# EVALUATION OF SORPTION BEHAVIOUR OF *Acacianilotica*LEAVES FOR REMOVAL OF HEAVY METALS FROM AQUEOUS SOLUTION

Supported by tetfund TERTIARY EDUCATION TRUST FUND

**[2]H. Ataitiya, [1]O. N. Maitera & [1]A. Akinterinwa**
[1.] Department of Chemistry ModibboAdamaUniversity Yola Adamawa State Nigeria.
[2.] Department of Chemical Sciences Federal University Wukari Taraba State Nigeria.

**Received: April 16, 2024 Accepted: June 28, 2024**

**Abstract**: Acacia nilotica was modified using eleven different treatments using ethanol, 0.1mol NaOH,0.1mol HCl and 10% glutarldehyde, it was reacted differently for 12hours on a magnetic stirrer then filtered and washed with distilled water till neutral pH. Preliminary studies was carried out, where 0.5g of biomass was reacted with 10ppm of Cadmium and Lead at pH 6 and stirred for 1hour then filtered, AAS was used for the determination of residual metal concentration to select the best treatment for batch sorption studies. Treatment 11 was selected as the best with 98% and 94% for Cadmium and Lead. Batch sorption studies were carried out to study the effects of pH, dosage, initial metal concentration and the effects of time on sorption capacity. The optimum sorption condition was found at pH 3, with 1g biomass dosage of Cadmium and Lead at 30mins equilibrium time. Characterization was also carried on the biomass using SEM-EDX, TGA and FTIR respectively. Acacia nilotica biomass showed a flaky rough surface with elongated thread like structures which contains carbon and oxygen, the amorphous nature of sorbent with the presence of Amine, Amide, Carboxyl and hydroxyl functional groups were present on the biomass. Thermal stability and decomposition of the biomass was also studied as a function of temperature. There was decrease in mass of the biomass in 4 steps which is the loss of volatile substances and moisture contents from $50^0$c to $150^0$c.

**Keywords:** Acacia nilotica; Biomass; Characterization; Metal ion; Sorption

## Introduction

High levels of toxic metals in the environment have become a worldwide issue for some time now (Pellera *et al.,* 2011).these heavy metals are introduced into the environment by human activities such as Mining, Electroplating, Petrol refining etc. they may pose health issues especially at high concentration (Ibrahim et al., 2010). Therefore it is very important to remove heavy metals from the environment, there are various techniques for removal of heavy metals from the environment this includes Membrane filtration, Ion exchange, Flocculation Adsorption and Chemical precipitation (Jimoh *et al.,* 2011). These techniques are costly so Adsorption has being proposed as a cost effective and efficient method for remediation. Recently many industrial and Agricultural waste have been studied as alternative adsorbent for clean-up and they are efficient, effective, and inexpensive, example Tea waste (Mahyi *et al*., 2005), Neem leaves (Vijay *et al*.,2013), Hazelnut hull (Ali *et al*., 2012), Maize cab and husk, Sun flower etc. *Acacianilotican* (Gabaruwa): Binomial name is *Vachellianilotica* (Kyalangalilwa *et al*., 2013) and by the vernacular names gum Arabic tree, babul thorn mimosa, thorny acacia and gabaruwa in hausa (Integrated Taxonomical Information System). Is a flowering tree in the family fabaceae. It is native to Africa, the middle East and the Indian sub-continent.. Acacia nilotica is a tree 5-20 meters high with a dense spherical crown, stems and branches usually dark to black colored, (www.fao.org 2017)

**Uses:** The plant has antimicrobial properties, effective against bacitracin producing microorganism (Ajayi *et al*., 2016). In traditional medicinal use the leaf sap is used as antidote against venoms, stings and bites. There is not a lot known about the physiological and agronomic aspectsof this plant, phytochemical observation shows that Alkaloids, Flavonoids, Tannins, Saponins, Anthraquinones, Anthocyanosides, Cardic glycosides, cardenolides and Steroidal nucleus are present (Bickel Sandkloter 2001).

