



# Intensified Pb(II) adsorption using functionalized KCC-1 synthesized from rice husk ash in batch and column adsorption studies

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## Abstract

An attempt to investigate the feasibility of 3-aminopropyltriethoxysilane (3-APTES)-functionalized KCC-1 (NH<sub>2</sub>/KCC-1) prepared from rice husk ash (RHA) for Pb(II) removal was executed. An effective functionalization of fibrous silica nanospheres (KCC-1) by NH<sub>3</sub> was confirmed by FTIR analysis. The optimized condition of Pb(II) adsorption in the batch system was at an initial Pb(II) concentration ( $X_1$ ) of 307 mg/L, adsorbent dosage ( $X_2$ ) of 2.43 g/L, and time ( $X_3$ ) of 114 min, with the Pb(II) removal ( $Y$ ) of 90.1% (predicted) and 91.2% (actual). NH<sub>2</sub>/KCC-1 can be regenerated by nitric acid (0.1 M) with insignificant decline of Pb(II) removal percentage (adsorption = 91.2–67.3%, desorption = 77.7–51.9%) during 5 cycles adsorption–desorption study. The examination of column adsorption study at a varying flow rate (1–3 mL/min) and bed height (10–20 cm) showed a good performance at a lower flow rate and higher bed height. Both Adam–Bohalt model and Thomas model displayed a good correlation with experimental data. However, Thomas model was more suitable due to the high correlation coefficient,  $R^2 = 0.91–0.99$ . This study revealed the intensified Pb(II) adsorption using NH<sub>2</sub>/KCC-1 synthesized from RHA in batch and column adsorption studies.

**Keywords** KCC-1 · Fibrous silica nanospheres · Pb(II) adsorption · Rice husk ash · Optimization

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## Introduction

Nowadays, due to the fast evolution of industrial development, environmental pollution has become one of the major concerns. The growth of the paper, batteries, tanneries, petrochemical, and fertilizer industries has directly or indirectly discharged heavy metals into the environment (Khan et al. 2021). Heavy metals are carcinogenic and have the potential to accumulate in living beings (Rehman et al. 2021).

Lead (Pb(II)) is known as the most poisonous and carcinogenic. Exposure to Pb(II) through inhalation and ingestion of food or water will cause severe diseases and permanent trauma to human's neurological system, deadly brain, kidney, and circulatory system (Rahman et al. 2019). Besides, Pb(II) can also affect aquatic life in physiology, biochemistry, behavior, and reproduction (Petitjean et al. 2019; El-Gendy et al. 2021). However, the maximum permissible limit (MPL) of Pb(II) concentration is only less than 0.5 µg/g, as itemized by World Health Organization (Loghmani et al. 2022). Therefore, the amount of Pb(II) emitted into the environment must be reduced because of its toxicity, even at modest levels of contamination.

The removal of Pb(II) has been examined using numerous techniques, for example, adsorption (Alqadami et al. 2020), chemical precipitation (Chen et al. 2018), membrane filtration (Jamshidifard et al. 2019), and ion exchange (Bezzina et al. 2019). Among these techniques, adsorption gained higher attraction due to its low cost, ability to minimize chemical and biological sludge, and flexible and straightforward design (Dotto and McKay 2020). However, selecting an appropriate adsorbent is critical to improving the adsorption process.

Mesoporous silica has attracted substantial attention as an adsorbent due to its variable pore size, wide surface area, and biocompatibility (Rechotnek et al. 2021). Lately, a new family of mesoporous silica, known as fibrous silica nanospheres (KCC-1), was introduced in 2010. KCC-1 features excellent characteristics, including nanosized, high surface area, large pore diameter, unprecedented fibrous surface morphology, hydrothermal stabilities, and high mechanical stabilities. Due to its excellent characteristics, KCC-1 has been explored in various applications, including dehydrogenation (Zhang et al. 2019), catalysis (Abdulrasheed et al. 2020), and drug delivery (Pishnamazi et al. 2021a; Follmann et al. 2020).

Additionally, previous studies proved that the KCC-1 has excellent potential for Pb(II) adsorption (Hasan and Setiabudi 2019). However, the cost for the manufacture of KCC-1 is high, which can be claimed on the cost of the silica source. Therefore, low cost and high availability of agricultural by-products with high silica content (Anuar et al. 2018; Hubadillah et al. 2018) will be a good choice. Rice husk is one of the major agricultural wastes with an annual production of 545 million metric tons (Mohseni et al. 2019), and has been demonstrated to have high silica composition after pyrolysis (Osman 2020). Therefore, employing rice husk ash (RHA) as an alternate silica source can be an excellent example of promoting the circular economy by converting low-value lignocellulosic materials into added-value products (Osman et al. 2020). This approach could also minimize solid waste pollution and consequently promote sustainable development.

Nevertheless, challenges arise when the performance of the silica materials synthesized from waste has low efficiency compared to commercial mesoporous silica. It has been reported that the efficiency of the adsorbent can be improved by surface modification of the adsorbent. Among others, 3-aminopropyltriethoxysilane APTES has been widely used as a surface modifier due to their possible interaction with the silanol group via siloxane bonds, electrostatic attraction, and hydrogen bonds (Cho et al. 2018). Therefore, in this research, KCC-1 synthesized from RHA was functionalized with an amino group to enhance the adsorption behavior of batch and column studies. To the best of the authors' knowledge, the current study is the first

to explore the adsorption performance of amino-modified KCC-1 from RHA.

## Materials and methods

### Extraction of sodium silicate from rice husk ash (RHA)

The extraction process was conducted according to Hasan et al. (2019a). In brief, the rice husk ash (RHA) was washed and dried (120 °C, overnight) to remove impurities. Then, the mixture of RHA and sodium hydroxide (NaOH, Merck) was calcined (550 °C, 1 h) to prepare NaOH-fused RHA. The NaOH-fused RHA was mixed with deionized water, agitated (24 h), and filtered. The supernatant liquid was used for the KCC-1 preparation. According to the X-ray Fluorescence Spectrophotometry (XRF, Bruker S8 TIGER), SiO<sub>2</sub> was identified as a substantial component of RHA with a composition of 88.52%.

