
Application of Activated Carbon Prepared From Sugarcane Bagasse for Lead Removal from Wastewater

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ABSTRACT

Agricultural wastes are known to cause various pollutions to the environment and as such their applications for the removal of pollutants from wastewater is helpful. Sugarcane bagasse activated carbon was produced by two step activation methods by subjecting it to the carbonization temperature of 350°C at a heating rate of: 10°C/min for the residence time of 1hour and impregnated with H₃PO₄ at the ratio of 2:1. The batch adsorption experiment was under agitation time of: 10-120mins, adsorbent dosage of: 0.2-1.0g, initial lead concentration of: 10-60mg/l and operating temperature of: 30-50°C. The results indicated that the optimum dosage for the lead adsorption was 0.2g; while the optimum agitation time was 10mins. Furthermore, the optimum initial lead concentration was 60mg/l, while the optimum operating temperature was 50°C. However, the adsorption process fitted well to freundlich, temkin and Devinin-Redushkevich isotherms, with freundlich maximum adsorption capacity of 3.7705mg/g. However, the adsorption process was endothermic owing to the fact that adsorption of lead increases with increase in the operating temperature coupled with positive ΔH value of: 39.998kJ/mol. Moreover, the negative value of ΔG obtained signified that the adsorption process was spontaneous; while the positive value of ΔS suggested increase in the degree of freedom of lead ion onto sugarcane bagasse activated carbon. It was concluded that sugarcane bagasse can be effectively utilized to remove lead from wastewater.

Key words: Agricultural waste, optimum, maximum adsorption capacity and freundlich isotherm

INTRODUCTION

Nigeria is placed as the second biggest producer of sugarcane after Ivory Coast in West Africa, with Kano State contributing about 30% of the country's production; while the neighboring States such as, Kaduna, Katsina and Jigawa produce about 13%, 13% and 8%, respectively (FAO, 2013). This is correlated with the large volume of by-product produced after the

removal of sugar from sugarcane which is termed sugarcane bagasse (Adamu and Ahmadu, 2015; Amores *et al.*, 2013). The presence of high quantities of sugarcane bagasse in the environment leads to environmental pollution and blockage our drainage system, hence the need to use it in a more effective way so as to minimize environmental pollution. The sugarcane bagasse contains many functional groups

including hydroxyl and carboxyl that can undergo chemical treatment in order to upgrade its adsorption potentials for higher efficiency (Martincigh *et al.*, 2013).

On the other hand, heavy metals such as lead (Pb), cadmium (Cd), mercury (Hg), nickel (Ni) and chromium (Cr) have serious negative health consequences. However, they are available in the environment through industrial practices and are resistant to conventional biological treatments that could reduce them to less toxic or nontoxic entities (Martincigh *et al.*, 2013; Gil *et al.*, 2007; Aliyah, 2012). Meanwhile, lead uptake by human being can result in a serious health effects in the body. It can greatly affect the function of kidneys, reproductive system, liver, brain, behavioral impairment and nervous systems (Aliyah, 2012; Lata and Samadder, 2014).

In addition, there are several methods of heavy metals removal but the adsorption onto activated carbon was proved to be more effective owing to the ease with which it can be produced, utilized and regenerated. However, due to the high cost of commercial activated carbon, researches have now been tilted towards the use of agricultural wastes because of their abundances as well as the environmental pollution they generate.

However, there are several previous researches on the application of sugarcane bagasse for adsorption of lead. Salihi *et al.*, (2017) carbonized sugarcane bagasse at 900°C and then used adsorbent dosages of 2-16g using initial lead concentration of 100mg/l. The reaserch concluded that adsorption of lead followed langmuir isotherm with maximum adsorption capacity of 23.4mg/g with an optimum pH of 5. However, Martincigh *et al.*, (2013) utilized

sugarcane bagasse for lead adsorption at the adsorbent dosage of 0.05g and initial lead concentration of 10-100mg/l and found out that the lead adsorption fitted well to Langmuir isotherm maximum adsorption capacity of 23.8mg/g with optimum pH of 4.5. Similarly, Tao *et al.*, (2015) pyrolysed sugarcane bagasse at 800°C and then impregnated with sodium hydroxide, using initial lead concentration of 50-250mg/l and dosages of 0.2-2.0g. The study found that the lead adsorption followed Langmuir isotherm with maximum adsorption capacity of 135.54mg/g with optimum p H of 4; while the adsorption kinetics followed pseudo-second order kinetics.

MATERIALS AND METHOD

Materials

The materials employed in this research are grouped into: glassware, chemical reagents as well as equipments or apparatus.

Glassware: Measuring cylinder (50, 100 and 1000ml), Pipette (25ml) and 250 ml conical flask.

Chemical reagent: Distilled water, de-ionized water, 0.1M NaOH, 0.1M HCl, 1M H₃PO₄ all of analytical grade.

