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Continuous Adsorption of Cd^{2+} and Cr (VI) Using a KOH-activated Corn Cob Carbon-Coal Fly Ash Composite

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Abstract

Industrial wastewater containing divalent cadmium (Cd^{2+}) and hexavalent chromium (Cr (VI)) poses a serious environmental threat because of the toxicity, persistence, and bioaccumulative nature of these heavy metals. This study aimed to evaluate the performance of a composite adsorbent, prepared from KOH-activated corn cob carbon (CCAC) and calcined coal fly ash (FA) at a mass ratio of 5:1, for the continuous adsorption of Cd^{2+} and Cr (VI). The adsorbent was characterized using Fourier Transform Infrared (FTIR) spectroscopy, while continuous adsorption experiments were performed in an up-flow acrylic column at an initial metal concentration of 100 mg/L, evaluating the effect of flow rate (5–25 mL/min) on Cd^{2+} removal and comparing it with Cr (VI) removal from real industrial wastewater at 5 mL/min. FTIR analysis confirmed that KOH activation and calcination modified the surface functional groups of the CCAC and FA, respectively, increasing the availability of oxygen-containing active sites. The composite adsorbent achieved a high and consistent Cd^{2+} removal efficiency of 91.13–91.51%, with an adsorption capacity of 4.18–4.19 mg/g, across all tested flow rates. In contrast, Cr (VI) removal was very low (0.797%), attributed to electrostatic repulsion between the anionic Cr (VI) species and the negatively charged adsorbent surface at near-neutral pH, as well as competition from co-existing ions in the real wastewater matrix. These findings indicate that the FA/CCAC composite is a promising, low-cost, waste-derived adsorbent for continuous Cd^{2+} removal, while further surface modification or pH adjustment is required to enhance its performance for Cr (VI) removal.

Keywords: *Cadmium, Chromium (VI), Continuous adsorption, Corn cob activated carbon, Coal fly ash*.

INTRODUCTION

The issue of industrial wastewater has attracted the attention of researchers over the past few decades as the volume of waste generated by various industrial sectors, such as the textile, pharmaceutical, leather tanning, electroplating, and aerospace industries, has increased. Wastewater from these sectors generally contains various contaminants, especially heavy metals, such as divalent cadmium (Cd^{2+}) and hexavalent chromium (Cr (VI)). In addition, heavy metals such as Cd^{2+} and Cr (VI) are also a concern because they are toxic, persistent, and easily accumulate in organisms. Exposure to Cd^{2+} has been reported to cause growth disturbances, reproductive issues, and gill damage in aquatic biota (Davidova et al., 2024), while Cr (VI), which has high mobility in water, can trigger oxidative stress, tissue damage, reproductive disorders, and even increase the mortality of aquatic organisms (Kamila et al., 2023). These various impacts highlight the importance of wastewater treatment before it is discharged into the environment.

One of the methods that has been widely developed to remove synthetic dyes and heavy metals is adsorption because it has high efficiency, relatively low operational costs, and a simple process. Additionally, continuous adsorption systems are more representative for application in industrial-scale wastewater treatment because they can operate continuously. The success of this process is greatly influenced by the characteristics of the adsorbent used. Activated carbon from corn cobs is a potential adsorbent because it is derived from abundant biomass waste and contains 43.42% carbon,

which can increase to 85.58% after the activation process, thereby supporting its potential as a carbon-based adsorbent material (Bavaresco et al., 2021). On the other hand, coal fly ash has considerable potential as a low-cost waste-derived material because it is primarily composed of SiO_2 and Al_2O_3 with varying amounts of CaO and can be converted into value-added adsorbent materials such as zeolites (Feng et al., 2018). Therefore, the utilization of corn cob-derived carbon and coal fly ash provides an opportunity to develop waste-derived adsorbents for wastewater treatment while supporting waste-to-resource approaches.

Earlier research has shown that activated carbon made from corn cobs can effectively remove different types of pollutants. Medhat and their team in 2021 looked at using activated carbon made from corn cobs to remove methylene blue from water. Then, Kumaravel and their group in 2024 tested chemically treated corn cob carbon to take out brilliant green dye from wastewater. Liu and their team in 2020 made carbon from corn cobs and used potassium hydroxide to activate it. They tested this activated carbon for removing mercury ions from water, showing that using KOH activation can enhance the ability of carbon made from corn cobs to absorb pollutants. Coal fly ash has also been studied as a cheap material to remove heavy metals, and researchers have tried modifying it to make it better at absorbing these metals (Sireesha et al., 2025). In addition, Liu and their team in 2023 made a composite material using biochar, which they created by heating corn straw and fly ash together, and they tested how well it works for removing Cd^{2+} ions. These studies show that both materials made from corn cobs and fly ash can be effective in removing contaminants.

