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Original Research

Assessment of Biochar Produced from Aquatic Plants for Environmental and Agricultural Applications by Multi-Analytical Characterizations

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Abstract

Biochar obtained through the processes of pyrolysis has become carbon-rich eco-friendly aromatic material and can be used as sorbents and soil amendment for organic and inorganic contaminants. This study investigated biochar's physical and chemical characteristics produced from commonly growing eight different aquatic plants. All biochars were obtained at 500 °C and characterized in detail to evaluate their appropriateness for agricultural and environmental utilization as well as applied to Cd-contaminated soil to assess the fixation of Cd to use as soil amendment. Herein, characteristics of biochars were described with multi analytical techniques, like Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), and thermogravimetric analysis (TG), respectively. Among the biochars, there were considerable variations in elemental composition, micrmorphology and surface area. Overall results indicate that biochars obtained from aquatic plants could be useful as soil amendment as well as fertilizer due to their higher content of mineral elements, highly porous structure and presence of aromatic and hydroxy functional

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groups and adsorption capacity of cadmium which are dominating in all biochars. All biochars produced from aquatic plants are better way to set out waste biomass to avoid eutrophication and have remarkable properties for environmental and agricultural applications.

Keywords: aquatic plants, biochar, micromorphology, pyrolysis

Introduction

Aquatic or wetland plants are floating and submersed plants which grow under the surface of water, and have been extensively grown for water cleaning in man-made floating islands and contaminated wetlands [1, 2]. Previously different species of wetland plants including Miscanthus sinensis Anderss, Vetiveria zizanioides, Acorus calamus, and Thalia dealbata were reported that have been functional to bioremediation of contaminated water using constructed wetlands [3, 4]. Most aquatic plants grow rapidly and are widely used in the management of domestic and swine wastewater [5, 6]. It was found that Miscanthus aquaticum sufficiently removes microorganisms/antibiotics from water [7]. However, to maintain nutrient evacuation efficiency of wetlands, a huge amount of aquatic plant biomass is required to be appropriately disposed of which has become a challenge since disintegration of plant biomass in water can boomerang all the nutrients and micro-organisms reverse back to the water. Though *Lemna minor* L. and *Pistia stratiotes* L. are widespread free-floating aquatic plants with fast reproduction and spreading rates, that can maintain high productivity of 10-30 tons of dry mass ha^{-1} $year^{-1}$ [8] and 60-110 tons ha^{-1} $year^{-1}$ [9]. However, overproduction of aquatic plants causes waterway clogging and acts as a secondary environmental pollutant [10]. Huge biomass of aquatic plants has been produced in the environment in greater quantities which need to be reused to avoid detrimental consequences. [11] reported that 6.75 million hectares of constructed wetlands in China produce about 107 Mt of wetland plants annually. With the frequent advancement and modernization of human society, water bodies have been fagged severely.

For waste biomass management, pyrolysis is emergent and time-consuming technology; thus, biochar is an aromatized carbon-rich product comprised through pyrolysis. Biochar was increasingly used as a promising environmentally useful material and received more attention [8, 12]. Therefore, producing biochar from aquatic plants not only solves the environmental pollution issue but is also helpful in waste management in a productive way [13]. Waste biomass has been renowned as the preferred raw material to produce biochar because of its extensive accessibility and cheap cost [14]. Biochar has been reported in many studies to improve carbon sequestration as well as water quality, and decrease greenhouse gas emissions in soils and water environments [15] because of its tremendous composition, elevated cation exchange capacity and larger surface area [12]. The characteristics of biochar determine the suitable application and potential of obtained biochars as sorbent to remediate soil or water. The current advancement in ecological and environmental protection had led to more attention towards aquatic plants [16]. Comprehensive studies have been carried out on biochar obtained from aquatic and wetland plant feedstock, to solve the problem of properly disposing of these plants and utilizing them as fertilizer and bioremediation. Therefore, the use of aquatic plants to produce biochar could not only consume these waste materials but also enhance environmental management.

Despite the use of different feedstock-derived biochars as a soil amendment, this study will focus more on biochars derived from different aquatic plants and evaluate these biochar amendments to determine the availability of Cd in soil. Presently, to evaluate the physicochemical properties of biochars, different methods or techniques have been followed for an accurate assessment of biochar characteristics. However, characterization techniques may vary from simple conventional analysis to advanced ones which usually depend on the selection and approachability of diagnostic instruments [17]. Fourier transform infrared spectroscopy (FTIR) has been used to obtain the functional groups of biochar [18]. And through scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) surface morphology and chemical composition can be explored [19, 20]. Thermogravimetric (TG) analyses can be carried out to identify the variations in the physicochemical characteristics of materials taking place during pyrolysis [9, 21]. [22] have completely characterized different biochars, including contiguous and eventual analysis, pH, SEM, FTIR, N_2 sorption and desorption isotherms. In this study, the objective was to characterize the physicochemical properties of the biochars obtained from aquatic plants commonly grown worldwide with a high rate of productivity, by using a multi-analytical approach. Besides the characterization of different biochar produced in the same conditions, evaluate their efficiency to reduce cadmium bioavailability in soil. The results obtained from this study might provide a more intensive understanding of biochar properties to potential environmental and agricultural applications.