### KCC-1 preparation

KCC-1 was synthesized consistent with Hasan et al. (2019b) with some modifications. Solution 1 [Na<sub>2</sub>SiO<sub>3</sub>-RHA, butanol (Merck), and toluene (Merck)] was mixed with Solution 2 [water, urea (Merck), and cetyltrimmonium bromide (CTAB, Aldrich)], stirred (room temperature, 45 min), and heated (120 °C, 5 h) in a hydrothermal synthesis reactor. The mixture was centrifuged and filtered. The solid particles were oven-dried (100 °C, 12 h) and calcined (550 °C, 6 h) to obtain KCC-1.

### Surface modification

KCC-1 was functionalized by mixing KCC-1 (3 g), toluene (50 mL), and 3-aminopropyltriethoxysilane (3-APTES) (2.4 g). The mixture was then refluxed (110 °C, 48 h) and filtered. The solid particles were then rinsed with ethanol and dried (100 °C, 12 h) to obtain NH<sub>2</sub>/KCC-1.

### Characterization

The crystallinity texture was analyzed by X-ray diffraction (XRD, Miniflex II Rigaku, 15 mA, 30 kV) with a Cu-K $\alpha$  radiation ( $\lambda = 1.54 \text{ \AA}$ ). The textural characteristics were studied using the Brunauer–Emmett–Teller (BET, Micromeritics®) method at 77 K. Fourier-Transform Infrared (FTIR, Thermo Scientific Nicolet iS5) was conducted for functional groups investigation. Transmission Electron Microscopy (TEM, Leo Libra-120) was employed for morphology analysis. The specimen was sonicated in ethanol,

and the solution was dropped onto an amorphous porous carbon grid.

### Batch adsorption

A stock Pb(II) solution (1000 mg/L,  $\text{Pb}(\text{NO}_3)_2$ , Sigma-Aldrich) was prepared prior to dilution to the necessary concentration (50–400 mg/L). The  $\text{NH}_2/\text{KCC-1}$  (0.5–5 g/L) was added to Pb(II) solution (200 mL, room temperature) while stirring for the adsorption process. The sample was collected at a specific time (0–140 min) and centrifuged (2 min, 3000 rpm). The Pb(II) concentration was determined using UV–Vis spectroscopy at 520 nm with a dithizone reagent.

The amount of Pb(II) adsorbed, and the percentage of Pb(II) removed were determined using Eqs. (1) and (2), respectively.

$$q_t = \left( \frac{C_o - C_t}{m} \right) \times V, \quad (1)$$

$$\text{Removal (\%)} = \left( \frac{C_o - C_t}{C_o} \right) \times 100, \quad (2)$$

where  $q_t$  (mg/g) signifies the quantity of Pb(II) adsorbed at a specific time,  $C_o$  (mg/L) represents the initial Pb(II) concentration, and  $C_t$  (mg/L) signifies the Pb(II) concentration at a specific time.  $V$  (L) represents the volume of the Pb(II) solution, and  $m$  (g) signifies the mass of  $\text{NH}_2/\text{KCC-1}$ .

### Experimental design and optimization

Response Surface Methodology (RSM) (Statsoft Statistica 8.0) with a central composite design (CCD) was chosen as the experimental design. The independent variables were the

initial concentration,  $X_1$  (50–400 mg/L), adsorbent dosage,  $X_2$  (0.5–5.0 g/L), and time,  $X_3$  (0–140 min). These independent variables were selected according to the findings obtained from the one-factor-at-a-time method (OFAT). A total of 16 different trials were carried out in triplicate. The result was analyzed by applying the analysis of variance (ANOVA), coefficient of determination ( $R^2$ ), and 3D response surface plots.

### Column adsorption

Figure S1 depicts the column system, whereby KCC-1 and cotton wool were placed in the column as illustrated. Following that, glass beads were placed on both sides of the filter to ensure that the influent solution was distributed uniformly from top to bottom. Then, the column was washed with pure distilled water before being fed with a Pb(II) solution.

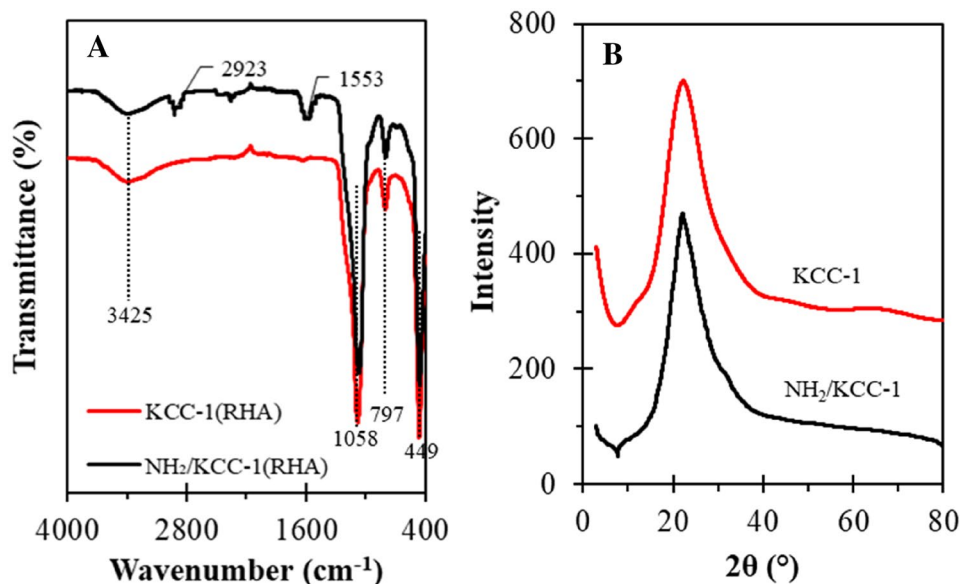
The continuous flow was used in the experiments, and the regular flow of the solution discharge volume was measured at specific times. 500 mL of a Pb(II)-containing solution was run through the column. At predefined intervals, samples were taken from the column output, followed by centrifugation and UV–vis analysis similar to the batch process.

