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Assessment of 6% And 12% NaOH-treated clay Samples for Composite Application in Wastewater Treatment

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Abstract
The present study aims to modify the surface characteristics of locally sourced kaolinite clay using 6% and 12% NaOH solution. The effect of NaOH activation on the surface characteristics, elemental composition and structure of the clay samples was investigated using scanning electron microscope coupled elemental dispersive X-ray spectroscopy (SEM-EDS) and Fourier transform infrared spectrometer (FTIR) techniques. The FTIR graphs showed that the intensity of the bands decreases with the concentration of NaOH. Furthermore, the SEM images revealed that the treated clay samples showed distinctive particle characteristics compared to the untreated sample. Thereby, improving the surface area and creating a rough surface topography that could enhance the adsorption of heavy metals in wastewater remediation and clay matrix interface polymer. Overall, the NaOH activation process improved the crystallinity of the treated samples. Additionally, the EDS analysis revealed that silicon, aluminium and iron were the prominent elements. These findings have proven the capability of NaOH-treated clay for use in wastewater remediation.

Keywords: Alkali treatment, SEM-EDS, Surface morphology, Kaolinite clay, Heavy metals**.**

Introduction

Water pollution has a significant negative impact on living things and the equilibrium of the entire ecosystem, making it a major global environmental problem. The health security of humans, animals and plants is seriously impacted by heavy metal contamination in the environment (Briffa et al., 2020). Heavy metals are not biodegradable and can be carcinogenic; hence, excess concentrations of these metals in water can pose a major health risk to living organisms (Qasem et al., 2021). Presently, several research have been conducted in this area to identify practical ways for remediation of heavy metals from industrial effluents. Nevertheless, the conventional treatment processes of heavy metals removal from industrial effluents are plagued with numerous limitations including high cost, high energy consumption, production of toxic sludge and inadequate removal of metal ions from the effluents. Therefore, the search for alternative adsorbents from local materials with effective properties that can solve the aforementioned problems is crucial. Moreover, clay, silica and zeolite are excellent options for cost effective wastewater remediation (Li et al., 2017). Although, clay has good surface hydrophilicity, high cation exchange capacity (CEC), swelling/expanding capacity, surface electronegativity and selectivity for cation exchange. Furthermore, pillar bearing, thermal treatment and acid treatment could improve the surface area, pore size and volume, which would lead to significant increase in adsorption efficiency (Zhang et al., 2020). Besides, it is important to use clay materials and their composites for the removal of heavy metals such as arsenic, chromium, mercury, cadmium, lead, copper, nickel and zinc. Further, clays and their modified forms have garnered great attention for use as adsorbents for the remediation of industrial effluents due to their low cost, availability and eco-friendliness. Therefore, several modification methods including impregnation and calcination have been suggested to increase the potential of mineral adsorbent in wastewater remediation. Thus, the aim of this study was to chemically modify kaolinite clay using

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different concentrations of NaOH to remove the adhering impurities and improve the surface properties of the clay specimen. Additionally, the surface characteristics and morphology of the clay samples were evaluated using SEM-EDS and FTIR techniques.

Materials and Methods

The clay specimen was obtained from River Kaduna along Patrick Yakowa Road, Kaduna State, Nigeria. The clay was finely grounded using a pestle and mortar. Further, the grounded clay was then sieved through a 0.06 mm sieve to eliminate heavy organic/inorganic particles and ensure uniformity for easy dispersion in water. The sieved samples were stored in airtight containers for further use.

100 g of the finely grounded dry clay samples was separately introduced into 500 ml of 6% and 12% NaOH solution and were intensively stirred for 4 hours. Then the clay slurry was thoroughly washed with distilled water and acetic acid solution before drying at room temperature. Furthermore, the surface morphology and molecular characteristics of the raw (untreated) and treated clay specimens were evaluated using scanning electron microscope coupled elemental dispersive X-ray spectroscopy (SEM-EDS) (PhenomWorld Eindhoven Company, Netherlands) and Fourier transform IR spectrometer (ThermoFisher Scientific, Holland). Figure 1 shows a pictorial diagram of the untreated (raw), 6% and 12% NaOH-treated clay samples.

