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EFFECTS OF ACTIVATION ON MOISTURE CONTENT AND BULK DENSITY OF LOCALLY-SOURCED BENTONITE SAMPLES IN NIGERIA

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Abstract

Efficiency of bentonite clay is usually dependent on its properties, especially moisture content and bulk density. The effects of activation on moisture content and bulk density of locally-sourced bentonite samples in Nigeria was investigated in this study. Locally-sourced bentonite was prepared and activated using standard alkaline and acid procedures. The physical characterization of the bentonite clay was determined using standard ASTM methods for apparent density, and moisture content. Obtained results showed that the moisture content of the activated bentonites varied significantly depending on the activation method. Alkali-activated bentonites exhibited significantly lower moisture content (0.3867% - 0.545%) compared to acid-activated bentonites (6.9683%). The control samples, raw bentonite and fuller's earth, had

[redacted]

Keywords;
Bentonite, acid activation, alkaline activation, moisture content, bulk density

intermediate moisture contents (0.61% - 0.62%). Hence, increasing the concentration of activating chemicals generally led to a slight increase in moisture content, with the most pronounced effect observed at the 0.1M concentration. In addition, the concentration of activating chemicals had a moderate effect on bulk density, with higher concentrations leading to slightly lower densities, with the most pronounced effect observed at the 0.1M concentration. Thus, the activation process significantly influenced the bulk density of the bentonites. Specifically, acid-activated bentonites (HCl, H_2SO_4) exhibited lower bulk densities (0.8433 g/cm³ - 0.9492 g/cm³) compared to alkali-activated bentonites (NaOH, KOH) and raw bentonite (1.000 g/cm³), while acetic acid and oxalic acid activated bentonites displayed intermediate values (0.9592 g/cm³ - 1.095 g/cm³). The above observations are recommended for use in the diverse industrial applications of bentonite clay, especially adsorption purposes.

INTRODUCTION

Nigeria is endowed with a wealth of substantial mineral resources that are equitably spread throughout the federation's states. The Geological Survey of Nigeria Agency claims that there are about 34 main mineral resources in Nigeria, spread out over the nation. For over 90 years, Nigeria has been exploring for a number of solid minerals, including tin, niobium, lead, zinc, and gold. However, only tin and niobium output has been ranked on a global scale. Crude oil, natural gas, limestone, salt, rocks, and clays are among the numerous non-metallic mineral resources found in the nation (Obaje, 2009).

Large bentonite clay deposits can be found in Nigeria (Igwilo *et al*., 2020). According to reports, there is a significant bentonite clay deposit in every part of Nigeria. According to Afolabi *et al*. (2017), the confirmed bentonite reserve in Nigeria is reasonably estimated to be over 700 million metric tons. The majority of this reserve is located in Afuze, Edo State, Mid-Western Nigeria, which contains roughly 70–80 million metric tons of bentonite clay (Nweke *et al.*, 2015). Bentonite is a modified volcanic ash that is an off-white montmorillonite clay. Its base-exchange characteristics and the way it absorbs and loses water make it particularly noteworthy. Its structure is sheet-silicate. Sodic bentonite can expand to eighteen times its dry volume and absorb up to ten times its own weight in water. Therefore, it can be used in situations where its colloidal qualities can be used to its advantage (Ezendiokwere *et al*., 2021a).

Foundry, animal feed, drilling mud, absorbent, industrial, and specialist applications are just a few of the many applications for bentonite. Bentonite additives have been utilized to increase the strength of ceramics and bricks, emulsify oils in asphalt and detergents, retain microparticles in papermaking, clarify water, wine, and juice, treat wastewater, deink paper, and reduce airborne stack emissions, as well as gel cosmetics and medications. Additionally, it has been an ingredient in a variety of cleansers, polishes, and sprays (Kelechukwu & Akaranta, 2021).

Bentonite has been referred to be a substance of one thousand (1000) uses for apparent reasons. Its absorption properties, which in turn depend on the chemical makeup of the clay, determine its applications. Around the world, bentonite is frequently utilized as drilling mud in the oil and gas industry. Typically, it is combined with water to create a slurry, which is then injected through the drill bit and drill string. Because of its high viscosity and yield strength, the drilling mud removes drill cuttings and carries them to the surface while also aiding in cooling and lubricating the drill bit. By doing this, the drilled hole is sealed and wall cave-ins are avoided. Additionally, abandoned drilled holes are filled and sealed with bentonite (Shabanzade *et al*., 2019).

