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Original Research

Biochar from Coconut Shell Biomass for the Removal of Sulfate and Cadmium Reduction in Acid Mine Drainage Treatment

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Abstract

The growing mining industry has led to environmental pollution, primarily from mining waste known as acid mine drainage (AMD). To effectively address AMD, a combination of constructed wetland and biochar treatment is necessary. This study aims to assess the ability of biochar in combination with wetland sediment to reduce sulfate and cadmium (Cd) heavy metals in AMD. The research method involved a laboratory-scale constructed wetland in a microcosmos and a treatment of: T1, biochar mixed with wetland sediment; T2, sediment; T3, biochar; and T4, control with no treatment. Observations included sulfate content, pH determination, heavy metal concentration, and scanning electron microscope (SEM) analysis of the biochar. Results after 30 days of observation showed that T1 reduced sulfate concentration by 72.03%, compared to 63.33% for T2, 63.33% for T3, and 2.50% for T4. The reduction in sulfate was accompanied by a consecutive increase in pH, with T1 at pH 6.9, T2 at pH 6.6, T3 at pH 6.4, and T4 at pH 3.6 after 30 days. T1 treatment reduced heavy metal Cd by 80.16% after 30 days, while T2 of 55.46%. T3 of 65.83% and T4 of 2.31%. This indicates that the constructed wetland method, combined with biochar, is more effective in reducing sulfate and the heavy metal Cd in AMD, compared to using only biochar or wetland sediment treatment.

Keywords: constructed wetland, acid mine drainage, biochar, wetland sediment, cadmium

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Introduction

The rapid growth of the mining industry has brought significant economic benefits. However, it has also resulted in environmental damage due to generated waste. One of the hazardous mining wastes is a sulfuric acid solution derived from residual ore, characterized by a low pH that can dissolve heavy metal ions, known as acid mine drainage (AMD) [1, 2].

AMD forms when mineral mining materials mix with the soil surface in the form of metal sulfides (MS), and react with oxygen, leading to oxidation and the formation of sulfate (SO_4). When exposed to water or rain, it transforms into sulfuric acid [3, 4]. The acidic nature of AMD can be lethal to aquatic organisms and disrupt plant growth on land. Additionally, the high solubility of heavy metals in AMD serves as a source of toxic heavy metal pollution, potentially impacting human health [5, 6].

Cadmium (Cd) is often present in AMD and commonly found in gold, nickel, copper, and coal mining areas [7]. Cadmium is toxic and can enter organisms' bodies through the food chain, subsequently accumulating in living organisms' tissues, indirectly affecting human health, particularly by causing liver toxicity and kidney failure [8].

Considering the impact of cadmium heavy metal, it is crucial to address AMD as a source of heavy metal pollution, especially cadmium. Traditionally, AMD mitigation has been achieved through chemical methods involving the addition of lime or calcium carbonate to neutralize pH or physical methods, such as redirecting AMD into large pits to prevent contact with oxygen [9]. Both of these methods are inefficient, costly, and may not always meet environmental requirements. An environmentally friendly and effective alternative is bioremediation using sulfate-reducing bacteria (SRB) to reduce sulfate and heavy metals [10].

SRBs are abundantly found in wetland sediments and have the potential to reduce sulfate and heavy metal levels [10]. On the other hand, biochar has been used as an adsorbent for various heavy metals in AMD. Based on this, a method combining constructed wetland with sulfate-reducing bacteria as sulfate reducers and biochar as a heavy metal adsorbent is needed to enhance the efficiency of AMD treatment [11].

Biochar is a form of activated carbon, derived from biomass waste, such as coconut shells, sugarcane bagasse, wood from forests, rice husks, and other biomass sources. This activated carbon is produced through pyrolysis, resulting in fine and porous characteristics [6, 12]. Among these raw materials, coconut shells are the preferred choice due to their abundance and availability. Additionally, coconut shell-based activated carbon is known for its abundant micropores, low ash content, high water solubility, and high reactivity [13, 14].

