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REMOVAL OF HEAVY METAL IONS FROM LOCAL BATTERY RECYCLING WASTEWATER USING FUNCTIONALIZED CORN HUSK DERIVED ACTIVATED CARBON

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Abstract

The synthesis of functionalized corn husk derived activated carbon was aimed at studying the potentials of the adsorbent in the removal of heavy metals from local battery recycling wastewater. The synthesis emphasizes on the pretreatment, particle size, amine functionalization and thermal treatment of the adsorbent while the effect of contact time, adsorbent dosage, effluent temperature, pH and concentration forms the basis of the study. The characterization of the raw corn husk and functionalized corn husk derived activated carbon was carried out using thermo gravimetric analysis (TGA), Fourier transform infrared spectroscopy (FT-IR), Branauer-Emmett-Teller analysis (BET), and scanning electron microscopy-elemental disperse spectroscopy (SEM-EDS). The results of TGA shows a temperature profile with steep degradation between 300 and 500 ^{0}C while the FT-IR results reveals the presence of various functional groups before and after modification, the SEM-EDS results also revealed enhanced pores in the activated carbon with broad elemental dispersion containing carbon, nitrogen, oxygen and hydrogen suggests the presence of related functional groups. Effect of contact time was studied between 5 and 140 min while the effect of temperature and dosage were studies between 28 to 70 ^{0}C and 0.5 to 5 g respectively. Further study on the effect of concentration and pH were conducted between the ranges of 10 to 100 % effluent initial concentration and 2 to 11 pH respectively. The experimental adsorption capacity (q_e) was 2.258, 1.492 and 1.065 mg/g for Pb Cu and Ni adsorption respectively. The Langmuir, fruendlich and D-R isotherms were used to describe the sorption process with a best fit in fruendlich isotherm which

suggests multi-layer adsorption, the study of pseudo-first and pseudo second order kinetic models reveals best fit for second order rate which suggests chemisorption. Further thermodynamic studies also suggest exothermic reaction with negative enthalpy and Gibbs free energy. The regeneration study also reveals the viability of the adsorbent for recycle and reuse as 99 % recovery efficiency was observed after three cycles.

***Keywords**: corn husk, activated carbon, local battery wastewater, adsorption, desorption*

Introduction

With rapid industrial development, problems related to pollution are becoming severe and water pollution is one of the most serious problems because inorganic and organic wastes are discharged to the aquatic environment either in water soluble or insoluble forms (Abdi and Kazemi, 2015). It has been discovered that nature can cope with small amount of pollutants but it will have high negative impact or almost becomes uncontrollable if the unquantifiable amount of waste water and sewages released into the environment continuously are not remedied before discharge, thus, effluent treatment reduces pollutants in waste water to an acceptable limit which will have no significant impact on human health (Zhou *et al*., 2013). Recently, heavy metal ions have become significant pollutants which have detrimental effects on the ecosystem due to the increasing number of production and processing companies (fuel producing, energy, fertilizer, mining, leather, pesticide, metal surface treating, and aerospace etc.) (Chaturvedi and Sahu, 2014). Wastewater treatment systems are designed to reduce metal contaminants to meet discharge requirements and achieve the water quality level needed for reuse and recycling (Adetukun *et al.*, 2018). As a result of improper treatment prior to discharge, many dissolved metals have been found in harmful concentrations in ground waters which are destined for potable drinking water. In small quantities, certain heavy metals are nutritionally essentially for a healthy life, many of these metals are required by human in trace amounts, but in larger, persistent dosages, these heavy metals become toxic when they are not metabolized by the body and accumulate in the soft tissues and may cause acute or chronic toxicity (Arquilada *et al.*, 2018).

Several treatment methods have been considered in treating wastewater which includes precipitation, ion exchange, electrochemical treatment, and adsorption (Lin *et al.,* 2019), most of the methods suffer some setback like generation of secondary pollutants, expensiveness or low efficiency for minute heavy metal concentration (Ofudje *et al.,* 2015).The trend in the research of corn husk powder (CHP) adsorbent has extensively proven the sweetability of the adsorbent for heavy metal remediation, various researcher have used several techniques to enhance the adsorption of different pollutants onto CHP adsorbent (Co-doped: Oana *et al.,* 2013; EDTA modified: Priya and Chandrajit 2014; Chemically Activated carbon: Anton *et al.,* 2018) using various modification processes. This research work seeks to further enhance the process of adsorption of lead ion onto CHP, a novel bio-adsorbent has been considered for activated carbon using CHP-based wastes materials as precursor. A modification process involving co-functionalization by certain amination reactions that will introduce several special nitrogen-containing functional groups is employed which will improve the adsorbent framework and its specific adsorption capacity and expand the scope of utilization of the adsorbent. The characteristics and performance of the prepared functionalized CHP-based activated carbon (F-CHPAC) are also investigated in the context of the above descriptions.