Material and Methods

Biochar Production

Commonly grown aquatic plants, including *Pistia stratiotes* (PS), *Trapa natans* (TN), *Myriophyllum* *crispatum* (MC), *Ceratophyllum demersum* (CD), *Hydrocotyle umbellate* (HU), *Lemna gibba* (LG), *Eichhornia crassipes* (EC), and *Azolla caroliniana* (AC) were obtained from the ponds of Zhejiang University, Hangzhou, China. Prior to pyrolyzed, all the aquatic plants were oven dried at 65ºC for 48 hr. All biochars were produced in pyrolyzing furnace model TH-01, China at 500ºC pyrolysis temperature at a rate of 10ºC min^{-1} for 2 hr. The biochar samples were ground to pass through a 0.5 mm sieve after pyrolysis.

Fundamental Characterization

The pH of all the biochars was analyzed by adding biochar to double distilled water at a ratio of 1:20 (biochar/water) [23]. Elemental analysis (N, C, H) was carried out through Elemental Analyzer (Flash-EA112, Thermo Finnigan, USA). The H/C atomic ratio was calculated to determine the aromaticity of biochars. According to the Brunauer Emmett Teller method, specific surface areas of the biochars were measured by N_2 adsorption isotherms at 77 K and by CO_2 isotherms at 273 K using a surface area analyzer (Quadrasorb Si-MP, Germany).

Thermogravimetric (TG) Analysis and Fourier Transform Infrared Spectrometer (FTIR)

Approximately 3 mg powdered sample of each dried aquatic plant was subjected to Thermogravimetric (TG) analysis was carried out under nitrogen as the carrier gas using a TG analyzer (Tagongsi SDTQ600, USA), heated from room temperature to 500ºC, at a heating rate of 10ºC min^{-1}, respectively.

FTIR analysis was carried out using FTIR spectrophotometer (Nicolet 6700, USA) for the identification of surface functional groups of biochars, at 25ºC from 400 and 4000 cm^{-1} spectral range using 50 scans being taken at 2 cm^{-1} resolutions, respectively.

Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS)

Microscopic imaging analysis was carried out to evaluate the biochar micromorphology by using a scanning electron microscope (SEM) model: Hitachi SU-8010 (Tokyo, Japan). Surface elemental analysis was also carried out at the same position by using SEM energy dispersive X-ray spectroscope (EDS, EDAX Inc. Genesis XM).

Soil and Amendments

The soil was taken from a depth of 0-20 cm of an uncontaminated and non-cultivated site. The soil was air-dried, grounded, screened through a 2 mm sieve and analyzed according to the Routine Analytical Methods of Agricultural Chemistry [24, 25] for physicochemical characteristics (Table S1).

Soil was spiked at the rate of 1 mg kg^{-1} with Cd as $Cd(NO_3)_2$ in an aqueous solution and mixed thoroughly. To maintain 70% water holding capacity distilled water was applied to the soil. Prior to the experiment, the spiked soil was left for two weeks to equilibrate then had been sieved again and the total Cd concentration was determined. 0.5 kg soil was amended with each biochar at the rate of 5% in each pot and homogenized thoroughly then incubated for two weeks with distilled water at 70% water holding capacity. Cd contaminated soil without biochar amendment used as control (CK).

Mehlich-3 Extraction

According to [26], the bioavailability of Cd was determined in soils with and without biochar amendment through Mehlich-3-extraction method. Briefly, prepared Mehlich-3 solution (0.2 mol L^{-1} CH_3COOH, 0.25 mol L^{-1} NH_4NO_3, 0.015 mol L^{-1} NH_4F, 0.013 mol L^{-1} HNO_3, 0.001 mM EDTA) than in 50 mL of Mehlich-3 solution, 5 g of each dry and sieved soil sample was shaken at 200 rpm for 5 min at 25ºC. The suspension was centrifuged at 4500 rpm for 10 mins (Sorvall, biofuge stratos, Germany) and filtered through 0.45 µm filter paper. Cd concentration in the filtrate was analyzed by ICP-MS (Agilent, 7500a, USA). Soil samples without biochar amendment were used as blank samples.

