International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 5 Issue 09, September-2016

Removal of Lead from Paint Effluent using Low Cost Activated Adsorbent (Fluted Pumpkin Seed Shells)

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Abstract **- Activated carbon produced from pumpkin seed shells was used as an adsorbent to remove lead ion from paint industrial effluent. The activated carbon produced was carbonized at 800ºc for 2hours and then chemically produced with 9M KOH solution. Batch adsorption experiments were conducted at different conditions to examine the effects of adsorbent dosage, P^H , contact time and temperature on adsorption of Pb^{2+} from paint effluent. The results obtained showed that, the adsorption of Pb^{2+} was dependent on the adsorbent dosage, P^H, contact time and temperature. The optimum adsorption was recorded at dosage of 0.3g, time of 60 minutes, P^H of 6 and temperature of 30ºC ; and 97.97% removal efficiency was obtained. The Linear regression coefficient R^2 of 0.997 for Langmuir model made the best fit.**

Key words: Activated carbon, Adsorption, Isotherm studies, lead (II) ions, paint effluent

INTRODUCTION

Environmental pollution problems caused by heavy metals cannot be over emphasized. Waste water from numerous industries such as paints and pigments, glass production, mining operations, metal plating and battery manufacturing processes are known to contain contained contaminants such as heavy metal. Heavy metals such as Pb(II), Cd(II), Hg(II), Ni(II), Zn(II), Cu(II) and Fe are present in industrial waste water (Mhemet et al, 2006).

Lead (Pb(II)) is one of the common heavy metals found in paint industrial waste water. Heavy metals are not biodegradable and tend to be accumulated in organisms and cause numerous diseases and disorders (Ozer and Pirincci, 2006). Chronic exposure to high amount of lead can result in various and considerable damages to system of the body, including nervous and reproductive systems and kidneys. Moreover, high blood pressure, anemia, lead poisoning, coma and death can be considered among the most substantial consequences (PCS, 2001). Lead as P_b^{2+} ion has a large affinity for the thio (-SH) and phosphate ion (PO_4) which contain enzymes, ligands and bio-molecules, thereby inhibiting the biosynthesis of heme units, affecting membrane permeability of kidney, liver and brain cells. These result in either reduced functions or complete breakdown of these organs. Lead form complexes with oxo-groups in enzymes to affect virtually all steps in the process of Hemoglobin synthesis and porphyrin metabolism (Ademorati, 1996).

Therefore, the elimination of heavy metals such as lead from the environment is important as a result of its high toxicity and at least to protect public health(Abdel-Ghani and El-Chaghaby, 2007; Abdel-Ghan et al.,2009; Resmi et al., 2010). Many researchers have reported the removal of heavy metals; P_b(II), using several physiochemical methods such as chemical precipitation, evaporation, ion exchange and reverse Osmosis (Ikhuoria and Omonmhenle, 2006). However, these conventional technologies appear to be inadequate and expensive over the years, adsorption have been shown to be an economically feasible alternative method for removing metal ions (Uzun and Guzel, 2000; Qader and Akhtar, 2005; Onundi et al, 2010, Okoye et al, 2010). The major advantages of an adsorption system for water pollution control are less investment in terms of initial cost and land, simple design and easy operation and no effect of toxic substances compared to conventional biological treatment processes (Markovska et al, 2006)

Agricultural by-products have been used as adsorbents in research work for removal of heavy metals from industrial effluents or other sources. This is due to the toxic effects of heavy metals and disadvantages of conventional methods of removal (Eze et al, 2013). Activated carbon has been used as adsorbent for removal of heavy metal pollutants from wastewater and has proved to be effective (Guen, et al., 2007; Goyal et al., 2008). This is due to its good adsorption properties which depend on its well developed porous structure and large active surface area (Kang et al., 2008) also it can be produced from cheap and locally available materials (Ochonogor and Ejikeme, 2005; Ejikeme and Ochonogor, 2008; Mahvi., 2008; Okpareke et al., 2009). The ability of activated carbon from Fluted Pumpkin seed shells (Telifairia Occidentalis) to remove lead from paint effluent is investigated in the work in the laboratory. Batch adsorption experiments and Adsorption Isotherm modeling were also carried out in the study.

