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Optimization of Activated Carbon Preparation from Corncob Wastewater Treatment

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***Abstract**: Activated Carbon were produced from corncob, using phosphoric (H_3PO_4) acid as activating agent, and it was used for the removal of heavy metal ion in waste water. The Activated Carbon was produced at a temperature of 800℃ for two hours and batch adsorption study was carried out on the corncob activated carbon and its confirmed that at a temperature of 450℃ with Acid concentration of 30% with an Impregnation Ratio of 1.5, the yield was found to be 55.7% and 528 in (Mg/g) which is good for the removal of lead (ii) metal ion in water. The Activated Carbon produced was characterized to have a good mesoporous structure and thus recommended for the treatment of waste water.*

Key Words: *Activated carbon, synthesis, corn cob, optimization*

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1.0Introduction

The composition of heavy metals from industrial wastewater is a major concern for the environment, based on the rich of copper (Cu), lead (Pb), chromium (Cr), cadmium (Cd), (Zwain et al., 2014). Population growth, an increase in development and the expansion of investment in the industrial sector has led to the rise in demand for industrial products. The expansion and development of various types of industries can result in generating huge volumes of wastewater along with complex toxic chemical compositions which demand advanced technological treatment techniques (Corcoran, 2010; Keng et al., 2013; Zwain et al., 2014).

Furthermore, most industries in less developed countries discharge huge volumes of raw wastewater to water bodies, causing environmental and health damage to the local population.

2.0 Materials and Methods

2.1 Preparation of adsorbent

Corn cobs were collected from local community house in Agarfa, in Kaduna State, Nigeria. The samples were washed with distilled and deionized water before oven drying at 105 °C for 24 hours. The dried samples were crushed to and sieved to obtained 2 mm size and later carbonized in a horizontal cylindrical furnace, which has provision for in flow and maintenance of nitrogen atmosphere at 800 °C (Awugchew, 2015).

Chemical activation of the sample was also carried out using phosphoric acid. 100 g of the carbonized samples were mixed with 100 ml of solution (85% phosphoric acid) at different impregnation ratio of 1:1,1: 1.5, and 1:2 and at various concentrations of the acid (i.e, 30, 60 and 90% by weight). The mixture was heated in a mechanical heating mixer at a temperature 120 °C for 1 h, after which the content was extracted and dried for 12 hours. This was succeeded by washing the sample to neutral pH.

3.2 Characterization of corn cobs activated carbon

3.2.1 Iodine number

In order to test for iodine number, the activated carbon sample was treated with 10.0 mL of 5 % HCl. This mixture was boiled for 30 s and cooled before adding 100.0 mL of 0.1 N (0.1 mol L^{-1}) iodine solution. The resulting solution was filtered and 50.0 mL of the filtrate was titrated with 0.1 N (0.1 mol L-1) sodium thiosulfate, using starch as indicator. The amount of iodine adsorbed per gram of carbon (X/M) was plotted against the iodine concentration in the filtrate (C), using logarithmic axes. The iodine number was estimated as the X/M value when the residual concentration (C) is 0.02 N (0.02 mol L^{-1}).

3.2.2 Carbon Yield

The total yields were determined after sample processing in terms of raw material mass. The dried weight, Wo of each pre-treated sample was determined using Mettler balance and the carbon yield was calculated as; Yield % = Wc/Wo*100

where: (Wo) is dry weight before carbonization and (Wc) is dry weight of activated carbon produced from produced corn cob.

3.2.3 Determination of moisture and ash content

The moisture content was determined using the method of difference (ref) while the ash content was determined directly through the mass of uncarbonized sample.

3.2.4 Determination of volatile content

Volatile content was estimated as the difference in weight between the sample before and after volatilization (at 105 °C)

3.3 Experimental design

The experimental data were analysed using a statistical software Design Expert software version 7.0.0 to initiate regression analysis and also for the evaluation of the statistical significance of the equations developed.

3.3.1 Factorial design methodology

Full factorial design with respect to three factors associated with batch adsorption techniques were used. These included the initial concentration of lead, pH and adsorbent dose In the full factorial design considered, twenty seven runs were conducted for production of corn cobs activated carbon (CCAC) where three factors each at three levels (high and low) were studied. The response variables considered were iodine number and carbon yield. The three factors considered were: activation temperature (AT°C), acid concentration (AC), and impregnation ratio (IR), The levels of each factor were activation temperature (A) were: 400 ,450 and 500°C. The three levels of acid concentration are 30, 60 and 90 % and the three levels for impregnation ratio were 1,1.5, and 2 w/w.

3.0 Results and Discussion

The effect of (60%) concentration on the yield of corn cob activated carbon at constant activation temperature of 450 °C, impregnation ratio of 1.5 w/w and activation time of 2h is represented in terms of a plot of % yield against concentration of H_3PO_4 in Fig. 1.

