

Research

Removal of lead in water by potassium hydroxide-activated biochar developed from *Syzygium cumini* stem

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Abstract

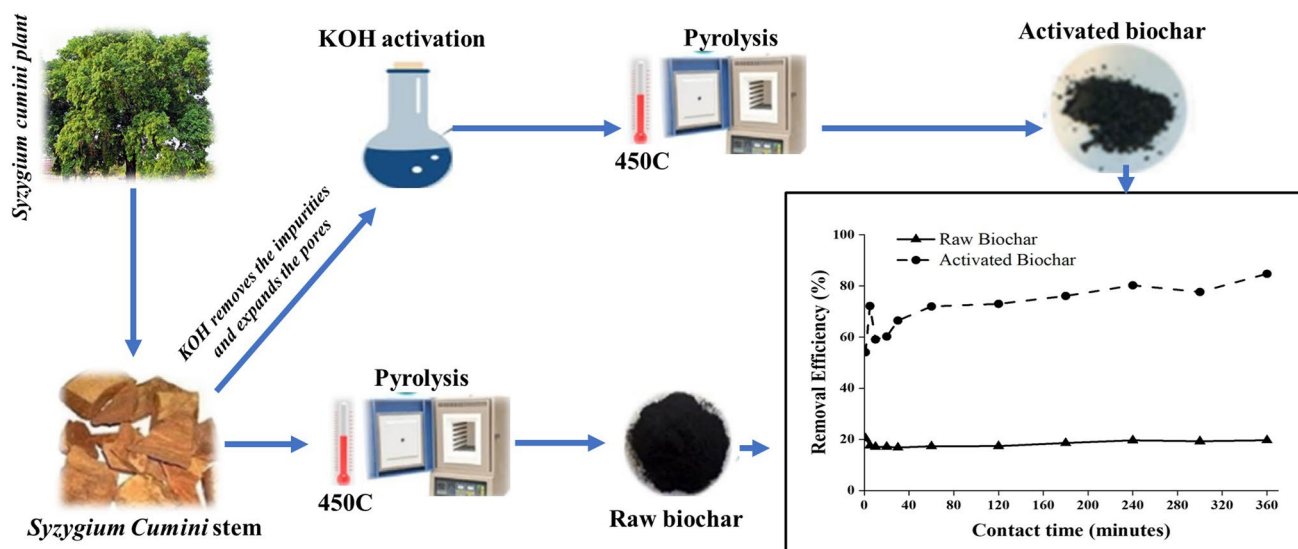
Lead (Pb) contamination in water poses a significant threat to public health across the globe which requires effective remediation strategies. The main objective of this study is to achieve a removal efficiency of Pb(II) ions from aqueous media using activated *Syzygium cumini* (java plum) stem biochar. It was prepared by slow pyrolysis at 400 °C after treating the biomass with potassium hydroxide for activation. These biochars were characterized thoroughly (SEM, SEM–EDX, TEM, FTIR, XRD, elemental analyses, and SBET) to conduct a set of batch experiments. The effect of several parameters such as pH, temperature, contact time, adsorbent dosage, initial lead concentrations, and co-existing ions were explored. The decrease in starting lead concentrations from 50 to 10 mg L^{−1} resulted in improved removal efficiency. The removal effectiveness of activated biochar was 97%, but non-activated biochar was just 19%. Lead adsorption increased considerably as pH increased from 3 to 5. Further, the activated biochar was optimized through various isotherms models, kinetic equations, and thermodynamic analysis. It was observed that the pseudo-second-order kinetic model and Temkin isotherms were the best-fitted models that identify the mechanism of chemisorption and monolayer sorption. According to this study, activated biochar is a promising biosorbent for removing lead from an aqueous solution.

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Graphical Abstract



Keywords Lead removal · *Syzygium cumini* biochar · Sorption · Isotherms

1 Introduction

Water pollution is a global environmental problem where more than 2.1 billion people do not have access to clean drinking water [1]. The water is getting polluted due to an increase in various pollutants in the surface and subsurface systems [2]. Among various contaminants, heavy metals are one of the major contaminants generated from either natural or anthropogenic activities causing contaminants to soil and existing water bodies [3, 4]. Among heavy metals, lead ion [Pb(II)] is one of the priority pollutants as it is considered a contaminant of emerging concern and ranked second (after arsenic) according to the Agency for Toxic Substances and Disease Registry (ATSDR). The Bureau of Indian Standards (BIS), World Health Organisation (WHO), and American Water Works Association (AWWA) have established 0.05 mg L^{-1} of lead as the ideal limit for drinking water [5]. Lead smelting, battery recycling, and coal combustion are prominent sources of lead pollution [6]. It damages the liver, kidney, bones, and reproductive system once it enters the human body through the food chain [7]. Lead can cause severe health problems for children such as delaying their mental or physical development and impairing their ability to pay attention and learn [8]. Therefore, over the last few decades, the effective removal of lead from polluted water has become an important research subject.

Lead removal technologies include ultrafiltration, ion exchange, membrane filtration, electrocoagulation, membrane separation, adsorption, and flocculation [9–12]. Among them, adsorption is one of the cost-effective techniques for the removal of lead or other contaminants from an aqueous solution [13–15]. Further, the adsorption process is simple, flexible, environment friendly, easy, capable of regeneration, and lacks toxic by-products [14, 16, 17]. Various adsorbents such as activated carbon, graphene oxide, bentonite, zeolite, and biochar have been applied for the removal of lead from aqueous solution [18–21]. The studies highlight that the removal efficiencies of Pb(II) are dependent on adsorbent dosages, initial Pb(II) concentration, pH, and contact time (Table 1). Among these adsorbents, biochar has been used as a feasible and alternative solution for the elimination of lead [22–25].

The development of biochar for the purification of water is a comparatively new practice [5, 26–29]. Biochar, a solid material enriched in carbon, is obtained upon the pyrolysis of biomass through slow pyrolysis, fast pyrolysis or gasification methods [30–32]. It possesses unique physiochemical features, such as high pore volume, big surface area, and rich functional surface groups [33]. This material is an effective, durable, and eco-friendly solution for the removal of contaminants from aqueous solution [34–36]. In pure form, biochar may have a lower adsorption capacity compared to traditional adsorbents [37]. Recently, researchers have enhanced the biochar properties, adsorption capacity, and metal