The Federal Government of Nigeria in 2003 limited the importation of foreign bentonite clays in order to tap into the enormous amounts of native bentonite that are found in the country's soil (Afolabi *et al*., 2017). Because there are few or no documented instances of using locally obtained bentonite clays for drilling operations, the oil and gas industry still lacks complete confidence in this practice notwithstanding this act. This one action has sparked more investigation into the employment of local clays for drilling mud in the oil and gas sector.

Prior research on the production of drilling fluids using only Nigerian bentonite has demonstrated that these fluids have a significant fluid loss rate. This can be because Nigerian-sourced bentonite is of low quality. Beneficiation is necessary since these clays have poor rheological and fluid loss qualities when they are fresh (Igwilo *et al*., 2020).

Furthermore, there hasn't been much of an improvement in the drilling mud's ability to successfully reduce fluid loss when locally available components and additives like guar gum are used. Enhancing the qualities of native Nigerian bentonite clays to satisfy the American Petroleum Institute (API) standard is becoming more and more necessary as oil and gas exploration moves to deep offshore, increasing the need for bentonite clays. A strong argument for more thorough investigation into enhancing the quality of these clay resources in Nigeria is made by these drawbacks as well as the possible economic ramifications of the nearby bentonite reserve.

One of the possible ways of improving the quality of Nigerian bentonite is activation using various alkaline and acidic solvents (Kelechukwu & Akaranta, 2021). Activation is carried out in an effort to improve the properties of the bentonite samples. The properties of bentonite clay are clearly divided into physical and chemical properties. Chemical properties of bentonite include composition, cation exchange capacity and pH, while physical properties include moisture content, density, texture etc. All things considered, bentonite's physical and chemical characteristics make it a very reactive and absorbent substance that may exchange cations and stabilize structures. Because of these qualities, it can be used in a variety of fields, such as environmental cleanup, drilling, building, agriculture, and cosmetics.

In this study, the moisture content and the bulk density of locally-sourced bentonite in Nigeria is investigated. This would help in assessing the effects of both alkaline and acid activation on the moisture content and bulk densities of Nigerian bentonite samples. Given that moisture content and bulk density are important physical properties of bentonite that strongly affects their rheological properties.

Materials and Methods

Preparation of Bentonite

The bentonite clay used for this study was obtained locally from Anambra State (supplied by Mansid Nig Ltd). The calcium bentonite was not beneficiated but analysed raw. It was washed with distilled water, dried, grinded and sieved through a 200μm sieve. It was then stored in an air tight bag for activation and bleaching.

Acid Activation

Acid activation of the raw bentonite was adopted from Ajemba *et al.* (2013), and Folleto *et al.* (2011). The mass of clay to acid ratio was 0.1g/ml and acid concentration range was 0.1M-5M (0.1, 0.5, 1, 2, 3.5, 5). The activation was carried out using inorganic acids (sulphuric acid and hydrochloric acid) and organic acid used where oxalic acid and acetic acid. The clay and acid were measured into a 250ml flask and heated in a magnetically stirred hot plate at a temperature of 90°C for 2 hours 30 minutes. The resulting slurry was poured into a Buchner funnel to separate acid and clay and washed severally with distilled water. The clay was dried in an oven at 105°C for 4hours and ground to 200 μm size. This was repeated for all the concentrations of the HCl, H_2SO_4, Oxalic acid, Acetic acid, labelled and stored in a tightly fitted container.

Alkaline Activation

The alkali activation was modified from Okwara & Osoka (2006), and Salawudeen *et al*. (2014). The activation was carried out using sodium hydroxide (NaOH) and potassium hydroxide (KOH). The mass of clay to alkali ratio was 0.1g/ml and alkali concentration range was 0.1M-5M (0.1, 0.5, 1, 2, 3.5, 5). The clay and alkali were measured into a 250ml flask and heated in a magnetically stirrered hot plate at a temperature of 90°C for 2hours 30mins. The resulting slurry was poured into a Buchner funnel to separate alkali and clay and thereafter washed severally with distilled water. The clay was later dried in an oven at 105°C for 4hours and ground to 200µm size. This was repeated for all the concentrations of the NaOH and KOH and stored in a tight fitted contained for adsorption studies.

Characterization

The physical characterization of the bentonite clay was determined using standard ASTM methods for apparent density (ASTM2 867-09), and moisture content (ASTM D2867-09).

