



## **Removal of Zinc from Aqueous Solution by Using *Desmostachya bipinnata* based Activated Carbon: Adsorption Isotherm and Kinetic Study**

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### **ABSTRACT**

*The performance of Activated carbon prepared chemically from Sungrass for the removal of Zn(II) from aqueous solution was investigated in batch technique. The activated carbon was characterized by FT-IR, XRD and SEM. In batch experiments, the influence of several operating parameters on the Zinc adsorption capacity was investigated including contact time, initial metal concentration adsorbent dosage and solution pH. Moreover, adsorption isotherm was found to be well fitted with Langmuir model. The maximum adsorption capacity of SGAC obtained from the Langmuir isotherm equation was 27.17mg/g. The adsorption kinetic followed the pseudo-second-order kinetic model. Thermodynamic study indicated that the adsorption of Zn(II) onto adsorbent surface was endothermic. This study indicates that Sungrass is a promising precursor for the production of low-cost and efficient activated carbon with a large surface area.*

**Keywords:** Heavy metal, adsorbent, activated carbon, adsorption isotherm, kinetic study,

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### **INTRODUCTION**

Heavy metal pollution has become one of the most serious environmental problems nowadays. The removal of these heavy metals from the environment is of utmost importance due to their persistence [1]. The main sources of heavy metals in water are from metal plating facilities, mining operations, battery manufacturing, production of pigments and paints etc. These heavy metals may enter into the human body directly or indirectly through food chain. Zinc is prior toxic pollutants in industrial wastewater discharged into the environment and ultimately become common ground water pollutants [2]. The presence of these pollutants in the environment can be detrimental to a variety of living species because these materials are non-biodegradable and tend to accumulate in living organisms, causing various diseases and disorders [3, 4]. The use of agricultural by-products for the preparation of adsorbents and its use in removal of heavy metal contamination from water has become increasingly popular because they are biodegradable, less expensive, abundant and efficient [5, 6]. Activated carbon prepared from those by products provides largest surface area and can be utilized not only for adsorption of heavy metals but also for different volatile organic compounds [7]. Adsorption on activated carbon is the most widely used process responsible for removal of inorganic and organic pollutants [8].

Activated carbons are amorphous in nature and to shows wide range of porosity and extended surface area. It is a result of an activation process of the carbon-rich material. The activation is initiated with the carbonization of the precursor to achieve high carbon content material [9]. An activated carbon with large surface area and high mesoporous volume can serve as an outstanding adsorbent for the removal of heavy metals from aqueous medium. The large surface area and mesoporous activated carbons are useful for an extensive application in separation, decoloration, deodorization, purification, and filtration operations [10]. The chemical composition, as well as the methods and the process conditions used during activation of biomass, play an important role in the formation of pores, pore volume, and surface area. Consequently, adsorption properties of the activated carbon vary from one species source to another [11]. In the present study activated carbon was prepared from sun grass by chemical activation method using  $H_3PO_4$  activating agent and evaluated its potential in removal of Zn(II) from aqueous

solutions through adsorption process. Chemical activation were employed to enhance the adsorption capacity of Sungrass activated carbon, it shows significant adsorption capacity for metal ions from aqueous solution. Batch experiments were used to evaluate the adsorption behaviour of the SGAC for metal ions with respect to contact time, initial ion concentration, adsorbent dose, and pH.

## MATERIAL AND METHODS

*Desmostachya Bipinnata* commonly known as sungrass was used for the preparation of activated carbon by chemical activation method. Sungrass powder was put in autoclave reactor and was heated in muffle furnace at the temperature of 250°C for 2 hours for the preparation of carbon. The resultant carbon was activated by  $H_3PO_4$  [12] and was again heated at the temperature of 500 °C for 4 hours. The activated carbon was washed by distilled water and dried in hot air oven. Activated carbon prepared from sungrass was designated as SGAC and was used as adsorbent for the removal of Zn(II). Various characterization techniques were used to characterize the surface of the SGAC. The FTIR spectra ( $400-4000\text{ cm}^{-1}$ ) of the SGAC before and after adsorption of Zn(II) were obtained as KBr pellet using a Perkin Elmer Spectrum-2 spectrometer. XRD pattern was carried out using X-ray diffraction technique (Regaku; MiniFlex300/600) using Cu  $K\alpha$  radiation and the morphology of SGAC were examined on (SEM) scanning electron microscopy (ZEISS SEM unit). Batch adsorption experiments were performed to evaluate the effect of the different parameters on the adsorption process. The parameters which have been optimized include contact time, metal ion concentration, adsorbent dosage and pH. All the solutions were analyzed for metal ion concentration using atomic absorption spectrophotometer (AAS; Perkin-Elmer, Analyst, origin USA). Removal percentage and adsorption capacity of adsorbent were calculated by using following equations; Metal adsorption efficiency (E %) was calculated by

$$E = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

Metal adsorption capacity was calculated by

$$q_e (\text{mg/g}) = \left( \frac{C_0 - C_e}{m} \right) V \quad (2)$$

Where  $C_0$  is the initial metal ion concentration (mg/L) and  $C_e$  is the concentration of metal in the solution after adsorption (mg/L) and V is the volume of solution and m (g) is the mass of adsorbent used.

## RESULT AND DISCUSSION

### Characterization

FT-IR spectroscopy was performed to validate the functional groups present on activated carbon and it augments in establishment of contact with the heavy metals mentioned above. The spectral data of activated carbon show the peaks at  $2926\text{cm}^{-1}$  and  $2887\text{cm}^{-1}$  corresponding to C – H asymmetric and symmetric stretching bands, the peak at  $1603\text{cm}^{-1}$  correspond to C=C stretching mode of aromatic ring. The peak at  $1507\text{cm}^{-1}$  and  $1436\text{cm}^{-1}$  is due to  $\text{CH}_2$   $\text{CH}_3$  stretching modes [13] and the peak at  $1403\text{cm}^{-1}$  is attributed to the oxygen containing functional groups. The band at  $1089\text{cm}^{-1}$  confers C-O stretching of hydroxyl group and the peaks between  $900-700\text{cm}^{-1}$  arise due to increase in aromaticity in the system [14]. After the adsorption of Zn(II), it could be found from the spectra that some primary peaks show evident shifts, reflecting the electronic attraction and electron transfer between these heavy metal ions and the functional groups (Fig. 1).

