Cadmium removal from aqueous solution by jackfruit seed bio-adsorbent

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Abstract

In this present study, jackfruit bio-adsorbent was synthesized and utilized as cost effective for the removal of cadmium from aqueous solution. Jackfruit bio-adsorbent was characterized using emission scanning electron microscope, energy dispersive X-ray spectroscopy, thermal gravimetric analysis, Fourier transform infrared spectroscopy, X-ray diffractive spectroscopy and inductively coupled plasma optical emissions spectrometer techniques. Effects of different parameters such as solution pH, dosage of adsorbent, stirring speed and concentration of cadmium removal were evaluated and optimized. The maximum adsorption was achieved at speed of 120 rpm, pH 7.6, 60 mg adsorbent dose and contact time for 1 h, the optimum adsorption efficiency of about 97.12% was observed even at lower adsorbent dosing of 60 mg. Activation energy of the jackfruit bio-adsorbent was also being evaluated, which was observed 130.69 kJ/mol using FWO method which attributed to a chemisorptions adsorption process.

Keywords Bio-adsorbent · Thermal kinetics · Jackfruit seeds · Cadmium removal

1 Introduction

Wastewater containing heavy metals are mainly released from the industries like electroplating, paint, cement, mining, dyeing etc. Chemical industries effluents mostly contain heavy metals such as cadmium, iron, copper, chromium, arsenic, lead etc. and its compounds. These heavy metals may bring long-term threat to environment and human being, when released in water [23]. Cadmium (Cd) is considered to be one of the most hazardous contaminants present in waste water. These contaminants generally enter into the water bodies through various industrial and agricultural practices and can adversely reacts with the ecosystem even at very low quantities [6–9, 11, 21, 41]. Therefore before discharging the wastewater in the river and lake from these industries, effluent treatment is required. After treatment the maximum limit of cadmium is equals 0.003 mg/L. There are different methodologies

available for wastewater treatment like coagulation, electro-coagulation, flocculation, filtration, membrane separation; adsorption reverses osmosis, precipitation and bioremediation [16, 17, 18, 29, 37]. There are different methodologies available for wastewater treatment like coagulation, electro-coagulation, flocculation, filtration, membrane separation; adsorption reverses osmosis, precipitation and bioremediation [16, 29, 37]. Among them, adsorption is one of the most common and commercially adopted techniques because of its good efficiency with simple procedure and low operational cost [4, 12, 20]. In the adsorption technology, an adsorbent is used in the powder form in the wastewater on which heavy metals adsorbs on the surface and settle down and clean water is on the top of the container [6–9, 41]. In the recent years, scientific community has been used a number of materials as an adsorbent including stones, shells, and various fruit seeds, sewage slags and biomass [1, 6, 9, 38]. The presence

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of this waste can cause undesirable effects on the health of the human body and other organisms primarily through interactions with enzymes in their cells [3, 16, 29, 37]. Due to the high cost of commercial carbons, researchers are searching for the material which is cheap, affordable and easily accessible to produce activated carbon. In this respect, agricultural and industrial wastes [6–9, 41] can be those appropriate raw materials to produce appropriate activated carbon for the removal of heavy metals from the waste water [3, 24, 30].

The present study is focused on the synthesis, characterization, thermal degradation kinetic of biomass to used material like jackfruit bio-adsorbent and its application in cadmium removal from aqueous solution.

2 Materials and method

2.1 Experimental setup

The self assembled experimental setup to perform in batch adsorption process [33, 34]. The experimental setup consist a magnetic stirrer of capacity 0 to 500 rpm with least count 5 rpm and 100 ml beaker consists of cadmium contained water with different concentration range varying from 0.5 to 2.5 ppm has been subjected to purify in presence of bio-adsorbent. The experiments have been performed with different adsorbent dosing. The experiments have been performed for 50 min to optimize

adsorbent dosing, rpm of magnetic stirrer, pH of solution and cadmium concentration of the solution. The removal of cadmium from the polluted water was studied in 10 min interval up to 2 h.

