Hydrochar Prepared from Aquatic and Terrestrial Biomass: Comparative Analysis of Characteristics and Applications

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The benefits of hydrothermal carbonization (HTC) for carbon sequestration, energy, and soil remediation are widely recognized. Up to the present, there has been much research on hydrochar from terrestrial biomass residues, but there is little research on hydrochar based on aquatic plants. In this study, the physical and chemical properties of water hyacinth (representative of aquatic plants) and corn stalk (representative of terrestrial plants) were systematically analyzed under the condition of single hydrothermal carbonization. The results showed that water hyacinth-based hydrochar (WHHC) had well-developed pores, rich functional groups, and high nitrogen content. Among them, the nitrogen content of WHHC was 3.83%, which was more than three times the nitrogen content of corn straw-based hydrothermal carbon (CSHC) (1.11%), and the number of micropores, mesoporous pores, and macropores were also higher than that of CSHC. These differences were attributed to the contrasting growing environments and main components of water hyacinth and corn stalk. These differences revealed their potential application directions: WHHC can be used as an adsorbent and soil amendment; CSHC is more suitable as a supplementary energy source because of its higher carbon content and stability.

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INTRODUCTION

With global attention to renewable energy and resource recycling, hydrothermal carbonization (HTC) is now more relevant. It is a new way to convert biomass into hydrochar with special properties under hydrothermal conditions and provides a new way for efficient utilization of biomass waste. The principle of HTC is that under the conditions of 180 to 300 °C and autogenic pressure, with water as the medium, the macromolecular organic matter in the biomass undergoes hydrolysis, dehydration, decarboxylation, and other reactions. As a consequence, some of the chemical bonds are broken, leading to the generation of sugar, organic acid, and other small organic molecules. In addition, condensation and polymerization take place in the reaction kettle to form the final product hydrochar. At the same time, CO_2, CH_4, and other gas products and water-soluble organic matter are produced (Soroush *et al.* 2023). HTC is affected by many factors, such as the difference in raw materials, reaction time, reaction temperature, solid-liquid ratio, and so on. With the in-depth exploration of the interaction mechanism of these factors, and the integration of HTC and diverse fields, such as materials science, environmental engineering, energy development, and other applications, HTC is expected to become a mainstay in the field of renewable energy and inject a steady stream of vitality for global sustainable development (Huang *et al.* 2023).

Hydrochar, which is the main product of HTC, has unique physical and chemical properties, and has shown a wide range of application prospects. Hydrochar in the process of preparation will form a rich structure of pores, large specific surface area, and provide many adsorption sites. It can be used as a high-quality adsorbent for the removal of heavy metal ions (such as Pd^{2+}, Cd^{2+}, *etc.*) or organic pollutants (such as dyes and pesticides) in water or soil, which is of great significance for environmental remediation (Soroush *et al.* 2023). There are abundant functional groups on the surface of hydrochar, which can be used to regulate the properties of the material. For example, hydroxyl, carboxyl, and carbonyl groups are typically present. These functional groups can chemically react with polymer monomers (such as esterification, etherification, *etc.*), or combine with polymer molecular chains through hydrogen bonding, thereby enhancing the interface compatibility of composite materials and improve the mechanical properties of composite materials (Liu *et al.* 2021). Hydrochar can be used as a high-quality solid fuel, and its high carbon content and energy density can release considerable energy in the combustion process to meet various energy needs (Afolabi *et al.* 2020). At the same time, the raw materials of hydrochar are mostly renewable resources, such as biomass, which realizes the effective use of resources, reduces the dependence on traditional fossil energy, reduces the net generation of greenhouse gas emissions, and has good environmental benefits. Hydrochar also has good chemical stability and can maintain relatively stable structure and performance in different environments. With its rich porosity and surface functional groups, it can be used as a catalyst and can also be applied to soil with nutrients to increase soil fertility and improve soil structure (Langone and Basso 2020). Due to the variety of raw materials of hydrochar, the differences of raw materials greatly affect the characteristics of hydrochar and then affect the potential and efficiency of hydrochar in different application scenarios.