The structural characterization of the prepared activated carbon was carried out using X-ray diffraction technique to determine the crystalline nature. The result indicated that all the diffraction peaks are indexed to face-centered cubic structure. Four characteristic peaks ( $2\theta = 21.94^\circ$ ,  $23.907^\circ$ ,  $27.217^\circ$ ,  $31.56^\circ$  and  $33.28^\circ$ ) marked by their Miller indices (122), (311), (101), (122) and (312) were observed and shows a crystalline structure and there was no other impurity phase or secondary phase in the XRD pattern. According to the XRD results, the average crystal sizes were estimated using the Debye–Scherrer equation and found to be 18.75 nm (Fig. 2).

Scanning electron microscopy was performed to visualise the surface morphology of SGAC. For scanning electron microscope studies samples were mounted on aluminium support using a double adhesive tape coated with gold in HUSSGB vacuum coating unit and observed in ZEISS SEM unit. It can be seen from the SEM micrographs that the surface of SGAC shows irregular and rigid structure (Fig. 3). The activated carbon (SGAC) possesses irregular grooves and porous structure. The development of pores on the surface of the activated carbon is attributed to  $H_3PO_4$  activation, thus leading to large surface area and porous structure of the activated carbon and these characteristics are responsible for adsorption of heavy metals [15].

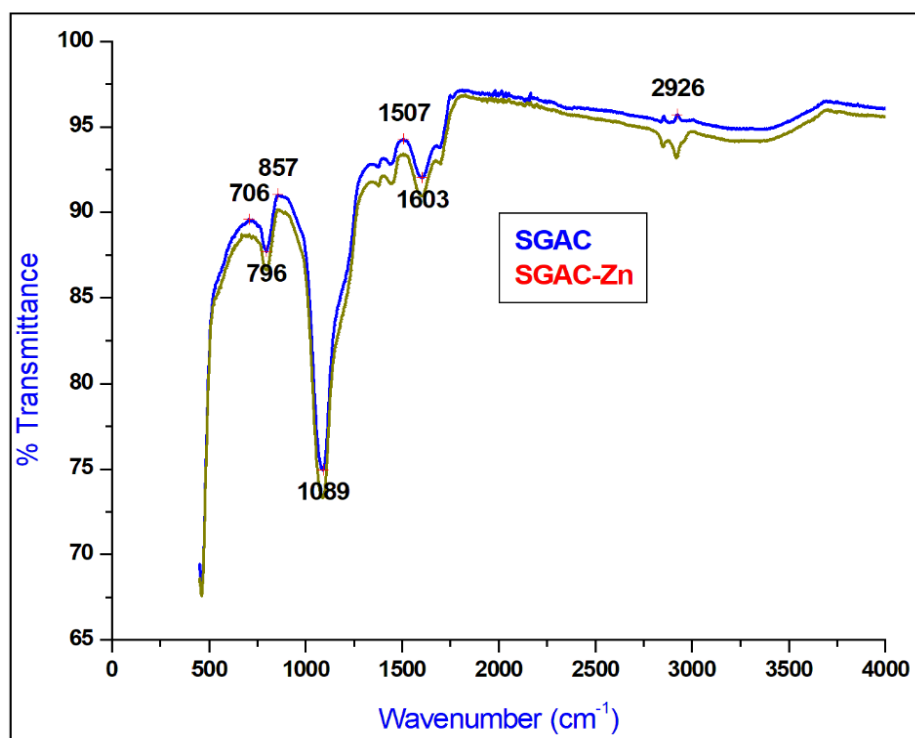


Fig. 1: FTIR spectra of activated carbon before and after the adsorption of Zn(II).

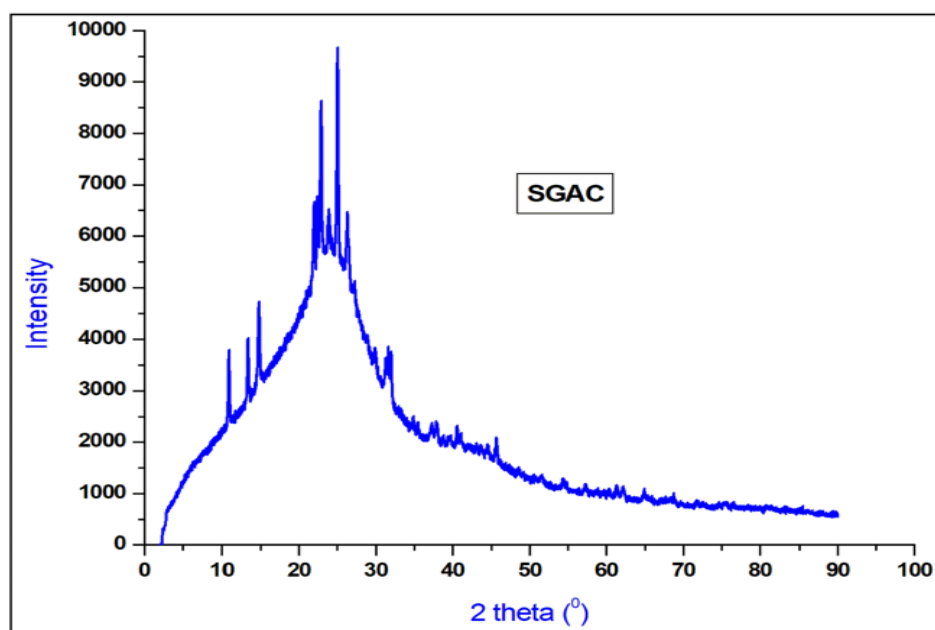


Fig. 2: XRD spectra of sungrass activated carbon (SGAC).