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MATERIALS AND METHOD

MATERIALS

Preparation of activated carbon

The fluted pumpkin seed was collected from New market Enugu, Nigeria. The shell was separation and was dried for 6 days under the sun light to reduce the moisture content of the seed shells. After this, it was collected and stored in a glass jar until use. The dried fluted pumpkin seed shells were crushed to desired mesh size of 1-2mm and then carbonized at 800^{0}C for 2hrs in a stainless steel vertical tubular reactor placed in a tube furnace. The char produced was crushed and sieved with 6OOnm sieve size. The char was soaked in 9M KOH solution with (1:1.5) char to KOH ratio. The mixture was then dehydrated in an oven at 105^{0}C to remove moisture and then was activated under the same condition as carbonization, but to a different final temperature of 850^{0}C for 1hrs. The activated product was then cooled to room temperature and washed with hot deionized water and 0.1 NHCL until P^H of washing solution reached 6.4. The preparation of the adsorbent was in accordance of the method used by (Tan et al; 2008) with slight modification.

Adsorbate

4 litres of effluent (waste water) was supplied by Buxtin Paint Ltd Uwani Enugu, Nigeria.

SORPTION EXPERIMENT

BATCH ADSORPTION STUDIES

Experiments/ Study on P^H, Dosage, Time and Temperature.

To study the effect of P^H on adsorption of lead on fluted pumpkin seed shell activated carbon, the experiments were carried out in five different batches. The first batch was at constant temperature of 30^{0}C, dosage of 0.1g and time of 20mins. Also at initial P^H values of 2,4,6,8 and 10. The second, third, fourth and fifth batches were at different temperatures of 40^{0}C, 50^{0}C, 60^{0}C, 70^{0}C, dosage of 0.3g, 0.3g, 0.4g and 0.5g and time of 40mins, 60mins, 80mins, 100mins respectively, but at the same P^H value as of the first batch. The P^H values were adjusted with 0.1m HCl or using P^H meter (Jenway model 3510, England), as centrifuged and the residues wastewater was analyzed using Atomic Absorption Spectrophotometer (AAS). All measurement was made at 661nm, a wave length corresponding to the maximum absorbance.

ISOTHERM STUDIES

Langmuir Isotherm

The Langmuir adsorption isotherm describes quantitatively the buildup of a layer of molecules on an adsorbent surface as a function of the concentration of the adsorbed material in the liquid in which it is in contact (Naseen rauf et al, 2012). This isotherm is based on the assumption that the adsorption process takes place at specific homogenous sites within the adsorbent surface and that once a dye molecule occupies a site, no further adsorption can take place at that site which concluded that the adsorption process is monolayer in nature (Ghaedi et al , 2012). The isotherm can be written as:

$$\frac{ql}{qo} = \frac{qlCAe}{1+KlCAe} \quad \text{.......................} \quad (1)$$

The linear form of the isotherm is given as:

$$\frac{\text{CAe}}{\text{qe}} = {}^{1}\!/_{\text{q}_\text{o}\text{K}_\text{L}} + \frac{\text{CAe}}{\text{q}_\text{o}} \quad \text{................} \quad (2)$$

Where qe is the amount of adsorbate adsorbed per gram of the adsorbent (mg/g), CAe is the concentration of adsorbate in liquid equilibrium, (mg/l). The constant K_L and q_o relate to energy of adsorption and maximum adsorption capacity respectively. K_L is a measure of heat of adsorption utilized to calculate dimensionless separation parameter $R_{L,}$ q_o is in mg/g.

To determine if the adsorption process is favorable or unfavorable, for the Langmuir type adsorption process, the isotherm shape can be classified by a term "R_L" a dimensionless constant separation factor which is defined as below (Malkoc and Nuhoglu., 2007).

$$R_L = \frac{1}{(1+K_L CAo)} \quad \text{.........................(3)}$$

Where KL (L/mg) is the Langmuir constant and Cao is the lowest initial solute concentration (mg/l). a plot of Cao / qe versus CAe from the linear relationship of the model will give ${}^{1}\!/_{(\text{q}_\text{o}\text{K}_\text{L})}$ and $1/q_o$ as intercept and slope respectively.