Terrestrial plants and aquatic plants have obvious differences in growth environment and organizational structure. Terrestrial plants usually have relatively well developed xylem, relatively high lignin content, and are relatively stable, and will retain part of their aromatic structure in the hydrothermal carbonization process, which makes the hydrochar prepared by terrestrial plants often have higher carbon content and greater thermal stability (Liu *et al.* 2017a). Aquatic plants generally contain more cellulose and hemicellulose, which are relatively easy to decompose in the hydrothermal carbonization process, and their decomposition products make important contributions to the pore structure and surface functional groups of hydrochar, so that the final product has a higher specific surface area and stronger adsorption properties (Li *et al.* 2015). In the research field of plant raw materials, the comparison between terrestrial plant raw materials has been extensively and deeply explored, and the related research results are quite fruitful. However, in sharp contrast, the research on aquatic plant raw materials is relatively scarce, still in its infancy, and there are a lot of unknowns waiting to be excavated. Recently, researchers looked at one particular aquatic plant and were keenly aware that its properties are quite different from those of terrestrial plants. These differences have significant implications for practical applications.

To address the situation just described, the present work systematically compared the physicochemical properties of hydrochar substances prepared from water hyacinth (representative of aquatic plants) and corn straw (representative of terrestrial plants) under the same conditions. The hydrothermal carbonization reaction conditions of 220 °C and 4 h were selected. At 220 ℃, cellulose and hemicellulose in biomass begin to degrade, and substances containing oxygen-rich functional groups are transformed into carbon networks with extended aromatic domain, which is conducive to improving the degree of hydrothermal carbon carbonization and is suitable for expected applications. The reaction time of 4 hours not only can ensure the full carbonization of biomass, but it also takes into account the production efficiency and cost, while avoiding the excessive accumulation of carbon structure to reduce product performance. Under these conditions, the differences in the preparation of hydrochar from different plant-based raw materials were considered, and their application potential was evaluated. It aims to achieve the goal of efficient and accurate use of biomass waste and promote the innovation and development of biomass energy and materials.

EXPERIMENTAL

Preparation of Hydrochar

Both corn stalks and water hyacinths were harvested from farmland and wetlands in Shanxi, China. Freshly harvested plants were rinsed three times with running water to remove dust and air-dried for a week. The air-dried sample was dried at 80 ℃ for 24 h and ground into powder. All hydrothermal carbonization experiments were conducted in the oven (101-00A, Tianjin Tongli Xinda Instrument Co., Ltd.). The mixture of 5 g raw material and 50 mL deionized water was added to a 100 mL reactor at a 1:10 ratio. Hydrothermal carbonization was carried out at 220 ℃ at a heating rate of 10 ℃ per minute, then maintained at peak temperature for 4 h, and then cooled to room temperature. The filtered hydrocarbon was washed with deionized water and dried at 80 ℃ for 12 h. The obtained hydrocarbon was ground and screened through a 0.25-mm sieve to obtain the final sample. Among them, the hydrothermal carbon prepared by water hyacinth is abbreviated as WHHC, and the hydrothermal carbon prepared by corn stalk is abbreviated as CSHC.

Analytical Methods

The yield of hydrochar is the ratio of biochar quality to raw material quality. The pH value of hydrogen carbon was determined with a pH meter, and the mass/volume ratio of hydrochar to deionized water was 1:20. An organic element analyzer (Elementar Vario EL III, Germany) was used to determine the chemical component content of CHNS in biochar using the CHNS model, and O element content was obtained *via* the difference subtraction method. The specific surface area and pore structure of biochar were measured with a V-Sorb X800TP specific surface area and pore size analyzer. The infrared spectrum of hydrochar was measured using an INVENIO Bruker infrared spectrometer in Germany. The hydrochar powder to be measured was mixed with KBr in a certain proportion, and the tablets were pressed and tested on the machine. The Fourier transform infrared spectrum of the sample was scanned with a resolution of 4 cm^{-1} in the range of 4000 to 400 cm^{-1}.

