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ORIGINAL PAPER

Polyaniline-coated biochar: Synthesis, characterization, and Cr(VI) adsorption studies

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Abstract

Polyaniline (PANI) is a promising adsorbent for heavy metal removal due to its high conductivity, large surface area, cost-effective synthesis, and environmental stability. Biochar, derived from organic waste, also shows potential for pollutant removal. In this study, a polyaniline-biochar composite (PANI-NSA/BC) was synthesized and assessed for Cr(VI) removal from aqueous solutions. SEM analysis showed rod-like polyaniline structures distributed within the biochar matrix. Batch adsorption studies were performed to evaluate the effects of adsorbent dosage, polyaniline-to-biochar ratio, and solution pH. Cr(VI) removal efficiency decreased with biochar fraction (maximum at 25% biochar) and increased with composite dosage, (maximum at 40 mg). The process was highly pH-dependent, achieving maximum removal at pH 2. These results demonstrate the potential of PANI-NSA/BC for Cr(VI) remediation in water treatment. Future work will focus on scaling up the synthesis, optimizing operational parameters, and exploring the composite's regeneration and reuse performance under continuous flow conditions.

Introduction

The widespread presence of chromium contamination in aqueous environments poses a significant threat to both ecosystems and human health. Chromium, particularly in its hexavalent form (Cr (VI)), is highly toxic, carcinogenic, mutagenic, and genotoxic to human beings and animals [1, 2]. It is non-biodegradable under the natural environmental conditions; hence, it stays in the environment for a long time [1]. Therefore, it is necessary to develop effective, sustainable, and low-cost adsorbents for the remediation of Cr(VI)-contaminated water.

Polyaniline (PANI), a well-known conducting polymer, has attracted extensive interest in environmental applications due to its high adsorption capacity, tunable surface properties, and redox-active nature [3]. It is environmentally stable, easy to synthesize, and has a high affinity for heavy metals thanks to its many imine and amine functional groups within the polymer chains [2, 4]. These properties enable it to adsorb and convert Cr(VI) to less toxic trivalent chromium (Cr(III)). However, the standalone application of PANI is limited by its low mechanical strength and high agglomeration tendency, which reduces its long-term reusability and adsorption efficiency. To overcome these challenges, many researchers have explored composites of polyaniline with other materials. Bhaumik [5] and her team explored the use of polypyrrole and polyaniline composite for Cr(VI) removal and observed a maximum adsorption capacity of 227 mg/L. In our previous study [6], we explored how polyaniline and nickel ferrite composite can be used to remove and reduce Cr(VI) to Cr(III). In an interest to find composites that can support column studies, we decided to explore a composite of polyaniline and biochar in this study.

Biochar is a carbon-rich material derived from biomass pyrolysis in an anaerobic environment [7, 8]. Since biochar can easily be derived from waste, it is considered low-cost and environmentally safe [9]. It has extensive applications such as environmental protection and agricultural production due to its nitrogen and carbon groups [10]. In the past ten years, significant progress has been made in studying biochar's use as an eco-friendly adsorbent for water treatment. This research highlights its potential as a cost-effective and sustainable alternative, offering enhanced removal efficiency

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compared to earlier methods [10]. It offers a promising solution as a support material for PANI due to its porous structure and high surface area. Biochar not only provides robust and stable support but also contributes to adsorption through functional groups on its surface, synergistically enhancing the composite's removal efficiency.

This study investigates the synthesis and characterization of a polyaniline-biochar (PANI-NSA/BC) composite for Cr(VI) adsorption. Although a similar composite was previously studied by Herath et al. [11], their work employed a different synthesis method and biochar source. In contrast, our study is preliminary in nature and is specifically limited to the synthesis of the PANI-NSA/BC composite and an initial assessment of its Cr(VI) adsorption performance. The primary objective at this stage is to evaluate the feasibility of the composite for future application in column studies. Initial batch experiments were conducted to assess the removal efficiency of Cr(VI) from aqueous solutions, with a view toward its potential in large-scale water treatment. The findings presented here lay the basis for more detailed investigations of adsorption kinetics, mechanisms, and long-term operational stability under dynamic conditions.