Results and Discussion

Fundamental Properties

The fundamental properties of biochars obtained from aquatic plants were given in Table 1. The elevated pH values of all biochars show that they have alkaline in nature. The N, C, and H contents measured in different biochars were ranging from of 45.6-62.3%, 1.39-2.85%, and 1.32-2.63%, respectively. Moreover, the H/C ratio of the biochars range from 0.34 to 0.57 and C/N ratios varied from 26.46 to 51.08. The surface area of the biochars ranges from 5.26 to 57.39 m^2 g^{-1}; however, the pore diameter of biochars was about 1-2 nm. It has been observed that PS-derived biochar had a higher surface area as compared to other biochars. All the biochars produced from aquatic plants in this study had alkaline pH due to the desiccation, aromatization, and decarboxylation which cause the reorganization of functional groups during the pyrolysis process which usually takes in a raise of pH [22]. This might also be due to the pyrolysis of biomass at completely closed environment at high temperatures, which is in agreement with [11], which suggests that the wetland plant-based biochars can be suitable for acidic soils and can improve soil quality and fertility. According to previous research, the soil amendment with biochar derived from plant-derived biochars had alkaline pH and their application as a soil amendment, in turn, enhance soil pH and reduced the heavy metal bioavailability

Table 1. Physicochemical properties of biochars derived from aquatic plants.

Biochar	pH	SA ($m^2 g^{-1}$)	Pore Size (nm)	C (%)	H (%)	N (%)	Atomic ratio	
							H/C	C/N
PS	9.81 ±0.01	57.39	1.45	59.91	2.85	1.9	0.570	36.78
TN	9.70 ±0.01	29.95	1.64	55.25	1.75	1.55	0.380	41.58
MC	9.74 ±0.01	48.07	2.93	61.95	1.51	1.75	0.292	41.3
CD	9.51 ±0.00	12.02	1.32	57.8	2.55	1.32	0.529	51.08
HU	9.92 ±0.01	35.85	2.04	45.6	2.07	2.01	0.544	26.46
LG	10.04±0.01	5.26	1.07	48.05	1.39	1.97	0.347	28.45
EC	9.13 ±0.00	13.89	1.53	58.51	2.46	2.41	0.504	28.32
AC	9.02 ±0.01	10.09	1.29	62.43	2.33	2.63	0.447	27.69

Biochars derived from *Pistia stratiotes* (PS), *Trapa natans* (TN), *Myriophyllum crispatum* (MC), *Ceratophyllum demersum* (CD), *Hydrocotyle umbellate* (HU), *Lemna gibba* (LG), *Eichhornia crassipes* (EC) and *Azolla caroliniana* (AC). Carbon (C), N (nitrogen), H (hydrogen), surface area (SA).

[9, 27]. The highly alkaline pH shows a moderately elevated ash content of alkali and alkaline earth metals [28]. The elemental analysis showed high C contents in all the biochars. However, MC biochar had the highest C content relatively than other studied biochars (Table 1). The H/C ratio of PS, CD, and HU biochar was ranged from 0.54-0.57, which suggested that in these biochars developed highly aromatic structure with the carbonization [29]. Thus, the low H/C ratio of biochars indicated a very strong aromatic structure [22]. Among all biochars PS biochar had a greater surface area (57.39 m^2 g^{-1}), whereas LG derived biochar had a lesser surface area (5.26 m^2 g^{-1}). When biochar is applied to soils with higher surface area can immobilize metals and reduce the sorption of metals by crops [30, 31].

Thermogravimetric (TG) Analysis

Mass loss was determined through thermogravimetric (TG) analysis, mass loss curves of PS, TN, MC, CD, HU, LG, EC, and AC were obtained under an inert atmosphere at 10ºC min^{-1}. At a specific temperature range, the TG curve showed weight loss. The biomass of feedstock initially decreased slightly although rapidly decreased from 250 to 400ºC (Fig. 1). LG and PS biochars were thermally more stable while TN and EC biochars became less stable than other biochar, respectively. During TG analysis, feedstock decomposes stepwise; firstly, dehydration (initial moisture loss), secondly volatilization and finally decomposition. In this study, after initial moisture loss, volatilization and decomposition took place in the range of 150ºC to 350ºC, respectively. However, all the biomass feedstock was thermally less stable and very little difference had been shown in the TG analysis which might be because all of them were aquatic plants. According to [9, 32], degradation characteristics vary with different feedstock during pyrolysis, i.e., biochars obtained from animal manure were thermally more constant than biochars obtained from plant biomass. It has been suggested that feedstock biomass can make a difference in the thermal decomposition of the materials.

Functional Group Analysis (FTIR)

The FTIR spectra of biochars were obtained to identify the nature of functional groups, and it can be seen that almost all biochars had the same peaks (Fig. 2). According to [33], the peaks of biochars at 3438 to 3444 cm^{-1} illustrate O-H hydroxyl group, at 1626 cm^{-1} showed C=C stretch and 1585 to 1592 cm^{-1} had C=C-C aromatic ring stretch. 1431 to 1433 cm^{-1} had methyl C-H asymmetric. The peak at 1111 cm^{-1} had C-O-C group and C-O stretch, respectively. Whereas, 617 to 875 cm^{-1} peaks showed aromatic C-H in plane blend and O-H group that was common among all biochars.