## Results and discussion

### Characterization analysis

Figure 1a compares the FTIR spectra of KCC-1 and  $\text{NH}_2/\text{KCC-1}$ . From the graph, all materials showed the presence of a peak around  $449 \text{ cm}^{-1}$ ,  $797 \text{ cm}^{-1}$ ,  $1058 \text{ cm}^{-1}$ , and  $3425 \text{ cm}^{-1}$  that corresponded to Si–O bending, unsymmetrical stretching of Si–O, symmetrical Si–O, and O–H

**Fig. 1** A FTIR spectra and B XRD pattern of KCC-1 and  $\text{NH}_2/\text{KCC-1}$



stretching vibration of Si–OH, respectively (Ershad et al. 2021). However, after surface modification with an amino group, addition peaks at  $2923\text{ cm}^{-1}$  and  $1553\text{ cm}^{-1}$  were detected that were ascribed as N–H stretching and N–H bending vibrations of the aminopropyl group, respectively. The presence of these peaks confirmed that the aminopropyl groups of 3-APTES were introduced into KCC-1. The presence of the amino group was also found by Zarei et al. (2019), who did a surface modification on KCC-1 and applied it to remove chromium.

Figure 1b displays the XRD pattern of KCC-1 and  $\text{NH}_2/\text{KCC-1}$ . The broad peaks at  $20^\circ$  and  $30^\circ$  proved the amorphous silica phase of the material (Hasan et al. 2019a). The XRD pattern showed no significant alterations, which suggested that the crystallinity texture of KCC-1 had not changed with the introduction of  $\text{NH}_2$ .

Figure 2a, b illustrates the TEM images of KCC-1 and  $\text{NH}_2/\text{KCC-1}$ , respectively. The synthesized KCC-1 is made up of uniform spheres with a fibrous morphology, similar to KCC-1 prepared from commercial silica sources, as reported by other researchers (Hitam et al. 2020; Zhang et al. 2019), indicating successful preparation of KCC-1 from RHA. The modification of KCC-1 with an amino group does not change the surface morphology of the KCC-1, as shown in Fig. 2b.

The BET analyses were employed to ascertain the surface area ( $S_{\text{BET}}$ ) and pore volume ( $V_p$ ). The  $S_{\text{BET}}$  of KCC-1 and  $\text{NH}_2/\text{KCC-1}$  were  $220\text{ m}^2/\text{g}$  and  $224\text{ m}^2/\text{g}$ , respectively.  $\text{NH}_2/\text{KCC-1}$  had a higher surface area, thus facilitating an effective adsorption process (Khantan et al. 2019). Notably, an increase in the  $V_p$  of  $0.94\text{ cm}^3/\text{g}$  (KCC-1) to  $1.12\text{ cm}^3/\text{g}$  ( $\text{NH}_2/\text{KCC-1}$ ) allowed for high loadings of metals or non-metals pollutants.

## Batch adsorption

### Statistical analysis

Table 1 summarizes the 16 experimental runs with the response values (Pb(II) removal). The regression coefficient was decoded using multiple analyses, as shown in Eq. (3). In the equation, a positive and negative sign denotes synergistic and antagonistic effects, respectively.

$$Y = 76.3648 + 0.0473X_1 + 2.1429X_2 + 0.0673X_3 - 0.0001X_1^2 - 0.4320X_2^2 - 0.0002X_3^2 - 0.0001X_1X_2 - 0.0001X_1X_3 - 0.0002X_2X_3, \quad (3)$$

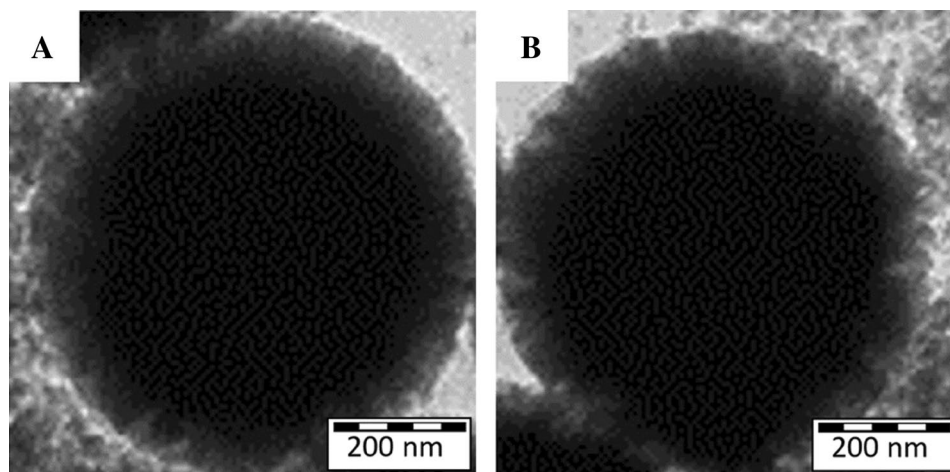
where  $Y$  is the estimated response, while  $X_1$ ,  $X_2$ , and  $X_3$  are the coded independent variables.

A comparison between the observed value (from the experiment) and predicted value (calculated from the regression model) of Pb(II) percentage removal is illustrated in Fig. S2. The good regression coefficient can be described with a close value of 1.0 or  $> 0.75$  (Storm 2019). From the graph, the obtained high correlation coefficient,  $R^2$  (0.9401), indicated that 94.01% of the data variability was considered for the model.

The understanding of the response surface quadratic model was further studied with variance analysis (ANOVA). ANOVA is a statistical tool that divides a collection of data into portions connected to specific sources of variation to test hypotheses associated with the model parameter (Jankovic et al. 2021). Table 2 lists the ANOVA data for this experiment. The tabulated  $F$ -value ( $F_{\text{table}} = 4.10$ ) was lower than calculated  $F$ -value ( $F_{\text{model}} = 10.42$ ) at 95% probability ( $p = 0.05$ ), indicating a significant of the model.