Materials and methods

All chemicals used were of analytical grade hence no further purification was done. The corn husk used was obtained from commercial corn roasters within minna town, Nigeria. It was washed with distilled water, grounded and segregated into 250 µm. it was further pretreated with 2% v/v HNO_3 and was stored for further modification

Amination

To synthesis an amine functionalized corn husk (AF-CH), the procedure described by Peng *et al.,* 2017 was used with some modifications. Brifly, the intermediate reaction was made by mixing 20 mL epichlorohydrin, 25 mL N, N-dimethylformamide and 11 mL ethylenediamine in a 250 mL round bottom flask at 80 ^{0}C on a rotary magnetic stirrer at 100 rpm for 60 min, 25 mL of trimethylamine was added to the solution afterwards and stirred for another 90

min. At the last step of the adsorbent synthetic protocol, 20 mL of pyridine (as the catalyzer) and 40 g treated corn husk was added to the mixture and stirred at 90 °C for yet another 90 min. After the reaction was completed, the resulting mixture was filtered and washed with 500 ml of ethanol (50%) and 2 liters of distilled water and then dried at 75 °C for 6 hrs.

Thermal treatment

The functionalized corn husk was heat treated in a muffle furnace at 300 ^{o}C for 2 hrs to produce a functionalized corn husk derived activated carbon

Batch adsorption

Batch adsorption study was carried out to investigate the effect of temperature, dosage, concentration, contact time and pH on the adsorption capacity of F-CHAC. In each experiment, 50 ml of the wastewater is placed in a beaker and adjusted to a desired pH (2-11) and concentration (10-100%) while a corresponding dosage (0.5-5 g) and temperature (28-70 ^{o}C) is preset in a water bath shaker for the experiment. A contact time rage of 5 to140 min was also studied correspondingly. The adsorbent was separated from the water after the desired contact time using whatman;s filter paper No 42 and the filtrate was analysed for residual heavy metal content using AAS analyzer. The corresponding % removal adsorption capacity of the adsorbent was calculated using the equations

$\%\, removal = \frac{\mathrm{Co}-\mathrm{Cf}}{\mathrm{Co}} \times 100\%$ and

$q = \frac{\mathrm{Co}-\mathrm{Cf}}{1000} \times \mathrm{V}\%$ respectively

Results and Discussions

The results of proximate analysis of raw corn husk shows the presence of various constituent in in distinguished percentages, from table 1, the fixed carbon constitute the highest percentage with over 50% , this implies that corn husk has high carbon content and hence is a good precursor for activated carbon.

Table 4.1 Proximate analysis result

S/N	Parameters	Values (%)
1	Moisture Content	12.50

2	Ash content	11.67
3	(VOC)	20.84
4	Fixed carbon	54.99

Functional Group Composition

The functional groups which were recorded by the FT-IR includes the O-H stretch, C-H stretch, C=O stretch, C-Br stretch, C-O stretch, C-C stretch, C=C stretch and the N-H stretch. Figure 1a shows the FT-IR spectra of freshly prepared corn husk with an intense and broad peak at 3862.72 cm^{-1} which can be attributed to the stretching of O-H group due to inter and intra molecular hydrogen bonding such as alcohols or phenols (Madhu *et al.*, 2014). Another peak observed at 3796.52 cm^{-1} can be associated with C–H asymmetric stretching vibration of the CH_2 group (Pachathu *et al.*, 2016). C-H bond of methyl, methylene and methoxy groups was noticed at peak 3441.30 cm^{-1}. 2780.61 cm^{-1} peak was attributed to the C-H associated with alkynes and aromatics (Dalia *et al,* 2014). FT-IR spectrum of the modified adsorbent shown in figure 1b reveal the presence of emerging peaks at 1390.27 and 1173.55 cm^{-1} which were attributed to the N–H bending vibration and C=O stretching of aldehydes and ketones which originates from the functionalization of the adsorbent.

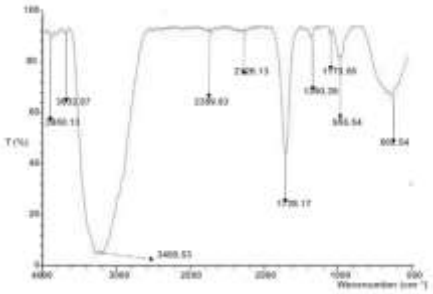

Fig 1a FT-IR Result of raw corn husk

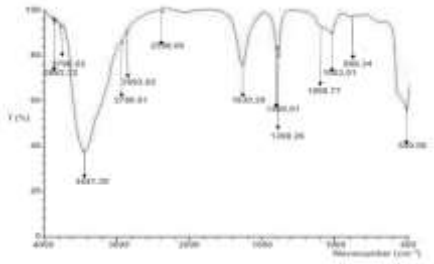

Fig 1b FT-IR Result of F-CHAC

Surface Morphology

Figure 2b and 2d, the presence of numerous pores were observed in the adsorbent after undergoing several modification, these pores were merely present in the raw corn husk adsorbent and was attributed to the formation of

extra pores when the adsorbent was subjected to chemical treatment which lead to the corrosion of several blockages on the surface of the adsorbent and high temperature. The EDS of the adsorbent obtained in fig 2e and 2f also shows the presence of numerous elemental compositions in the fresh and modified husk, the presence of new components in the modified husk (2f) was attributed to the amination reaction which introduced some Nitrogen containing groups into the adsorbents framework. (Mishra *et al.*, 2019).