Materials and methods

Materials and methods

Aniline (C_6H_7N), ammonium persulfate ($(NH_4)S_2O_8$), potassium dichromate ($K_2Cr_2O_7$), 2-naphthalene sulfonic acid ($C_{10}H_8O_3S$) and 1,5-Diphenylcarbazide (DPC) were purchased from Sigma-Aldrich. Sulfuric acid (H_2SO_4, 98 wt-%), hydrochloric acid (HCl, 98 wt-%), sodium hydroxide (NaOH), and methanol (CH_3OH) were purchased from Glassworld. Biochar (from pinewood) was commercially sourced from Organics Matter, South Africa.

Composite synthesis

The polyaniline-biochar composite (PANI-NSA/BC) was synthesized using a modified procedure adapted from Long et al. [12]. In brief, biochar pellets were ground using a mortar and pestle to obtain a fine powder. Different amounts of biochar powder, corresponding to final weight fractions ranging from 0.25 to 0.75, were dispersed in 80 mL of deionized water containing 0.416 g of 2-naphthalene sulfonic acid. Subsequently, 0.2 mL of aniline was added to the solution, and the mixture was ultrasonicated for 15 min. After this, 0.456 g of ammonium persulfate - dissolved in 5 mL of deionized water - was gradually introduced into the reaction mixture, followed by an additional 1-minute ultrasonication. The mixture was then left undisturbed for 24 h to complete the polymerization process. The resulting product was thoroughly washed with deionized water and methanol to remove any unreacted species and then dried in an oven at 60°C for 24 h.

Characterization of the composite

Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Spectroscopy (EDS) were employed to analyze the surface morphology and elemental composition of the PANI-NSA/BC composite. These complementary techniques provided valuable insights into the structural features and elemental distribution within the material.

Batch adsorption studies

Batch adsorption experiments were conducted to evaluate the effects of composite dosage, biochar weight fraction, and initial solution pH on Cr(VI) removal efficiency. All tests were performed in a thermostatic water bath shaker set at 200 rpm for 24 h to ensure equilibrium conditions. The initial Cr(VI) concentration was maintained at 50 ppm, and the temperature was held constant at 25 °C. Each experiment was conducted in duplicate using 100 mL glass bottles containing 50 mL of Cr(VI) solution, and average values were reported.

Effect of biochar weight fraction studies

The influence of biochar weight fraction in the composite on adsorption performance was assessed by varying the initial amount of biochar added prior to polymerization (see "Composite synthesis" section). Weight fractions ranging from 0.25 to 0.75 were studied. All tests were conducted at an initial pH of 2, as suggested by Herath et al. [11], using a fixed composite dosage of 40 mg. The percentage removal of Cr(VI) was calculated using Eq. 1

$$\%removal = \frac{C_o - C_e}{C_o} \times 100 \tag{1}$$

Effect of dosage studies

To investigate the effect of composite dosage on Cr(VI) adsorption, the mass of the PANI-NSA/BC composite was varied between 10 mg and 50 mg. Dosages were measured using a digital analytical balance with four-decimal precision. Experiments were conducted at an initial pH of 2 and a fixed biochar weight fraction of 0.5.

