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Suitability of Feldspar as an Adsorbent

[1]Shiru Adama Grace, [2]Manase Auta & [3]Bello Friday Ohiani
[1,2&3] Department of Chemical Engineering,
Federal University of Technology, Minna

Abstract

Availability of clean and safe drinking water has been compromised by pollutants. Among these sources of pollutants are domestic sources popularly termed domestic wastewater? Clay and clay minerals have proven to be effective adsorbents; however, their adsorption capacity differs. The aim of this study is centred on the suitability of feldspar as an adsorbent as opposed to other clay minerals such as Plastic clay, Red clay and Barite. Comparative BET analysis of the clay minerals considered in this study revealed that feldspar has the highest surface area and pore volume of 332.8 and 0.218 respectively. Similarly, the clay minerals were used in adsorption study with feldspar producing the best removal efficiency of 90.00 and 73.96% for ammonia and phosphate respectively. The comparative BET results and the favourable adsorption result showed that feldspar was a better adsorbent than other adsorbents. Thermal activation of feldspar was carried out at temperatures of 500, 600 and 700 °C. The BET results of the thermally activated feldspar revealed that feldspar activated at 600 °C has the highest surface area and pore volume of 533.9 and 0.272 as opposed to the surface area and pore volume of raw feldspar, feldspar activated at 500 and 700 °C.

Keywords: *Adsorbent, Adsorption, Clay, Feldspar, Wastewater*

Corresponding Author: Shiru Adama Grace

Background to the Study

Water is a universal resource and due to its free occurrence in nature, it has over time been taken for granted and abused, especially in third world nations where information is neither readily accessible nor disseminated to society [1], [2]. According to [3] the importance of water in human life cannot be overstated as it a precursor to life. As such there is a need to preserve and sustain the supply of clean water.

Reference [1] reported that water that has been spent (used) is called wastewater. Sources of this wastewater comprise of liquid waste discharged from domestic residences, commercial properties, industrial and agricultural sources. According to [5] this wastewater constitutes an environmental pollutant as they contain pollutants such as heavy metals, ammonia and nitrates. Similarly, [5] reported that the type of pollutants found in wastewater depends on the source of the wastewater.

The major sources of wastewater include domestic sources and process industries. Sources of domestic wastewater include human waste, food scraps, oil, soaps, chemical detergents and runoffs while sources of process industries wastewater include electroplating industries, hospitals, pharmaceuticals, power plant, refineries, leather tanning, mining, dyes and pigments, steel fabrication, canning industries and inorganic chemical production plants[4]. This work shall focus primarily on domestic wastewater as this a major cause of degradation of receiving water bodies such as rivers, lakes and streams. To deal with this major threat to the environment, much work has been done to address water pollution. To this effect wide variety of physical, chemical, and biological techniques has been developed; these methods include but are not limited to flocculation, precipitation, ion exchange, membrane filtration, irradiation and ozonation [1] [13]. Similarly, [2], [9] reported that processes such as chemical precipitation, lime coagulation, ion exchange, reverse osmosis, adsorption and solvent extraction can be classified as a conventional approach to the removal of wastewater pollutant while Biosorption and Bioaccumulation can be termed as a non-conventional approach to the removal of wastewater pollutants.

Table 1: Advantage and disadvantages of current treatment technologies for wastewater treatment [2].

Methods	Advantages	Disadvantages
Oxidation	A rapid process for toxic pollutants removal	High energy costs and formation of by-products
Ion exchange	Good removal of a wide range of heavy metals	Adsorbents require regeneration or disposal
Membrane filtration technologies	Good removal of heavy metals	Concentrated sludge production and expensive
Adsorption	Flexibility and simplicity of design, ease of operation and insensitivity to toxic pollutants	Adsorbents require regeneration
Coagulation /flocculation	Economically feasible	High sludge production and formation of large particles.
Electrochemical treatment	Rapid process and effective for certain metal ions	High energy costs the formation of large particles.
Ozonation	Applied in gaseous state; alteration of volume	Short half-life
Photochemical	No sludge production	Formation of by-product
Irradiation	Effective at lab scale	Require a lot of dissolved O_2
Fentons reagents	Effective and capable of treating a variety of wastes and no energy input necessary to activate the hydrogen peroxide	Sludge generation

According to [3], [8] the adsorption technique for the removal of pollutants from wastewater remains the best technique as it is inexpensive, readily available and easy to operate. Adsorption is the transfer process of a substance (adsorbate) that is in solution in a liquid phase to the surface of a solid phase (adsorbent). The adsorption process can occur by means of physical forces, in which the van der Waals forces are involved. It can also occur through chemisorption, which involves chemical interaction between the adsorbate molecules and the surface of the adsorbent[6].