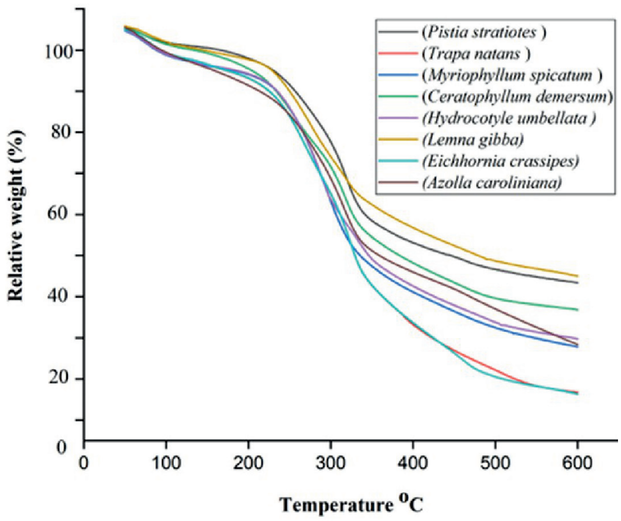


Fig. 1. Thermogravimetric analysis of biochar derived from different aquatic plants feedstock.

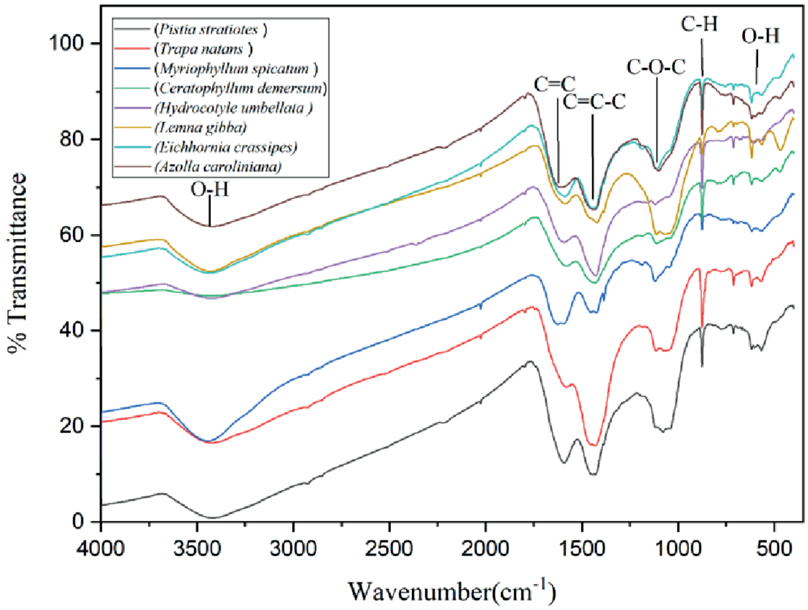


Fig. 2. FTIR spectra of biochars derived from different aquatic plants feedstock.

In this study, at 500ºC pyrolysis temperature biochars were produced, because this temperature was selected on the basis of previous studies. According to them, it was considered that pyrolysis at 500 ºC is an appropriate temperature which gives the high-value biochar [14, 34]. On FTIR spectra all of the biochars had almost similar patterns, which may be ascribed to the same feedstock and same production process. These findings are consistent with [11] in which under the same temperature different wetland plant species were pyrolyzed, thus the variations in function groups of biochar may possibly be ascribed to the variation in lignin, hemicelluloses, and cellulose contents. According to [35], functional groups present in biochar itself are dependent on the characteristics of surface charge which may affect the sorption capability of biochar by plugging transition and non-transition metals onto the surface. It has been investigated that hydroxy and aromatic are the foremost functional groups in all biochars. It was previously reported that these functional groups are responsible for establishing complexes with heavy metals, thus increasing the adsorption of the metals in biochar-amended soils [36].

Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy (SEM-EDS)

The electron microscopy images of all biochars have different micro-morphology due to the difference in plant material (Fig. 3 A1-H1). From the SEM image of TN and LG, it can be seen that they have irregular granular structure with fine pores. The micro-morphology of HU and MC has a smooth mesoporous structure. While, PS and CD have crystalline irregular structures and EC has sponge-like porous structure. AC has fine pores with smooth surface, respectively. Moreover, the EDS spectrum of the surface of each biochar expressed C, P, K, and O in the highest amount, whereas other elements including Mg, Ca, Al, and Fe were present at different proportions (Fig. 3 A2, A3, A4-H2, H3, H4). The different coloured dots having elemental map show the presence of basic elements. The presence of O, C, and Ca in high amount subsequent to the carbonate groups, respectively. SEM images showed the porous texture of all studied biochars. The porous structure in biochar enhances its tendency to adsorb or removal of contaminants [12]. It was observed that MC and HU derived biochars had mesopores (2-50 nm) but in other biochars microporous (1.5 nm) structure, which are possibly useful for metal adsorption [37]. It has been reported that biochar porosity is an outcome of the liberation of minute volatilizable compounds (i.e., H_2O, CH_4, CO_2, and CO) throughout the procedure of thermal conversion [22]. The micromorphology of all biochars showed different structures due to the difference in plant material which is in consistent with [11].

