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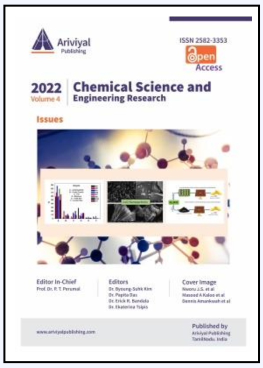

Preparation, Characterization and Antimicrobial Activity of Bentonite Clay Obtained from Sabon Kaura Deposit in Pindiga, Gombe State, Nigeria

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Abstract: The Usual techniques such as X-ray diffraction (XRD), X-ray Flourescence (XRF), Fourier Transform Infrared spectral (FTIR) analysis, Scanning electron microscopy (SEM), and UV-visible were employed to study bentonite sample from Sabon Kaura Pindiga (Gombe State, Nigeria). From the XRF analysis, the most abundant oxides in the sample were Al_2O_3 and SiO_2. The XRD analysis showed that the bentonite from Sabon Kaura region is composed of minerals such as kaolinite, quartz, montmorillonite, chlorite, clinochlore and illite. The average crystal size of the prepared bentonite as calculated by the Debye–Scherer formula was found to be 37.54 nm which indicates that the particles are in Nano size. Sabon Kaura (SK) sample shows a SEM image with a soft surface and more phase separation between the particles. From the FTIR spectra, the sample shows OH vibrational stretching picks in the Si-OH and Al-OH groups of the tetrahedral and octahedral sheets. Bands were also noticed by the stretching mode of Si–O. The result of the analysis by UV-visible shows that the SK bentonite spectrum was detected around 300 nm. The sample shows a good antibacterial and antifungal activity when tested against gram-positive and negative bacteria, *Staphylococcus aureus, Mycobacterium* and *Bacillus subtilis Escherichia-coli, Helicobacter pylori* and *Klebsiella pneumonia*. And also two fungi *Aspergillus niger* and *Candida albicans*, the Minimum inhibitory concentration (MIC) and Minimum bactericidal concentration (MBC) of the sample shows both bactericidal and bacteriostatic effects.

Keywords: Bentonite clay; Characterization; Antibacterial; MIC; MBC

1. Introduction

Bentonites are aluminum phyllosilicate formed frequently from the formation of volcanic ash, predominantly consisting of smectite minerals, usually montmorillonite (MMT) (80-90% by weight). Bentonites are versatile material for geotechnical engineering, due to its special properties. Bentonites are in high demand for different industrial applications.[1-2] Geological/Mineralogical and chemical composition affects the properties of bentonite. The measured physical characteristics are frequently used to ascertain the mineralogical contents of bentonite.

The types of bentonite exist usually in two forms: swelling bentonite (sodium bentonite) and non-swelling bentonite (calcium bentonite). Most notable of bentonites are of the calcium type.[3-7] Research by the Nigerian Mining Corporation revile the existence of bentonitic clay reserves of over 700 million tonnes in different parts of the country which include the North-east quadrant; Adamawa, Borno, Taraba and Yobe.[8]

The form of clay particles is perceived in layers where each layer is composed of two types of structural sheets: tetrahedral and octahedral. The layers present in MMT mostly composed of a 2:1 structure type i.e. two tetrahedral silica sheets sandwiching a central octahedral alumina sheet (TOT). Largely due to isomorphic substitution within the layers (e.g., Al^{3+} for Si^{4+} in the tetrahedral sheet and Fe^{2+} or Mg^{2+} for Al^{3+} in the octahedral sheet) the bentonites clay layers consists a negative crystal charge which is balanced by exchangeable cations such as Na^+, K^+, Ca^{2+} in the interlayer together with water molecules bonded by ion-dipole forces. The hydration of these in-organic cations causes the clay mineral surface to be hydrophilic.[9-11]