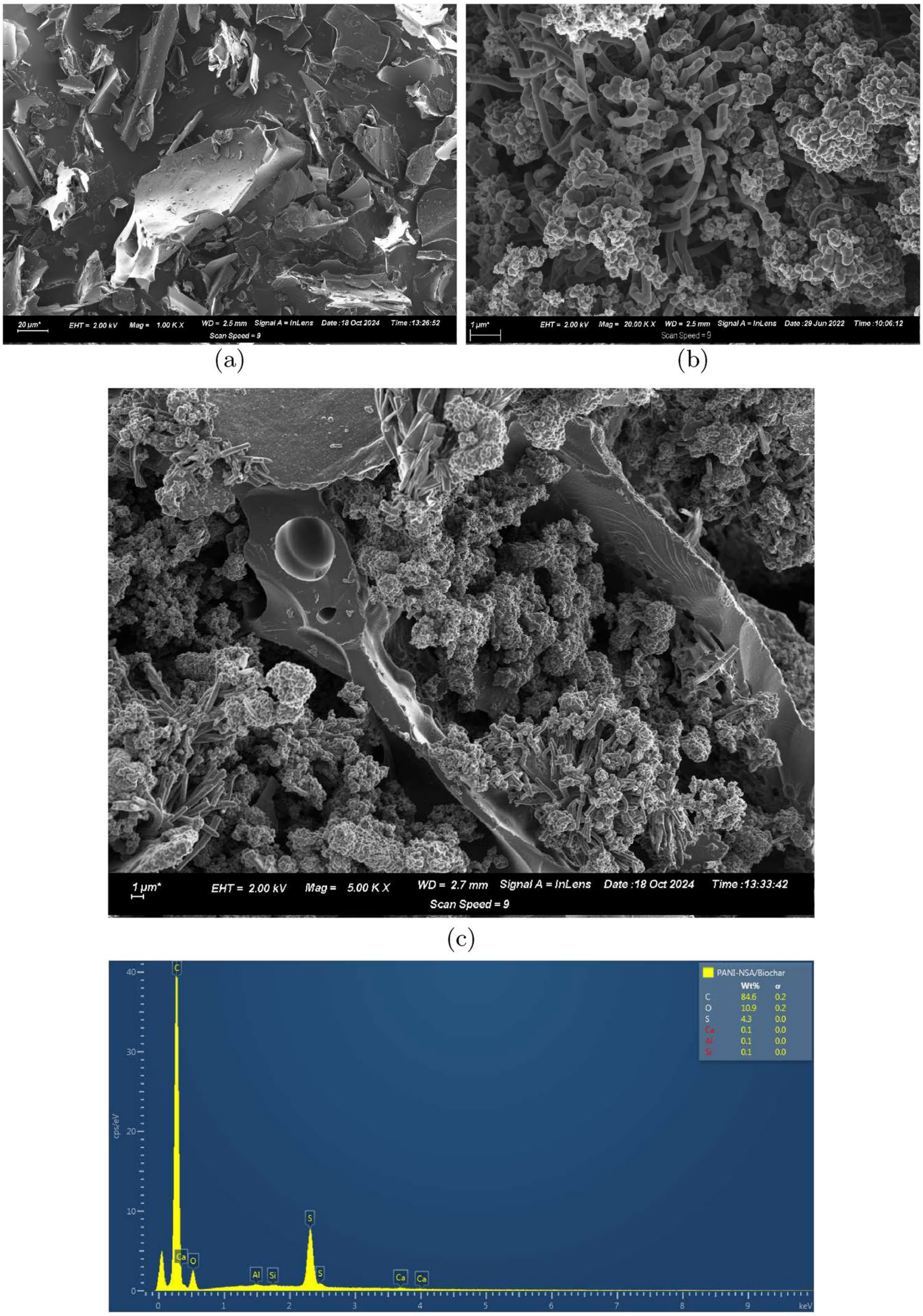
Effect of pH studies

The effect of solution pH on Cr(VI) removal was studied by adjusting the initial pH from 2 to 10 prior to the addition of 40 mg of the composite with a biochar weight fraction of 0.5. pH adjustments were made using 1.0 M HCl and 1.0 M NaOH.

Results and discussion

Characterization

The SEM images of biochar, PANI-NSA, and the PANI-NSA/BC composite are presented in Fig. 1a–c. Pure biochar (Fig. 1a) displays a smooth, compact surface with no visible porosity. In contrast, the composite (Fig. 1c) shows



(a)
(b)
(c)
(d)

Fig. 1 SEM images of pure biochar as purchased (**a**), synthesized PANI-NSA (**b**), synthesized PANI-NSA/BC (**c**), and EDS of the PANI-NSA/BC (**d**) polyaniline chains interwoven throughout the biochar matrix. These chains exhibit the characteristic rod-like morphology of PANI-NSA, as shown in Fig. 1b and previously reported in our earlier work [6]. The retention of this structure within the composite suggests that polymerization in the presence of biochar preserves the native morphology of polyaniline while enhancing its dispersion and potential for adsorption. The morphological contrast between the composite and its individual components supports the successful formation of a polyaniline-biochar composite.

EDS analysis (Fig. 1d) confirms the carbon-rich nature of the composite and detects sulfur, originating from the naphthalene sulfonic acid dopant. This sulfur incorporation enhances both structural stability and functionality, contributing to the observed rod-like features of polyaniline [6].

Additionally, the increase in material weight from 0.2 g of biochar to 0.3268 g after polymerization corresponds to a composition of approximately 39% polyaniline and 61% biochar, further confirming the successful synthesis of the PANI-NSA/BC composite.

Effect of biochar amount in the composite

Fig. 2 illustrates the impact of varying biochar content in the PANI-NSA/BC composite on the Cr(VI) removal efficiency from aqueous solutions. As the proportion of biochar in the composite increases, a clear decline in adsorption efficiency is observed. The highest Cr(VI) removal (≈ 98%) occurs at a composition containing 25% biochar and 75% polyaniline, while the lowest removal (≈ 79%) is recorded at 75% biochar content. This inverse relationship highlights the dominant role of polyaniline in driving the adsorption process.