This plant *Acacia nilotic* is a very good plants used as biosorbents due to its large surface area, their ion-exchange, adsorption, high versatility, easily manipulated properties, and low costs materials in water pollution remediation processes (Bhattachary *et al.,* 2014). The objective of this research is to develop low-cost sorbent for removal of Cadmium and Lead from aqueous solution, the effects of pH, initial metal concentration and dosage were studied , the equilibrium data were fitted into Freundlich and Langmuir isotherm models, the effects of time was study to know the rate of the reaction. SEM-EDX, XRD, TGA, and FTIR analysis were performed to elucidate the sorption mechanisms.

## Materials and Methods

### *Plant Materials collection and treatment*

The plants *Acacia nilotica* leaves was collected from Ngbalang along Numan – Guyuk road in Adamawa state the leaves was authenticated in Plant Science Department of Modibbo Adama University Yola it was washed with water under a running tap then rinsed with distilled water to remove adhering particles, it was dried at room temperature for 14days then it was ground and sieved and keep in air tight container and was used in this Research to determined residual Heavy Metals (Cadmium and Lead) concentrations aqueous solutions. (Igwe & Abia 2006)

**Figure1**: Acacia nilotica leaves

***Modifications and Characterization of plant Material.***
Eleven modifications methods as listed in Table 1 were used. Five grams*Acacianilotica* was soaked in ethanol for three days it is being shaken at some interval for proper phytochemical extraction then it was further reacted with 500ml various chemical solutions separately then stirred for 12hours, the modified biomass were then filtered washed with distilled water till neutral pH and dried in an oven at $60^0$c. the biomass prepared were powdered, the chemically modified biomass using Ethanol, 0.1mol NaOH and 10% Glutaraldehyde was selected for characterization and batch sorption studies after preliminary studies.

***Fourier Transforms Infrared Analysis***
The functional groups as well as the binding sites were examined using Shimadzu FTIR spectrometer 8400S, the spectrum was obtained at the range of 400 - 4000$cm^{3.}$

***Scanning Electron Microscopy- Electron differential Xray (SEM-EDX)***
The surface morphology and elemental composition was probe using SEM-EDX machine, 500- 2000 magnifications were used

***Thermogravimetric Analysis (TGA).***

TGA analysis is a technique used to study thermal decomposition and weight changes of material as a function of temperature, NAN JING DA ZHAN instrument was used at 10 degree /min weight loss was measured as temperature was increased from room temperature to 280degree.

***Heavy metals stock solution*n**
Method of stock preparation of Heavy Metals was adopted from Dawodu *et al.*, (2012). A stock solution of 1000 mg/L of Heavy metals was prepared by dissolving 1.6308 g of Lead and 1.790g of Cadmium in 1000 cm3 volumetric flask and made up to the mark with deionized water. Several standard concentrations (10, 20, 30, 40 and 50 mg/L).

***Batch Sorption Studies.***
The experiments were carried out in the batch mode for measurement of adsorption capacities. From 100ppm of Cadmium and Lead ions solution, 50ml was measured into 250ml conical flask and 0.5g of *Acacia nilotica* biomass was added and shaken for two hours at the range of pH 1-9 for lead and Cadmium under room temperature on a magnetic stirrer. The optimum pH was determined. The solution pH was adjusted using 0.1mol/L of HCl and 0.1mol/L NaOH respectively. The effect of initial metal concentration was studied from 10ppm- 50ppm. The residual metal ions concentrations were determined using atomic absorption spectrophotometer (AAS), Percentage removal was calculated using

% Removal=$\frac{CO-Ce}{CO} \times 100$

**Results and Discussions**
The sample *Acacia nilotica* leaves was subjected to eleven (11) different modifications as shown in Table 1 below