However, most studies have looked at single adsorbents, modified fly ash, or various biomass-fly ash mixtures, but using KOH-activated corn cob carbon mixed with coal fly ash as a combined adsorbent has not been studied much. Even though activated carbon is used in both batch and continuous adsorption systems (Budianto et al., 2023), there is not enough research on how well a KOH-activated corn cob carbon–coal fly ash composite works in continuous-flow situations. The way Cd^{2+} and Cr (VI) attach to surfaces is likely different because Cd^{2+} is a positively charged ion, while Cr (VI) mainly exists as a negatively charged ion in water. So, checking how well the composite works with both metal types is key to understanding both its strengths and its weaknesses when it comes to removing heavy metals.

So, this study creates a new type of adsorbent made from KOH-activated corn cob carbon and coal fly ash, and it checks how well this material works for removing Cd^{2+} and Cr (VI) from water continuously. The unique part of this study is testing this waste-made composite under a steady flow of water to remove both positively charged Cd^{2+} and negatively charged Cr (VI), which helps understand how well the composite works and its limits when dealing with metals that have different chemical properties. The study also checks how well the composite removes pollutants and how much it can absorb under various flow rates to see if it can be used for ongoing wastewater treatment.

METHODS

Materials and Equipment

The raw material for activated carbon in the form of corn cobs was obtained from Toko Berkah Jagung, Batujajar, Bandung, while coal fly ash was obtained from PT Safilindo Permata, Bandung Regency, West Java, Indonesia. The wastewater used consisted of Cr (VI)-containing wastewater obtained from PT Jabil Circuit Indonesia, Bandung, and a synthetic Cd^{2+} solution prepared from cadmium nitrate tetrahydrate ($Cd(NO_3)_2{\cdot}4H_2O$). Other chemicals used in this study included potassium hydroxide (KOH) as the activating agent for activated carbon, nitric acid (HNO_3), potassium dichromate ($K_2Cr_2O_7$), and other analytical-grade (PA) reagents. The main instruments used were a UV-Vis spectrophotometer, an Atomic Absorption Spectrophotometer (AAS), Fourier Transform Infrared Spectroscopy (FTIR), and a Energy Dispersive X-ray Spectroscopy (EDS).

Adsorbent Preparation

The fly ash was washed thoroughly with distilled water until a clear filtrate was obtained, followed by sun drying to remove excess moisture and oven drying at 105 °C for 24 h (Tiadi et al., 2020). The dried material was cooled in a desiccator and calcined at 500 °C for 1 h (D. Liu et al., 2019). After cooling to room temperature, the calcined fly ash was ground and sieved to obtain a particle size of 325 mesh before being stored in a sealed container for further use.

Similarly, corn cobs were washed with distilled water until a clear filtrate was obtained, sun-dried, and further dried in an oven at 105 °C for 1 h (Agustina et al., 2023). The dried corn cobs were cut into approximately 5 mm pieces (Zegeye et al., 2023) and carbonized in a furnace at 600 °C for 3 h (Aritorang et al., 2025). After cooling in a desiccator, the resulting biochar was chemically activated using 1 M KOH at a solid-to-liquid ratio of 1:10 (w/v). The suspension was heated at 60 °C under continuous stirring for 30 min (Sankaranarayanan et al., 2025), followed by impregnation at room temperature for 25 h (Zegeye et al., 2023). The activated carbon was then washed with 0.1 M HCl and

distilled water until the filtrate reached a neutral pH (6–7), oven-dried at 105 °C for 24 h (Zegeye et al., 2023), ground, sieved to the desired particle size, and stored in a sealed container until use (Agustina et al., 2023).

Continuous Adsorption Experiments

The continuous adsorption experiments were carried out using an acrylic column with a height of 48 cm and an inner diameter of 3.5 cm (Hummadi et al., 2022). The column was packed with 6 g of a composite adsorbent consisting of coal fly ash (FA) and KOH-activated corn cob carbon (CCAC) at a mass ratio of 5:1 with a particle size of 90 mesh. The influent solution was introduced into the column in an up-flow mode using a peristaltic pump (Golander BT50S-1, Version V126S110, AC 100–240 V, 50/60 Hz). The initial concentration of both Cd^{2+} and Cr (VI) was maintained at 100 mg/L. The adsorbent composition, adsorbent mass, particle size, and initial metal concentration were kept constant throughout the experiments. For Cd^{2+} adsorption, the flow rate was varied at 5, 10, 15, 20, and 25 mL/min to evaluate its effect on the continuous adsorption performance (Dominguez et al., 2024) and the duration is 60 minutes (Yaashikaa and A. Saravanan., 2026). Meanwhile, Cr (VI) adsorption was carried out only at a flow rate of 5 mL/min under identical operating conditions for comparison with Cd^{2+} adsorption. Effluent samples (20 mL) were collected every 5 min (Hummadi et al., 2022).

Adsorbent Characterization (FTIR)

The surface functional groups of the activated carbon before and after KOH activation, as well as the coal fly ash before and after calcination, were characterized using Fourier Transform Infrared (FTIR) spectroscopy over the wavenumber range of 4000–400 cm^{-1}. The obtained spectra were used to identify changes in the surface functional groups after the activation and calcination processes.

