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Active carbon prepared from vegetable wastes for the treatment of Pb(II) in aqueous medium

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Abstract

Activated carbon was prepared from vegetable wastes collected from the residential halls of University of Dhaka, Bangladesh. The carbon, produced by pyrolysis of vegetable wastes at 400^oC to 600^oC for 30 min under N_2 atmosphere, was chemically activated with zinc chloride. The activated carbon samples were characterized by X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FT-IR). Topographical and morphological characteristics were examined by Scanning Electron Microscopy (SEM). The surface area of the activated charcoal was determined by adsorption of Methylene Blue from alcoholic solution and was found to be 53 m^2g^{-1}. The zero point charge (pH_{zpc}) was also determined by pH titration means and obtained to be 6.10±0.1 at ambient temperature. Analyses of these experimental data lead us to conclude that the activated carbon prepared from vegetable wastes materials is a high grade active carbon in comparison with commercially available one. Activity of generated active carbon as an adsorbent was tested through adsorption of Pb(II). The results indicate that adsorption of Pb(II) on active charcoal was very fast and it followed Langmuir type of adsorption isotherm, and the dimensionless separation parameter, R_L, was found to be $0<R_L<1$ at 30^oC. It indicated that activated charcoal obtained from vegetable wastes is an effective adsorbent for removal of toxic heavy metal from industrial waste water.

Keywords: Active carbon; Zero point charge; Heavy metal; Adsorbent; Adsorption

Introduction

Adsorption plays an important role in the physico-chemical treatment of municipal wastewaters. Advantages of adsorption by utilizing various adsorbent over biological treatment systems include, among others, lower land area requirements, lower sensitivity to diurnal flow and concentration variations and to toxic substances, potential for significant heavy metal removal, greater flexibility in design and operation; and superior removal of organic wastes (Walter *et. al.*). Active charcoal has been attracted as a promising adsorbent for removal of heavy metals (Babel *et. al.*, 2003), organic pollutants (Das *et. al.,* 2004 and Naghizadeh, *et. al.*, 2011). Physico-chemical systems using adsorption with activated carbon consistently produce high levels of treatment and have a high degree of stability and reliability. The adsorption is enhanced by *in-situ* partial regeneration affected by biological growth on the surfaces of carbon. However, active charcoal has been prepared by various ways due to its wide applications (Zhang *et. al.,* 2007). Cost effective and easily reachable adsorbent for removing heavy metals is a crucial demand for practical applications. Besides, waste management is a burning issue in Bangladesh, especially, in municipal area. It is, thus, of interesting to find a route utilizing these waster materials in efficient way as commercially viable materials like active carbon.

Presence of heavy metals in the industrial water has been of great concern to researchers and engineers because of harmful effects that might not have been anticipated (Fleeger *et. al.*, 2003, Weis *et. al.*, 2004, Jarup *et. al.*, 2003 and Samarghandi *et. al.*, 2011). Hexavalent chromium (Cr^{6+}) (Uddin, *et. al.*, 2007 and Sen *et. al.*, 2010) and divalent lead (Pb^{2+}) are highly priority toxic species that are classified as carcinogenic, mutagens and teratogens in biological system (Pamila *et. al.*, 1991, Alvarez-Ayuso *et. al.*, 2007, Baral *et. al.*, 2007 and Srivastava *et. al.,* 2007). Indiscriminate disposal of effluents containing Cr^{6+} and Pb^{2+} without adequate treatment poses a severe threat to public health and environment. Effluent from leather industries as well as from textile industries contains Cr^{6+} bearing species which is a big problem in Bangladesh. A number of methods have been developed for removal of heavy metals from industrial wastes. These include, among others, precipitation, electroplating,

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evaporation, ion exchange, membrane separation, coagulation, floatation, reverse osmosis, membrane filtration, solvent extraction and various biological processes (Wang *et. al.*, 2009, Hota *et. al.*, 2008, Afkhami *et. al.*, 2002 and Afkhami *et. al.*, 2008). These methods are expensive and have many disadvantages such as incomplete removal, requirements of large amount of reagents and generation of toxic sludge or other waste products that require further disposal or treatments (Nameni *et. al.*, 2008). It is therefore, quite urgent to develop simple, innovative, more efficient as well as economically viable materials for removal of harmful Cr (VI) and Pb (II) from industrial effluents. In our previous work (Deb *et. al.*, 2010) we have found that 93% of chromium can be removed from aqueous solution using adsorbent generated from vegetable wastes.