Bentonites deposits and clay minerals usually vary in nature and frequently different samples of clay from the same deposits differ. Their chemical and physical properties (swelling ability, plasticity, cation exchange capacity, etc) vary typically within and between deposits due to the differences in the degree of chemical substitution within the smectite structure and nature of exchangeable cations present, and also due to the type and amount of impurities present.[12-14]

Quartz, Calcite, Feldspar, Cristobalite, Biotite, Kaolinite, Mica and Organic matter are the most common impurities in bentonite clay, while hydrated iron oxide, ferrous carbonate and pyrite are being the minor impurities depending on the nature of their genesis.[14-15]

2. Materials and Methods

2.1. Materials

All reagents used were of analytical grade which are as follows: Hydrogen peroxide (H_2O_2), NaOH, Mueller Hinton Broth, Mueller Hinton Agar, Potato dextrose Agar Distilled water and Deionised water. The equipment's used are: pH Meter, Micrometer Sieve, Muffle Furnace, Pestle, Mortar, and Crucibles. Bentonite was obtained from Sabon Kaura Village, Pindiga, and Akko Local Government Area Gombe State, Nigeria. The field sampling exercise was carried out during the dry season. Fresh sample of the clay was collected.

2.2. Methods

The bentonites lay ascertain in this study was obtained from Sabon Kaura deposits, Pindiga, Gombe State Nigeria and was labelled as SK. Large chunks of the bentonite was crushed with a hammer and then pulverized with pestle and mortar. The powder was then sieved using 250 µm sieve (Scientific sieve). The powder obtained was sieved to a size of 74 micrometres (200 mesh).[16] The powdered bentonite sample was pre-treated with 10% H_2O_2 to oxidize any coloured organic and inorganic matter in the bentonites clay.[17] The suspension was then agitated and allowed to stand for 2 hr. The clear supernatant liquid was decanted and the clay re-suspended in 1 L 0.5 M NaOH solution for a period of 2 hr with moderate agitation using a mechanical stirrer. The supernatant was also decanted and rinsed with deionized water, sun dried, and then calcined at 600°C for 2 hr and ground. The physicochemical properties of the bentonites were reported elsewhere.[18]

2.3. Minimum Inhibitory Concentration (MIC) using Broth Dilution method

MIC of the extracts was carried out using broth dilution method as adopted by Shah et al 2013.[19] Nine serial two-fold dilu-tions of extracts or conventional antibiotic (gentamycin) were made from the stock concentration to obtain final concentration ranges of 500 µg/mL to 3.9 µg/mL. The ninth test tubes (n°9) served as negative control (broth + inoculums). For each bacterial, three columns of nine test tubes were used. Each well has the culture medium (2 ml) + plant extract or gentamicin (1.8 ml) + 0.2 mL standardized bacterial inoculums (106 µg/mL). The test tubes were covered and incubated at 37°C for 24 hours for bacteria and observed for turbidity or growth. The lowest concentration which showed no turbidity in the test-tube was recorded as the minimum inhibitory concentration (MIC).

2.3.1. Minimal Bactericidal Concentration (MBC)

Minimum bactericidal concentration (MBC) was carried out according to the method described by Abdallah et al., 2011[20] and was recorded as a lowest extract concentration killing 99.9% of the bacterial inocula after 24 h incubation at 37°C. MBC values were determined by removing 100 µl of bacterial suspension from subculture demonstrating no visible growth. The plates were labelled with the test microorganism (each plate with a test microbe). The microbes were spread evenly over the surface of a prepared MHA media with the aid of a swab stick. The plates were incubated at 37°C for 24 hours for bacteria and observed for colony growth. The MBC was the plate with the lowest concentration of the plant extract or gentamicin and without colony growth. The experiment was carried out in triplicate.

2.3.2. Fungal culture

28 g of potato dextrose agar (PDA) and was dissolved in 1000 ml of deionized water in 1000 ml conical flask. The mixture was sterile in autoclaved for 15 minutes at 121°C. The mixture was allowed to cool and then and 0.2 g of Chloramphenicol was added to the solution and was then stirred and poured on petri dishes. After the PDA had solidified inside the petri dish, pure isolate of the fungi *Aspergillus niger* and *Candida albicans* were grown on different agar plates at 27°C for 48 hours in an incubator.