Concentration Analysis and Calculation

The residual Cd^{2+} concentration was determined using atomic absorption spectrophotometry (AAS) according to SNI 6989.16:2009 using atomic absorption spectrophotometry (AAS) at a wavelength of 228.8 nm, whereas the residual Cr (VI) concentration was determined according to SNI 6989.71:2009 using the diphenylcarbazide method at 530–540 nm using UV–Visible spectrophotometry. The experiments were continued for 60 min or until the adsorbent reached saturation. The experimental data were used to calculate the removal efficiency and adsorption capacity, as well as to construct the breakthrough curves. The removal percentage and adsorption capacity were calculated using Equations (1) and (2):

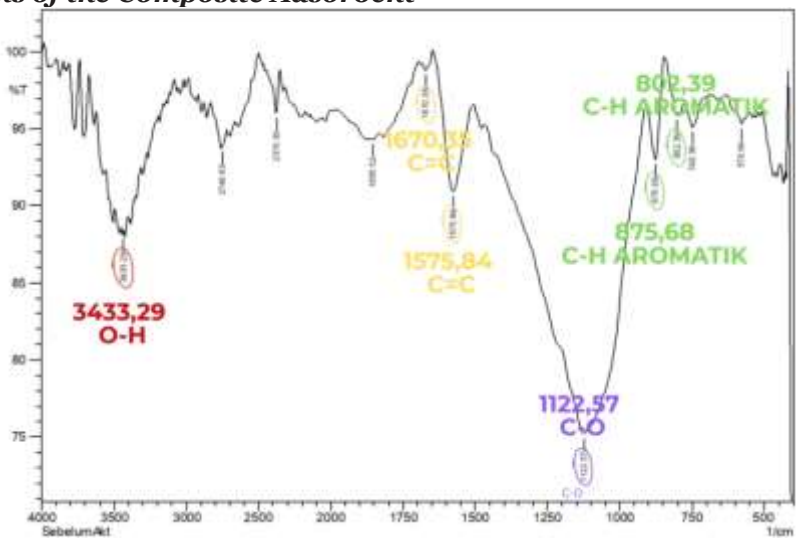
$$\%\text{Removal} = \left(\frac{C_0 - C_e}{C_0}\right) \times 100\% \quad (1)$$

$$q_e = \frac{(C_0 - C_e) \times V}{m} \quad (2)$$

where C_0 and C_e are the initial and equilibrium adsorbate concentrations (mg/L), V is the solution volume (L), and m is the adsorbent mass (g).

RESULTS AND DISCUSSION

FTIR Analysis of the Composite Adsorbent



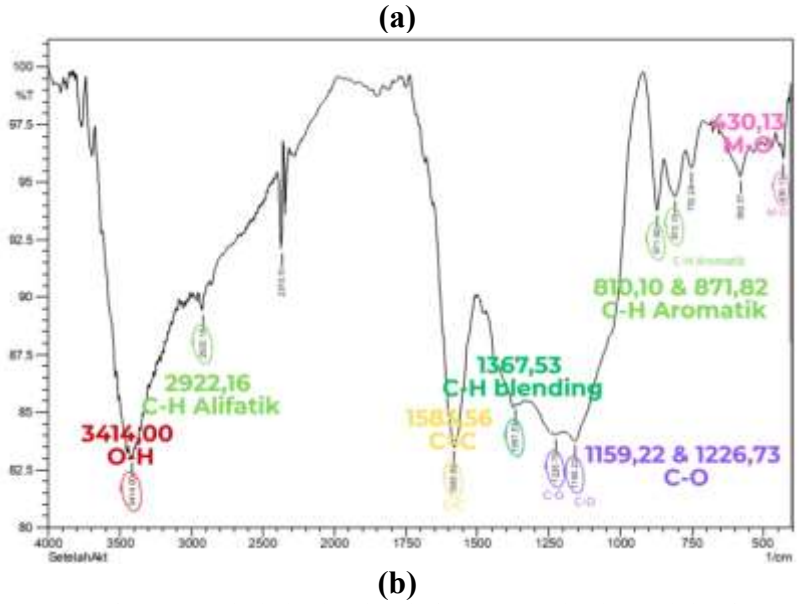


Figure 1. FTIR spectra of (a) corn cob-derived carbon before KOH activation and (b) after KOH activation

Table 1. Assignment of FTIR absorption bands of corn cob-derived carbon before and after KOH activation

Functional group	Before activation (cm^{-1})	After activation (cm^{-1})	Observation	Interpretation
O–H stretching	3433.29	3414.00	Shift to lower wavenumber	Change in hydrogen bonding after KOH activation
Aliphatic C–H stretching	-	2922.16	New band appeared	Formation/exposure of aliphatic carbon groups
Aromatic C=C stretching	1670.35; 1575.84	1583.56	Slight shift	Aromatic carbon framework retained
Alkyl bending	-	1367.53	New band appeared	Surface structural modification
C-O stretching	1122.57	1226.73; 1159.22	Shift and reduced intensity	Modification of oxygen-containing functional groups
Aromatic C-H blending	875.68; 802.39	871.82; 810.10	Slight shift	Changes in aromatic environment
Metal-oxigen	-	430.13	New band appeared	Interaction between KOH and carbon surface.

