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Influence of 9% Sodium Hydroxide Solution on Kaolinite Clay

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Abstract
This study investigated the effect of sodium hydroxide (NaOH) solution on the surface characteristics and molecular structure of kaolinite clay. Scanning electron microscope coupled energy dispersive spectroscopy (SEM-EDS) and Fourier-transform infrared spectroscopy (FTIR) were employed in analyzing the raw and 9% NaOH-treated clay samples. The outcome of the SEM-EDS analysis showed a distinct difference between the raw and treated kaolinite clay samples in atomic concentration and weight concentration. In addition, the FTIR spectra of the raw clay exhibited characteristic peaks indicating the presence of O-H stretching vibrations and aromatic compounds. In contrast, the treated clay displayed new peaks suggestive of alkane groups and C-Cl stretching vibrations, indicating potential modifications induced by the NaOH solution treatment. These alterations in the chemical structure of the clay would have significant implications on the adsorption properties, particularly in the removal of contaminants from wastewater. Therefore, alkali-treated clay materials can be used as reinforcement in the production of nanocomposite for wastewater treatment processes.

Keywords: Heavy metals, SEM, FTIR, Alkali treatment, Surface morphology

Introduction
Heavy metals are widely recognized as environmental contaminants because of their high toxicity, ability to accumulate in living organisms and long-lasting presence in the environment (Ali et al., 2019). Heavy metals have become more prevalent in the environment due to rapid industrialization and urbanization since the 1940s. Industrial wastewater effluents contain harmful heavy metal pollutants, which are mostly caused by human and anthropogenic activities. In addition, the adverse impacts of heavy metals such as lead, cadmium, mercury, and arsenic at trace levels can disrupt aquatic life and bio-accumulate in the food chain posing severe health risks to humans (Renu & Singh, 2017). Currently, over 700 million individuals worldwide reside in nations or regions that suffer from a persistent scarcity of water (Tortajada, 2020). According to the United Nations Water (UN Water), the number of people experiencing water scarcity is projected to rise to almost 1.8 billion by 2025 (Awad et al., 2019). Also, more than 50% of the global population is being compelled to reside in regions where they confront substantial water scarcity due to the escalating menace of climate change (Dutta et al., 2021). Regions in sub-Saharan Africa are more likely to be affected because they currently account for the largest number of water-stressed countries in the world (Silva, 2023). According to the United Nations, Africa is projected to experience significant water shortages affecting a population ranging from 70 million to 250 million people (Chojnacka et al., 2020). Thus, early action including wastewater treatment, may help in reversing the devastating impacts of living without clean and safe water for drinking, agricultural, or industrial usage (Silva, 2023). Thus, effective wastewater treatment is essential for addressing this concern and safeguarding ecosystems. However, conventional methods for heavy metal removal, including precipitation, ion exchange and membrane filtration, often face limitations such as high costs and inefficiency in treating low-concentration pollutants (Zamora-ledezma et al., 2021). As a result, the quest for alternative adsorbents made from local materials with effective qualities capable of resolving the aforementioned issues is critical. Thus, the use of various natural clays and their composites to remove metals such as manganese, arsenic, cadmium, iron, selenium, lead, uranium, tungsten, chromium, and zinc is of great concern.

Furthermore, clays and their modified forms have recently attracted a lot of attention for usage as metal ion adsorbents in aqueous mediums due to their ease of availability, low cost, and environmentally favourable efficiency. Teğin and Saka (2021) employed calcination and acid treatment methods in the modification of raw clay for the removal of methylene blue. The findings from the SEM (scanning electron microscope) and Fourier-transform infrared spectroscopy (FTIR) analyses showed that the modified clay significantly improved the adsorption capacity of the treated clay samples. Moreover, the preparation and application of NaOH-treated clay as a potential adsorbent for wastewater treatment, specifically targeting heavy metal removal is an innovative area that should be explored. Therefore, this study aimed to modify the characteristics of kaolinite clay surface to improve the adsorption capacity. The modification of clay minerals, particularly kaolinite, with sodium hydroxide presents a promising avenue for enhancing adsorption capacity and matrix interface for composite applications in wastewater treatment. Kaolinite has a high surface area and available active sites that offer suitable substrate for modification (Bhattacharyya & Gupta, 2008). This research is significant due to the potential environmental and economic benefits it offers. The proposed modification could provide a cost-effective, sustainable, and efficient means to mitigate heavy metal pollution in wastewater while contributing to cleaner water resources and healthier ecosystems.

