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- EDUCATIONAL TECHNOLOGY
- ENGINEERING SCIENCES
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General Information

Background Brief: The Nigerian Journal of Technological Research (NJTR) is the official journal of the Federal University of Technology, Minna, Niger State, Nigeria. It was first published in June 1989.It has since made giant strides in its effort to provide an avenue for the dissemination of relevant modern up-to-date research information in the core areas of discipline available in The University at inception; namely, Pure and Applied Sciences, Engineering Technology, Environmental Technology and Agricultural Technology.

Philosophy: As a strictly scientific and technological journal, it tends to provide information on problem solving technology to its immediate environment and the international community.

Development: The journal being responsive to the dynamic nature of research and development in the Federal University of Technology, Minna and its environs, has widen its scope of information dissemination to include but not limited to Information Communication Technology (ICT), Management Technology, Educational Technology and Entrepreneurship. It has developed electronic communication procedures to ensure that, it has the capacity to reach a larger community at a faster rate. It is the anticipation of the journal that scientific data which will provide very current information to problem solving in the identified areas of The University program will be found in it.

Management: The Nigerian Journal of Technological Research has a unique management structure which enables it to carry out its functions promptly. This include; The Management Board, Editorial Board, Editorial advisory Board, Regional editors, Associate editors and a Business Manager. These groups bring to beer their vast knowledge which ensures the quality and reputable academic output from the journal.

Finally, The Management Board and The Editorial Board of The Nigerian Journal of Technological Research believe firmly in quality of information that will benefit mankind, hence their commitment to ensuring productivity and quality of information dissemination.

Editorial Comments.

This edition coming immediately after the twentieth anniversary of the journal is expected to give a consolidation to the standard and quality of research works show cased by the Editorial Board in the last issue. Consequently, the Board has painstakingly made selections of manuscripts considered worth being reviewed and consequently published. This is an indication of the desire of The Board to ensure that The Nigerian Journal of Technological Research remains valuable to both researchers and productive industry.

The relevance of verifiable research data is important for a steady developmental growth of any national economy. Manuscripts selected in this edition, intend to help focus on relevant issues which will ensure standard growth of an economy. Relevant data on flood or flood prone indices have been highlighted in addition to engineering factors that have the capacity to influence variables in the developing economy. The manuscripts have been well synchronized to ensure they have data relationships which will link the various authors.

As we close the year 2015, The Editorial Board will wish to inform all our authors that some new editorial procedures will be introduced commencing from volume 1 of 2016. This will require that authors visit the journal webpage early enough to avail themselves with the detailed information of reviewing procedures. This is important so that authors will derive full benefits emanating from the journal. Also, prospective authors should always visit the journal website, regularly on update news concerning the journal and manuscript developments.

The Editorial Board is very appreciative of all who have contributed to all editions and reviewers of our manuscript in the year 2015. We are ever grateful and hope to give you the best always. A happy and prosperous new year as you move into another calendar year.

Table of Contents

Page

Articles

Agriculture

Engineering

Environmental Technology

Entrepreneurship Technology

Educational Technology

Karim et. al 2015 vol 10 no 2:27-33

Optimization of lead (ii) ions adsorption on to chemically activated carbon from sugarcane bagasse.

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Abstract.

The adsorption of Lead (II) ion on to chemically activated carbon has been studied and optimized in a batch reactor system. The zinc chloride impregnated sugarcane bagasse was thermal activated in a fixed bed reactor in the presence of argon gas. The surface morphology, surface functional group and thermal stability were determined. The SEM micrograph revealed porous structures which enhance the sorption capacity of the developed activated carbon. The derivative thermal analysis (DTA) and thermogravimetric analysis (TGA) profile of the activated carbon were employed in the proximate analysis. The BET surface area shows a high microporous surface area and micropore volume of 840.38 m^2/g *and 0.30 cc/g respectively which aids sorption efficiency. The adsorption of lead ion was optimized using* 2^k *factorial experimental design where pH, mass of absorbent, temperature and time was studied in a batch reactor. pH has the highest positive impact on the adsorption of* Pb^{2+} *while considering single effect as studied using atomic absorption spectrophotometer. After estimating the main effects, the interacting factors affecting the removal of* Pb^{2+} *were determined by performing the analysis of variance (ANOVA) with R-Square of 99.97%. This study has demonstrated that Zinc chloride activated carbon has high affinity for lead ion sorption.*

Keywords: Sugarcane Bagasse, Activated Carbon, Characterization, Adsorption and Optimization.