In this study, activated charcoal generated from biomass has been used to remove Pb(II) from industrial waste water. Activated charcoal from vegetable wastes has been characterized and its activity toward removal of Pb(II) from industrial effluents is reported herein.

Materials and methods

Materials

Hydrochloric acid (Merck, Germany), methylene blue (Janssen Chimica, Belgium), silver nitrate, (Universal Chemicals, India), zinc chloride (Scharlav C10160), sodium hydroxide (Merck Pellets Purifies, India) and lead chloride (E. Merck, India) were used without further purification. De-ionized distilled water and analytical grade acetone (BDH, England) were used frequently to dissolve the necessary reagents.

Preparation of raw materials for carbonization

Vegetable wastes mainly composed of cabbage, green banana peels, tomato, onion peels etc. were collected from different canteens of Dhaka University, Bangladesh. The raw biomass materials were cut into small pieces and washed several times with water for removal of adhered extraneous matter. After washing the pieces were dried in open air for few days. Then the dried raw materials were heated in an oven at 120°C for complete removal of moisture. Finally, the dried pieces were powdered with the help of a grinding machine.

Preparation of charcoal

The powdered raw materials obtained from vegetable wastes were taken in a stainless steel tube measuring 0.45 inch in inner diameter and two feet in effective length. This is the main part of the reactor employed for carbonization. This reactor was situated horizontally in an electrically heated tube furnace. The carbonizations were carried out with 25 to 30 g of the feed species in the temperature range of 400 to 600°C for 30 min. The atmosphere inside the furnace was made inert by passing a stream of nitrogen gas slowly into the reactor tube.

Activation of charcoal

The finely powdered charcoal was washed with dilute HCl followed by de-ionized distilled water for complete removal of chloride ion. The presence of chloride ion was frequently tested with silver nitrate solution and confirmed that chloride ion, if present, was beyond the test limit. Then it was dried in the oven at 120°C for about 3 h. The activation process was carried out by mixing of zinc chloride with the prepared charcoal. Three different type of activated charcoal were prepared in which the ratios of charcoal to zinc chloride were 5:1, 10:1, and 15:1 (w/w). A systematic investigation of the effect of impregnation ratio on the properties of active charcoal was carried out by FT-IR. It was found that the activated charcoal with 10:1 (charcoal: zinc chloride) is better active charcoal (Sutradhar *et. al.*, 2008). Finally, this ratio was maintained for further activation of charcoal for characterization and applications. The mixtures were made to paste and heated into a muffle furnace in the temperature range of 400-500°C for 20 min. It was then washed, dried and grounded to powder.

Characterization of the activated charcoal

The activated charcoals were characterized for better understanding the surface property and morphology. The X-ray Diffraction (XRD) (X'Pert PRO XRD PW 3040, PHILIPS, Netherlands) was used to analyze the phase composition and the FT-IR (Shimadzu FT-IR IR-Prestige-21) was used for molecular characterization of the activated charcoal. Scanning Electron Microscopy (SEM) (Hitachi 3400N) was used for morphological analysis. The surface area of the activated charcoal was measured by using methylene blue as an adsorbate. The zero point charge (pH$_{zpc}$) was also determined by pH-titration method (Bolt, 1957; Park and Bruyn, 1962).