Reference [4] reported that adsorption dynamics consists of four steps namely (i) the transportation of the adsorbate from the bulk solution to the external surface of the adsorbent by diffusion through the liquid boundary layer. (ii) the internal (interphase) mass transfer through the pore by diffusion from the outer surface of the adsorbent to the inner surface the porous structure (iii) Surface diffusion along the porous surface and (iv) Adsorption of the adsorbate on the active sites on the internal surface of the pores. However, the overall rate of adsorption is controlled by either film or intraparticle diffusion, or a combination of both.

Reference [2] have identified various adsorbents suitable for the adsorption process. They include activated carbon (i.e. organic material with high carbon content such as periwinkle shell, coal, wood, cocoa pod husk, peach stone, walnut shell, physic nut waste, coconut shells, bamboo stem wastes palm kernel shells, sugarcane bagasse), activated alumina (made from aluminium ore so that it becomes porous and highly adsorptive), Silica Gel (which is derived from alkali silicate solution where the porous silica gel with an inorganic synthetic polymeric matrix is developed), Hydrogels (which are simply three-dimensional polymeric crosslinked networks that do not dissolve in water but can absorb a large volume of water or aqueous solution) and low-cost adsorbents (this are adsorbent that requires little processing and are ubiquitous such as clays and clay minerals, agricultural wastes, cast iron filings and animal shells)

Reference [2] also reported that clays and clay minerals are preferable amongst the low-cost adsorbents as they possess good adsorption properties (high surface area and porosity) and they require little or no processing and are readily available. Furthermore, [6], [11] reported that clay's adsorption mechanism is dependent upon ion exchange, physical adsorption or both. The surface of clay contains anions and cations like Ca^{2+}, Mg^{2+}, H^{+}, NH^{4+}, Na^{+}, SO_4^{2-}, Cl^{-} and NO^{3-}that can be exchanged with other ions without affecting the mineral structure of clay.Clay and clay mineral are first-class adsorbent properties due to clay's large specific surface area, chemical and mechanical stability, layered structure, high cation exchange capacity (CEC)[3], [10]. Plastic clay, Red clay, Barite and Feldspar have all been identified as clay minerals with desirable adsorption properties.

A preliminary adsorption process to determine the clay mineral with the highest adsorption capacity was set up. This study shall focus on the use of the selected suitable adsorbent as an adsorbent for the adsorption of phosphate and ammonia from domestic wastewater [12], [13].

Methodology

a. Sample Preparation

The clay mineral samples (Plastic clay, Red clay, Barite and Feldspar) was treated by hand picking to remove dirt and other foreign bodies and after which it was sieved with a 125 μm mesh sieve. The powdered sample was then oven-dried at 100°C for 24h.

b. Preliminary Investigation on the different clay minerals

2 g of each of the four clay minerals (Plastic clay, Red clay, Barite and Feldspar) were added to 100ml of the wastewater at operating conditions of a temperature of 30 °C and time of 30 minutes.

c. Wastewater characterization

The physical and chemical properties of the domestic wastewater used were determined in other to have a complete overview of the constituents and nature of the effluent. Properties such as pH, Turbidity, Total Dissolved Solids (TDS), Conductivity, Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD), Total Alkalinity, Total hardness, Nitrates, ammonia and Magnesium were all determined in accordance to the standard methods for the Examination of Water and Wastewater [4].

d. **Characterization of the adsorbent**

i. **Thermo-gravimetric analysis (TGA)**

This analysis was carried out based on the method reported by [7] where thermal transition and decomposition of the sample was done via TGA analysis using Perkin Elmer TGA 4000 thermogravimetric Analyzer at 10 °C/min constant heating rate in nitrogen atmosphere following the ASTM D6370 standard procedure.

ii. **Brunauer Emmet Teller (BET) analysis**

Specific surface area and pore volume analysis of the adsorbent was carried out using BET surface area Nitrogen adsorption procedure. The prepared adsorbent was outgassed under vacuum condition at 300 °C for 4 hours. Out gassed carbon sample was tested for surface area (m^2/g) and pore volume (m^3/g) at 77 K using a 15-point BET NovaWinQuantachrome, 2013 version 11.03[7].

iii. **X-ray diffraction (XRD) analysis**

This was carried out according to the method reported [7], [8] where an Empyrean X-ray diffractometer DY 674 was used with 40 mA, 45 VA and 240 mm tube current, voltage rating and goniometer radius, respectively. The sample was prepared using the sample preparation block and compressed in the flat sample holder to create a flat, smooth surface. The sample was then mounted on the sample stage in the XRD cabinet. The X-ray of Cu Kα radiations was collimated and directed onto the sample. The sample was then analyzed using the reflection-transmission spinner stage and Theta-Theta settings scanning range of 4 to 75.000 degrees with a two-theta step of 0.026 at 13.7700 seconds per step. The intensity of the diffracted X-rays was continuously recorded automatically on a chart and the appropriate 2-Theta (Θ) and intensity values were then obtained [7].

iv. **Determination of Ammonia and Phosphate**

This was carried out using the colourimetric method and was determined using a photometer (Palintest Photometer 7100). For ammonia 5mL of reagent NH4-1was pipette into a test tube. 0.20mL of the pre-treated sample was added and mixed. 1 level blue micro spoon of reagent NH4-2 was added and shaken vigorously until the reagent was completely dissolved. It was then left to stand for 15 minutes. Then the sample was filled into a cell and measured in the photometer.