Table 1 Pb(II) removal efficiency using common adsorbents and activated biochar

Adsorbent	Pb(II) removal efficiency (%)	Adsorbent dose (g/L)	pH	Initial Pb(II) concentration (mg/L)	Contact time (min)	References
KOH activated biochar	85–97	0.5–3	3–7	10–50	5–360	This study
Biochar	90	2	2	400	3600	[25]
Exothermic padding waste (activated carbon + Fe ₂ O ₃ + vermiculite)	96.4	1	5	100	1400	[76]
Spent biological activated carbon	99	0.2	5–7.5	5	300	[21]
Clay + Fe ₂ O ₃ composite	99.8	3	5	800	240	[77]
Zeolite	99.6	2.5	7.3	40	60	[20]
nZVI + kaolin	98	2	4.5–6.5	50	10	[78]
Polyacrylamide coffee ground	82	20	4	103	1440	[79]
Chitosan	89	1.9	4.5	4	76	[19]

remediation efficiency through modifications, metal impregnation, magnetization, and/or activation [24, 35, 38–41]. The surface area of the biochar, pore structure, and functional groups associated with biochar enhanced after the modifications [42]. The adsorption of lead from aqueous solution by activated carbon [21, 43] and by agricultural waste-derived biochar have been reported by various researchers as well [25]. However, research on biochar modifications through a variety of techniques is still an emerging area of study [37]. Researchers are increasingly recognising the benefits of biochar modifications through the activation process. Activation can be done either through physical activation where biochar can be activated at a high temperature (> 900 °C) in the presence of steam or CO₂ [44], or by chemical activation under which biochar can be activated through various agents such as potassium hydroxide [45], potassium carbonate (K₂CO₃), zinc chloride (ZnCl₂), and phosphoric acid (H₃PO₄) [46]. Specifically, chemical activation with KOH has been widely developed for the synthesis of activated biochar by various researchers [47–49]. Biochar developed from tobacco stems, celery stalks, and canola straws has high efficiency in removing lead from aqueous solution [50, 51]. Unfortunately, there is currently a lack of information on the effect of biochar washing on the elimination of released compounds from biochar after chemical treatment.

This study aims to evaluate the efficiency of the adsorption of lead onto activated biochar obtained from *Syzygium cumini* stem (java plum) upon a slow pyrolysis process. The present study aims to assess the adsorption of Pb(II) onto low-cost activated biochar which was synthesized in a single-step process. The primary goal of this research is to (a) analyse the physiochemical properties of both raw and activated biochar, and (b) use batch experiments to evaluate Pb(II) removal efficiency under various conditions such as pH, initial concentration, biochar dose, contact time, temperature, and co-existing anions. Furthermore, adsorption isotherms, kinetics, and thermodynamic behaviour are used to estimate the Pb(II) removal mechanisms using activated biochar in this study.

2 Materials and methods

2.1 Reagents and equipment

Analytical or guaranteed reagents are used during the experiments which were purchased from either Merck or unless otherwise specified. Pb(II) stock solution was prepared by dissolving [Pb(NO₃)₂] into de-ionized water. The pH 7 was achieved using either 0.1 M HNO₃ or 0.1 N NaOH using a pH meter (Hanna instruments). A static water bath (BUCHI, B-461) was used for adsorption studies. A vortex mixer (Scientific Industries, T3-1280) was used to stir the samples.

2.2 Feedstock and biochar preparation

The *Syzygium cumini* (java plum) stem part was collected from the local market of Rajgir, India (25° 00' 44.4" N 85° 24' 37.1" E). A small piece of dried stem was cut and washed with ultra-pure water 4–5 times, and soaked in ultra-pure water for 72 h to remove the color and impurities from the biomass. The prepared biomass was oven-dried for 72 h

at 75 °C to remove moisture as much as possible and was kept in a container after cooling. Further, the pyrolysis experiments were conducted in a fixed-bed reactor (muffle furnace). The *Syzygium cumini* stem biomass was pyrolyzed in the muffle furnace at 400 °C for 2 h. The *Syzygium cumini* stem biochar (SCB) was soaked in ultra-pure water for 24 h and then washed properly to leach color, and then oven-dried at 75 °C for further adsorption studies.

2.3 Preparation of activated *Syzygium cumini* stem biochar (ACB)

In this study, potassium hydroxide chemical was selected for the activation of biochar as a result of a large improvement in the metal sorption [52]. The schematic diagram for the development of activated biochar is shown in Fig. 1. The activated *Syzygium cumini* stem biochar (ACB) was prepared through single-step activation process according to the method described by Basta et al. [53]. Briefly, 2 g of oven dried *Syzygium cumini* stem was soaked into 250 mL of 2 M KOH solution and stirred for 12 h at 120 revolutions per minute (rpm). The resulting biomass was repeatedly washed with ultra-pure water and oven dried at 75 °C for 24 h. The activated *Syzygium cumini* stem biomass was pyrolyzed in the muffle furnace at 400 °C for 2 h. The ACB was soaked in ultra-pure water for 24 h, washed properly to leach color, and oven-dried at 75 °C for further adsorption studies.

2.4 Proximate and ultimate analysis

Total C, N, and H contents were measured with EUROM EA3000 elemental analyzer. The moisture content, volatile matter, and ash content were determined according to the D1762-84 method. Feedstock and biochar moisture content were estimated by oven drying for 2 h at 105 °C. The samples were heated to 950 °C for 11 min (covered crucibles) to determine the volatile matter and at 750 °C for 2 h (uncovered crucible) for the ash content [54]. The results are reported in weight percent on an ash basis.

2.5 Characterization of raw and activated biochar

The morphology of raw and activated biochar was investigated by Scanning electron microscopy (SEM) (Make: deMicro, Orchid, India), at a 20,000-V accelerating voltage. All samples were Au-coated before mounting on a copper stab with double-stick carbon tape. Elemental sample compositions were analyzed to a ~2–3 µm depth by SEM/EDX (EDX, EDAX Inc., USA). Similarly, the structural and chemical characterization of raw and activated biochar was carried out using Fourier transform infrared (FTIR) spectra (Agilent Technologies, USA) and X-ray diffraction (Rigaku Corporation, Japan),

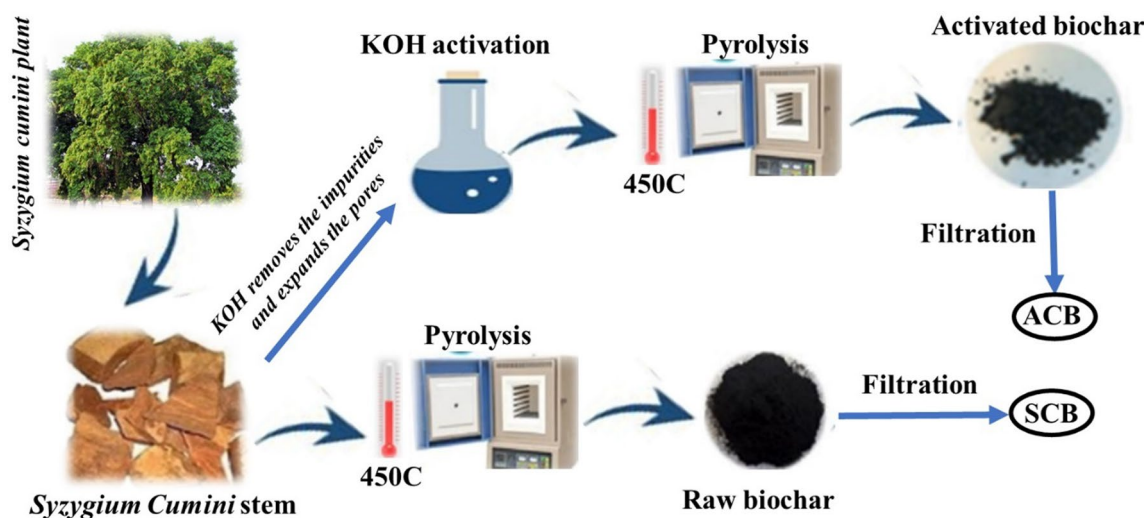


Fig. 1 The schematic development of SCB and ACB from the pyrolysis process of *Syzygium cumini* stem biomass

respectively. BET surface area for SCB and ACB was determined by a micrometric ASAP 2020 surface area analyzer. The samples (0.15 g) were degassed at 250 °C for 12 h at $> 10^{-3}$ Torr.

