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Response Surface Methodology as a Tool for Optimization of Metal Adsorption by Banana Peel Biochar

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A B S T R A C T

Agricultural wastes are considered as viable alternatives because of their abundant availability, low cost and also with various functional groups such as carboxylic acid, ester, carboxylate, hydroxyl, phenolic and amino that can act as adsorption sites for heavy metal ion. Banana peel waste based biosorbents to remove cadmium from waste water was taken up for the present study. Banana peel biochar was used as biosorbent and biosorption study was conducted at different pH (4, 5, 6, 7 and 8), different concentration (0, 10, 25, 50, 75 and 100 ppm) and different contact time (0, 1, 2, 3, 4, 6 and 8 h). The adsorption of Cd by BPB was increased along with increase of pH from 4 to 7, followed by pH 8. The highest adsorption (91.51 %) was observed at pH 7 and 75 ppm concentration. The adsorption of Cd (86.69 %) was recorded in 6 hours contact time. The FTIR spectrum of BPB after Cd adsorption revealed a large number of peaks within the interval of 4000-400 $cm^{-1.}$ Alkane functional group with (C-H) band was detected at 2918.73 cm^{-1} frequency before adsorption but after adsorption of Cd, it was decreased to 2917.77 cm^{-1}. Alkene functional group with (=C-H) band was reported at 997.982 cm^{-1} adsorption frequency before adsorption but after adsorption of Cd, the alkene functional group was changed to ether with (C=O) band at 1013.41 cm^{-1} frequencies. There were no changes in Alkyl Halide functional group before adsorption and after adsorption of Cd. Hence, the presence study with BPB for Cd removal is effective due to the negative surface charge on the of the biochar. It also an ecofriendly technique to remove heavy metal from the wastewater.

Introduction

Heavy Metal contamination is gradually more being recognized now a day in the developing world mainly in India and China (Cheng, 2003).Cadmium, the seventh most toxic metal as per according to ATSDR (2007), instigates cancer, damage to bones, kidney and mucus membrane. It also causes vomiting and affects hormone secretion. The search for new technologies to remove toxic metals from wastewaters has directed attention to biosorption, which is an economically affordable alternative, remains one of the key priorities among researchers. Only within the past decade the potential of metal biosorption by plant based materials have been recognized. Many crop residues are good adsorbents better than activated carbon and ion exchangers and remain unused. These waste materials contains various functional groups such as carboxylic acid, ester, carboxylate, hydroxyl, phenolic and amino that can act as adsorption sites for heavy metal ion (Ashraf *et al.*, 2011).

Banana (*Musa sapientum*) is one of the most popular fruit crop grown in more than 130 countries, particularly in tropical and sub-tropical regions of the world. Banana peel, represents about 40% of total weight of the fresh fruit, totally 40 million tons of banana peel generated annually (Anhwange and Joseph, 2009), is considered to be a waste material. Exploring alternative uses of banana peel would thus bring an additional value to the industry. As reported by Mohapatra *et al.,* 2010, banana peel is found to be a good source of pectin (10-21%), lignin (6-12%), cellulose (7.6-9.6%), hemicelluloses (6.4-9.4%), and galactouroninc acid.

Annadurai *et al*. (2002) reported that acid (HNO_3) and alkali (NaOH) treated banana peels showed adsorption capacity of divalent heavy metal ions and the maximum metal adsorptions were recorded as 7.97 (Pb^{2+}), 6.88 (Ni^{2+}), 5.80 (Zn^{2+}), 4.75 (Cu^{2+}), and 2.55 mg g^{-1} (Co^{2+}). Practical applicability of banana peel to remove arsenic from contaminated water samples collected from 8 different areas of Sindh, Pakistan was reported by Jamil *et al.*, (2008).

Anwar *et al.*, (2010)studied the effect of adsorbent dosage 10-90 g/L on the adsorption of Cd^{2+} and Pb^{2+} and the maximum removal was observed at the doses of 30 and 40 g/L, respectively, for Cd^{2+} (89.2%) and Pb^{2+} (85.3%). Thorough scanning of literatures showed that only limited work has been carried out with reference to banana peel as adsorbent especially for cadmium. Hence an attempt to develop banana peel based adsorbent may provide a viable solution to removal of cadmium from wastewater.