**Table 1:** List of Modifications and % Removal for Cadmium and Lead

| Treatments | Chemical Modifier Used | Lead % | Cadmium % |
|---|---|---|---|
| | | ACA | ACA |
| 1 | Raw Biomass + Ethanol | 51.96 | 87.96 |
| 2 | Raw Biomass + Ethanol + 10% glutaraldehyde | 67.13 | 93.07 |
| 3 | Raw Biomass + 10% glutaraldehyde | 55.30 | 67.13 |
| 4 | Raw Biomass + 0.1M NaOH | 81.04 | 85.30 |
| 5 | Raw Biomass + 0.1M HCl | 87.36 | 81.04 |
| 6 | Raw Biomass + 0.1M NaOH + 10% glutaraldehyde | 62.09 | 87.36 |
| 7 | Raw Biomass + 0.1M HCl + 10% glutaraldehyde | 62.06 | 82.07 |
| 8 | Raw Biomass + Ethanol + 0.1M NaOH | 57.02 | 82.06 |
| 9 | Raw Biomass + Ethanol + 0.1M HCl | 87.36 | 57.02 |
| 10 | Raw Biomass + Ethanol + 0.1M NaOH + 10% glutaraldehyde | 89.76 | 92.42 |
| 11 | Raw Biomass + Ethanol + 0.1M HCl + 10% glutaraldehyde | **94.93** | **98.64** |

**ACA =** *Acacia nilotica*

***Batch Sorption Studies***

The experiment were carried out in the batch mode for the measurement of adsorption capacities. Working solutions were prepared from stock solution ranging from 10-50ppm of both cadmium and lead solutions, 50ml was taken into 250ml conical flask and 0.5g of *Acacia nilotica* was added covered with aluminum foil and shaken for 2hours at room temperature and 180rpm speed.

***Effects of Solution pH***

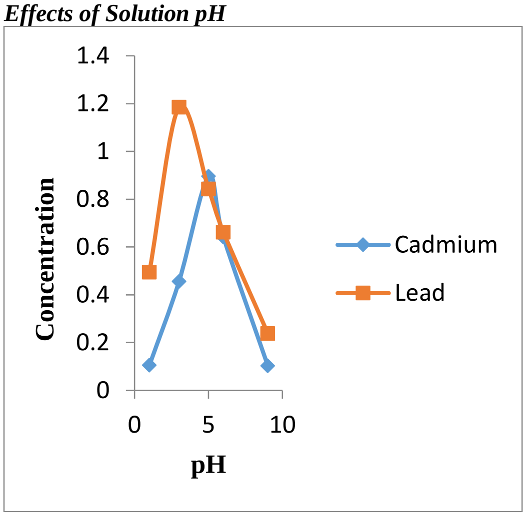


**Figure 2:Effects of pH on Adsorption of Cadmium and Lead on Acacia**

The result in Figure 2 above showed that highest percentage removal for Lead was 99.82% at pH 3 and that of Cadmium was 99.78% at pH 5, the sorption capacity increases with increase in pH until it reached maximum values at pH 3 for lead and pH 5 for cadmium , at higher pH values from 3-5 the metal biosorption increased due to the ionization of functional groups on the cell surface that serves as the binding sites related to isoelectric point for protein of the cell wall which may be due to repulsive effects between biomass and metal ions in solution got stronger ( Guo- Xiu Xing *et al*., 2006). At low pH values, more $H_3O^+$ ions will be available to available to compete with Cadmium and Lead ions for the adsorption site of the biosorbents , at low pH also most of the functional groups are protonated. The results obtained from all the adsorbents showed that optimum removal was achieved between pH 3-5 which may be due to electrostatic attraction between the adsorbate and adsorbent (Bolat *et al*., 2010). From the results obtained the increase in adsorption at low pH could be explained that acidic pH Favors cation exchange that occurs between the sorbate and sorbents ( Dawodu *et al*., 2012) at low pH values , the solution is strongly acidic, where a lot of protons are thus present in solution for cation exchange with Cadium and Lead ions further increase in pH will leads to precipitation of the metal ion into the solution.