Determination of the Equilibrium time for the adsorption of Pb²⁺ in aqueous solution

The adsorption equilibrium was determined by adding 25 mL of 10 ppm Pb^{2+} solution in each of four reagent bottles containing 0.05 g of the adsorbent at a definite pH. The reagent bottles were shaken in a mechanical thermostated shaker at 30±2°C. After definite interval of time each of the bottles was withdrawn from the shaker. The supernatant was removed from the sorbent as soon as possible and centrifuged repeatedly until a clear solution was obtained. Then the concentration of the solution was measured by Atomic Absorption Spectroscopy (Analyst 800, Perkin Elmer). The same procedure was repeated for the 5, 20, and 30 ppm of Pb^{2+} solutions.

Determination of the adsorption isotherm of Pb²⁺ on activated charcoal

Batch adsorption studies were conducted to determine adsorption isotherm of Pb^{2+} on activated charcoal. 0.05 g of the activated charcoal was taken in a series of reagent bottles. Freshly prepared 25 mL of Pb^{2+} solutions of varying initial concentrations ranging from 5 ppm to 50 ppm prepared by diluting of 100 ppm Pb^{2+} solution with de-ionized distilled water was added to each bottle. As kinetics studies have shown that, 15-20 min time was sufficient to attain equilibrium adsorption; the bottles were shaken by a thermostated mechanical shaker for 20 min. Thereafter, all the bottles were removed from the shaker and the supernatants were removed from the sorbent as soon as possible and centrifuge repeatedly until a clear solution, in each case, was obtained. The concentration of Pb^{2+} solutions were measured by the Atomic Absorption Spectrophotometer. The amounts of Pb^{2+} adsorbed per gram of the adsorbent were calculated from the difference between concentration of Pb^{2+} solution before and after adsorption.

Results and discussion

FT-IR analyses

The FT-IR spectroscopic data of non-activated charcoals and activated charcoal are shown in the Figs. 1(a) and 1(b) respectively. It is found that the activated charcoal with ratio 10:1 to $ZnCl_2$ has few major absorption bands whereas FT-IR spectrum for non-activated carbon exhibits multiple bands indicating clear activation of charcoal. The spectrum shows three major absorption bands at 472, 565, and 605 cm⁻¹ (Fig. 1(b)). The band at 472 cm⁻¹ is due to the absorp-

tion of zinc molecules as result of a Zn-O stretching. Peaks at 565 and 605 cm⁻¹ are assigned to the out-of-plane C-H bending mode (Socrates, 1994). These spectra were also suggested to be due to alkaline groups. It is also noted that, the absorption band in the range 540-760 cm⁻¹ is found for the chloro-alkanes groups (Pavia *et. al*., 2007).
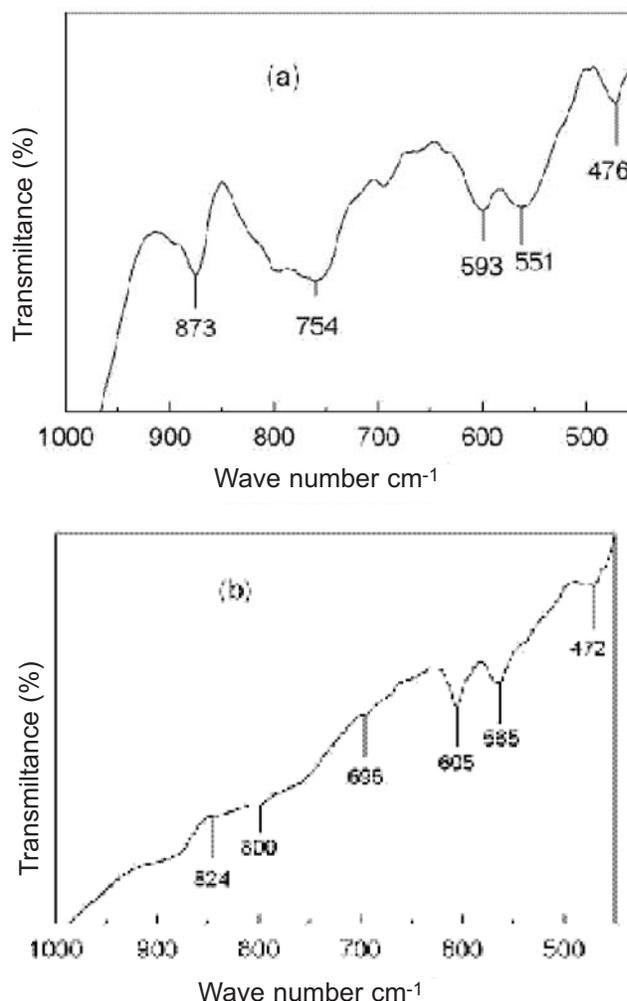

