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Research Article

Evaluation of adsorption isotherms and kinetics of chloride ion in water using biochar derived from locally available agro-waste

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ABSTRACT

The uses of agricultural byproduct solid wastes to develop low cost sorbents are advantageous and promising for the removal of water contaminants. It serves the purposes of both environmental remediation and appropriate management of agricultural waste generated during agricultural processing. In this study, locally available apricot seed shell and Salix Alba leaves were utilized as agro-waste for the preparation of adsorbents. The biochar was prepared at 300-400°C via pyrolysis and 80 mesh particle sizes were modified by 1N HCl. The unmodified and acid modified local Salix Alba leaves and Apricot seed shell biochar were used to study the adsorption of chloride ion in water, which can damage appliances of industries and also poses health issues at elevated concentration. Adsorption kinetics including pseudo 1st and 2nd order and equilibrium studies including Langmuir and Freundlich isotherm were conducted at pH 7. The adsorption efficiency of modified biochar was much higher than the unmodified biochar due to induce surface positive charge. The Langmuir maximum adsorption of modified Salix Alba leaves sorbent was found to 22.98 mg/g, while modified Apricot seed shell biochar was found to 25.83 mg/g. The experimental data were simulated and applied to fit adsorption isotherm and kinetics models and found a better compliance with Langmuir isotherm model and pseudo 2nd order kinetics model. The RL value and KL value indicated favorable sorption and enhanced sorption affinity. The kinetics also indicated that there is interaction between adsorbate and adsorbent active sites and the result indicated a significant potential of both adsorbents for the removal of chloride ion.

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INTRODUCTION

Adsorption is the most common and effective separation technique used in the water and wastewater treatment [1-3]. The commercially available activated carbon (AC) is commonly uses in the water and wastewater treatment methods due to its large porous structure, huge surface area, high capacity, and hydrophobic properties, however, commercially activated carbon is expensive and has major regeneration concerns [1, 2, 4]. Therefore there are a significant number of reports are available on the development and uses of organic, inorganic, nanomaterial adsorbents and ion exchange resins for wastewater treatment. One of the known materials is biochar has regarded six times less expensive than commercially available activated carbon [5]. However, modern research has concentrated on using materials of biological origin, particularly agricultural waste, to remove contaminants due to many beneficial reasons. Agricultural byprod-

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uct solid wastes, such as fruit and vegetable wastes, leaves, seeds, tree waste, fibers, fruit peels, dates, sawdust, bark, etc., are being utilizing for the development of an effective adsorbent alternative to costly activated carbon and other inorganic and organic materials [6, 7]. The use of agricultural wastes in water and wastewater treatment serves the purpose of both environmental remediation and suitable management of agricultural waste generated during processing [8, 9]. However a large number of studies have reported that the adsorption capacity of raw agricultural waste materials for pollutants is rather lower, due to their inherent chemical composition and structure [9, 10]. As a result, chemical and physical surface changes are done to improve their adsorption properties. Chemical surface modification approaches include protonation, amine grafting, metals or metal oxides impregnation and surfactants, whereas physical modification involves heating materials to remove impurities and volatile matter [9-13].

Biochar, an inexpensive carbonaceous porous material obtained by heating agricultural wastes without oxygen, is an effective sorbent for the removal of a variety of pollutants, due to its improved adsorbing properties [4, 5, 7, 12]. However, commercial anionic ion exchange resins such as Indion NSSR, Amberlite IRN-78, Amberlite IRA 400, Imac HP555, and Purolite A100 are available but are more expensive [14]. A significant number of studies available on the use of biochar and activated biochar for the treatment of cations, metals and dyes. Rice husk activated biochar was used to remove ranitidine [2]. The biochar of rice husks treated with HCl and NaOH increases the surface area and used to remove Mn (II), Se (II), and Fe (II) [5]. Banana pith, an agricultural waste, was utilized to make biochar, which was used to remove As (V) [6]. The biochar of bagasse, bamboo, poplar wood and wheat bran impregnated with Fe and Ca have 12-70 times higher adsorption efficiency for phosphate ion than unmodified biochar [11]. Similarly, biochar from pecan shells, pistachio shells and wood sawdust treated with KOH and FeCl3 were used to remove phenol [12]. The biochar of Conocarpus green waste removed 90% of NO3- ion on impregnation with magnesium and iron oxides [13]. Apricot shell biochar has high adsorption capacity for atrazine [15]. Similarly biochar obtained from reed have enhanced adsorption capacity toward pentachlorophenol by six-fold on treatment with 1 M HCl [16]. Similarly, NaOH impregnation of wheat straw biochar enhances adsorption capacity of toluene by three-fold [17]. The biochar derived from various agricultural wastes have been employed for adsorption of chloride ions from aqueous solution by many researchers. The non- modified biochar and chemically modified biochar derived from date palms were utilized to removal free residual chlorine from aqueous solution [18]. Similarly biochar derived from malt spent rootlets, walnut shells, and kernels of apricot, olive, carob and grape were also utilized for the removal of free and total chlorine from water [19]. Though the presence of chloride ions in water has no significant health issues, but an excess of chloride ions enhances the hardness, conductivity and saltiness of the water, which can damages appliances. The present study work employed biochar and acid activated biochar derived from locally available agricultural wastes including Salix alba leaves and apricot seed shell for the removal of chloride ions from aqueous solution using batch adsorption model.