2.6 Preparation of stock solution and analytical method for Pb(II)

Pb(II) (1000 mg L⁻¹) stock solution was obtained by dissolving lead nitrate salt [Pb (NO₃)₂] into ultra-pure water. 0.1 M HNO₃ and 0.1 N NaOH were used to adjust the pH. The concentration of Pb(II) was measured by UV–VIS spectrophotometer at 212 nm (Agilent Technology, ALIGENT CARY100) [55, 56]. The proposed wavelength for Pb(II) analysis using Spectrophotometer is based on calibration and validation processes using preliminary experiments.

3 Results and discussion

3.1 Biochar pyrolysis

Based on the weight of the initial biomass, slow pyrolysis produced 27 weight percent of stem biochar from *Syzygium cumini*. Table 2 provides proximate analyses of feedstock, SCB, and ACB. ACB consists of higher carbon content and hydrogen content than SCB [C (70.27 vs 45.70%) and H (1.99 vs 1.92%)]. Biochar properties are generally dependent on the nature of feedstock, temperature, residence time, and reactor type. For example, the tea residue and paddy straw biochar were prepared at 650 °C and 550 °C, respectively, contributing the highest carbon content in tea residue biochar. The molar H/C ratio describes the degree of char carbonization [57, 58]. The H/C molar ratio for *Syzygium cumini* stem was 0.12, whereas those for SCB and ACB were 0.02 and 0.03, respectively (Table 2). Biochar's reduced H/C molar ratio contained low organic wastes (like cellulose) and a high carbonization value [57]. Because it indicates the polar-group content produced from carbohydrates, the molar ratio of oxygen to carbon (O/C) is employed as an indication for surface hydrophilicity [57]. The molar O/C ratio was 1.11 for *Syzygium cumini* stem and 0.20 and 0.32 for SCB and ACB, respectively. SCB and ACB surface areas were found as 145.11 and 168.98 m² g⁻¹, respectively (Table 2). Pyrolysis at a lower temperature (400 °C) resulted in SCB with a lower surface area and higher ash content. Biochar surface area normally increases with a higher pyrolysis temperature [59, 60].

3.2 Biochar characterization

The SEM micrographs of the SCB and ACB showed their irregularly shaped structures (Fig. 2). The SEM images of SCB showed microscopic sizes of the particles with irregular shapes and sharp edges (Fig. 2A, B). The ACB were seen as larger particles and appeared as an agglomerated form of pristine biochar. (Fig. 2C, D). The XRD spectra of SCB and ACB are presented in Fig. 3. The spectra showed a broad hump region between 2θ of 18–28° corresponds to the presence of amorphous carbon [61]. This represents crystal plane index C(002) which is formed due to azimuthal and

Table 2 Elemental and proximate analysis of biomass feedstock and biochar

Sample	Elemental composition ^a (wt %)				<i>S</i> _{BET} (m ² /g)	Proximate analysis ^a				Biochar yield (%)	Atomic ratio		
	C	H	N	O*		Ash (%)	Volatile matter (%)	Moisture content (%)	Fixed carbon (%)		C/N	H/C	O/C
<i>Syzygium cumini</i> stem	41.12	4.94	6.01	45.65	–	2.22	80.90	5.11	10.67		6.84	0.12	1.11
SCB	71.27	1.99	4.04	14.44	145.11	5.22	3.93	1.89	88.90	27	17.64	0.02	0.20
ACB	72.91	2.78	1.36	23.89	166.98	6.29	1.71	1.59	90.40		53.61	0.03	0.32

Note: To maintain uniformity, a number (n = 5) of biochar samples were randomly picked from thoroughly mixed (using Quadrate method) bulk biochar. These samples were then mixed well again using quadrate method at least 10 times then a small sample size is picked for analysis

SCB: *Syzygium cumini* stem biochar; ACB: activated *Syzygium cumini* stem biochar

^aOn the dry basis

Fig. 2 Scanning electron micrographs (SEM) of **a, b** SCB and **c, d** ACB. Microscopic particle size with irregular shapes and sharp edges for SCB are visible, while agglomerations with large particles are present in ACB

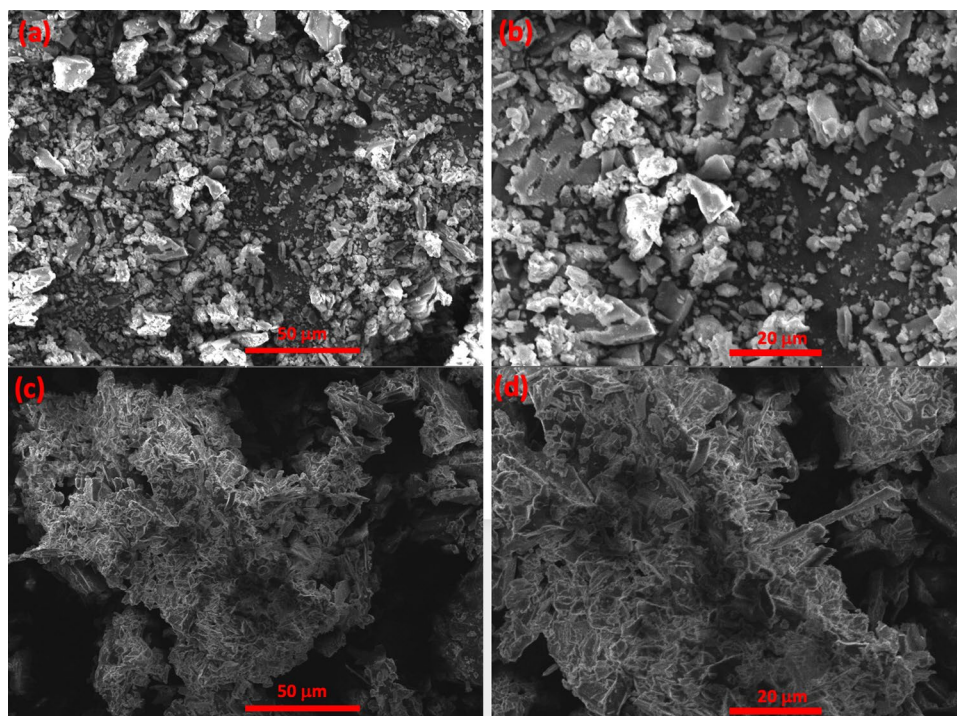
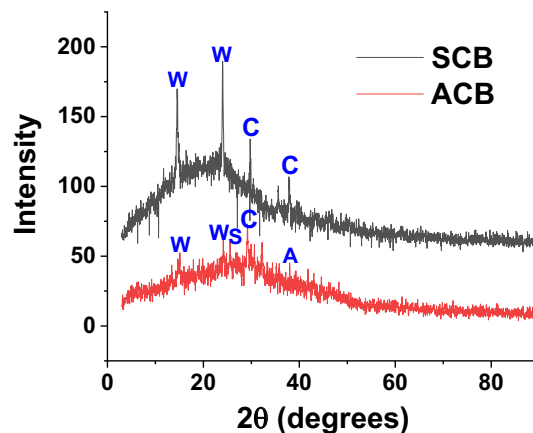