Fig. 1. FT-IR spectra of (a) non activated charcoal and (b) activated charcoal

XRD analysis

X-ray diffractograms of activated charcoals and commercial charcoal (Uni-Chem/chemical reagent, China) are shown in the Fig. 2. The XRD spectra of the activated carbon are similar with that of commercial charcoal under similar experimental conditions.
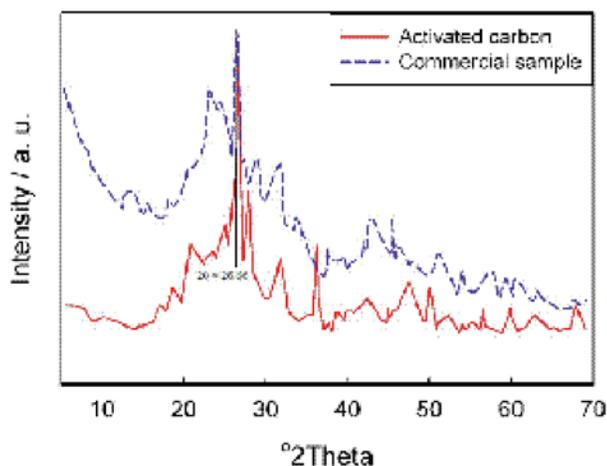
Fig. 2. XRD spectra of prepared activated charcoal and commercial charcoal

The graphite form was observed at $2\theta = 26.66$ (Mopoung *et. al.,* 2008; Qodah *et. al.*, 2009) with relative intensity of 100%. The results reveal that high grade active charcoal can be obtainable from vegetable wastes collected from the kitchen. This research thus opens new window for waste disposal in Bangladesh both in terms of economic feasibility as well as from scientific point of view.

SEM image of activated charcoal

SEM photograph (Fig. 3) shows that wide variety of pores is present in the activated carbon along with fibrous structure.
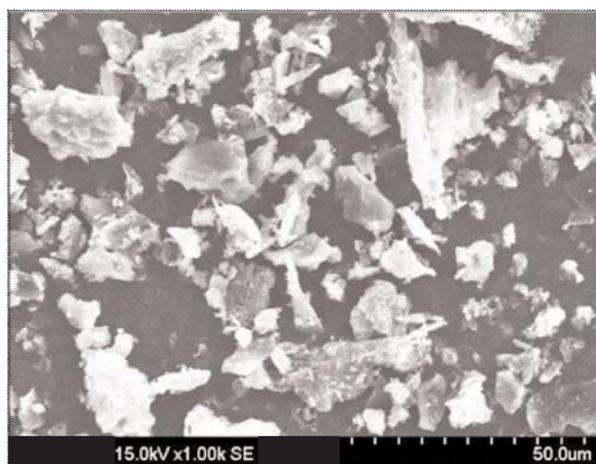

Fig. 3. SEM image of activated charcoal

$ZnCl_2$ impregnated carbon consists of more canals like structure. In cases of $ZnCl_2$ impregnated activated carbon, surfaces are pitted and fragmented. Although, it is not possible to predict the exact size of the individual particle from the

SEM image, which can be done through TEM analysis but from the following image it can be observed that the particle size of the activated charcoal is in the range of 5-10 μm.