Figure 1 and Table 1 present the FTIR spectra and the corresponding functional group assignments of corn cob-derived carbon before and after KOH activation. Prior to activation, the carbon exhibited characteristic absorption bands corresponding to O–H, aromatic C=C, C–O, and aromatic C–H functional groups. After activation, noticeable shifts in the O–H, aromatic C=C, and C–O bands, together with the appearance of new absorption bands assigned to aliphatic C–H, alkyl bending, and metal–oxygen (M–O) vibrations, indicate that KOH activation successfully modified the surface chemistry of the carbon. These changes are associated with dehydration, restructuring of the carbon matrix, and pore development during chemical activation (Karaer Yağmur., 2026).

The modification of oxygen-containing functional groups, particularly hydroxyl and C–O groups, is expected to increase the availability of active adsorption sites on the carbon surface. These functional groups can facilitate electrostatic attraction, ion exchange, and surface complexation with dissolved metal ions, while the preserved aromatic carbon framework provides structural stability during adsorption. Therefore, the FTIR results suggest that KOH activation enhanced the chemical properties of the activated carbon, which is expected to contribute to the continuous adsorption performance of the FA/CCAC composite adsorbent discussed in the following section.

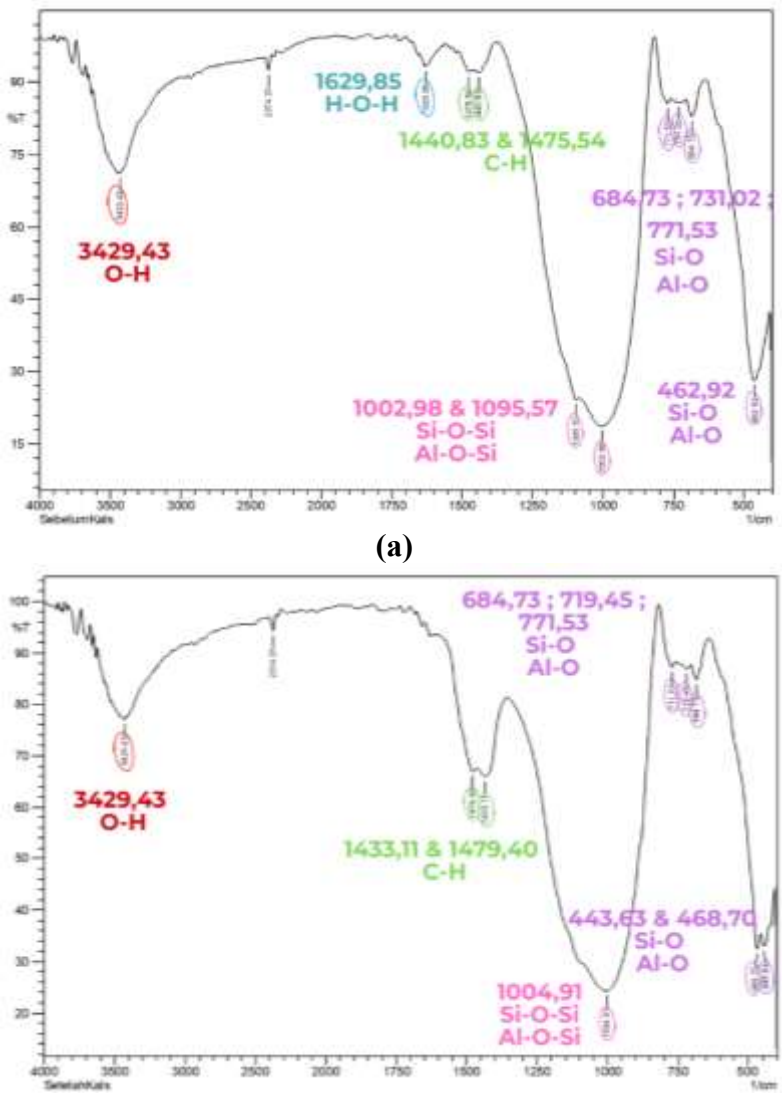


Figure 2. FTIR spectra of (a) coal fly ash before calcination and (b) after calcination

Table 2. Functional group assignments of coal fly ash before and after calcination