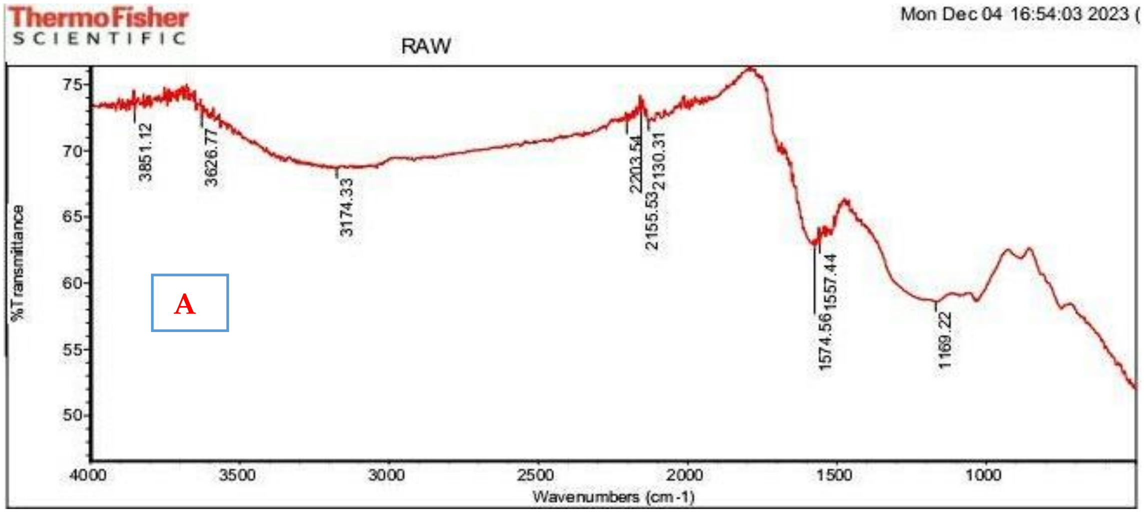
Figure 1: Untreated (raw), 6% and 12% NaOH-treated clay samples

Results

Figure 1 shows the untreated (raw), 6% and 12% NaOH treated of the clay samples. It was noted that the untreated clay sample was darker in colour compared to the treated clay samples. Also, the untreated sample had a rough and coarse texture in contrast to the 6% and 12% NaOH-treated samples. However, the treated samples were observed to be lighter in colour and smoother in texture. Additionally, the size particles of the 6% NaOH treated clay were fine and less coarse in texture compared to the untreated clay. While the particle size of the 12% treated sample was smoother than the 6% NaOH-treated clay.

FTIR spectra for the untreated and treated clay samples

FTIR spectroscopy is used in identifying different functional groups in the clay structure. These functional groups are associated with specific vibration modes, which can be identified by the characteristic absorption bands seen in the FTIR spectrum. Additionally, the FTIR spectrum is a plot of transmittance against wavenumber (cm^{-1}). Figure 2 shows the FTIR spectra of the untreated (raw), 6% and 12% NaOH-treated clay samples in the range of 4000-540 cm^{-1}.