Fig. 3 XRD spectra of the developed SCB and ACB. The intensity of SCB is higher than ACB with broad humps between 18 and 28°, sharp peak at 37.7°, and a small hump around 43°



parallel orientation of partially carbonized aromatic clusters [62]. Another small hump around 2θ of 43° represents the C(100) plane of carbonized aromatic carbon planes [62]. Further, SCB showed sharp peaks at 2θ of 14.9 and 24.13 corresponds to whewellite, and 2θ of 29.65 and 37.7 corresponds to the presence of calcite (Fig. 3). ACB has lowered the strength of the whewellite and calcite peaks. Submerging the biochar in KOH activates it, causing calcite and whewellite to dissolve and potassium-containing minerals such as sylvite to emerge. This tendency is also consistent with the EDX data (Fig. 4), which revealed the presence of calcium in SCB and potassium in ACB.

Broad peaks of -OH stretching bands from organic or inorganic components observed from 4000 to 3000 cm^{-1} are seen in the FTIR spectra of SCB and ACB (Fig. 5). Small peaks in SCB and ACB at 2854 cm^{-1} and 2927 cm^{-1} , respectively, are attributed to the C-H symmetric stretching vibration in organic carbon. The presence of carbon dioxide is the cause of sharp peaks in SCB and ACB at 2360 cm^{-1} . The normal range of 2000–2400 cm^{-1} is associated with triple bond stretching of $\text{O}=\text{C}=\text{O}$, $\text{-C}\equiv\text{C-}$ and $\text{-C}\equiv\text{N}$. According to Reig et al. [63], there are multiple asymmetric peaks in the wide range of 1926–1314 cm^{-1} , with the peak maxima at 1685 cm^{-1} in SCB and ACB. These comprise contributions of complex conjugated C=C systems in the samples as well as C=O stretching of different functional groups in ketones, carboxylic acids, esters, and anhydrides. The aromatic ring's C=C bond stretching is responsible for the peak observed at 1560 cm^{-1} in both SCB and ACB [62]. Reig et al. [63] ascribed the -C-O bond to the SCB peak at

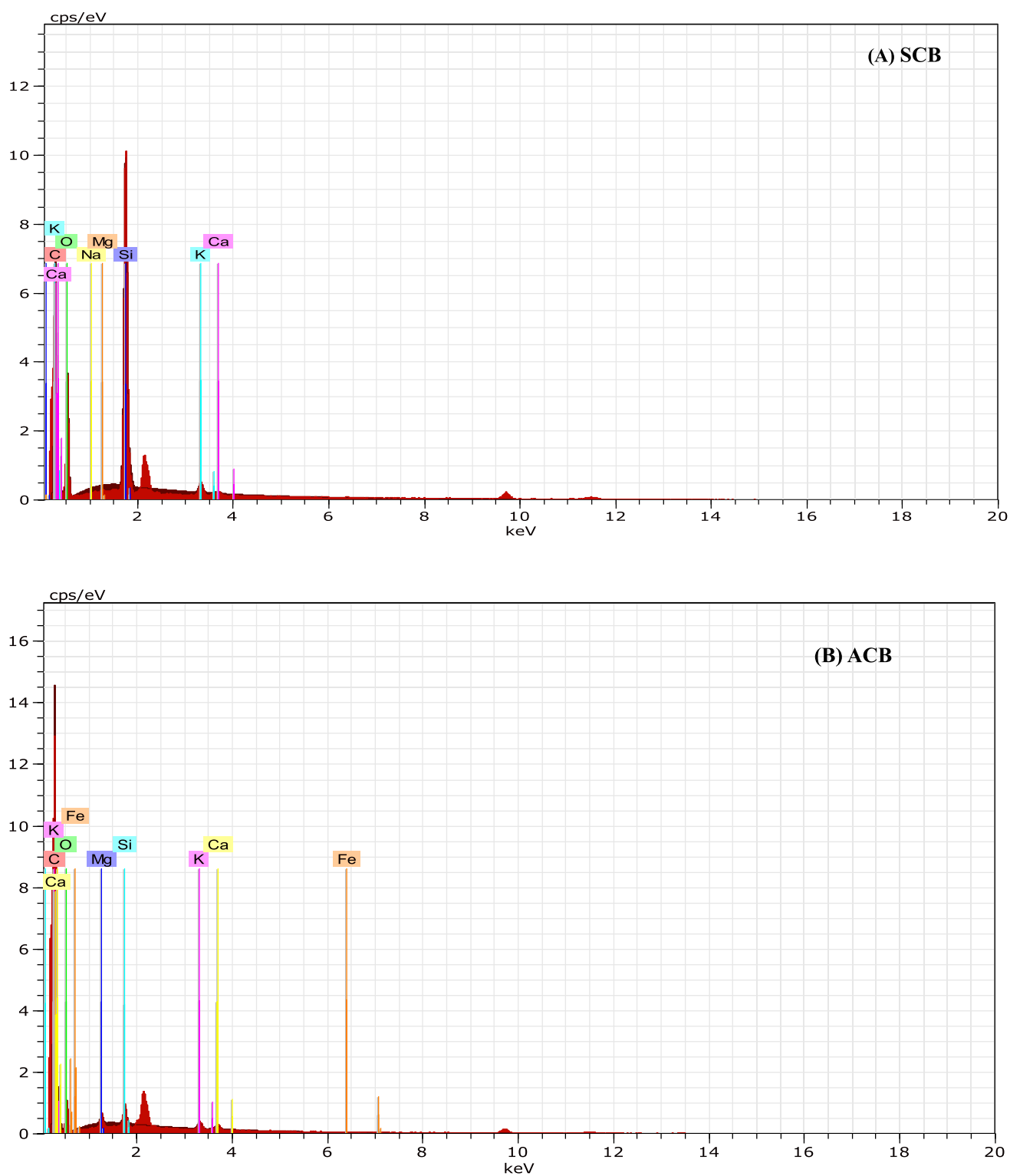
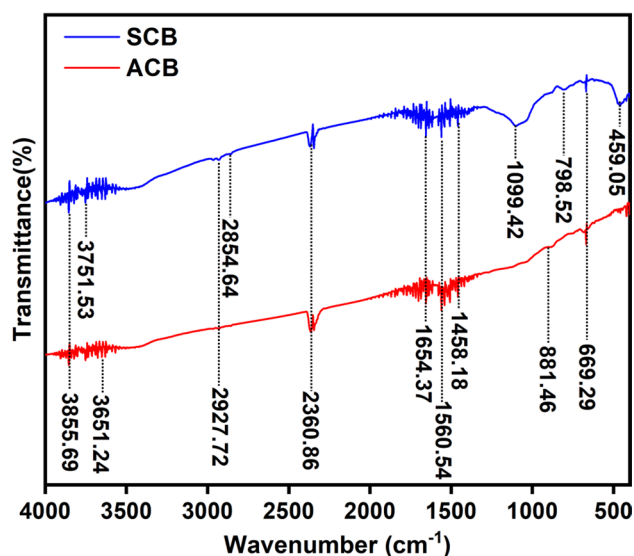