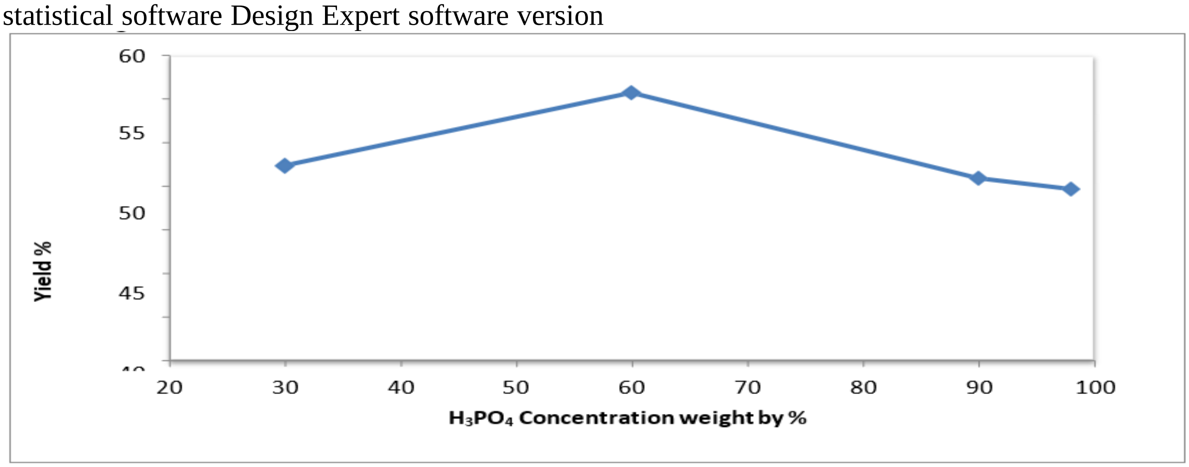

Fig. 1: Variation of % yield with concentration of H_3PO_4 (at a temperature of 450 °C, impregnation ratio of 1.5 w/w and activation time of 2h)

The plot reveals initial rise in the yield of CCAC shows strong dependent on concentration of H_3PO_4. The yield was highest at concentration of 60% and then decreases for other concentration. This implies that the optimum concentration of the acid required for efficient production of CCAC is 60%. Fig. 2 shows similar plot with respect to temperature.

From the figure, it can also be seen that best yield was obtained at 450 °C while percentage yields for other temperature were low.

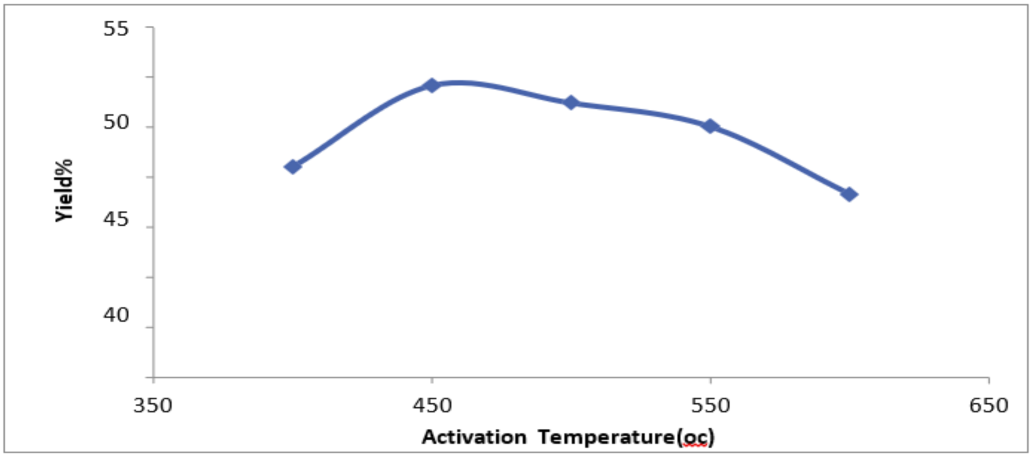

Fig. 2: Effect of activation temperature on the yield of corn cob activated carbon

The observed effect of activation temperature and H_3PO_4 concentration on the yield of the produced activated carbon can be linked to the dependency of iodine absorption on temperature as shown in Figs. 3 and 4 respectively.

From Fig. 3, it is evidence that maximum iodine absorption occurs at this optimum temperature and that outside this temperature (450 °C <T<450 °C), the yield is lower than optimum.