Availability of Chloride in Water

Chloride in water increases electrical conductivity and corrosivity of water and can damage water pipelines, industrial appliances and would raise metal ions level in water. Chloride ions in water are associated with sodium, potassium, and calcium. The presence of chloride in groundwater can be caused through weathering of rock, salt spray deposition, wastewater, inorganic fertilizer, septic tanks, and intrusion of ocean water into the groundwater sources in coastal areas [8, 20]. The usage of salt for de-icing the roads is one of the most common anthropogenic sources of chloride ions in water [20]. Chloride levels in drinking water greater than 250 mg/l are considered undesirable according to regulation [20].

Local agricultural Waste

Many species of Salix grows naturally in the cold, arid region of Ladakh in the Trans-Himalaya. Salix Alba (SA), often known as white willow, is a deciduous tree whose leaves are utilized mainly as cattle feed in the region. Similarly, Ladakh is the largest Apricot producing state in India, producing 15,789 tonnes of Apricot annually [21]. A significant volume of seed shell is producing during the processing. The Apricot shell shells (ASS) are hard material not been use in any form, other than being burned in the local heating system to warm rooms during winter. Figure 1 depicts the Salix leaves and see shell.

Figure 1. a) Salix Alba leaves b) Apricot seed shell

The main purposes of this study were to: i) evaluate the adsorption removal of anion (Cl^- ion) from synthetic water using raw Salix alba leaves biochar (SALB) and acid activated Salix Alba leaves biochar (ASALB), (ii) evaluate the adsorption removal of anion (Cl^- ion) using Apricot seed shell biochar (ASSB) and acid activated Apricot seed shell biochar (AASSB), (iii) study the adsorption kinetics and isotherms.

MATERIALS AND METHODS

All the chemicals and reagents of analytical grade were purchased from Biological museum , Agra, India and were used. Synthetic water was prepared by dissolving Potassium Chloride (KCl) in distil water to obtain 100 mg/l concentration. 0.05N KCrO4 and 0.01N AgNO3 solution were used in the analysis. Fresh Salix Alba leaves and Apricot seed shell were were collected from Kargil, UT Ladakh. Muffle furnace (model- MSW 25) and Bruker Alpha II- FTIR spectrometer were used as instrumentation.

Preparation of biochar

The fresh leaves were washed with tape water and then with deionized water to remove dirt. The leaves were cut in to small pieces and then dried in an oven at 100 oC for 24 hour. Similarly Apricot seed shells were collected from the oil processing unit at Hardas, Kargil of UT Ladakh and were washed with water and deionized water to remove dirt. 300g of leaves and seed shells were placed in the reactor of muffle furnace (model- MSW 251). The biochar of the materials were prepared by subjecting the material to pyrolysis at a temperature of 300°C, 330°C, 350°C, 370°C and 400°C with a continuous flow of nitrogen gas at the flow rate of 100-200 ml/min. The gases formed during pyrolysis were bubbled in water. The temperature of the reactor was adjusted by using the temperature knob. After 5 hour the material were allowed to cool down and then powdered the materials to get a mesh size of 80. The yield of biochar was found higher at 370°C and get decreased with the temperature rise. A systematic representation for processing of biochar is depicted in figure 2 and 3.

Figure 2. Apricot seed shell biochar

Figure 3. Salix Alba leaves biochar

10g of Salix Alba leaves biochar (SALB) and Apricot seed shell biochar (ASSB) were soaked in 1N HCl solution for 3hr and separated. The adsorbents were washed with deionized water till pH become neutral and then dried in oven at 60 0C.

Characterization with Zero point of charge (ZPC)

A base electrolyte of 0.01 M NaCl was taken in a series of volumetric flasks and adjusted the pH of solution with pH range (2-10) by using 0.1N HCl and 0.1N NaOH solution. 1g of adsorbents were added in 50 ml of each flask and kept at the shaker for 8h and recorded the pH using pH meter as pHf. A plot of pH (pHf- pHi) against pHi was plotted and at zero ΔpH, the ZPC of the adsorbents were recorded.