According to Yin et al. [15], biochar also shows advantages for AMD treatment. It serves as an excellent carrier for the growth of sulfate-reducing bacteria for sulfate reduction and acts as an adsorbent for heavy metals. Furthermore, biochar, with its stability in water, has the ability to raise pH and provide nutrients for bacterial growth [6, 16]. Additionally, Wibowo et al. [17] have reported that biochar is more effective and cost-efficient as an adsorbent for various heavy metals, such as Fe, Mn, Al, Mg, Cu, Zn, Ca, K, Ba, Li, Pb, Ni, and Si, in AMD.

Sediments contain a high level of organic matter, which benefits growth and provides a favorable environment for sulfate-reducing bacteria to support the complex metal reduction process [10, 18]. Furthermore, biochar applications can be divided into several categories and the most prevalent utilization of biochar is as a catalyst and catalyst support for environmental remediation [19-21].

Several studies have explored the application of biochar as a heavy metal adsorbent. For instance, research by Yin et al. [15] demonstrated that the application of biochar to AMD was able to significantly reduce Cd by up to 40.7%. Similarly, in a study by Lu et al. [16], the use of sludge-derived biochar was effective in absorbing Pb^{2+}, achieving rates of 45-60%. Another study by Shakya and Tripti [22] involving biochar derived from poultry litter pellets successfully reduced Zn, Mn, and Cu by up to 98%. Given the success of biochar applications as heavy metal adsorbents and the use of sediments as a source of sulfate-reducing bacteria inoculum for sulfate reduction in AMD, the combination of biochar with sediment treatment offers an effective solution for AMD treatment.

Experimental

Sampling

The acid mine drainage (AMD) was collected from the Lamuru mining area, Massenrempulu District, Bone Regency (Fig. 1). Sampling of acid mine drainage was carried out using the integrated sample method, with one collection at three sampling points of the pond and pit. Samples were taken from former mining areas, indicated to have low acidity using a dipper, referring to Indonesian National Standards 8520: 2018. Then, the acid mine drainage samples were put together, ready for further examination. Wetland sediment was sourced from the Tallo Makassar Mangrove Forest area, while coconut shells were obtained from the Daya Traditional Market in Makassar City.

Initial Characterization of Sediment and AMD

Characterization of sediment samples included: total organic carbon was measured using a TOC analyzer with the method referring to the Indonesian National Standardization Agency; total nitrogen using the Micro Kjeldahl method, with the nitrogen decomposition stage initially carried out in the acid mine water sample, then the addition of base to convert NH^{4+} to NH_3 through condensation, with the amount of nitrogen calculated from the number of ammonia ions in the solution through titration; phosphorus content using the Stannous Chloride method is based on the reaction that forms a complex with ammonium molybdate followed by reduction with