***Effects of Initial Metal Concentration***

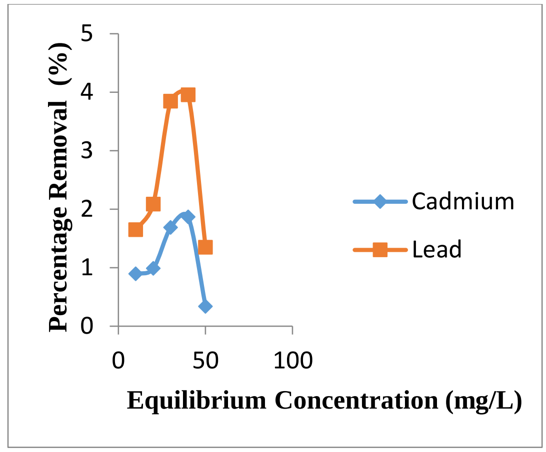


**Figure 3: Effects of Initial Metal Concentration on *Acacianilotica*.**

The effects of initial metal concentration on adsorption of Cadmium and Lead for *Acacianilotica* as shown in Figure 3 above. The percentage removal increase when initial concentration increases from 89.96% at 10mg/L to 99.97% at 40mg/L then with further increase in the metal concentration leads to decrease in the percentage removal to 84% at 50mg/L, these findings indicates that the sorbents is effective in dilute metal solutions and can remove metal concentration up to 10ppm similar results was reported by Kumar 2012. Increase in concentration leads to strong competition for adsorption site (Bhattacharyya and Gupta , 2011; Jiang *et al* 2011).

***Effects of dosage***

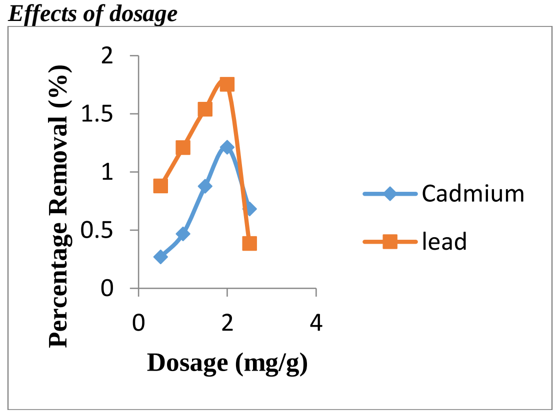


**Figure 4: Effects of Sorbent dose of Acacia nilotica**

Theeffectsofadsorbents dosage on percentage removal of Cadmium and lead for *Acacianilotica* can be seen in figure 4 above which showed that as the dose increases from 0.5g - 2.5g adsorption increases with increase in adsorbents dose the percentage removal increase from 42.20%for Cadmium and 46.87% for Lead with 0.5g biomass to 86.50%for Cadmium and 84.23% with 2.0g of biomass then the percentage removal reduced to 67.89% for Cadmium and 54.78% for Lead. The increase in adsorbent dose with increase in adsorption is generally due to availability of wider surface area and availability of binding sites. The result obtained is similar to results reported by Torab-Mostaedi *et al*., 2013 which looked at biomass dosage effects on biosorption of Cd(ll) and Ni(ll) by grape fruits peel . Additional available adsorption sites which leads to higher capacity for adsorption (Nanganoa *et al*., 2014).

***Effect of Contact Time on Adsorption of Cadmium and Lead.***

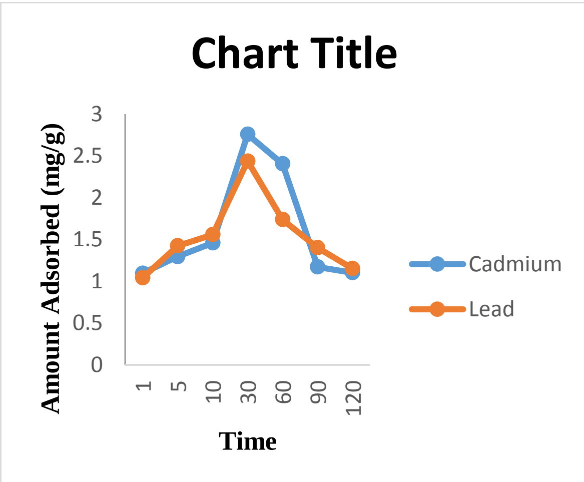


**Figure 5. Effects of Time on *Acacia nilotica*.**

The effects of Time on sorption capacity of *Acacia nilotica* as shown in Figure 5 above shows that there was increase in percentage removal of Cadmium which is 54.00% and 53.89% for Lead at 1min to 95.60% for Cadmium and 95.24% for Lead at 30mins agitation time, then there was decrease in percentage removal to 87.70% for Cadmium and 84.67% for lead after 60mins contact time, the reaction has reached equilibrium with 30mins agitation then with further increase in contact time from 30min to 60min, the further increase in contact time to 120 mins leads to decrease in percentage removal of Cadmium to 62.76% and 67.74% for Lead.The increase in contact time increases the removal efficiency until equilibrium concentration is reached, The equilibrium state occurs when the biomass is saturated with metal ions. Moreno- Pirajan *et al*., (2011) found out that the removal of heavy metals (Mn, Fe, Ni) from waste water by activated carbon from coconut shell was increased with increase contact time and reached a maximum at just 8miniutes of solution agitation.