RESULTS AND DISCUSSION

Physicochemical Properties of Hydrochar

In the process of hydrothermal carbonization, hydrochars prepared from different raw materials show significant differences in elemental composition, and these differences have a profound impact on the properties and applications of hydrothermal carbon. Due to the difference in growth environment, structure, and main composition of water hyacinth and corn straw, the hydrochar obtained by hydrothermal carbonization must be different in element content. This difference not only reflects the characteristics of the raw material itself, but it also is closely related to the reaction mechanism of hydrothermal carbonization.

Table 1. The Physicochemical Properties of Hydrochar

Samples	C (%)	H (%)	O (%)	N (%)	S (%)	H/C	O/C	pH	Yield (%)
CSHC	69.28	6.28	23.09	1.11	0.24	1.09	0.25	4.36	46.19
WHHC	54.91	5.59	35.29	3.83	0.38	1.22	0.48	5.85	41.81

Table 1 shows the basic physicochemical properties of hydrothermal carbon prepared from the two different raw materials. It can be seen from the table that CSHC had a higher content of C and H elements, while WHHC had a higher content of O, N, and S elements. This is due to the high lignin content in corn straw, which is not easy to degrade (Li *et al.* 2020). The structure of lignin is complex and the main connection mode is ether bonds. The molecular arrangement is extremely irregular, so the water temperature is greater than 240 ℃, which is the main reason why the CSHC yield is greater than WHHC. High carbon content means that the CSHC has a higher calorific value, while the higher H content also helps to burn more fully, reduce the production of harmful gases, such as CO, improve combustion efficiency, and can be used as supplementary energy (Afolabi *et al.* 2020). WHHC raw materials are grown in water bodies rich in O, N, and S, so the WHHC has a high O, N, and S content. Biochar with high oxygen content usually contains rich oxygen-containing functional groups and has high hydrophilicity. When applied to soil, it can improve the aggregate structure of soil and increase the aeration and water retention of soil. At the same time, oxygen-containing functional groups have good adsorption capacity for heavy metal ions. They can provide more active sites, which provides great potential for application in pollution adsorption and composite material preparation (Zhang *et al.* 2024). Nitrogen is one of the most essential elements for plant growth. Biochar with high nitrogen content can be used as a slow-release nitrogen fertilizer to provide a continuous nitrogen source for plant growth. Biochar containing nitrogen and sulfur can provide more capacitance and improve the electrochemical performance of the electrode in the preparation of supercapacitors and battery electrode materials (Huang *et al.* 2021).

The main components of biomass are cellulose, hemicellulose, and lignin. In the process of hydrothermal carbonization, cellulose and hemicellulose will undergo a hydrolysis reaction and decompose into formic acid, acetic acid, propionic acid, and other organic acids. At the same time, some ether bonds and double carbon bonds in lignin will be broken and recombined to produce small molecular substances containing acidic functional groups, such as organic acids containing hydroxyl groups, which will also make hydrothermal carbon acidic (Zhuang *et al.* 2019). Because terrestrial plants need stronger mechanical support than aquatic plants to resist gravity and external influences, and cellulose and hemicellulose are important components of plant cell walls, terrestrial plants contain more cellulose and hemicellulose than aquatic plants. Therefore, the CSHC is expected to be more acidic than WHHC. Acid biochar can be used to improve alkaline soil and adjust the pH of composite materials. However, over-acidic biochar may destroy the ecological balance of soil, accelerate the corrosion process of metal materials, and reduce the service life of composite materials. H/C and O/C represent the aromaticity and stability of biochar, respectively, and the lower the value, the stronger the performance. The aromaticity and stability of CSHC are higher than WHHC, and it can maintain good performance in high temperatures or harsh environments and is not easy to change (Liu *et al.* 2017c). Figure 1 shows the relationship between the H/C and O/C atomic ratios of biomass, hydrochar, peat, lignite, coal, and anthracite. CSHC is close to the lignite region, and the WHHC is close to peat, both of which obtain better carbonization effects.