Equipment/ apparatus: Electrical weighing balance (Mettler P160N), Electric Furnace, Centrifugation machine, Magnetic stirrer, Scan electron microscopy (Phenom ProX), Fourier transform infrared spectroscopy (FTIR-8400S, Shimadzu), pH meter (Pocket size, Hanna), Atomic absorption spectroscopy (AA6800 Shimadzu), Membrane micro filter (0.45µm), Whatt man filter paper, 30 mesh sieve (0.60mm).

METHOD

Collection and preparation of sugarcane bagasse

The sugarcane bagasse was collected from Gaskiya Road along Nuhu Bamalli Polytechnic, Zaria of Kaduna State. It was then manually broken and then placed into electrical blender to reduce them into smaller sizes. They were then washed thoroughly with tap water, followed by distilled water to impurities after which it was then placed into electrical oven where it was oven dried overnight at 100°C (Gaikwad and Mane, 2013); Mohd Adib *et al.*, (2014); Pareira *et al.*, (2010). It was grinded using electrical blender and then sieved using standard sieve test (30mesh sieve).

Production of sugarcane bagasse activated carbon

The prepared sugarcane bagasse (SB) was then placed into an electrical furnace and carbonized at a temperature of 350°C at a heating rate of 10°C/min for a residence time of 1 hour. It was placed into a Desiccator and allowed to cool. The SB was then impregnated with concentrated H₃PO₄ (1M) at the impregnating ratio of 2:1 and stirred at 85°C for 3hours (Mohammed, 2015; Pradahn, 2011). It was then filtered with whatman filter paper and washed thoroughly with distilled to remove excess acid until the pH was 7. It was then placed into the oven and dried at a temperature of 100°C for the period of 4 hours to ensure proper drying.

Preparation of stock solution of Lead (100mg/l)

0.1599g of Pb(NO₃)₂ was measured and dissolved in 10ml of 0.1M HNO₃ placed in a 100ml volumetric flask. De-ionized water was then added to make it exactly 100ml. From this, working solutions of 10mg/l,

20mg/l, 35mg/l, 50mg/l and 60mg/l were prepared.

Determination of surface functional group and surface morphologies

The surface functional group for the sugarcane bagasse activated carbon was obtained using Fourier transform infrared spectroscopy (FTIR-8400S, Shimadzu); while the surface morphologies was determined using Scan electron microscopy (Phenom ProX).

Batch adsorption experiment

Batch adsorption of Pb onto SBAC

0.2, 0.6 and 1.0g of sugarcane bagasse activated carbon were placed into 250 ml conical flask containing 100ml of: 10, 20, 35, 50 and 60mg/l of Pb solution and agitated continuously in a magnetic stirrer at the speed of 150rpm. 10ml of Pb solution was withdrawn through 10ml syringe at the agitation times of: 10, 30, 60, 90 and 120mins, respectively. The residual Pb concentration was analyzed using Atomic Absorption Spectrophotometer (AA 6800). This experiment was also carried out at pH values of 2, 4, 6, 8 and 10 as well as operating temperature of 30, 40 and 50°C at the optimum conditions.

RESULTS AND DISCUSSION

The results obtained in this research are presented in Figures 1 to 12 as well as Tables 1 to 3. Figure 1 shows the Scan Electron Microscopy (SEM) micrographs of the sugarcane bagasse activated carbon. The Figure indicates the nature of pore present on the surface of the sugarcane bagasse activated carbon. From the figure, the sugarcane bagasse activated carbon surface possesses porous surface which is heterogeneous in nature. The presence

various pores could be attributed to the fact that the sugarcane bagasse was carbonized to a temperature of 350°C which play great role in librating the volatile matter as well as moisture content on its surface thereby

opening the pores. These pores play important role in adsorption of pollutant from wastewater onto the surface of the sugarcane bagasse activated carbon.