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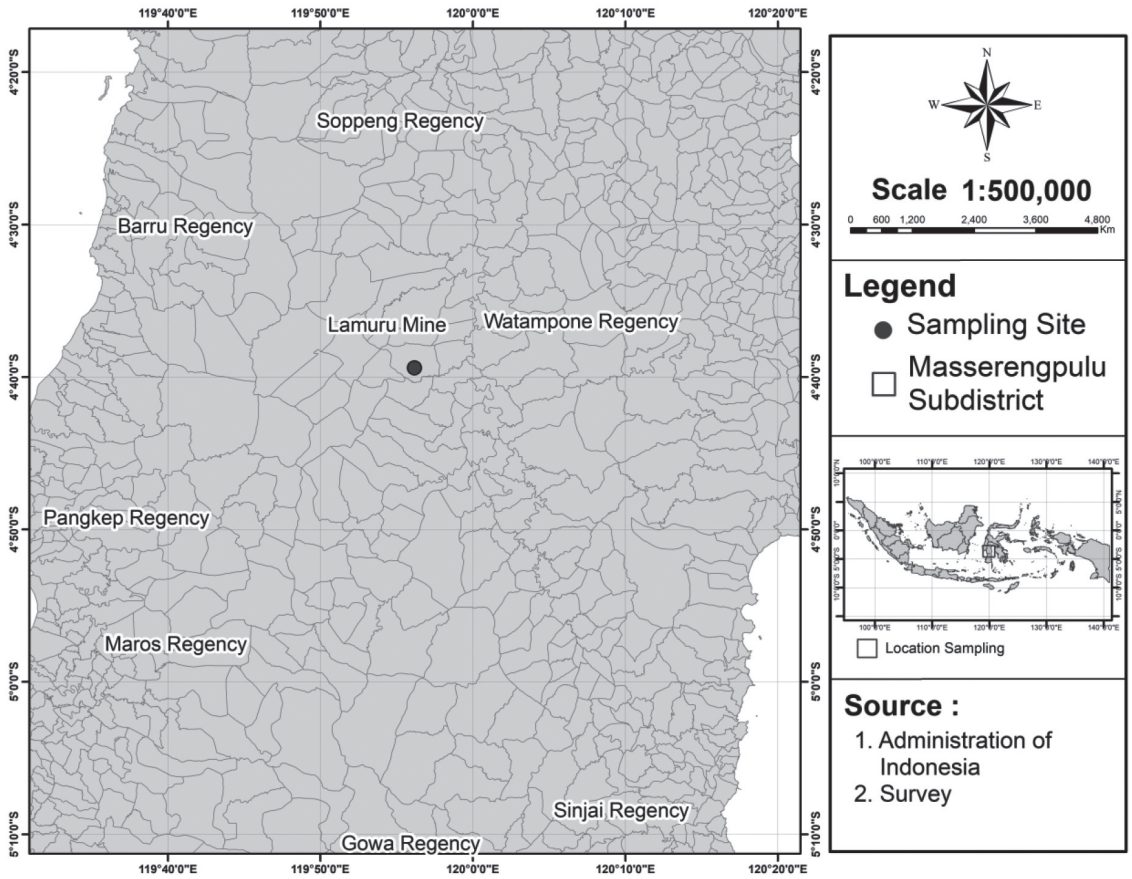

Fig. 1. Map of the sampling site of acid mine drainage on the Lamuru Mine, Bone Regency, South Sulawesi, Indonesia.

stannous chloride, then measured on a spectrophotometer at a wavelength of 690 nm [23].

Characterization of AMD samples included: measuring sulfate content is measured by the addition of barium chloranilate, which reacts with sulfate ions to liberate the chloranilic acid, with the sulfate concentration measured using a spectrophotometer at a wavelength of 420 nm; determining pH using a pH meter, the pH electrode was first calibrated with a standard buffer solution with a pH value of 7, then, the electrode was immersed into the sample solution until a stable reading was achieved [23].

Carbonization

The coconut shells are cleaned from the fibers that are still attached, crushed into smaller sizes, then dried to reduce the water content. Next, carbonation is carried out in a furnace supplied with inert gas at a temperature of 400 °C for 2 hours to remove volatile substances. The resulting coconut shell charcoal was allowed to cool in a desiccator. After that, the charcoal was crushed and sieved to 200 mesh size until carbon powder was produced as biochar.

Chemical Activation of Coconut Shell Charcoal

The coconut shell charcoal resulting from combustion was pulverized into 200-mesh particles using mechanical steel ball milling. Subsequently, the carbon particles were activated by immersing them in a 0.2 N NaOH solution for 18 hours, followed by rinsing with deionized water and filtration using a Buchner funnel. The charcoal precipitate was washed repeatedly with deionized water until the filtrate reached a near-neutral pH and then dried. The activated carbon from coconut shell charcoal, as biochar, was ready for use in the treatment [24].