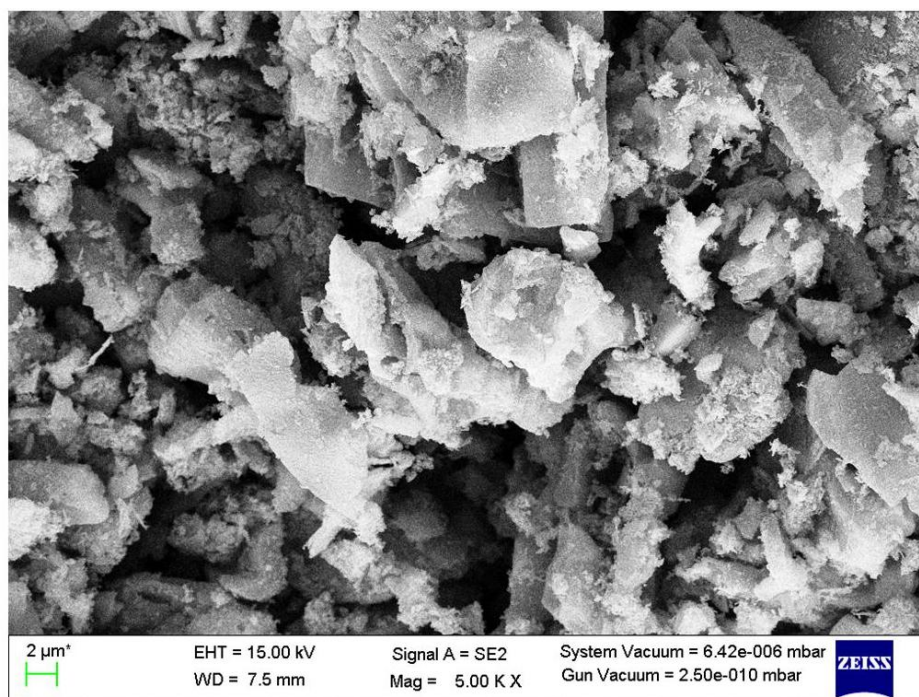


Fig. 3: SEM image of Sungrass activated carbon (SGAC).

#### BATCH ADSORPTION STUDIES

Batch adsorption was performed in a series of Erlenmeyer conical flasks containing 100 mL of metal solutions with different metal initial concentrations (10-100 mg/L), stirred at contact time between (15-150 min.), adsorbent dose were added between (0.2-4.0 g/L) and pH was adjusted in the range of (2-12) and each flask were kept on a shaker at room temperature. The parameters optimized were contact time, metal ion concentration, adsorbent dosage and pH.

##### Effect of contact time

The effect of contact time on removal of Zn(II) by SGAC was investigated at different contact time ranged between 15- 150 minutes for a fixed concentration of metal ions (10mg/l) and adsorbent mass (2 g/L) [16]. It was evident from the results that the removal percentage increases with increases in contact time and shows maximum removal of 76% at 90 minutes (Fig. 4). It is obvious from the fact that by increasing time, the adsorption rate increases at first depends on unoccupied sites on the surface of adsorbent therefore, metal adsorption was relatively high. Further increasing contact time, the percentage removal did not changed and showed steady state which is the indication that equilibrium is attained. An initially large number of vacant sites are available and therefore favours the adsorption. Also the difference of metal ion concentration in the solution and solid liquid interface is high which results in faster adsorption of metal ions at initial phase of contact time [17]. However, few minutes later the remaining free surface sites are hard to be occupied due to repulsive forces between the metal ions on the solid and the aqueous phases, [18, 19].

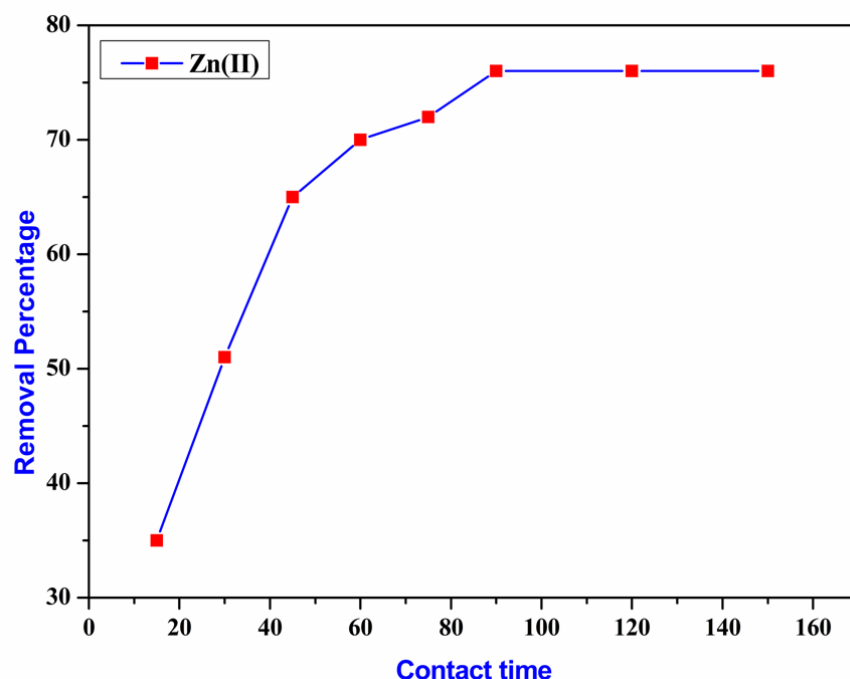


Fig. 4: Effect of contact time on removal of Zn(II) using SGAC.