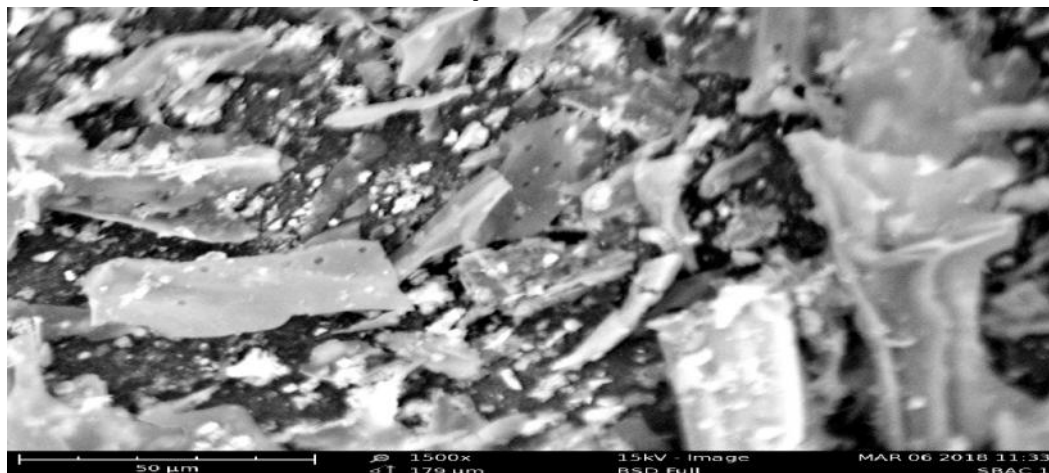


Figure 1: SEM micrographs of sugarcane bagasse activated carbon

Figure 2 shows the Fourier Transform Infrared Spectroscopy (FTIR) of sugarcane bagasse activated carbon. From the figure, some peaks at 3942.63, 3911.77 and 3826.90 cm^{-1} could be attributed to N-H stretch of amine groups; while the peak at 3333.1 cm^{-1} could be attributed to C-H of alkynes group, O-H of carboxylic acid, O-H of alcohols and N-H of amides. Similarly, the peak at 3271.38 cm^{-1} could be associated to the presence of C-H of alkynes group or N-H of amides group; while the peak at 3201.94 cm^{-1} could be attributed to O-H of carboxylic acid or N-H of amide group (Coates, 2000). In addition, the presence of O-H in the SBAC is a clear indication that it could bind with metal ions in aqueous solution (Sovattei *et al.*, 2013).

Moreover, the peak at 3132.5 cm^{-1} could be attributed to the presence of =C-H stretch of alkenes and O-H of carboxylic acid; while the peak at 2376.38 cm^{-1} could be

attributed to the presence of C C or C N. Similarly, the peak at 1627.97 cm^{-1} could be due to the presence of N-H of amines and amides groups; while the peak at 1411.94 cm^{-1} could be attributed to vinyl C-H in-plane bend of alkenes, carboxylate of carbonyl and carbonate ions. In addition, the peak at 1118.75 cm^{-1} could be attributed to C-F of alkyl halides, C-O stretch of alcohol and C-O-C of ethers. Similarly, the peak at 856.42 cm^{-1} could be attributed to p-distributed C-H of aromatic compounds; while the peak at 779.27 cm^{-1} could be due to the presence of C-Cl of alkyl halides of m-distributed C-H of aromatic compounds. The peak at 678.97 cm^{-1} could be attributed to C-Br of alkyl halides or C-H bend of alkynes; while the peaks at 601.81 and 563.23 cm^{-1} could be due to the presence of C-Br of alkyl halides. Moreover, the peak at 470.65 cm^{-1} , could be attributed to the presence of C-I stretch alkyl halides (Coates, 2000).

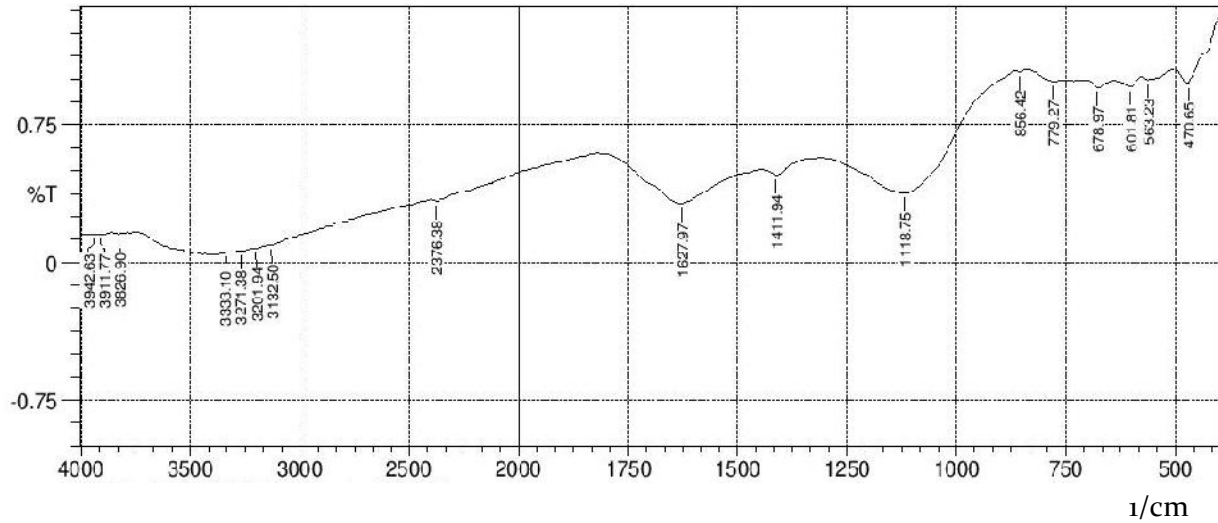


Figure 2: FTIR for SBAC

Figures 3 and 4 showed the effect of adsorbent dosages on adsorption capacity and removal efficiency at fixed initial lead concentrations of 10, 35 and 60mg/l. From Figures 3, it could be observed that, when the adsorbent mass increased from 0.2 to 1.0g, the amount of lead adsorbed decreased from 4.9985 to 0.9811mg/g, 17.4208 to 3.4867mg/g and 29.9660 to 5.9751mg/g, at fixed initial lead concentration of 10, 35 and 60mg/l, respectively. The result indicated that at fixed initial lead concentration, the amount of lead adsorbed onto the sugarcane

bagasse activated carbon decreased. This could be attributed to the fact that at higher adsorbent mass and unchanged initial lead concentration; the amount of lead in solution would not be able to satisfy all the sorption sites on the sugarcane bagasse activated carbon surface, hence the decreased in the adsorption capacity with increased in adsorbent dosage. This result is in agreement with Mohammed, (2015); Nsami and Mbadcam, (2013); Parate and Talib, (2015); Dargo *et al.*, (2014).