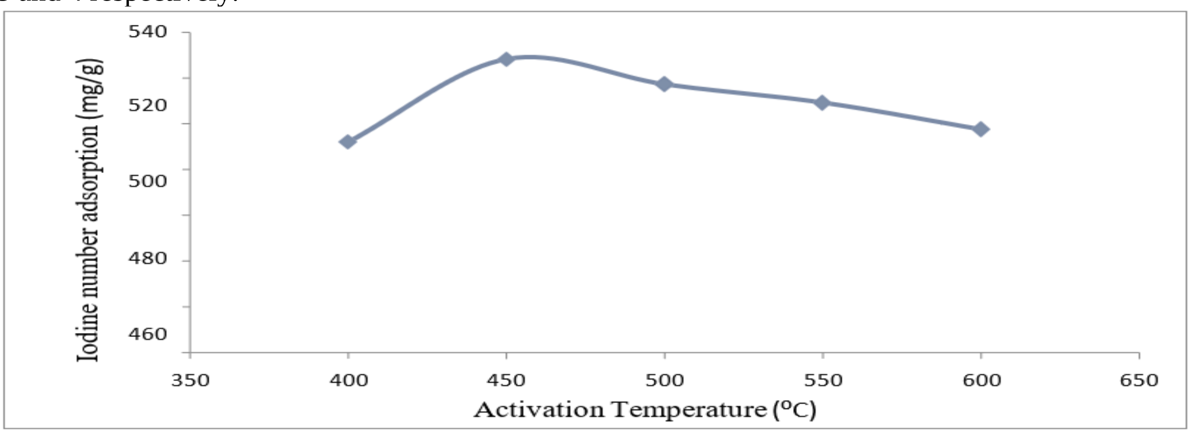

Fig. 3: Variation of iodine number with activation temperature

The observation can be attributed to structural deformation when the temperature is below the optimum and incomplete activation when the temperature is above the optimum. On the other hand, Fig.4 also reveals that the optimum concentration of H_3PO_4 (60%) that gave best yield also correspond to the concentration that gave highest iodine absorption. Therefore, under these optimum temperature and concentration optimum porosity of the produced activated carbon is also observed.

The iodine values obtained in this study ranged from 211 to 528 mg/g. The corn cob activated carbon produced chemically using phosphoric acid has the highest iodine number of 528 mg/g. This indicates highly active surfaces present on the activated carbon, with high porosity and surface area. The optimum conditions were similar to those reported by Yahaya *et* al. (2015). The observed analytical differences in iodine values maybe be attributed to differing adsorptive characteristics arising from differences in specific surface area, pore size and pore volume of the carbon. (Zahra, 2012).

The results obtained from the effect of impregnation ratio on the iodine number is represented in Fig. 5 and it reveals that the highest iodine value was obtained at optimum impregnation ratio of 1.5. Values less or higher than this optimum gave lower iodine value. Therefore, impregnation ratio is a significant parameter that affects the development of micro porous structure in the synthesized activated carbon.

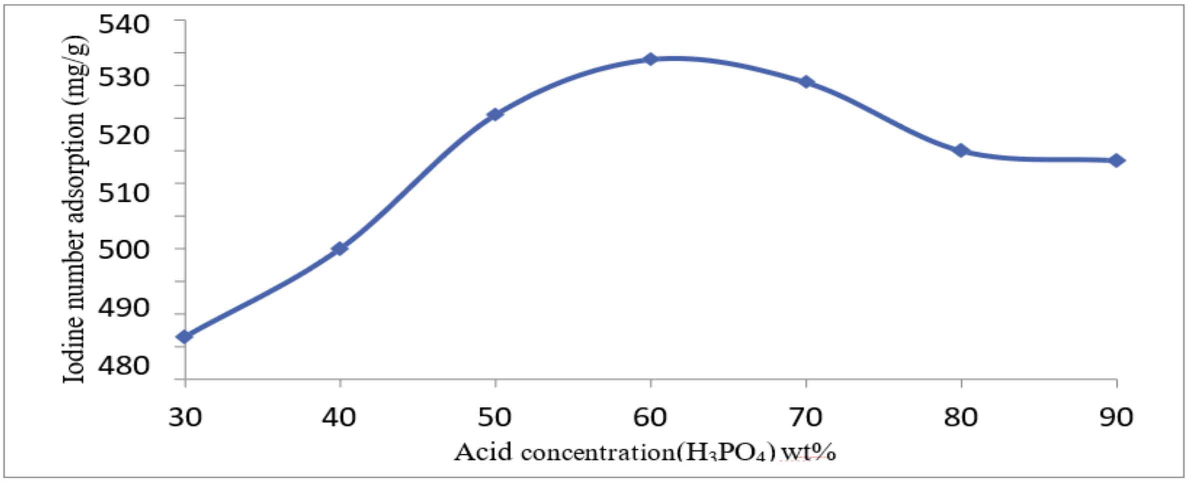

Fig. 4: Variation of iodine number with concentration of H_3PO_4

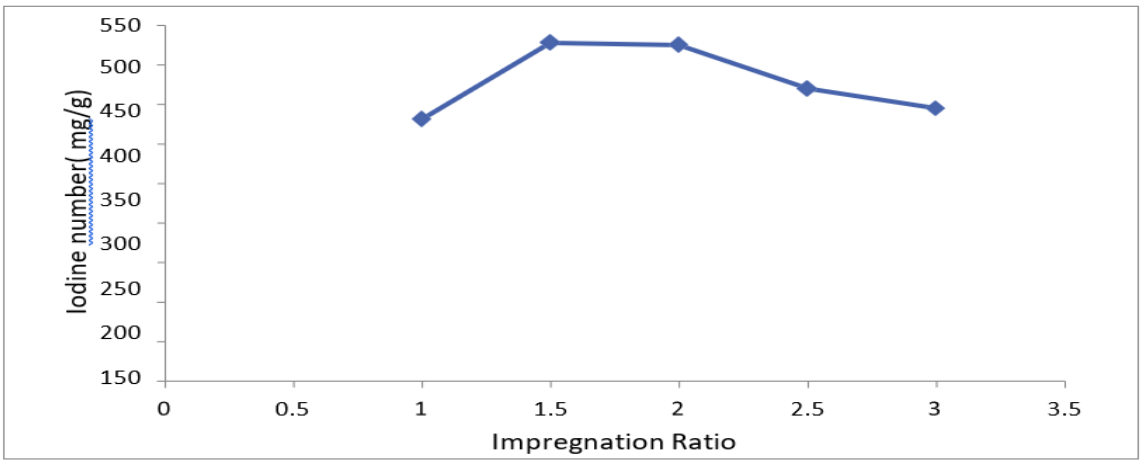

Fig. 5 Effect of impregnation ratio on iodine number adsorption

From the analysed results, it can be stated that optimal conditions required for best production of CCAC are acid concentration impregnation ratio of 1.5 and activation temperature of 450 °C.