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Introduction.

Heavy metals are individual metals that impede human health (Sabine and Wendy, 2009). Arsenic, cadmium, barium, chromium, mercury, lead, silver and selenium are most prominently identified common heavy metals which occur naturally with lower concentration as regard their environmental presence with hazardous effects at higher concentration (Sabine and Wendy, 2009). They are in areas where mining operation, fertilizer production plants, metal plating factories, chemicals and pharmaceutical industries are located (Bhattacharya *et. al.*, 2006).

Activated carbon is a carbonaceous material that possesses high degree of porosity and surface area for adsorption capacity. Granular activated carbon (GAC) and powdered activated carbons (PAC) are major types of activated carbon used as adsorbents for the removal of heavy metals from domestic and industrial wastewater owing to their large surface area, micro porous structure, nonpolar character, high adsorption capacity, high degree of surface reactivity (Nakagawa *et. al.*, 2003; Williams and Reed, 2006) and due to its economic viability.

Bagasse is a solid waste material removed during the consumption of sugarcane stem. Owing to their excellent specific surface areas and well-designed porosity, activated carbons are employed as adsorbent in organic chemicals and metal ion removal from environmental or economic concern from air, gases, and wastewater (El-Hendawy, 2003; Musa *et. al.*, 2012).

Wide range of agricultural precursors and waste products has been extensively investigated for activated carbon production. Such precursors include Olive stones (Rodriguez-Reinoso *et. al.*, 2001), pecan shells (Rengarag *et. al.*, 2002) palm seed (Garcia *et. al.*, 2003), rubber seeds (Legrouri *et. al.*, 2005) and Molasses (Khalili *et. al.*, 2000). Several studies have shown the advantages of chemical activation method over physical methods of activation owing to the fact that the former yields a material of high degree of porosity, improved surface area, and high yield (Deng *et. al.*, 2009; Guo *et. al.*, 2005).

This study focuses on chemical activation of sugarcane bagasse using Zinc chloride and pyrolysis in the presence of an inert environment, argon to reduce the degree of ash formation by oxidation and its characterization for morphology, surface area and pore size, surface functional group present and TGA.

Materials and methods

Reagents and Materials

Sugar bagasse was collected at sugarcane market at Kasua Gwari, Minna, Niger State, Nigeria. All the chemicals were of analytical grade with percentage purity in the range of 95-99.99% and were used without further purification. The chemical used include Zinc chloride, Lead nitrate, hydrochloric acid and distilled water.

Methodology.

Prior to activated carbon synthesis, sugarcane bagasse was soaked in hot water for 30 minutes. This is to ensure the removal of soluble impurities and dirt from the bagasse. This procedure was repeated three times, thereafter; it was washed with distilled water at ordinary temperature and dried at 110 °C in a static air oven. The dried samples were milled with the aid of a blender then sieved using 1.70 µm sieve size. 5.0 g of the sieved bagasse was added to a solution containing 15.0g of Zinc chloride dissolve in 50 ml of distilled water. The resulting solution was stirred using a magnetic stirrer and hot plate operating at a temperature of 100 °C for 1 hr then oven dried at a temperature of 110°C for 6 hrs. Zinc chloride impregnated sample was further exposed to Humidity for 5 hrs to enhance pore structure development. In an inert environment, using argon gas at a flow rate of 30 SCCF, the impregnated activated carbon was calcined for 45 minutes at 500 °C. The sample was removed after being cooled in the argon environment, crushed using mortar and pestle. Excess activating agent, Zinc chloride and residual organic mineral matters were removed by adding 250 ml of 0.6 N HCl then 250 ml of distilled water. The resulting Zinc chloride activated carbon from Sugarcane bagasse was then dried at 120 °C for 4 hrs and sieved with 150 µm sieve size, kept in an air tighten container and then characterized using SEM, FTIR, BET and TGA.

Adsorption Process

Batch adsorption experimental setup was employed in this study. 0.002mol/L standard solution of lead (II) was prepared from Lead (II) nitrate salt with distilled water. The pH of the solution was adjusted with the aid of 0.05 mol/L of sodium hydroxide. 25 ml of the solution was transferred into 100 ml beakers for each experimental batch following the 2^K experimental design and placed in a water batch shaker at specified temperature and time. After the completion of the adsorption process, 15 ml of the aliquots were drawn from each of the samples. The aliquots was then centrifuged at 6000 rpm for 1 hr then filtered using a filter unit into a sample bottle for Atomic Absorption Spectrophotometer. For easy adsorption process development, a number of influencing factors such as adsorbent dosage, temperature, pH, and residence time affecting the process were studied using 2^k factorial experimental design.