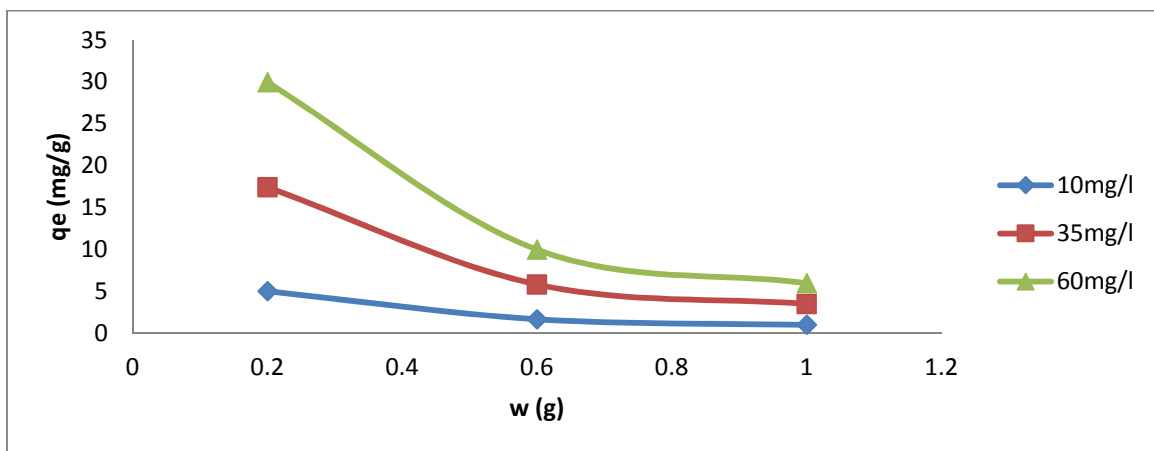


Figure 3: Effects of adsorbent dosage on adsorption capacity

On the other hand, it could be observed in Figure 4 that at the adsorbent dosage of 0.2g, 99.887% of lead ion was removed from the solution at fixed initial lead concentration of 60mg/l. This signified that the optimum dosage for the lead removal from the aqueous solution was 0.2g. However, the increased in adsorbent dosage

was accompanied with increase in the removal efficiency of lead onto sugarcane bagasse activated carbon. This could be attributed to the fact that the increase in the adsorbent dosage creates more adsorption sites for the lead removal, hence the increase in the removal efficiency.

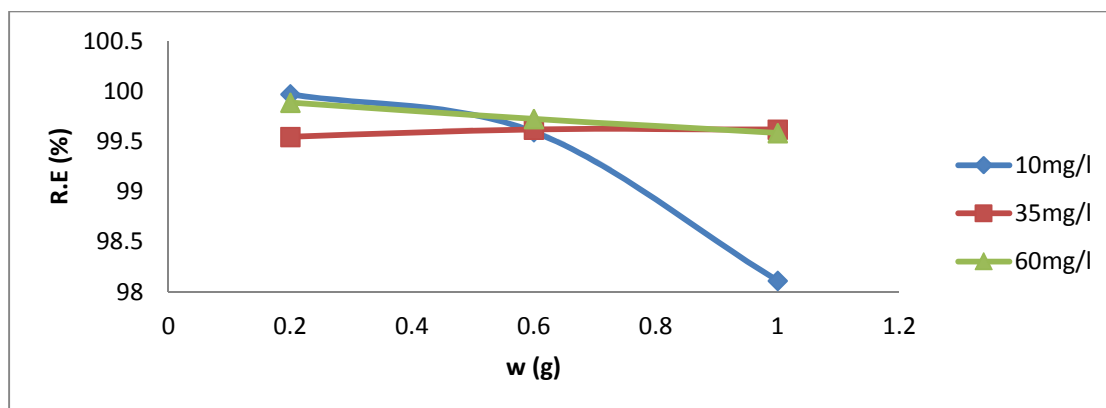


Figure 4: Effects of adsorbent dosage on removal efficiency

Figures 5 and 6 depict the effect of initial lead concentration on the adsorption capacity and removal efficiency. From Figure 5, when the initial lead concentration increased from 10 to 60mg/l, the adsorption capacity increased from 4.9985 to 29.9660mg/g, 1.6599 to 9.9724mg/g and 0.9811 to 5.9751mg/g for adsorbent dosages of 0.2, 0.6 and 1.0g, respectively. The result indicated that increased in the initial lead

concentration was accompanied with increased in the adsorption capacity. This could be attributed to the fact that increase in initial lead concentration at fixed adsorbent mass, yield increased in the driving force that neutralized the resistance of solute transfer from bulk solution onto the surface of the sugarcane bagasse activated carbon, hence the increase in the adsorption capacity.