#### Effect of concentration

The effect of metal ion concentration on the adsorption uptake quantities shows that the adsorption equilibrium capacity of the activated carbon. The effect of initial metal ion concentration on percent removal from aqueous solution was evaluated at different concentration ranged between 10 to 100 mg/L. It was observed that with increasing metal ion concentration the percentage removal decreases from 74% to 41% for Zn(II) on increasing metal ion concentration from 10-100 mg/L (Fig. 5). The maximum removal was observed the concentration of 10mg/L with the removal percentage of 74%. At the initial stage, metal adsorption takes place quickly that is related to the presence of adsorption active sites on the adsorbent which can adsorb the metal ions quickly and then gradually decreases. However, the number of these adsorption active sites gradually decreases with increase in the adsorption time and the number of metal ions adsorbed on the adsorbent, as well [20].

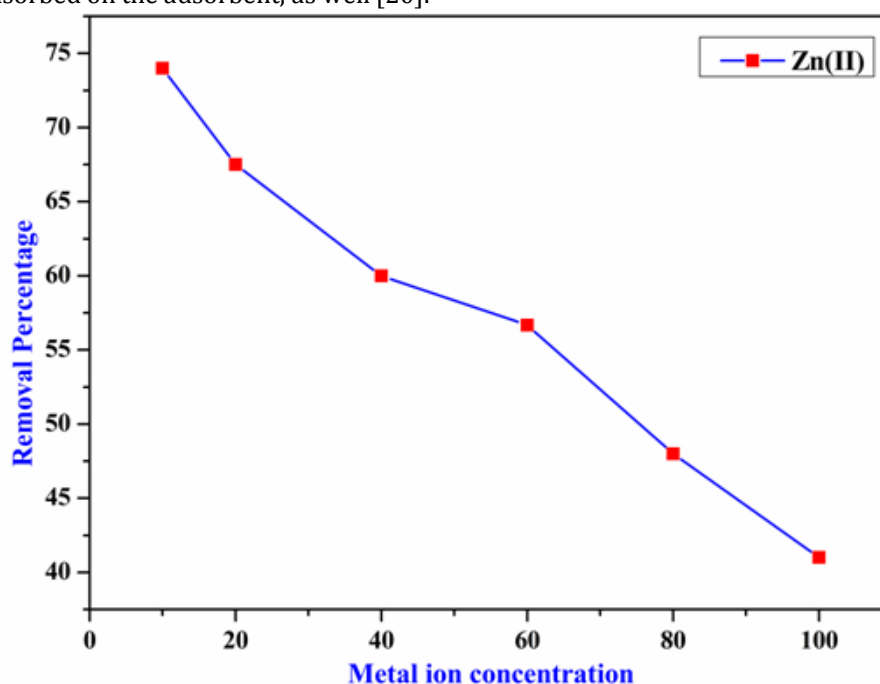


Fig. 5: Effect of metal ion concentration on removal of Zn(II) using SGAC.

### Effect of dose

The dependence of metal adsorption on the amount of adsorbent dosage (SGAC) was studied at different adsorbent mass (0.2 – 4.0g/L) and the other optimized experimental conditions. The effect of adsorbent dosage on Zn(II) ion removal and as well as on the adsorption capacity of SGAC was evaluated on the experimental conditions such as metal ion concentration, pH and contact time. It was observed that removal percentage of Zn(II) increases from 35-76% and amount of residual metal ions decreases sharply. The maximum removal of 76% were observed for Zn(II) at an adsorbent dosage of 2.0 g/L (Fig. 6). However, after attaining equilibrium, percentage removal did not seem to increase significantly due to equilibrium limitation i.e. for a given initial metal ion concentration, further increase of the adsorbent dosage did not showed any effect on the adsorption rate of metal ions, instead showed slight decline which may be attributed to overlapping of available binding sites results that results in decrease in total surface area of the adsorbent [21].

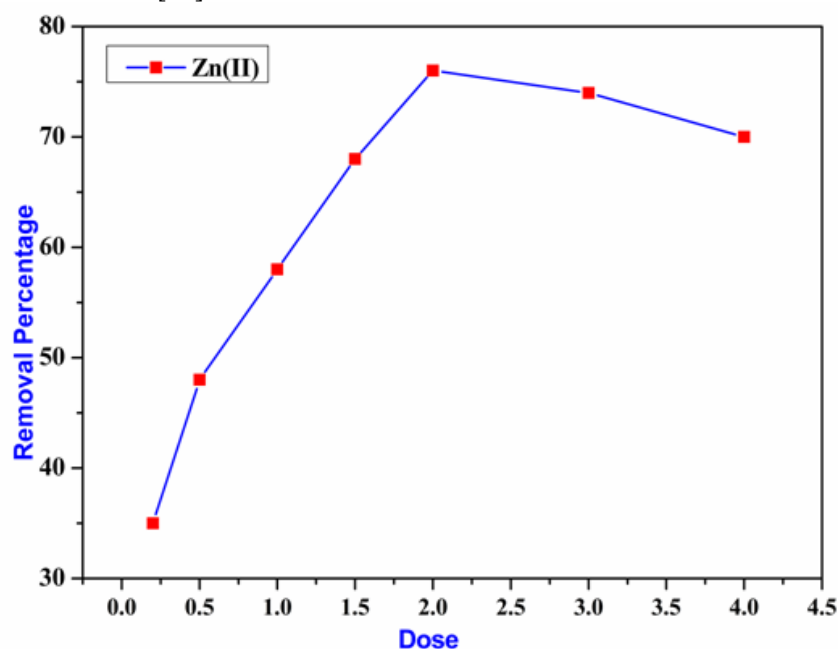


Fig. 6: Effect of adsorbent dose on removal of Zn(II) By using SGAC.