Determination of equilibrium time for the adsorption of methylene blue on activated charcoal

The effect of contact time on the adsorption of methylene blue (MB) on the carbon sample is shown in Fig. 4. The experiments were carried out at 30ºC with a fixed adsorbent dose (0.05g/25 mL. The initial concentration of MB was (10 mgL⁻¹) and the agitation speed (110 rpm) was kept constant. Initially the rate of adsorption increased rapidly initially, and the optimal removal efficiency was reached within 15 min then almost remained at level off position until the experimental time was over.
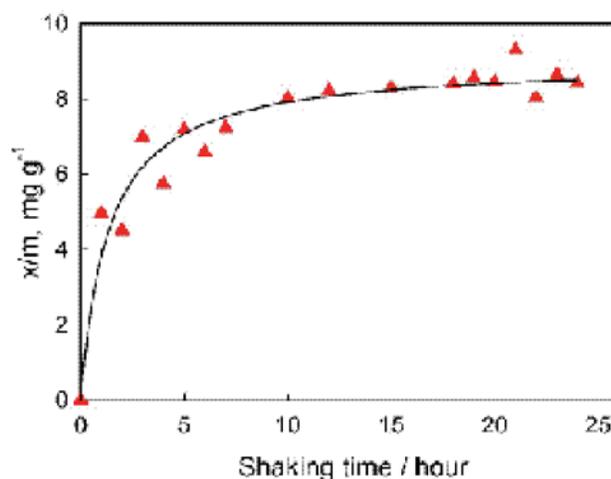

Fig. 4. Effects of shaking time on adsorption of methylene blue on activated biomass adsorbent

Therefore, it was concluded that the MB adsorption equilibrium reached at 15 min under this condition.

Adsorption isotherm of MB on activated charcoal

The adsorption studies conducted at fixed adsorbent dose and by varying the initial concentration of MB and the equilibrium data were fitted to the Langmuir isotherm in Fig. 5.

The experimental data were analysed by Langmuir equation:

$$\frac{C_e}{Q} = \frac{1}{K_a Q_m} + \frac{C_e}{Q_m} \tag{1}$$

where $x/m = Q$ is the amount adsorbed g⁻¹ of adsorbent at an equilibrium concentration of C_e and Q_m is corresponding
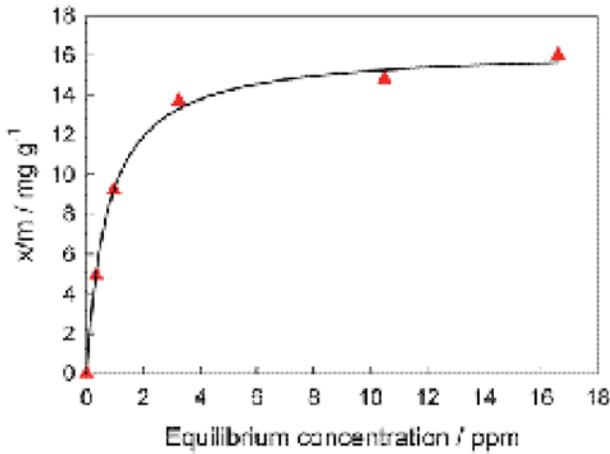
Fig. 5. Langmuir isotherm for adsorption of methylene blue on activated charcoal

amount for the formation of a monolayer. A plot of C_e/Q against C_e is expected to give a straight line.
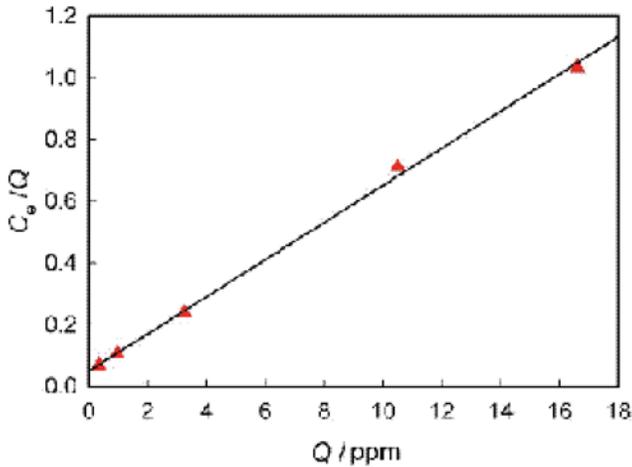