Materials and Methods

Collection and preparation of banana peel biochar

The banana peels were collected from the nearby chips industry and the peels were thoroughly washed with tap water to remove external dust particles and kept in sunlight for drying. After sun drying, the banana peels were kept in an oven at 70°C for 2h and used for biochar preparation. Banana Peel biochar (BPB) was prepared through the process of slow pyrolysis at 400-600°C at the Department of Bioenergy, Tamil Nadu Agricultural University, Coimbatore to obtain a stable product that was used as biochar.

The charcoal obtained in the reactor is calculated as,

$$\text{Charcoal Yield, \%} = \frac{\text{weight of charcoal}}{\text{weight of biomass}} * 100$$

Characterization of banana peel biochar

The Banana Peel Biochar (BPB) was subjected to various characterization analyses such as electrochemical properties, biochemical components, elemental composition and Fourier Transform InfraRed Spectroscopy (FTIR). The electrochemical properties like pH, EC and CEC ((Jackson, 1973), Organic carbon ((Jackson, 1973)), Total Nitrogen, Crude fiber and Protein content (Sadasivam and Manickam, 2009), Moisture content, Ash content (Gupta, 2007) of the BPB was analysed as per the standard methods. Pectin was extracted from sample through Gravimetric Method (Sadasivam and Manickam, 2009).

Elemental composition in BPB was determined as per standard methods. Total Nitrogen was determined as per Micro-Kjeldahl Method and Total Phosphorus and Total Potassium was estimated in Flame photometer (Jackson, 1973). Calcium and Magnesium was estimated by Versenate titration (EDTA) and Sodium was determined by Flame photometer method (Gupta, 2007).

Iron, Manganese and Zinc was determined in atomic absorption spectrophotometer method (Tandon, 2013). The elemental composition of specimen surfaces and the percentage weight of chemical compositions were analysed in EDX analyser (Jamil *et al.,* 2008).

Before and after adsorption of Cd the banana peel biochar was analyzed for the presence of functional groups by FT-IR(Model 8400S of Shimadzu, Japan) using Attenuated Total Reflectance (ATR) technique having wavelength source of (400-4000 cm^{-1}) (Trakal *et al.,* 2014). The surface charge of BPB samples were determined by measuring the zeta potential (Ucar *et al.,* 2014).

Optimization of adsorption potential of BPB

A batch study was carried out to optimize the various incubation conditions for metal adsorption. Adsorption of Cadmium was studied at different incubation time, different metal concentration and various pH. The dose of adsorbents was 1g in 100 ml working standard solution, size was 0.5 mm and equilibration time was 24 h at room temperature. The various pH employed here are 4 to 8, contact time from 0 to 8 hrs and concentrations were 25, 50, 75 and 100 mgL^{-1}. $CdCl_2$. H_2O was used to prepare different concentrations of Cd. All the treatments were replicated thrice with and without banana peelbiochar.

AAS analysis

The samples were analyzed spectrophotometerically using Atomic Absorption Spectrophotometer (Perkin Elmer) in the Department of Environmental Science, TNAU by using appropriate instrumental parameters. The amount of heavy metal adsorbed per unit mass of the adsorbent (Q) was calculated by using the following equation (Priyanka, 2017):

$$\text{Adsorption \%} = \frac{(Ci - Cf)}{Ci} \times 100$$

Where, C_i is the initial concentration of metal ion in solution before sorption (mg L^{-1}), C_f is the final metal ion concentration after the sorption analysis (mg L^{-1}).

Response Surface Methodology (RSM)

Optimization of various conditions for the biosorption of cadmium (Cd) was determined by response surface methodology (RSM). Central composite design (CCD) was used to study the effects of three independent variables *viz.,* initial metal concentration, contact time and initial pH on the Cd removal(%)(Ghosh *et al.,* 2015).The central composite design was used due to its suitability to fit quadratic surface which usually works well for process optimization.

For statistical calculation, the variables were coded as given below:

$$x_i = \frac{X_i - X_0}{\Delta X} \quad (1)$$

Where X_i is the dimensionless coded value of the *I* th independent variable, X_0 is the value of X_i at the centre point and ΔX is the step change value. The behaviour of system is explained by the following empirical second-order polynomial model

$$Y = b0 + b1A + b2B + b3C + b12AB + b13AC + \cdots \quad (2)$$

Where *Y* is the predicted response, bn is the coefficient associated with factor n, and the letters, A, B, C,…. represent the variables in the model.