2.4. Characterization of the sample

X-ray fluorescence analysis (XRF) - The chemical composition of the bentonite sample was carried out using X-Ray fluorescence analysis (X-supreme 8000) instrument. This was done at Ahmadu Bello University Zaria, Kaduna State Nigeria.

Fourier Transform Infrared spectroscopic analysis (FTIR)- The infrared spectra measurements were carried out using a Perkin Elmer spectrophotometer model 10.03.09 at the Department of Pharmaceutical Sciences Gombe State University Gombe, Gombe State Nigeria to identify the possible nanocomposites responsible for the reduction, capping and stabilization of the metal NCs. The FT-IR spectra were recorded in the range of 4000 – 450 cm^{-1} at a resolution of 4 cm^{-1}.

X-ray diffraction analysis (XRD) - The Bragg angular zone was explored by X-ray diffraction (XRD) by Rigaku EMFMA (Enhanced Miniflex Material) Analyzer, Goniometer radius 240 mm, 0 kV and 0 mA, Cu Kα radiation source (λ = 1.54060). The diffraction angle 2θ was scanned from 3o to 90° at scanning rate of 2°/min and step size of 0.02° in order to calculate the distance between the silicate layers, Bragg's law was used.

Scanning Electron Microscope (SEM) - (Phenom Proxy, PW 100-002, magnification-300x, Accelerating Voltage- 15 KV) was used to evaluate the surface morphology of the nanocomposites samples. Thin films of the samples were prepared on a carbon coated brass stub by adding a drop of the samples onto the cover slip mounted on the stub and air-dried for 10 minutes. The samples on the stub were then sputter coated with gold for approximately 20 minutes.

UV-visible Spectroscopy Analysis - The UV spectra were recorded on a Perkin Elmer UV-Visible spectro-photometer model 725. The reduction process of silver ions in aqueous solution was measured by the sampling of 1 ml aliquot compared with 1 ml of distilled water used as blank and subsequently measured the UV-visible spectrum of the solution. UV-visible spectrum was monitored on Cary Series UV-vis spectrophotometer Perkin Elmer, operated within the wavelength range of 200 to 800 nm.

The susceptibility of the bacteria to the samples and in combination with conventional antibiotic (gentamycin) varied according to microorganism. As for the micro well dilution assay

Table 1. Chemical Composition of Bentonite

Chemical Oxide	S.K (wt %)
SiO_2	56.588
Al_2O_3	32.006
TiO_2	4.773
Fe_2O_3	4.223
Cl	0.645
ZrO_2	0.550
BaO	0.386
K_2O	0.205
V_2O_5	0.199
CaO	0.137

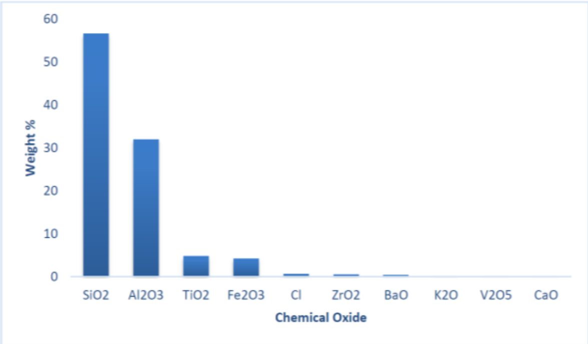

Fig. 1. Chemical Composition of the Bentonite Sample Analysed by XRF

Table 2. Minimum inhibitory concentration (MIC) and Minimum bactericidal concentration (MBC) of Bentonite

Bacteria	MIC	MBC	Bactericidal	Bacteriostatic
S. aureus	125	500	0	_
B. subtillus	125	250	+	0
E. coli	250	1000	0	_
K. pheumoniae	250	500	+	0