Materials and Methods

The clay sample was obtained from River Kaduna along Yakowa Road, Kaduna State. The kaolinite clay sample collected was pulverized and reduced to fine particles by grinding, using a pestle and mortar; the ground clay sample particles were sieved with a fine mesh sieve and stored in a well-labelled sample container.

Purification of the kaolinite Clay Sample: 100g of the clay particles was weighed using a weighing balance and washed with distilled water to dissolve solid impurities. The slurry was allowed to dried at room temperature, followed by surface treatment using 9% sodium hydroxide solution (NaOH) for 4 hours. The slurry was washed with distilled water and acetic acid solution was used to remove the residual NaOH solution on the clay samples. The treated clay samples were dried and stored in a well-labelled sampling container.

Characteristics of the Raw and Untreated Clay Samples: The properties of the raw and treated clay samples were analysed using FTIR and SEM. FTIR analysis was performed using a Nicolet 10 FTIR spectrometer (ThermoFisher Scientific, Holland). SEM was conducted using the Phenom-World proX G6 model (Phenom-World Eindhoven Company, Netherlands) instrument coupled with the EDS (Energy-dispersive X-ray spectroscopy) software Bruker QUANTAX 70 (see Figure 1).

Figure 1: Diagram of the SEM-EDS Machine

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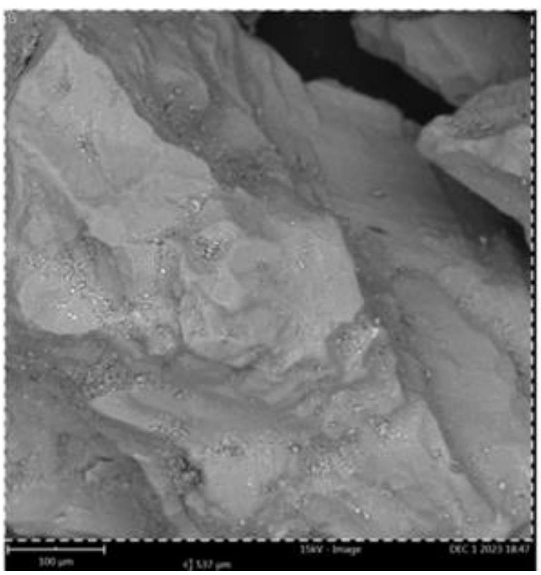
Results

Figure 2 shows the raw clay sample and treated clay with a 9% sodium hydroxide (NaOH) solution.

Figure 2: Raw and 9% NaOH Treated Sample

From Figure 2, it was observed that the 9% NaOH treated clay was light brown compared to the dark brown of the raw clay sample. Also, it was observed that the treated clay was smoother with fine particle size compared to the raw clay sample. Surface Characteristics of the Raw and Untreated Clay Sample: SEM-EDS analysis provided detailed information on the surface morphology and elemental composition of the clay materials. The analysis revealed the presence of several elements. Figures 2 and 3 present the SEM diagrams and the elemental compositions of the raw and treated clay samples present with the atomic and weight concentrations.



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	77.86	74.25
13	Al	Aluminium	10.79	9.89
26	Fe	Iron	4.57	8.66
20	Ca	Calcium	1.63	2.21
11	Na	Sodium	1.47	1.15
12	Mg	Magnesium	1.32	1.09
15	P	Phosphorus	0.78	0.82
16	S	Sulphur	0.70	0.76
17	Cl	Chlorine	0.45	0.54
19	K	Potassium	0.26	0.34
22	Ti	Titanium	0.18	0.29

Figure 3: SEM and Elemental Composition of the Raw and Untreated Clay Samples

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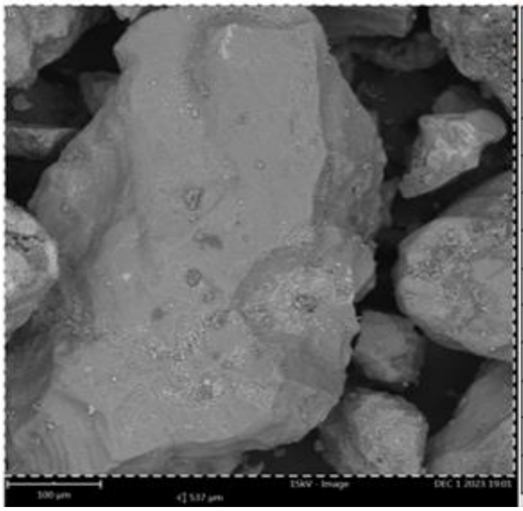