The DESIGN EXPERT 6.07 (Stat-Ease Inc., Minneapolis, cMN, USA) software was used for regression and graphical analysis of the data. By solving regression equation of the desired variables, the optimum values of the selected variables were obtained as the optimization criteria. The variable A (Initial metal concentration) varied from 25 to 100, B(Contact time) varied from 0 to 8 and variable C (pH) varied between 4 to 8.

Results and Discussion

Characterization of Banana Peel Biochar (BPB)

The biochar was prepared from the dried banana peel by slow pyrolysis process and the final product obtained was used as banana peel biochar. Banana Peel Biochar (BPB) had alkaline pH (9.96) with an EC of 2.95 dS m^{-1}. The alkaline pH of biochar may be effected by the release of alkali salts from feedstock during pyrolysis (Ahmad *et al.*, 2012). The CEC of BPB was higher and the reason for high CEC in biochar might be due to increase in surface area after pyrolysis and also increase in charge density on the surface (Gomez-Eyles *et al.*, 2013, Natalia *et al.*, 2017). The zeta potential measurement indicated that BPB are negatively charged which indicates stability of the particles in banana peel (Kokila *et al.*, 2015).

The Banana Peel Biochar showed a carbon content of 20.32 % and this may be due to evaporation of some amount of carbon as CO_2 or CO during pyrolysis.

Adsorption efficiency at varied pH and concentration

The adsorption behavior of biochar for different contaminants (i.e., heavy metals, organic pollutants and other pollutants) are different and well correlated with the properties of contaminants. In addition, the adsorption mechanism may also depend on various properties like surface functional groups, specific surface area, porous structure and mineral components.

As for as heavy metals are concerned the possible adsorption mechanisms usually involves integrative effects of several kinds of interactions including electrostatic attraction, ion-exchange, physical adsorption, surface complexation and/or precipitation. The appropriate sorption of banana peel biochar makes a great contribution to the adsorption of heavy metals. Therefore, the adsorption efficiency of BPB at different pH, varied Cd concentrations and different equilibration time were studied as batch experiment.

In the present experiment the adsorption of Cd by BPB was studied at different metal concentrations (25, 50, 75, 100 ppm along with control) and varied pH. The cadmium removal percentage was highest at 75 ppm (77.85 %) and the lowest was detected at 25 ppm (57.19 %).

The removal of Cd was increased along with increase of pH from 4 to 7. The maximum Cd removal (80.56%) was observed at pH 7, followed by 75.53% at pH 8 and the lowest adsorption was recorded at pH 4 (55.76%).

Interaction effect of different concentrations and pH was found to be significant on removal percentage of Cd by BPB. The pH is one of the main variable affect the adsorption process, influencing not only the speciation of the metal ions, but also the surface charge of the metal ions, surface charge of the sorbent and the degree of ionization of the adsorbent during the reaction (Kilic *et al.*, 2013).

In case of BPB, there was an increase in the adsorption of Cd (Fig. 1) at pH 7.0 and minimum adsorption was at pH 4.0. Similar observations of higher adsorption at higher pH > 6 were reported by Kilic *et al.*, (2013) and Ucar *et al.*, (2014).

Liu and Zhang (2010) that reported at lower pH, the surface functional groups (mainly oxygen containing groups) bind to the H^+, making these inaccessible for Pb ions. With increasing pH, the deprotonating functional groups provided the chance to co-ordinate with Cd and Pb ions resulting in higher removal percent.

The removal efficiency of Cd by BPB was also increased with the increasing in the concentration from 25 to 75 ppm, and then decreased with the increasing of concentration.

The maximum adsorption of Cd (91.51%) was observed in 75 ppm and the minimum was at 25 ppm solution. The capacity of banana peel increases with increasing concentration of metal ions. But if the substrate dose remains constant the removal efficiency may be reduced even if the metal concentration increases (Zhou *et al.*, 2007).

At higher dose, overcrowding of adsorbent particle leads to overlapping and aggregation at adsorption sites reduces the adsorption capacity. BPB showed more efficiency, which may be due to creation of large internal surface area in a limited volume leads to small sized pores which might be the reason for more adsorption by biochar. The existence a macrospores also might have facilitated the adsorption.