The percentage removal of Pb^{2+} was calculated by using the following equation:

$$\% R = \left(\frac{C_u - C_v}{C_u}\right) X\ 100 \qquad (1)$$

Where C_u and C_v are the Pb^{2+} concentrations in grams per litre initially and at a given time t respectively while %R denote percentage removal.

Table 1: Experimental ranges and levels of the factors used in the factorial design.

Independent variable	Coded symbol	Range and level	
		-1	+1
Adsorbent dosage (g/l)	A	0.3	0.6
Temperature (°C)	B	31	60
p^H	C	3	7
Residence time (hrs)	D	1	3

Results and Discussion.

The activated carbon and raw sugarcane bagasse were characterized for physiochemical properties such as moisture content, ash content, carbon content, volatile content, degradation temperature range, peak temperature and temperature at which 50 % of the material degraded.

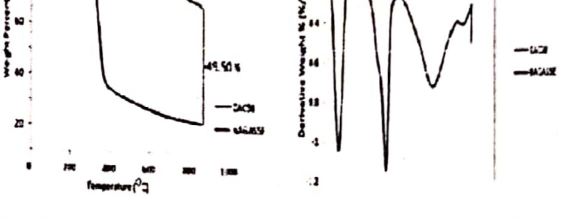

Fig 1: (a) TGA of Bagasse and Activated Carbon synthesized at 500 °C (b) DTG of Bagasse and the Developed Activated Carbon

Table 2: Proximate analysis of bagasse and the developed adsorbent under N_2 environment

Properties	**Sugarcane Bagasse**	**Activated Carbon**
Moisture Content (%)	2.271	7.187
Volatile content (%)	68.688	21.524
Ash content (%)	9.824	2.994
Peak Temperature, Tp (^{O}C)	354.07	626.74
T_{50} (^{O}C)	357.11	> 850
Degradation temperature profile (^{O}C)	163.11-466.58	179.69-775.41

Figure 1 (a) shows the TGA and DTG of both the raw activated carbon and Zinc Chloride activated carbon developed from sugarcane bagasse. The higher percentage moisture content of activated carbon is as a result of its exposure to light and humidity during the process of pore opening and enlargement. The TGA results show that activated carbon possesses an improved properties as regards it thermal stability. During the calcination process, huge quantities of amorphous material inclusive of cellulose material are removed from the bagasse. These said impurities lower the degradation temperature of material as shown in the bagasse TGA curve. The sugarcane bagasse was subjected to series of physical and chemical processes to remove water soluble impurities and HCl treatment after pyrolysis in the presence of argon gas to remove the accompanied amorphous material. The argon gas functions in two ways; evacuating gases from the quartz tube environment and also to reduce the degree of amorphous material that will be produced during the process by lowering the rate of oxidation process. These led to the reduction of ash and volatile content from 9.824 % to 2.994 % and from 68.688 % to 21.524 % respectively. The major weight loss in the bagasse profiles indicates the percentage loss of lignocellulosic compounds comprises of cellulose, hemicellulose and lignin with 68.688 %. The TGA/DTG reveals that 31.34 % of the lignin and other non-fibrous materials were removed during the pyrolysis experiment in argon environment. The degradation at 462-775.42 ^{O}C of the bagasse profile indicates the loss in the lignin content (Aksu, 2006). The peak, T_{50} and degradation temperatures of the synthesized activated carbon were greater than 850 ^{O}C as compared to raw bagasse with 357.11 ^{O}C. The improvement in the thermal stability of the synthesized activated carbon was as a result of the acid and chemical treatments during activation method and reduction in hemicellulose and lignin of the starting material.

In adsorption process, surface area, pore size and pore volume are important parameters which affect the rate of adsorption. In this research work, the surface area of the developed activated carbon from sugarcane bagasse was 840.38 m^2/g which is in accordance with the specification provided by Shoba, 2015. The micropores of 0.30 cc/g possessed by the adsorbent provides superb conditions for adsorption to take place due to simultaneous adsorption onto the pores of the adsorbent. The adsorption energy is the heat of adsorption determined at constant quantity of adsorbate adsorbed used in the estimation of design parameters during adsorptive process.