Fourier transform infrared spectrometry (FTIR)

The surface functional groups and the effect of chloride on the surface functional groups of the adsorbents were characterized by using Bruker Alpha II- FTIR spectrometer in the wavelength of 4000-500 cm^{-1}.

Chloride analysis

Mohr's method of chloride determination was used [22, 23]. The volumetric method involves the titration of chloride solution against standard solution of silver nitrate (AgNO3) in the presence of potassium chromate as indicator. During the titration a white precipitate of silver chloride (AgCl) was formed and at the equivalence point a red brown color of silver chromate appeared.

$$Cl^- + Ag^+ \rightleftharpoons AgCl\,(s)$$

$$2Ag^+ + CrO_4^{2-} \rightleftharpoons Ag_2CrO_4(s)$$

The analysis were carried at neutral pH, as below pH 6 chromate get converted to dichromate and above pH 8 silver get converted to silver hydroxide.

Adsorption kinetics

The adsorption kinetics of unmodified and acid modified biochar were conducted by adding 1.0 g adsorbent in to 100 ml of 100 mg/l chloride solution at neutral pH and shaked at different time intervals of 10, 30, 50, 70, 90, 110, 130, 150, 170, 190 minutes. At each time intervals the biochar was separated and amount of chloride ion in the filtrate was determined as per the procedure described in chloride analysis section. The amount of chloride adsorbed per gram of adsorbent (mg/g) and removal percentage were calculated using the following formula.

$$Q_t = \frac{(C_i - C_t) * V}{\mathrm{m}}$$

$$\text{Removal (\%)} = \frac{(C_i - C_t)}{C_i} * 100$$

Qe: amount of chloride adsorbed per gram of adsorbent at equilibrium (mg/g).

Ce: equilibrium concentration of chloride ion (mg/l).

Ci: initial Concentration of Chloride ion (mg/l).

The adsorptions of nitrate were fitted to Langmuir isotherm and Freundlich isotherm models using the equations.

Langmuir model: $\frac{C_e}{Q_e} = \frac{1}{Q_{max} K_L} + \frac{1}{Q_{max}} * C_e$

Qmax: maximum amount of chloride ion adsorbed (mg/g).

KL: Langmuir constant (L/mg).

RL: is a dimensionless quantity calculated from KL by the equation, Rl = 1/1+Kl Ci) . The parameters of the Langmuir isotherm with RL values 0< RL <1, favours and improved adsorption [30].

Freundlich model: LogQe = log Kf + 1/nlogCe

Kf and n are Freundlich constants (mg/g) (L/mg) 1/n).

RESULT AND DISCUSSION

Characterization with Zero point of Charge (ZPC)

The adsorption of charged ions depends on the zero point of charge of the adsorbent. When pH of the solution is greater than zero point of charge (ZPC), the surface of the adsorbent becomes negatively charged and favors the adsorption of cations, and vice versa [24]. Therefore pH of the solution plays an important role in the adsorption. The zero point charge of unmodified and acid modified Salix alba leaves biochar were recorded to 4.6 and 8.4, indicating modified sorbent could better adsorb negatively charged species up to pH 8.4. Similarly the ZPC of pure and acid modified apricot seed shell biochar were shown to shift from 5.8 to 8.9. Therefore the acid modified biochar shows better adsorption efficiency for negatively charged ions than unmodified biochar at neutral pH.

Fourier transform infrared spectrometry (FTIR)

The IR spectra of Apricot seed shell biochar (ASSB), acid treated, and chloride loaded are shown in figure 4. A prominent peak around 1580-1700 cm-1 indicated the presence of many functonal groups. The peak around 1636 cm−1 is ascribed to (C=O) stretching vibration of aromatic carbonyl group [2]. The peaks around 1690-1700 cm^{-1} indicates stretching vibration of amide group and 1540–1640 cm^{-1} corresponds the existence of carbonyl groups (C=O) and carboxylic acid, while as the absorption peak of methylene in long-chain saturated alkanes (-CH2-) appears at 2838–2843cm^{-1} [15]. The peak around 1600 cm^{-1} also ascribed to C=C stretching of aromatic component and the peak at 1457 cm^{-1} and 1376 cm^{-1} are assigned to –CH2 and –CH3 bending vibrations, respectively [27]. The IR bands in 1300-1000 cm^{-1} are observed and are assigned to C–O or C–O–C stretching in acids, alcohols, phenols, ethers or esters groups [29] There are no prominent changes observed in the spectra on treatment with acid, however loaded with chloride changes the peak pattern in the regions 2400 cm^{-1} and 800-600 cm^{-1}. The various groups on the surface of biochar served for the adsorption of solute.