Functional group	Before activation (cm^{-1})	After activation (cm^{-1})	Observation	Interpretation
O–H stretching	3429.43	3429.43	Reduced intensity	Removal of physically adsorbed moisture and changes in hydroxyl environment
H–O–H bending	1629.85	-	Disappeared	Elimination of physically adsorbed water after calcination
C–H bending	1475.54; 1440.83	1479.40; 1433.11	Slight shift	Minor modification of residual organic species
Si–O–Si / Al–O–Si stretching	1095.57; 1002.98	1004.91	Merged into a sharper band	Structural rearrangement of the aluminosilicate framework
Si–O / Al–O bending	771.53; 731.02; 684.73	771.53; 719.45; 684.73	Minor shift	Rearrangement of Si–O and Al–O bonding environment
Si–O / Al–O bending	462.92	468.70; 443.63	Peak splitting	Modification of the aluminosilicate

	structure after calcination

Figure 2 and Table 2 present the FTIR spectra and the corresponding functional group assignments of coal fly ash before and after calcination. Prior to calcination, the fly ash exhibited characteristic absorption bands assigned to O–H stretching, H–O–H bending, C–H bending, and Si–O–Si/Al–O–Si vibrations, confirming the presence of hydroxyl groups, adsorbed water, and an aluminosilicate framework. After calcination, the H–O–H bending band disappeared, while slight shifts were observed in the C–H and Si–O–Si/Al–O–Si bands. In addition, the absorption band at 462.92 cm^{-1} split into two bands at 468.70 and 443.63 cm^{-1}, indicating structural rearrangement of the aluminosilicate framework following thermal treatment.

The disappearance of the H–O–H band indicates the removal of physically adsorbed water, whereas the changes observed in the aluminosilicate bands suggest a more ordered and accessible surface structure after calcination. These modifications are expected to expose additional adsorption sites associated with silanol (Si–OH) and aluminol (Al–OH) groups, thereby improving the interaction between the fly ash surface and dissolved metal ions. Consequently, calcined fly ash is expected to complement the KOH-activated carbon by providing additional active sites and enhancing the overall adsorption performance of the FA/CCAC composite adsorbent during the continuous adsorption process. FTIR analysis of activated carbon.

EDS Analysis of the Composite Adsorbent

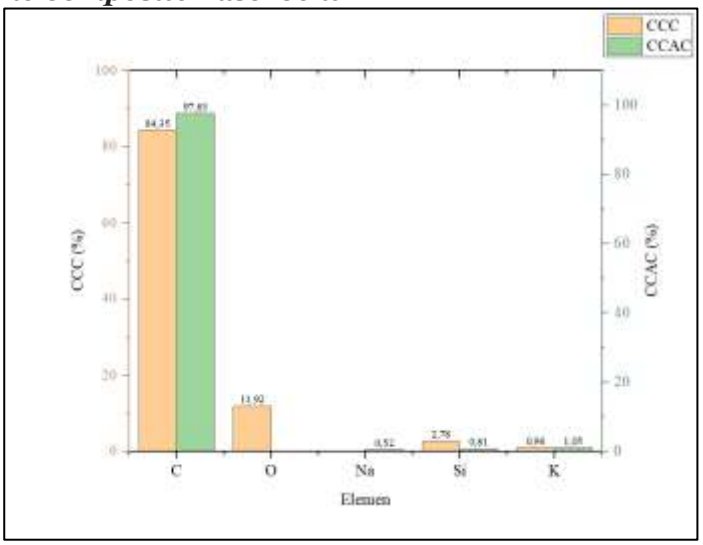


Figure 3. EDS of corn cob-derived carbon before KOH activation (CCC) and after KOH activation (CCAC)

The data from EDS in Figure 3 also back up the explanation regarding the elements present. The carbon content went up from 84.45% in the raw carbon to 97.63% in the activated carbon, showing that the amount of carbon increased after the carbonization and activation steps. At the same time, the amount of oxygen went down from 11.92% to a level that could not be detected, and the amount of silicon dropped from 2.78% to 0.81%. After activation and washing, there is less of the oxygen-containing parts and silica-based inorganic parts on the surface that was studied. The potassium level went up a little, from 0.96% to 1.05%, and this was probably because some KOH was left behind after washing. This result is consistent with Mopoung et al. (2015), where they showed that the main elements in the activated carbon made from biomass using KOH were carbon, oxygen, silicon, and potassium. The EDS did not detect any oxygen, but the FTIR still showed oxygen-containing groups like –OH.This isn't a problem because EDS tells us about the elements present, while FTIR looks for specific chemical bonds that indicate certain groups.