Freundlich Isotherm

The Freundlich Isotherm equation is based on the assumption that the adsorbent has a heterogeneous surface composed of different classes of adsorption site (Marsal et al., 2012). This isotherm does not predict any saturation of the sorbent by the sorbate, indicating multilayer sorption of the surfaces (Donat et al, 2005). The freundlich isotherm is expressed by the following equation.

$$qe = K_F\ CAe \text{...............................} \quad (4)$$

A linear form of the freundlich can be obtained by taking the logarithm to the above equation.

$$\log qe = Log\ K_F + {}^{1}\!/_{n} Log\ CAe \text{ .... } (5)$$

Where K_F = freundlich constant. Where K_F (mg/g) is the constant related to the adsorption capacity. The value of 1/n ranging between 0 and 1 is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero (Zeynep et al.,2007). A plot of Log_{qe} versus CAe will give intercept as $LogK_F$ and slope as 1/n.

Temkin Isotherm Model

The heat of the adsorption and the adsorbate – adsorbate interactions in a adsorption process were studied by

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Temkin and Pyzhev (Temkin et al; 1940), and its equation is given as follows:

$$qe = BlnA + BlnCe$$
$$B = (RT/b) \quad \ldots\ldots\ldots\ldots\ldots \quad (6)$$

Where T is the absolute temperature in K and R is the universal gas constant 8.3143J/molk. The constant b is related to the heat of adsorption qe(mg/g) and Ce(mg/l) and the equilibrium concentration respectively. A and B are constants related to adsorption capacity and intensity of adsorption. A plot of qe versus Ince yields a slope of B and intercept of BlnA.

Dubinin – Raduschkevich (D – R)

Dubinin – Radushkevich model is similar to the Langmuir model but does not assume a homogenous surface or constant energy potential (Marsal et al., 2012). The D – R equation can be written as:

$$qe = \varepsilon_{max} \exp(-B_D \varepsilon^2) \ldots\ldots\ldots\ldots \quad (7)$$

Where ε is the Polanyi potential (J/mol) given by: $\varepsilon = RTLn(1 + 1/Ce)2$ ……………………. (8)

Linearizing it gives

$$Lnqe = In\ qmax - 2B_D RT\ Ln(1 + 1/Ce) \ldots\ldots \quad (9)$$

Where qe is the amount of sorbate adsorbed at equilibrium (mg/g), qmax is the theoretical saturation capacity (mg/g), B_D is a constant related to the adsorption capacity (mg^2/l^2). A plot of Lng versus (RTLn(1 + 1/Ce) yields a straight line with a slope of -2B_D and intercept of Lnqmax. The value of B_D can be used to calculate the sorption mean free energy E (KJ/Mol).

$$E = (2B_D)^{-1/2} \ldots\ldots\ldots\ldots \quad (10)$$

Taking the logarithm of the D – R equation, it becomes

$$Lnqe = Lnq_{max} - 2B_D \varepsilon \ldots\ldots\ldots \quad (11)$$

A plot of Lnqe against ε gives slope of -2B_D and intercept of Lnq_{max}.

RESULTS AND DISCUSSION

Table 1: ANALYSIS OF PAINT EFFLUENT

S/N	Parameter Physical Analysis	Unit	Waste water
1	Temperature	^0C	30.4
2	Odour	-	-
3	Conductivity	Ns/Cm^3	468
4	P^H	-	6.01
5	Turbidity	NuT	18.01
6	Acidity	Mg/L	Nil
7	Alkalinity	Mg/L	20
8	Total solid	Mg/L	0.5
9	Dissolved solid	Mg/L	0.2
10	Suspended solid	Mg/L	0.3
11	Copper	Mg/L	0.40
12	Iron	Mg/L	0.15
13	Zinc	Mg/L	Nil
14	Lead	Mg/L	0.148
15	Chloride	Mg/L	21.3
16	Sulphate	Mg/L	24.6
17	COD	Mg/L	12.4
18	DO	Mg/L	19.7
19	BOD	Mg/L	143.7
20	Phosphorus	Mg/L	0.029
21	Aluminum	Mg/L	Nil

The analysis was performed physically to ascertain its temperature, odour, conductivity, P^H and Turbidity. Also chemically to determine its acidity, alkalinity, total solid, dissolved solid, suspension solid, BOD, COD, DO and presence of some metals such as Cu^{2+}, Fe^{2+}, Zn^{2+}, Pb^{2+}, Cl^-, sulphate, phosphorus and aluminum.

APPARATUS AND EQUIPMENTS USED ARE AS FOLLOWS:

conical flasks, beakers, P^H meter, weighing balance, crucible, electric shaker, sintered glass crucible, platinum crucible, muffle furnace, desiccators, graduated cylinders, thermometer, filter paper, Atomic Absorption Spectrophotometer, pipette, volumetric flask, shaker incubator, UV Visible spectrophotometer.