**Adsorption Kinetics**

***Adsorption Rate Curves***

The plots in Figures 6a and 6b showed that adsorption capacity increased with increase in contact time until equilibrium was achieved for Pb and Cd at 30 minutes using *Acacianilotica* biomass.

The rate curves in figure 5. for adsorption of Cadmium and Lead using *Acacia nilotica* showed that the adsorption capacity increased from 1-30mins until the equilibrium was reached at 30mins. Adsorption rate appeared to be very fast at the beginning of the reaction these could be attributed to the fact that the available adsorption sites were sufficient at the starting point. reported similar results. The highest adsorption capacity of both Camium and Lead were recorded at 30mins given as4.992mg/g and 4.956mg/g using *Acacia nilotica* respectively. The ions adsorbed occupied the active sites on the adsorbent which as the contact time increased, the active sites on the adsorbent becomes saturated similar studies was carried out by Ahmed et al., (2015) on characterization and application of Kaolinite for removal of copper ions from environmental waste water samples.

***Adsorption kinetic model***

Adsorption kinetics model data were obtained from the effects of contact time on adsorption of Cadmium and Lead using *Acacia nilotica* were fitted into the kinetics models in Figures 5a and 5b showed that the adsorption of cadmium and lead on*Acacia nilotica* was described by both first and second order models. Based on the correlation coefficient $R^2$, it was observed that the Blanchard Second-order gave a better fit than the Lagergren First-order kinrtice model. $R^2$ values are 0.999 Cadmium and0.988 Lead for *Acacia nilotica* compared to the Lagergren Pseudo-First order which gives lower $R^2$ values for *Acacia nilotica* ranging from 0.0134 Cadmium and 0.0003 for Lead. The high value for regression coefficient $R^2$ and rate constant for the Blanchard Second order showed that the equation best describes the entire adsorption process (Ahmed et al., 2015). This suggested that the adsorption may occur through a chemical process which may involve valence forces of shared or exchanged electrons (Ajay et al., 2015).