Effects of metal ions concentration and contact time

The adsorption of Cd(II) by BPB was analyzed based on contact time. It ranged from 0-8 hours under shaking at 250 rpm of Cd solution. The control (0 ppm or without metal) solution was not detectable at different time interval. Removal percentage of Cd by BPB increased with the increase in contact time from 0-6 h, after then decreased with increase of time. The highest removal percentage of Cd (75.20%) was recorded at 6h. The lowest adsorption (26.31%) of Cd was noted at 0h equilibration period. Interaction effect of time and Cd ions concentration was also found to be significant. The highest removal of Cd (86.69%) was observed at 6h and 75 ppm.

Present study is well correlated with the observation of Anwar *et al.*, (2010) who determined that adsorption of Cd by banana peel increases with increase in contact time and agitation speed (Priyanka, 2017). The adsorption of metal ions gradually decreases as time progressed. This might be due to ions have to pass through the deeper surface of the pores, which encounter substantial resistance leading to decreased adsorption during the later phase of the study (Srivastava *et al.*, 2016). And also the transportation rate of ions from the exterior to the interior sites of the adsorbent particles actually determines the adsorption rate of later phase (Shafaghat *et al.*, 2012).

Fitting the model

The results obtained with the experimental design that was focused at identifying the best levels of the selected variables, contact time (0-8), pH (4-8) and initial metal ion concentration (25-100 ppm). The second – order polynomial equation was used to find out the relationship between variables and response (Table 1 and 2).

Table.1 Electrochemical and biochemical characteristics of BPB

S.No.	Parameter	BPB*
1	pH	9.96
2	EC (dSm^{-1})	2.95
3	CEC (c.mol (P^{+}) kg^{-1})	10.4
4	Particle size (nm)	11315.6
5	Zeta potential (mV)	-20.0
6	Organic Carbon (%)	20.32
7	Crude protein (%)	15.31
8	Crude Fiber (%)	-
9	Protein (%)	-
10	Carbohydrate (%)	-
11	Cellulose (%)	-
12	Hemicellulose (%)	-
13	Pectin (%)	-
14	Moisture content (%)	7.5
15	Dry Matter (%)	92.5
16	Ash content (%)	17

Table.2 Mineral composition of BPB

S. No.	Element	BPB (mg g^{-1})*
1	Total Nitrogen	15.2
2	Phosphorus	5.51
3	Potassium	36.42
4	Calcium	12.43
5	Magnesium	10.87
6	Sodium	25.65
7	Iron	0.18
8	Manganese	42.14
9	Zinc	0.13

*Value represents means of three replications.

Table.3 ANOVA for quadratic model of banana peel biochar

Source	Sum of Squares	df	p-value (< 0.0001-significant)
Model	5680.69	5	< 0.0001
A-Concentration	1095.54	1	< 0.0001
B-Time	3229.91	1	< 0.0001
AB	0.6191	1	0.7457
A^2	210.33	1	0.0001
B^2	1276.37	1	< 0.0001
Residual	55.70	10	
Lack of Fit	55.70	5	
Pure Error	0.0000	5	
Cor Total	5736.40	15	

Table.4 Fit statistics

Banana peel biochar			Banana peel biochar	
Std. Dev.	2.36		R^2	0.9903
Mean	59.86		Adjusted R^2	0.9854
C.V. %	3.94		Predicted R^2	0.9660
			Adeq Precision	45.5423

Table.5 Significance of regression coefficients for the removal of Cd

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	F-value	p-value
Intercept	75.28	1	1.14	72.74	85.31	< 0.0001
A-Concentration	12.94	1	0.9225	10.88	111.05	< 0.0001
B-Time	20.43	1	0.8483	18.54	170.66	< 0.0001
AB	0.4587	1	1.38	-2.61	7.44	0.0213
A^2	-8.59	1	1.40	-11.71	29.73	0.0003
B^2	-19.98	1	1.32	-22.92	40.59	< 0.0001

Table.6 Chemical compositions of BPB before and after adsorption using EDX analyzer

Element	Before adsorption (Wt %)	After Cd adsorption (Wt %)
C	68.78	70.71
N	03.66	7.23
O	24.61	15.84
Zn	-	-
Na	0.36	0.00
Mg	0.19	0.54
Al	0.23	0.22
Si	0.21	0.62
P	0.00	0.34
S	0.00	0.00
Pb	0.00	0.33
Cl	0.14	0.23
Cd	01.12	2.21
K	00.70	1.29