The Pareto chart was utilized to determine which variable was significant in this model. The predicted effect of each independent variable was indicated as proportional length

**Fig. 2** TEM images of **A** KCC-1 and **B**  $\text{NH}_2/\text{KCC-1}$

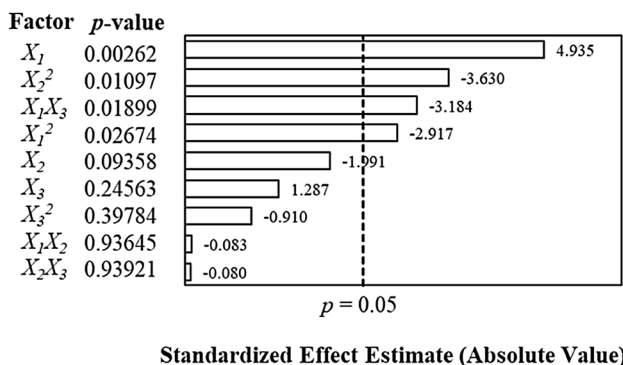


**Table 1** Experimental design and experimental results of Pb(II) removal

Run	Independent variables						Response
	Initial concentration (mg/L), $X_1$		Adsorbent dosage (g/L), $X_2$		Time (min), $X_3$		Pb(II) removal (%), $Y$
	Uncoded	Coded	Uncoded	Coded	Uncoded	Coded	
1	50	-1	0.5	-1	60	-1	82.99
2	50	-1	0.5	-1	180	1	86.28
3	50	-1	5	1	60	-1	81.77
4	50	-1	5	1	180	1	84.91
5	400	1	0.5	-1	60	-1	87.92
6	400	1	0.5	-1	180	1	86.75
7	400	1	5	1	60	-1	86.54
8	400	1	5	1	180	1	85.31
9	50	-1	2.75	0	120	0	85.09
10	400	1	2.75	0	120	0	89.78
11	225	0	0.5	-1	120	0	87.38
12	225	0	5	1	120	0	86.63
13	225	0	2.75	0	60	-1	88.66
14	225	0	2.75	0	180	1	88.63
15 (C)	225	0	2.75	0	120	0	90.81
16 (C)	225	0	2.75	0	120	0	90.49

**Table 2** ANOVA for removal percentage of Pb(II)

Sources	Sum of square (SS)	Degree of freedom (df)	Mean square (MS)	F-value
Regression (SSR)	90.053	9	10.0	10.42
Residual	5.7411	6	0.96	
Total (SST)	95.794	15		

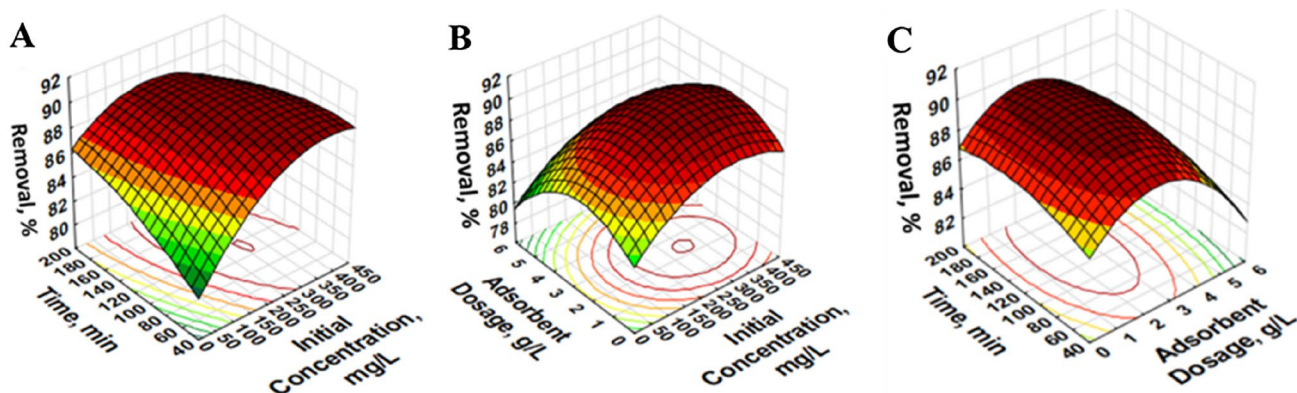


**Fig. 3** Pareto chart for Pb(II) removal onto  $NH_2/KCC-1$

corresponding to the absolute value, as illustrated using the horizontal bar chart shown in Fig. 3. The independent variables were examined using the  $p$ -value and  $t$ -value, in which the variables with a larger  $t$ -value magnitude or/and a lower  $p$ -value had a greater impact on the regression model

(Setiabudi et al. 2013). The linear term of initial concentration ( $X_1$ ), quadratic term of adsorbent dosage ( $X_2^2$ ), interactive term between initial concentration and time ( $X_1X_3$ ), and quadratic term of initial concentration ( $X_1^2$ ), were statistically significant due to the large  $t$ -value magnitude and low  $p$ -value ( $p < 0.05$ ). The other terms of the variables were regarded as less significant because of the larger  $p$ -value ( $p > 0.05$ ). In short, initial concentration ( $X_1$ ) had the greatest influence on Pb(II) removal, while the relation between adsorbent dose and time ( $X_2X_3$ ) had the least.

Figure 4a depicts the interaction impacts of initial concentration ( $X_1$ ) and time ( $X_3$ ) on Pb(II) elimination. The elliptical form of the response surface demonstrated outstanding interaction amidst initial concentration and time. An increase in the initial concentration (from 50 to 300 mg/L) and time (from 40 to 100 min) increased the Pb(II) removal percentage until the optimum conditions ( $X_1 = 300\text{--}350$  mg/L,  $X_3 = 100\text{--}150$  min,  $Y = 90\%$ ) were achieved, and decreased at elevated initial concentration ( $> 350$  mg/L) and time ( $> 150$  min). At the beginning of the time, the percentage removal increased with increasing time until it achieved the optimal (100–150 min) and continued to decrease at a higher time. This result can be clarified by a higher quantity of accessible active sites at the beginning of the time and decreases with increasing time. Additionally, when the initial concentration of Pb(II) increased, the percentage of Pb(II) removal increased as well, which was related to the sufficient active sites on the  $NH_2/KCC-1$ 's surface to attract Pb(II) ions. However, after the adsorption on the active site



**Fig. 4.** 3D plots show the effects of **A** initial concentration and time, **B** initial concentration and adsorbent dosage, and **C** adsorbent dosage and time

achieved the saturated point, raising the initial concentration would not affect the Pb(II) removal.