Apparent Bulk Density

The apparent bulk density (AD) was determined for the raw and activated bentonite clays using ASTM 2854-09, 2g of the bentonite clay was weighed separately in a graduated cylinder of known weight. The side of the cylinder was tapped to maintain a constant clay volume. The volume of the tapped clay was taken and apparent density calculated. The apparent density is given by:

$$AD\left(\frac{g}{cm^3}\right) = \frac{(clay\ mass, g)}{(clay\ volume, cm^3)} \tag{1}$$

Moisture Content

According to ASTM D2867-09, a clean porcelain crucible was dried in an oven at 105°C, and cooled in a desiccator. Then, 2g of the bentonite clay was weighed into a crucible, and dried in an oven at 110°C for 3 hours to attain a constant weight. The percentage moisture was calculated using the following equation:

$$\text{MC (\%)} = \frac{W_{wet} - W_{dry}}{W_{wet}} \times 100 \qquad (2)$$

Where W_{wet} = weight of wet bentonite clay and W_{dry} = weight of dry bentonite clay

Results and Discussion

Effect of Concentration on the Moisture Content of Activated Bentonite

Figure 1 shows the effect of different activating chemicals on the moisture content of activated bentonite clay. From the figure, moisture content for HCl-activated bentonite is 0.545%; H_2SO_4–activated bentonite is 6.9683%; NaOH-activated bentonite is 0.3867%; KOH-activated bentonite is 0.4067%; CH_3COOH-activated bentonite is 0.4033%; $C_2H_2O_4$–activated bentonite is 0.445%; as compared to raw bentonite 0.62%, and fuller's earth 0.61%. The moisture content of the activated bentonites varied significantly depending on the activation method. Alkali-activated bentonites exhibited significantly lower moisture content (0.3867% - 0.545%) compared to acid-activated bentonites (6.9683%). Figure 2 shows the results of the activated bentonite in comparism with the control samples, which are raw bentonite and Fuller's earth. From the figure, raw bentonite and Fuller's earth had intermediate moisture contents (0.61% - 0.62%).

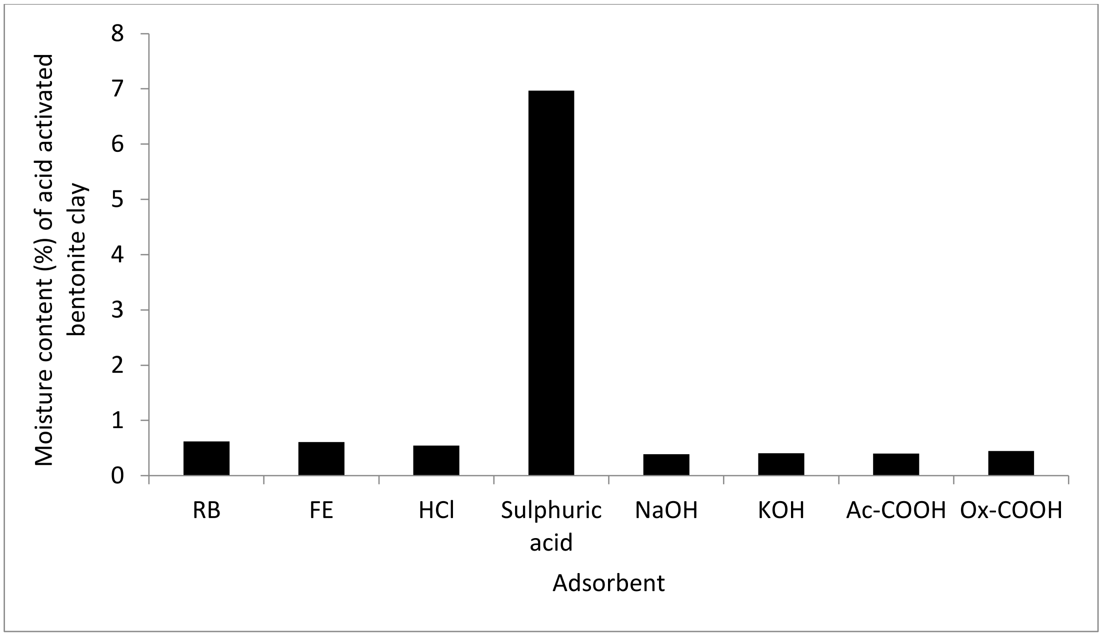

Figure 2: Effect of different activating chemicals on the moisture content of activated bentonite clay

Figure 3 shows the effect of adsorbent concentration on the moisture content of activated bentonite clay. The mean concentration of chemicals affected the moisture content of the bentonites as follows: 0.1M concentration: 1.5783%; 0.5M concentration: 1.615%; 1M concentration: 1.605%; 2M concentration: 1.4633%; 3.5M concentration: 1.45%; 5M concentration: 1.4433%; as compared to the controls: Raw bentonite: 0.62%, and Fuller's earth: 0.61%. Increasing the concentration of activating chemicals generally led to a slight increase in moisture content, with the most pronounced effect observed at the 0.1M concentration. The concentration of activating chemicals had a moderate effect on moisture content, with higher concentrations leading to slightly higher moisture levels. This trend, however, was not consistent across all activation methods.

