

BIOCHAR AND HYDROCHAR AS ADSORBENTS FOR THE REMOVAL OF CONTAMINANTS OF EMERGING CONCERN FROM WASTEWATER

Aleksandra Adamović¹, Mirjana Petronijević^{2*}, Sanja Panić², Dragan Cvetković¹, Igor Antić², Zoran Petrović³, Nataša Đurišić-Mladenović²

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¹University of Niš, Faculty of Technology, Leskovac, Serbia

²University of Novi Sad, Faculty of Technology Novi Sad, Novi Sad, Serbia

³University of East Sarajevo, Faculty of Technology, Zvornik, Republic of Srpska, Bosnia and Herzegovina

Carbon materials have been used extensively in water treatment, and among them, biochar (BC) and hydrochar (HC) have been studied in recent decades as eco-friendly adsorbents in water treatment. Specific physicochemical characteristics of chars allow for the effective removal of a wide range of water contaminants, including emerging contaminants (e.g. pharmaceutically active compounds, agrochemicals, and endocrine disrupting compounds). BC and HC can be synthesized from different biomass sources, even from biowaste, which makes them low-cost materials. In fact, the application of waste biomass for char production can be designated as a promising way towards achieving sustainable development goals concerning solid waste management. The selection of feedstock material and optimization of the reaction conditions of the carbonization process (pyrolysis, hydrothermal carbonisation, etc.) influence the performances of char-based materials leading to the products with desired characteristics suitable for the removal of a certain group of compounds. The objective of this review is to provide an overview of the current status of research regarding the use of BC and HC as adsorbents in the removal of contaminants of emerging concern (CECs) from wastewater. The physicochemical properties of the substrates, types of synthesis, characterization techniques, as well as mechanism and influence of process parameters in the removal of selected CECs from wastewater, will also be addressed.

Keywords: biochar, hydrochar, adsorption, contaminants of emerging concern (CECs), water treatment

Introduction

Population growth demands the invention of new technologies and/or the improvement of existing ones in order to provide enough energy and food for mankind. At the same time, constant industrialization is among the main sources of harmful pollutants in the environment. An indispensable product of industrial and living activities is the production and discharge of wastewater containing various contaminants. Contaminants that can be found in wastewater are pharmaceutically active compounds, currently used pesticides, endocrine-disrupting compounds, phenols, X-ray contrasting agents, food additives, surfactants, personal care products, veterinary products, steroids and engineered nanomaterials. Most of the mentioned organic contaminants have been identified as potentially dangerous to environmental health and/or humans recently. The commonly accepted name for such compounds is compounds of emerging concerns, CECs. CECs enter the environment in different pathways. These contaminants can be found in surface waters in a wide concentration range (from a few ng/L to µg/L), and up to a few mg/L in wastewater [1, 2]. However, the impact of all these components on the environment

and human health, as well as the impact of their decomposition and transformation products, is still unknown. The majority of these compounds are not covered by environmental quality standards (EQS) based on annual average (AA-EQS) and maximum allowable (MAC-EQS) concentrations defined by Directive 2013/39/EU under the currently active European Union (EU) water legislation, the Water Framework Directive (WFD, 2000/60/EC) [3]. Only for a limited number of pharmaceutically active compounds, does the appropriate data exist on ecotoxicity that is adequate for risk assessment. The European regulation failed to impose allowed limits for CECs presence in the environment with the exception of some compounds (five pharmaceuticals, one synthetic and two natural hormones, one antioxidant and one organic UV filter, and eight pesticides) listed under the EU Watch List for Which Predicted No-Effect Concentrations (PNECs) where established [4].