The interaction impacts of initial concentration ( $X_1$ ) and adsorbent dosage ( $X_2$ ) on the elimination of Pb(II) are depicted in Fig. 4b. An increase in initial concentration and adsorbent dosage increased the removal of Pb(II) until it passed the optimum values around  $X_1 = 300\text{--}350$  mg/L and  $X_2 = 2\text{--}3$  g/L, respectively, with Pb(II) percentage removal around 90%, and declined at the elevated initial concentration ( $> 350$  mg/L) and adsorbent dosage ( $> 3$  g/L). This phenomenon increased the percentage removal of Pb(II) until a specific value. Then, it started to decrease as the formation of adsorbent aggregation reduced the viability of the active sites attached by Pb(II) ions (Ain et al. 2020). By comparing both independent variables, the initial concentration of Pb(II) displayed a more significant impact in Pb(II) removal than adsorbent dosage. This outcome was compatible with the Pareto chart analysis (Fig. 3), which indicated that the initial concentration had a higher  $t$ -value (4.935) than the adsorbent dosage with  $t$ -value of  $-1.991$ . A comparable result was described by Bahrami et al. (2019) for Pb(II) removal using starch phosphate. In their study, the Pb(II) removal improved with increasing initial Pb(II) concentration and adsorbent dosage, reaching the optimum at 63.57 mg/L and 0.25 g/L, respectively, but dropped at elevated values.

Figure 4c displays the impacts of adsorbent dosage ( $X_2$ ) and time ( $X_3$ ) on the elimination of Pb(II). According to the plot, increasing the adsorbent dosage and time concurrently increased the Pb(II) removal until it reached the optimum percentage (90%) at the adsorbent dosage of 2–3 g/L and time of 100–150 min, and then decreased as the adsorbent dosage and time were increased further. This finding can be explained by a higher quantity of accessible active sites at the beginning of the time and decreases with increasing time. Lingamdinne et al. (2018) reported a comparable trend for the Pb(II) adsorption

using nickel ferrite-reduced graphene oxide (NFRGO) nano-composite, in which the removal of Pb(II) increases with time and NFRGO dosage until the optimal condition was reached. It is interesting to note that  $\text{NH}_2/\text{KCC-1}$  showed good adsorption performance owing to its exciting morphology that contains well-defined and ordered fibers coming out from the center of the particles and uniformly distributed in all directions. This advantage allows  $\text{NH}_2/\text{KCC-1}$  to adsorb the pollutant more effectively.

The optimization modeling study suggested the optimal conditions were at initial Pb(II) concentration ( $X_1$ ) of 307 mg/L,  $\text{NH}_2/\text{KCC-1}$  dosage ( $X_2$ ) of 2.43 g/L, and time ( $X_3$ ) of 114 min, with Pb(II) removal ( $Y$ ) of 90.1%. Triplicate experiments were conducted under optimal conditions to validate the optimization modeling findings. The removal of Pb(II) was attained at 91.2%, with a percentage error of 1.1%.

### Kinetic study

In this study, three types of kinetic models [pseudo-first-order, pseudo-second-order (Ezzati 2020), and Elovich (Ealias and Saravanakumar 2018)] were utilized. The kinetics study was conducted at optimum conditions ( $\text{NH}_2/\text{KCC-1}$  dosage of 2.43 g/L and time of 114 min) with different initial Pb(II) concentrations ( $C_0 = 100\text{--}500$  mg/L). The kinetics of heterogeneous chemisorption were characterized by the Elovich, while pseudo-first-order and pseudo-second-order defined adsorption behavior in physisorption and chemisorption, respectively (Nayak and Pal 2019). The equation of the models is expressed as follows:

$$\text{Pseudo - first - order: } \log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t, \quad (4)$$

Pseudo - second - order:  $\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t,$  (5)

Elovich:  $q_e = \left(\frac{1}{\beta}\right) \ln(\alpha\beta) + \left(\frac{1}{\beta}\right) \ln t,$  (6)

where the quantities of Pb(II) adsorbed at time,  $t$ , and equilibrium represent as  $q_t$  (mg/g) and  $q_e$  (mg/g), respectively.  $k_1$  (L/min) represents pseudo-first-order's rate constant, while  $k_2$  (g/mg·min) represents pseudo-second-order's rate constant.  $\alpha$  (mg/(g min)) signifies adsorption rate constant, and  $\beta$  (g/mg) represents desorption constant.

The linear regression coefficient ( $R^2$ ) and other parameters are tabulated in Table 3. The tabulated data was the average value of triplicate experiments. Hence, the pseudo-second-order model was the best-fitted isotherm attributable to the highest  $R^2$  ( $\geq 0.9986$ ) and the closest value of  $q_e$ . In contrast, the Elovich model was the weakest due to the lowest  $R^2$  value. The pseudo-second-order model also best fitted the Pb(II) adsorption onto common Guar gum/bentonite bio-nanocomposites (Ahmad and Mirza 2018), as well as in our previous study for Pb(II) adsorption using KCC-1 (Hasan et al. 2019a). In a nutshell, the Pb(II) adsorption onto NH<sub>2</sub>/KCC-1 is a chemisorption process, and the adsorption rate is proportionate to the accessibility of the active sites on the NH<sub>2</sub>/KCC-1's surface.

**Isotherm study**

The isotherm models of Freundlich (Al-Ghouti and Da'ana 2020), Temkin (Aharoni and Ungarish 1977), Dubinin-Radushkevich (Dubinin 2002), and Langmuir (Langmuir 2002) were utilized in this study. The isotherm study was conducted at optimum conditions (NH<sub>2</sub>/KCC-1 dosage of 2.43 g/L and time of 114 min) with different initial Pb(II) concentrations ( $C_0 = 100\text{--}500$  mg/L). Temkin is employed to examine how adsorbent–adsorbate interactions influence the adsorption process (Liu et al. 2020). Meanwhile, the

Dubinin–Radushkevich is accredited to the heterogeneous surface assumption theory for the adsorption mechanism. In contrast, Langmuir and Freundlich explained that monolayer adsorption happens on the homogenous adsorbent's surface and multilayer adsorption over the heterogeneous adsorbent's surface, respectively (Benkaddour et al. 2018). The linearized forms of these isotherms were expressed as the following equations.