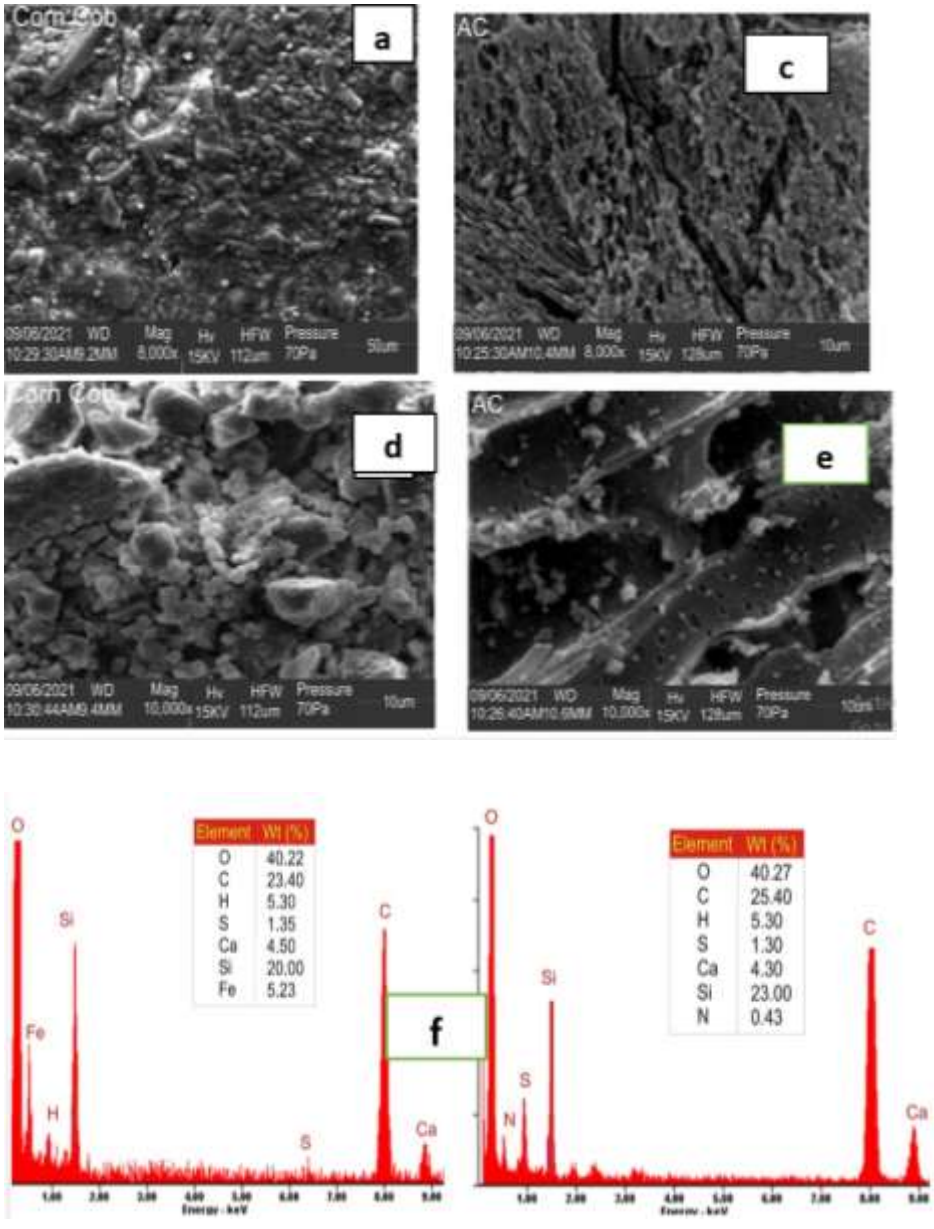

Fig 2a-f: SEM-EDX results for the raw husk corn husk F-CHAC

Surface Area and Pore Volume

From Table 2, it shows that unmodified corn husk which has an initial surface area of 92.1 m^2/g has increased by a very significant factor (more than 400%) after it was modified, this could be attributed to the increased porosity on the modified husk surface which is as a result of the chemical and thermal treatment it went through. These findings are in agreement with the work of Mishra *et al.*, 2019 where corn husk derived magnetized activated carbon was used for the removal of phenol and para-nitrophenol from aqueous solution.