Fig. 6. Langmuir plot for the adsorption of methylene blue on activated charcoal

The plot in Fig. 6 shows a linear relationship between C_e/Q and C_e indicating that the adsorption isotherm is of the Langmuir type.

Measurement of surface area

The Langmuir equation was used to calculate the specific surface area of activated charcoal. The general form of Langmuir isotherm is:

$$Y = \frac{KC}{(1+KC)} \tag{2}$$

Where Y is the fraction of activated carbon surface covered by adsorbed methylene blue molecules, K is a constant, and C is the equilibrium MB solution concentration. In this case, $Y = N/N_m$, where N represents the number of moles of MB adsorbed per gram of carbon at equilibrium concentration, C and N_m is the number of moles of MB g^{-1} of carbon required to form a monolayer. Equation (2) can be rearranged to obtain

$$\frac{C}{N} = \frac{C}{N_m} + \frac{1}{KN_m} \tag{3}$$

For all adsorption isotherms of MB on activated charcoal of biomass, the plot of C_e/Q vs. C_e gives a straight line with slope equal to $1/N_m$, and intercept equal to $1/KN_m$. Therefore, the Langmuir isotherm is an adequate description of the adsorption of the MB on biomass charcoal. The specific surface area was calculated from the following equation (Chongrak *et. al.*, 1998 and Itodo *et. al.*, 2010).

$$S_{mb} = \frac{N_g \times a_{mb} \times N \times 10^{-20}}{M} \tag{4}$$

Where, S_{mb} is the specific surface area in 10^{-3} km^2 kg^{-1}, N_g is the number of molecules of methylene blue adsorbed at the monolayer of fibers in kg kg^{-1} (or $N_g = N_m*M$), a$_{mb}$ is the occupied surface area of one molecule of methylene blue, 197.2 Å^2 (Graham *et al.*, 1955). N is Avogadro's number, 6.023 x 10^{23} and M is the molecular weight of methylene blue, 373.9 g mol^{-1}. The calculated value for the surface area was found to be 52.86 m^2 g^{-1}.

Determination of zero point charge

The zero point charge (pH$_{zpc}$) of activated carbon was determined by the pH titration method as describe by previous report (Chongrak *et. al.*, 1998 and Itodo *et. al.*, 2010). The experiment was carried out by measuring pH of the suspension and filtrate with 0.01 M NaCl and 0.001 M NaCl solution at room temperature. Two bottles of suspension and supernatants were titrated with 0.05 M HCl and 0.05 M NaOH using micro burette. Finally, the mean volumes of acid and alkali with the corresponding pH were estimated from the pair of titration curves correspond to suspensions and supernatants. The net titrations curves including solutions two different ionic strength were shown in Fig. 7. From the Fig. 7, the zero point charge for the activated charcoal was found to be 6.10±0.1 at ambient temperature. pH$_{zpc}$ for the activated charcoal was found to be 6.23±0.1 and 6.16±0.1 respectively for 0.01 M NaCl and 0.001 M NaCl under similar experimental conditions.
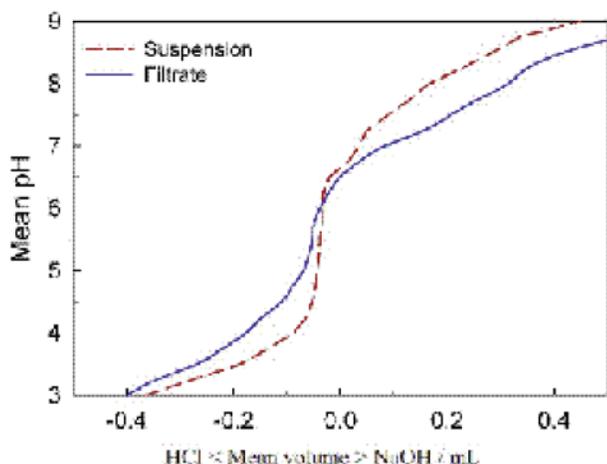
Fig. 7. Net pH$_{ZPC}$ of active charcoal by pH titrametric method with different concentrated NaCl solution

