tablet formulation. World J. Pharm. Pharm. Sci. **4**(7), 976–984 (2015)
17. Shahabuddin, M.D., Al Mamun, A., Tasnu, T., Asaduzzaman, M.: In-process, and finished products quality control tests for pharmaceutical tablets according to Pharmacopoeias. J. Chem. Pharm. Res. **7**(9), 180–185 (2015)

18. Aroke, U.O., El-Nafaty, U.A., Osha, O.A.: Properties and characterization of kaolin clay from Alkaleri, NorthEastern Nigeria. Inte. J. Emerg. Technol. Adv. Eng. **3**, 387–392 (2013)
19. Archer, J.D., Douglas, W.M., Brad, S.: The effects of instrument parameters and sample properties on thermal decomposition: interpreting thermal analysis data from Mars. Planet. Sci. **2**(2), 1–21 (2013). https://doi.org/10.1186/2191-2521-2-2
20. Twyman, R. M. Wet Digestion. Elsevier Ltd. Online (2005)
21. Potts, P.S.: A Handbook of Silicate Rock Analysis, pp. 47–58. Blackie and Sons Ltd., Glasgow (1992)
22. King, R.E.: Tablets, capsules and pills. In: Oslo, A. (ed.) Remington's Pharmaceutical Sciences, pp. 1559–1560. Mack, Philadelphia (1980)
23. Robert, J.P., Toshiaki, M., Frederick, M.F.: The dispersibility and stability of carbon black in media of low dielectric constant, Electrostatic and steric contributions to colloidal stability. Colloids Surf. **7**(3), 183–207 (1983)
24. IS 6940: Indian Standard Methods of Test for Pesticides and their Formulations (FAD 1): Pesticides and Pesticides first revision (1982)
25. Hongfei, C., Qinfu, L., Jing, Y., Songjiang, M., Ray, L.F.: The thermal behavior of kaolinite intercalation complexes-A review. Thermochim. Acta **545**, 1–13 (2012)
26. Bontle, M., Nadiye-Tabbiruka, M.S.: Chemical and thermal characterization of a clayey material found near gaborone dam. J. Appl. Sci. Environ. Manage. **11**, 77–80 (2007)
27. Hu, P., Yang, H.: Insight into the physicochemical aspects of kaolins with different morphologies. Appl. Clay Sci. **47**, 58–65 (2013)
28. Peter, A.A., Yahaya, M.S., Wan, M.A.W.D.: Kaolinite properties and advances for solid acid and basic catalyst synthesis. RSC Adv **5**, 101127–101147 (2015). https://doi.org/10.1039/c5ra18884a
29. Kenne, D.B.B., Elimbi, A., Cyr, M., Dika, M.J., Tchakoute, H.K.: Effect of the rate of calcination of kaolin on the properties of metakaolin-based geopolymers. J. Asian Ceram. Soc. **3**, 130–138 (2015)
30. Goulash, M., Buhl, J.-C.H.: Geochemical and mineralogical characterization of the Jabal Al-Harad Jordan for its possible utilization. Clay Miner. Mineral. Soc. **45**(4), 281–294 (2010)
31. Liew, Y.M., Kamarudin, H., Mustafa Al Bakri, A.M., Luqman, M., KhairulNizar, I., Ruzaidi, C.M., Heah, C.Y.: Processing and characterization of calcined kaolin cement powder. Constr. Build. Mater. **30**, 794–802 (2012)
32. Bundy, W.M., Murray, H.H.: The effect of aluminum on the surface properties of kaolinite. Clays Clay Miner. **21**(5), 295–302 (1973)
33. https://www.indiamart.com/proddetail/olinpowder7107133755.html. Accessed June 2018
34. Burhan, D., Emin, C.: Investigation of central anatolian clays by FTIR Spectroscopy. Int. J. Nat. Eng. Sci. **3**, 154–161 (2009)
35. Giese, R.F.: Kaolin minerals: structure and stabilities. In: Bailey, S.W. (ed.) Hydrous Phyllosilicates, pp. 29–66. Mineralogical Society of America, Washington DC (1988)
36. Kissa, E.: Stabilization with dispersants and dispersibility. In: Dispersions Characterization, Testing, and Measurement; (surfactant science no, 84), p. 173. Marcel Dekker, New York (1999)
37. Attwood, D., Florence, A.T.: Surfactant System Their Chemistry, Pharmacy, and Biology, EC4P 4EE, pp. 31–37. Chapman and Hall, London (1983)