Table.7 FTIR analysis of BPB before adsorption

Frequency (cm^{-1})	Bond	Functional Group	Characteristic Adsorptions (cm^{-1})	Type of Vibration	Intensity
2918.73	C-H	Alkane	2850-3000	stretch	strong
997.982	=C-H	Alkene	675-1000	bending	strong
771.387	C-Cl	Alkyl Halide	600-800	stretch	strong

Table.8 FTIR analysis of BPB after Cd adsorption

Frequency (cm^{-1}) for after Cd adsorption	Bond	Functional Group	Characteristic Adsorptions (cm^{-1})	Type of Vibration	Intensity
2854.13	C-H	Alkane	2850-3000	Stretch	Strong
1015.34	C-O	Ether	1000-1300	Stretch	Strong
770.423	C-Cl	Alkyl Halide	600-800	Stretch	Strong

Fig.1 Removal % of Cd by BPB at different pH

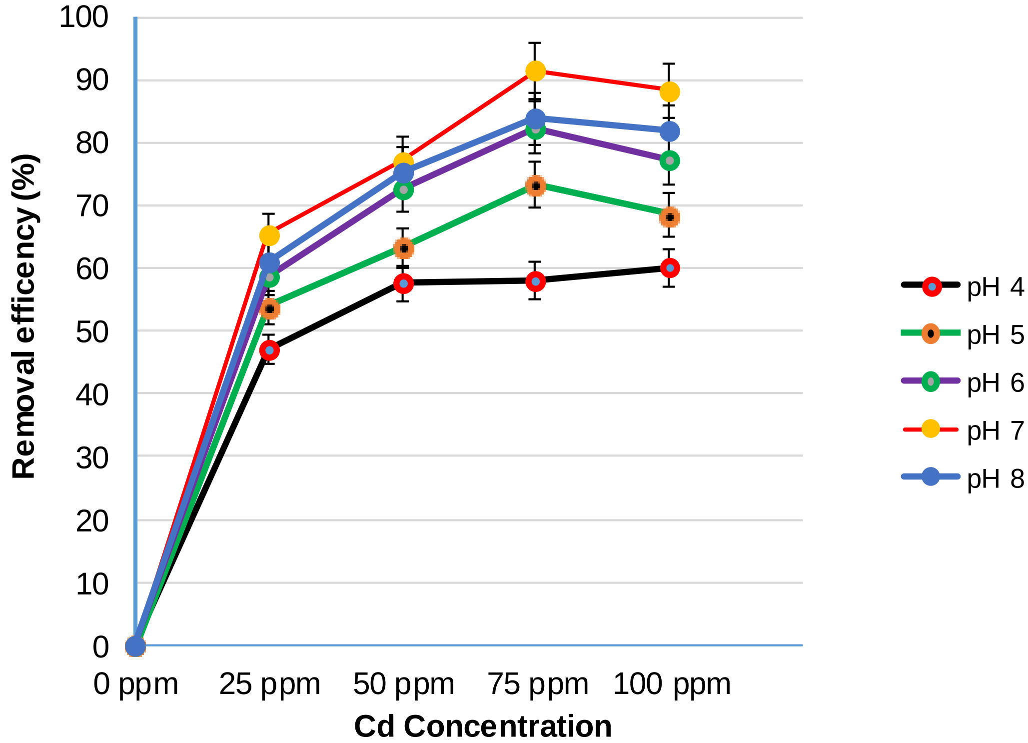


Fig.2 Removal % of Cd by BPB at different contact time

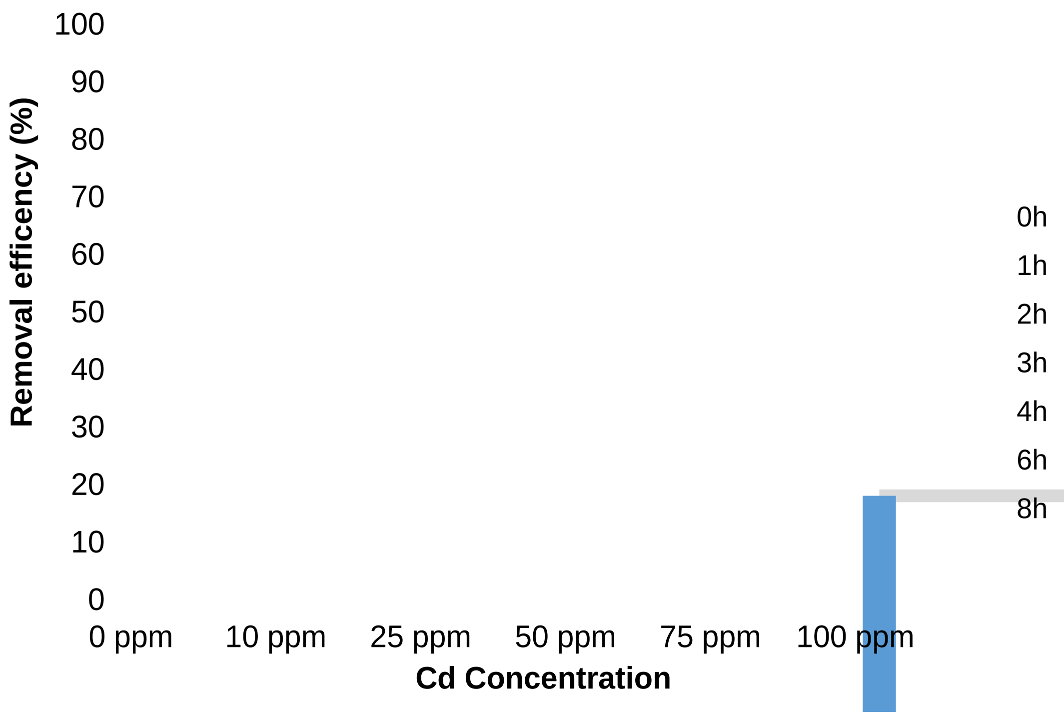


Fig.3 Effect of initial metal concentration and pH on the % adsorption of Cd (BPB)

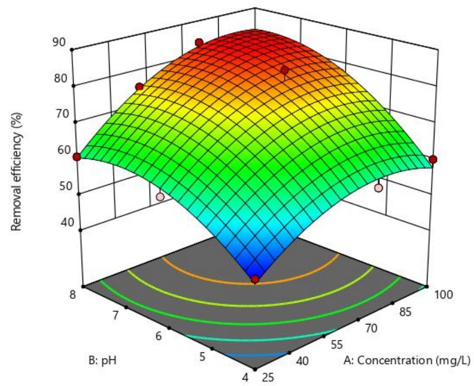


Fig.4 Overlay plot of perturbation of all the variables for BPB

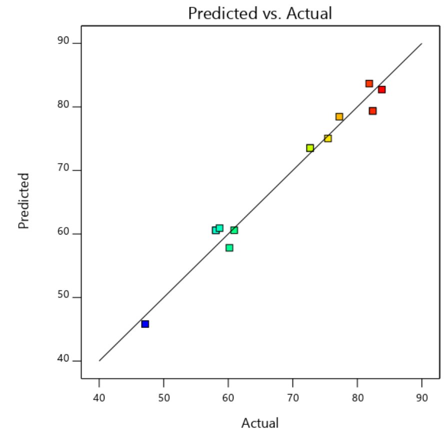


Fig.5 Effect of initial metal concentration and contact time on the on the % adsorption of Cd(BPB)