There are several techniques that could be used for organic contaminants removal from water matrices, such as different oxidation processes, coagulation, adsorption, etc. However, liquid-solid adsorption stands out

***Author address:** Mirjana Petronijević, University of Novi Sad, Faculty of Technology Novi Sad, Bulevar cara Lazara 1, Novi Sad, Serbia;
email: mirjana.petronijevic@uns.ac.rs
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among them as an economically profitable process that can be used to achieve a high degree of pollutant removal [5]. At the same time, adsorption is a technologically simple process that could be easily implemented as a new one or within the existing water treatment systems. The efficiency of pollutant removal by adsorption occurs on active sites located on the surface of the adsorbent material. Numerous factors, including temperature, pH, and adsorbate concentration, can influence the capacity of the adsorption process. Fluctuations of these factors can also result in the desorption of pollutants from the surface back into the water [6]. The primary mechanisms involved in the adsorption of organics are hydrophobic and electrostatic interactions, the formation of hydrogen bonds, partitioning, pore-filling, and π - π interactions between the adsorbent and organic pollutant molecules [7].

Carbon materials represent a group of very important cost-effective and eco-friendly sustainable adsorbents with high efficacy for organic pollutants removal [6, 8-10]. Biochar (BC) and hydrochar (HC) are carbon materials obtained by dry/wet carbonization of biomaterial, respectively. In general, characteristics such as specific surface area (SSA) and porosity are significantly higher for BC than HC. However, those parameters are affected by the type of raw material and reaction conditions. Still, there are cases where HC with a high SSA is also obtained, or HCs have to be activated. For example, authors Kabakci and Baran synthesized HC from different lignocellulosic biowastes (wood sawdust, olive pomace, walnut shell, apricot seed, tea stalk, hazelnut husk) [11]. The HCs yield was in the range of 55.7-71%, and the SSA of HCs after KOH activation was 308.9-666.7 m²/g [11]. Carbonization encompasses a variety of technologies which can be classified into three main processes - pyrolysis, hydrothermal processes and gasification [12, 13]. BC is produced by pyrolysis in the absence of oxygen and generates bio-oil and combustible gas as by-products. On the other hand, HC is obtained from hydrothermal processes, such as hydrothermal carbonization (HTC) and hydrothermal liquefaction (HTL) [10, 13]. The production of HCs is significantly higher when using the HTC method (12-91% from lignocellulosic biomass and 31-94% from sewage sludge) [14], compared to HTL (16-38% from lignocellulosic biomass [14] and 29-43% from soils [16]). The advantage of the hydrothermal process in relation to pyrolysis is that wet biomass can be used directly, without the need for drying. The yield and characteristics of chars largely depend on the conditions of the applied process (pressure, temperature, reactor design, heating rate, residence time, and the nature of the raw biomaterial) [14, 17, 18]. Also, the properties of chars could be tailored by using different catalysts during their production [19].

Biomass from a wide range of sources can be used to produce both BC and HC with favourable characteristics. Lignocellulosic biomass, such as crop and forestry residues, as well as non-lignocellulosic materials, such as sewage sludge, algae, sugar, hair, bone, and meat

residues, can all be utilized to produce high-quality chars [14, 20]. BC produced from lignocellulosic biomass at temperatures ≥ 500 °C is characterized by a high content of carbon (>60%) and low to medium ash content (10-21%) [20]. In contrast, BC produced from animal manure has high ash content (48-67%) and a very high pH value due to the inorganic materials present in the biomass sources [9, 20]. HCs derived from different lignocellulosic biomass (crop and forestry residues) are mostly characterized by low ash content (up to 13%) [14]. The total carbon content in the structure of HCs obtained from lignocellulosic biomass is high (38-91%) [14]. HCs obtained from non-lignocellulosic biomass (e.g. sewage sludge), are characterized by a wide range of ash (23-82%) and the total carbon content (11-85%) [14]. The application of waste biomass for char production can be designated as a promising way towards achieving sustainable development goals in relation to solid waste management.

This review aims to provide the latest research data in the area of CECs removal from water matrices using BC, HC and their composites. The methods for preparation and physicochemical characterization of BC and HC are comprehensively reviewed, along with the adsorption mechanisms of organic pollutants (pesticides, pharmaceuticals, etc.) on the appropriate chars.