**Instrumental characterazations of adsorbents**

***FTIR Results for both Modified and Unmodified Biomass***

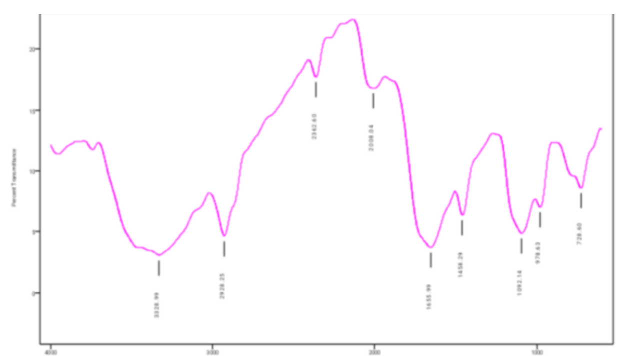

**Figure 6. Spectrum for Unmodified *Acacia nilotica***

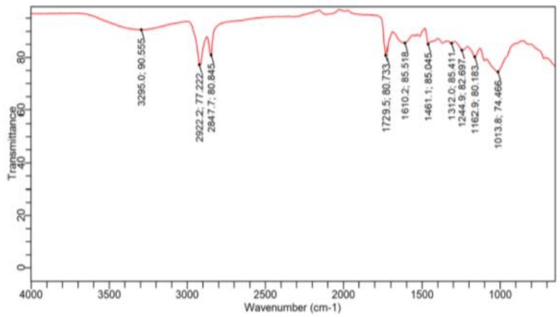


**Figure 7; Spectrum for Modified *Acacia nilotica***

The FTIR spectrum gives information about the functional group in the sample which can be use to identify chemical composition in the sample. Functional group can play a vital role in adsorption). The spectrum was recorded in the spectral range of 4000-600 cm-1. The biomass as shown in Figures 6 and 7 showed a broad peakat 3396 cm and 3328 respectively corresponds to O-H intra and interbonds stretching (3500-3000 cm-1). The sharp peaks at 2926, 2928 cm-1 is due to C-H asymmetrical stretching and bending vibrations. The double bond region has peaks at 1688cm-1 and 1655cm-1 which corresponds to C=O and –COOH, the peak at 1600cm-1 peaks corresponds to O-H, -COOH which might have overlapped N-H peaks . The peaks at 1458, 1423 are complementary peaks for C-C, O-H and C-H bendind and wagging respectively there was also absorbance peaks in the finger print region at 1200-600 cm-1 , There was addition of some peaks corresponding to N-H at 1602cm-1, C-N at 1028 cm-1, N-O at 1520cm-1 and C-O at 1013cm-1 which appeared in the modified biomass that were not present in the unmodified biomass, the FTIR is in line with the characteristics spectra reported by Sharma *et al*., 2020.

***Scanning Electron Microscopy Energy Dispersive Spectroscopy of Adsorbents.***

The results in Figures 8a.- 8d. showed the surface morphology and elemental composition of the adsorbents of *Acacia nilotica*.The SEM-EDS analysis allows imaging and EDS analysis in the SEM chamber on the spot areas selected, ascanning chemical images of a micro area of the sample surface using SEM is performed then some areas are analyzed identifying the presence or absenceof chemical elements using EDS.