The FTIR spectra of salix alba biochar is illustrate in figure 5. The peak around 3400-3200 cm^{-1} correspomd to stretching vibration of O-H group could have originated from the alcohols, phenols, and carboxylic acid content, while the absorption peak of methylene in long-chain saturated alkanes appears at 2838–2843cm-1 [15]. The characteristic methyl (C–H) stretching vibration can be seen at 2927 cm^{-1} [28]. The peak around 2346 cm^{-1} correspond to stretching vibration of to C –N triple bond. The band in 1690–1700 cm^{-1} is the stretching vibration band of –CO-N amide groups and 1540–1640 cm−1 corresponds the existence of carbonyl groups (C=O) and carboxylic acid [15]. The peak around 1636 cm^{-1} is ascribed to (C=O) stretching vibration of aromatic carbonyl group [2]. The peak around 1600 cm^{-1} is also ascribed to C=C stretching of aromatic component [27]. The peaks at 1457 cm^{-1}1 and 1376 cm^{-1} are assigned to –CH2 and –CH3 bending vibrations, respectively [27]. The temperature does not cause any appreciable impact on the spectra.

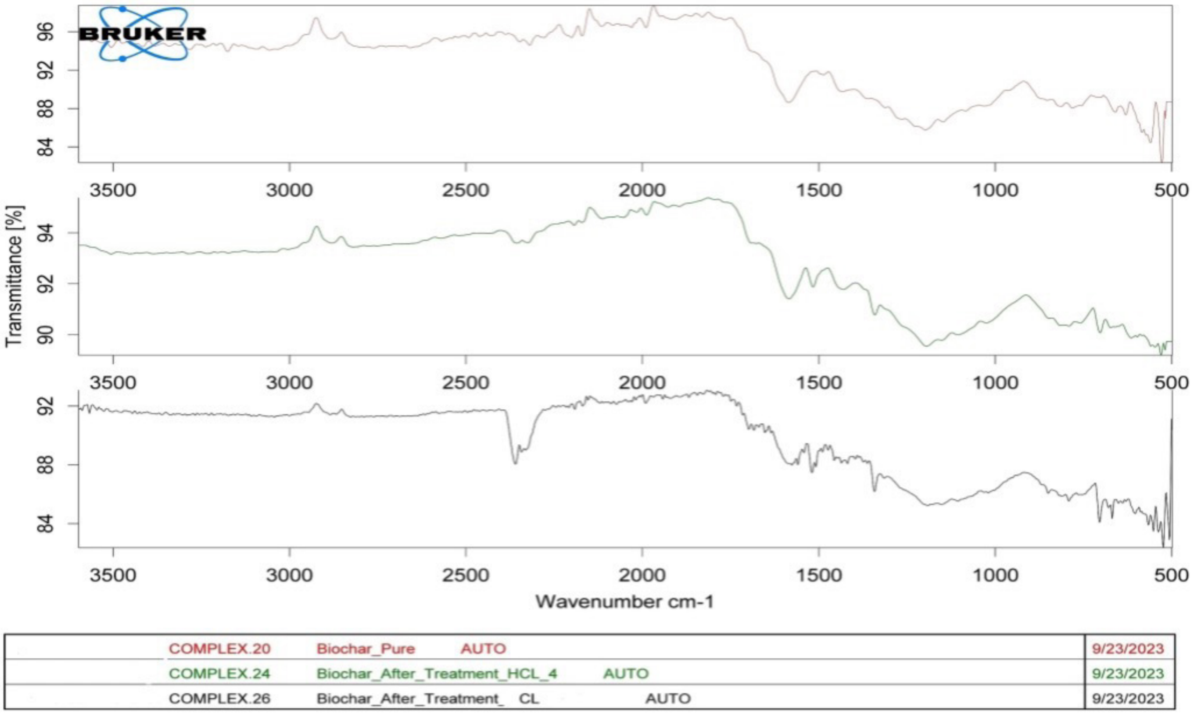

Figure 4. FTIR Spectra of apricot seed shell biochar

Adsorption Kinetics

The rate of adsorption of chloride ion on to the pure and acid modified Salix Alba leaves and Apricot seed shell adsorbent at neutral pH were analyzed for contact time period of 10-190 minute. The pure biochar sorbents have very less adsorption as the surface have negatively charged functional groups which caused repulsion. However acid modified biochar exhibited an enhanced adsorption at neutral pH due to protonation of the surface functional groups and induced surface positive charge. The adsorption slightly faster at the beginning and increases with increasing contact time and attained equilibrium around 150 min. The % removal of chloride ion by the adsorbents are illustrated in figure 6.