PRODUCTION OF BIOCHAR AND HYDROCHAR

The most widely used biomass conversion technique for the production of BC is pyrolysis. Formation of BC also follows the process of biomass gasification, but in much lower yields than is the case for pyrolysis. On the other hand, HCs are primarily produced through hydrothermal processes such as HTC and HTL. The reaction conditions such as temperature, pH, heating rate, residence time, substrate concentration and feedstock type play significant roles in the carbonization process, influencing yield and physicochemical characteristics of the produced chars. The parallel between the two mean carbonisation processes and obtained products is present in Figure 1.

Pyrolysis

Pyrolysis represents a method for producing BC from biomass due to its versatility and effectiveness. This thermal decomposition process is carried out at high temperatures (typically 400-800 °C) without the presence of oxygen [21]. This process breaks down the natural polymers present in biomass such as cellulose, hemicellulose, and lignin, resulting in the production of biochar, bio-oil, and non-condensable gases [22, 23]. Natural polymers undergo several transformations [23], such as disintegration, cross-linking, and then fragmentation [24]. Hemicellulose decomposes at temperatures between 200-260 °C, followed by cellulose (240-350 °C), and lignin (280-500 °C) [25]. Pyrolysis can be classified into different categories based on product requirements, operating conditions, and feedstock type, which are described below.

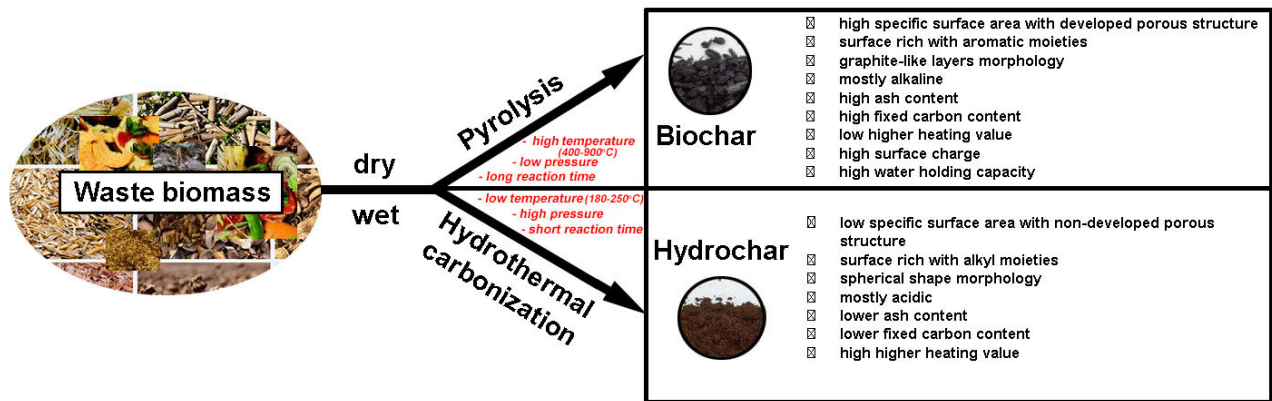


Figure 1. Main characteristics of chars produced by pyrolysis and hydrothermal carbonization.

Slow pyrolysis is a conventional method performed at temperatures < 700 °C and is characterized by a long retention time (> 1 h) and a slow heating rate ($5\text{--}7$ °C/min). The main product at the end of the process is BC (35–45% yield), along with bio-oil (25–35% yield) and gas products (20–30% yield) [26–28].

Fast pyrolysis is popular in the biofuels industry and is characterized by high reaction temperatures (300–1000 °C), high heating rates (typically > 300 °C/min) and short retention time (< 2 s) [29]. Primarily, it is used in the biofuel industry to produce bio-oil (65–70% yield), while BC and gas yields are lower (10% and 20%, respectively) [28].

Flash or ultra-fast pyrolysis represents an improved type of fast pyrolysis utilizing a higher heating rate (> 1000 °C/s) and a very short reaction time (0.1–1 s). The reaction temperature can vary between 700–1000 °C (even up to 1200 °C) [30]. The main product of flash pyrolysis of biomass is bio-oil (70–80% yield), while BC and gas are obtained in a markedly lower percentage (10–15 and 5–20%, respectively) [28, 31]. Bio-oil is viscous and contains high levels of oxygen-containing compounds [33] and it can be used as a bioliquid for heating or as biofuel in transport, but after appropriate treatment [32].