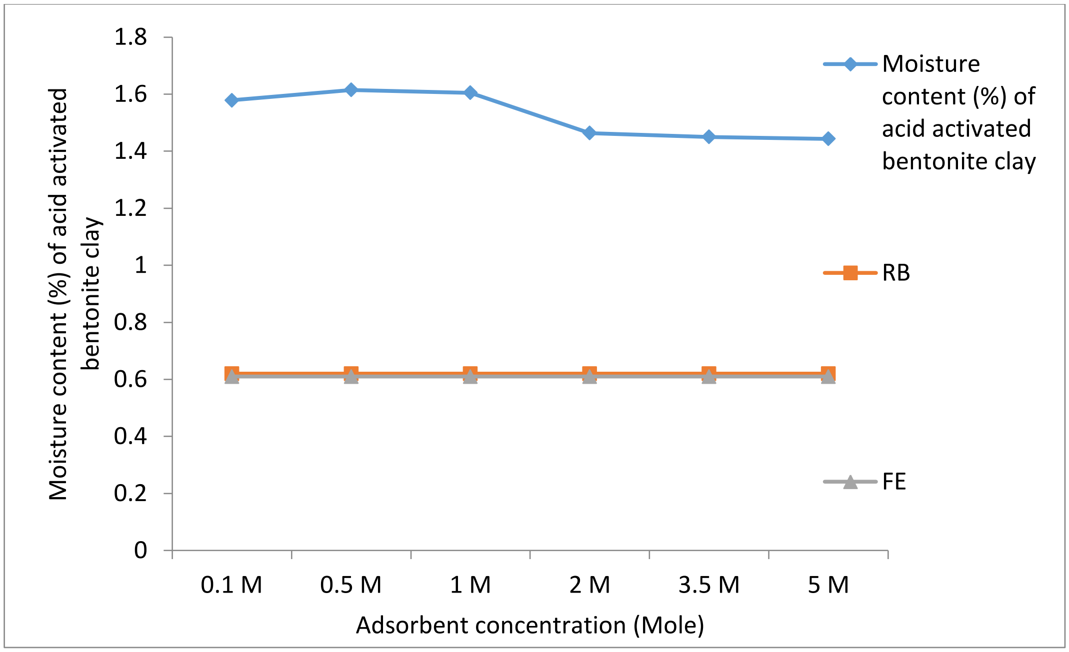

Figure 4: Effect of adsorbent concentration on the moisture content of activated bentonite

The moisture content of the activated bentonites varied significantly depending on the activation method. Alkali-activated bentonites exhibited significantly lower moisture content compared to acid-activated bentonites. Raw bentonite and fuller's earth had intermediate moisture contents. Increasing the concentration of activating chemicals generally led to a slight increase in moisture content, with the most pronounced effect observed at the 0.1M concentration. However, this trend was not consistent across all activation methods. The observed differences in moisture content can be attributed to several factors.