Langmuir:  $\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m},$  (7)

Freundlich:  $\log q_e = \log K_F + \frac{1}{n} \log C_e,$  (8)

Temkin:  $q_e = B \ln A + B \ln C,$  (9)

Dubinin – Radushkevich:  $\ln q_e = \ln q_m - K_{DR} \varepsilon^2,$  (10)

where  $q_e$  (mg/g) represents the equilibrium adsorption capacity, while the maximum adsorption capacity is symbolized as  $q_m$  (mg/g).  $n$  is an empirical constant,  $C_e$  (mg/L) is the Pb(II) concentration at equilibrium, while  $K_L$  (L/mg) and  $K_F$  ((mg/g) (L/mg)<sup>1/n</sup>) are the Langmuir and Freundlich constant, respectively. For the Temkin isotherm,  $B$  represents Temkin constant, while an equilibrium binding constant is symbolized as  $A$  (L/g). For the Dubinin–Radushkevich isotherm,  $K_{DR}$  (mol<sup>2</sup>/kJ<sup>2</sup>) represents Dubinin–Radushkevich constant, while Polanyi potential is symbolized as  $\varepsilon$  (J/mol).  $\varepsilon$  can be calculated from  $\varepsilon = RT \ln(1 + 1/C_e)$ , where  $R$  (J/mol·K) is gas constant and  $T$  (K) is the absolute temperature. The Langmuir equilibrium parameter ( $R_L = 1/(1 + K_L C_0)$ ) is used to indicate the isotherm shape either irreversible, favorable, linear, or unfavorable, for  $R_L = 0$ ,  $0 < R_L < 1$ ,  $R_L = 1$ , or  $R_L > 1$ , respectively [44].

Table 4 lists the computed parameters for the studied models. The tabulated data was the average value of triplicate experiments. Thus, the Langmuir isotherm

**Table 3** Kinetic parameters for removal of Pb(II) onto NH<sub>2</sub>/KCC-1

Models	Parameter	100 ppm	200 ppm	300 ppm	400 ppm	500 ppm
Experimental	$q_{e,exp}$ (mg/g)	41.237	42.779	51.143	52.836	54.734
Pseudo-first-order	$q_{e,cal}$ (mg/g)	6.2661	9.4842	9.8175	9.3240	10.678
	$k_1$ (min <sup>-1</sup> )	0.0392	0.0343	0.0309	0.0161	0.0306
	$R^2$	0.8888	0.9853	0.9845	0.8767	0.9939
Pseudo-second-order	$q_{e,cal}$ (mg/g)	41.494	43.668	51.813	53.191	55.249
	$k_2$ (g/mg min)	0.0189	0.0092	0.0087	0.0070	0.0084
	$R^2$	0.9999	0.9998	0.9997	0.9986	0.9998
Elovich kinetic	$\alpha$ (mg/(g min))	2E+09	3E+07	4E+08	8E+08	2E+08
	$\beta$ (g/mg)	0.6263	0.5060	0.4713	0.4774	0.4241
	$R^2$	0.9295	0.8795	0.9363	0.9068	0.9269

**Table 4** Isotherm models for Pb(II) adsorption onto NH<sub>2</sub>/KCC-1

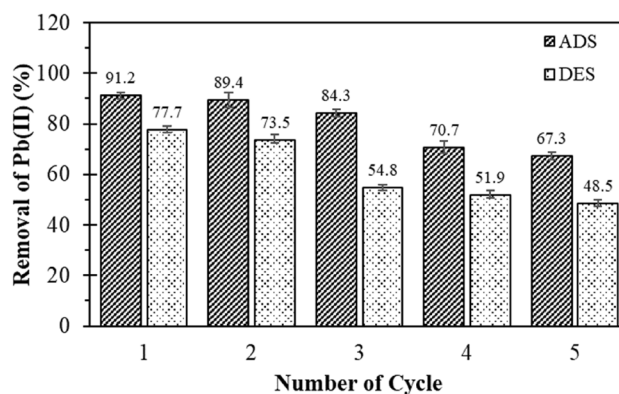
Isotherm	Parameters	Value
Langmuir	$q_m$ (mg/g)	28.169
	$K_L$ (L/mg)	0.0749
	$R^2$	0.9927
	$R_L$	0.1178
Freundlich	$n$	1.5083
	$K_f$ (mg/g) (L/mg) <sup>1/n</sup>	488.65
	$R^2$	0.9309
Temkin	$B$ (J/mol)	31.312
	$A$ (L/g)	154.95
	$R^2$	0.9223
Dubinin-Radushkevich	$q_m$ (mg/g)	58.621
	$K_{ad}$ (10 <sup>-4</sup> )	1
	$R^2$	0.9898

was the best-fitted model attributable to the highest value of  $R^2$ , which indicated that Pb(II) uptake occurs on the homogenous surface by monolayer adsorption. The Langmuir's  $q_m$  value,  $K_L$  constant,  $R_L$  value was 28.169 mg/g, 0.0749 L/mg, and 0.1178, respectively. The observed results verified that the synthesized NH<sub>2</sub>/KCC-1 considerably favored the Pb(II) adsorption with Pb(II) initial concentration = 307 mg/L, adsorbent dosage of NH<sub>2</sub>/KCC-1 = 2.43 g/L, and time = 114 min. Furthermore, by comparing the maximum adsorption capacity ( $q_m$ ) of NH<sub>2</sub>/KCC-1 (this study) with KCC-1 synthesized from RHA (Hasan et al. 2019a), NH<sub>2</sub>/KCC-1 has a higher  $q_m$  (28.169 mg/g) than the KCC-1 (26.954 mg/g), indicated an excellent performance of NH<sub>2</sub>/KCC-1 in removing Pb(II). The excellent performance of NH<sub>2</sub>/KCC-1 can be explained by the positive role of amino groups in improving the surface area and functional groups that responsible in the adsorption process.

The  $q_m$  value for NH<sub>2</sub>/KCC-1 (this study) was compared to the  $q_m$  values reported in the literature for various silica adsorbents, as shown in Table S1. It was noticed that NH<sub>2</sub>/KCC-1 has a superior adsorption capacity, which is attributed to the exceptional physicochemical features (nanosized, high surface area, large pore diameter, and unprecedented fibrous surface morphology), showing the considerable potential of NH<sub>2</sub>/KCC-1. Interestingly, NH<sub>2</sub>/KCC-1 was produced using RHA as a silica source, which benefited environmental impacts.