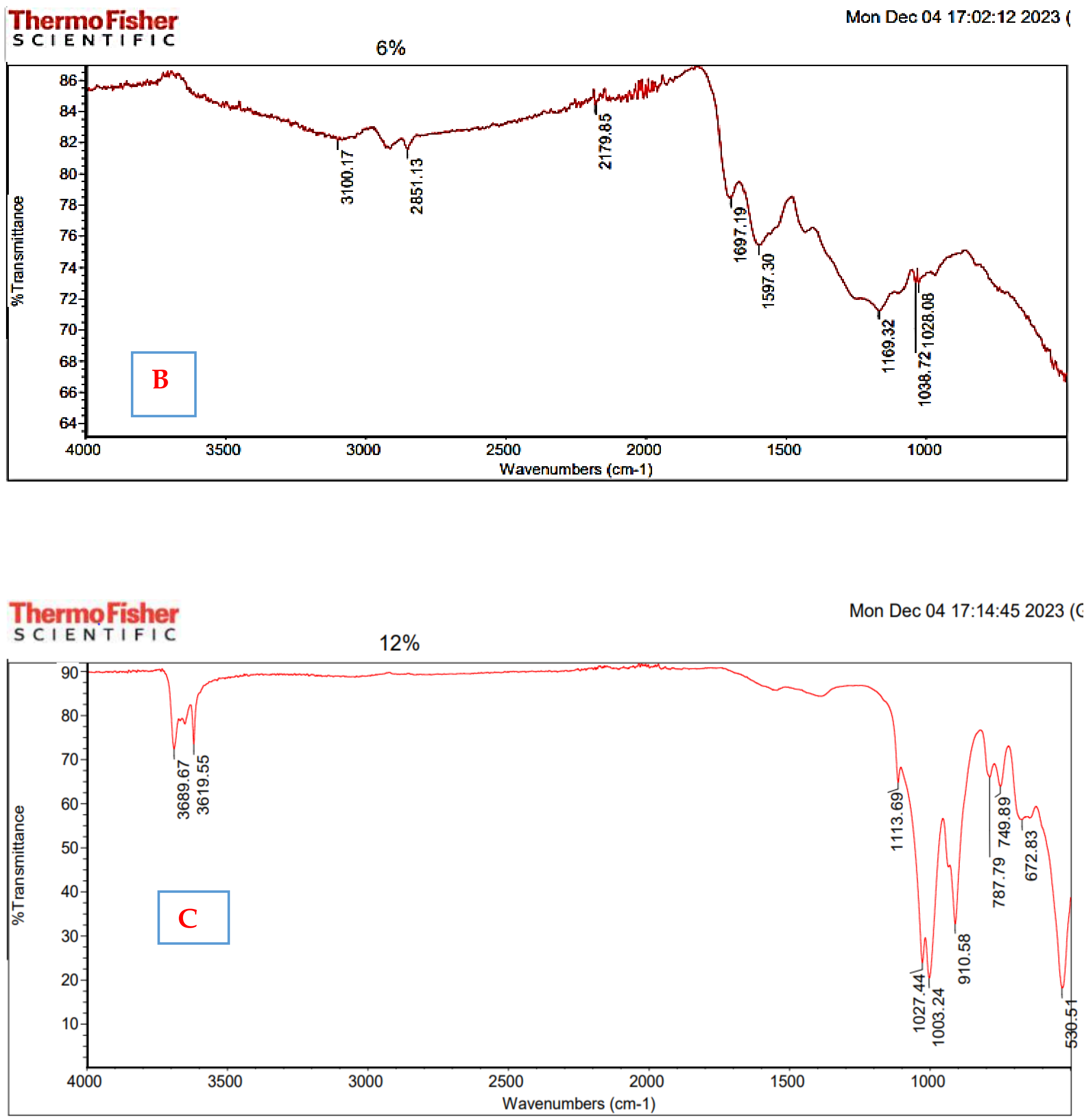