The results are the means of number of the colonies ± standard deviations **+:** bactericidal effect (MBC/MIC = 1 or 2)**–:** bacteriostatic effect (MBC/MIC = 4 or 16) [14]. S. aureus = *Staphylococcus aureus, B. subtillus = Bacillus subtilis, E. coli = Escherichia coli, K. pneumonia = Klebsiella pneumonia.*

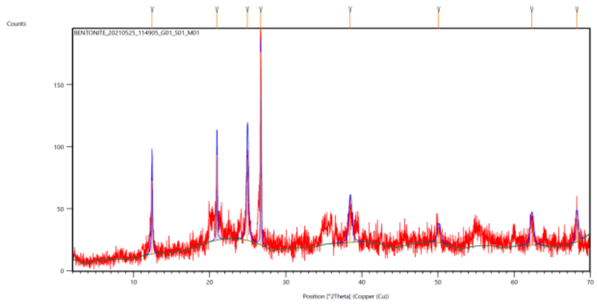

Fig. 2. X-ray diffraction pattern of prepared Bentonite sample.

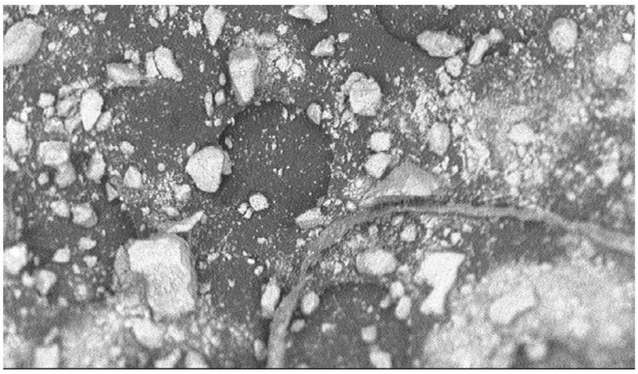

Fig. 3. Scanning electron microscopy (SEM) micrographs of prepared Bentonite.

(MIC) and Minimum bactericidal concentration (MBC) of samples and in combination, result varied according to the microorganism (Table 2). The MIC values were ranged from 500 µg/ml to 31.25 µg/ml and for the MBC values were ranged from >1000 µg/ml to 62.5 µg/ml. The bactericidal and bacteriostatic effect of samples and their combination with conventional antibiotics was determined using the ratio MBC/MIC (Table 2).[21]

3. Results and Discussions

For the XRF result, the chemical compositions are shown in Table 1 and Fig. 1. The result of the chemical composition of Sabon Kaura bentonite sample is higher to those of previous researchers.[5,22-30] The most abundant oxides in Sabon Kaura bentonite sample is SiO_2 and Al_2O_3. The silica (SiO_2) value of the Sabon Kaura bentonite (56.59 wt %) is higher than the values reported by for Wyoming bentonites.[5,23-30] The calcium content of the bentonite sample (Sabon Kaura) from this study (0.14 wt %) has value lower than those of Wyoming bentonite. Fe_2O_3 value reported for the Sabon Kaura bentonite sample (4.22 wt %) in this study is lower than those reported[23-31] (4.80 and 5.12); (8.26) but higher than those reported[20,4] (3.82 wt %); (3.84 wt %); for the Wyoming bentonites. All the values for the other oxides from this study were similar to the values obtained for the different Wyoming bentonites. Sabon Kaura bentonite was found to be a non-swelling bentonite that is a calcium bentonite.

Fig. 2 showed that the XRD spectra of bentonite of Sabon Kaura area are a mixture of several minerals, such as kaolinite, quartz, montmorillonite, chlorite, clinochlore and iolite. From the XRD patterns of the sample, basal reflections with an angles 5.90°, 12.42°, 20.94°, 24.96°, 26.71°, 38.46°, 50.08°, 62.30° and 68.24° were observed which are associated to the presence of smectite and impurities, quartz and iolite in the Sabon Kaura bentonite sample. Reflection at 29.50° was seen which is due to the presence of Feldspar in the sample. The result is with-in the range of expected values (1.2 to 1.6 nm) for typical smectite minerals observed under ambient temperature and humidity.[31] This XRD result is also similar with previous studies.[2-5] The average crystal size of the prepared bentonite as calculated by the Debye–Scherer formula was found d = 37.54 nm. This indicates that the particles are in Nano size.