Effect of Biochars on Soil pH and Cd Bioavailability

After two weeks of the incubation period, the application of all aquatic plants derived biochars significantly increased the soil pH (Fig. 4a). Mainly, the highest pH value was observed in the LG and PS derived biochar treatments as compared to the pH of control soil.

The concentration of Mehlich 3-extractable Cd in soil with biochar amendment was determined. All biochars were significantly ($P<0.05$) reducing the Cd bioavailability in the soil (Fig. 4b). PS derived biochar showed a maximum reduction of Cd bioavailability in soil by 60.21%, respectively. On the other hand, LG derived biochar showed the least reduction in Cd bioavailability by 16.88%, as compared to the control.

In this study, all biochars had higher pH and reduced Cd bioavailability in soil but the concentration varied from each other. According to [9], biochars have alkaline pH due to pyrolysis of feedstocks at higher temperature which cause immobilization of heavy metals in soils but it is depended on type and concentration of bioachar in soil. Concentration of soil bioavailable Cd was significantly ($p<0.05$) reduced by the addition of different biochar which varied with types of biochar. In this study, it was concluded that all biochar have the potential to reduce extractable Cd in soil due to their highly alkaline pH with aromatic and alcoholic functional groups as well as higher porosity and irregular structure proposing higher adsorption ability. Previously studies have been carried out on the mechanism of reduction in extractable Cd of soil