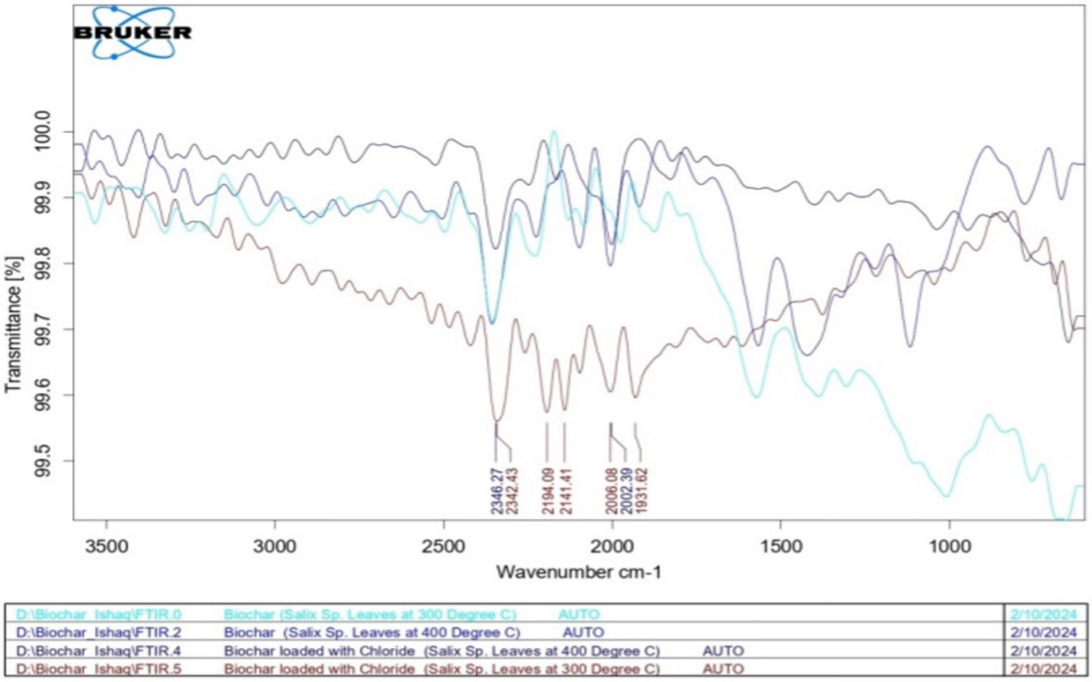

Figure 5. FTIR Spectra of Salix Alba leaves biochar

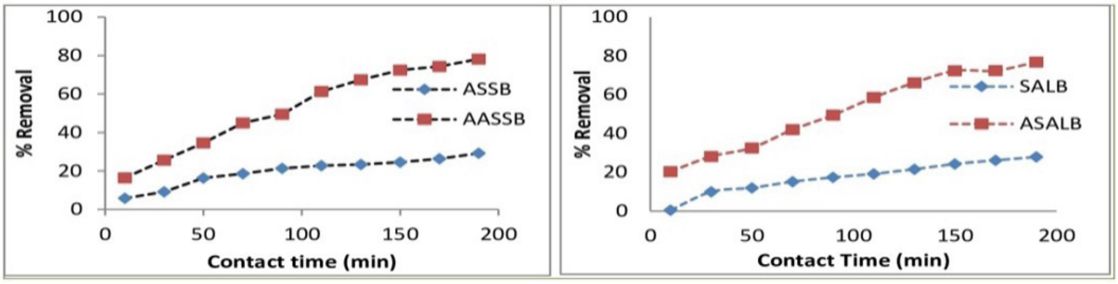

Figure 6. Effect of Contact time.