Table 4.5 BET analysis result for raw corn husk and F-CHAC

S/N	Sample type	Surface area (m^2/g)	Pore volume (cc/g)	Pore size (nm)
1	Raw Corn husk	92.11	0.453	2.015
2	F-CHAC	442.70	0.217	2.132

Adsorption Studies

The adsorption studies was carried out to study the effect of five parameters (concentration, temperature, dosage, time and pH) on the removal efficiency of functionalized corn husk derived activated carbon

Effect of contact time

The contact time for adsorption studies on F-CHAC for the removal of selected heavy metals from battery recycling process wastewater was between for 5 to 140 min as shown in fig 3, in this study, the removal efficiencies of the heavy metals rapidly increased during the initial concentration stage as the equilibrium time was attained. The maximum percentage removal of the pollutants which justify the equilibrium time for Pb, Cu, and Ni ions was 80, 79 and 87 % and at an optimum time of 95, 125 and 125 min respectively. The gradual reduction in the rate of removal as it approaches equilibrium could be as a result of the saturation of the adsorbent active sites and the weakening of the binding force as a result of reduced sorbate concentration (Mishra *et al.* 2019). It was also observed from fig 3 that the removal of heavy metals from local battery recycling wastewater is in the order of Ni>Pb>Cu. Ni ion had highest

percentage removal from local battery recycling wastewater and this behavior could be associated to ionic radii of the interested metal ions in the wastewater.

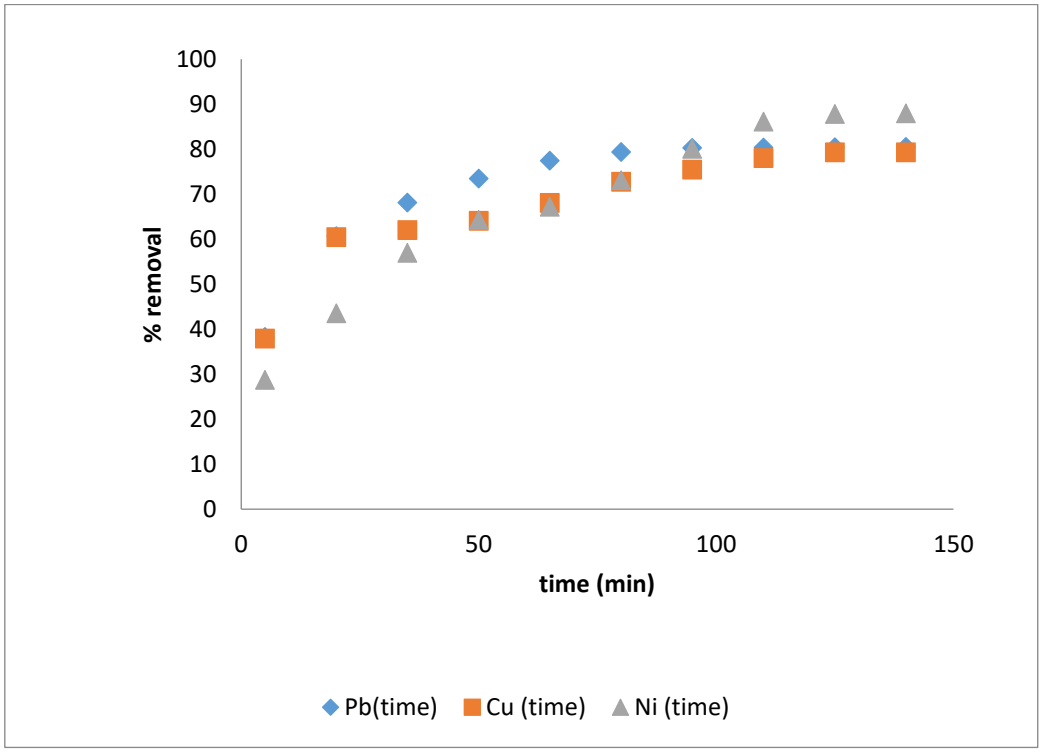

Fig 3 Effect of contact time on the adsorptive removal of Pb, Cu, and Ni

Effect of adsorbent dosage

The effect of adsorbent dosage which ranges from 0.5 to 5 g on the removal of Pb, Cu, and Ni ions from Local battery recycling wastewater is presented in fig 4. The removal percentage was observed to have increased with increasing F-CHAC dosage. The removal efficiencies of Pb, Cu and Ni ions increased from 41 to 99.36 %, 36 to 97.95 % and 38 to 95.92 % respectively at a dosage of 3 g, this implies optimum amount of F-CHAC needed to achieve equilibrium for the three metals. The trends as shown in figure 4 could be attributed to slow adsorption rate at lower adsorbent-adsorbate interface while the increase in percentage removal at higher dosages and fast equilibrium could be attributed to abundance of sorption sites as a result of wider surface area (Mustapha *et al.* 2021).