The value of pH$_{zpc}$ was determined by pH-metric (Periasam *et. al.*, 1991) and potentiometric titration (Parfitt *et. al.*, 1983 and Tewari, 1981). Both the methods provide the accurate values of pH$_{zpc}$. It has been reported that pH of carbon, 6-8 is acceptable for most of the applications (Ekpete *et. al.,* 2011). It was further established that generated active carbon exhibited characteristics of high grade commercially available carbon materials. This value of pH$_{zpc}$ suggests that the surface release H$^+$ ion into the bulk only when pH of the solution is above 6.10 and the surface becomes positive when the solution pH is below 6.10. It thus indicates that below the pH 6.10 the water donates more protons than hydroxide groups, and so the adsorbent surface is positively charged (attracting anions). Conversely, in the event of pH is higher than 6.10 the surface would be negatively charged (attracting cations /repelling anions). Higher is the pH of the solution than 6.10, higher is the electrostatic attraction between surface and adsorbate Pb^{2+} ions.

Effect of contact time on the adsorption of lead

The effect of contact time on the adsorption of Pb^{2+} ions with concentrations is shown in Fig. 8. In the case of methylene blue, Pb^{2+} ions also reached equilibrium in about 15 min at all concentration studied in the research. The experiments were carried out at 30°C with a fixed adsorbent dose (0.05g/25 mL) and the agitation speed (110 rpm) was kept constant.
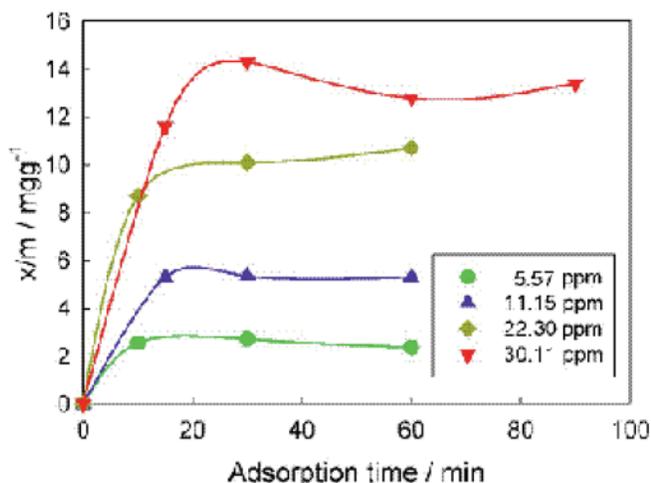

Fig. 8. Effects of shaking time on adsorption of Pb(II) on activated biomass
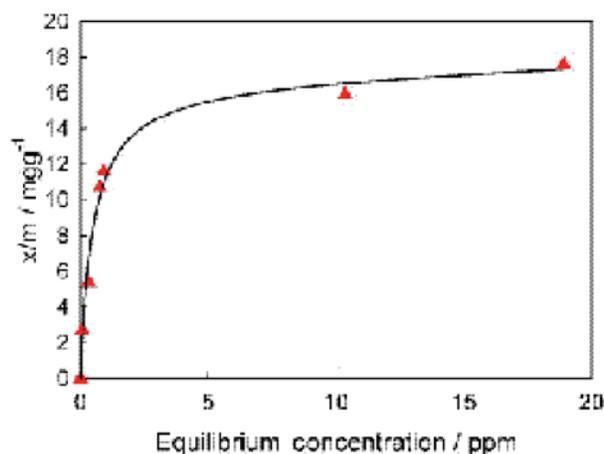