3.1 Characterization of Corn Cobs Activated Carbon

Characteristics of the activated carbon prepared under the optimal conditions mentioned above, are presented in Table 1. The results are in line with those reported by some researchers in activated carbon (Awugachew, 2015; Khan, 2015). However, the observed yield (55.7), defined as the weight ratio on a dry basis, of the resulting activated carbon to that of the original corn cob waste is significantly higher than the value reported for other lignocellulose materials, which give an edge over others since increase in yield reduces production cost and point towards raw materials optimization

The volatile constituents of our CCAC is also lower than those reported by Awugachew (2015) and by Khan (2015) but is in good agreement with the work of Hiremath and Shivayoginath (2012).

The bulk densities observed in this work are in close agreement with values of 0.3876 g/m^3 reported for other agricultural raw materials. (Khan *et al*., 2015). The moisture contents of 10.5% for acid corn cob activated carbon. This observation is in agreement with studies by who reported the moisture content of corn cob (Zhang *et al*.,2012). The observed porosity value of 48.8% shows that the surface area for the activated carbons fall within the range of 400 to 900 m^2/g. Possession of high surface area by adsorbent is essential factor since it is a limiting factor to the number of materials that can be adsorbed. The surface areas were higher for acid activated carbons and this is expected because chemical activation normally develops more porosity and gives high surface area when compared with thermal activation (Barakat, 2011).

Table 1: Compared characteristics of the optimal activated carbon

	Activating agent	Adsorbent characteristics									Reference
		pH	Porosity (%)	MC (%)	AC (%)	VC (%)	FC (%)	Yield (%)	IN (mg/g)	BD (kg/m^3)	
Corncobs	Chemical (H_3PO_4)	6.2	48.8	10.5	1.5	78.2	24.8	55.7	528	287.6	This work
Bamboo stem	Chemical (H_3PO_4)	6.4	-	7.6	5.6	24.4	62.4		-	650	(Khan *et al.*, 2015)
Coffee husk	Chemical (H_3PO_4)	5.4	57.3	6.3	9.4	13.2	71.1	57.09	396	690	Awugachew (2015)

3.2 FTIR-characterization of the corn cob activated carbon

Pore structure of activated carbon is a significant parameter in assessing the adsorption properties of an adsorbent and can be assessed through the functional groups present in the adsorbent. The FTIR spectra of the adsorbent (Fig. 6) before and after activation reveals that the OH stretch at 3400 shifted to 3403 cm^{-1} with increasing intensity, the C-H stretch at 3000 shifted to 2938 cm^{-1} with increasing intensity, the C=C stretch at 1500 shifted to 1649 cm^{-1} with increasing intensity and the band at 730 shifted to 752 cm^{-1} with increasing intensity after activation. According to Essien and Eddy (2015), suitable functional groups are essential for effective adsorption. Therefore, the activation process enhanced the adsorption capacity of the corn materials.

3.3 Full factorial design for corn cobs activated carbon production

Tables 2 represent the design matrix, main effects, and interactions between factors, the generated regression models and the ANOVA analysis. Results in Table 2 shows a significant (positive) effect activation temperature (factor), acid concentration (factor) and a moderate (positive) effect of the IR (factor), on the carbon yield and iodine number. These results prove that the three factors contributed significantly towards optimum operating conditions for the production of corn cobs activated carbon.

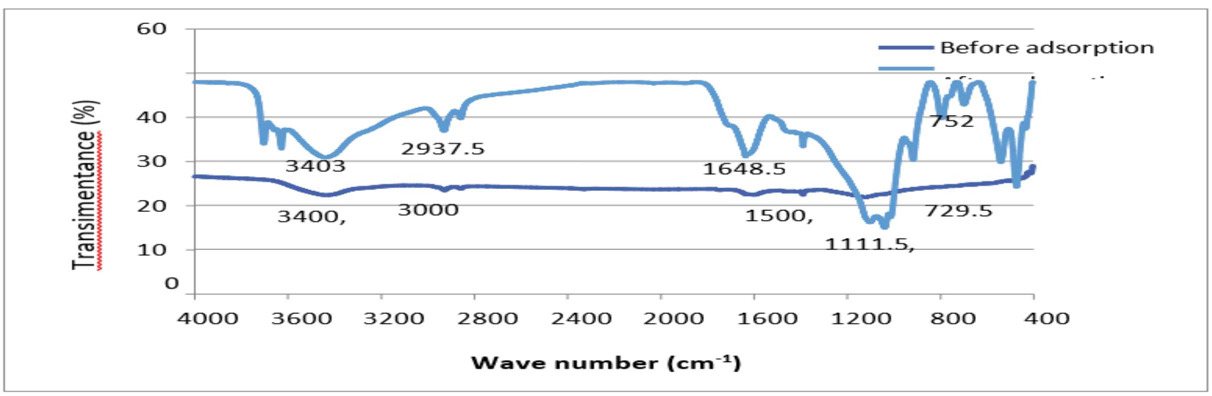

Fig. 6: FTIR of the corn cob before and after activation

The ANOVA analysis shown in the lower part of the table indicated that at there is a linear relationship between the calculated carbon yield and the three factors considered.3-D surface plots and contour plots for the response variable were also generated and are shown in Figs. 7 and 8 for carbon yield. These graphs prove the validity of conclusions presented earlier regarding the interactions between the three factors.