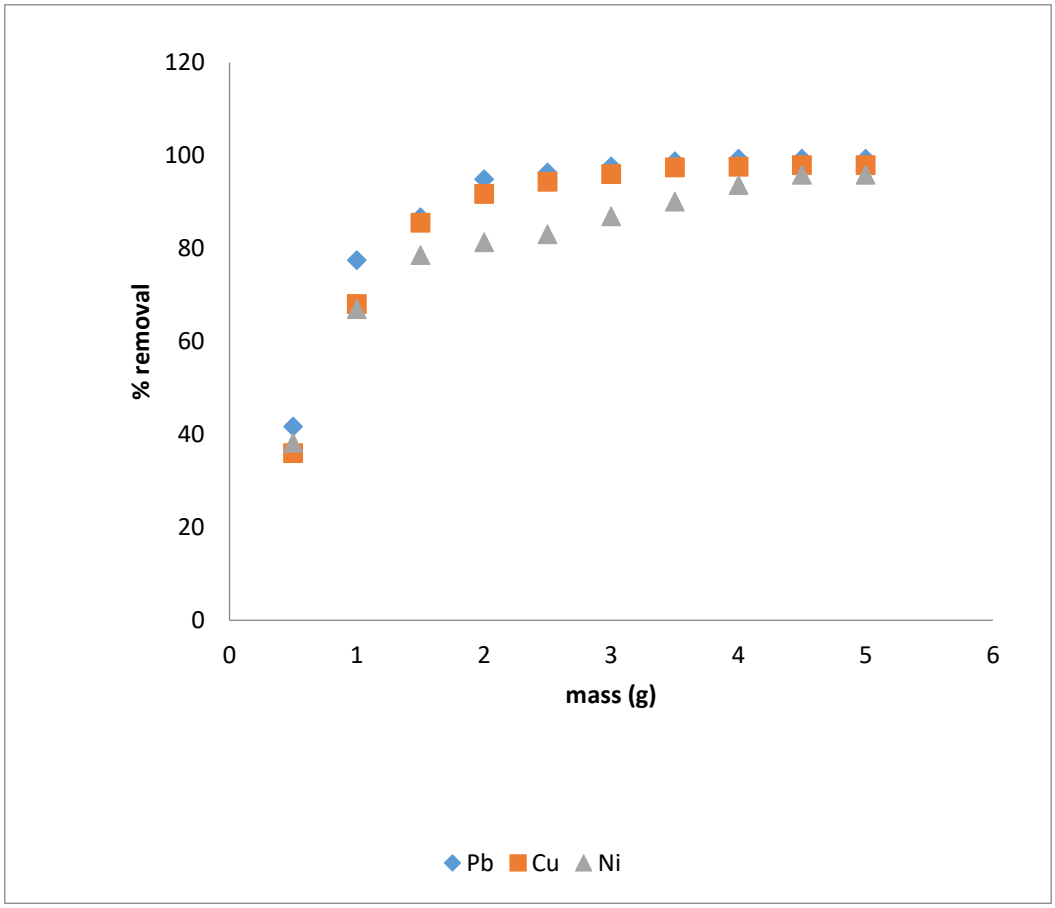

Fig 4: effect of adsorbent dosage

Effect of temperature

The effect of temperature on the removal of Pb, Cu, and Ni at temperature range of 28 to 70 ^{0}C was investigated and presented in fig 5. It was observed that adsorption of heavy metals increased with temperature and a significant increase was observed for temperatures up to 40 ^{0}C and this could be due to increase mobility of the adsorbate onto the active sites of the adsorbent at higher temperatures. However, the magnitude of such increase continue to decline as temperature is increased above 45 ^{0}C, this is because with increase in temperature, the attractive forces between biomass surface and metal ions are weaken and the sorption decreases, the thickness of the boundary layers are also reduced at higher temperatures hence the tendency for desorption of the already adsorbed metals suggesting further reaction as exothermic. (Horshfall and Spiff 2018).