Table 2: Characterization of fluted pumpkin seed shells

S/N	**Parameter**	**Unit**	**Raw fluted pumpkin seed shells**	**Activated fluted pumpkin seed shells**
1	Bulk Density	g/ml	0.28	0.48
2	Ash Content	%	4.0	2.0
3	Moisture Content	%	8.1	6.6
4	Iodine Number	Gl_2/100g	290.4	411.4
5	P^H	-	6.4	6.4
6	Tapped Density	g/ml	0.32	0.52
7	Volatile matter	%	48.9	28.0
8	Fixed carbon	%	39.0	63.4

The fluted pumpkin seed shells were characterized based on its raw state and after activation.

Apparatus and Equipments used are as follows:

Stainless steel vertical tabular reactor, tube furnance, crusher, sieves and weighing balance

EFFECT OF PROCESS VARIABLES ON ADSORPTION

Effect of Dosage

Figure1 shows the effect of dosage of the adsorbent used on the adsorption of lead from paint effluent.

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International Journal of Engineering Research & Technology (IJERT)
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Vol. 5 Issue 09, September-2016

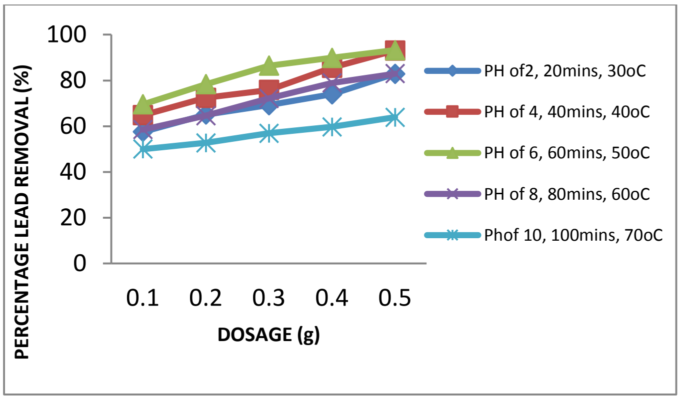

Figure 1 Effect of dosage on percentage removal of lead

From the figure 1, it can be seen that the percentage quantity of lead adsorbed increases linearly with increase in adsorbent dosage. This is as a result of the fact that the quantity of sites for adsorption increases with increase in adsorbent weight (Dahya et al, 2008) similar result was observed by Dave et al 2009) for the adsorption of Cu^{2+} on commercial activated carbon.

Also, the quantity of lead adsorbed per gram of adsorbent increases with increase in the dosage of the adsorbent used. This is as a result of the fact that the adsorbate ions concentration was constant at all the site available for adsorption on the adsorbent. At the dosage of 0.5g, the highest degree of adsorption was recorded and the lowest degree was recorded at dosage of 0.1g.

Effect of P^H

Figure 2 shows the effect of P^H of the paint effluent on the adsorption of lead by the activated carbon of fluted pumpkin seed shells.

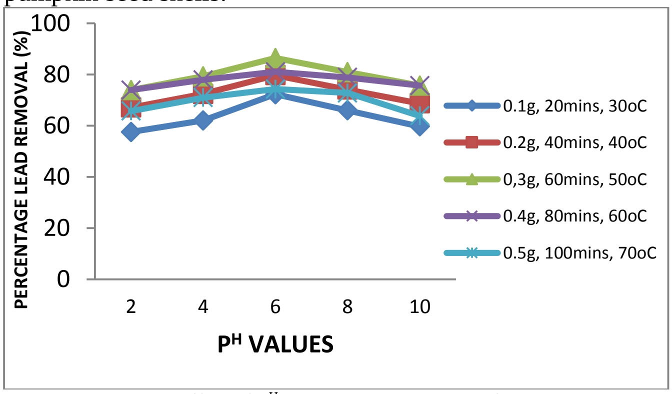

Figure 2 Effect of P^H on percentage removal of Lead

The initial P^H of an adsorption medium is one of the most important parameters affecting the adsorption process. The effect of P^H on the adsorption of lead was studied varying the P^H from 2 to 10 shown in figure 2. It was observed that the amount of lead adsorbed occurred at P^H 6 for fluted pumpkin seed shells activated carbon.