Fig. 3 showed the SEM image of SK bentonite. Massive plates with some phase separations are observed as a heterogeneous surface morphology. It showed a soft surface where large lamellas tend to form big agglomerates with a layer disposition. Sample of Sabon Kaura shows a SEM image with more phase separation between the particles.

The FTIR spectra of the bentonite sample (Fig. 4) shows that the bands at 3696 cm^{-1} and 3621 cm^{-1} are as-signed to OH stretching vibrations in the Si-OH and Al-OH groups of the tetrahedral and octahedral sheets of the Sabon Kaura clay sample. Bukit et al., Abdullahi et al., Kwaji et al., Dibala et al.,[12-15] reported values of 3698 cm^{-1} and 3620 cm^{-1}; 3693 cm^{-1} and 3620 cm^{-1}; 3695 cm^{-1}; 3698 cm^{-1} and 3620 cm^{-1} for the OH stretching vibrations in the Si-OH and

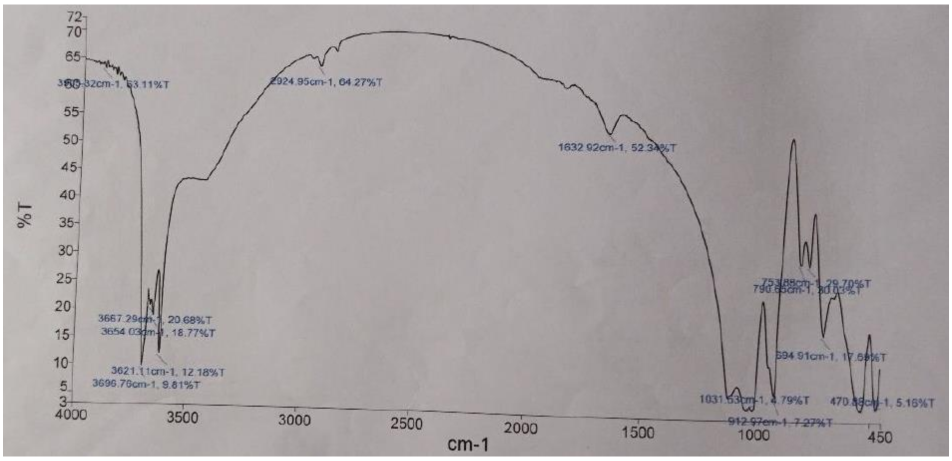

Fig. 4. FTIR spectra of Sabon kaura Bentonite sample

Table 3. Anti-fungal activity of Bentonite.

Tested Organism	Concentration µg/mL 500	250	125	62.5	Control
A. niger	20.50	18.50	14.00	10.00	18.00
C. albicans	22.00	19.00	17.00	14.50	20.00

A. niger = *Aspergillus niger*, *C. albicans* = *Candida albicans*, Control = Ketoconazole (50µg/mL)

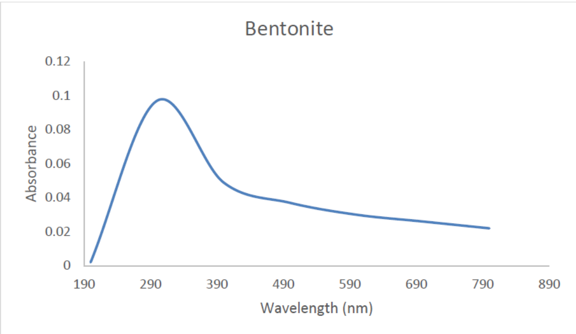

Fig. 5. UV-spectrum of Bentonite supernatant acquired on a PerkinElmer 725 UV-visible spectrophotometer in 1 cm path length quartz cuvette against deionized water in the reference beam.