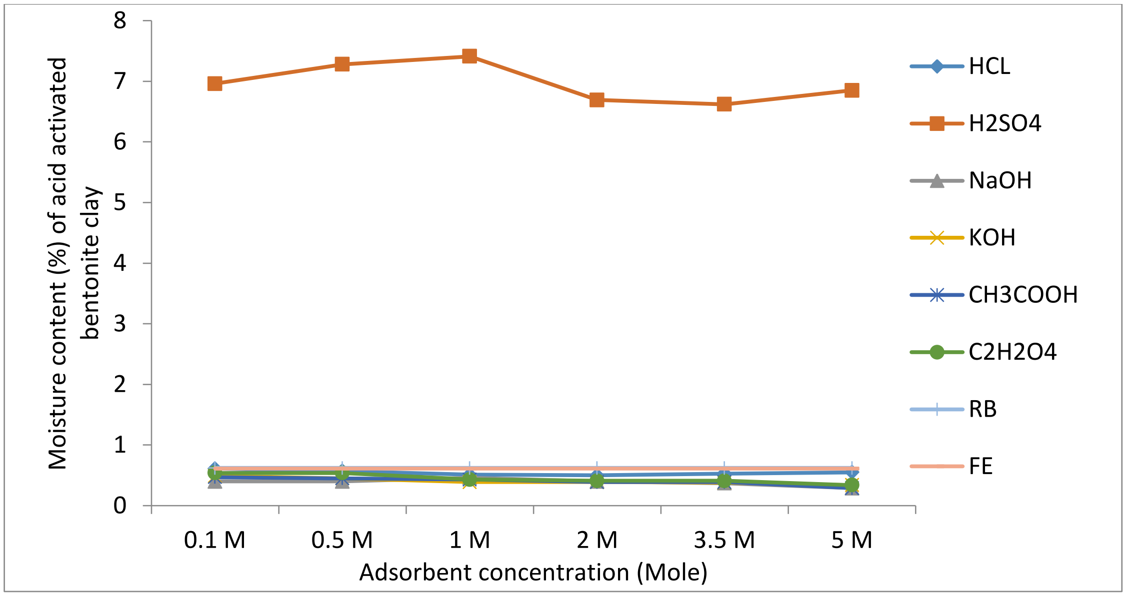

Figure 5: Effect of adsorbent concentration on the moisture content of activated bentonite clay

The activation process can alter the hydrophilicity of the bentonite surface, affecting its ability to retain water (Ezendiokwere *et al.*, 2021b). A more porous structure can increase the surface area available for water adsorption, leading to higher moisture content. The intercalation of activating ions can influence the water retention capacity of the bentonite. Selim and his co-workers (Selim *et al.*, 2020) have iterated that chemical activation of bentonite increases its surface porosity and reduces its bulk density. One of the effects of this is that the open pores helps remove water and other impurities attached to the clay particles.

These physico-chemical reactions modify bentonite morphology as the pores open up and the surface appears as more porous and homogenous. The slight increase in moisture content with increasing concentration of activating chemicals suggests that the activation process becomes more effective at higher concentrations, leading to a more hydrophilic and porous structure. However, the lack of a consistent trend across all activation methods indicates that other factors, such as the nature of the activating chemical and the specific properties of the bentonite, also play a role in determining moisture content.

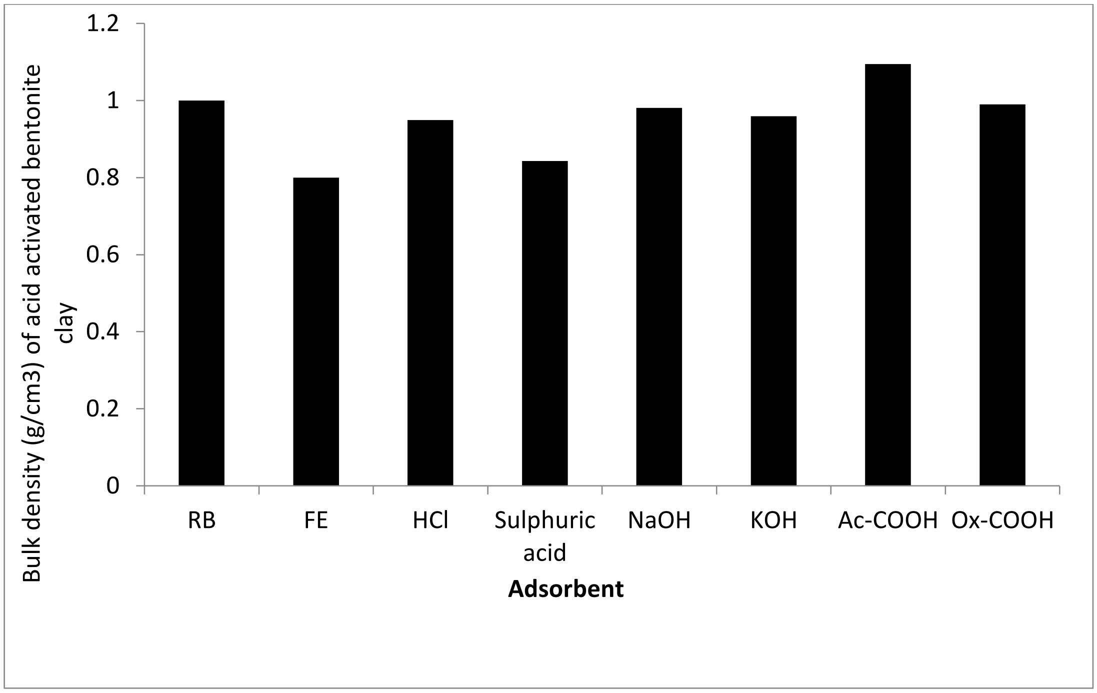

Figure 6: Effect of different activating chemicals on the bulk density of activated bentonite