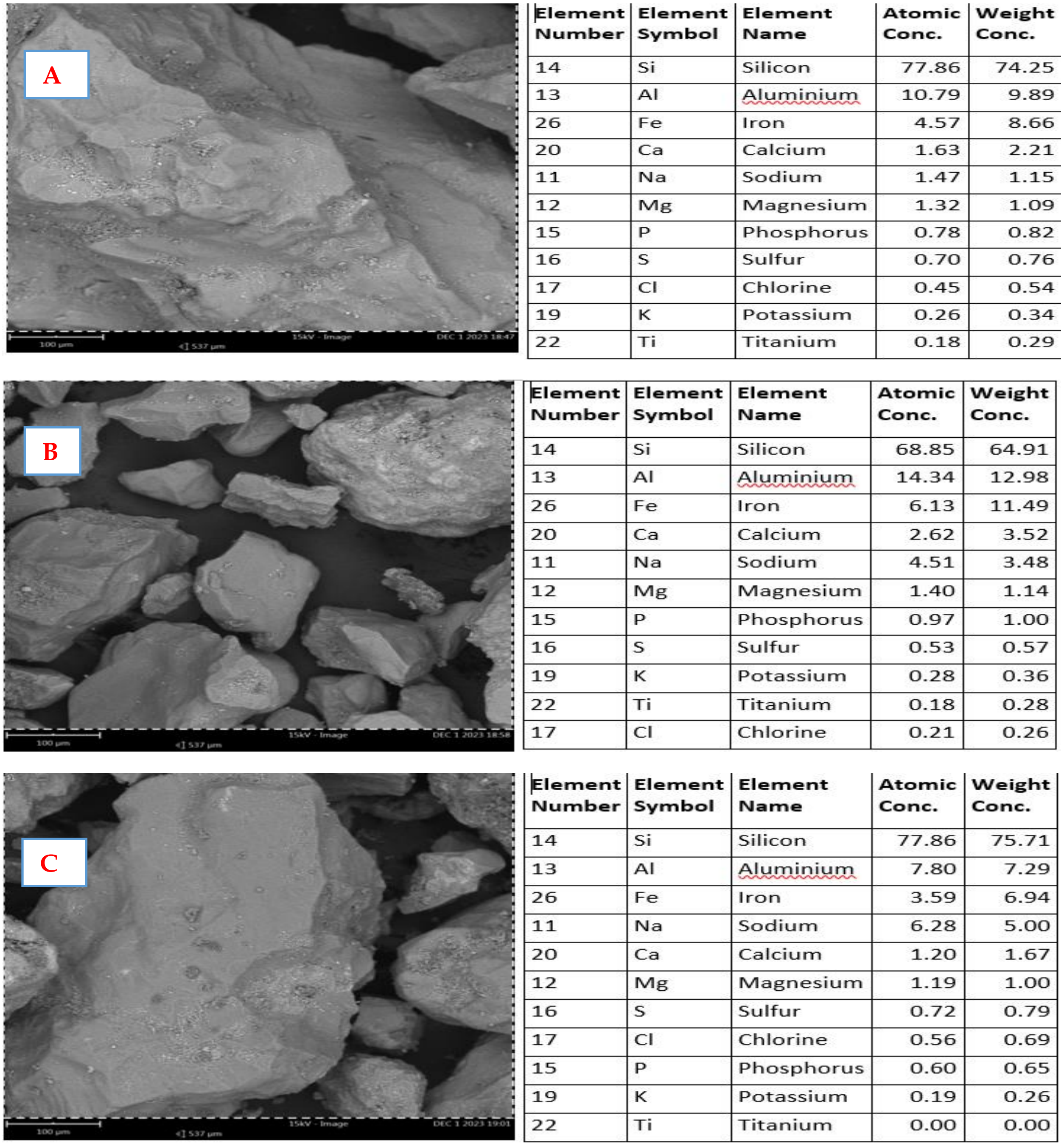