Scanning Electron Microscopy (SEM) Analysis

Characterization of the physical properties of biochar from coconut shell carbon was carried out using scanning electron microscope (SEM) type Thermo Fisher Phenom ProX, which has a magnification range of up to 300,000 times with a resolution of 6 nanometers at 5000x magnification. This is used to determine the surface morphology of activated carbon by looking at the distribution and characterization of its pores. This SEM is equipped with a secondary electron detector, and has 2 operational modes, namely Low Vacuum for non- conductive samples, and High Vacuum for conductive samples.

Treatment and Experiment

The combination of biochar and sediment treatment in acid mine drainage using the constructed wetland

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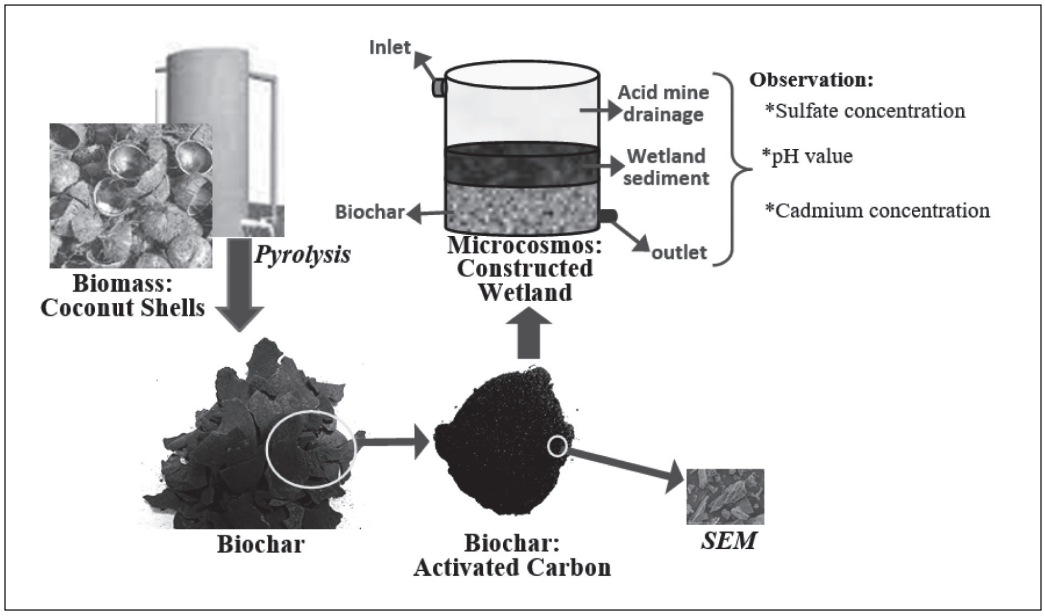

Fig. 2. The research stages start from making biochar to application in the treatment of acid mine drainage in constructed wetland method.

(CW) method was carried out in vertical microcosms, constructed with a column diameter of 15 cm and a height of 35 cm [25]. The treatments included: T1, comprising 20% sediment and 15% biochar; T2, with 20% sediment; T3, with 15% biochar; and T4, containing only acid mine drainage as a control group. Each treatment was duplicated and placed at room temperature (27 °C). Observations included pH, sulfate content, and the cadmium (Cd) heavy metal content. Overall, the research stages start with preparing biochar for application in the treatment of acid mine drainage in constructed wetland method (Fig. 2)

pH Measurement

pH values in the treatments were measured using a pH meter. The pH electrode was rinsed with deionized water, calibrated using buffer solutions, and allowed to stabilize for 15 minutes. Subsequently, the pH meter electrode was immersed in the acid mine drainage treatment sample, and the pH value was determined when it appeared on the pH meter scale.