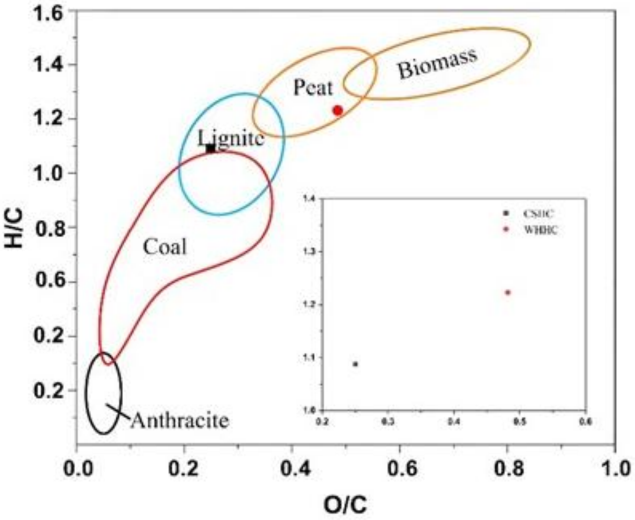

Fig. 1. The Van Krevelen diagram of hydrochars, in which biomass, peat, lignite, coal, and anthracite represent the atomic ratio range of their raw materials

Surface Structure Characteristics of Hydrothermal Carbon

Figure 2 and Table 2 summarize the test results of the specific surface area and pore size of the two types of hydrothermal carbons. As shown in Fig. 2 (a), when P/P_0 was very low, the adsorption capacity rose sharply, and the subsequent curve was nearly horizontal. The adsorption capacity was close to a limit value, which is a typical Langmuri/ Type I isotherm. In the medium relative pressure region, the mesoporous filling became the main adsorption process. Capillary condensation occurred in the mesopores, and hysteresis loops appeared in the isotherm, which accords with the characteristics of type IV isotherm (Tian *et al.* 2020). In the high relative pressure region, the adsorption capacity increased abruptly, mainly in the macroporous adsorption and multilayer adsorption stages. Therefore, the number of micropores, mesoporous pores, and macropores in WHHC was larger than that in CSHC, while the pores in CSHC were underdeveloped. It can be seen from Fig. 2(b) that WHHC had higher porosity in smaller pore size, indicating that the WHHC had more pore volume and micropores in the micropore region. The microporous structure enables WHHC to have a higher specific surface area and can provide more adsorption sites, which can effectively adsorb small molecular pollutants in the environment, such as heavy metal ions and organic small molecules. When the WHHC is applied to the soil, its rich pore structure can increase the aeration and water permeability of the soil and promote the respiration and growth of plant roots (Garbuz *et al.* 2021). At the same time, the larger specific surface area can provide more capacitance, on account of the electrical double layers at the charged surfaces. The pore structure is conducive to the rapid diffusion and migration of electrolyte ions, to improve the specific capacitance and charge and discharge performance of supercapacitors. The porosity of CSHC was relatively low and flat throughout the pore size range, indicating that the pore volume distribution of CSHC was relatively uniform, but the overall pore volume was small. The more uniform pore structure of CSHC is conducive to the dispersion of active components so that the catalyst can be evenly distributed in the pore, improve the activity and stability of the catalyst, and promote the catalytic reaction (Tao *et al.* 2021). Although the CSHC micropores were not as abundant as the WHHC, the relatively uniform pore structure can adapt to the adsorption of macromolecules of a certain size, such as the adsorption of biological macromolecules such as proteins in bioengineering.