Effect of Concentration on the Bulk Density of Activated Bentonite

Figure 7 shows the effect of different activating chemicals on the bulk density of activated bentonite. From the figure, the bulk density results based on the activation methods are: HCl-activated bentonite: 0.949167 g/cm³; H_2SO_4-activated bentonite: 0.843333 g/cm³; NaOH-activated bentonite: 0.980833 g/cm³; KOH-activated bentonite: 0.959167 g/cm³; CH_3COOH-activated bentonite: 1.095 g/cm³; $C_2H_2O_4$-activated bentonite: 0.99 g/cm³; as compared to raw bentonite: 1.00 g/cm³, and Fuller's earth: 0.80 g/cm³. From the obtained results, it can be deduced that the activation process significantly influenced the bulk density of the bentonites. Consequently, acid-activated bentonites (HCl, H_2SO_4) exhibited lower bulk densities (0.8433 g/cm³ - 0.9492 g/cm³) compared to alkali-activated bentonites (NaOH, KOH) and raw bentonite (1.000 g/cm³) and Filler's earth (0.80 g/cm³). But acetic acid and oxalic acid activated bentonites displayed intermediate values (0.9592 g/cm³ - 1.095 g/cm³).

Figure 8 shows the effect of adsorbent concentration on the bulk density of activated bentonite clay. From the figure, the mean concentration of chemicals affected the bulk density of the bentonites as follows: 0.1M concentration: 1.0292 g/cm³; 0.5M concentration: 0.9508 g/cm³; 1M concentration: 0.9283 g/cm³; 2M concentration: 0.945 g/cm³; 3.5M concentration: 0.9708 g/cm³; 5M concentration: 0.9933 g/cm³; as compared to the controls: Raw bentonite: 1.00 g/cm³, and Fuller's earth: 0.80 g/cm³. The concentration of activating chemicals had a moderate effect on bulk density, with higher concentrations leading to slightly lower densities. Increasing the concentration of activating chemicals generally led to a slight decrease in bulk density, with the most pronounced effect observed at the 0.1M concentration. This trend, however, was not consistent across all activation methods.

The activation process significantly influenced the bulk density of the bentonites. Acid-activated bentonites (HCl, H_2SO_4) exhibited lower bulk densities (0.8433 g/cm³ - 0.9492 g/cm³) compared to alkali-activated bentonites (NaOH, KOH) and raw bentonite (1.000 g/cm³). Acetic acid and oxalic acid activated bentonites displayed intermediate values (0.9592 g/cm³ - 1.095 g/cm³). Increasing the concentration of activating chemicals generally led to a slight decrease in bulk density, with the most pronounced effect observed at the 0.1M concentration. However, this trend was not consistent across all activation methods. The observed variations in bulk density can be attributed to several factors. Acid activation may lead to increased porosity due to the dissolution of mineral components, resulting in lower bulk density.