### Effect of pH

Since the solution pH affects the surface charge of adsorbent and ionization degree [22]. The negative charges on the surfaces of adsorbent are affected by the pH of the solution [23]. Different adsorbents shows different tendency for the removal of different metals from the solution depends upon the pH of the solution [24]. Since SGAC was used as an adsorbent, the results revealed that the removal percentage for Zn(II) increases with increase in pH from 2 to 6. The maximum removal of Zn(II) was observed at pH 6 with removal percentage of 70% and beyond pH 6, percentage removal shows slight decline (Fig. 7). A decline was observed probably due to saturation of all active sites present on the adsorbent surface on or beyond pH 6. It is apparent that optimum percentage removal was obtained at pH 6; therefore pH 6 was selected for further studies [25].

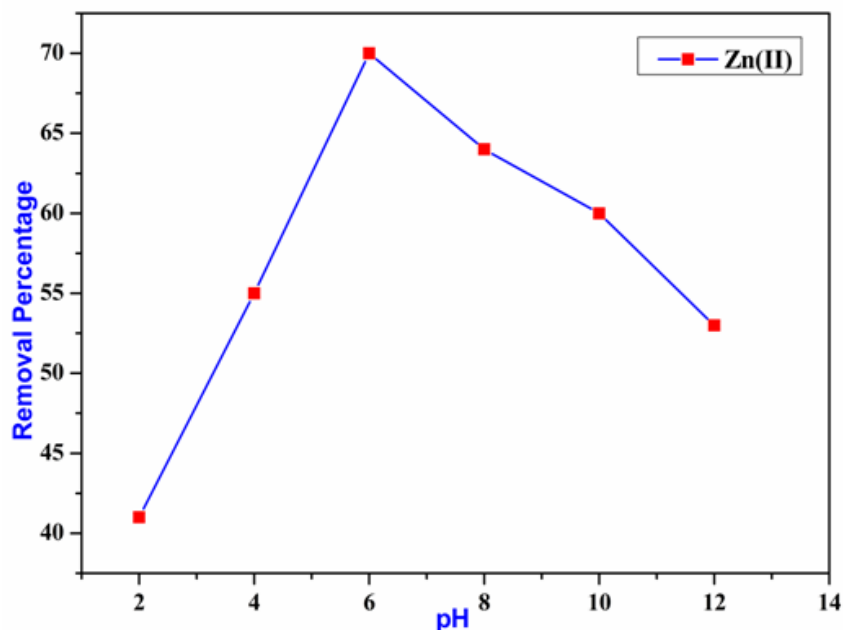


Fig. 7: Effect of pH on the removal of Zn(II) by using SGAC.

### Adsorption Isotherm

The adsorption isotherm is used to determine the distribution of metal ion between solution and the adsorbent surface at equilibrium [26]. To determine the suitable model for the design process isotherm data was analysed by fitting them to the two models i.e. Freundlich and Langmuir isotherm models. Freundlich isotherm expects multilayer adsorption happens over a heterogeneous surface of adsorbent with non-uniform distribution of adsorption heat [27]. The well-known logarithmic form of the Freundlich isotherm is given by the following Equation;

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (3)$$

Where  $C_e$  the equilibrium metal ion concentration (mg/l),  $K_F$  provides an indication of the adsorption capacity ( $\text{mg g}^{-1} (\text{L mg}^{-1})^{1/n}$ ) and  $n$  is related to the intensity of adsorption [28]. The value of the parameters  $K_F$  and  $n$  was obtained using linear fit of the plot  $\log q_e$  vs  $\log C_e$  (Fig. 8a). The Langmuir isotherm assumes monolayer adsorption onto a surface containing a limited number of available adsorption sites of uniform strategies of adsorption. Langmuir isotherm was used to fit the equilibrium adsorption data and linear form of this isotherm model is given by the following Equation;

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (4)$$

Where  $q_e$ ,  $q_m$  are equilibrium adsorption capacity under given condition and maximum adsorption capacity, respectively.  $C_e$  is the equilibrium concentration.  $K_L$  ( $\text{L mg}^{-1}$ ) is the Langmuir constant which is related to the energy of adsorption [29]. Maximum adsorption capacity,  $q_m$  ( $\text{mg g}^{-1}$ ) and  $K_L$  ( $\text{l/g}$ ) were obtained from slope of the graph plotted between  $C_e/q_e$  versus  $C_e$  (Fig. 8b). It was observed that the adsorption of Zn(II) are well fitted by Langmuir isotherm, which indicates that there is occurrence of monolayer adsorption of Zn(II) on the surface of SGAC. The maximum adsorption capacity ( $q_m$ ) obtained were 27.17 mg/g for Zn(II) as depicted in Table 1.



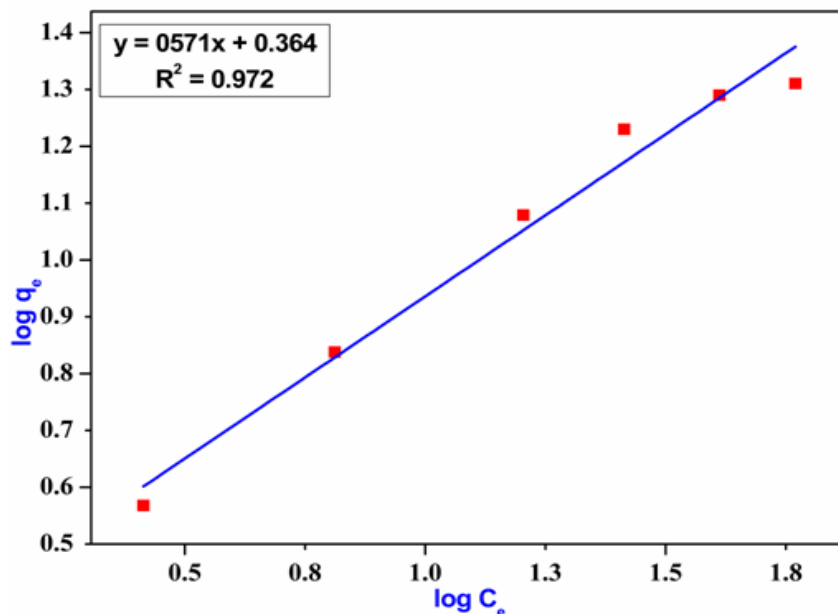


Fig. 8a: Freundlich isotherm of Zn(II) on SGAC.