Determination of Sulfate Concentration

The sulfate concentration was measured using the turbidimetry method. Acid mine drainage samples from the treatments were forced to react with barium chloride (BaCl2) and vigorously shaken using a vortex mixer for 1 minute to form a turbid BaSO4 suspension, creating a colloid. The absorbance of the solution was then measured with a UV-Vis spectrophotometer at a wavelength of 420 nm. The absorbance data obtained was used to determine the sulfate content using a linear equation derived from a standard sulfate solution.

Analysis of Cadmium Heavy Metal Concentration

The analysis of the cadmium (Cd) heavy metal content involved initial sample digestion. Subsequently, the digested samples were transferred to a 50 mL volumetric flask, washed with metal-free water, and then added to the flask. Deionized water was added to reach a final sample volume of 25 ml, followed by homogenization. The solution was then filtered for the analysis of cadmium heavy metal content using Atomic Absorption Spectroscopy (AAS) at a wavelength of 228.8 nm.

Results and Discussion

Characterization of Sediment and AMD

The initial characterization of the mangrove sediment samples revealed the following: organic carbon content of 289,000 mg/L, total nitrogen content of 18,210 mg/L, and total phosphorus content of 9.71 mg/L. Similarly, the acid mine drainage samples exhibited a pH of 3.2 and a sulfate content of 1.21 ppm. This characterization was conducted to establish the baseline conditions for the treatment of acid mine drainage using biochar in combination with sediment. The carbon, nitrogen, and phosphorus content in the sediment serve as nutrient sources for microbial growth and development during the processes of cadmium heavy metal reduction and sulfate reduction in the acid mine drainage treatment. Hydrogen molecules originating from the sediment's organic material act as electron donors required by microbes for sulfate reduction through the oxidation reaction into sulfite [25, 26].

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Scanning Electron Microscope (SEM) Analysis of Biochar

The results of the SEM analysis of coconut shell biochar after activation, at a magnification of 1000x, revealed a surface morphology characterized by small, dense, and irregularly shaped pores, in contrast to the non-activated biochar, which displayed almost no discernible pores (Fig. 3). The formation of these porous structures in activated biochar is related to its ability to adsorb metals and influences the creation of immobilized cells on the biochar surface [6, 27].

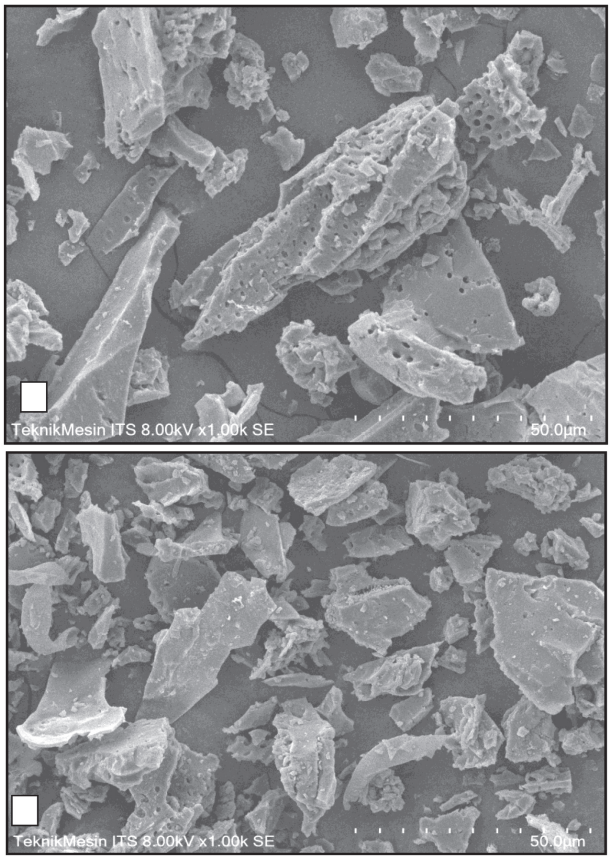

Fig. 3. SEM analysis results at 1000x magnification of activated biochar (a) and non-activated biochar (b).