The significance and adequacy of the models were further justified through analysis of variance (ANOVA). F-value is the ratio of the mean square owing to regression to the mean square owing to error. The higher the F-value, the greater is the significance of the corresponding variable to cause effect. In addition, if Prob.>F less than 0.05, the model terms are considered as significant. (Azmier *et al.*, 2011). Therefore, the interaction effects between the acid concentrations are considered high. The model F-value of 3.62 and Prob.>F of 0.0135 implied that this model was significant.

Table 2; ANOVA for yield of corn cobs activated carbon (CCAC)

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F
Model	578.5111	6	96.4185	3.6214	0.0135
Activation temperature(x_1)	30.7067	1	30.7067	1.1533	0.0029
Acid concentration(x_2)	29.8507	1	29.8507	1.1212	0.0302
Impregnation ratio(x_3)	159.0733	1	159.0733	5.9746	0.0239
	5.3067	1	5.3067	0.1993	0.6600
	104.9617	1	104.9617	3.9422	0.0610
	248.612	1	248.612	9.3376	0.0062

3.4 Effect of independent variables on the yield of CCAC

Results obtain from factorial analysis show that the absolute effect of the activation temperature (AT) and acid concentration (AC) are negative while that of Also, observed was a moderately positive value corresponding to significant interactions between the acid concentration (AC) and impregnating ratio (IR).

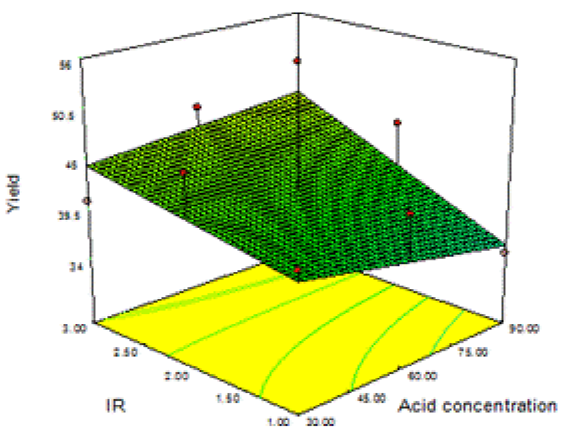

Fig. 7: Effect of activation temperature and impregnation ratio on the CCAC yield

Acid concentration and impregnation ratio are the important factors that govern the development of porosity of the prepared carbon. The interaction effect of these two parameters on the carbon yield of the adsorbent is shown in Fig. 8, which reveals that the carbon yield increases rapidly with increasing acid concentration upto 60%.

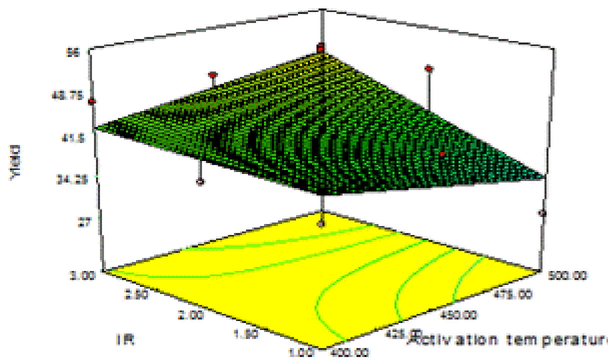

Fig. 8: Effect of acid concentration and impregnation ratio on the CCAC yield

From Fig. 9, it can be observed that all three factors work very well at the high acid concentration 60% and impregnation ratio of 1.5. None of the factors perform very well at the lowest temperature 400°C.

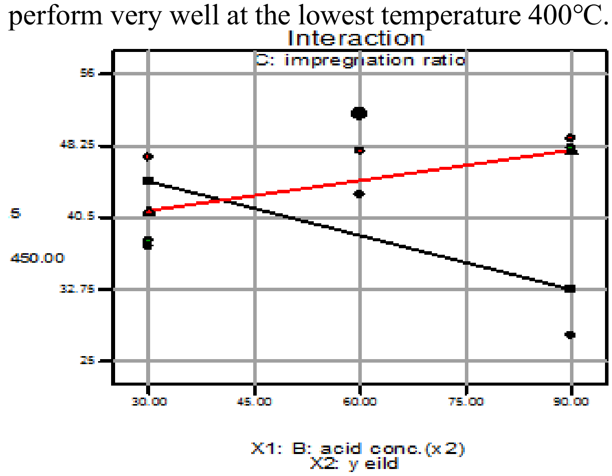

Fig. 9: Interaction effect acid concentration and IR on the CCAC yield

3.5 Effect of independent variables on the iodine number adsorption of CCAC

The ANOVA analysis shown in the lower part of Table 3 proves the results discussed above at a confidence level of 95%. The regression model generated for the full factorial designs using the twenty-seven runs conducted using each factor. As we observe the agreement between the experimental and the predicted values is excellent. This indicates that there is a linear relationship between the calculated iodine number adsorption and the three factors considered.