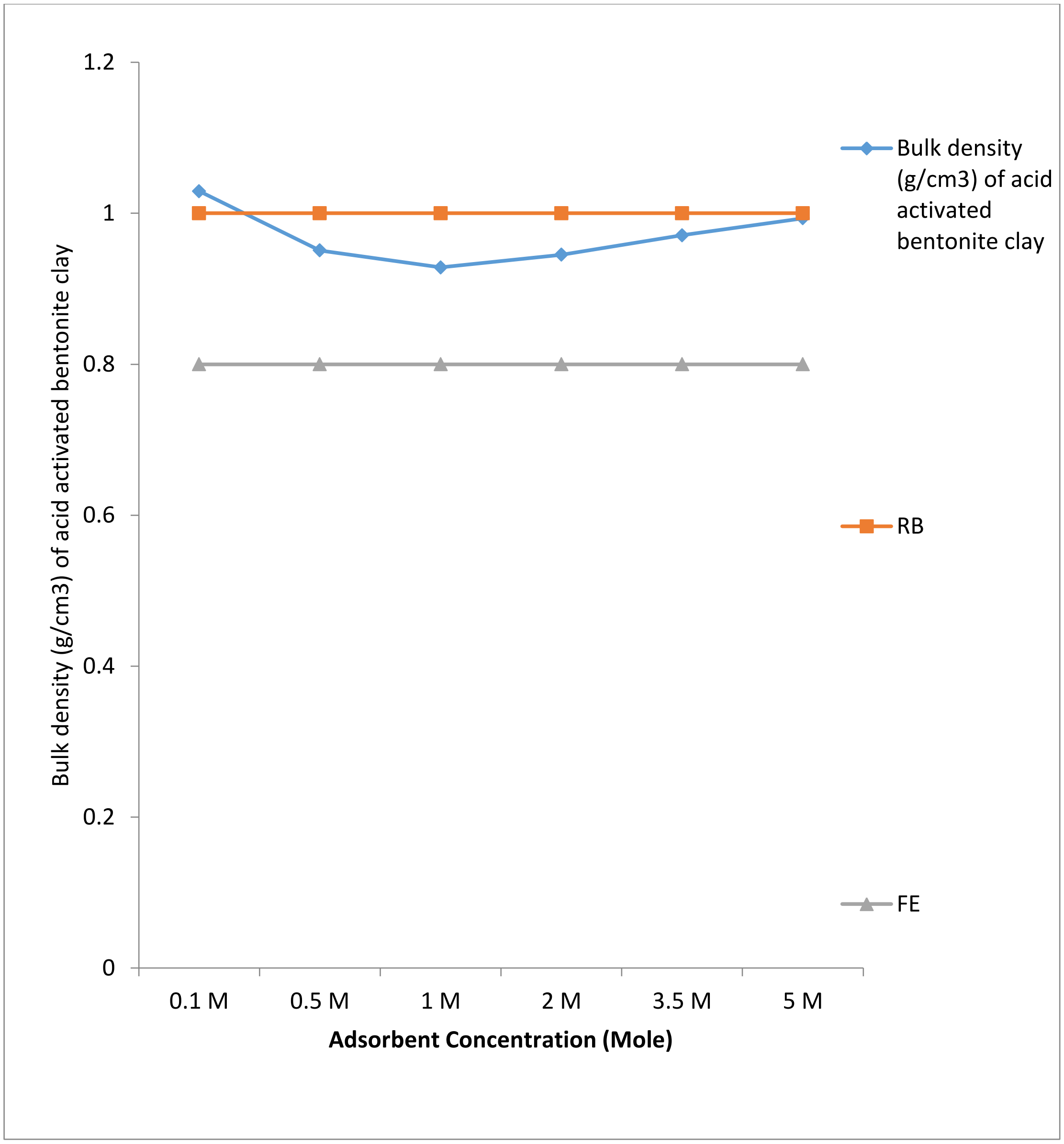

Figure 9: Effect of adsorbent concentration on the bulk density of acid activated bentonite clay

The activation process can alter the particle size distribution of the bentonite, which can influence bulk density. The intercalation of activating ions between the clay layers can affect the packing density and, consequently, bulk density. The slight decrease in bulk density with increasing concentration of activating chemicals suggests that the activation process becomes more efficient at higher concentrations, leading to a more porous structure. However, the lack of a consistent trend across all activation methods indicates that other factors, such as the nature of the activating chemical and the specific properties of the bentonite, also play a role in determining bulk density.