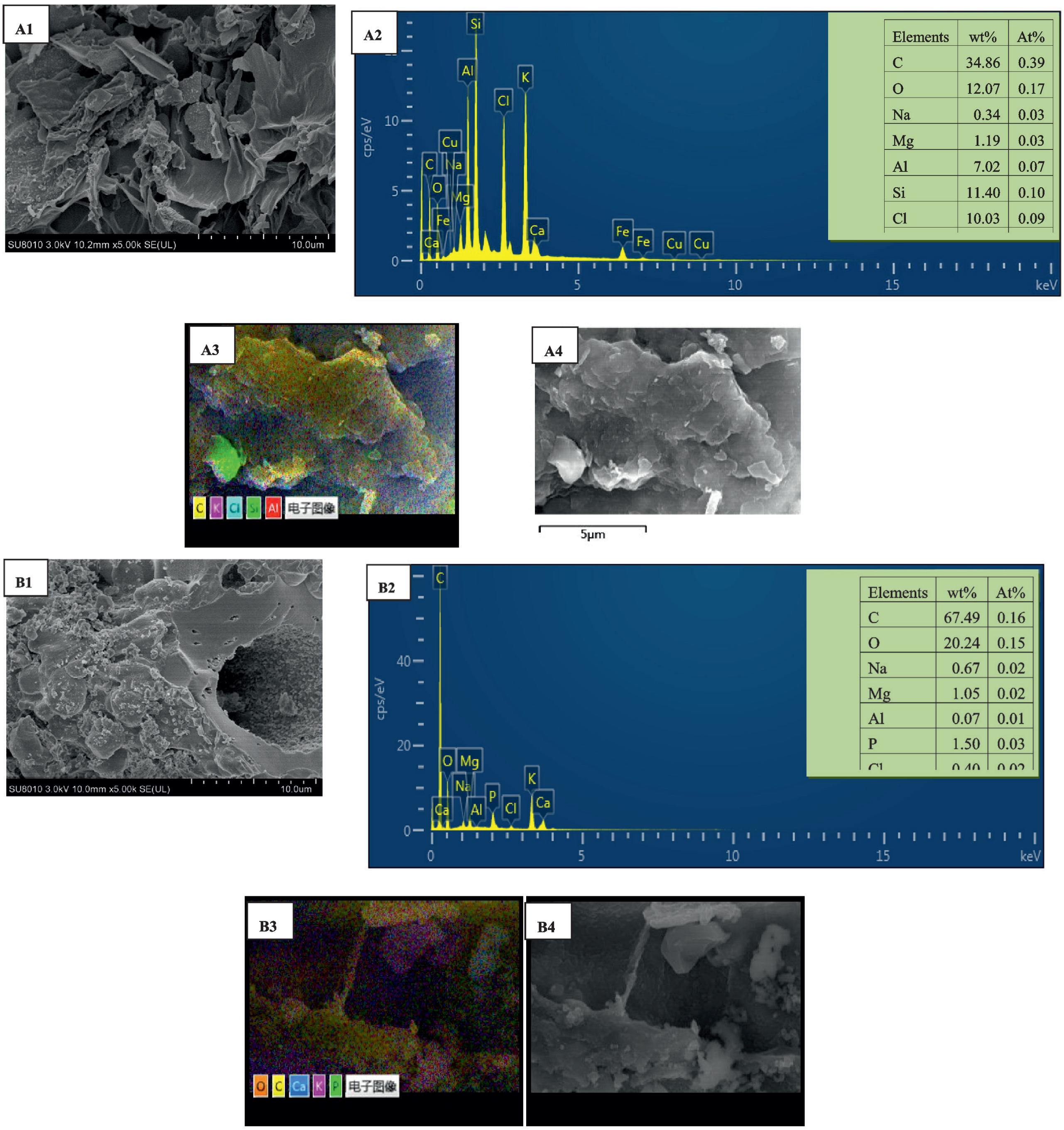
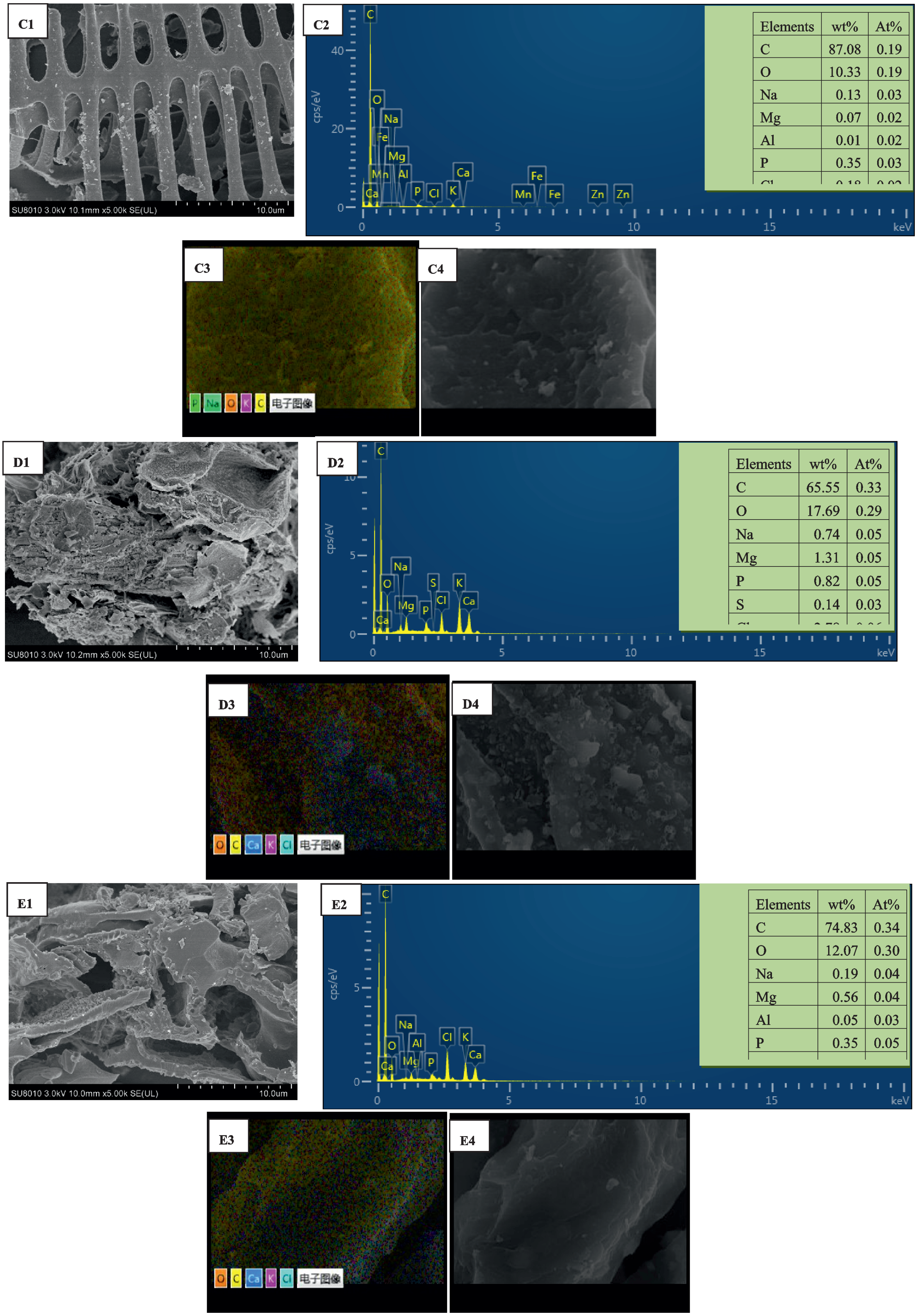