3-D surface plots and contour plots for the response variable were also generated and are shown in Fig. 10 for iodine number. These graphs prove the validity of conclusions presented earlier regarding the interactions between the three factors. Referring to F-value shown in Table 3 the main effect between the activation temperature and impregnation ratio were considered high. The model F-value of 2.99 and Prob.>F of 0.0298 implied that this model was significant.

Table 3: ANOVA for iodine number adsorption of corn cobs activated carbon (CCAC)

	Sum of		**Mean**	**F**	**p-value**
Source	Squares	Df	Square	Value	**Prob > F**
Model	578.5111	6	96.41852	3.6214	**0.0135**
Activation temperature(x_1)	30.70667	1	30.70667	1.1533	**0.0029**
Acid concentration(x_2)	29.85069	1	29.85069	1.1211	**0.0302**
Impregnation ratio(x_3)	159.0733	1	159.0733	5.9746	**0.0239**
	5.3067	1	5.3067	0.1993	**0.660**
	104.9617	1	104.9617	3.9422	**0.061**
	248.612	**1**	**248.612**	**9.3376**	**0.0062**

The effects of activation temperature and impregnation ratio are the important factors that govern the development of micro porous of the prepared carbon as shown in Fig. 1 which reveals that the quantity of iodine number adsorbed increase with increase in activation temperature upto the optimal at 450 °C Several investigations have ~~has~~ established that in the case of H_3PO_4 activation of other agricultural wastes (woods, coconut shell, coffee husk, grain sorghum) were observed at a temperatures close to 450 °C (Yun *et al*., 2011)

3.6 Batch adsorption study

The equilibrium adsorption capacity and removal efficiency were determined using batch adsorption experimental methods. The results obtained are presented in Table 4.

The initial metal ion concentration provides an important driving force to overcome all mass transfer resistances of metal ion between aqueous and solid phases.

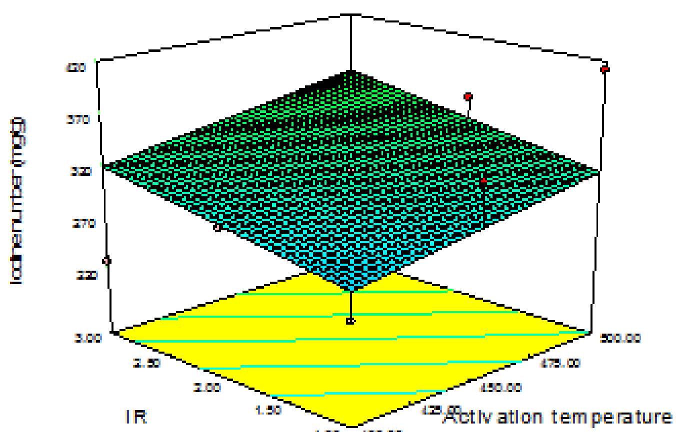

Fig. 10: Effect of activation temperature and impregnation ratio on the iodine number

The observed increase can be attributed to increase concentration gradient which led to increase in the number of metal ions diffusing to the surface of the adsorbent coming in contact with the adsorbent

Table 4; the average experimental design matrix for CCAC Pb^{2+} removal efficiency and adsorption amount from three replication data

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F
Model	82337.64	6	13722.94	2.9929	0.0298
Activation temperature(x_1)	22840.97	1	22840.97	4.9814	0.0372
Acid concentration.(x_2)	8192	1	8192	1.7866	0.1963
Impregnation ratio(x_3)	45300.5	1	45300.5	9.8796	0.0049
x_1 x_2	752.0833	1	752.0833	0.1640	0.6898
x_1 x_3	5166.75	1	5166.75	1.1268	0.3011
x_2 x_3	85.33333	1	85.3333	0.0186	0.8929

The percentage removal efficiency of lead (II) decreases from 96.8% to 41% as the initial concentration is increased from 10 mg/L to 100 mg/L. The removal efficiency at a fixed adsorbent dose on the effect of initial concentration of Pb^{2+} ions is depicted in Fig. 11. The trend indicates that the amount of lead ions removed decreases as the initial concentration increases which may be attributed to the competition between forces of adsorption and desorption (Odoemelam *et al*., 2018).