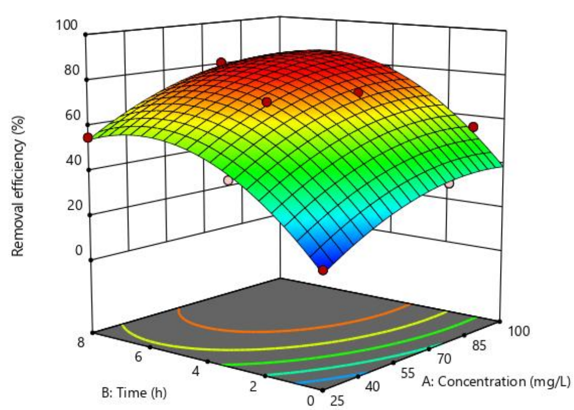


Fig.6 Overlay plot of perturbation of all the variables for BPB

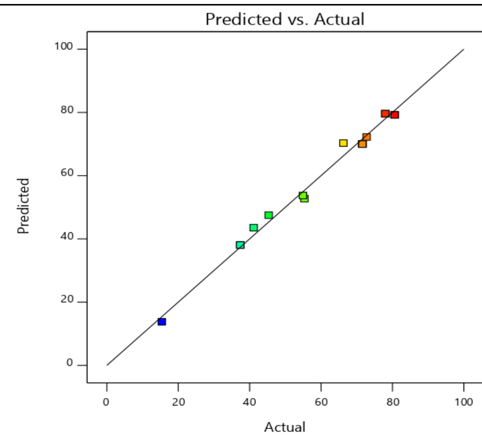


Fig.7 EDX micrograph before Cd adsorption

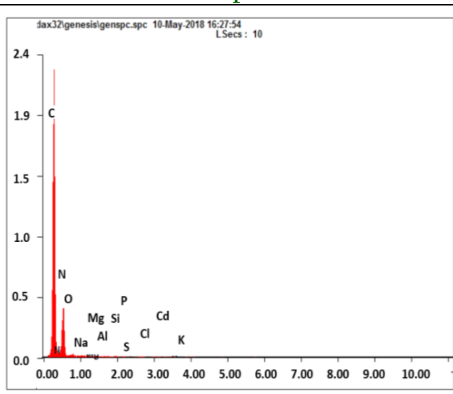


Fig.8 EDX micrograph after Cd adsorption

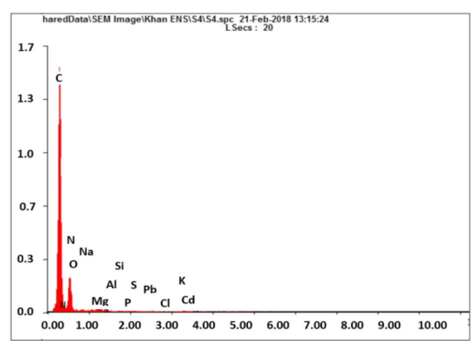


Fig.9a FTIR analysis of BPB before Cadmium adsorption

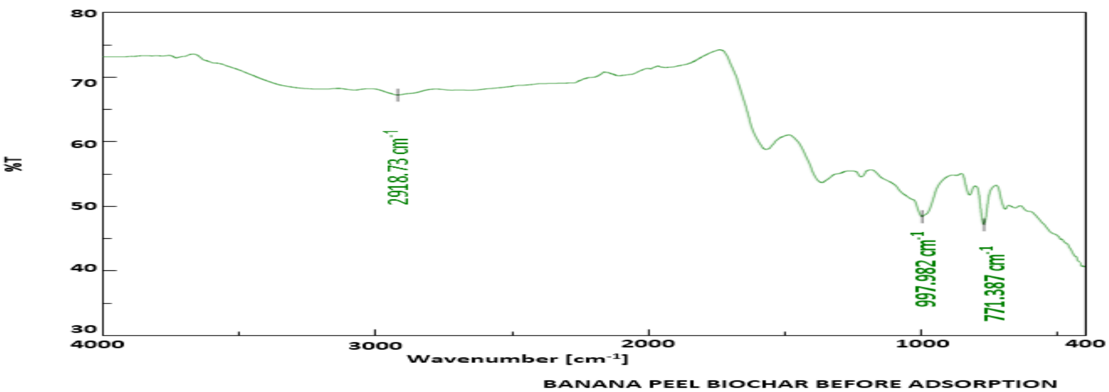
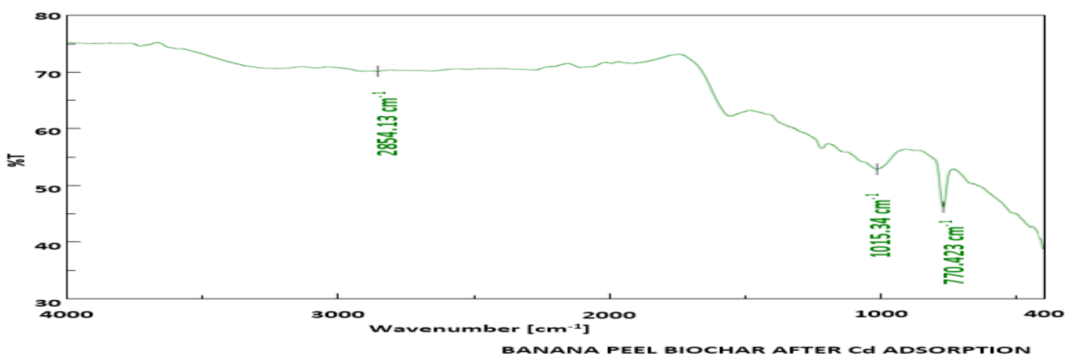


Fig.9b FTIR analysis of BPB after Cadmium adsorption



The regression equation coefficient was calculated and data was fitted to a second – order polynomial equation. The flat surface on the three-dimensional response indicates an optimum condition for the biosorption (Fig. 3-6).