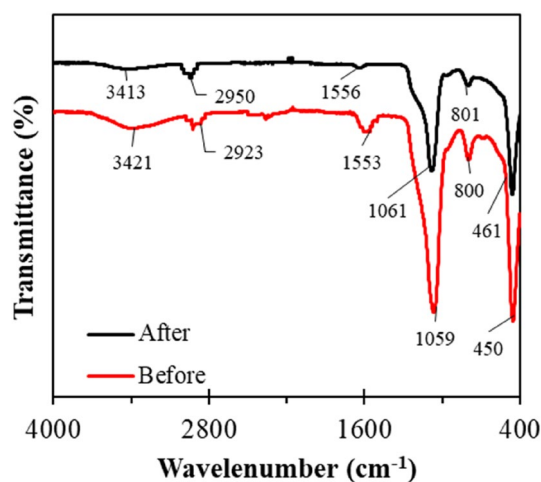
### Reusability study

Figure 5 depicts the performance of NH<sub>2</sub>/KCC-1 over five adsorption–desorption experiments at optimum conditions (Pb(II) concentration of 307 mg/L, NH<sub>2</sub>/KCC-1 dosage of 2.43 g/L, and time of 114 min). The desorption method was

**Fig. 5** Regeneration and reusability of NH<sub>2</sub>/KCC-1

studied using a chemical treatment of nitric acid (HNO<sub>3</sub>) (0.1 M) because of its effectivity, as published by Place et al. (2018). The adsorption–desorption displayed a decreasing pattern with an increasing number of cycles. This finding could be due to the blockage of the active sites by Pb(II) molecules and changes in the adsorbent's structure and the chemical composition after the adsorption process (Fu et al. 2019). Abdelrahman and Hegazy (2019) reported a similar phenomenon for adsorption–desorption cycles of Pb(II) removal by hydroxy sodalite/chitosan composites.

Even though the adsorption–desorption showed a decreasing pattern, the percentage reduction was less significant (3.4%) as compared to Pb(II) removal by KCC-1, almost 7% for the fifth cycle, as described in our earlier publication (Hasan et al. 2019a). Additionally, the efficacy of KCC-1 toward Pb(II) adsorption and Pb(II) desorption was lower than NH<sub>2</sub>/KCC-1, with percentage differences of 19.3% and 4.8%, respectively. The better performance of

**Fig. 6** FTIR spectra of NH<sub>2</sub>/KCC-1 before and after Pb(II) adsorption



NH<sub>2</sub>/KCC-1 compared to KCC-1 could be ascribed to the positive role of the amine group in the adsorption process.

The FTIR spectra of NH<sub>2</sub>/KCC-1 (before and after adsorption) revealed identical infrared peaks, as illustrated in Fig. 6. However, the peak intensity had altered after adsorption. Both FTIR spectra showed the existence of O–H stretching vibration of Si–OH (3421 cm<sup>-1</sup> and 3413 cm<sup>-1</sup>), stretching vibration of N–H (2923 cm<sup>-1</sup> and 2950 cm<sup>-1</sup>), bending vibration of N–H (1553 cm<sup>-1</sup> and 1556 cm<sup>-1</sup>), symmetrical stretching of Si–O (1059 cm<sup>-1</sup> and 1061 cm<sup>-1</sup>), unsymmetrical stretching of Si–O (800 cm<sup>-1</sup> and 801 cm<sup>-1</sup>), and Si–O bending (450 cm<sup>-1</sup> and 461 cm<sup>-1</sup>), respectively (Pishnamazi et al. 2021b). After adsorption, the peaks were shifted to a higher wavenumber, attributable to the electrostatic interaction, complexation, and coordination of functional groups with metal ions (Shahabuddin et al. 2018). The amino and hydroxyl groups are implicated in Pb(II) adsorption by surface complexation (Wu et al. 2019).

### Column adsorption

#### Effect of flow rate

The impact of flow rates at a fixed value of bed height (15 cm) and initial Pb(II) concentration (307 mg/L) was illustrated by the breakthrough curve in Fig. 7a. With increasing flow rate, the plotted curve showed a reduction in residence time and adsorption capacity. Therefore, the adsorption process can be considered the most effective at lower flow rates due to the higher residence time, leading to higher Pb(II) elimination from an aqueous phase. Bo and their team (Bo et al. 2020) conducted the adsorption of Pb<sup>2+</sup> in batch and column systems using amine (N/O)-enrichment of adsorbent (CM<sub>0.5</sub>AP<sub>0.75</sub>). Their research showed

that insufficient contact between CM<sub>0.5</sub>AP<sub>0.75</sub> and Pb<sup>2+</sup> in a higher flow rate resulted in decreasing adsorption capacity. Consequently, employing a lower flow rate was recommended for the system to achieve high adsorption of Pb<sup>2+</sup>.

#### Effect of bed height

The influence of column bed height was examined with a fixed initial Pb(II) concentration (307 mg/L) and flow rate (1 mL/min). The result of the continuous study is shown in Fig. 7b. From the graph, the adsorption capacity and breakthrough curve showed that increasing bed height (from 10 to 20 cm) increased the adsorption capacity and sharpened the breakthrough curve shape. The quantity of adsorbent in the column affected the Pb(II) removal. Besides, Pb(II) solution spent more time in contact with the adsorbent, and an enormous number of active sites were accessible when a higher/more extended bed height was used.

#### Adsorption modeling

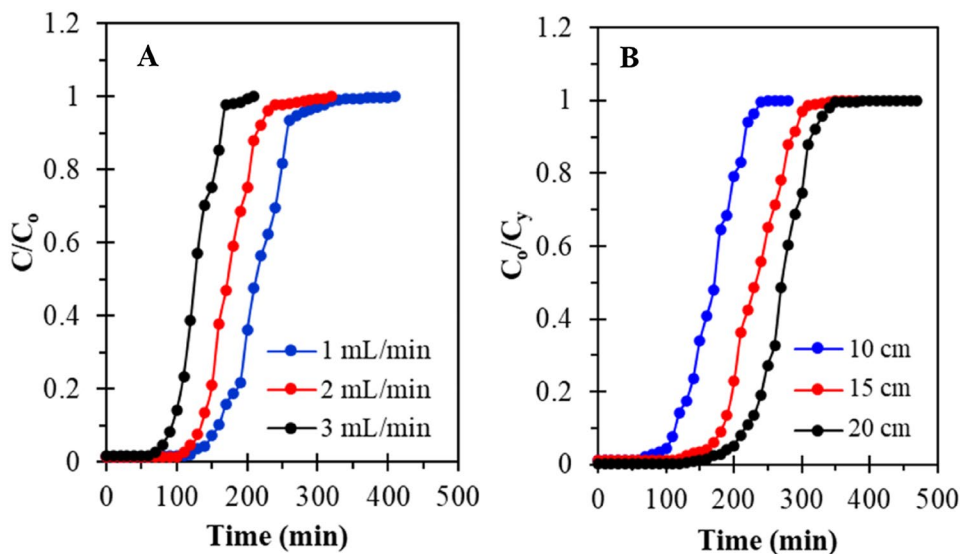
The adsorption modeling was examined using the Adam–Bohart model and Thomas model. The linearised forms of these models are expressed as the following equations.