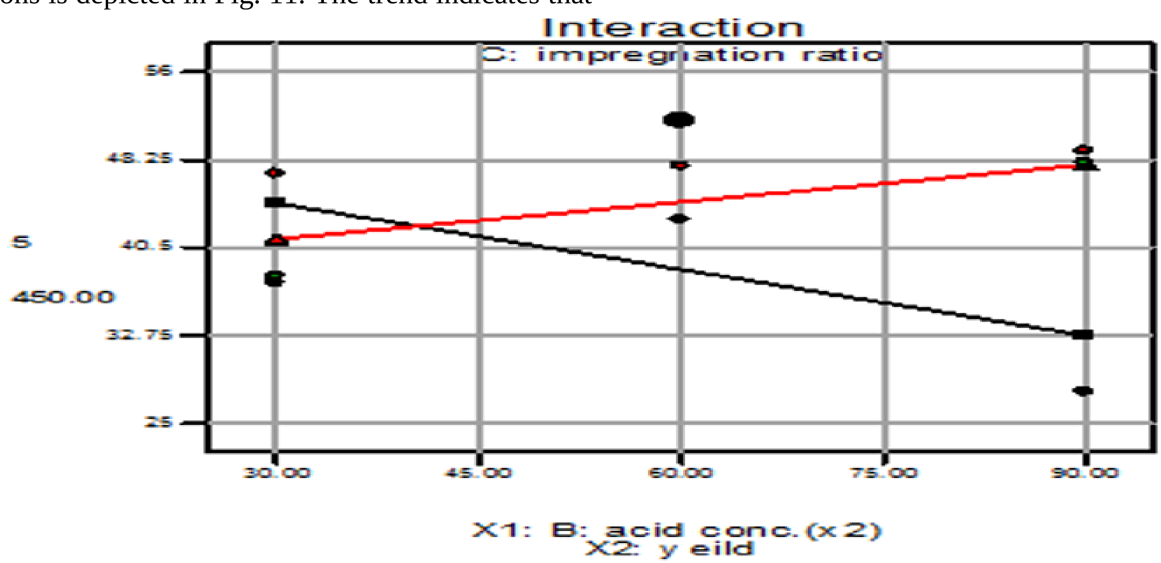

Fig. 11: Interaction effect acid concentration and IR on the CCAC yield

Effect of contact time on the removal of lead is illustrated in Fig. 12, which shows that the amount of lead ions adsorbed increases with contact time upto 30 minutes, after which, further increase in time did not significantly increase the amount of lead ions adsorbed. This may be attributed to the saturation of available adsorption sites (Farooq, 2013). pH is an important parameter in the adsorption process. The pH of solution has a significant impact on metal uptake since it determines the surface charge of adsorbent and the degree of ionization and speciation of adsorbate (Alves *et al*., 2013).

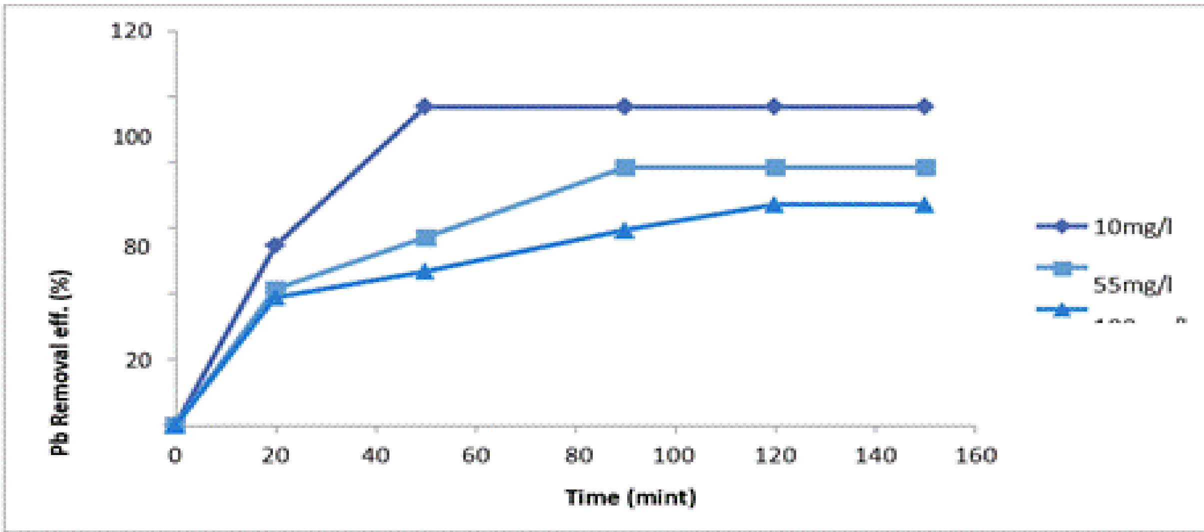

Fig. 12; Effect of contact time on the removal efficiency of lead (II) by CCAC. Initial concentrations: 10mg/L, 55mg/l and 100mg/L; adsorbent dose: 3g and pH; 5:

Variation of the amount of lead ions adsorbed with pH is shown in Fig. 13. Lead uptake was observed to increase with increasing pH up to optimal pH of 5. Acidic pH has been reported to enhance adsorption by several pH and is mostly attributed to the presence of hydroxonium ion in the solution. However, as acidity decreases, concentration of hydroxyl ions increases while that of oxonium ion decreases leading to decreasing adsorption capacity (Aziz *et al*., 2005; Geetha & Belagali, 2013).