The analysis of variance (ANOVA) for biosorption study of Cd(II) ions with banana peel adsorbent was used in order to ensure a quadratic model. The test for significance of regression model and the results of ANOVA test for significance of regression model and the results of ANOVA are in Table 3. prob >F less than 0.05indicated model terms are significant (Table 3-5).

The predicted and adjusted R^2 values of BPB was0.9660, 0.9854, which are closer to 1, indicates the better fitness of model in the experimental data (Fig. 3-4). The regression equation after the analysis of variances (ANOVA) gave the level of cadmium ion removal as a function of the initial cadmium ion concentration, initial solution pH and contact time. The results of the multiple regression analysis of the data were fitted with a second-order full polynomial equation (Amini, *et al.*, 2008). The empirical relationship between cadmium ion removal (*Y*) and the independent variables in coded units obtained by the application of RSM is given by

Cd Removal percentage (BPB) $=75.28+12.94\times A+20.43\times B+0.4587\times A\times B-8.59\times A^2-19.98\times B^2$ (4).

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings.

Characterization of banana peel biochar before and after adsorption of cadmium

EDX analysis of BPB before and after adsorption

The elemental composition of BPB before adsorption of Cd was analyzed through EDX analyzer and the results are given in Table 6 and figure 7. Cadmium to the level of 1.12 on per cent weight basis was observed in BPB. The percentage weight of chemical composition present on the surface of BPB after adsorption of Cd is given in Table 6 and figure 8. It showed increases in the weight percentage Cd adsorption. The Cd content also increased and it was 2.21% in BPB. This strongly proves that the adsorption of Cd ions in the selected substrates. There are variations in other elements also and this may be due to the interference in the adsorbed Cd ions. To our surprise before adsorption of Cd, the Na was 0.36 % in BPB, but after adsorption there was no sodium at all in BPB. This may be due to Na, the most suitable exchange ion for the removal of Cd from solution (Maria and Rodney, 1987).

Functional characteristics of biosorbents before and after Cd adsorption

The BPB was subjected to FTIR analyses and the results indicated that, the FTIR spectrum peak was observed within 4000-400cm^{-1} (Table 7). Alkane, Alkene and Alkyl Halide are the functional groups in BPB before adsorption. The Alkane ring structure (C-H stretch) has been recorded at the band, 2918.73 cm^{-1} in BPB. The band corresponding to Alkene (=C-H) was detected at 997.982 cm^{-1} and the band related to Alkyl Halide (C-Cl stretch) was detected at 771.387 cm^{-1} in BPB (Fig. 9a).

The FTIR spectrum of BPB after Cd adsorption revealed a large number of peaks within the interval of 4000-400 cm^{-1}, with the complex chemical nature of the bio-material (Table 8 and Fig. 9b). The Alkane ring structure (C-H stretch) has been recorded at the band, 2854.13 cm^{-1}. The band corresponding to Ether (C-O) was detected at 1015.34 cm^{-1} and the band related to Alkyl Halide (C-Cl stretch) was detected at 770.423 cm^{-1} in BPB after Cd adsorption.

Alkane functional group with (C-H) band was detected at 2918.73 cm^{-1} frequency before adsorption but after adsorption of Cd, it was decreased to 2854.13 cm^{-1} and similar C-H stretching vibration functional group also observed by Hai *et al.,* (2016) before and after adsorption of Cd.

Alkene functional group with (=C-H) band was reported at 997.982 cm^{-1} adsorption frequency before adsorption but after adsorption of Cd, the alkene functional group was changed to ether with (C=O) band at 1015.34 cm^{-1}. There were no changes in Alkyl Halide functional group before adsorption and after adsorption of Cd. In Cd adsorption, C-H group was reduced but =C-H increased without change in C-Cl.

The presence of negative charge on the surface of the biochar may be the reason for higher adsorptive capacity of the substrates. Not only the charges, but the functional groups detected in the biochar also increased the potential for adsorbing the metal cations.

In conclusion, detailed study with banana peel biochar indicates that the adsorption of cadmium from aqueous solution spiked with different concentration was higher in biochar. The pH 7, solute concentration 75 mg L^{-1} with a contact time of 6 h was found to be optimum for the maximum removal of Cd. Hence ecofriendly wastes like banana peel can be used for heavy metal removal from the wastewater.

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