2.2 Preparation of bio-adsorbent

Jackfruit seeds (JFS) was collected from the fruit market near the campus of the Birla Institute of Technology, Mesra, Ranchi, Jharkhand, India. The seed was washed with tap water followed by distilled water to remove dust particles and other impurities from the surface. After washing, seeds were dried in an air oven for 48 h at 60 °C for removing moisture. Figure 1 shows the dried seeds which is ready for crushing and milling to make it powder form. The powder has been kept in sheave shaker and all equal size particles have been distinguished. The higher size particles have been again crushed to find minimum particle size. After getting all equal size particles the powder sample has been send for different types of characterization and again calcined it at 400 °C for 3 h [16].

2.3 Adsorbent characterization

FESEM-EDX was recorded on Sigma-300 with EDX (Ametek) Field Emission Scanning Electron Microscope instrument. An accelerating voltage of 15 kV and a magnification of 1000X were used. Thermo gravimetric analysis (TGA) and Differential Thermal Analysis (DTA) performed on a Shimadzu (Japan;

Fig. 1 Flow chart of jackfruit seeds based bio-adsorbent

DTG-60) in Nitrogen environment, at a different heating rate of 10–50 °C/min. FTIR spectrum of the prepared adsorbent was recorded in the range of 400–4000 cm^{-1} using Shimadzu (Corporation, Japan; IR-Prestige 21). The elemental analysis by Inductively Coupled Plasma Optical Emissions spectrometer in Perkin Elmer, Optical 2100DV ICP-OES (USA) with spectral range of 160–900 nm, spectral resolution of 0.009 nm at 200 nm and speed of 20–25 elements in less than 5 min.

2.4 Experimental design

All the experiments have been designed with the help of design-expert 6.0.8. Response surface methodology has been used to optimize the process parameters for maximizing the percentage pollutant removal from aqueous solution. Within this methodology, a four factor and five level central composite design (CCD) has been implemented to study the combined effect of all the four parameters [pollutant concentration (A), mixing speed (B), pH (C) and adsorbent dose (D)] on the percentage removal of pollutant from aqueous solution [35]. The range and levels of all the four selected parameters studied in the experiments have been presented in Table 1.

2.5 Thermal kinetic

The common rate equation governs in all kinetic studies can be generally written as follows:

$$\frac{d\alpha}{dt} = k(T)f(\alpha) \quad (1)$$

where $\frac{d\alpha}{dt}$ is the rate of conversion at a constant temperature, $f(\alpha)$ is the reaction model which describes the changes in the physical or chemical properties, k is the rate constant, dependent on temperature T .

The conversion rate α (using TGA records) is defined as:

$$\alpha = \frac{w_o - w_t}{w_o - w_f} \quad (2)$$

where w_o , w_t and w_f are the initial mass of the sample at $t=0$, mass of the sample at time t and mass of the sample at final degradation respectively.

According to the Arrhenius relationship, the rate constant is the function of temperature and can be written as follows:

$$k(T) = k_0 \exp\left(\frac{-E}{RT}\right) \quad (3)$$

where k_0 is the pre-exponential factor, R is the gas constant (8.314 J/K mol), and E is the activation energy (kJ/mol).

Using Eqs. (1) and (3) following equation can be derived:

$$\frac{d\alpha}{dt} = k_0 \exp\left(\frac{-E}{RT}\right)f(\alpha) \quad (4)$$

Equation (4) describes the relationship between the conversion rate, rate constant and temperature which is used for recording the experimental data. To conduct kinetic study in TGA one of the ways is to use a heating rate, $= dT/dt$. Equation (4) can be written as follows:

$$\frac{d\alpha}{dT} = \frac{k_0}{\beta} \exp\left(\frac{-E}{RT}\right)f(\alpha) \quad (5)$$

Equation (5) is most basic equations used for kinetic study of thermal degradation.

E , activation energy can be estimated using various models. Generally used methods are Kissinger, Friedman, Flynn and Wall, Coats-Redfern, and Flynn-Wall-Ozawa (F-W-O) [15, 19, 25]. In this study, we used F-W-O method which gives slope $(-E/RT)$ from the line obtained by plotting $\log \beta$ against $1/T$ at fixed degree of conversion.