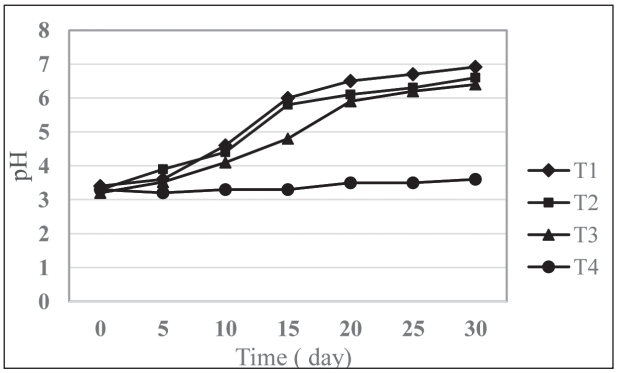

Fig. 4. Changes in pH values in the treatment of acid mine drainage, where T1 is biochar and sediment, T2 is sediment, T3 is biochar, and T4 is the control.

The activation process involves the release of many volatile compounds, which leads to pore formation and reduces hydrocarbon coverage on the coconut shell biochar. The creation and enlargement of pores in activated carbon result from the evaporation of degraded cellulose components, leading to differences in pore structure between activated and non-activated carbon [28].

Pyrolysis at temperatures above 400 ^{0}C will cause thermal degradation of biomass organic matter. Then, it will release more volatile matter and substances that block the pores of the biomass matrix. SEM analysis shows the creation of more pores and channel-like structures in biochar. SEM analysis also reveals that biochar has a larger surface area obtained at pyrolysis temperatures of 400-600 °C [29]. This affects the alkalinity property of the biochar which increases with pyrolysis temperature as acidic and polar functional groups are successively removed, resulting in a more hydrophobic and well-organized carbon-coated biochar. A highly organized aromatic structure is formed in coconut shell biochar, which has larger and more defined pores than rice husk biochar, which is less defined and smaller [30].

This is also confirmed by the results of research by Zaitun et al. [31]. Carbon and oxygen content correlate with the gasification temperature. The higher temperature in coconut shell biochar correlates with an increase in carbon content and a decrease in oxygen in biochar. Increasing the gasification temperature will increase the volatilization of light compounds of the material.

pH Changes and Sulfate Concentration

During pH measurements, all treatments exhibited an increase in pH (Fig. 4). The most significant increase was observed in treatment T1, involving biochar with sediment, which increased from an initial pH of 3.4 to 6.9 by day 30. In comparison, treatment T2, consisting of sediment only, reached a final pH of 6.4. Treatment T3, involving biochar alone, resulted in a pH of 6.6, while the control treatment, T4, maintained a pH of 3.6.

In the acid mine drainage treatment, treatment T1 showed a significant reduction in sulfate concentration from an initial level of 1.18 ppm, gradually decreasing to 0.33 ppm by day 30 (72.03%). Treatment T2 exhibited a sulfate reduction to 0.44 ppm (63.33%), T3 showed a sulfate decrease to 0.57 ppm (52.10%), while the control treatment, T4, experienced minimal reduction (2.50%) (Fig. 5).

Based on the pH and sulfate content measurements, it is evident that the reduction in sulfate levels corresponds with an increase in pH. This suggests that sulfate content in acid mine drainage is acidic, indicated by its low pH. It is known that lower sulfate levels are associated with higher pH values.