Fig. 9. Adsorption isotherm for the Pb^{2+} on activated charcoal

The adsorption isotherm of Pb^{2+} on activated charcoal (Fig. 9) indicates that with increasing initial concentration of lead solution, the amount of the adsorbate g^{-1} of adsorbent increases initially at a fast rate, however, finally reached nearly a constant amount of adsorption on further increase of concentration of Pb^{2+}. It is concluded that the type of adsorption may be of Langmuir type. However, nature of adsorption isotherm varies with the adsorbent and also interactions between adsorbent and adsorbate. Thus, it is necessary to confirm further whether it follows Langmuir type or any other models. To establish our preliminary assumptions, we made characteristic plot for various types of isotherm. Fig. 10 shows a linear plot of C_e /(x/m) against C_e.

The fitting of the experimental data to a straight line is excellent, indicating the validity of the Langmuir model of
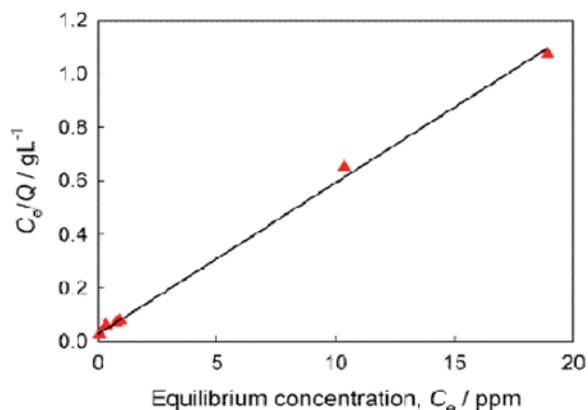
Fig. 10. Langmuir plot for the adsorption of Pb (II) on activated biomass (charcoal)

adsorption. The value of the Langmuir constants, Q_m and K_a were determined from the plot and found to be 17.314 mgg^{-1} and 2.108 Lmg^{-1} respectively. This amount of adsorption to be comparable to any other studies pertaining adsorption studies.

Table I. Data for the determination of equilibrium parameters from Langmuir

Initial concentration	K_a		
C_o $mg\ L^{-1}$	$L\ mg^{-1}$	$1+K_a\ C_o$	R_L
5.57		12.74	0.078
11.15		24.50	0.041
22.30		48.01	0.022
30.11	2.108	64.48	0.015
44.60		95.02	0.011
55.75		118.5	0.008

The essential characteristics of Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter, R_L which is defined by the relation:

$$R_L = 1/(1+K_a\ C_o) \qquad (5)$$

Where K_a is the Langmuir constant and C_o is the initial concentration of Pb (II). The equilibrium parameter, R_L can be easily calculated using the values C_o and K_a as found earlier. It is found that the values of equilibrium parameter, R_L is always lies between 0 to 1, which indicates the favorable adsorption of Pb^{2+} on activated charcoal of biomass in all concentration.

Conclusion

The results of this study showed that activated carbons with relatively high surface areas can be obtained from vegetable wastes by direct chemical activation with $ZnCl_2$. Especially, the XRD analysis confirmed that the diffractogram resembles to commercial charcoal. In SEM analysis, it is seen that, the particle size of the prepared activated charcoal is in the range of 5-10 micrometer. It was found that adsorption of Pb^{2+} on activated charcoal requires only 15 min to reach the equilibrium whereas it requires more than one hour to reach equilibrium for chromium adsorption. At ambient condition activated charcoal reduce 99% Pb^{2+} from the aqueous solution. Thus it can be concluded that, activated charcoal obtained from vegetable wastes is an ideal adsorbent for removal of Pb^{2+} from contaminated water. Further studies are underway to clearly understand the mechanism of adsorption on the activated carbon.

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