Table 1. Relevant parameters of adsorption kinetics

Adsorbents	Pseudo 1st order kinetic				Pseudo 2nd order kinetic		
	Qecal (mg/g)	K1	R2	Qe exp(mg/g) (at contact time of 150 min)	Qecal(mg/g)	K2	R2
SALB	2.948	0.0236	0.9191	2.62	3.637	0.003	0.9548
ASALB	8.800	0.0217	0.8352	6.88	10.822	0.001	0.8915
ASSB	2.856	0.017	0.9605	2.74	3.898	0.003	0.9704
AASSB	7.446	0.0185	0.9006	6.82	9.813	0.0013	0.9495

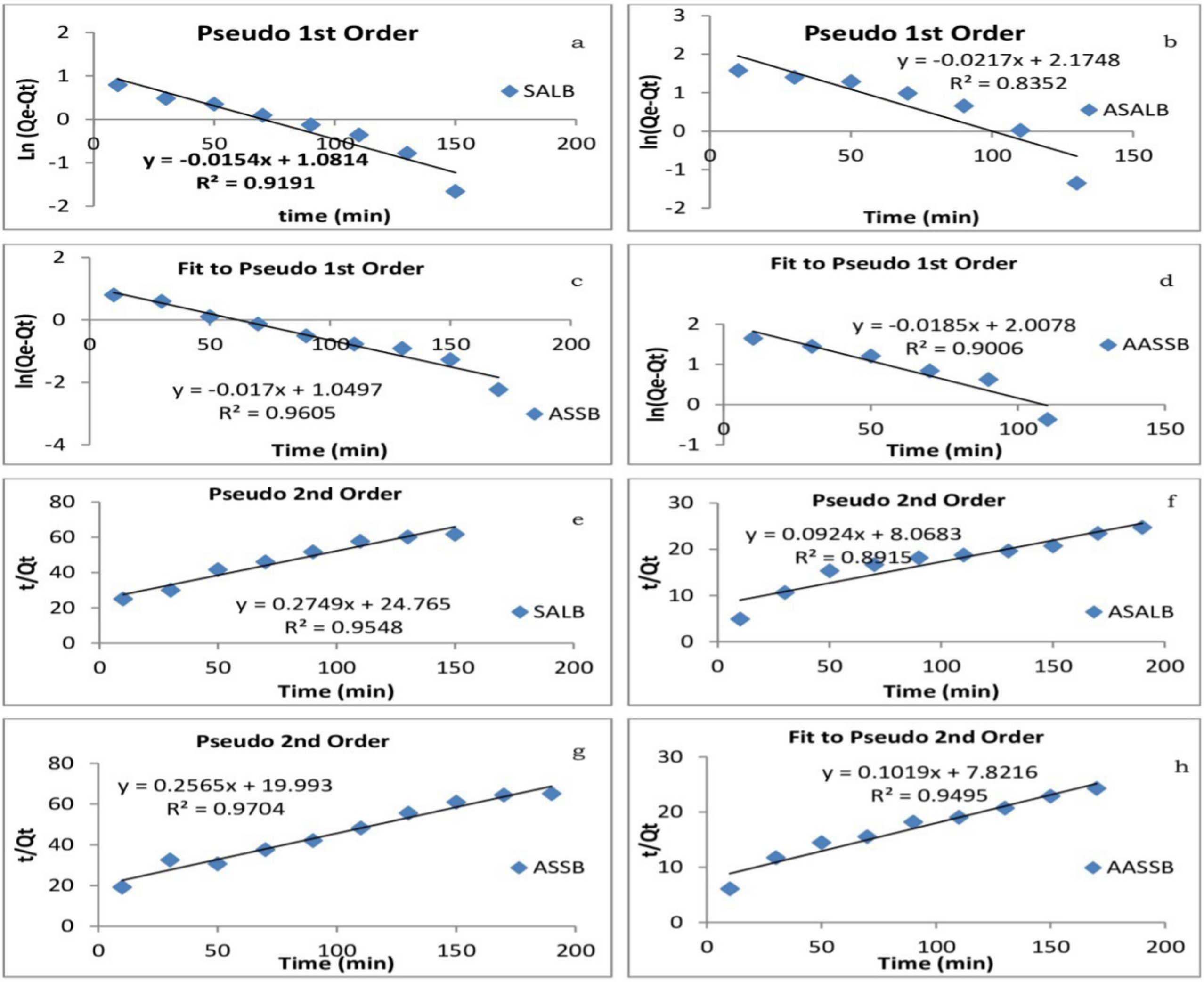

Figure 7. Fit to adsorption kinetics- Pure (a,c,e,g) and activated (b,d,f,h) adsorbents.

Adsorption equilibrium

The % removals of chloride ion by the pure and acid activated biochar were analyzed at different dosage of adsorbents (0.5-2 g), using 100mg/L of chloride solution at neutral pH and the result is depicted in figure 8. The adsorption increases with dosage at neutral pH due to increasing available surface sites of adsorbent and more and more adsorbate get adsorbed.