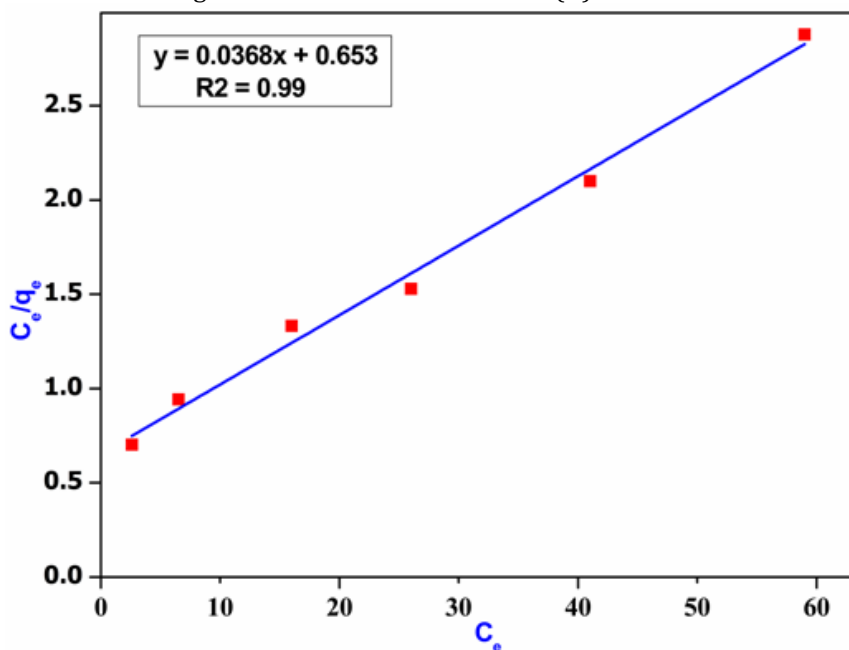


Fig. 8b: Langmuir isotherm of Zn(II) on SGAC.

**Table- 1: Adsorption isotherm parameters of Zn(II) adsorption on SGAC.**

Metal ion	Freundlich Isotherm			Langmuir Isotherm		
	$K_f$	$N$	$R^2$	$K_L \text{ mg}^{-1}$	$q_m \text{ mg/g}$	$R^2$
Zn(II)	2.312	1.751	0.97	0.056	27.17	0.99

### Kinetics study

The kinetics of adsorption is of significant importance in understanding the dynamics of adsorption, therefore, plays an important role in design and modelling of adsorption process. Kinetic investigation have been carried out using pseudo-first order, pseudo second- order and intraparticle diffusion models to investigate the adsorption of Zn(II) ion from 10 mg/L onto sungrass activated carbon (SGAC). The linearized forms of pseudo first-order and pseudo second-order kinetic equations are given as following Eqs. (5) and (6), respectively [30, 31].

$$\log(q_e - qt) = \log q_e - \frac{K_{1ad}}{2.303} t \quad (5)$$

$$\frac{t}{qt} = \frac{1}{K_{2ad} q_{e2}} + \frac{1}{q_e} t \quad (6)$$



The pseudo-first-order rate constant  $K_{1ad}$  ( $\text{min}^{-1}$ ) was determined experimentally by plotting  $\log (q_e - q_t)$  versus  $t$  (Fig. 9a) and the second order rate constant  $K_{2ad}$  ( $\text{g mg}^{-1} \text{m}^{-1}$ ) was calculated by plotting a curve of  $t/q_t$  vs  $t$  (Fig. 9b). Among the two kinetic models adsorption follows the pseudo second-order kinetic model with higher correlation coefficient  $R^2 = 0.99$  as compared to that of pseudo first order kinetic model which suggests that the rate-limiting step may be the chemisorption. Moreover, the experimental  $q_{e \text{ exp}}$  values agreed well with the theoretical  $q_{e \text{ cal}}$  values the adsorbent studied. In addition, Weber and Morris model is a widely used intraparticle diffusion model to understand mechanism and predict the rate controlling step [32]. The rate constant of intraparticle diffusion ( $K_{id}$ ) was determined using the following equation:

$$q_t = K_{id} t^{0.5} + C \quad (7)$$

The intraparticle diffusion rate constant  $K_i$  and the parameter  $C$  were obtained by slope and intercept of a plot of  $q_t$  vs  $t^{0.5}$  (Fig. 9c). It can be seen that the obtained curve is subdivided into linear part and non-linear part indicating more than one process such as external diffusion, intraparticle diffusion etc. are involved in the adsorption of metals. In the beginning, magnitude of  $q_t$  increases sharply with increase of time due to the immediate availability of active adsorption sites on large surface area of SGAC that signifies the external surface adsorption. The second subdued portion of the plot represents the steady adsorption capacity, where rate-controlling step is the intraparticle diffusion [33]. The intraparticle diffusion is not the only rate controlling step for the adsorption of metal ions onto SGAC. The calculated values of pseudo first order, pseudo second order kinetic and intra particle diffusion parameters are summarized in the Table 2.