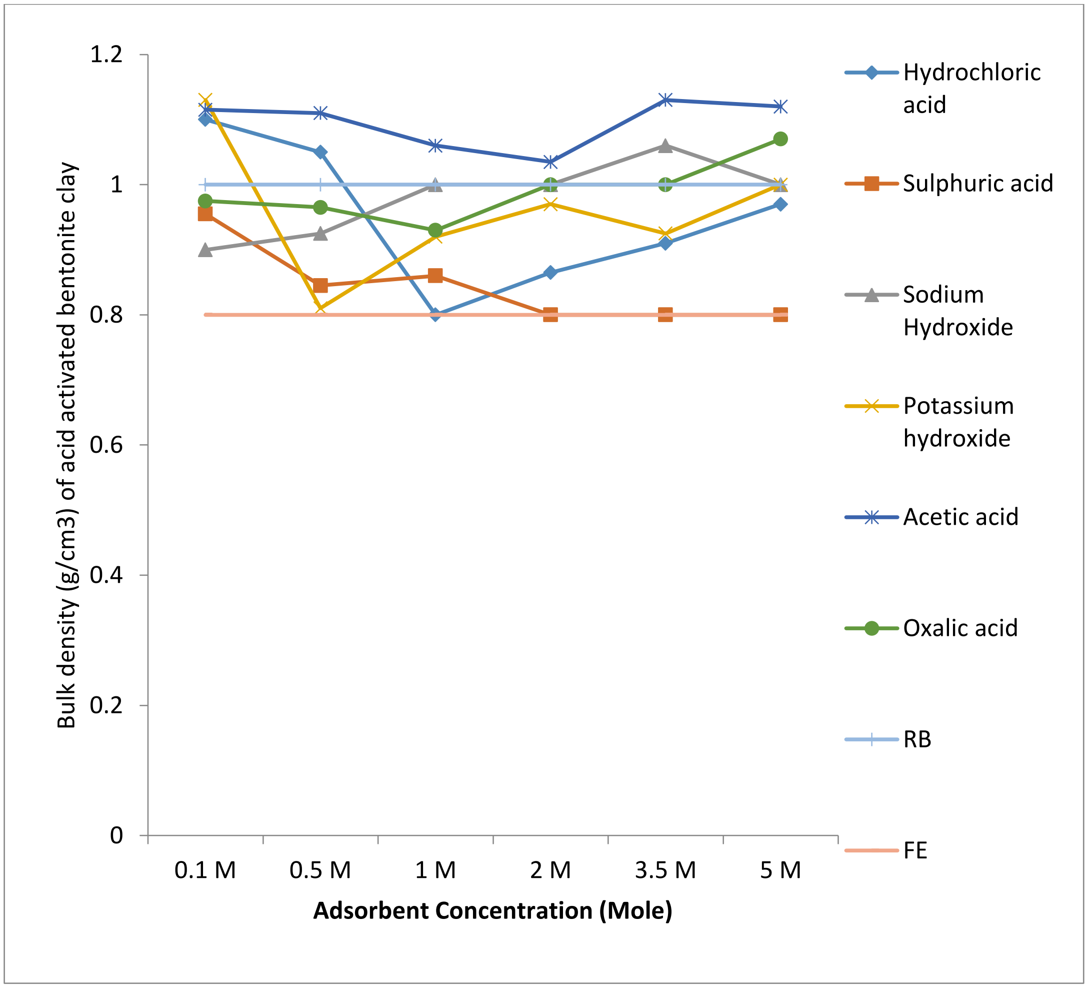

Figure 10: Effect of adsorbent concentration on the bulk density of activated bentonite

Considering that bulk density has an inverse relationship with porosity, these findings are in line with the reports of Selim *et al*. (2020). According to their findings, the leaching of cations upon acid activation results in voids in the bentonite, increasing the porosity of the clay surface at low acid concentrations and forming clumps of uneven, low-porosity surfaces. The surface becomes extremely porous with evenly distributed pores as the acid concentration rises. They clarified that the elimination of contaminants and the substitution of H+ ions for exchangeable cations were the causes of the small pore establishment. They added that as the pores widen and the surface seems more porous and uniform, these physico-chemical interactions alter the morphology of bentonite. This proves that clay that has been acid activated and then thermally activated is more porous than clay that has merely been acid activated.

A different team of researchers used SEM to examine both natural and acid-activated bentonite samples in order to identify any changes in surface morphology following acid activation (Al-Khatib *et al*., 2012). In contrast to the flakes with low porosity for the natural bentonite, they discovered clumps of uneven surface with pore distribution for the acid-activated bentonite. They clarified that this discrepancy results from the cations' leaching through acid activation, which leaves the bentonite with voids and raises its surface porosity while decreasing its bulk density. Meanwhile, Taher *et al*. (2018) also found that chemical activation of bentonite increased the porosity of bentonite. This is evident in their study which showed that the activated bentonite had increased pore volume from 0.0959 cm^3 g^{-1} for natural bentonite to 0.1118 cm^3 g^{-1} for activated bentonite.

Conclusion

In conclusion, the moisture content of the activated bentonites varied significantly depending on the activation method. Such that alkali-activated bentonites exhibited significantly lower moisture content compared to acid-activated bentonites, while raw bentonite and fuller's earth had intermediate moisture contents. More so, the concentration of activating chemicals had a moderate effect on bulk density, with higher concentrations leading to slightly lower densities. Hence, increasing the concentration of activating chemicals generally led to a slight decrease in bulk density. In addition, the activation process significantly influenced the bulk density of the bentonites. As acid-activated bentonites (HCl, H_2SO_4) exhibited lower bulk densities compared to alkali-activated bentonites (NaOH, KOH) and raw bentonite. But acetic acid and oxalic acid activated bentonites displayed intermediate values. The above observations are valuable insights, with obvious implications for the diverse industrial applications of bentonite clay.

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