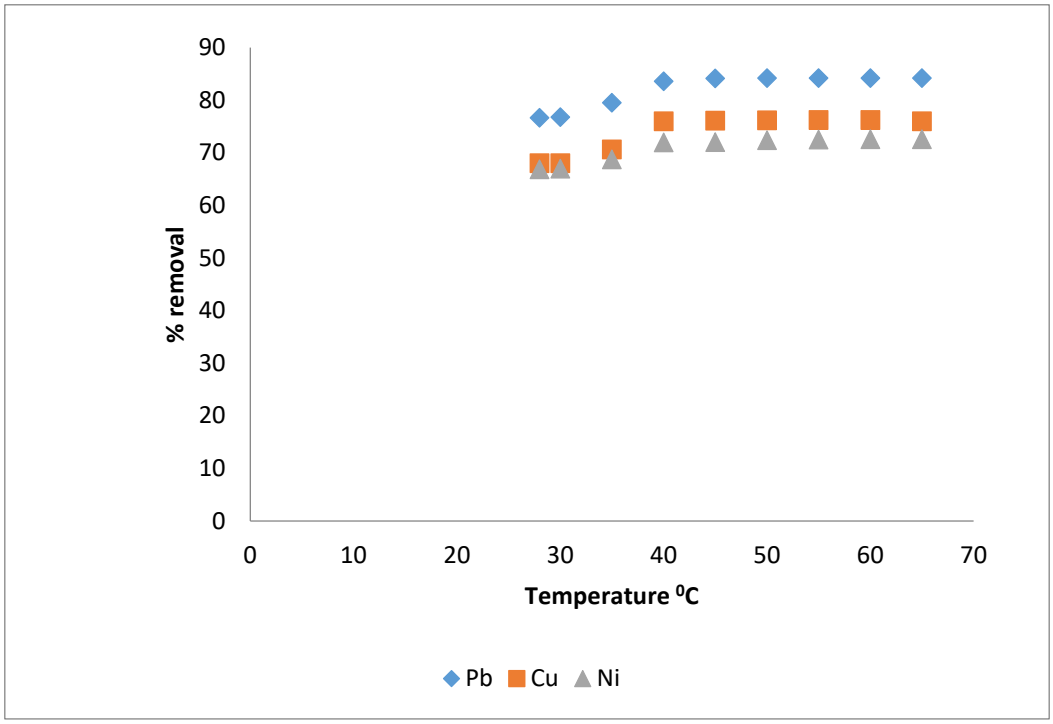

Fig 5: effect of temperature

Effect of concentration

The effects of the initial adsorbate concentration were studied. 1 g of F-CHAC was used for all concentrations of Pb, Cu and Ni. The initial concentrations of the adsorbate were determine using a dilution rate of 10 % at a pH of 2 and a temperature of 30 ^{0}C. The adsorption behavior of the adsorbent at different initial concentrations is shown in Fig.5. The result shows that the adsorption percentage of all the metals tends to decrease with increasing initial concentration. The maximum adsorption of Pb, Cu and Ni was observed at the lowest initial concentrations of 16, 12 and 10 mg/L respectively. The maximum adsorption removal of Pb, Cu and Ni was found to be 94.8 %, 99.7 % and 99.8 % respectively. However, on increasing the initial concentrations of the metals, there was a sharp decrease in adsorption percentage, and the lowest removal percentage was observed at 100 % concentrations (Pb:160 mg/L, Cu:122.2 mg/L, and Ni:100 mg/L). This is because at lower concentrations, the number of adsorbate molecules was less than the number of adsorbent molecules providing a high surface area for adsorption (Mishra *et al*., 2019).

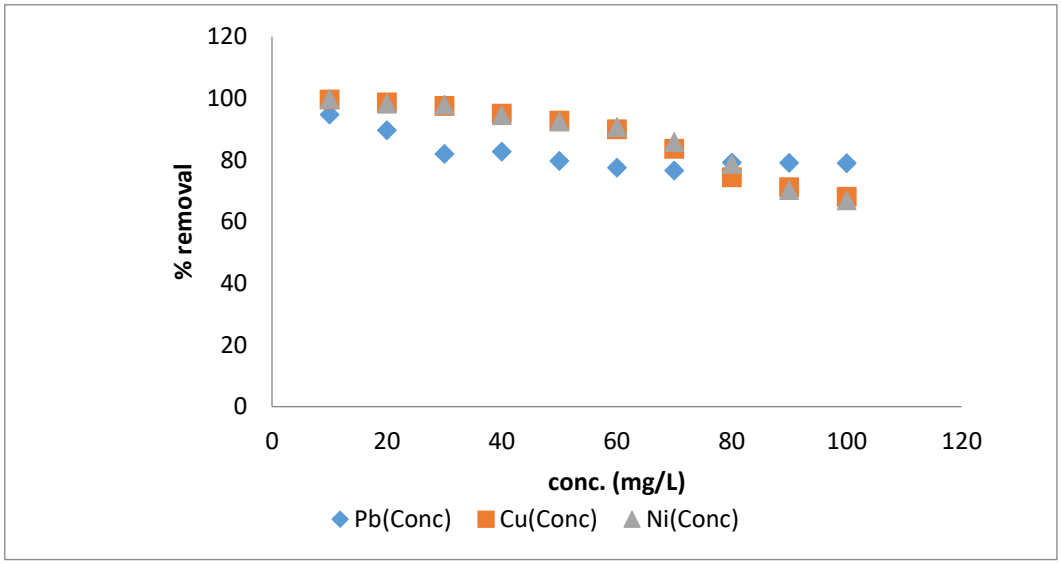

Fig 4.7: effect of concentration

Effect of solution pH

The solution pH is important parameter that determines the rate of adsorption, adsorption of all heavy metal ions is generally affected by pH value. In most cases it increases with pH value increase and reaches a plateau value at pH 4-6. A similar trend was observed in this study as the percentage removal increased steadily at pH below 5, the rate of adsorption became almost the same at pH of 4-8 for Pb, 5-8 for Cu and 6-8 for Ni as shown in figure 7 (Mustapha *et al.* 2021).