F-W-O fitting equation is given below [31]:

$$\log f(\alpha) = \log \left\{ \frac{k_0 E}{R} \right\} - \log \beta - 2.315 - 0.4567 \frac{E}{RT} \quad (6)$$

3 Result and discussion

3.1 Field emission scanning electron microscopy (FESEM)

The morphology of the jackfruit seed powder (JFSP) after calcination at 400 °C for 3 h, they were subjected to Field Emission scanning electron microscopy (FESEM) analysis of three different magnifications shown in Fig. 2a–c its diameter is around 3 μm and spherical shape obtained in all magnifications [6–9, 41].

Table 1 The range and levels of selected parameters studied in the experiments

Variables	Range and level	
	Low	High
Pollutant concentration (A) (ppm)	0.5	2.5
mixing speed (B) (rpm)	110	150
pH (C)	3	11
Catalyst dose (D) (mg)	20	100

Fig. 2 FE-SEM images of jackfruit seeds calcined at calcined at 400 °C for 3 h

3.2 Energy dispersive X-ray spectroscopy (EDX) and inductively coupled plasma optical emissions spectrometer (ICP-OES)

Energy dispersive X-ray spectroscopy analysis was conducted to quantify the near surface amount of elements present in the bio-adsorbent. From Fig. 3, it can be seen that 71.23% oxides, 4.94% calcium, 2.5% calcium 1.5% manganese and rest are carbon present in the sample on weight percentage basis [39]. Inductively Coupled Plasma Optical Emissions spectrometer analysis of the JFSP before the calcination of sample the major composition are magnesium, iron, calcium, copper, zinc, manganese, nickel and cobalt etc. [6–9, 41]. Theivasanthi also studied and shown

Fig. 3 EDX spectra of jackfruit seeds

similar trace elements like carbon, oxygen, magnesium, aluminium, phosphorus, potassium, titanium, iron, nickel and molybdenum.

3.3 Fourier transform infrared spectroscopy (FTIR) analysis

The FTIR spectral analysis revealed several functional groups on the surface of biomass shown in Fig. 4. The absorption peak observed at wave-number 3279 cm^{-1} is associated with O–H stretching vibration. The wave-numbers around 2927 cm^{-1} and 2364 cm^{-1} are corresponding to the C–H aliphatic stretching vibration. The peak displayed at 1643 cm^{-1} is attributing to the stretching band of the carboxyl double bond from carboxyl functional group. The peak around 1150 cm^{-1} is indicative of P=O stretching present in phosphate group. It has been observed by the [6–9, 22, 41]. [22] the carboxyl groups present in jackfruit bio-adsorbent plays a significant role in the cation cadmium removal. The peak displayed at wave number 1007 cm^{-1} is assigned to the O–H stretching of primary alcohol group. The peak at 929 cm^{-1} shows the C–C links in the bio-carbon. The presence of polar groups on the bio-carbon surface is likely to provide the considerable cation exchange capacity to the absorbent [2].

3.4 Thermo-gravimetric analysis (TGA)

The micrograph of TGA and DTA were used to study the thermal degradation behaviour of JFSP, as shown in Fig. 5. The DTA plot of JFSP shows three major endothermic peaks which indicated decomposition occurs in three main processes. The initial weight loss of 10% in the

Fig. 4 FTIR spectra of jackfruit seeds **Fig. 5** TG-DTA thermograms of jackfruit seeds

temperature range of 30–150 °C, mainly corresponds to the evaporation of physically absorbed aqueous solution. DTA curve also shows first sharp endothermic peak at 59 °C with change in enthalpy of -1.4 kJ/g. TG curve revealed that above 150 °C, thermal stability is gradually decreasing and decomposition of the jackfruit bio-adsorbent occurs with the complete decomposition at 626 °C. The second stage decomposition process occurs in the temperature range of 150–332 °C (weight loss ~64%), showing second

endothermic peak at 152 °C with change in enthalpy of -1.67 kJ/g [6–9, 41]. This result is attributed to the thermal depolymerisation of hemicelluloses, pectins, the cleavage of glycosidic linkage of cellulose and some parts of lignin [5, 28, 39]. At a temperature range of 350–625 °C, third and last stage decomposition process occur at peak 415 °C (change in enthalpy of -3.02 kJ/g) showing weight loss of ~99%. This indicates the thermal degradation reactions taking place for the major constituent of the biomass that is cellulose and remaining lignin [10, 36].