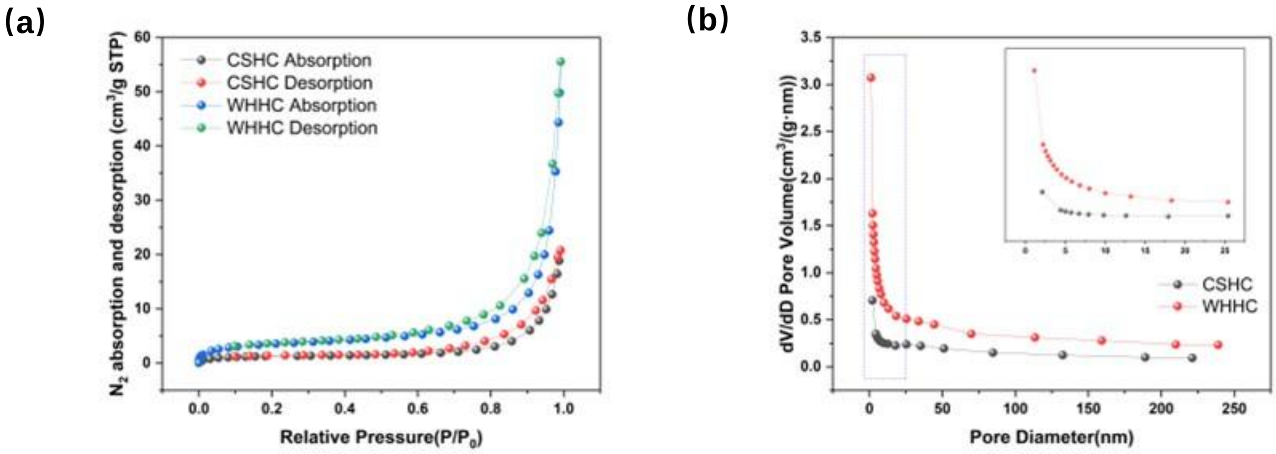

Fig. 2. (a) Adsorption and desorption curve of the specific surface area of hydrochar; (b) Hydrochar pore volume adsorption curve

Table 2. Textural Properties of Hydrochar

Samples	Surface Area ($m^2 \cdot g^{-1}$)	Pore Size (nm)	Pore Volume ($cm^3 \cdot g^{-1}$)
CSHC	4.2573	26.3339	0.0322
WHHC	5.7151	31.4649	0.0517

Infrared Analysis of Hydrothermal Carbon

It can be seen from the spectrum diagram of the two kinds of hydrothermal carbon in Fig. 3 that there was a strong absorbance peak at 3400 cm^{-1}, which was triggered by the O-H stretching vibration on hydroxyl and carboxyl groups, indicating that the hydrothermal carbon contained -OH and the wide spectrum band was due to the presence of hydrogen bonds. There are two characteristic absorption peaks at 2832 and 2717cm^{-1}, which are relatively weak and generally induced by C-H stretching vibration on the aldehyde group (Liu *et al.* 2017b; Qiu *et al.* 2021). Near 1585 cm^{-1}, the absorption peak is strong and there is a large amount of C=C. This is due to the dehydration reaction of cellulose, hemicellulose, and lignin in the hydrothermal carbonization process of biomass, resulting in changes in the structure of carbon chains and the formation of carbon-carbon double bonds between adjacent carbon atoms (Choi *et al.* 2021). At the same time, lignin contains a benzene ring structure, and aromatization may occur during the reaction process, indirectly forming carbon-carbon double bonds. The peak at 1361 cm^{-1} is a C-H symmetric deformation vibration absorbance peak, indicating the presence of -CH_3 in hydrothermal carbon, which may be caused by the retention of methoxy groups in lignin, or the decomposition of complex organic compounds in biomass to generate some small molecular fragments containing methyl groups, thus forming methyl functional groups (Jacob *et al.* 2013; Getnet *et al.* 2020). At 1106 cm^{-1}, the CSHC had a characteristic absorbance peak, while WHHC did not. This shows that there are C-O groups connected with alkyl groups in CSHC, that is, ether bonds, because the ether bonds of lignin in raw materials may not be completely broken under relatively mild hydrothermal carbonization conditions and can be retained (Li *et al.* 2020). The absorbance peak at 775 cm^{-1} corresponds to aromatization C-H, and there was interbenzene disubstitution.