Fig. 4 EDX spectra of **A** SCB and **B** ACB indicated the presence of Ca, Mg and K including other minerals

Fig. 5 FTIR spectra of the developed SCB and ACB. The $-\text{OH}$, $\text{C}-\text{H}$, $\text{O}=\text{C}=\text{O}$, $-\text{C}\equiv\text{C}-$, $-\text{C}\equiv\text{N}$, and $\text{C}=\text{O}$ are present at different wavenumbers including symmetric stretching vibrations of $\text{Si}-\text{O}-\text{Si}$ and $\text{Si}-\text{O}$ at low wavenumbers



1099 cm^{-1} . The carbonate $-\text{C}=\text{O}$ stretching found in calcite is the cause of the band at 881 cm^{-1} in ACB biochar [63]. According to Li et al. [64], the 'asymmetric bending vibrations' of $\text{Si}-\text{O}-\text{Si}$ and the 'symmetric stretching vibrations' of $\text{Si}-\text{O}$ are responsible for the peaks in the ranges 798 cm^{-1} to 669 cm^{-1} and 459 cm^{-1} , respectively.

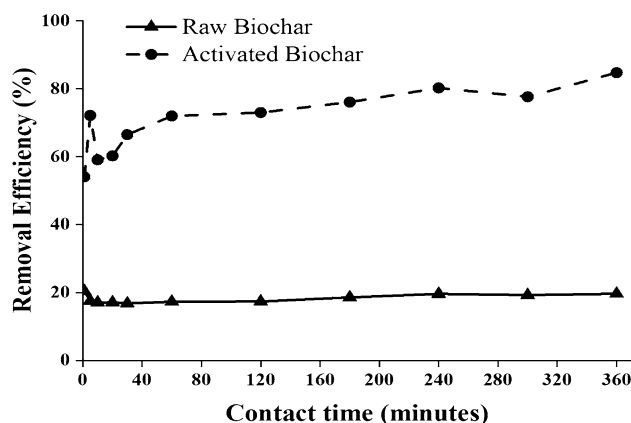
3.3 Effect of contact time on Pb(II) sorption

Removal efficiency (%) of Pb(II) using raw and activated biochar was studied as a function of contact time (360 min). The sorption experiments were performed for SCB and ACB at $\text{pH } 7.0 \pm 0.1$ with an initial Pb(II) concentration of 50 mg L^{-1} and biochar dosage of 0.5 g L^{-1} at temperature 302 K (Table 3). It was inspected that the removal efficiency (%) of Pb(II) is high and increased with increase in contact time in an aqueous solution. The sorption of Pb(II) with a concentration of 50 mg L^{-1} on ACB is more than SCB (Fig. 6). The rapid increase in removal efficiency for ACB at an early stage occurs because of the existence of vigorous sites. The removal efficiency also enhances the sorption capacity due to instant mass transfer. Pb(II) removal efficiency increased from 54 to $\sim 85\%$ for ACB whereas, SCB shows negligible change in removal efficiency ($\sim 20\%$) after 360 min (using equation S1). The sorption capacity of Pb(II) is 1.97 mg g^{-1} over the raw biochar and 8.47 mg g^{-1} for

Table 3 Experimental conditions for sorption of Pb(II) with *Syzygium cumini* stem biochar (SCB) and activated *Syzygium cumini* stem biochar (ACB) through batch experiment of contact time 360 min

Exp. no	Adsorbent material	Adsorbent dosage (mg/L)	Initial Pb(II) conc. (mg/L)	pH	Temperature (K)
1	SCB	0.5	50	3	302
2	SCB	0.5	50	5	302
3	SCB	0.5	50	7	302
4	ACB	0.5	10	5	302
5	ACB	0.5	20	5	302
6	ACB	0.5	30	5	302
7	ACB	0.5	40	5	302
8	ACB	0.5	50	5	302
9	ACB	1	50	5	302
10	ACB	1.5	50	5	302
11	ACB	2	50	5	302
12	ACB	2.5	50	5	302
13	ACB	3	50	5	302
14	ACB	0.5	50	5	310
15	ACB	0.5	50	5	320

Fig. 6 Effect of contact time on removal efficiency of Pb(II) using SCB and ACB at pH=7.0±0.1, initial Pb(II) concentration = 50 mg L⁻¹, biochar dosage = 0.5 g L⁻¹, temperature = 302 K



activated biochar (using equation S2). The increase in removal efficiency for activated biochar might be ascribed to the electrostatic force of attraction between Pb(II) ions and active site availability on biochar surfaces and large surface area [24, 65].

3.4 Effect of pH on sorption and Pb(II) removal mechanism

The effect of Pb(II) sorption on SCB and ACB is shown in Fig. 7. The pH effect of Pb(II) on SCB and ACB was studied at a pH range of 3–7. To understand the effect of pH on the removal efficiency of Pb(II), the sorption experiments were performed over SCB and ACB at varying pH of 3, 5, and 7 with an ‘initial lead concentration’ of 50 mg L⁻¹, biochar dosage of 0.5 g L⁻¹, and 302 K temperature in an aqueous solution. The maximum sorption appeared at pH 5. Sorption of Pb(II) increased consequently as the pH increased from 3 to 5 and became constant from pH 5 to 7. However, the maximum removal of Pb(II) occurred at pH 3 (~46%) for SCB and at pH 5 (~86%) for ACB, respectively (using equation S1). The sorption capacity of Pb(II) over SCB at pH 3 was 4.64 mg g⁻¹ and 5.74 mg g⁻¹ over ACB (using equation S2) at a metal concentration of 50 mg L⁻¹ and an adsorbent dose of 0.5 g L⁻¹. At high pH values, Pb(II) removal over ACB sorption has been inhibited as a result of competition between hydrogen and metal ions, due to which the metal cations had been restricted as an outcome of repulsive force. This indicates that an ion-exchange mechanism is dominant for the sorption process. At pH 7.0, Pb(II) ions can be solvated, hydrolyzed, and polymerized into insoluble hydroxide/oxide species, where precipitation can succeed as shown in Eqs. (1–3) [66], given below:

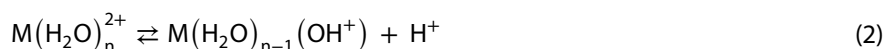


Fig. 7 Effect of pH on the removal efficiency of Pb(II) over SCB and ACB at initial lead concentration = 50 mg L⁻¹, contact time = 360 min, biochar dosage = 0.5 g L⁻¹, and temperature = 302 K

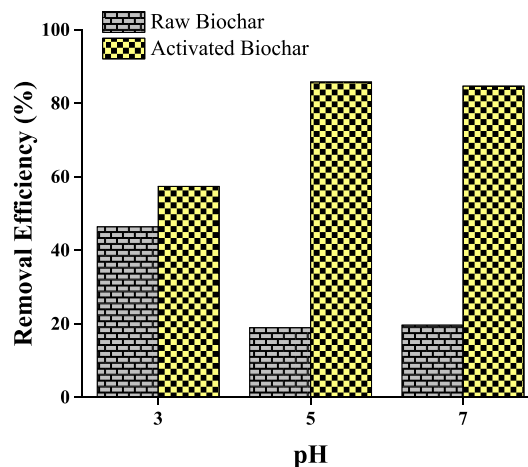


Fig. 8 Effect of initial Pb(II) concentration on removal efficiency using ACB at pH=5 ± 0.1, contact time=360 min, biochar dosage=0.5 g L⁻¹, temperature=302 K

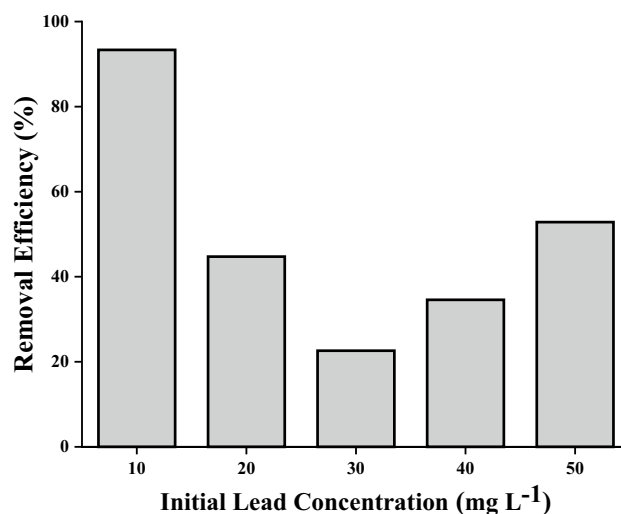
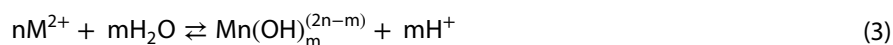
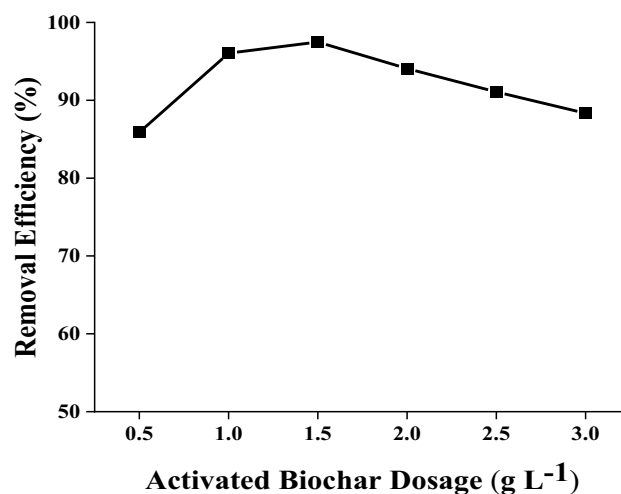


Fig. 9 Influence of ACB dosage over removal efficiency of Pb(II) ions at pH=5 ± 0.1, initial lead concentration=50 mg L⁻¹, contact time=360 min, and temperature=302 K



With an increase in pH, the adsorbent surfaces are negatively charged, so it loses protons from carboxylic acids, enols, and acidic alcohol groups which attract and bind Pb(II). At low pH (< 7), metal ion gets precipitated and form lead hydroxide/oxide. Hence, sorption and absorption are affected by the competition of precipitation; therefore, they cannot be independently studied [5]. The same observation has been documented in the literature with various biomass under similar conditions [67, 68]. As a result, equilibrium and kinetic studies were carried out at an initial pH of 5.

3.5 Effect of initial Pb(II) concentrations

The effect of initial Pb(II) concentration on removal efficiency using activated biochar was studied at pH 5 and a biochar dose of 0.5 g L⁻¹. As shown in Fig. 8, the removal efficiency for Pb(II) concentrations of 10 mg L⁻¹ onto activated biochar was 94% (using equation S1), and the sorption capacity was 9.87 mg L⁻¹ (using equation S2). The increase in removal efficiency (%) of lower concentrations of Pb(II) attributed to the active sites generated at the surface of ACB to the increase in removal efficiency (%) at lower concentrations of Pb(II) [69–71].

3.6 Effect of ACB dose on Pb(II) removal

Figure 9 illustrates the effect of ACB dosage on Pb(II) removal at pH 5 ± 0.1 , 'initial lead concentration' of 50 mg L^{-1} , biochar dosage of $0.5\text{--}3.0 \text{ g L}^{-1}$, and temperature of 302 K. The removal effectiveness of Pb(II) increased from ~ 86 to $\sim 97\%$ with different biochar dosages at a concentration of 50 mg L^{-1} . Biochar doses increased but Pb(II) ion concentrations in an aqueous solution remained constant due to the abundance of active sites available for Pb(II) sorption. The maximum sorption ($\sim 97\%$) was achieved (using equation S1) and the sorption capacity was 9.74 mg g^{-1} (using equation S2) at 1.5 g L^{-1} as compared to 0.5 and 1 g L^{-1} doses of ACB because of a huge number of active sites available over the biochar surfaces. However, the removal efficiency decreased as the dose increased from 1.5 to 3 g L^{-1} due to less mass transfer at the adsorbate and adsorbent phase in an aqueous solution. Similar findings have been documented in the literature for Pb(II) removal with KOH-activated camphor leaves [72].

3.7 Effect of temperature on Pb(II) removal

Figure 10 depicts the effect of temperature on the efficiency of Pb(II) removal. Pb(II) sorption decreased slightly as temperature increased at pH 5. The removal efficiency (%) of Pb(II) at 302 and 310 K was 86%, while at 320 K it was 77% (using equation S1), and the sorption capacity at 302 and 310 K was 8.58 mg g^{-1} , while at 320 K, it was 7.74 mg g^{-1} (using equation S2). Therefore, a lower temperature was found to be more advantageous in Pb(II) sorption from an aqueous solution. Pb(II) ions have an exothermic nature, which influences their proclivity to desorb over the biochar. A similar experiment was conducted for the sorption of Pb(II) ions using pecan shell biochar, and the results showed that increase in temperature can reduce the sorption of Pb(II) ions [68].