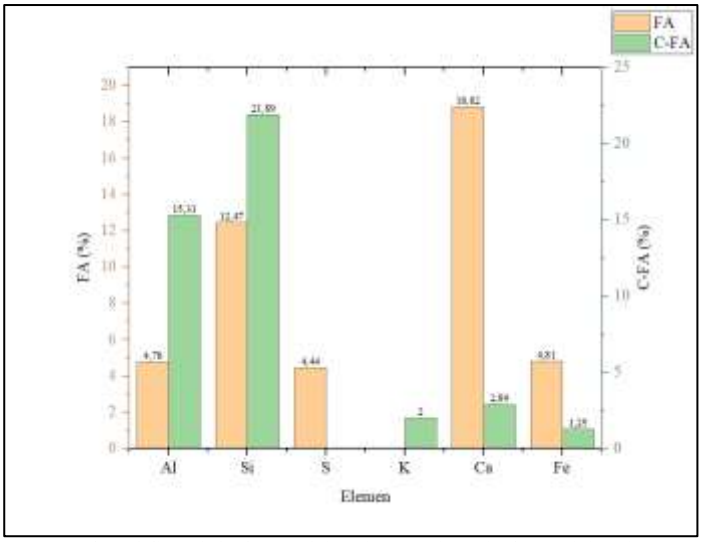


Figure 4. EDS of coal fly ash before calcination (FA) and after calcination (C-FA)

At the same time, the makeup of the fly ash after it was heated in Figure 4 showed a different kind of pattern. The amount of silicon went up from 12.47% to 21.89%, and aluminum increased from 4.78% to 15.31%. Calcium went down from 18.82% to 2.89%. Sulfur was no longer found, going from 4.44% to 0%. Iron also decreased, from 4.81% to 1.25%. The rise in the amounts of silicon and aluminum, along with a drop in other elements, shows that the surface makeup of the fly ash changed after it was heated. The loss of sulfur and the drop in calcium and iron could mean that some parts with these elements were changed or taken away during heating. This result matches what Kutchko and Kim (2006) found, stating that coal fly ash is mostly made up of amorphous aluminosilicate materials, and elements like calcium and iron can be found in different types of minerals. After calcination, sodium and potassium showed up, which weren't found in the original fly ash. This might be because the mix of elements changed or because some surfaces became exposed that EDS didn't pick up before.

The different amounts of elements in the activated carbon and fly ash show that they have useful traits that work well together when making the FA/CCAC composite. The activated carbon was mostly made up of carbon, with 97.63% of its content being carbon. The calcined fly ash, on the other hand, had a higher amount of silicon and aluminum, with silicon making up 21.89% and aluminum 15.31%. So, the two materials have different elements, with one coming from activated carbon as a carbon structure and the other from fly ash containing aluminosilicate parts. These materials work together to make them useful as a composite adsorbent, and the surface features come from both parts.

Continuous Adsorption

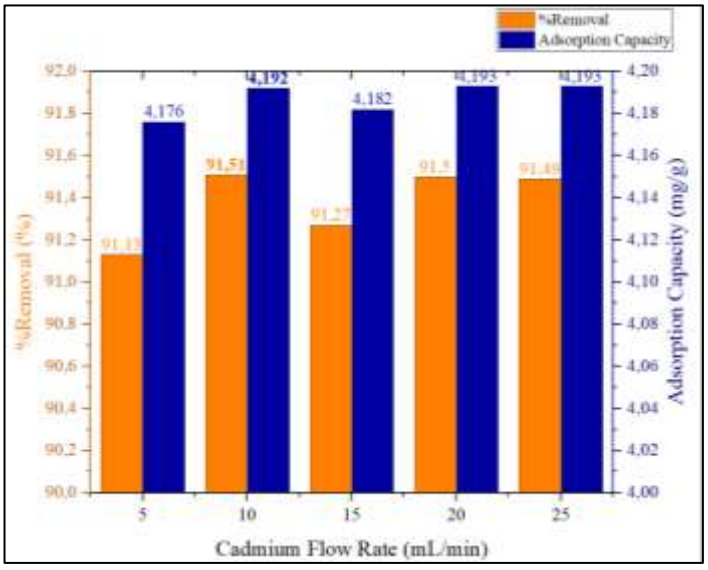


Figure 5. %Removal and adsorption capacity of cadmium at varying flow rate

Figure 5 shows that the removal efficiency of Cd^{2+} remained consistently high, ranging from 91.13% at 5 mL/min to 91.51% at 10 mL/min, and stabilizing at approximately 91.5% at 15–25 mL/min, while the corresponding adsorption capacity increased only marginally from 4.176 to 4.193 mg/g. This near-constant performance across a five-times change in flow rate indicates that, within the range and 60-minute duration examined, the FA/CCAC composite bed still possessed substantial unused adsorption capacity and had not yet approached exhaustion, so that variation in flow rate exerted little influence on the overall removal efficiency.

This interpretation is supported by the breakthrough curves in Figure 6, where the C_t/C_o ratio for Cd^{2+} remained low and comparatively flat (0.082–0.092) throughout the 60-minute run for all five flow rates, without the characteristic S-shaped rise associated with the mass transfer zone approaching the column outlet. In fixed-bed adsorption, an increase in flow rate is generally expected to shorten the empty-bed residence time and cause earlier breakthrough, since the fluid spends less time in contact with the adsorbent (Samanth et al., 2024). However, a higher flow rate can simultaneously reduce the thickness of the liquid boundary layer surrounding the adsorbent particles, thereby lowering external mass transfer resistance and partly compensating for the shorter contact time (Samanth et al., 2024). Because the mass transfer zone in the present system had evidently not yet reached the end of the bed within the tested period, this compensating effect appears to explain why the removal efficiency of Cd^{2+} did not decline noticeably as the flow rate was increased from 5 to 25 mL/min.