Significant reduction in removal efficiency was observed for further increase in P^H. This may be attributed to the fact that at low P^H (1-3), the metal ions (Pb^{2+}) had to compete with H^+ ions for adsorption site on the adsorbent surface. As the P^H increased, this competition weakened and more metal ions were able to replace H^+ ions bound to the adsorbent surface (Ibrahim et al, 2006).

Lead considered in this study has pka value of 8. When the P^H of a solution goes beyond the pka, lead chiefly exists as a negative ion and as neutral molecules below pka. Therefore at high P^H value greater than 6, for fluted pumpkin activated carbon, the H^+ ions becomes more competitive to Pb ion for sorption site. The H^+ ions could populate the surface resulting in repulsion of lead ions and thereby causing decreased adsorption.

Therefore, from the experimental results, P^H 6 was selected as an optimum P^H.

Effect of Time

Figure 3 show the effect of contact time on the adsorption of lead (Pb^{2+}) from the waste water.

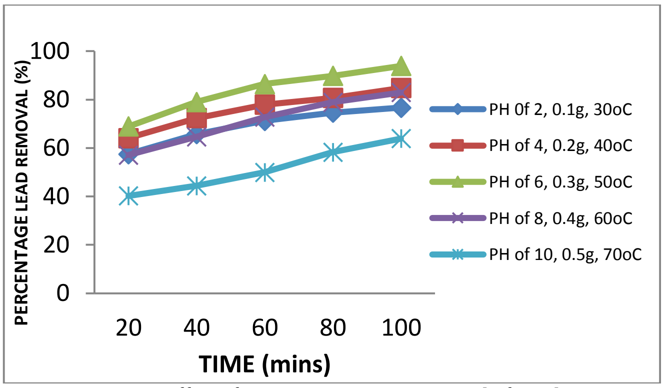

Figure 3 Effect of Time on percentage removal of Lead

In figure 3, there was an initial rapid sorption for about 20 minutes of agitation. After this, the rate of sorption became slower. After 75-85 minutes of agitation, equilibrium was attained. This can be explained by the fact that within the first 20 minutes, the adsorbent still had a vast, number of unoccupied sites unto which the Pb^{2+} adsorbate could adsorb. As a result, there was a high probability of adsorption for every diffusing molecule of the adsorbate. The lead absorbed within the first 20 minutes lead to a decrease in number of unoccupied sites, this made the adsorption to become slower since adsorbent surface was approaching, saturation. After 85 to 100 minutes, the adsorbent sites were completely occupied by the adsorbate particles. At this stage, the system attained equilibrium at which the rate of adsorption becomes equal to rate of desorption.

Effect of Temperature

Figure 4 shows the effect of adsorption temperature on the adsorption of lead by the activated carbon.

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International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 5 Issue 09, September-2016

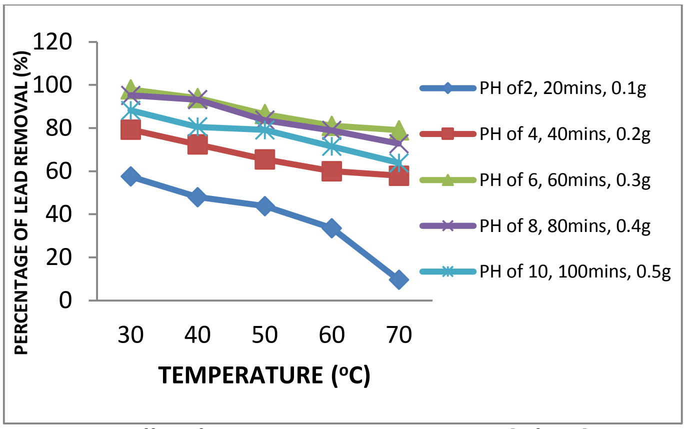

Figure 4 Effect of Temperature on percentage removal of Lead

From the results and figure 4, it is seen that the adsorption decreased as temperature increased. This is in agreement with the result obtained by (Teker et al, 1999). This decrease in adsorption capacity with increase in temperature indicates that the adsorption process were exothermic in nature as there is decrease in the number of microstates and decrease in the freedom of movement of molecules.

Because of this reason, the highest absorption of lead occurred at 30^0c with approximately 98% removal.

ISOTHERM STUDIES

The isotherm data obtained for the adsorption processes were analyzed using the Langmuir, Freundlich, Temkin and Dubinin Kadushkevich isotherms.