Fig. 3. SEM spectra of biochars derived from different aquatic plants feedstock, (A1)*Pistia stratiotes, (*B1) *Trapa natans,* (C1) *Myriophyllum crispatum,* (D1) *Ceratophyllum demersum,* (E1) *Hydrocotyle umbellata,* (F1) *Lemna gibba,* (G1) *Eichhornia crassipes,* (H1) *Azollacaroliniana* . EDS spectra of biochars derived from different aquatic plants feedstock, (A2,3,4) *Pistia stratiotes, (*B2,3,4) *Trapan atans,* (C2,3,4) *Myriophyllum crispatum,* (D2,3,4) *Ceratophyll umdemersum,* (E2,3,4) *Hydrocotyle umbellata,* (F2,3,4) *Lemna gibba,* (G2,3,4) *Eichhornia crassipes,* (H2,3,4) *Azolla caroliniana.*



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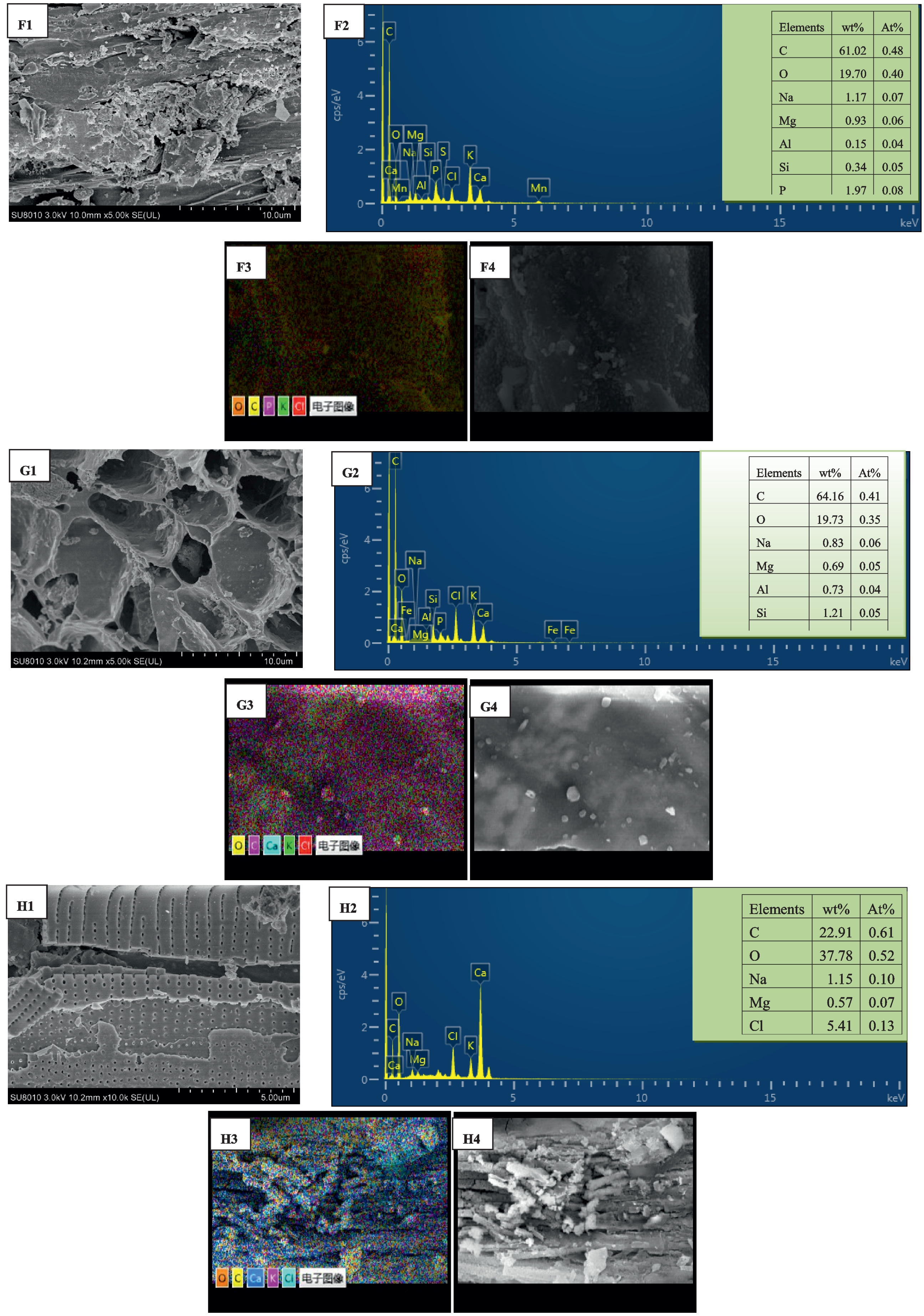