3.8 Effect of interfering ions

The effect of interfering ions, for example, Cl^- , NO_3^- , SO_4^{2-} , and PO_4^{3-} was investigated by mixing 50 mg L^{-1} concentration from different salts, such as NaCl, NaNO_3 , Na_2SO_4 , and Na_3PO_4 , with 50 mg L^{-1} of 'initial lead concentration' at pH 5 and a biochar dose of 0.5 mg L^{-1} . The co-existing ions negligibly influenced the removal of Pb(II) using ACB in an aqueous solution (Fig. 11). Chloride and nitrate are spherically sorbing ions and have a removal efficiency of 85%, and the sorption capacity was 8.55 mg g^{-1} . As a result, the sorption of Pb(II) has observed the least effect. At the same time, phosphate and sulphate are spherically sorbing ions with removal efficiencies of 76% and 84%, respectively, and sorption capacities of 7.65 mg g^{-1} and 8.42 mg g^{-1} , which were able to form complexes on the active sites of the functional groups of biochar surfaces as compared with the lead ions.

Fig. 10 Effect of temperature on removal efficiency of Pb(II) ions using ACB at pH 5 ± 0.1 , initial lead concentration = 50 mg L^{-1} , contact time = 360 min, and biochar dosage = 0.5 g L^{-1}

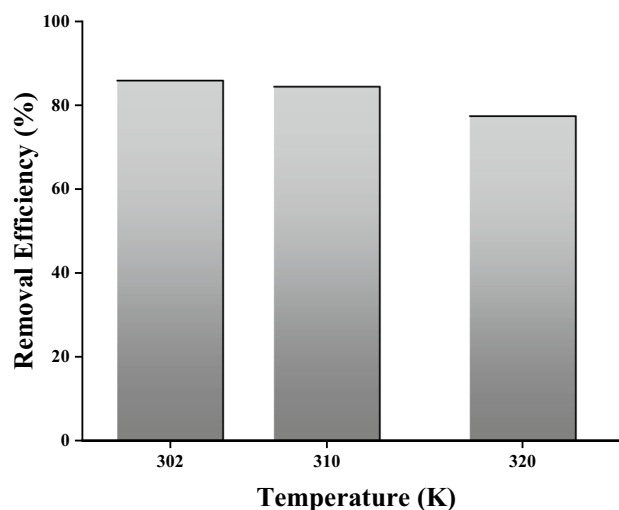


Fig. 11 Effect of co-existing ions on the removal efficiency of Pb(II) ions over SCB and ACB at pH=5±0.1, co-existing ions concentration=50 mg L⁻¹, lead concentration=50 mg L⁻¹, contact time=360 min, biochar dosage=0.5 g L⁻¹, and temperature=302 K

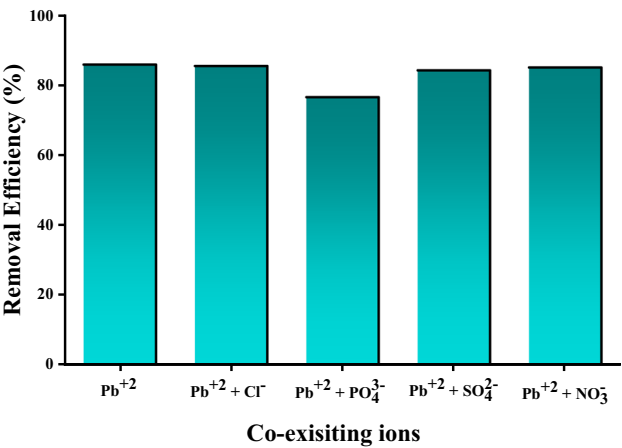


Table 4 Comparison between the isotherms model for activated *Syzygium cumini* stem biochar where, pH=5±0.1, contaminant concentration=50 mg/L, biochar dosage=0.5 g/L, temperature=302 K, residence time=360 min

Isotherm parameter	Temperature (302 K)
Freundlich	
K_F (mg/g)	20.7179
$1/n$	2.480774
R^2	0.9276
Langmuir	
Q_o (mg/g)	0.5739
β	1.5121
R^2	0.0311
Temkin	
B (L mg ⁻¹)	455.5034
K_t (J mol ⁻¹)	1.3229
R^2	0.9604

3.9 Sorption isotherms

Pb(II) sorption equilibrium studies were conducted on ACB at an initial pH of 5 and 302 K temperature. Then all the data were analysed with the help of Langmuir, Freundlich, and Temkin isotherm model along with their correlation coefficient obtained for Pb(II) removal onto activated biochar as given in Table 4. Out of these three sorption models, the Temkin isotherm model best fitted the data ($R^2=0.96$). The justification of the Temkin isotherm model as the best-fitted model is indicated by the value of R^2 (using Eqs. 4–6).

$$q_t = \frac{q_o \beta C_E}{1 + \beta C_E} \tag{4}$$

$$q_e = K_f C_e^{1/n} \tag{5}$$

$$q_e = \frac{RT}{B} \ln K_t C_e \tag{6}$$

3.9.1 Sorption kinetics

Pseudo-1st-order and pseudo-2nd-order kinetic models were used to identify the rate parameters required for Pb(II) metal sorption. Pseudo-1st and pseudo-2nd-order kinetic models were employed to simulate Pb(II) sorption on ACB. Table 5 displays the values for k_1 , q_e , and the regression coefficients. The pseudo-1_{st}-order regression coefficient

Table 5 Pseudo first order and pseudo-second order rate constants for activated *Syzygium cumini* stem biochar with Pb(II) concentration = 50 mg/L, contact time = 360 min, biochar dosage = 0.5 g L⁻¹, and temperature = 302 K

Q _e (mg g ⁻¹)	K ₁ /K ₂ (min ⁻¹)	R ²
Pseudo first order		
1.2328	0.0059	0.6411
Pseudo-second order		
9.3844	0.0985	0.999

was 0.64 only (using Eq. 7), i.e., poorly correlated between log (q_e-q_t) vs t. At the same time, the adsorbate amount (on the adsorbent) and the equilibrium amount have a significant impact on pseudo-2nd-order energy. 2nd-order regression coefficients predominate the 1st-order coefficients (0.999) (using Eq. 8).

$$\ln(q_e - q_t) = \log(q_e) - \frac{K_1}{2.303} t \quad (7)$$

$$\frac{t}{qt} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \quad (8)$$

The q_e values and regression coefficients for both kinetics are summarized in Table 5. Where, q_e and q_t are the sorption capacity of the adsorbent (mg g⁻¹), t is the time (minutes), and K₁ and K₂ are constant for pseudo-1st-order and pseudo-2nd-order kinetics, respectively.