Table 2: BET parameters showing their respective quantity

PROPERTIES	VALUES
Average Half pore width (Å)	20.507
Adsorption energy (KJ/mol)	6.339
Micropore Volume (cc/g)	0.299
Micropore surface area (m^2/g)	840.376

The morphology of the synthesized activated carbon was characterized as shown in figure 3.

Fig. 2 SEM micrograph of activated carbon at varied magnification

The SEM micrograph of activated carbon was shown in figure 2. The images show an irregular particle shapes and pores. Adsorption on the metal (Pb^{2+}) ions takes place in the pore size of the activated carbon. Also, the presence of numerous pores in the samples aid the rate of adsorption capacity of the material as large quantity of metal ions is adsorbed. The application of Zinc chloride improves the pore activation in the process. The presence of low percentage of moisture content, ash content and volatile contents aid in the adsorption capacities as more crystalline materials are formed which when activated form a branch of carboxyl groups. The particle size diameter range was determined to 1.3-18.2 μm using ImageJ coupled with Origin6.0.

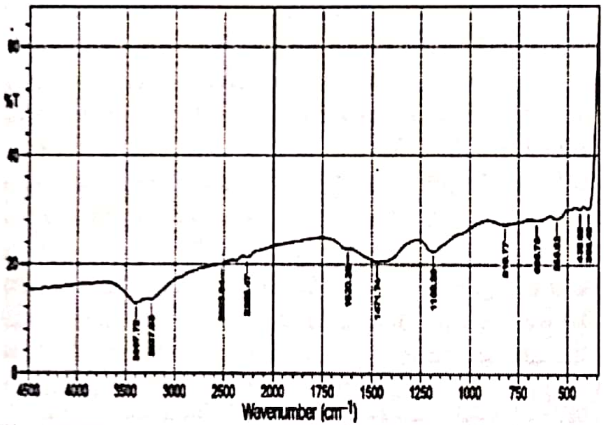

Fig. 3 FTIR spectral of the synthesized activated carbon

The functional groups present which give the surface chemistry in the developed activated carbon were characterized with the aid of Fourier Transform Infra-Red Analysis. Figure 3 shows the spectra of the existing functional materials present in the sample represented by distinct peaks with complex surface. The peak 3397.72 cm^{-1} represents the absorption peak on the surface of the activated carbon implies the presence of O-H stretching vibration in Carbon whereas the peaks at 2523.94 cm^{-1} indicates -CH stretching of aliphatic hydrocarbon. Peak 1620.26 cm^{-1} and 1471.74 cm^{-1} indicate carboxyl group and –C=O stretching respectively while peak 1186.26 cm^{-1} implies ether linkages. According to Yalcin *et. al.* (2012), peak 819.77 cm^{-1} indicates –CH groups which are out of plane.

The levels and ranges of the studied process parameters (A – adsorbent dosage, B – temperature, C - pH, D- residence time) affecting lead removal employed in the experiment are given in Table 3. The observed and predicted percentages of Pb^{2+} removal by both activated carbons were presented in Table 4. pH has an adverse effect on the response for activated carbon adsorption of Pb^{2+}. The results were analysed using the software Minitab 16 and along with the main effects, the interactions of different factors were determined. The coded mathematical model for 2^4 factorial designs is given as:

$$\% Y = X_0 + X_1A + X_2B + X_3C + X_4D + X_5AB + X_6AC + X_7AD + X8BC + X_9BD + X_{10}CD + X_{11}ABC + X_{12}ABD + X_{13}ACD + X_{14}BCD + X_{15}ABCD \quad (2)$$

Where Y (%) is the percentage removal of Pb^{2+}, X_0 is the global mean, X_i represents the other regression coefficients and A, B, C, D stands for adsorbent dosage, temperature, pH, residence time respectively.