The main product obtained through slow pyrolysis is biochar, while bio-oil is the main product of flash pyrolysis. Generally, the yield of bio-oil increases as the formation of biochar decreases, irrespective of the pyrolysis method employed, whether it is slow, intermediate, fast, or flash pyrolysis. On the other hand, the gas fraction does not exhibit significant variations among different conventional pyrolysis techniques. Notably, the introduction of catalysts alters the distribution and composition of the pyrolysis products, typically promoting deoxygenation reactions. The techno-economic analysis demonstrates the potential for scaling up the pyrolysis process. However, current research on this topic remains fragmented, with limited comprehensive studies that compare conventional, advanced, and emerging approaches to biomass pyrolysis from an economic perspective. This highlights the need for extensive research in the future to address this gap and provide a more comprehensive understanding of biomass pyrolysis in terms of its eco-

nomical viability [33].

Gasification

Unlike pyrolysis, gasification is a thermochemical process that includes the degradation of carbonaceous feedstock at high temperatures, typically ranging between 700–1500 °C. The process takes place in the presence of an oxygen-based gasification agent (air, pure oxygen, steam, etc.) [18, 28, 34, 35]. While biomass conversion to BC via gasification is possible, it is not commonly reported, as the primary product of gasification is syngas (i.e. H_2 , CO , CO_2 , CH_4), with a yield of approximately 85% [28]. Biochar and bio-oil are considered by-products with a much lower yield (10% and 5%, respectively) [18, 28, 34].

Hydrothermal carbonization

In contrast to the aforementioned thermochemical methods of char production, hydrothermal processes for biomass conversion involve suspending biomass in water under moderate temperature conditions, typically between 180–350 °C and at pressures ranging from 2–22 MPa [36, 37]. These processes are characterized by relatively short retention times (5–240 min) and involve a series of reactions such as decarboxylation, dehydration, condensation, and hydrolysis, which are initiated by the presence of water in a subcritical state [38]. The primary product of HTC is a solid residue (HC), which accounts for 35–80% of the product yield [37]. Other by-products include liquid bio-oil and gases, mainly consisting of CO_2 and CO [39].

When biomass is subjected to hydrothermal processes, hemicellulose is broken down into oligosaccharides, pentoses and hexoses, while cellulose breaks down into hexose. The process of converting monosaccharides into furfural and hydroxymethylfurfural involves several reactions, including ring opening, intramolecular cyclization, dehydration, etc. [40]. Polymerization of furfural and hydroxymethylfurfural results in the formation of hydrocarbon microstructures. The hydrochar network is created through solid-solid conversions of the unhydrolyzed fraction of cellulose and hemicellulose, as well as lignin

[40]. Due to its low solubility in water, lignin, an amorphous heteropolymer, undergoes hydrolysis to form soluble fragments, including aromatic monomers, dimers, and oligomers. This hydrolysis process plays a limiting factor in determining the formation of secondary char. The remaining insoluble lignin undergoes mild dehydration and decarboxylation reactions, resulting in the formation of pyrolysis char [41]. Hydrochars derived from non-lignocellulosic biomass, such as sewage sludge, typically exhibit characteristics such as low heating value, low surface area, limited dewatering ability, and high concentrations of heavy metals [14].

Temperature plays a crucial role in the hydrothermal process, as it directly affects the conversion of biomass to HC. Higher temperatures lead to higher conversion rates of biomass to HC, but this is accompanied by a reduction in the HC yield and an increase in bio-oil and gas yields [40]. Increasing the residence time of biomass in the process also influences the conversion rate similarly to temperature [42].

However, the improvement of the quality of HCs, such as sewage sludge-derived HCs, can be done by applying the HTC process that involves treating two different raw materials simultaneously (Co-HTC). During Co-HTC, the interaction between these raw materials leads to synergistic effects. This approach offers several advantages over single-feedstock HTC. It overcomes the limitations of relying solely on a single feedstock, mitigates any undesirable characteristics of individual raw materials, etc. [14].