Figure 2: FTIR Analysis for (A) Untreated, (B) 6% and (C) 12% NaOH Treated Clay Samples

From Figure 2, the absorbance peaks of 3851, 3626, 3174, 2203, 2155, 2130, 1574, 1567 and 1169 cm^{-1} were detected in the FTIR graph of the untreated clay samples. The peak at 3626 cm^{-1} represented the hydroxyl (O-H) group stress vibration. In contrast, SiO stress frequencies of quartz were observed below 1000 cm^{-1} in the spectrum of 6% NaOH-treated clay samples. The peak between 1068-1027 cm^{-1} displayed strong absorption bands attributed to Si=O bonds, corresponding to silicate structure. A discernible band was linked to OH deformation resulting from adsorbed water containing exchangeable cations present in the clay samples. While at 12% treated clay samples, peaks of 672 and 530 cm^{-1} corresponded to the bending vibrations of Si-O-Si.

Scanning Electron Microscopy/Energy Dispersive Xray Spectroscopy (SEM-EDS) Analysis of the untreated and treated clay samples

Figure 3 depicts the surface morphologies and elemental composition of the untreated and treated clay samples. The SEM-EDS technique allowed high-speed qualitative elemental analysis of the clay samples.



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	Sulfur	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

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	68.85	64.91
13	Al	Aluminium	14.34	12.98
26	Fe	Iron	6.13	11.49
20	Ca	Calcium	2.62	3.52
11	Na	Sodium	4.51	3.48
12	Mg	Magnesium	1.40	1.14
15	P	Phosphorus	0.97	1.00
16	S	Sulfur	0.53	0.57
19	K	Potassium	0.28	0.36
22	Ti	Titanium	0.18	0.28
17	Cl	Chlorine	0.21	0.26

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	Sulfur	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 3: SEM Micrographs: (A) Untreated, (B) 6% and (C) 12% Treated clay samples

The surface characteristics of the untreated clay, 6% and 12% NaOH treated clay samples are shown in Figure 3. It was discovered that the SEM images of the treated clay samples showed distinctive particle characteristics compared to those of the untreated sample. Whereas the uniform thick layers firmly piled on the clay's surface became increasingly heterogeneous, as shown in the micrograph of the raw clay sample. As such the irregular particles of the untreat clay were removed by leaching in the 6% and 12% NaOH solution.

Discussion

A decrease in the quantity of the treated clay sample was observed when compared with the untreated clay sample. This might be ascribed to the action of NaOH on the clay sample, indicating improvement in the characteristics of the kaolinite clay after the alkali treatment. These outcomes coincide with the work of (Jozefaciuk & Bowanko, 2002; Lakevics et al., 2014). In general, NaOH in contact with clay attacks not only the amorphous phase consisting of organic matter, ferric, silicon and aluminium compounds, but also the texture of the clay materials (Nguemtchouin et al., 2018).

Additionally, SiO groups were observed at 910, 787 and 749 cm^{-1} in the spectrum. As a result, structural alterations in the tetrahedral cations caused variations in the peaks corresponding to Si-O and Si-O-Si. Furthermore, the NaOH treatment drastically altered the position and density of all distinctive peaks. Protons permeated the clay layers and attacked the OH groups, creating alterations in the adsorption bands. When compared to the untreated clay samples, the peak intensities of Si-O-Si Si-O-Al, and Si-O were found to be lower. Also, it was observed that the intensity of the bands decreases with the concentration of NaOH. Similar findings were reported by Nguemtchouin et al. (2018). In general, the aluminosilicate (Si, Al, Fe and Mg) elements were compactly arranged in the brighter areas displayed on the surface of the SEM diagrams. Additionally, the SEM micrographs of the 6% NaOH-treated clay samples formed agglomerates of flaky particles piled together. Furthermore, clustered particles were found when the morphological structures of the untreated and treated clay were compared. These particles were formed due to the simultaneous replacement of trivalent with divalent ions and trivalent ions in the octahedral and tetrahedral layers, respectively. In other words, cations react with acid to form oxides, which are easily lost from the clay structure during the acid treatment. Besides, during the acid treatment process, several cations including Na, Ca and K were leached. Thus, the NaOH treatment altered the morphological structures, thereby increasing the surface area of the clay particle size.

In addition, the energy-dispersive X-ray spectroscopy **(**EDS) technique provided a qualitative evaluation of the elemental components present in the untreated and treated clay samples. Aluminium and silicon were observed to be the most common elements, forming the aluminosilicate structure of the clay minerals. This outcome aligns with the findings of Ismadji et al. (2015). The presence of other elements including Na, K, Fe, and Mg was observed. These elements act as cations and help balance the excessive surface charge of clay materials (Tetteh et al., 2022). Overall, the NaOH activation process improved the crystallinity of the clay compared to the untreated clay samples.

Conclusion

This study demonstrated that NaOH treatment can alter the physical and chemical properties of kaolinite clay, which could make it effective for use as reinforcement in composites. The FTIR and SEM results showed that the alkali treatment caused changes in the hydroxyl groups and surface morphology of the clay. These findings highlight the potential of NaOH-treated clay as an effective adsorbent for wastewater treatment applications. Additionally, the application of surface-modified clay materials in wastewater treatment processes will offer a sustainable and cost-effective solution for environmental remediation.

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Recommendations

Further study can focus on the development of nanocomposites with the treated NaOH clay samples as reinforcement. In addition, the effects of acid/alkali activation on other types of clay materials should be investigated.

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