In treatments involving the addition of biochar mixed with sediment and treatments with sediment alone, a sharp increase in pH was observed. This indicates the activity of sulfate-reducing bacteria sourced from the sediment, which reduce sulfate to hydrogen sulfide (H_2S). Consequently, sulfate levels decrease, and bicarbonate

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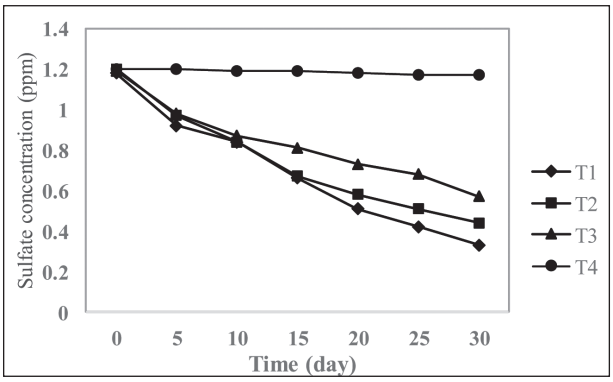

Fig. 5. Sulfate concentration in the treatment of acid mine drainage, where T1 is biochar and sediment, T2 is sediment, T3 is biochar, and T4 is the control.

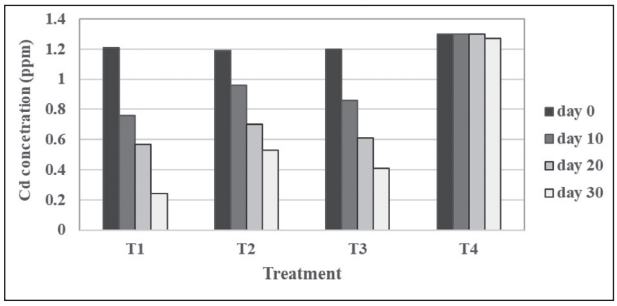

Fig. 6. Cd concentration in acid mine drainage treatment, where T1 is biochar and sediment, T2 is sediment, T3 is biochar, and T4 is the control.

ions (HCO^{3-}) are produced as a buffering agent, causing the pH to rise in the treatment [32, 33]. The formation of bicarbonate serves as evidence of the sulfate-reducing bacteria's ability to control pH in the acid mine drainage treatment [34, 35]. On the one hand, the organic material in biochar acts as an electron donor in sulfate reduction to sulfide, leading to a decrease in sulfate content [36].

In the treatment involving the addition of biochar, an increase in pH was also observed. This can be attributed to the release of basic cations, such as alkali metal oxides like Na^+, Ca^{2+}, Mg^{2+}, and K^+, as part of the metal-biochar complex, which is associated with the pH increase in acid mine drainage [37, 38]. In the control treatment, there was no significant increase in pH, as this is because of the absence of sediment and biochar treatments that play a role in the sulfate reduction process.

The reduction in sulfate content observed in treatments with both biochar and sediment and treatments with sediment alone is related to the influence of sulfate-reducing bacteria that convert sulfate (SO_4^{2-}) into hydrogen sulfide (H_2S). In this reduction process, sulfate serves as an electron acceptor by sulfate-reducing bacteria, and the organic material present in the sediment acts as a carbon source for metabolic processes to occur [4].

During this sulfate reduction process, hydroxyl ions (OH^-) are released, which trigger an increase in pH [9]. Additionally, the organic material in biochar acts as an electron donor in sulfate reduction to sulfide [39]. This is in line with the findings of Yoon et al. [40] stating that the functional groups on the biochar surface can initiate active radical species that play a vital role in the degradation of contaminants as catalysts and in the removal of adsorbents through electron transfer or redox processes, released from a photocatalyst or metal.

Cadmium (Cd) Heavy Metal Concentration

In the measurement results of cadmium (Cd) concentration, treatment T1, involving both biochar and sediment, showed a decrease to a concentration of 0.24 ppm (80.16%) by day 30. Treatment T2, which included sediment alone, exhibited a Cd concentration of 0.53 ppm (55.46%), and treatment T3, with biochar alone, experienced a reduction to 0.41 ppm (65.83%). In contrast, treatment T4 as the control showed a relatively insignificant decrease, with the initial concentration dropping from 1.30 ppm to 1.27 ppm (2.31%) (Fig. 6).