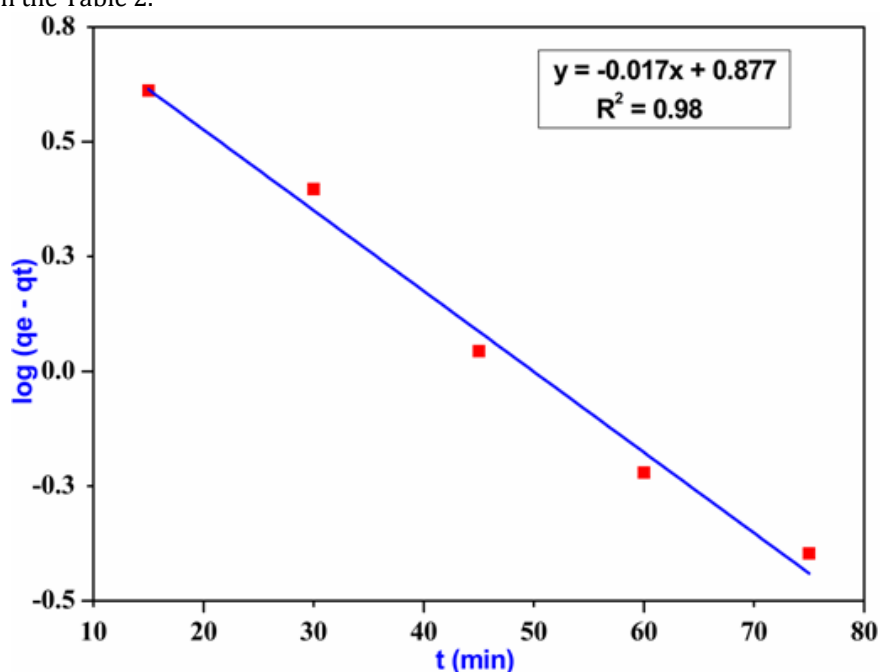


Fig. 9a: Pseudo first order kinetics of Zn(II) on SGAC.

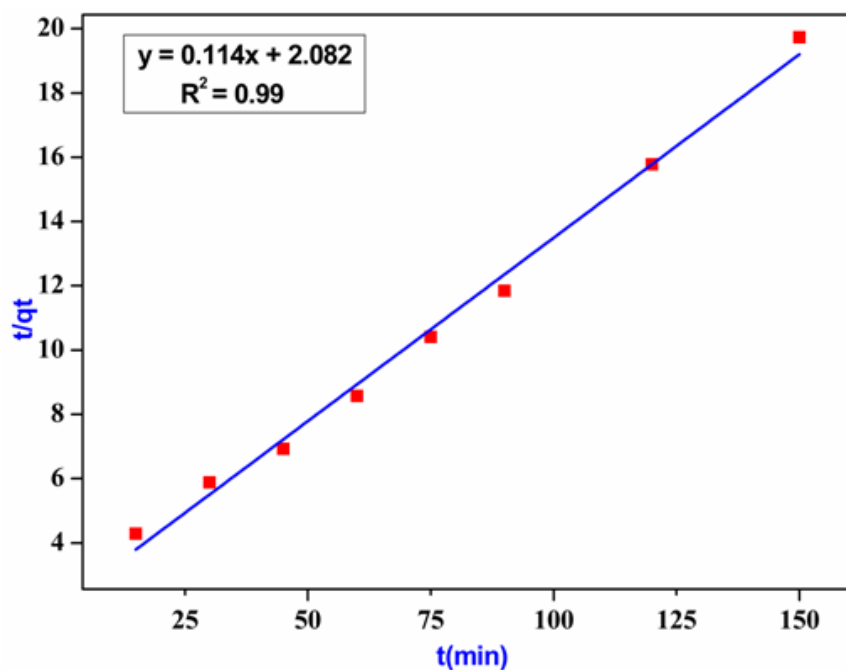


Fig. 9b: Pseudo second order kinetics of Zn(II) on SGAC.

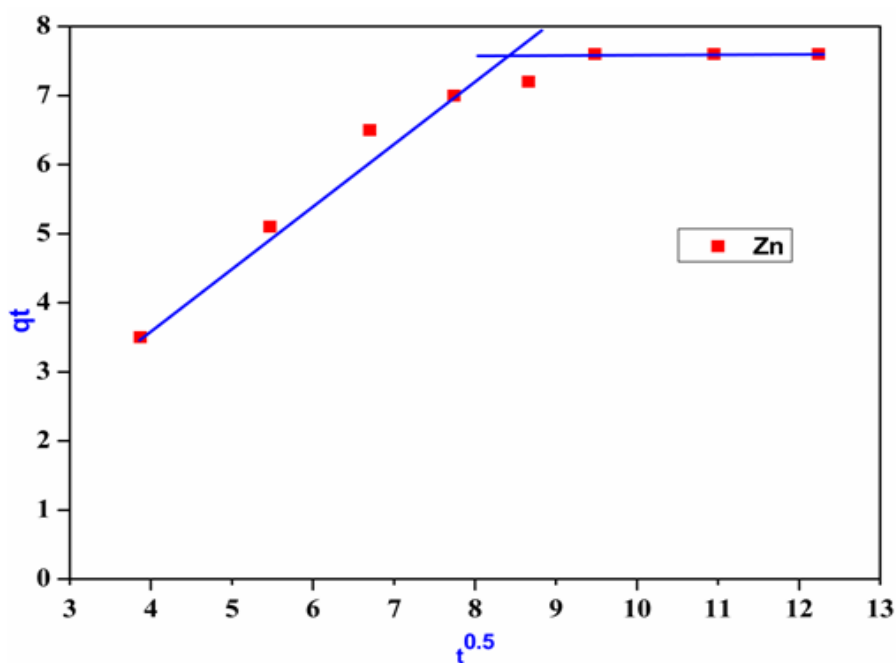


Fig. 9c: Intraparticle diffusion plot for Zn(II) ion removal from aqueous solution using SGAC.