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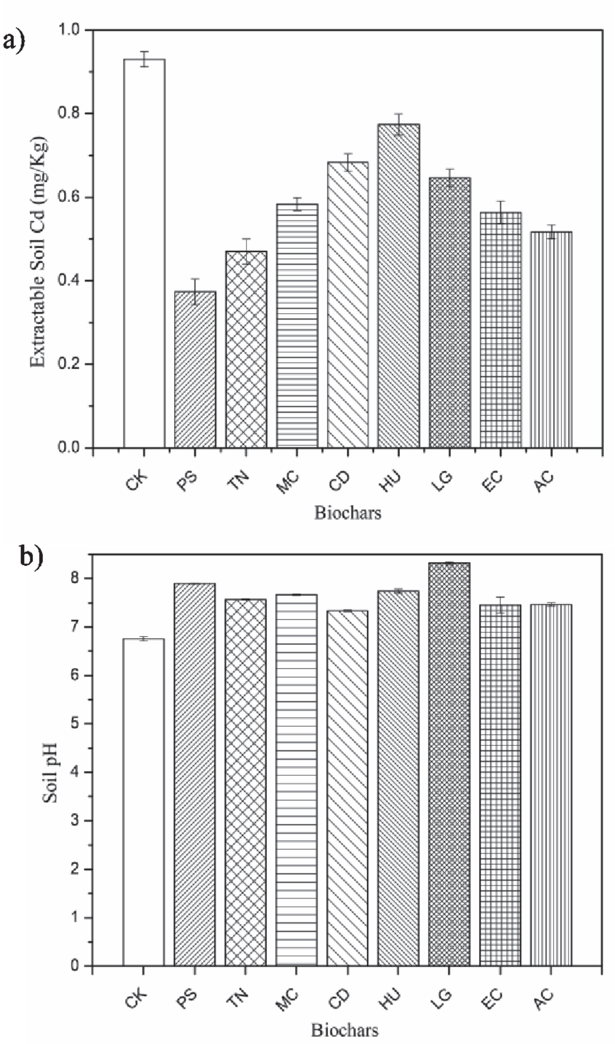

Fig. 4. Extractable cadmium in soil a) and soil pH b) after the application of different biochars. Values are means ± standard error (n = 3).

through different biochar [38, 39]. It has been reported that the mechanism of biochar to directly adsorb Cd includes large specific surface area, functional groups, pore size, chemical precipitation, ion exchange and mineral composition [40, 41]. There are some vital macronutrients like K, P, and Ca essential for better plant growth, therefore their presence in biochar can make the biochar appropriate to use as a soil fertilizer. Among the detected elements in EDS, mainly C, O, K, Ca, and P were dominant in all studied biochars (Fig. 3). According to [13] aquatic plants biochar is rich in essential nutrients and exhibited sorption capability to absorb different types of environmental pollutants which makes it suitable for soil amendment. However, beside biochar aquatic plants are also important biosorbents from water bodies [42, 43]. [44, 45] reported that elemental composition of biochar is affected by the feedstock type and also investigated that after the pyrolysis process some metals (Fe, Al, Ni, Mg, Ca, Zn, Co, Mn, Cu, Cr) and heavy metals (Cd, Pb) remain unaffected and present in the solid biochar. Therefore, it is important to analyze feedstock and biochar as well before applying as water or soil amendment. The SEM-EDX spectrum revealed high amount of C and Ca in all studied biochars which represents the significance of carbonate phases [46]. It has been suggested that SEM and SEM-EDX spectrum appears as a potentially effective way to study micromorpology as well as elemental contents in biochar.

Conclusions

A range of elements and organic compounds were present in all biochars. The physical and chemical characteristics of biochars derived from aquatic plants are mainly influenced by the plant species. SEM-EDX spectrum revealed that all the biochars enclosed high concentrations of nutrients like P, K, Mg, and Fe. FTIR analysis showed higher content of aromatic and alcoholic functional groups proposing higher adsorption ability. Micromorphology showed a higher porosity and irregular structure of almost all biochars in SEM analysis, which may be a highly beneficial characteristic to use as sorbent material and soil amendment especially in Cd-contaminated soils. Thermal analysis suggested less thermally stability of biochar. However, due to the highly alkaline pH of biochars makes it apposite as soil amendment in low pH soils. It has been concluded that biochars obtained from aquatic plant might be valuable as a low-cost approach to use as soil fertilizer and a better way to dispose aquatic plant biomass to avoid eutrophication and due to their beneficial properties can be utilized for agro-environmental purposes as adsorbents for contaminants.

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Conflict of Interest

The authors declare no conflict of interest.

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Supplementary Material

Table S1. Basic physic-chemical properties of soil.

Soil type	Mollisol
pH	6.79±0.03
OM (%)	6
Silt (%)	16
Clay (%)	25
Sand (%)	60
EC (μS/cm)	369±0.91
Total N (g kg^{-1})	1.44±0.02
Total P (g kg^{-1})	0.89±0.04
Total K (g kg^{-1})	5.17±0.01
Total Cd (mg kg^{-1} soil)	0.03±0.02