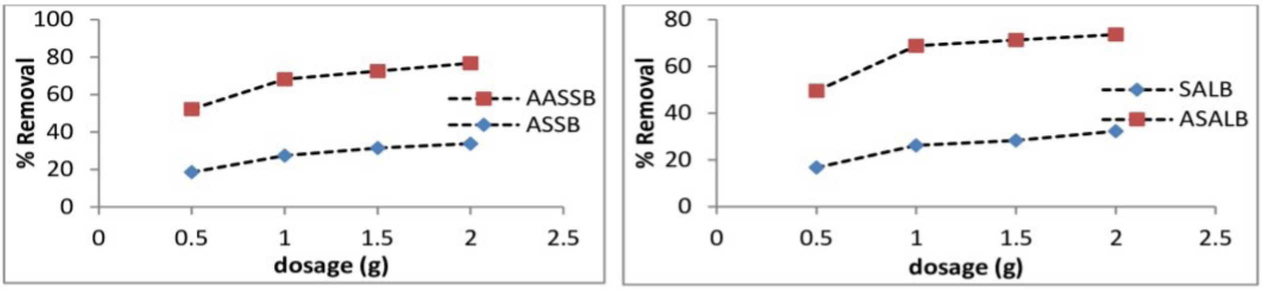

Figure 8. Effect of dosage on % removal of ion

The experimental data obtained by using 100 mg/l at different adsorbent dosage of 0.5,1,1.5 and 2g and contact time period of 150 min at neutral pHwere simulated by using ms excel and fitted to Langmuir and Freundlich isotherm models (figure 9). The Langmuir maximum adsorption capacities (Qmax) of pure and acid activated Salix Alba leaves biochar were found to be 5.537 mg/g and 22.988 mg/g while that of pure and acid activated Apricot seed shell were found to be 6.373 mg/g and 25.839 mg/g respectively.