For phosphate 5ml of the pre-treated sample was pipette into a test tube. 5 drops of PO4-1was added and mixed. 1 level blue micro spoon of PO4-2 was added. It was then shaken vigorously until the reagent was completely dissolved. After that, it was left to stand for 5 minutes. Then the sample was filled into the cell and measured in the photometer.

Results and Discussion

This section deals with the results obtained from the physiochemical analysis of the domestic wastewater, preliminary analysis of the various clay minerals and selection of a suitable adsorbent and the thermal activation of the selected adsorbent.

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Table 4: Preliminary analysis of the raw domestic wastewater using various clay minerals (Plastic clay, Red clay, Barite and Feldspar) as adsorbents.

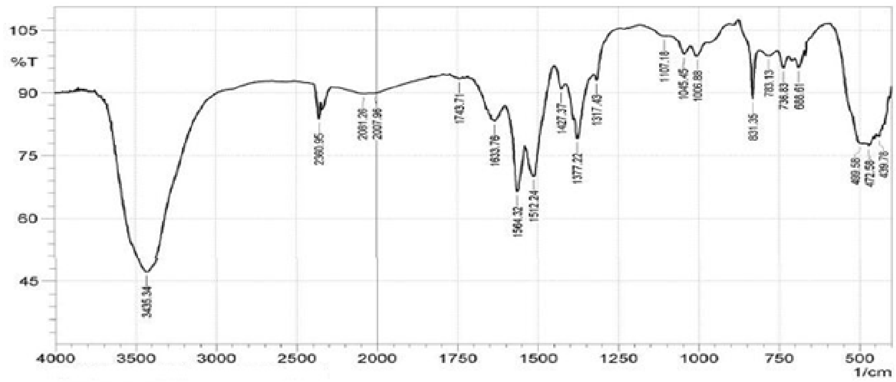
Parameter	Raw Sample	Plastic Clay	Red clay	Barite	Feldspar	WHO Limit for effluent	F.M.E limit for effluent
PH	6.689	7.443	7.49	7.601	7.527	6.5-8.5	6-9
Temp (°C)	26.10	24.40	24.00	23.70	23.30	< 40	<40
Conductivity	580.0	548.0	570.0	672.0	643.0	1250	
NH_4^3	0.50	0.15	0.25	0.25	0.05	0.1	0.2
PO_4^3	14.40	3.00	2.20	0.60	3.75	5	5
Fe^{2+}	2.00	1.15	0.80	0.25	1.05	1	
COD	750.00	120.00	300.00	200.00	280.00	80	80
BOD	350.00	40.00	120.00	80.00	120.00	30	50
TSS	1000.00	40.50	40.00	12.00	41.00	30	30

Source: F.M.E: Federal Ministry of Environment

Table 5: Shows the percentage removal of ammonia and phosphate by each of the various clay minerals (Plastic clay, Red clay, Barite and Feldspar).

Parameter	**Plastic Clay**	**Red clay**	**Barite**	**Feldspar**
NH_3	70.00%	50.00%	50.00%	90.00%
PO_4^3	79.17%	84.72%	95.83%	73.96%

From table 5 it was observed that the percentage removal of ammonia and phosphate when feldspar was used as the adsorbent gave the best results. Based on this data feldspar was selected as our preferred adsorbent