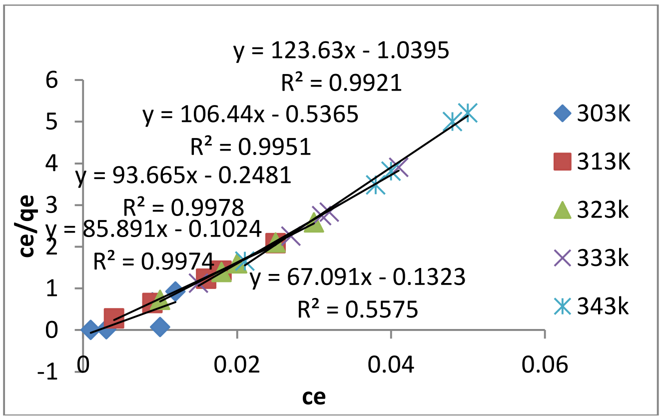

Figure 5, Langmuir isotherm plot for Lead Adsorption of FPSS activated carbon

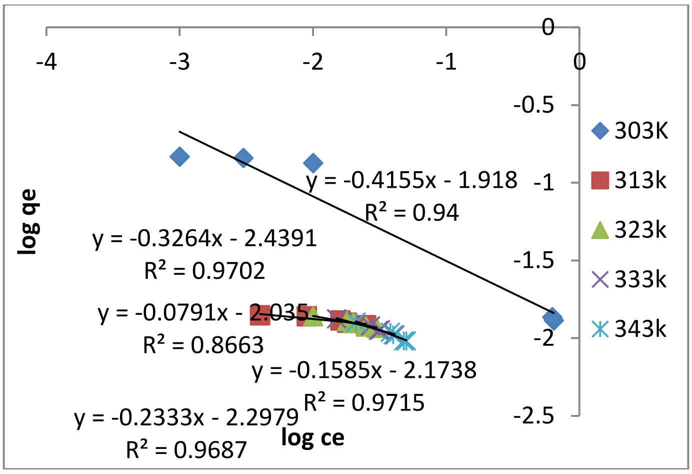

Figure 6, Freundlich Isotherm plot for Lead Adsorption on FPSS activated carbon

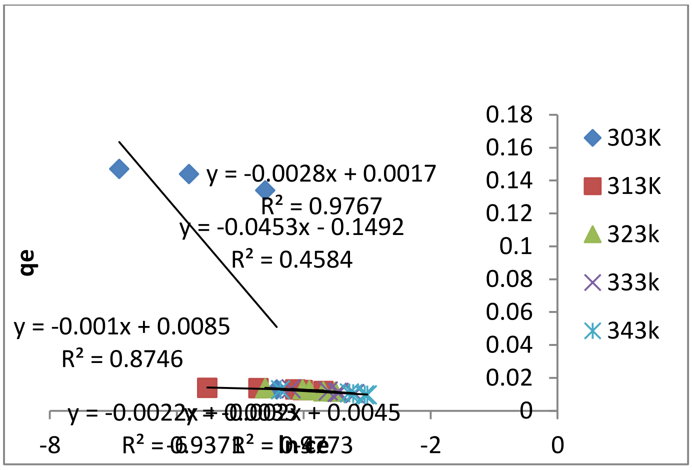

Figure 7, Temkin Isotherm plot for Lead Adsorption on FPSS activated carbon

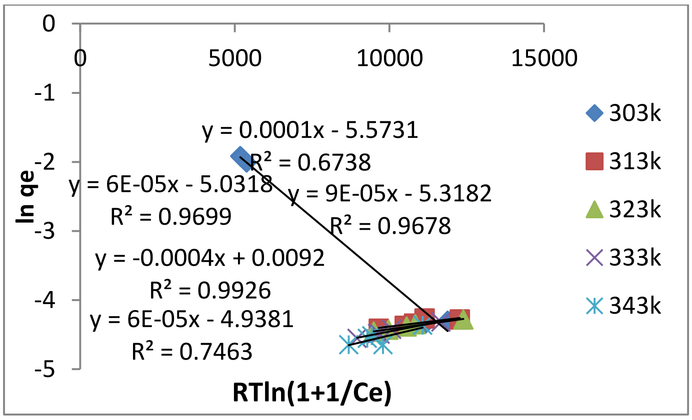

Figure 8, Dubinin Radushkevich Isotherm plot for Lead Adsorption on FPSS Activated carbon

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International Journal of Engineering Research & Technology (IJERT)
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Table 3: Isotherm Parameters for lead Adsorption on fluted pumpkin seed shells activated carbon.