3.5 Thermal kinetic

Activation energy was estimated using TGA data at five different heating rates (10–30 °C/min). The average activation energy was determined using isothermal FWO method from the thermal degradation conversion range of 0.1–0.9. Figure 6 shows the linear plot jackfruit bio-adsorbent at various conversion rates. At various thermal degradation conversion rates, Activation energy was estimated from the slope of the curves. Summary of the activation energy values with R^2 values are given in Table 2. From the result, it can be observed that the activation energy varies with the varying rate of conversion, indicating high degree of probability to present more than one step reaction. Higher activation values are observed in the middle conversion rate (0.3–0.6) whereas lower activation energy values were observed for lower and higher conversion rate [6–9, 41]. Activation energy is the minimum amount of energy required to initiate a reaction. Activation energy is dependent on the pyrolysis reaction mechanism; higher value of

Fig. 6 Curves of fitting to kinetic model proposed by Flynn–Wall–Ozawa (F–W–O) to various conversion percentages

Table 2 Kinetic parameters obtained from F–W–O method with fitted equation for Jackfruit (JF) power

Conversion rate (α)	Equations	R^2	E, (kJ/mole)
0.1	$y = -2.5565x + 6.9645$	0.97	46.54
0.2	$y = -5.4866x + 11.498$	0.98	99.88
0.3	$y = -8.0076x + 15.621$	0.99	145.77
0.4	$y = -8.3145x + 15.835$	0.99	151.36
0.5	$y = -8.5099x + 15.862$	0.99	154.92
0.6	$y = -7.8898x + 14.329$	0.99	143.63
0.7	$y = 6.5171x - 8.9333$	0.99	120.02
0.8	$y = -5.4511x + 8.5543$	0.97	99.23
0.9	$y = -5.8823x + 8.1026$	0.97	107.08

activation energy indicates slower reaction [25]. This variation in activation energy is probably observed due to the mechanism involving the thermal decomposition of the jackfruit bio-adsorbent matter is different [18, 21, 18, 29, 40]. This could be explained by the TGA curve (Fig. 6). The thermal degradation temperature for $\alpha = 0.1$ –0.2 is around 270 °C and for $\alpha = 0.5$ is around 312 °C. This is the part of the TGA curve where maximum thermal degradation takes place. The observed activation energy is 130.69 kJ/mol, which corresponds to the degradation of hemi-cellulose, cellulose and lignin [31]. According to [26], value of the activation energy indicates about the type of adsorption taking place, which is physical or chemical adsorption. For the physical adsorption processes activation values usually found in the range of 0–40 kJ/mol, while higher activation energies values i.e. in the range of 40–800 kJ/mol indicates chemical adsorption processes [6–9, 39].

3.6 Statistical analysis

The results were obtained in terms of percentage removal of cadmium from the aqueous solution for all the individual experiments and then statistical analysis has been performed by using the Design-Expert. The optimisation study has been conducted with Design-Expert software to maximize the percentage removal and is presented in the following subsections. According to the design discussed in the earlier section, a total number of 30 experiments have been conducted. For the further study these values have been fed to the Design-Expert software, which helps to predict the model (quadratic equation). This equation relates the independent variables (A, B, C and D) and dependent variable (% removal) as shown in Eq. (7).

$$\begin{aligned}
 Y = & +33.61 + 13.22A + 0.39B + 3.67C \\
 & + 0.322D - 2.54A^2 - 0.0001B^2 - 0.158C^2 - 1.27D^2 \\
 & - 0.062A * B + 0.187A * C + 0.0187A * D \\
 & - 0.015B * C - 0.0015B * D + 4.68C * D
 \end{aligned} \quad (7)$$

where Y is the cooling rate and A, B, C and D are Pollutant concentration, mixing speed, pH and bio-adsorbent dose respectively. The value of correlation coefficient R^2 , used to check the model validity, is found to be 0.92.