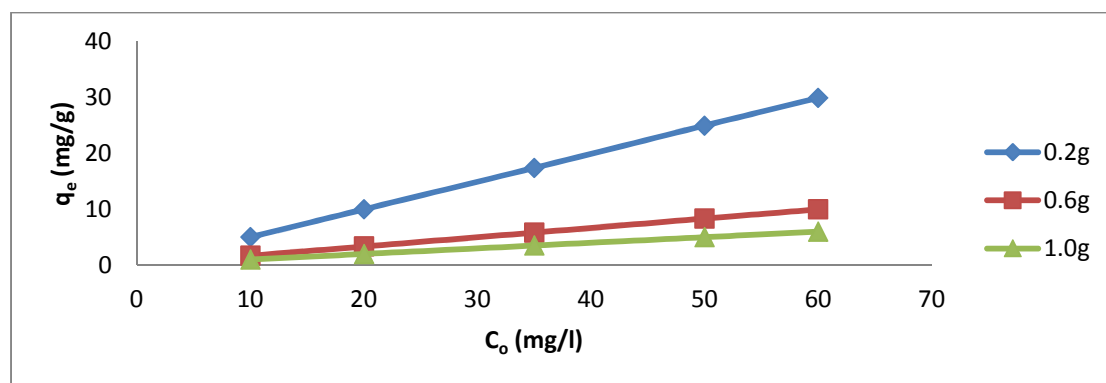


Figure 5: Effects of initial concentration on adsorption capacity

In Figure 6, it could be observed that when the initial lead concentration increased from 10 to 50mg/l, the removal efficiency increased from 99.969 to 99.979% and slightly declined to 99.887% at a constant adsorbent dosage of 0.2g. In addition, when the initial lead concentration increased from 10 to 60mg/l, the removal efficiency increased from 99.594 to 99.724% at a constant adsorbent dosage of 0.6g. However, when the initial lead concentration increased

from 10 to 60mg/l, the removal efficiency increased from 98.113 to 99.585%, respectively at unvaried adsorbent dosage of 1.0g. The increased in the removal efficiency with increased in the initial lead concentration signified the presence of multilayer adsorption in which the lead ion is adsorbed on already adsorbed layer. This result is in agreement with Mohammed, (2015).

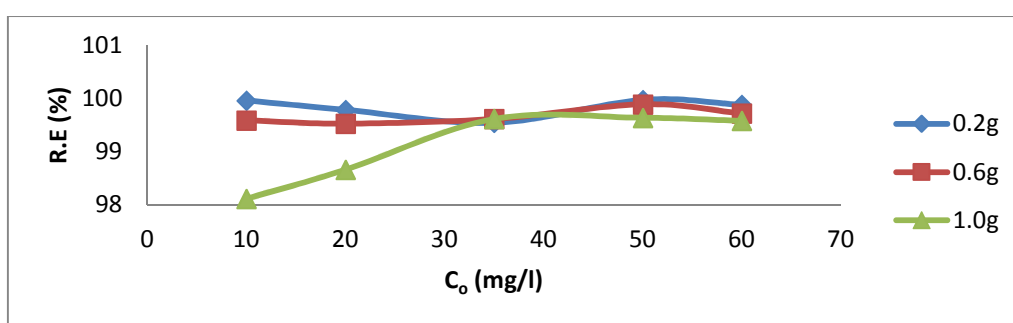


Figure 6: Effects of initial concentration on removal efficiency

Figures 7 and 8 show the effect of agitation time on the adsorption capacity and removal efficiency. From Figure 7 and 8, it could be seen that, the maximum adsorption capacity and removal efficiency of 29.9906mg/g and 99.969%, respectively were achieved within 10mins of agitation time. However, the fast rate of adsorption at the initial stage could be attributed to the presence of enough adsorption sites on the

adsorbent for lead uptake and the quantity of lead ion was high, but it decrease thereafter due to decrease in the sorption sites leading to limited transport of lead ions from the bulk solution onto the surface of sugarcane bagasse activated carbon. This observation is in agreement with the report by Surchi, (2011); Mehdizadeh *et al.*, (2014); Giwa *et al.*, (2013); Mohammed, (2015).