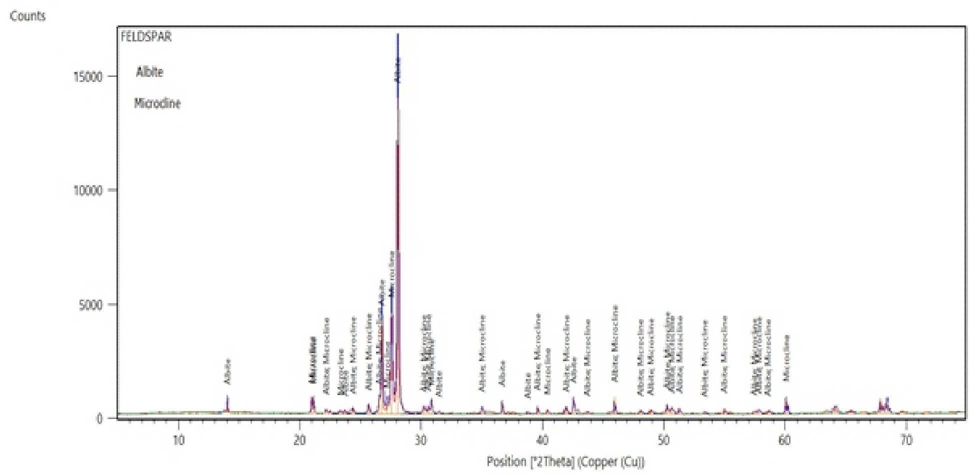


c. **FT-IR analysis of feldspar adsorbent**

The FT-IR analysis of the raw feldspar adsorbent was carried out in order to identify the functional group present in the feldspar adsorbent. Figure I show FT-IR analysis of feldspar adsorbent. The FTIR spectra for the raw felspar gave a characteristic wide band of 3456.55 cm-1 which corresponded to the O-Hhydroxy group. Likewise, the band observed at 2360.95 cm-1 corresponded to the N-H group stretching. The band at 1637.62cm-1 corresponded to C=O as reported by[3].

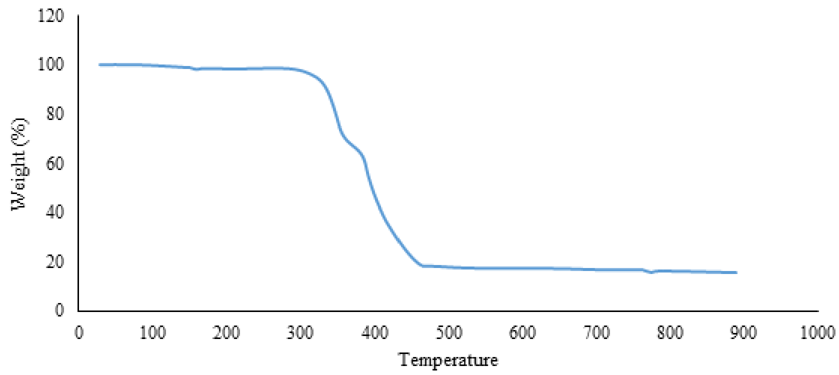
d. X-ray diffraction (XRD) analysis of feldspar adsorbent

The XRD analysis of feldspar adsorbent was carried out in order to determine the adsorbent crystallinity. From the XRD analysis in Figure II, it was observed that the characteristics feldspar peaks were observed at 2 theta (ɵ) = 28 °C.



e. Thermal activation of the feldspar adsorbent

The feldspar adsorbent was calcined (thermally activated). The thermal activation of the feldspar improves its chemical and physical properties of the feldspar adsorbent by liberating of inter-layer water molecules and organic matter as such leading to improved surface area and pore volume [8].Figure III shows the Thermogravimetric analysis (TGA) plot, of feldspar adsorbent.



From the plot it was observed that the region from 25 °C to 470 °C correspond to the loss of interlayer water molecules and volatile organic matter. At temperatures above 500 °C, it was observed that a straight-line trend occurred indicating that thermal activation of feldspar occurs at temperatures above 500 °C. The Feldspar adsorbent was calcined at three different temperatures of 500, 600 and 700 degrees for 1hr. The BET analysis of the thermally activated feldspar at different temperatures is as presented in Table VI.

Table 6: BET of Thermally activated felspar

Sample	Surface Area (m^2/g)	Pore Volume (cc/g)	Pore width (nm)
Raw Feldspar	332.8	0.218	2.433
Feldspar at 500 °C	352.5	0.216	2.433
Feldspar at 600 °C	533.9	0.272	2.105
Feldspar at 700 °C	418.3	0.211	2.100

Conclusion

In conclusion, the various clay minerals considered in this study all have the potential to be used as adsorbents where Plastic clay has a percentage removal of 70.00 and 79.17% for ammonia and phosphate, Red clay has a percentage removal of 50.00 and 84.72% for ammonia and phosphate. Barite has a percentage removal of 50.00 and 95.83% for ammonia and phosphate and Feldspar has a percentage removal of 90.00 and 73.96% for ammonia and phosphate. However, the use of feldspar as an adsorbent has proven to yield the highest percentage removal for the simultaneous adsorption of ammonia and phosphate amongst the clay minerals considered.

Similarly, the comparative BET analysis of the different clay minerals showed that feldspar has the largest surface area and pore volume which are good indicators of a good adsorbent. The thermal activation of the feldspar adsorbent further increases the adsorption capacity of the raw feldspar adsorbent as the thermal treatment increases the feldspar surface area and pore volume. Based on the clay minerals considered feldspar have proven to be a preferred choice based on its surface area, pore volume and percentage removal of ammonia and phosphate.

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