3.7 Effects of pH

The effect of pH was studied from a range of 3–11 under the precise conditions at an optimum contact time of 1 h with 25 mg of the adsorbents used for 100 mL of aqueous solution, and at a room temperature and it was observed that with increase in the pH (3–8) of the aqueous solution,

Fig. 7 Effects of pH on cadmium removal

the percentage removal of cadmium ion increased up to the pH 7–8 as shown in Fig. 7. At pH 7.5, maximum removal was obtained for cadmium ions, with 96.7%. The increase in percentage removal of the cadmium ions may be explained by the fact that at higher pH the functional groups in the adsorbent surface were deprotonated and become negatively charged and while at lower pH, adsorption rate decreases and this is because at lower pHs, there is competition between H^+ ions and cadmium ions [6–9, 26, 41].

3.8 Effects of concentration and steering speed

The effect of initial concentration 0.5–2.5 ppm was investigated at ambient temperature with regular intervals of 10 min to 2 h for the adsorbent dosage of 20–100 mg and steering speed ranges 110–150 rounds per minutes. From the obtained result (Fig. 8), no significant change has been observed for the percentage removal of cadmium ions with the increase in initial concentration of cadmium ions. This is probably because of the removal of cadmium ions by forming complex with bio-adsorbent [6–9, 27, 41]. This observation indicates that the adsorption of cadmium by jackfruit bio-adsorbent is mostly depends upon the

number of active sites present which can be able make complex with the metal ion. Similar result has also been reported by Mohammad et al. [27], which shows saturation point at the same time interval for different initial conc. This corresponds to the availability of more vacant binding sites for cadmium adsorption during initial contact times [32].

3.9 Effects of adsorbents dosing

For the present study, effect of variation in dosing (25–100 mg) was analyzed under the specified conditions at ambient condition. Sample was collected for the analysis of percentage removal of cadmium in the regular interval of 10 min for the period 2 h. From the experimental results shown in Fig. 9, it can be clearly seen that the percentage removal of cadmium ions has been increased with the increase in adsorbent dosing. Maximum adsorption efficiency of about 97.12% was observed even at lower adsorbent dosing of 60 mg. Further increase in adsorbent dosing has not been shown any significant effect on the sorption [6–9, 41]. This is probably because of the availability of more binding sites at higher dose of bio adsorbents [13].

Fig. 8 Effects of Concentration and Steering Speed on cadmium removal

Fig. 9 Effects of dosing of adsorbent on cadmium removal

4 Conclusion

Successfully prepared jackfruit bio-adsorbent for the removal of Cadmium from the aqueous solution. FTIR analysis shows the presence of carboxyl group in jackfruit bio-adsorbent which plays a significant role in the metal removal and the presence of polar groups on the bio-adsorbent surface is likely to provide the considerable cation exchange capacity to the adsorbent. TGA analysis shows maximum thermal decomposition in the range of 150–380 °C. Activation energy was observed 130.69 kJ/mol using FWO method which attributed to a chemi-sorption adsorption process. Jackfruit bio-adsorbent was used successfully to remove cadmium ions from aqueous solution ~ 97% in 1 h at 120 rpm speed and pH 7.6. This study shows a new trend for using waste materials as a jackfruit bio-adsorbent for the benefit of pollution control.

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Compliance with ethical standards

Conflict of interest There are no conflicts of interest between authors.

Ethical standard There is no involvement of human or animal cell in this work. All the co-authors have seen the final manuscript and agreed with the submission to the journal. The manuscript has not been published elsewhere. The article is not consideration under any other journal in full or in part. No data or figures have been fabricated or manipulated. The funding agencies (NPIU/AICTE New Delhi, India) have been duly acknowledged. All the authors agree to transfer copy right to SN Applied Science's.

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