Al-OH groups respectively. The band of low intensity located at lower frequencies at 1633 cm^{-1} for bentonite sample is produced by the bending vibration mode of adsorbed water, this was in agreement with the values reported[5,29-31] of 1639 cm^{-1} and 1640 cm^{-1} respectively. Ahonen et al., and RMRDC[5-6] reported values of 1630 cm^{-1} and 1636 cm^{-1} respectively.

The most intense bands of the spectrum were found in the low-frequency region. The bands at 791 cm^{-1} and 754 cm^{-1}; for Sabon Kaura bentonite clay was produced by the stretching mode of Si–O (out-of-plane) for Montmo-rillonite and Si–O stretching (in-plane) vibration for layered silicates. Band at 1032 cm^{-1}, was reported from different literatures for the Si-O-Si stretching out of plane and in-plane respectively.[29-33] Bands at 912 cm^{-1} are associated with the bending vibration of Al-Al-OH for both the sample which are in close agreement with values reported in some literatures; 914, 915 cm^{-1}, 919 cm^{-1} 919 cm^{-1} and 920 cm^{-1}.

Table 4. Determination of Helicobacter pylori and Mycobacterium

Organisms	Bentonite
Helicobacter pylori	+
Mycobacterium	+

UV-Vis spectra of Sabon kaura bentonite showed (Fig. 5) a peak 300 nm (strong) corresponding to tetrahedrally coordinated ions Fe.[33,34]

The minimum inhibitory concentration (MIC) of SK bentonite showed less susceptibility in both grams positive, *S. aureus* and *B. subtillus* and also gram negative, E. coli and K. pheumoniae. While the Minimum Bactericidal Concentration (MBC) showed both bactericidal and bacteriostatic effects.

Sabon Kaura Bentonite showed the zone of inhibition ranging from 19 – 22 mm at concentration range of 125 – 500 mm µg/mL. This zone of inhibition is remarkably higher than the zone of inhibition of the control drug (Ketoconazole) with is 18.00 and 20.00 mm for *Aspergilus niger* and *Candida albicans* respectively.

3.1. Evaluation of bactericidal and bacteriostatic capacity

Actions of an antibacterial (samples) on the bacterial strains are characterized with two parameters such as Minimum inhibitory concentration (MIC) and Minimum bactericidal concentration (MBC). If the ratio MBC/MIC = 1 or 2, bactericidal, but if the ratio MBC/MIC = 4 or 16, bacteriostatic.[22]

From Table 4, the sample is reactive against the test bacteria. The test result indicated positively using polymerase chain reaction (PCR) and nucleic acid amplification test method (NAAT) for Helicobacter pylori and Mycobacterium respectively.

4. Conclusions

It can be concluded that, the Sabon kaura bentonite was prepared from the clay and characterized using XRF, XRD, SEM, UV-visible spectrophotometry and FTIR analysis. The Sabon kaura bentonite was found to be calcium type. XRD shows that the sample is composed of different types of minerals, and the average crystal size was found to be 37.54 nm. The sample shows a SEM image with a soft surface and more phase separation be-tween the particles. From the FTIR spectra, the sample shows OH stretching vibrations in the Si-

OH and Al-OH groups of the tetrahedral and octahedral sheets. While the UV-Visible shows that the Sabon kaura bentonite spectrum was detected around 300 nm. It shows a good antibacterial and antifungi activity when tested against gram-positive and negative bacteria, *Escherichia-coli, Klebsiella pneumonia, Staphylococcus aureus, Bacillus subtilis* and *Klebsella pneumonia, Helicobacter pylori, Mycobacterium*. And also two fungi *Aspagillus niger* and *Candida albicans*.

Conflicts of Interest

The authors declare no conflict of interest.

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