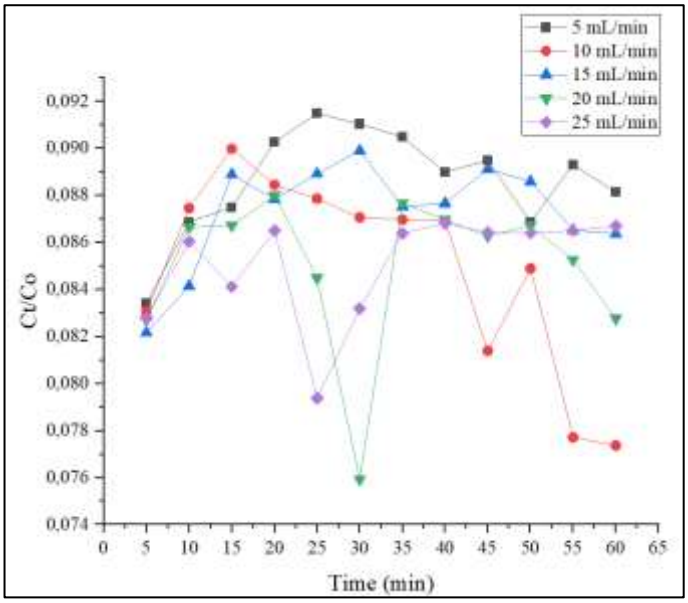


Figure 6. Breakthrough curve of cadmium adsortion at varying flow rate

The high and stable removal of Cd^{2+} is consistent with the FTIR results discussed in the preceding section. The oxygen-containing functional groups generated on the CCAC surface after KOH activation (hydroxyl and carboxylate species) and the silanol/aluminol groups exposed on the calcined FA surface provide negatively charged sites that readily interact with the divalent Cd^{2+} cation through electrostatic attraction, cation exchange, and surface complexation (Ge et al., 2024). Because Cd^{2+} exists as a simple hydrated cation across a wide pH range, its uptake is comparatively insensitive to the small pH fluctuations that may occur along the column, which further supports the stable

removal performance observed at all tested flow rates.

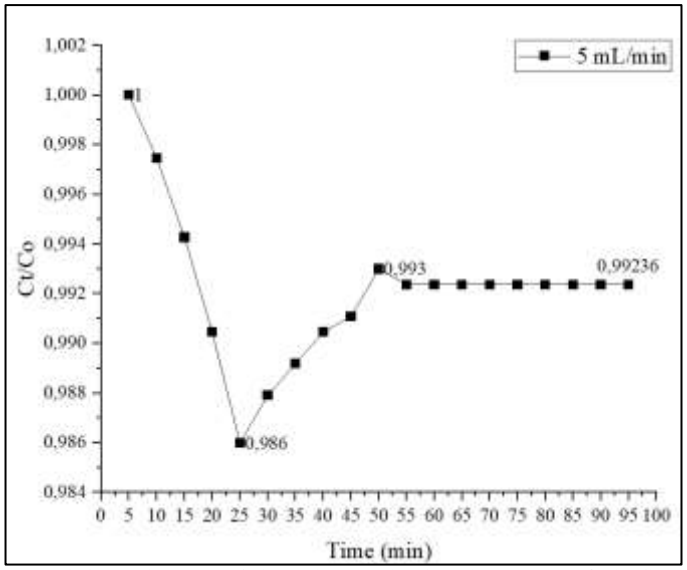


Figure 7. Breakthrough curve of chromium (VI) adsortion at 5 mL/min flow rate

A very different behavior was seen for Cr(VI), as shown in Figure 7. The Ct/Co ratio began close to 1, dropped just a little to a minimum of 0.986 at 25 minutes, which is about 1.4% removal, and then went up to a steady level between 0.992 and 0.993 after 55 minutes, which means 0.7 to 0.8% removal. So, the column showed a very quick start in removing Cr(VI), but in contrast, it kept removing Cd^{2+} at over 91% efficiency under the same conditions.