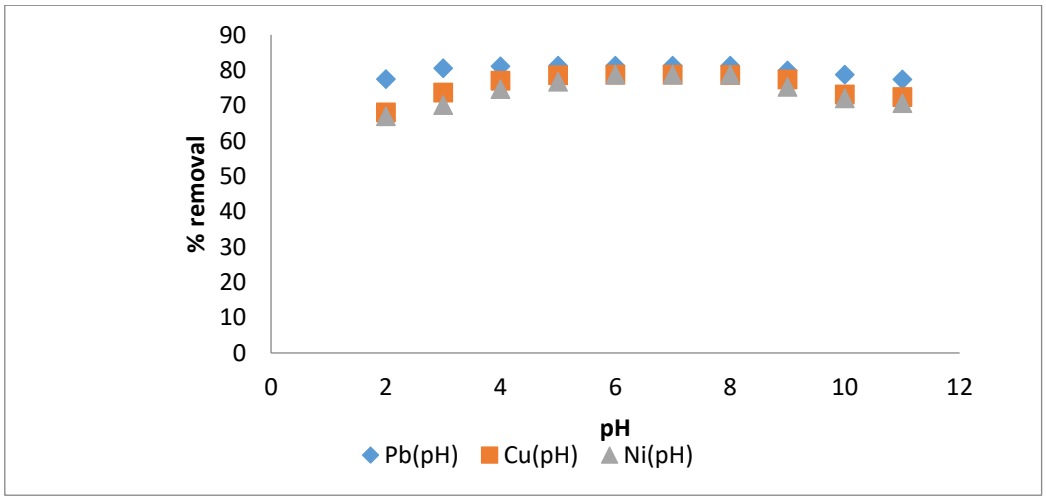

Fig 4.8: effect of pH

Adsorption Isotherm Models

Adsorption isotherms of Pb, Cu and Ni were obtained from the equilibrium sorption experiment investigated at adsorbent dosage ranging from 0.5 to 5 g. The experimental data were fitted using linearized regression co-efficient according to the Langmuir, Freundlich, Harkin-Jura, Elovish and D-R isotherm models as presented in Table 3. The fitted linear plot in Appendices suggests that adsorption rate increased with an increase in dosage. The fitness of the experimental data was determined with the regression correlation coefficient. It was found that the Freundlich model is the most suited model compared to Langmuir and D-R isotherm. The fitted parameters in Table 3 show that highest correlation coefficients were ascribed by Freundlich model. The Freundlich model explains the heterogeneous active sites present in the adsorbent and the results are in agreement with the finding of Xu *et al.* (2020). The adsorption intensity (1/n) values in Freundlich model for the pollutants lies between 0> (1/n) <1 indicating identical adsorption process and adsorption energy for all sites, it also suggests that the adsorbent is good for removal of the heavy metals. With regards to the correlation coefficient, the D-R and Harkin-Jura isotherm models did not fit well with the experimental data. In addition it is also stated that the use of these isotherms could tacitly change the error variance and normality assumption of standard least squares (Almasian *et al.,* 2017). For authenticity of the fitness of isotherm models, the fitness of equation to the experimental data error function was evaluated. The sum of square error (SSE) tests was used to buttress the best isotherm model as presented in Table 3a. The lower the error function, the better the isotherm model used for the adsorption of pollutant on to the adsorbent. Overall observation on the three isotherm model employed in terms of correlation coefficient and error analysis revealed that this study follows the trend Freundlcih > Elovich >Langmuir> Harkin-Jura> D-R. Table 3b shows comparison of this research with previous wors from literature.

Table 3a: Adsorption isotherm parameters for Pb, Cu and Ni

Isotherm	Parameter	Pb	Cu	Ni
Langmuir	Q_m	2.8137	0.7236	0.3369
	Q_e	2.2580	1.4920	1.0650

	K_L	0.5392	0.1526	0.1104
	R^2	0.9056	0.9716	0.8490
	SSE	0.31	0.59	0.53
Freundlich	K_F	1.7968	1.0321	0.6978
	Q_e	2.2580	1.4920	1.0650
	$1/N$	0.3139	0.3745	0.5287
	R^2	0.9743	0.9316	0.9266
	SSE	0.2127	0.2115	0.1348
Harkin-Jura	B	1.6874	1.6806	1.6623
	A	5.988	2.7655	1.1429
	Q_e	2.2580	1.4920	1.0650
	R^2	0.8058	0.7109	0.9135
	SSE	13.91	1.6218	0.0607
Elovich	K_e	2.3535	1.2054	1.2399
	Q_m	1.6984	1.5929	2.9069
	Q_e	2.2580	1.4920	1.0650
	R^2	0.9466	0.9221	0.6252
	SSE	0.313	0.0101	3.3925
D-R	K_{D-R}	3.0×10^{-7}	1.0×10^{-6}	3.0×10^{-6}
	E	-1290	-707	-408
	Q_e	2.2580	1.4920	1.0650
	q_m	4.4777	3.4573	2.2922
	R^2	0.7087	0.8849	0.5754
	SSE	4.9371	3.8624	1.5060

Table 3b: Comparison of F-CHAC's adsorption capacity with literature works.