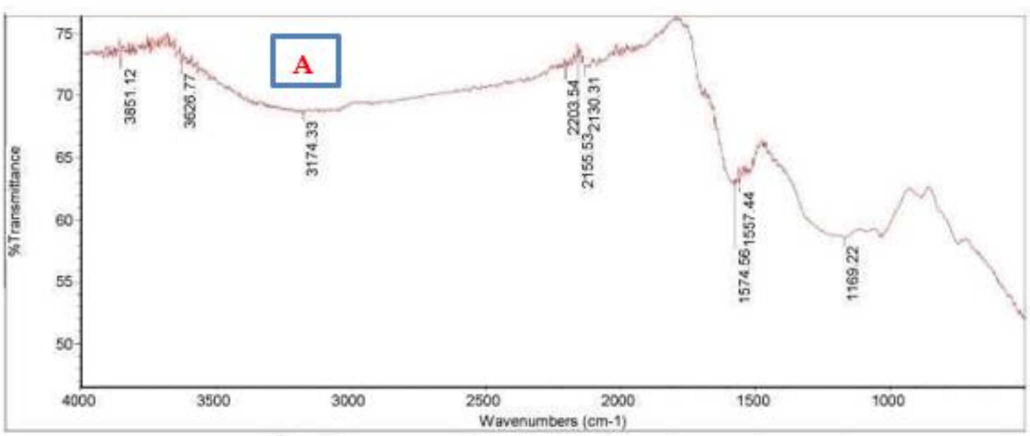
Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	77.86	75.71
13	Al	Aluminium	7.80	7.29
26	Fe	Iron	3.59	6.94
11	Na	Sodium	6.28	5.00
20	Ca	Calcium	1.20	1.67
12	Mg	Magnesium	1.19	1.00
16	S	sulphur	0.72	0.79
17	Cl	Chlorine	0.56	0.69
15	P	Phosphorus	0.60	0.65
19	K	Potassium	0.19	0.26
22	Ti	Titanium	0.00	0.00

Figure 4: SEM and Elemental Composition of the 9% NaOH Treated Clay Samples

The SEM photomicrograph of the raw clay revealed a network structure in which the particles are bonded together. Also, the SEM microstructure of the raw clay sample showed a predominant filmy stack in layers, while the micrographs of the treated clay sample exhibited variable cluster of largely flake-like particles that are loosely packed with some level of porosity which will improve the adsorption capacity of the clay.

Fourier-Transform Infrared Spectroscopy (FTIR) Analysis of the Clay Sample

The FTIR analysis provided valuable insights into the molecular composition of the raw and treated clay samples. Figure 5 presents the FTIR graphs of the raw and untreated clay samples. The figure is a graphical representation of transmittance (%) against the wavenumber (cm $^{-1}$) of vibrational peak intensities.



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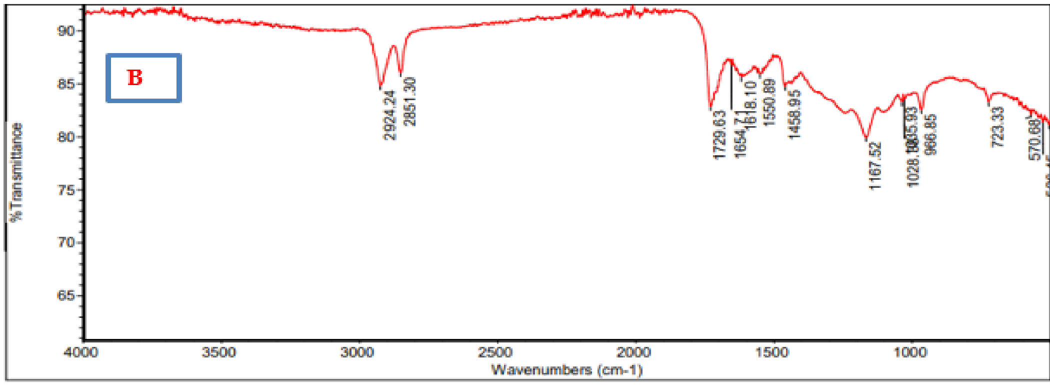

Figure 5: Graph showing the FTIR graphs of the; (A) raw and (B) treated clay sample