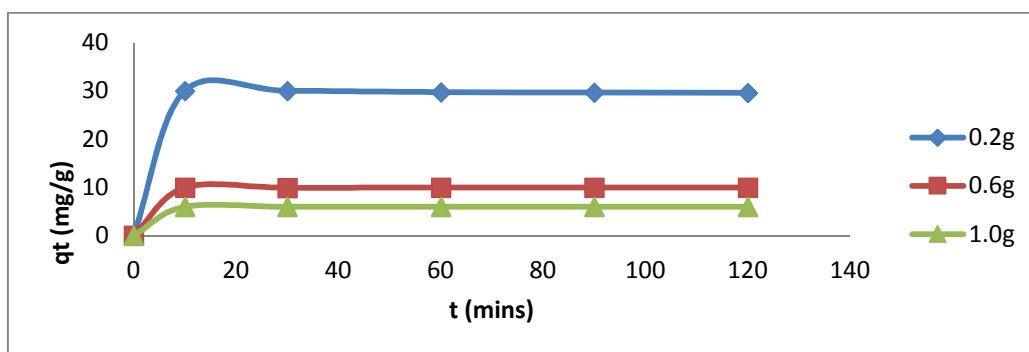


Figure 7: Effects of contact time on adsorption capacity

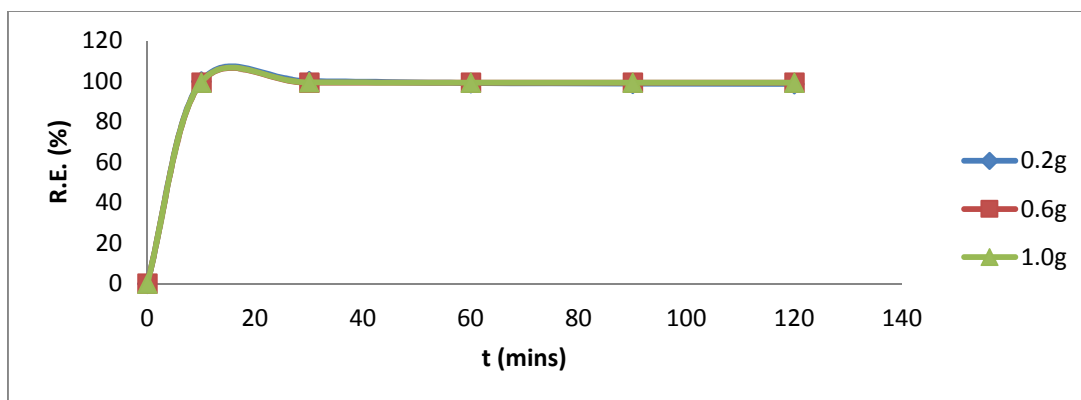


Figure 8: Effects of contact time on removal efficiency

Figures 9 and 10 show the effect of solution pH on the adsorption capacity and removal efficiency. From Figure 9, it could be observed that the minimum adsorption capacity of 29.8020mg/g was recorded at pH of 2 after which a maximum adsorption

capacity of 29.9995mg/g was obtained at pH of 4. This implies that the optimum solution pH for adsorption of lead from aqueous solution onto sugarcane bagasse activated carbon was 4.

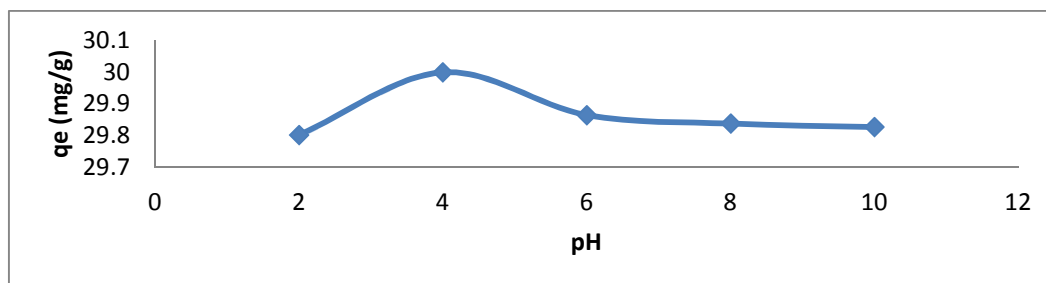


Figure 9: Effect of pH on adsorption capacity

Figure 10 presents the effect of solution pH upon the removal efficiency of lead onto sugarcane bagasse activated carbon. From the Figure, it could be observed that the minimum removal efficiency of 99.340% was recorded at pH of 2 after which a maximum removal efficiency of 99.998% was obtained at pH of 4. This

implies that the optimum solution pH for removal of lead from aqueous solution onto sugarcane bagasse activated carbon was 4. This result is in agreement with Ayub and Cahcanga, (2014); Salihi *et al.*, (2017); Moyo *et al.*, (2013); Akgun, 2005); Abdel-Ghani *et al.*, (2007).