**Table- 2. Kinetic study parameters of Zn(II) adsorption on SGAC.**

	Pseudo first order reaction				Pseudo second order reaction		
	$q_{e \text{ exp.}}$ (mg/g)	$K_1$ (g/mg min)	$q_{e \text{ cal.}}$ (mg/g)	$R^2$	$K_2$ (g/mg min)	$q_{e \text{ cal.}}$ (mg/g)	$R^2$
Zn(II)	7.6	0.039	7.533	0.98	0.0099	6.94	0.99
	Intra particle diffusion						
	$C$	$K_{id}$ (g/mg min)	$R^2$				
	Zn(II)	2.652	0.4744	0.76			

### Thermodynamics

Thermodynamic behaviour was studied to determine the thermodynamic parameters including the change in standard enthalpy ( $\Delta H$ ), standard entropy ( $\Delta S$ ) and Gibbs energy ( $\Delta G$ ) due to transfer of unit mole of solute from solution onto the solid-liquid interface. The thermodynamic parameters were calculated using the following equations (8, 9, 10) [34-35]:

$$\Delta G^\circ = -RT \ln K_d \quad (8)$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (9)$$

$$\ln K_d = \frac{\Delta S^\circ}{R} + \frac{\Delta H^\circ}{RT} \quad (10)$$

Where  $R$  ( $8.314 \text{ J K}^{-1} \text{ mol}^{-1}$ ) is the universal gas constant,  $T$  (K) is the absolute solution temperature and  $K_d$  is the is distribution coefficient. It can be seen that the negative values of  $\Delta G$  are clearly obtained for adsorption of Zn(II) at different temperatures with adsorbents studied, suggesting that the this adsorption process can occur naturally [36]. For  $\Delta S$  values, they are positive which are attributed to spontaneous and irreversible processes for metal adsorption. In the case of  $\Delta H$  values, they also are positive that indicate the endothermic nature of the adsorption of Zn(II) on the adsorbents studied [37].

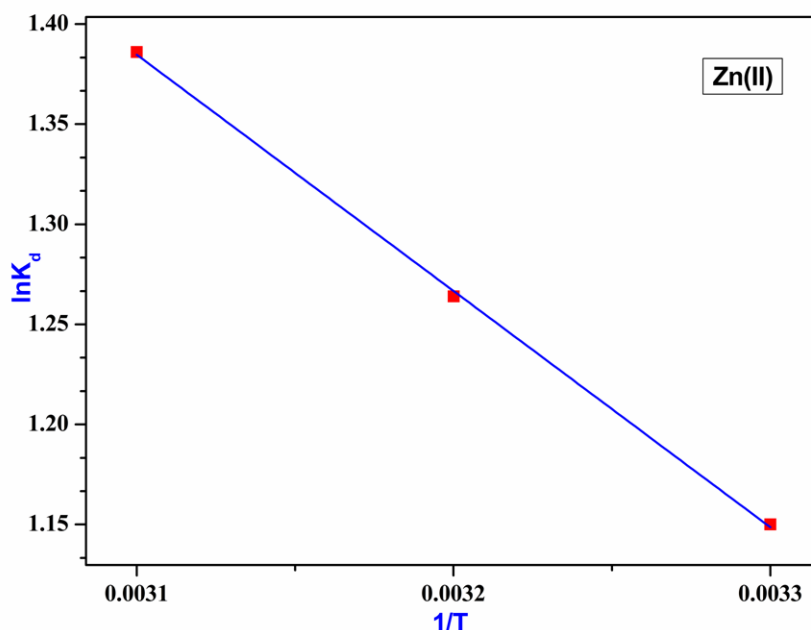


Fig: 10: Thermodynamics of Zn(II) ion removal from aqueous solution by using SGAC.

**Table- 3: Thermodynamic parameters of Zn(II) adsorption on SGAC.**

	$\Delta G^\circ \text{ KJmol}^{-1}$	$\Delta H^\circ \text{ KJmol}^{-1}$	$\Delta S^\circ \text{ Jmol}^{-1}\text{K}^{-1}$
<b>Zn(II)</b>	- 2.614	9.727	0.041
	- 2.871		
	- 3.145		

### CONCLUSION

In the present investigation, performance of activated carbon prepared from sun grass as an adsorbent was evaluated for the removal of Zn(II) ion from aqueous solution. The activated carbon was prepared by chemical activation method. The large surface area and multiple functional groups enhance the adsorption performance of activated carbon. The probable interaction of metal ions and adsorbent were displayed by FTIR and SEM studies. It was also observed that the percentage removal of metals increased with increase in adsorbent dosage, contact time but decreased with increase in concentration of the metal solution and the maximum metal ion adsorption was obtained at pH 6. Sun grass activated carbon shows different removal efficiency for different metals with initial ion concentration of 10 mg/L. Analysis of different adsorption model like Langmuir, and Freundlich gave the impression that Langmuir isotherm model fitted best over the other two isotherms models. This suggests that metal adsorption is limited with monolayer adsorption. It was also found that the rate of adsorption can be better described by pseudo second-order model which is an indication for chemisorptive rate-limiting reaction and intraparticle diffusion model indicates that external force along with the intraparticle diffusion are responsible for the adsorption of Zn(II) on SGAC. Thermodynamics study revealed that the adsorption

process are spontaneous and exothermic. Sungrass (*Desmostachya bipinnata*) activated carbon thus serves as a suitable clean and green technology on its application for removal of metal ion contamination from drinking water as it requires no power supply, is easy available and cost effective.

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