$$\text{Adam - Bohart model: } \ln \left( \frac{C_t}{C_o} \right) = K_{AB} C_o t - \frac{K_{AB} N_o Z}{U_o}, \tag{11}$$

$$\text{Thomas model: } \ln \left( \frac{C_o}{C_t} - 1 \right) = \frac{K_{Th} q_o m}{Q} - k_{Th} C_o t, \tag{12}$$

where C<sub>o</sub> (mg/L) and C<sub>t</sub> (mg/L) represent inlet and outlet Pb(II) concentration, respectively. t (min) represents the time

**Fig. 7** Effect of **A** flow rate and **B** adsorbent height on continuous removal of Pb(II) onto NH<sub>2</sub>/KCC-1



of the breakthrough,  $Z$  (cm) signifies the column height,  $Q$  (mL/min) represents flow rate, and  $N_0$  (mg/L) indicates saturation concentration. Meanwhile,  $K_{AB}$  (L/mg min) and  $K_{Th}$  (mL/min mg) represent the kinetic constant of the Adam–Bohart and Thomas models, respectively.

Adam–Bohart model explained the theory about surface reaction, and the equilibrium was assumed not instantaneously. Therefore, the reaction rate was proportionate to the remaining fraction of adsorption capacity on the adsorbent. To study this model, the graph of  $\ln(C_t/C_0)$  versus  $t$  (not shown) was plotted, and the calculated parameter from Eq. (11) is tabulated in Table 5. The obtained data indicated that the value of  $K_{AB}$  reduced with an increment in an inlet Pb(II) concentration, whereas the value of  $N_0$  increased with an increment in the inlet Pb(II) concentration. This result indicated that, at initial column adsorption, the external mass transfer had dominated the overall kinetic system (Zhang et al. 2018). In addition, the Adam–Bohart model provided a comprehensive and straightforward approach to analyzing column adsorption. However, this model was valid in a limited range of conditions used. Besides, the  $R^2$  was also relatively low, which was between 0.91 and 0.98.

Thomas model has been extensively utilized in explaining the performance of the column adsorption process because this model assumes the internal and external diffusions will not be the limiting step. The graph of  $\ln((C_0/C_t)-1)$  versus  $t$  was plotted (not shown), and the calculated parameter from the graph is shown in Table 5. According to the findings, as the inlet Pb(II) concentration increased, the  $K_{Th}$  value decreased, while  $q_0$  increased. This phenomenon happened as a result of the driving force effect between Pb(II) attached to the  $NH_2/KCC-1$ 's surface and Pb(II) in solution at different inlet concentrations, thus increasing the  $q_0$  value. Besides, the  $R^2$  was also high, which was between 0.91 and

0.99. Therefore, the Thomas model was more suitable to describe this column adsorption. Zhao et al. (2020) also reported the same for Pb(II) adsorption onto MOFs-DHAQ.

## Conclusion

This study successfully functionalized KCC-1 derived from rice husk ash (RHA) with an amino group to produce  $NH_2/KCC-1$ . According to response surface methodology (RSM), the optimal condition was discovered to be at an initial Pb(II) concentration ( $X_1$ ) of 307 mg/L, adsorbent dosage ( $X_2$ ) of 2.43 g/L, and time ( $X_3$ ) of 114 min, with Pb(II) removal ( $Y$ ) of 90.1% (predicted) and 91.2% (actual). The Pareto chart revealed that the significant effect was an initial Pb(II) concentration ( $X_1$ ), while the minor effect was the interaction between adsorbent dosage and time ( $X_2 X_3$ ).  $NH_2/KCC-1$  had the prospect of being employed in multiple cycles with adsorption performance of (91.2–67.3%) and desorption performance of (77.7–51.9%). According to the column adsorption study, the adsorption process showed good performance at a lower flow rate (1 mL/min) and higher bed height (20 cm). Additionally, the adsorption process was well-fitted Thomas model, and was presented by a high correlation coefficient ( $R^2$ ) of 0.91 to 0.99. In summary, the  $NH_2/KCC-1$  adsorbent performed well in Pb(II) adsorption and was effectively used in both batch and column adsorption systems. The successful preparation of  $NH_2/KCC-1$  derived from RHA significantly revealed the ability of agricultural wastes as a silica precursor, which could minimize solid waste pollution and promote sustainable development. With these advantages, further studies can be conducted to discover the effectiveness of other silica-rich solid wastes as silica's precursors in synthesizing KCC-1 or other mesoporous silica families.

**Table 5** The parameter of Adam–Bohart and Thomas models

Adam–Bohart model						
$C_0$ (mg/L)	$Z$ (cm)	$Q$ (mL/min)	$U_0$ (cm/min)	$K_{AB}$ (L/mg min)	$N_0$ (mg/L)	$R^2$
100	30	1	0.1111	2.40E–05	64.1985	0.9193
200	30	1	0.1111	1.83E–05	78.9556	0.9885
300	30	1	0.1111	1.11E–05	164.5113	0.9360
400	30	1	0.1111	8.40E–06	548.6678	0.9430
500	30	1	0.1111	5.30E–06	642.9420	0.9384
Thomas model						
$C_0$ (mg/L)	$m$ (g)	$Q$ (mL/min)	$K_{Th}$ (mL/min mg)	$q_0$ (mg/g)	$R^2$	$C_0$ (mg/L)
100	2.1	1	4.5251E–05	834.50	0.9176	100
200	2.1	1	3.0270E–05	8145.80	0.9911	200
300	2.1	1	1.2570E–05	12,781.65	0.9393	300
400	2.1	1	1.0101E–05	44,123.07	0.9520	400
500	2.1	1	7.2277E–06	29,292.21	0.9390	500

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## Declarations

**Conflict of interest** There is no conflict of interest.

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