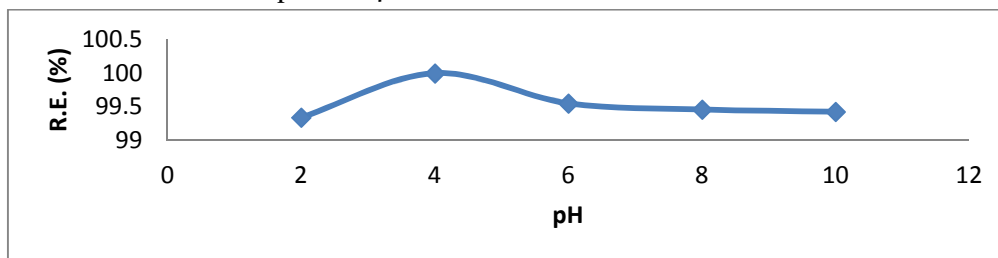


Figure 10: Effect of pH on removal efficiency

Figures 11 and 12 depict the effect of operating temperature on adsorption capacity and removal efficiency. From Figure 11, it could be discovered that when the operating temperature increased from 30 to 50°C, the adsorption capacity also increased from 29.8692 to 29.9516mg/g. This revealed that higher adsorption capacities were

obtained at higher operating temperatures which also implied that the batch adsorption process of lead adsorption onto sugarcane bagasse activated carbon was endothermic in nature. This observation was in agreement with Mengistie *et al.*, (2008); Chowdhury *et al.*, (2015); Rahman and Sathasivam, (2015).

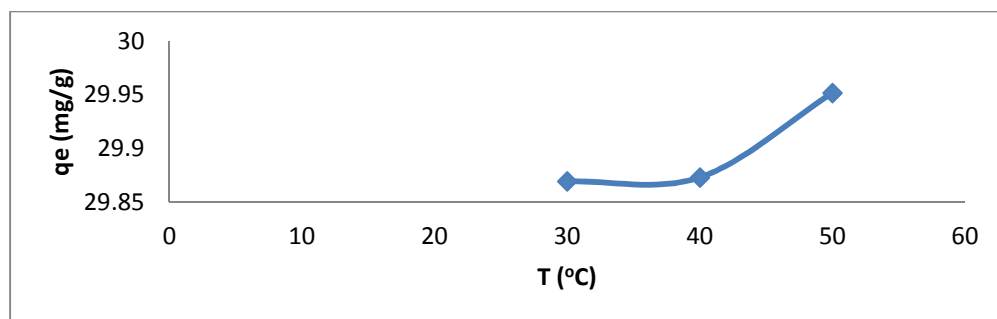


Figure 11: Effect of operating temperature on adsorption capacity

Fig. 4.63b shows the effect of the operating temperature on the removal efficiency of Pb onto SBAC. The figure revealed that when the operating temperature increased from 30 to 50°C, the removal efficiency also increased from

99.564 to 99.839%. This reveals that higher removal efficiencies were obtained at higher operating temperatures which imply that the batch adsorption process was endothermic in nature.

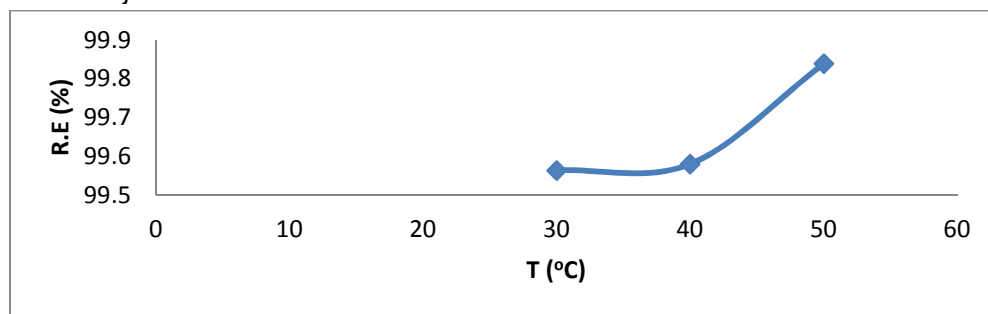


Figure 12: Effect of operating temperature on removal efficiency

Table 1 contains the adsorption isotherm data for the lead adsorption onto sugarcane bagasse activated carbon. The Table contained four adsorption isotherms: Langmuir, freudlich, temkin and Devinin-Redushkevich (D-R) model. The Table indicated that the batch adsorption data fitted well to freudlich, temkin and D-R isotherms with corresponding R² values of

0.9503, 0.9940 and 0.9503, respectively. The maximum adsorption capacity (q_m) of 3.7705mg/g was revealed by both the freudlich and D-R isotherms as could be seen from the Table. In addition, the 1/n value of 0.1954 revealed by the freundlich isotherm indicated normal and favourable adsorption. On the other hand, the mean free energy value of 1.0541kJ/mol (<8kJ/mol) revealed by

the D-R isotherms suggested that the adsorption process is physical (Itodo and Itodo, 2010). Moreover, with E values of $<16\text{kJ/mol}$ also suggested that the mechanism of ion exchange could be film-diffusion controlled. This observation is in agreement with Rahman and Sathasivam,

(2015); Dada *et al.*, (2012); Itodo and Itodo, (2010). Similarly, the temkin isotherm indicated the temkin adsorption potential value of 0.7334l/mg and heat of sorption values of 6.495J/mol as could be observed from Table.