By substituting the coefficients X_i in Equation (2) by their values we get:

Equation in terms of coded factors

$$= +68.81 + 0.82 * A + 0.94 * B + 13.22 * C - 4.68 * D + 4.17 * A * B + 14.00 * A * C - 11.04 * A * D - 11.60 * B * C - 1.27 * B * D + 15.18 * C * D + 5.60 * A * B * C - 4.73 * A * B * D + 0.37 * A * C * D + 6.84 * B * C * D \quad (3)$$

Table 3 Factorial design matrix of four variables along with experimental and predicted responses for Pb^{2+} removal by AC

Run No.	Coded values of independent variables				Pb^{2+} Removal (%)		
	A	B	C	D	Observed	Predicted	Residual
1	-1	-1	-1	-1	59.3346	55.29	4.0446
2	+1	-1	-1	-1	50.2343	49.37	0.8643
3	-1	+1	-1	-1	95.0912	92.17	2.9212
4	+1	+1	-1	-1	97.1212	86.25	10.8712
5	-1	-1	+1	-1	77.2837	60.25	17.0337
6	+1	-1	+1	-1	98.2345	110.33	-12.0955
7	-1	+1	+1	-1	14.7941	23.37	-8.5759
8	+1	+1	+1	-1	95.7877	73.45	22.3377
9	-1	-1	-1	+1	50.2343	60.69	-10.4557
10	+1	-1	-1	+1	12.3453	10.61	1.7353
11	-1	+1	-1	+1	70.4123	70.21	0.2.23
12	+1	+1	-1	+1	9.9015	20.13	-10.2285
13	-1	-1	+1	+1	98.0012	99.01	-1.0088
14	+1	-1	+1	+1	97.2312	104.93	-7.6988
15	-1	+1	+1	+1	78.7823	89.43	-10.7077
16	+1	+1	+1	+1	96.1232	95.41	0.7132

Table 4 Estimated regression coefficients of significant factors (coded units) and their effects for Pb^{2+} adsorption.

Terms	Sum of Square	Degree of Freedom	Mean Square	F-value	p-value Prob > F
Model	16017.45	14	1144.10	273.59	0.0474
A	10.64	1	10.64	2.54	0.3565
B	14.28	1	14.28	3.41	0.3158
C	2797.44	1	2797.44	668.95	0.0246
D	350.16	1	350.16	83.73	0.0693
AB	277.74	1	277.74	66.42	0.0777
AC	3135.59	1	3135.59	749.81	0.0232
NMI. These knowledge types are used by man;			1951.50	466.66	0.0294
BC	2153.91	1	2153.91	515.07	0.0280
BD	25.76	1	25.76	6.16	0.2439
CD	3688.31	1	3688.31	881.99	0.0214
ABC	502.27	1	502.27	120.11	0.0579
ABD	358.00	1	358.00	85.61	0.0685
ACD	2.22	1	2.22	0.53	0.5993
BCD	749.65	1	749.65	179.26	0.0475
Residual	4.18	1	4.18		
Cor Total	16021.63	15			

After estimating the main effects, the interacting factors affecting the removal of Pb^{2+} were determined by performing the analysis of variance (ANOVA). Sum of squares (SS) of each factor quantifies its importance in the process and as the value of the SS increases the significance of the corresponding factor in the undergoing process also increases (Table 4). The main and interaction effects of each factor having P values <0.05 are considered as potentially significant.

The effect of each factor was statistically significant at $P < 0.05$ (abdel-Ghani *et al.*, 2009) The main effect C and interactions AC, AD, BC, CD and BCD are of higher statistical significance. Based on F ratio and P-value statistically insignificant factors were discarded. The removal of 4-way interaction factor (ABCD) was essential due to the hierarchical nature of the model. After discarding insignificant terms, the resultant models can be represented as:

The empirical relation in terms of coded factors;

$$\text{Yield} = +68.81 + 13.22*C + 14.00*A*C - 11.04*A*D - 11.60*B*C + 15.18*C*D + 6.84*B*C*D \qquad (5)$$

The empirical relation in terms of actual factors;

$$\text{Yield} = -61.19952 + 37.60145*\text{pH} - 14.42230*\text{Dosage}*\text{pH} + 19.12424*\text{Dosage}*\text{Residence Time} - 1.45175*\text{Temperature}*\text{pH} - 3.70640*\text{pH}*\text{Residence Time} + 0.23603*\text{Temperature}*\text{pH}*\text{ResidenceTime} \qquad (6)$$

It can be seen that the effect of pH was characterized by a greater degree of departure and also had a positive effect on the response (Y %). The interaction plots showed that interaction of AC, AD, BC, and CD played major role and also these two factors interacted strongly with other factors indicating predominant influence in removal of Pb^{2+} concentration.