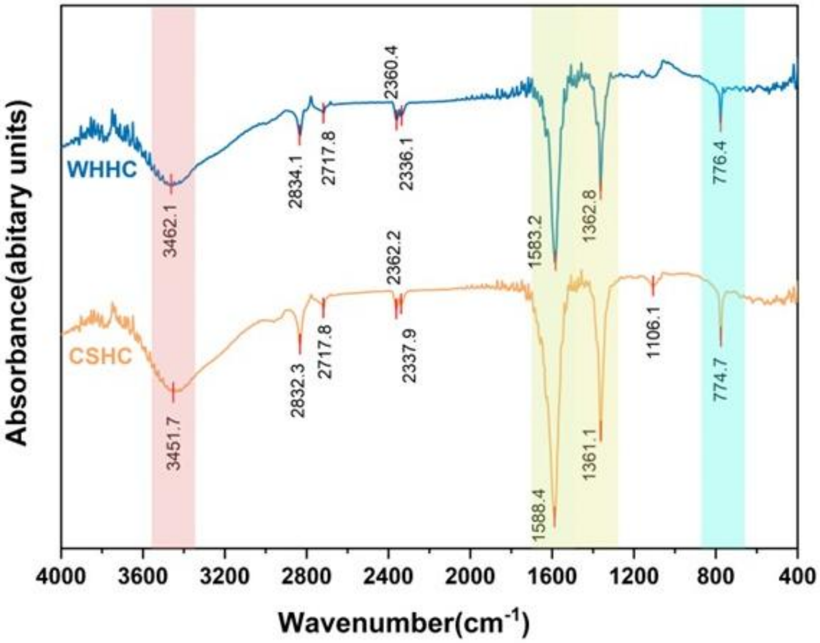

Fig. 3. The FTIR spectra of hydrochar

Combined with the infrared spectra of hydrochar and atomic ratio analysis, CSHC showed good stability because of its unique molecular structure and composition. Compared with WHHC, the CSHC will undergo obvious structural changes in the face of some acid-base substances or at higher temperatures during pyrolysis and is suitable as a catalyst carrier. At the same time, the aromatic structure of CSHC is not easy to decompose and volatilize prematurely in the combustion process. It can be continuously and stably burned to reduce energy fluctuations in the combustion process and improve combustion efficiency (Jin *et al.* 2023). However, incomplete combustion may exist in the combustion process, resulting in partial energy cannot be fully released, thus limiting the further improvement of energy conversion efficiency. The WHHC has more oxygen-containing functional groups, which can adsorb organic pollutants through hydrogen bonding and Π-Π interaction. It can also interact with mineral particles and organic matter in the soil, increase the cation exchange capacity (CEC) of the soil, and improve the soil microbial ecological environment. At the same time, hydroxyl and carboxyl groups and other oxygen-containing functional groups can undergo reversible REDOX reactions, so WHHC can be used as electrode materials for supercapacitors to improve the performance of capacitors (Amjadipour *et al.* 2020). It can also be used as a negative battery material to provide more active sites for the adsorption and desorption of lithium electrons to improve battery performance.

CONCLUSIONS

In this study, the hydrothermal carbonization products of water hyacinth and corn straw were systematically compared under the same conditions, but the conditions were single and had limitations, so it will be necessary to continue to optimize the reaction conditions for different application directions, providing a reference for the precise application of plant-based hydrothermal carbonization. The research conclusions are as follows:

1. The comparison of hydrothermal carbon content showed that corn straw-based hydrothermal carbon (CSHC) can be used as a supplementary energy source due to its complex lignin structure, high C and H content, and strong aromatics. By contrast, the water hyacinth hydrothermal carbon (WHHC) that had been prepared under the same conditions had high O, N, and S content, and weak acidity, and can be used as a soil amendment.
2. The number of micropores, mesoporous pores, and macropores of WHHC was greater than that of CSHC, with abundant pores and high specific surface area, which can provide adsorption sites and effectively adsorb small molecular pollutants in the environment. The pore distribution of CSHC was more uniform, which is conducive to the dispersion of active components, and can be used as a catalyst carrier and adsorption of macromolecular substances.
3. CSHC surface functional groups contained a large number of C-H bonds. The CSHC exhibited good stability in the face of some acid-base substances or high-temperature reaction. During such processes it remained relatively stable, and it was not easy to change significantly. By contrast, WHHC had more oxygen-containing functional groups and can be used as electrode material for supercapacitors in reversible REDOX reactions.

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