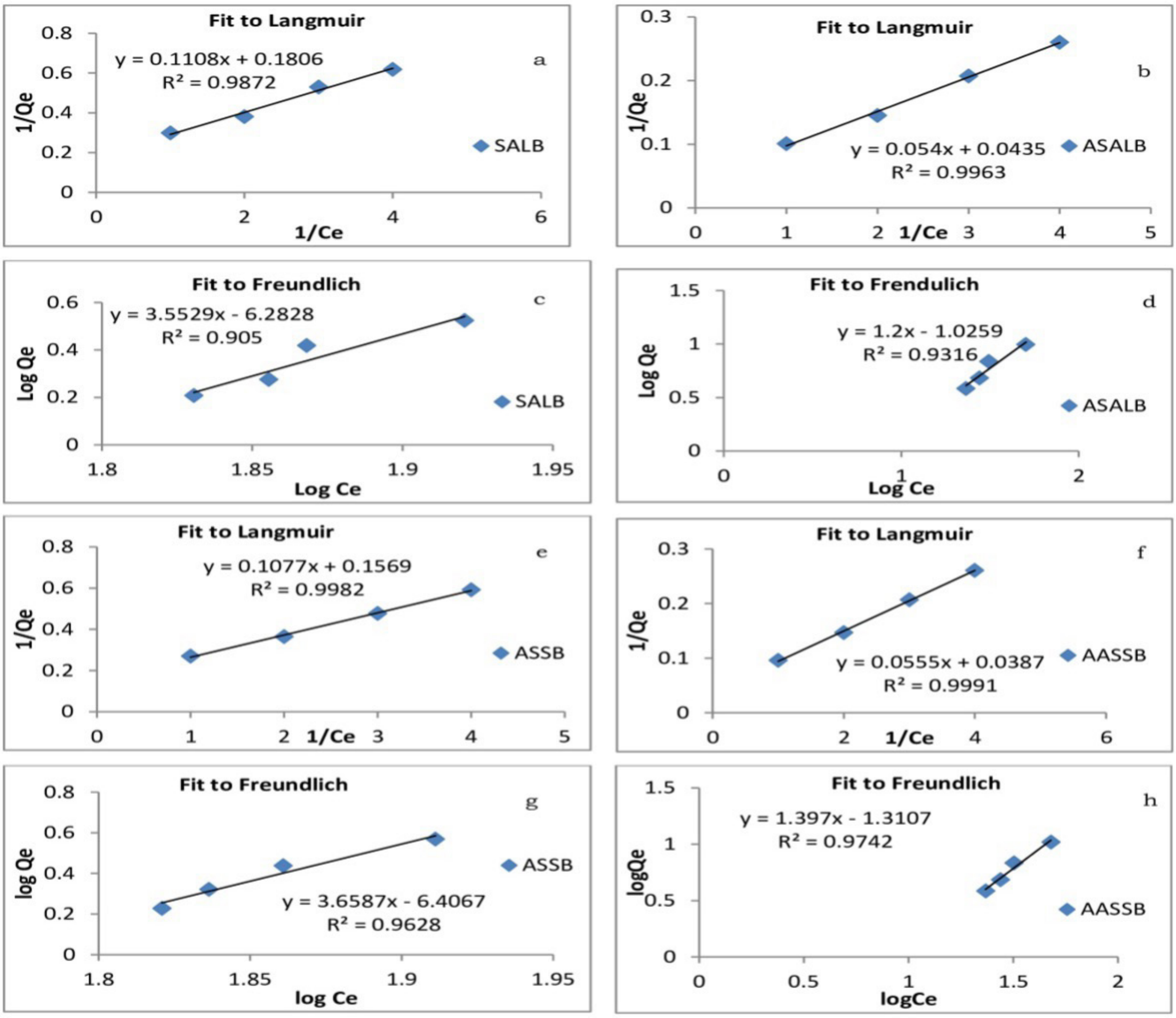

Figure 9. Fit to isotherm models- Pure biochar (a,c,e,g), Activated biochar (b,d,f,h)

The relevant parameters of Langmuir and Freundlich isotherm models are shown in table 2. The coefficient of determination (R2) for Langmuir and Freundlich isotherm model for SALB and ASALB were 0.9872, 0.9050, 0.9963 and 0.9316 respectively, while as for ASSB and AASSB were 0.9982, 0.9628, 0.9991 and 0.9742 respectively. The Langmuir model has two dimensionless constants- RL and KL values. The RL values indicate the favorability, if ($0 < RL < 1$), unfavorable, if ($RL > 1$), linearity, if ($RL = 1$), or irreversibility, if ($RL = 0$) of a given system [24, 30]. The RL was found to be less than 1 indicated a favourable sorption and relatively showed a better compliance with Langmuir isotherm model.

Table 2. Relevant parameters of adsorption isotherms

Adsorbents	Langmuir isotherm				Freundlich isotherm		
	Qmax (mg/g)	K_L	R_L	R^2	n	K_f	R^2
SALB	5.537	1.563	0.012	0.9872	0.281	533.78	0.9050
ASALB	22.98	0.805	0.024	0.9963	0.833	2.789	0.9316
ASSB	6.373	1.456	0.013	0.9982	0.273	605.89	0.9628
AASSB	25.84	0.697	0.278	0.9991	0.715	0.269	0.9742

CONCLUSION

In light of the global water pollution crisis, scientists/researchers are focusing on developing strategies and technology for preventing and reducing contaminants in water ecosystems. In this study, biochar produced from locally available agricultural wastes (Apricot seed shell and Salix Alba leaves) were used to investigate sorption capacities towards anionic contaminant (chloride ions) using a batch adsorption model. The experimental results showed that acid activated biochar are more effective for removing anion (chloride ion) from water than pure biochar at neutral pH. The Langmuir maximum adsorption capacity (Qmax) of activated Salix Alba leaves biochar recorded to 22.98 mg/g, which is significantly greater than the 5.537 mg/g of the unmodified biochar of Salix Alba leaves. Similarly the Langmuir maximum adsorption capacity (Qmax) of activated Apricot seed shell biochar becomes 25.84 mg/g, which is much greater than the unmodified Apricot seed shell biochar's value of 6.373 mg/g. The experimental data were simulated and fitted to the Langmuir and Freundlich isotherms, and the results were in a close consistent with the Langmuir isotherm. The adsorption kinetics experimental data were also simulated using MS Excel and fitted to pseudo first and second order kinetics and the results exhibited a close agreement with pseudo 2nd order kinetic, showing the involvement of surface contact diffusion, electrostatic interaction, and ion exchange in the adsorption process. Both agricultural wastes have little utility in the region, and their conversion to potential sorbents incurs a minimal capital cost. As a result, these agricultural wastes might be utilized into an effective and sustainable sorbents for the removal of anionic pollutants from water and wastewater.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available within the article and more raw data are available on request from the corresponding author.

AUTHORS CONTRIBUTION

Mohd Ishaq conceived and designed the study. He conducted the experimental work, simulated data, compiled and drafted the manuscript. RC Chippa provided critical feedback, participated in interpretation of data and drafting the manuscript. Anupama Sharma reviewed selected literature. Gh. Ali supported in experimental works.

CONFLICT OF INTEREST

The authors have no conflict of interest to declare in respect of content and publication of this article. Also there is no financial interest to report.

USE OF AI FOR WRITING ASSISTANCE

Not declared.

ETHICS

The authors declare that there is no ethical issue with the publication of this article.

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