Table 1: Adsorption isotherm data

Isotherm model	Parameters	Values
Langmuir isotherm	-b (l/mg)	0.0171
	q_m (mg/g)	16666.7
	R^2	0.0007
Freundlich isotherm	1/n	0.1954
	k_f (mg/g)	3.7705
	R^2	0.9503
Temkin isotherm	At (l/mg)	0.7334
	Bt (J/mol)	6.4951
	b	381
	R^2	0.9940
D-R isotherm	B_D (mol^2/kJ^2)	0.4500
	q_m (mg/g)	3.7705
	E (kJ/mol)	1.0541
	R^2	0.9503

Table 2 shows the adsorption kinetics of the lead adsorption onto sugarcane bagasse activated carbon. The Table indicated that the adsorption kinetic data fitted well to pseudo-second order than the pseudo-first order kinetics with R^2 values of 0.8357 and 1.0000, respectively. However, the rate constants, K_1 and K_2 for the pseudo-first order and pseudo-second order kinetics were: 0.0288min^{-1} and 0.1064 (mg/g/min), respectively with the corresponding equilibrium adsorption capacity values of: 0.0190 and 29.4985. Furthermore, the Table also shows that the intra-particle diffusion was not the only rate controlling step owing

to the fact the intercept ($C \neq 0$) which suggested that the intra-particle diffusion plot did not pass through the origin. This also implied that the adsorption mechanism is also dependent upon the movement of lead ions from the bulk solution to the outer surface of the sugarcane bagasse activated carbon. However, the transfer of solute in adsorption process could be by intra particle diffusion or external mass transfer or both (Mohammed, 2015). This result is in agreement with Surchi, (2015); Mohammed, (2015); Ferreira *et al.*, (2015); Gupta and Mote, (2014).

Table 2: Adsorption kinetic data

Kinetic model	Parameters	Values
Pseudo-first order	K_1 (min^{-1})	0.0288
	q_e (mg/g)	0.0190
	R^2	0.8357
Pseudo-second order	K_2	0.1064
	q_e	29.4985
	R^2	1.0000
Intra particle diffusion	K_{id}	-0.0547
	C	30.177
	R^2	0.8659

Table 3 shows the batch adsorption thermodynamic data for lead adsorption onto sugarcane bagasse activated carbon. The enthalpy change (ΔH) recorded positive value of 39.988kJ/mol indicating that the adsorption process was endothermic. However, the enthalpy value obtained also suggested that the adsorption process was both physical and chemical one (Rahman and Sathasivam, 2015). This result is in agreement with Chowdhury *et al.*, (2012); Surchi, (2011); Zhang *et al.*, (2017); with Rahman and Sathasivam, (2015); Mengistie *et al.*, (2008); Ferreira *et al.*, (2015). On the other hand, the change in entropy (ΔS) also

recorded positive value of: 0.0205kJ/mol, which implied increase in the degree of freedom of the lead ion onto the sugarcane bagasse carbon. Furthermore, it could also be attributed to the increase in affinity at solute-adsorbent interface as reported by Chowdhury *et al.*, (2012); Zhang *et al.*, (2017). On the other hand, the Gibbs free energy (ΔG) recorded negative values of: -11.546 to -14.948kJ/mol as the operating temperature increased from 30 to 50°C. The negative value of ΔG implied that the adsorption process was spontaneous and feasible (Purkait *et al.*, 2007; Zhang *et al.*, 2017).

Table 3: Adsorption thermodynamic data (SBAC-Pb)

T (K)	1/T (K^{-1})	ln k (g/l)	ΔG (kJ/mol)	ΔS (kJ/mol)	ΔH (kJ/mol)
303	0.00330	2.871	-11.546	0.0205	39.988
313	0.00319	2.292	-13.247	0.0205	39.988
323	0.00310	1.749	-14.948	0.0205	39.988

CONCLUSION

Based on the results obtained in this research work, it could be concluded that sugarcane bagasse can be effectively used to produce activated carbon and use it to remove lead from aqueous solution. The research revealed that the optimum dosage

for the removal of lead from the wastewater considered was 0.2g; while the optimum agitation time and pH were: 10mins and 4, respectively. The removal efficiency of lead onto sugarcane bagasse activated carbon recorded maximum value of: 99.998% at pH of 4. In addition, the adsorption isotherm

data fitted well to freundlich, temkin and D-R models with R^2 values of: 0.9503, 0.9940 and 0.9503, respectively. However, the freundlich and D-R isotherms recorded maximum adsorption capacity of 3.7705mg/g, implying that 1g of sugarcane bagasse carbon removed 3.7705mg of lead from aqueous solution. Similarly, the adsorption process was endothermic in nature owing to the fact that adsorption of lead increases with increase in the operating temperature which was justified by positive ΔH value of 39.998kJ/mol. In addition, the negative value of ΔG obtained signified that the adsorption process was spontaneous; while the positive value of ΔS suggested increase in the degree of freedom of lead ion onto sugarcane bagasse activated carbon.

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