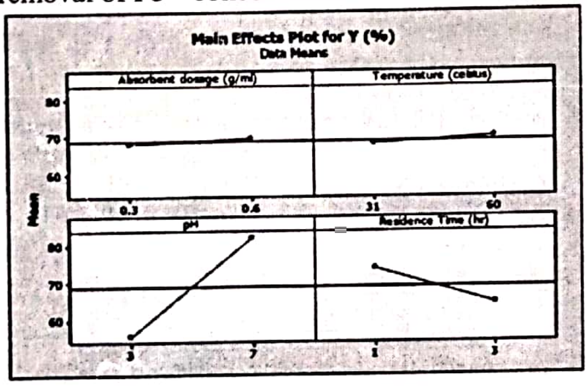

Fig. 4. Main effect plots of Lead ions sorption by activated Carbon

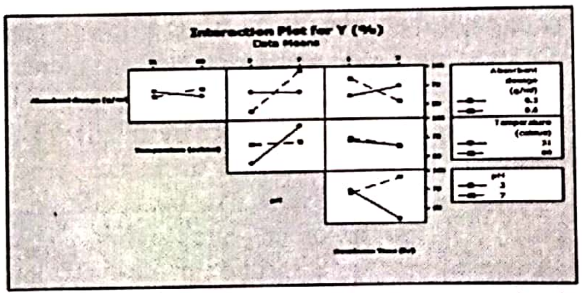

Fig. 5. Interactive effect plots of Lead ions sorption by activated Carbon

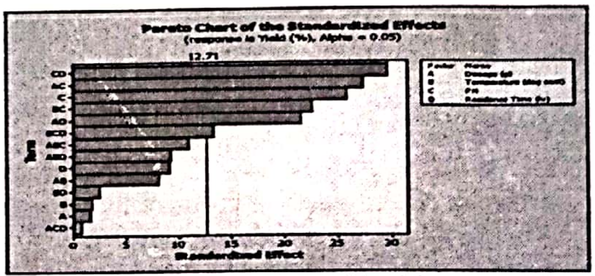

Fig. 6. Interactive effect plots of Lead ions sorption by activated Carbon

Std. Dev.	2.04	R-Squared	99.97%
Mean	68.81	Adj R-Squared	99.61%
C.V. %	2.97	Pred R-Squared	93.32%
PRESS	1070.55	Adeq Precision	45.129

R square measures the proportion of total variability explained by the model. The value of R-square is 99.97 %. The Prediction Error Sum of Squares, PRESS, indicates that the model is expected to explain about 93 % of the variability.

Effect of Temperature on Pb $^{2+}$ Adsorption

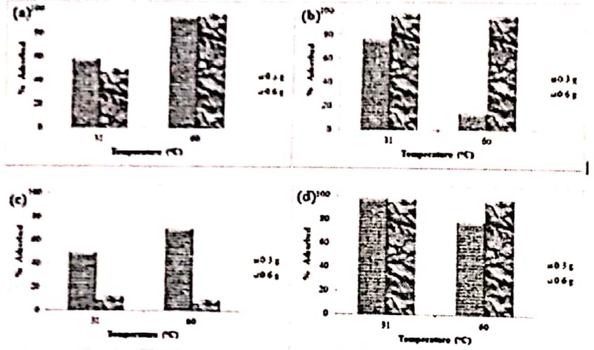

Figure 7: Effects of Temperature at (a) pH of 3 and 1hr (b) pH 7 and 1hrs (c) pH of 3 and 3hrs (d) pH of 7 and 3hrs

The effect of temperature on the adsorption efficiency of the synthesized zinc chloride activated carbon in lead (II) ion solution was studied. Result as presented in figure 7 shows the effect of temperature on percentage adsorption of lead ion. The effect of temperature on the removal of Pb^{2+} in aqueous solution by alkaline based activated carbon was studied by varying the temperature between 31 and 60 °C. Typically, Data as presented in figure 7(a) shows the behaviour of a biosorption of many ions in an aqueous solution at an increased temperature (Ho, 2003). However, the above deduction is only valid at lower pH of ion solution (pH 3) and lower residence time (1 hour) of sorption process. At this condition, the pores of the adsorbent are active and are susceptible for the adsorption of Lead (II) ion at higher temperature.