The reduction in cadmium (Cd) concentration in the acid mine drainage treatment can be attributed to several factors. One of them is the biological processes, specifically the sulfate-reducing bacteria found in the sediment, which engage in the reduction reaction of the heavy metal Cd. Another biological mechanism is the formation of a complex of bacteria-immobile cells on the biochar, resulting in a bioabsorption mechanism for Cd. In addition to these biological mechanisms, there are chemical processes, with the activated carbon in biochar acting as an adsorbent for heavy metal Cd [13, 15].

In the reduction reaction of heavy metal Cd with sulfate by sulfate-reducing bacteria, highly reactive hydrogen sulfide (H_2S) is produced, accelerating the reaction with metals to form insoluble metal sulfide compounds. Some heavy metals were precipitated in this reaction, while others became bound to immobile cells and biochar [39, 41].

Even in the treatment involving the addition of biochar, there was a decrease in cadmium concentration. This occurred because biochar particles contain numerous pores that act as adsorbents for heavy metals. Smaller particle sizes result in larger pore surfaces on biochar, allowing more ions to be absorbed onto its surface [17]. In contrast, the control treatment showed no significant reduction in cadmium (Cd) concentration because it lacked sulfate-reducing bacteria, as well as activated carbon to bind the heavy metal Cd.

Based on these observations, a comparison can be drawn between the roles of sediment and biochar in sulfate and cadmium heavy metal reduction. Treatment T2, involving sediment alone, had the most significant impact, reducing sulfate concentration by 64.81% while also raising the pH compared to treatment T3 with biochar, where sulfate decreased by only 46.90%.

In terms of the decrease in heavy metal Cd concentration, Treatment 3 (T3) plays a more significant role as it can reduce it by 58.67%, compared to Treatment 2 (T2), which reduces the concentration of heavy metal Cd by 53.85%. This indicates that sulfate-reducing bacteria

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have a more crucial role in reducing sulfate through the conversion of sulfate to sulfite. Conversely, biochar plays a more substantial role in the removal of heavy metal Cd through an adsorption mechanism. Therefore, combining biochar with sediment is necessary to enhance the effectiveness of acid mine drainage treatment.

Overall, the combined treatment of biochar with sediment proves to be more effective, both in reducing sulfate and decreasing the concentration of heavy metal Cd. Biochar is known to operate through chemical and biological mechanisms, serving as an adsorbent and creating a medium for immobile cells to form biochar-cell complexes. Additionally, it physically absorbs pollutants through its pores. As per Zhang et al. [38], biochar, acting as a carrier for immobile cells, enhances the binding of heavy metals. This aligns with Du et al. [6] statement that biochar facilitates electron transfer between microbial cells and contaminants, utilizing the organic carbon within the sediment. According to Xu et al. [42] certain bacterial species, particularly *Pseudomonas sp*, can serve as adsorbents for heavy metals through the immobilization of lead (Pb).

Based on this discussion, it is evident that biochar is an effective adsorbent with substantial capabilities in its interactions with contaminants. Various mechanisms are involved in the interactions between biochar and organic and heavy metal contaminants, including electrostatic attraction, polar and non-polar organic attraction to the biochar carbonized phase, and partitioning into the non-carbonized phase [42].

Conclusions

The application of biochar with sediment in the constructed wetland method proves to be more effective in reducing sulfate and heavy metals in the treatment of acid mine drainage. In this context, biochar acts as an adsorbent, while wetland sediment serves as a source of sulfate-reducing bacteria for sulfate reduction. The combination of biochar with sediment in the constructed wetland method complements the formation of immobile cells in the mechanism of sulfate and heavy metal reduction in acid mine drainage. Therefore, treatments that combine biochar with sediment are significantly more effective in lowering sulfate concentrations, achieving up to 72.03% reduction while increasing the pH value from 3.4 to 6.9. Similarly, their ability to reduce heavy metal Cd concentrations reaches 80.16%, surpassing treatments that solely use biochar (55.46%) or sediment alone (65.83%).

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Conflict of Interest

The authors declare no conflicts of interest

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