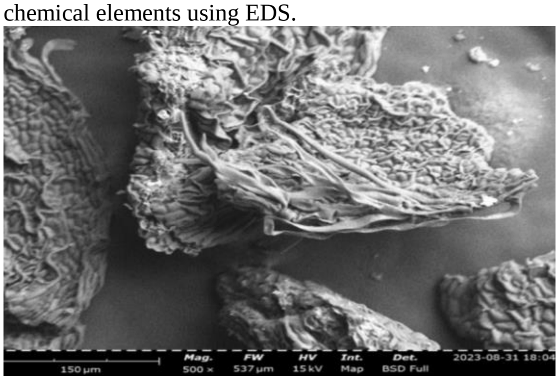


**Figure 8a. SEM-EDS of *Acacia nilotica* Leaf 500X Magnification**

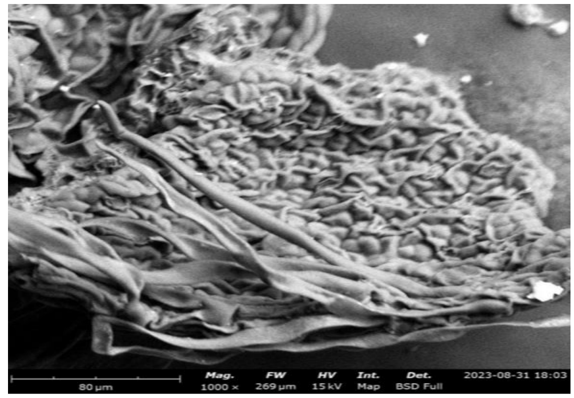


**Figure 8b. SEM-EDS of *Acacia nilotica* Leaf 1000 X Magnifications**

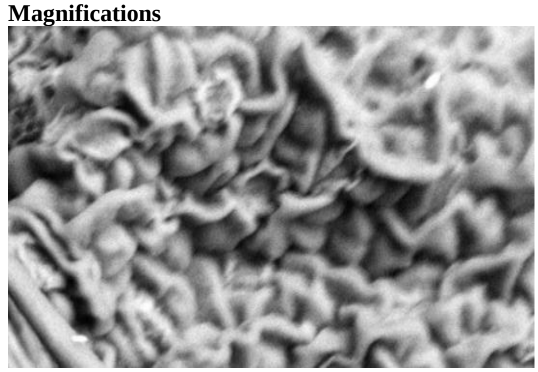

**Figure 8c. SEM-EDS of *Acacia nilotica* Leaf 2000 X Magnifications**

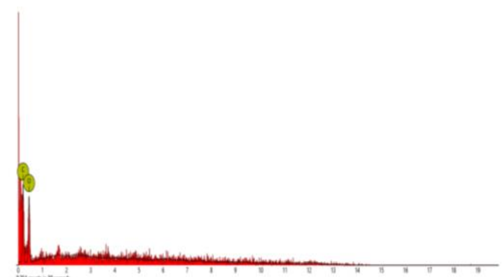

**Figure 8d. SEM-EDS of *Acacia nilotica* Leaf.**

The results in Figure 8a- 8d above showed morphological features of the biomass, from the result of SEM-EDS obtained it showed that the biosorbent has a flaky rough surface structures with elongated thread-like structures which are suggested to be hemicellulose ,there were well arranged pores on one side then flake with thread-like structures on the side and the edges showed more amorphous and branched structured biomass whice provides external surface of porous active sites which can enhance its adsorption capacity similar results were reported by (Moradi *et al* ., 2015) . The pore diameter of the biomass ranges from 2.456nm, 2.960nm, micro pore volume 0.239cc/g and pore area of 444.68m2/g and 488.063m2/g, the EDS result showed that *Acacia nilotica* biomass contained Carbon 77.02 % and Oxygen 22.98% . According to (Gupta and Nayak 2011) adsorbents with fine porous structures are good for adsorption process the EDS result showed that *Acacia nilotica* biomass contained Carbon 77.02 % and Oxygen 22.98% .

***Thermogravimetric (TGA) Analysis of the Adsorbents.***

Thermogravimetric analysis is a technique used to determine thermal decomposition and weight changes of materials as a function of temperature. Figures 9 showed *Acacia nilotica*

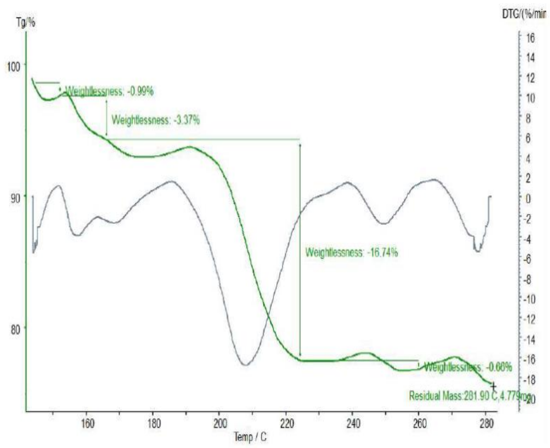


**Figure 9. Thermogravimatric Analysis (TGA)of *Acacia nilotica***

Figure1 0above showed Thermogravimetric analysis (TGA) of *Acacia nilotica* it was subjected to different temperature from 50 – 280 degree centigrade at interval of 10 degree/min there was an observed weight loss in 4 steps, the first loss was (0.99%) at 150C then (3.37%) at 168C then at $170^0$C- $230^0$C there was great loss of upto (16.74%) then finally at $260^0$C there was a weight loss of (0.66%). (7.23%), at $255^0$Cthe weight loss was recorded as (0.86%) and finally at $273^0$C (6,28%). Weight loss at temperatures below $100^0$C is usually due to loss of moisture content from the biomass, from the TGA analysis of *Acacia nilotica* biomass, the weight loss recorded started from $150^0$C which is due to loss of volatiles subtances, then with further increase in temperature there was another weight loss which was recorded at temperature above $250^0$C and it is said to be due to degredation of hemicellulose and cellulose in the samples Gerola *et al.,* 2016. Similar trend of weight loss were reported for other biomass. *Acacia nilotica* showed good thermal stability.

**Conclusion and Recommendation**

The results obtained from this study on *Acacia nilotica* leaves can be used as an alternative low-cost biosorbent for the removal of cadmium and lead from aqueous solution. The equilibrium data fitted well with Langmuir isotherm model with monolayer sorption. The presence of functional groups in the sorbents favors metal ion binding. The sorption and characterization studies showed the feasibility of *Acacia nilotica*leaves for water treatment. It is recommended that the effectiveness of the biomass to treat industrial effluents be evaluated.

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