Effect of pH on Pb $^{2+}$ Adsorbtion

The sorption of lead by activated carbon from sugarcane bagasse and characterization of biosorptive ability were significantly affected by pH solution (Sheng et al., 2004). The effect of lead on the sorption potential of the synthesized activated carbon is shown in figure 8. The effect of activated carbon weight as well as PH of the solution was depicted. The graphs show an increment in the sorption capacity at higher pH of 7 than 3. I all cases, the adsorption of lead ion to the activated carbon increase as the pH increases from 3 to 7. This result is in agreement with the behaviour reported by Cabrera, *et. al.*, 2005 and Shibata, 1997 which is as a result of excessive protonation of the active part of the adsorbent at lower pH.

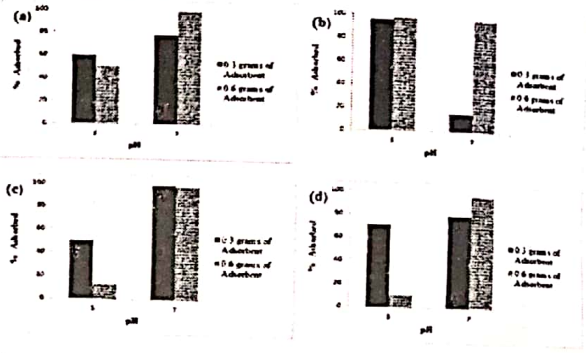

Figure 8: Effect of pH at (a) 31 °C and 1 hour (b) 60 °C and 1 hour (c) 31 °C and 3 hours (d) 60 °C and 3 hour

Effect of Adsorbent Weight on Pb $^{2+}$ Adsorption

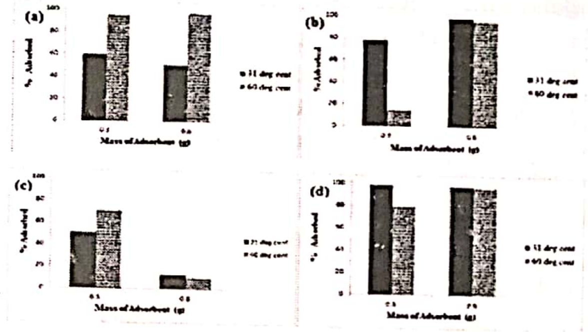

Figure 9: Effects of Mass of Adsorbent at (a) pH of 3 and 1hr (b) pH 7 and 1hrs (c) pH of 3 and 3hrs (d) pH of 7 and 3hrs

Figure 9 represents the effect of adsorbent dosage on the adsorption capacity of the produced activated carbon using zinc chloride. From the result of the experiment, figure 9 it was showed that the percentage sorption of lead ions onto the pores of the activated carbon reduces at 0.3g activated carbon loading, pH of 7, 3 hours of residence time and 60 °C. The reduction in sorption capacity is due to the desorption of the metals ions at higher temperature into the solution.

Effect of Residence Time on Pb $^{2+}$ Adsorption

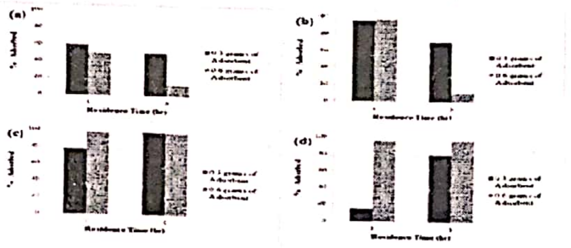

Figure 10: Effect of residence time at (a) 31 °C and pH of 3 (b) 60 °C and pH of 3 (c) 31 °C and pH of 7 (d) 60 °C and pH of 7

Considering figure 10a, at residence time of I hour, 31 ^{O}C and pH of 3, the rate of adsorption of lead ions is high. This is an indication of presence of active pores in the activated carbon for adsorption at the beginning of the process. As the surface adsorption sites become dissipated, the uptake rate was controlled by the rate at which the adsorbate is transported from the outer to the interior pores of the adsorbent particles and thus, the sorption rate decreases.

Conclusions

Adsorption process as widely regarded as one of the effective and efficient heavy metal ions removal technology. In this study, adsorbent of low cost and high sorption capacity was successfully developed from sugarcane bagasse using chemical activation process. The developed adsorbent possesses high surface area and pore volume for effective adsorption of heavy metals. The best adsorption parameters were determined to occur at high dosage (0.6g), low temperature (31), high pH (7.0) and low residence time (1hr). The adsorption of metal on activated carbon is built on the principle of physical/physical adsorption, which is caused by Van der Waals forces, a relatively weak bond.

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