From Figure 5, the absorbance peak at 3626.77 cm^{-1} indicated O-H stretching vibration for the raw sample. This suggests the presence of functional groups like alcohol, phenol or carboxylic acid. These functional groups are often associated with organic compounds. Further, the absorbance peak at 3174.3 cm^{-1} indicated a double bond stretching vibration, suggesting the presence of an aldehyde group functional group of organic compounds. Also, the absorbance peak at 2203.54 cm^{-1} indicated the presence of sulphur and sulphonamide compounds at a stronger intensity. The FTIR for the treated clay observed absorbance peak at 2924.24 cm^{-1} which indicated the presence of alkane C-H stretching vibrations. This suggests that the treatment with 9% NaOH solution has introduced or modified alkane groups in the clay structure. Also, an absorbance peak of 1729.63 cm^{-1} showed the presence of ester stretching vibrations with a stronger vibrational intensity suggesting the 9% NaOH treatment has introduced $\alpha - \beta -$ unsaturated Lactone groups in the clay structure. Additionally, the 1654.71 cm^{-1} absorbance peak represents aliphatic compounds at a stronger stretching vibration. The absorbance peak at 723.33 cm^{-1} indicated C-Cl stretching vibration, suggesting the presence of halogens. The introduction of halogens might be a result of the 9% NaOH solution treatment or could be naturally occurring in the clay. These findings indicated that the NaOH treatment altered the structure of the raw clay. Hence, The FTIR spectra of the raw clay exhibited characteristic peaks indicating the presence of O-H stretching vibrations and aromatic compounds. In contrast, the treated clay displayed new peaks suggestive of alkane groups and C-Cl stretching vibrations, indicating potential modifications induced by the NaOH solution treatment.

Discussion

From the Figures (3 and 4), Silicon (Si) remains the same and most prevalent element (77.86%) for both the raw and treated clay samples. Silicon is a fundamental component of clay minerals and its prevalence is expected in both raw and treated samples. Similarly, to the treated clay, the raw clay sample contains various other elements, such as potassium (K), iron (Fe), titanium (Ti), calcium (Ca), sodium (Na), phosphorus (P), sulphur (S), aluminium (Al), magnesium (Mg), and chlorine (Cl). Whereas, the weight concentrations indicated that Si was the major component for both the raw and treated clay samples. Also, the weight concentration of iron is slightly higher in the raw clay compared to the treated clay. Therefore, the treatment with 9% NaOH solution has led to changes in the weight concentration of silicon, suggesting alterations in the composition of the clay, possibly due to the introduction of new compounds or modifications in existing ones. The comparative analysis of atomic and weight concentrations provides insights into the elemental composition of the clay samples and the impact of the NaOH solution treatment on specific elements. Furthermore, The SEM diagrams showed that the NaOH-treated clay particles clustered together when compared with the raw samples. This finding corroborates with the work of (Teğin & Saka, 2021) who reported that the cluster particles observed in the acid-treated clay samples could be attributed to the reaction of some cations with the acid to form sulphates, chlorates and oxide that can be easily lost from the clay structure.

In addition, the strong absorption bands obtained at 3626.77 cm^{-1} and 3174.3 cm^{-1} represents stretching of the surface hydroxyl groups of the inner hydroxyl group and dioctahedral layer located in the plane between the octahedral and tetrahedral sheets (see figure 5). These peaks confirmed that the clay is kaolin and corroborates with literature report of (Lawal et al., 2020). Furthermore, the absorption at this band was diminished by the treatment of

NaOH due to dehydroxylation. Peaks at 1729.63 cm^{-1} associated with C = O stretching in carbonyl group, signified potential C-O stretching between the mineral composition. Peaks in the range of 1654.71 cm^{-1} to 1550.89 cm^{-1} indicated C = C stretching in aromatic rings. The 1167.52 cm^{-1} absorption band can be ascribed to Si-O-Si stretching, 1028.45 cm^{-1} signified alternating Si-O-Si and Al-O-Al stretching vibrations, and 966.85 cm^{-1} maybe due to Al-OH vibrations. These outcomes aligns with previous research findings, providing a comprehensive understanding of the vibrational characteristics of the analyzed samples (Lawal et al., 2020; Shuma et al., 2019).

Conclusion

The outcome demonstrated that sodium hydroxide treatment of the clay has a significant impact on the surface characteristics and molecular composition of the treated clay, as revealed by the SEM-EDS and FTIR analysis. The introduction of alkane groups and halogens suggests potential modifications that could enhance the clay adsorption properties for contaminants in wastewater. Furthermore, additional analysis such as X-ray diffraction can be conducted to provide more detailed insights into the structural changes induced on the clay samples by the sodium hydroxide treatment.

Recommendations

1. This study recommends adsorption studies to evaluate the efficacy of the treated clay in removing specific contaminants from industrial wastewater.
2. Further analysis with X-ray diffraction spectroscopy to provide detailed information of the crystal structure and physical properties of the clay samples.

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