The observed trend can be attributed to several factors. Firstly, polyaniline contains abundant redox-active functional groups, such as amine and imine moieties, which facilitate both the adsorption and partial reduction of Cr(VI) to the less toxic Cr(III) form [13]. As the proportion of biochar increases, the relative amount of polyaniline–and hence the number of active sites–decreases, leading to reduced interaction with Cr(VI) ions. Secondly, while biochar provides a stable and porous support structure, it contributes less directly to the chemical binding of Cr(VI), especially when used in higher proportions that dilute the polyaniline component.

Moreover, the presence of polyaniline is known to enhance the structural integrity and stability of the composite, improving its dispersion and surface accessibility. This structural synergy likely explains the superior adsorption performance observed at lower biochar loadings. Similar findings have been reported in studies where conductive polymers dominate the adsorption mechanism due to their affinity for metal ions [14].

These results underscore the importance of optimizing the polyaniline-to-biochar ratio when designing composites for water treatment applications. While increasing polyaniline content improves performance, it also increases synthesis costs. Therefore, future work should aim to identify the most effective and economically viable composition that balances adsorption efficiency, material stability, and reusability.

Effect of dosage

In Fig. 3 illustrates the effect of adsorbent dosage on the removal efficiency of Cr(VI) from aqueous solutions. In this study, a 50 mL solution containing 50 ppm of Cr(VI) was treated with varying masses of the PANI-NSA/BC composite. The removal efficiency increased from 70.49% with a 10 mg adsorbent mass to 99.21% with a 40 mg adsorbent mass. This enhancement is attributed to the increased surface area and the availability of more active sites for adsorption as the adsorbent mass increases. Similar trends have

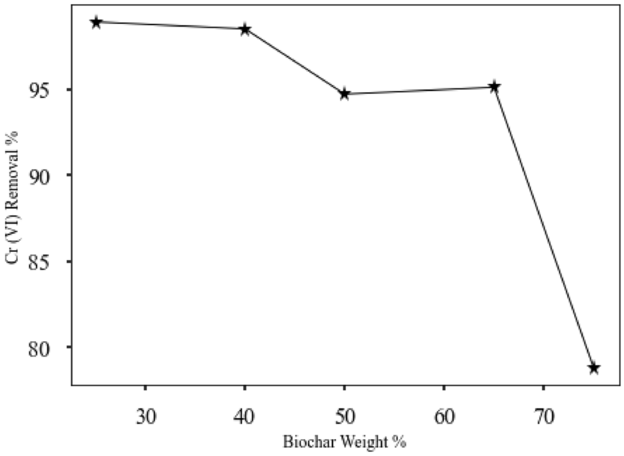


Fig. 2 Effect of biochar percentage in the composite on Cr(VI) adsorption

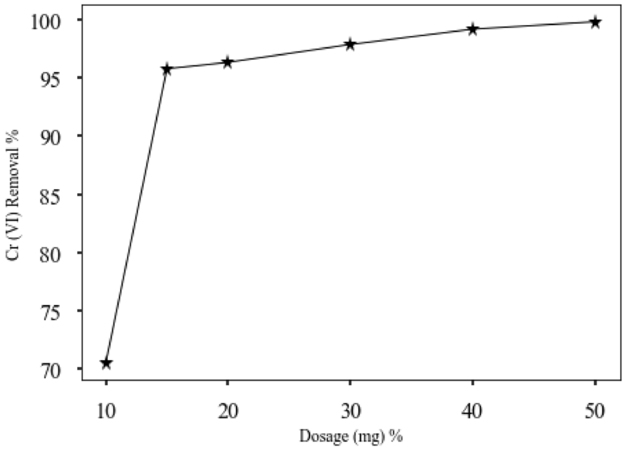


Fig. 3 Effect of adsorbent dose on the adsorption of Cr(VI)