3.9.2 Sorption thermodynamics studies

The sorption thermodynamics studies show the nature or accuracy of the sorption mechanism, whether endothermic or exothermic. The data was plotted between lnK vs 1/T for the study of Pb(II) sorption over ACB (using Eqs. 9–11).

$$\log(K_c) = \frac{\Delta S^\circ}{2.303R} - \frac{\Delta H^\circ}{2.303R} \cdot 1/T \quad (9)$$

$$K_C = \frac{C_{Ae}}{C_e} \quad (10)$$

$$\Delta G = \Delta H^\circ - T \Delta S^\circ \quad (11)$$

where R is the “universal gas constant” and T is the absolute temperature. The sorption capacity at 302 and 310 K was 8.58 mg g⁻¹ and 7.74 mg g⁻¹ for 320 K (using equation S2). Sorption on ACB is shown by the Thermodynamic Parameters (ΔH°, ΔS°, and ΔG°) for Pb(II) (Table 6). The negative value of ΔH° (– 25,982.7 kJ mol⁻¹) indicated that the Pb(II) sorption over ACB follows exothermic mechanisms under the experimental conditions, and the negative value of ΔS° (– 70.5649 kJ mol⁻¹) showed a random decrease in the surface area of ACB sorption process. The fact that the ΔG° value is negative which indicates that Pb(II) sorption is spontaneous. When Pb(II) ions were removed utilizing an activated date seed and banana stem, a similar outcome was documented [73, 74].

Table 6 Thermodynamic parameter for Pb(II) sorption with activated *Syzygium cumini* stem biochar at different temperatures where, pH = 5, initial Pb (II) concentration = 50 mg L⁻¹, contact time = 360 min, biochar dosage = 0.5 g L⁻¹

ΔS° (kJ mol ⁻¹)	ΔH° (kJ mol ⁻¹)	ΔG° (kJ mol ⁻¹)		
		302 K	310 K	320 K
– 70.5649	– 25,982.7	– 4672.08	– 4107.56	– 3401.91

4 Conclusions and future recommendations

The sorption results demonstrated that Pb(II) sorption was influenced by pH of the solution. From pH 3 to 5, Pb(II) sorption increased dramatically before stabilizing between pH 5 to 7. The electrostatic attraction and repulsion that exist between the Pb(II) and biochar could adequately explain this behavior. The removal efficiency of Pb(II) ions over ACB and equilibrium state of the system were determined by examining the impact of the initial Pb(II) concentration and contact time, respectively. It was discovered that as initial Pb(II) concentrations decreased, the removal efficiency of Pb(II) over the surface of ACB was increased. During the batch sorption experiments, the effect of adsorbent dosage on sorption was investigated, and it was discovered that sorption of Pb(II) across the surface of adsorbents increased with an increase in adsorbent dosage. The improved surface area and more active sites for interactions lead to improved removal efficiency when the dosage of adsorbent increased. At pH 5, the sorption of Pb(II) reduced somewhat with increasing temperature. The removal efficiency (%) of Pb(II) of concentration 50 mg L⁻¹ at 302 and 310 K temperature was found ~86% only, whereas the removal efficiency was observed at ~77% at 320 K, however, the sorption capacity at 302 and 310 K temperature was 8.58 mg g⁻¹ and it was 7.74 mg g⁻¹ at 320 K temperature.

The sorption equilibrium for Temkin, Langmuir, and Freundlich isotherms has been analyzed and the data fits perfectly well with the Temkin isothermal model confirming the capacities of the multi-layered sorption. A pseudo-1st-order and pseudo-2nd-order kinetics were used to determine the sorption kinetic process. Sorption of pseudo-2nd-order kinetics over an activated biochar has been discovered, which means that Pb(II) sorption over the active biochar surface constitutes a multisectoral process and involves an external sorption and Pb(II) ion diffusion in the interior of the absorbent.

The thermodynamics analysis with the negative value of ΔH° (−25,982.7 kJ mol⁻¹) indicated that the Pb(II) sorption over activated biochar follows exothermic mechanisms under the experimental conditions, and the negative value of ΔS° (−70.5649 kJ mol⁻¹) showed a random decrease in the surface area of activated biochar during the sorption process. The negative ΔG° value specifies that the sorption of Pb(II) was feasible and spontaneous. The removal efficiency of Pb(II) was compared between raw biochar and activated biochar. When compared to standard biochar production, biochar modification necessitates more energy and materials, i.e., raising the production cost. As a result, future aims include achieving a balance between optimizing performance by lowering production costs, and creating new technologies and methodologies. Based on this study, the following recommendations are proposed for future studies.

- As a novel sorbent, nanoparticles tend to agglomerate in aqueous solutions, reducing particle reactivity and making recycling more challenging. Biochar-based compounds can help nanoparticles to operate in a better way by acting as support [75]. Metal/metal oxide-biochar nanocomposites have gained popularity in recent years due to their ability to execute the combined adsorption benefits of biochar and nanomaterials, making them appealing and useful for environmental applications.
- Because the use of a modifying agent and a different process method increases production costs, procedures must be addressed before scaling up the production and putting it into practice to resolve the cost-adsorption capacity conflict. To obtain better quality products of interest, pyrolysis parameters must be tuned to correspond to different modification processes, making biochar both economically and environmentally beneficial. Furthermore, future advances should aim to improve the modified biochar's adsorption capacity in the face of interference and competition from single metals and other contaminants in natural wastewater.
- It's also important to think about the stability of the modified biochar composite. This necessitates long-term tests to determine whether the composite's bonding strength can withstand scouring and soaking by water while maintaining outstanding adsorption capabilities which may be verified by adsorbent analysis after each cycle of batch experiment.
- If metals/metal oxides, organics, and nanoparticles are present in water systems, the toxicity of modified biochar should be assessed. Future research should also look into pollutant recovery and the reuse of saturated biochar which may be confirmed by characterization of adsorbent after completion of batch experiment.

Biochar is not only effectively removes Pb(II) from water but also mitigates greenhouse gas emissions by sequestering carbon. Its low-cost production and abundance make it an attractive option for water treatment in resource-constrained regions. By utilizing biochar as an adsorbent, these countries can address water pollution issues affordably

while simultaneously reducing their carbon footprint and promoting sustainable development. Therefore, developing low-cost biochar with high adsorption capacity would be beneficial for pollutant remediation, especially in the low-income countries, where water pollution severely impacts the population. Developing biochar capable of adsorbing several heavy metals/water pollutants would be helpful for remediating water with multiple pollutants. Also, such multipurpose materials would be useful for pollution remediation of different locations affected by different pollutants. All these works need to be supported with proper risk analysis prior to large scale and widespread implementation in natural environment.

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Permissions to collect the plants/plant parts The collection of *Syzygium cumini* stem used in this study complied with local or national guidelines.

Author contributions Prabhakar Sharma conceived and planned the study, wrote and reviewed the manuscript, and supervised the research. Abhilasha performed the experiments, prepared the figures, and wrote the initial draft of the paper. Kumar Abhishek wrote and reviewed the paper. Sayan Bhattacharya wrote and reviewed the paper. Shubhalakshmi Sengupta prepared SEM images, wrote and reviewed the paper. Chandra Shekhar Seth wrote and reviewed the paper.

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Declarations

The author has read, understood, and complied as applicable with the statement on "Ethical responsibilities of Authors" as found in the Instructions for Authors and is aware that with minor exceptions, no changes can be made to authorship once the paper is submitted.

Ethics approval and consent to participate Not applicable.

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