Adsorbent	**Adsorption capacity/removal efficiency (mg/g)**	**Metal ion**	**Experimental condition**	**Surface area (m^2/g)**	**Reference**
Functionalized Magnetic Nanoparticles Supported	80 % Pb 53 % Cr	Cr(VI) and Pb(II) Ions	Contact time 3 h, pH 2, Temperature 25 ^oC	51.1	Fatehi *et al.*, 2017

on Activated Carbon		from Saline Solutions			
magnetic activated carbon incorporated with amino groups	104.20	Pb(II)	Dosage 1.0 g/L, pH 2	48.9	Fu *et al.*, 2016
EDTA modified yeast biomass coated with magnetic chitosan micro particle	134.90	Pb(II)	Contact time 120 min, Temperature 30 ^{o}C		Li *et al.*, 2013
Magnetic iron oxide (Fe3O4) nanoparticles from tea waste	4.81	Arsenic	Contact time 30 min, pH 6, Dosage 3 g/L, Temperature 30 ^{o}C		Lunge *et al.*, 2014
Corn husk derived magnetized activated carbon	96 % phenol 94 % para-phenol	Phenol and Para-phenol	Dosage 0.15 g, pH 3.2, Concentration 50 mg/L		Mishra *et al.*, 2019
Magnetic Activated Carbon Derived	3.23	Toxic dyes	Contact time 2 h, Dosage 2 g/L, pH 6.1,		Cazetta *et al.*, 2016

from biomass (Coconut shell) Waste Functionalized corn husk derived Activated carbon	2.26 1.49 1.07	Pb Cu Ni	Temperature 25 °C Contact time 60 min, Dosage 1 g, Temperature 30 °C, pH 2	442.70	This work

Adsorption Kinetic Models

The adsorption data on the removal of heavy metals from local battery recycling wastewater was subjected to kinetic analysis and the results presented in Table 4, from the tables, the kinetic models parameters were fitted by pseudo-first-order, pseudo-second-order models and intra-particle diffusion kinetic models. The correlation coefficient of the pseudo-second-order model described the data better, suggesting that the reaction depends on the contact time and the concentrations of the heavy metals in the wastewater. This suggests that Chemisorption played a significant role in the adsorption process (Xu *et al.*, 2020). Noticeability, the calculated k_2 value was highest for Pb(II) ions followed by Ni(II) and Cu(II) ions. The intra-particle diffusion factor K_i also suggest adsorption at intra-particle level (Mustapha *et al.* 2021)

Table 4: Adsorption kinetic parameters of Pb, Cu and Ni

First-order	k_1	$q_{e\ theo}$	$q_{e\ exp}$	R^2	**SSE**
Pb	0.002	5.2735	6.422	0.727	1.32
Cu	0.004	3.0063	4.844	0.718	3.38
Ni	0.007	1.9073	4.393	0.825	6.18
Second-order	k_2	$q_{e\ theo}$	$q_{e\ exp}$	R^2	**SSE**
Pb	0.0065	6.826	6.422	0.9908	0.16
Cu	0.0035	5.173	4.844	0.9453	0.11
Ni	0.0028	3.368	4.393	0.9812	1.05
Intra-particle diffusion	K_i	$q_{e\ theo}$	$q_{e\ exp}$	R^2	**SSE**

Pb	0.3197	3.1954	6.422	0.8317	10.41
Cu	0.2405	2.2159	4.844	0.9195	6.91
Ni	0.3203	0.8183	4.393	0.9867	12.78

Regeneration Study

The regeneration experiment for F-CHAC was investigated and the adsorption capacity of the adsorbent after each cycle is presented in table 5. The result shows that the F-CHAC has high adsorption capacities for Pb, Cu and Ni ions after the first 3 repeated cycles with near 100 % adsorbent regeneration, this however reduces gradually after the third run as the desorption efficiency drops from 33 to 25%. This further suggests collapse of the inner and outer pores or gradual blockage of the active sites that leads to change in structural composition of the adsorbent due to extensive exposure to HCl over time of desorption recycle. (Gautam *et al.*, 2014).

Table 5: Desorption and recovery efficiency of F-CHAC

No. of recycles	Adsorption capacity Q_m			% Desorption	%Re
	Pb (mg/g)	Cu (mg/g)	Ni (mg/g)		
1	6.1995	4.1515	3.3485	33.32	100
2	6.1585	4.1505	3.3155	33.15	99.33
3	6.1545	4.147	3.3005	32.97	99.27
4	5.371	3.634	3.1595	28.00	86.63
5	4.349	3.199	2.899	25.25	70.15

Conclusions

The study of heavy metal removal from local battery recycling wastewater using functionalized corn husk derived activated carbon was achieved with high efficiency. The introduction of nitrogen containing groups into the framework of the activated carbon confirms the amine functionalization of the corn husk derived adsorbent. The optimum contact time of 95 and 125 min was established for Pb and Cu, Ni removal respectively with removal efficiency of 99.36, 97.95 and 95.92 % at an optimal dosage of 3 g and the adsorption efficiency as influenced by temperature and pH shows exothermic sorption process as increased removal efficiency was achieved at certain high temperature and pH

while further increase in lead to lower sorption. The fruendlich isotherm established the multilayer adsorption The second order kinetic model was the best fit for the adsorption kinetics which confirms chemisorption played the most significant role in the sorption study and the values of enthalpy and Gibb's free energy further establish the exothermic and spontaneous nature of the adsorption process with high removal efficiency after three cycles of reuse.

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