ISOTHERM MODEL		PARAMETERS			
Langmuir	Temp (K)	qo(mglg)	KL(^{L}/mg)	R_L	R^2
Isotherm	303	0.0149	-0.0019	1.0	0.557
	313	0.0116	-1.1832	1.0	0.997
	323	0.0107	-0.0027	1.0	0.997
	333	0.0094	-0.0050	1.0	0.995
	343	0.0081	-0.0084	1.0	0.992
Freundlich	Temp (K)	$\frac{1}{n}$	n	KF	R^2
Isotherm	303	-0.415	-2.41	0.012	0.940
	313	-0.079	-2.66	0.009	0.866
	323	-0.158	-6.34	0.007	0.971
	333	-0.233	-4.29	0.005	0.968
	343	-0.326	-3.07	0.004	0.970
Temkin Isotherm	Temp (k)	B	A	b	R^2
	303	-0.045	27.41	-55.98	0.458
	313	-0.001	0.00	-2602.3	0.874
	323	-0.002	0.14	-1342,111	0.977
	333	-0.002	0.61	-1384381	0.976
	343	-0.002	0.22	-1425,851	0.937
Dubinin Radushkevich Isothern	Temp(k)	Q1 (mg/g)	BD	E (kJ/mol)	R^2
	303	1.00	0.005	0.00	0.992
	313	1.04	2.469	0.45	0.746
	323	1.04	2.516	0.46	0.969
	333	1.06	2.149	0.48	0.967
	343	1.00	2.787	0.42	0.673

Langmuir Isotherm

The value of a dimensionless separation factor, RL, indicates the type of the isotherm to be either unfavourble if (RL > 1, linear (RL = 1), favourable (O < Rl <1) or irreversible (RL = 0). The plot of $\frac{ce}{qe}$ versus Ce indicated the application of Langmuir adsorption isotherm Langmuir constants q_0 and K_1 which were calculated from the slopes and intercepts are given in table 3 along with the correlation coefficient (R^2) and separation factor (R_L). The average value of Rl for each of the temperature and P^H used were 1.0, which indicates a linear adsorption of lead. This was not in agreement with the work done by (Emad et al, 2006). However, the correlation coefficient R^2 which is highest at temperatures of 313k and 323k (0.997) respectively indicates that it fitted well with the model.

Freundlich Isotherm

The values of freundlich constants and correlation coefficients (R^2) are given in table 3 The value of Kf and n changed with the rise in temperature. The values on n less than 1, did not strongly favour the nature of the adsorbent. The correlation coefficient which ranges from 0.866 to 0.971 shows that the data fit well with Freundlich equation at temperature of 323k where R^2 0.971 is closer to unity.

The Freundlich constant n indicates the degree of favourablility and should lie in the range 1-10 to be classified as favourable adsorption (Rao et al 2001; Raji et al, 1997). This work was not in line with the work done by (Okoye et al, 2010).

Temkin Isotherm

The Temkin isotherm constants and coefficients of determination are presented in table 3. This shows that the equilibrium binding constant; A (L/mg), corresponds to the maximum binding energy. This energy was maximum at P^H of 2. The constant b which relates to heat of adsorption, increased with increase in temperature. The correlation coefficient R^2, 0.977 at temperature of 323 fitted well with Temkin isotherm model.

Dubinin – Radushkevich Isotherm

The Dubinin-Radushkevich Isotherm gives information about the chemical or physical properties of the sorption which mean free energy, E (Naseenrauf et al, 2012). The values of the mean adsorption energy that is less than 8 kglmol, indicates that the adsorption of lead was more a physical process rather than a chemical process (Marsal et al, 2012; Chen et al, 2009).

The correlation coefficient R^2 if the isotherm in table 3 shows that Dubinin Radushkevich equation was well fitted. The R.D was fitted well at temperature of 303k where its R^2 is 0.992. This work was in agreement with the work done by (Ejikeme, 2010).

CONCLUSION

The result obtained from this work shows the possibility of production of activated carbon with good properties from fluted pumpkin seed shells. Adsorption decreased with increase in adsorption temperature and increased with increase in dosage and time in all the cases considered. Adsorption also increased with increase in P^H of the waste water up to P^H of 6, beyond which metal ions were precipitated out of their solutions. The kinetic model was best described using first order equation as it has the best R^2 values. The isotherm analysis showed that the adsorption process of Langmuir, isotherm fitted better than other studies.

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