This big difference in behavior is probably because of how Cr(VI) changes with pH and the way the adsorbent's surface charges work. In water solutions, chromium in the +6 oxidation state mainly exists as oxygen-containing ions, such as $HCrO_4^-$, CrO_4^{2-}, and $Cr_2O_7^{2-}$, and the form it takes depends on the acidity and amount of the solution. Electrostatic adsorption works best when the surface of the adsorbent is positively charged and has protons, which happens more often in acidic conditions (Ali et al., 2023). For coal-fly-ash-based adsorbents, the point of zero charge has been reported at around pH 7–8; below this value the surface is positively charged and favors Cr (VI) adsorption, whereas near or above it the surface becomes negatively charged and repels Cr (VI) oxyanions (Umejuru et al., 2023). Since the Cr (VI) wastewater used in this study was treated without acidification, the FA/CCAC surface, rich in the same oxygen-containing hydroxyl and silanol/aluminol groups that favor Cd^{2+} uptake is expected to remain negatively charged, resulting in electrostatic repulsion toward the anionic Cr (VI) species and, consequently, the very low removal efficiency observed. Figure 8 below shows the average removal percentage was 0.797%, and the adsorption capacity was 0.061 mg/g.

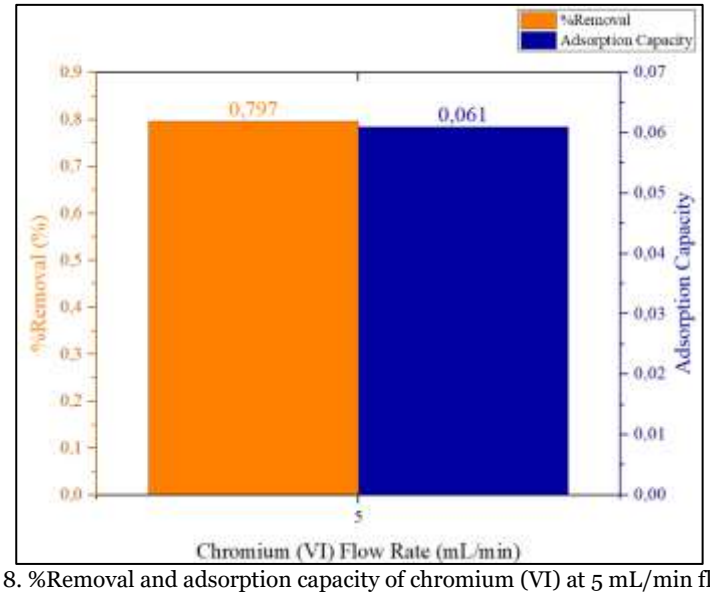


Figure 8. %Removal and adsorption capacity of chromium (VI) at 5 mL/min flow rate

An additional contributing factor is that the Cr (VI) solution used in the continuous column was

real industrial wastewater obtained from PT Jabil Circuit Indonesia, rather than a single-solute synthetic solution as used for Cd^{2+}. Real wastewater often has other ions and chemicals that can take up or cover the spots where heavy metals usually stick, which makes it harder to remove those metals effectively compared to simple lab-made solutions that only have the metal ion (Yadav et al., 2025). This matrix effect, combined with the unfavorable electrostatic interaction mentioned earlier, probably made it even harder for the composite adsorbent to take up the limited amount of Cr (VI) it was already able to absorb.

Taken together, these results indicate that the FA/CCAC composite adsorbent developed in this study performs effectively for the continuous removal of Cd^{2+}, achieving high and flow-rate-independent removal efficiencies above 91% under the tested conditions, but is not yet suitable for the simultaneous removal of Cr (VI) from untreated, near-neutral-pH industrial wastewater. This finding contrasts with the complementary removal mechanism anticipated for the two metals, and suggests that pH adjustment of the influent, or further functionalization of the adsorbent surface with cationic or amine-based moieties to promote anion exchange, would be necessary to enable effective simultaneous Cd^{2+} and Cr (VI) removal in future applications of this composite adsorbent.

CONCLUSIONS

A composite adsorbent prepared from KOH-activated corn cob activated carbon (CCAC) and calcined coal fly ash (FA) at a mass ratio of 5:1 was successfully evaluated for continuous adsorption of Cd^{2+} and Cr (VI). FTIR analysis confirmed that KOH activation and fly ash calcination enhanced the adsorbent surface by increasing oxygen-containing functional groups, including –OH, C–O, Si–OH, and Al–OH, which provide active sites for adsorption.

The FA/CCAC composite exhibited excellent performance for Cd^{2+} removal, achieving 91.13–91.51% removal efficiency with adsorption capacities of 4.18–4.19 mg g^{-1}, while the breakthrough curve indicated that column exhaustion had not yet been reached. In contrast, Cr (VI) removal was very limited, with only 0.797% removal efficiency and an adsorption capacity of 0.061 mg/g, as the breakthrough curve rapidly approached saturation. This poor performance is attributed to electrostatic repulsion between anionic Cr (VI) species and the negatively charged adsorbent surface under near-neutral pH, together with competition from co-existing ions in real wastewater.

These findings demonstrate that the adsorption performance of the FA/CCAC composite depends not only on surface functional groups but also on the chemical speciation and ionic charge of the target metal. The composite shows strong potential as a low-cost, waste-derived adsorbent for continuous Cd^{2+} removal, whereas further optimization, such as pH adjustment or surface functionalization, is required to improve Cr (VI) adsorption.

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