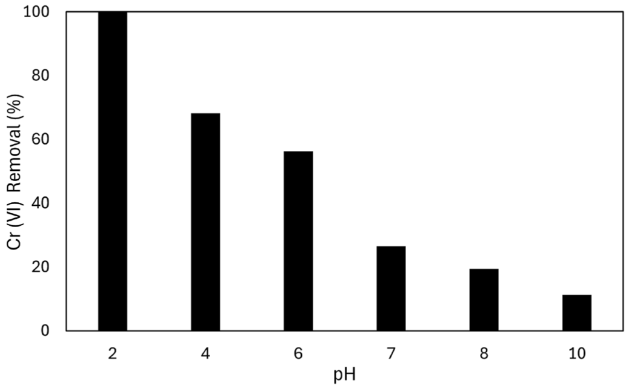


Fig. 4 Effect of solution pH on the adsorption of Cr(VI)

been observed in other studies; for instance, [15] reported that increasing the adsorbent dose led to higher Cr(VI) removal efficiencies due to the greater availability of adsorption sites. However, beyond a certain adsorbent mass, the removal efficiency plateaus, indicating that equilibrium has been reached. At this point, the concentration of Cr(VI) becomes the limiting factor, and additional adsorbent does not result in a significant increase in removal efficiency. This phenomenon is consistent with findings by [16], who observed that after reaching equilibrium, further increases in adsorbent dosage did not enhance Cr(VI) removal efficiency. Therefore, while increasing the adsorbent mass initially leads to higher removal efficiencies due to more available active sites, there exists an optimal dosage beyond which no significant improvement is observed as the system reaches adsorption equilibrium.

Effect of Solution pH

In Fig. 4 the effect of the initial pH of the solution on the removal efficiency of Cr(VI) is demonstrated. It is clear that with an increase in the initial solution pH, the percentage of Cr(VI) removal by PANI-NSA/BC is decreased. The maximum removal of Cr(VI) is observed at pH 2. The removal efficiency of Cr(VI) is significantly influenced by the pH of the solution, as it affects both the surface charge of the adsorbent and the speciation of the Cr(VI) ions in the solution. At lower pH levels, particularly around pH 2, the surface of the PANI-NSA/BC composite becomes highly protonated, resulting in a positively charged surface. This positive charge enhances the electrostatic attraction between the adsorbent and the negatively charged Cr(VI) oxyanions, such as $HCrO_4^-$ and $Cr_2O_7^{2}-$, leading to higher removal efficiencies. For instance, studies have shown that at pH 2, the adsorption capacity of polyaniline-based composites for Cr(VI) is significantly higher due to this electrostatic attraction [13]. As the pH of the solution increases, the degree of protonation of the adsorbent surface decreases, resulting in a reduction of positive charge. Consequently, the electrostatic attraction between the adsorbent and Cr(VI) oxyanions diminishes, leading to decreased adsorption efficiency. Moreover, at higher pH levels, the dominant species of Cr(VI) in solution shifts toward $CrO_4^{2}-$, which experiences greater electrostatic repulsion from the less positively charged or neutral adsorbent surface. This phenomenon has been observed in various studies, where the adsorption capacity of Cr(VI) decreases with increasing pH [17].

Conclusion

This preliminary study demonstrated the successful synthesis and characterization of a polyaniline-biochar (PANI-NSA/BC) composite and its potential application in the removal of Cr(VI) from aqueous solutions. The composite exhibited favorable adsorption performance, achieving a maximum removal efficiency of 99.21% under optimal dosage and pH conditions. The improved removal is attributed to the synergistic effects between biochar's porous structure and polyaniline's redox-active functional groups, which provide abundant active sites for Cr(VI) interaction.

Among the key findings, the study revealed that lower biochar content (25%) in the composite enhances removal efficiency, confirming polyaniline's dominant role in adsorption. Similarly, dosage and pH were found to be critical factors, with optimal adsorption occurring at 40 mg of composite and pH 2, respectively. These observations are consistent with known electrostatic interactions and redox mechanisms.

Given the early-stage nature of this work, the focus was limited to synthesis, characterization, and preliminary batch adsorption tests. Future research will include kinetic and thermodynamic analyses, mechanistic studies using advanced techniques such as XPS, and column experiments to evaluate performance under continuous flow. The ultimate goal is to optimize the composite for cost-effective, scalable, and sustainable application in industrial wastewater treatment.

Author contributions Ruth Kasavo: Conceptualization, Investigation, Methodology, Data curation, Validation, Visualization, Writing - original draft, Formal analysis, Writing - review & editing. HG Brink: Formal analysis, Writing - original draft, Writing - review & editing, Funding acquisition, Resources, Supervision.

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Data availability Data will be made available on request.

Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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