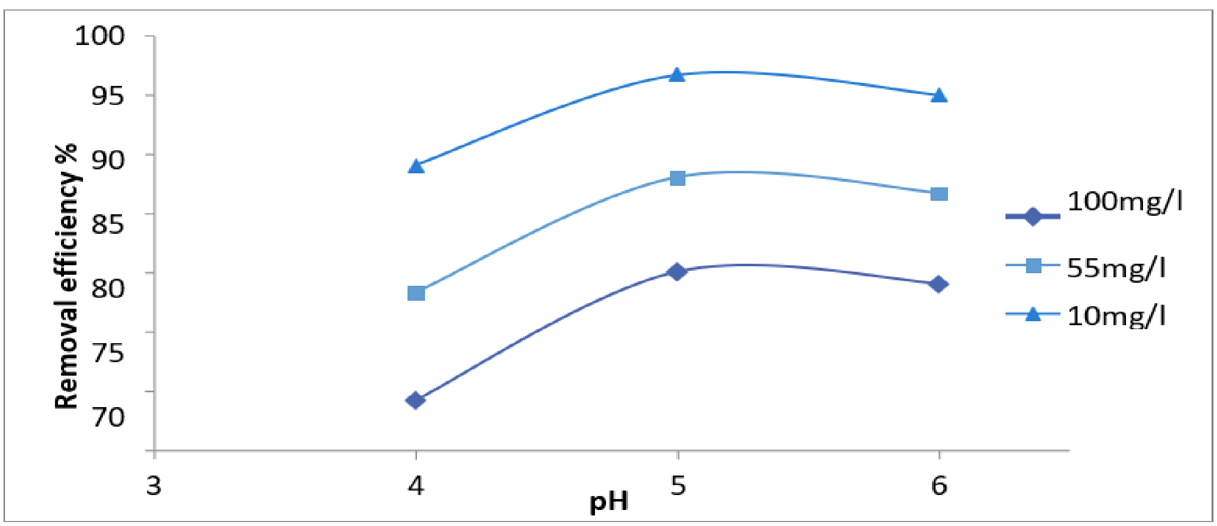

Fig. 12: Effect of the pH on sorption of Pb^{+2} onto CCAC

The dose of adsorbent is another important parameter in the adsorption process. Fig. 13 shows the variation of amount of lead ions adsorbed with adsorbent dosage. The variation of adsorption capacity is also shown in the same graph. It is evident from the plots that adsorption of Pb^{2+} increased from 41.00 to 96.8 % when the adsorbent dosage was increased from 1 to 3 g/L, respectively.

This is because for a fixed initial metal concentration, increase in adsorbent dosage led to increase in surface area and the number of adsorption sites (Okwunodulu and Eddy, 2013; Kundu *et al*., 2014). However, the plot of capacity (metal uptake per adsorbent unit) versus adsorbent dose revealed that the capacity was high at low doses and low at greater dose of adsorbent, which shows increase in adsorption with the growth of adsorbent. Similar results were reported by Khan *et al*. (2015) for the adsorption of Pb^{2+}using bambo as adsorbent

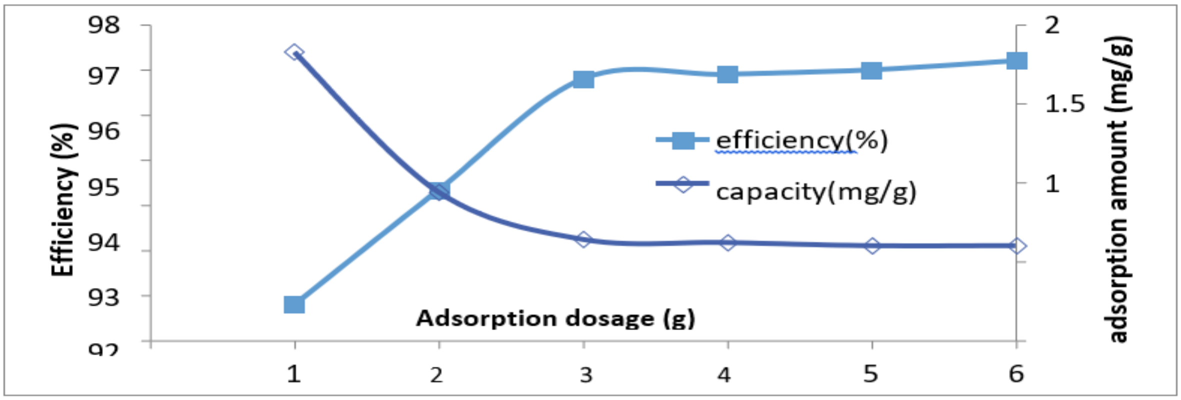

Fig. 12: Adsorption amount and removal efficiency of Pb^{2+} at different adsorbent dose

4.0 Conclusion

Based on the result obtained within the frame work of this study it appears that the activated carbon prepared from corn cobs constitutes a good adsorbent for removing of heavy metal Pb^{2+}ions from it aqueous solution. The main conclusions that can be drawn from current investigation are given below: The optimum pH for lead removal was found to be 5. The percentage uptake was found to be highly dependent on the initial concentration of the adsorbate and sorbent fractional adsorption becomes dependent on initial concentration. For fixed adsorbent dose, the total available adsorption sites are limited thereby adsorbing almost the same amount of sorbate thus resulting decrease in percentage removal of the adsorbate corresponding to an increase initial sorbate concentration.

Adsorption capacity of adsorbate had seen to decrease with increasing adsorbent dose while the efficiencies increased. In addition, a decrease in efficiency of adsorbent was observed with increasing initial metal ion concentration. This study indicated that lead ion could be removed by an inexpensive agricultural waste product—the CCAC with the minimum equilibrium time<2h.

This result is also confirmed in the ANOVA analysis given by the p-value. For the selected response variables studied using the regression models generated, good agreement between the experimental and predicted values was observed.

It can therefore be concluded that agricultural by-products which are inexpensive, readily available are the effective adsorbents for the adsorption process is expected to be economically for wastewater treatment.

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Conflict of Interest

The authors declared no conflict of interest