Hydrothermal liquefaction

Hydrothermal liquefaction (HTL) is a process that involves the use of supercritical water and high temperatures (250–375 °C) and pressure (10–22 MPa) to convert biomass into bio-oil, with relatively low yields of HC and gas [40]. The yield of HC is dependent on various factors, such as feedstock characteristics and reaction conditions, and can reach up to 15% [43]. However, there are examples where by using HTL a high yield of HC was obtained. Ahmad et al (2019) subjected the antibiotic fermentative residue (lincomycin residue) to microwave-assisted hydrothermal liquefaction and obtained HC yield in the range of 45-93% [44]. The effect of temperature on biomass components can differ in HTL, with cellulose, hemicellulose, and lignin reacting differently to temperature changes [37]. The reaction conditions of the HTL process also have a significant impact on the resulting products. Panchal et al. (2017) found that increasing the temperature from 250 °C to 350 °C led to a decrease in the yield of bio-oil, while increasing the pressure from 10 MPa to 22 MPa led to an increase in the yield of bio-oil. Additionally, the composition and properties of the bio-oil were found to be affected by the reaction conditions, with higher temperatures leading to a higher concentration of oxygen-containing compounds and lower concentrations of nitrogen-containing compounds [45].

CHARACTERIZATION OF BIOCHAR AND HYDROCHAR

The properties of BC and HC are influenced by several factors including the raw material, synthesis process, and reaction conditions. To assess the quality of the obtained chars and determine their potential application, it is necessary to carry out their detailed characterization (e.g. SSA, porosity, and elemental composition). To achieve this, various instrumental techniques need to be applied. A thorough understanding of the relationship between the production conditions and physicochemical properties of the chars is essential for their optimal utilization.

Characterization techniques

The properties of chars can be classified into four main categories: chemical properties, physical properties, surface structure and morphology, and thermal stability properties [9]. The instrumental techniques that are most often used in the characterization of chars are present in Table 1.

Table 1. Instrumental techniques for char characterization (modified from [9, 12, 20])

Properties of char	Techniques	
Chemical property	Point of zero charge (PZC) analysis	
	Electrical conductivity	
	Boehm titration (Surface acidity/alkalinity)	
	Ion chromatography (Cation exchange capacity)	
	CHNS analysis, EDS, XPS (Elemental composition)	
	Gravimetry (Proximate analysis: Moisture, Ash, Volatile organic matter, Fixed carbon)	
	TGA (Proximate analysis)	
	FTIR, Raman spectroscopy (Surface functionality)	
	¹³ C NMR, Raman spectroscopy (Surface aromaticity)	
	Physical property	Textural characteristics (Specific surface area (BET), pore volume, pore size distribution)
Laser sizing (Particle size distribution)		
Mercury porosimetry for macro- and meso-porosity analysis, Pycnometer (Density)		
Thermal stability		TGA-DSC
		SEM
Surface structure and morphology	TEM	
	XRD, Raman spectroscopy (Structure of crystallites)	

CHNS analysis - Content of Carbon, Hydrogen, Nitrogen and Sulphur analysis; EDS - Energy-dispersive X-ray spectroscopy; XPS - X-Ray photoelectron spectrometry; TGA-DSC - Thermal gravimetric analysis and Differential scanning calorimetry; FTIR - Fourier transform infrared spectrometry; NMR - C wide-cavity solid-state nuclear magnetic resonance spectroscopy; SEM - Scanning electron microscopy; TEM - Transmission electron microscopy; XRD - X-Ray diffraction spectroscopy

Biochar and hydrochar properties

BC and HC show different physical and chemical properties that significantly affect their potential application. It is important to note that BC and HC have unique properties that vary depending on several factors, such as the type of biomass used, the reaction temperature, and the residence time [46]. The synthesis process involves complex chemical reactions, leading to significant differences in the physicochemical properties of BC and HC. Table 2 and Table 3 provide a summary of the characteristics of BCs and HCs derived from various biomass. It is crucial to understand the correlation between char production conditions and their properties to determine their potential applications accurately. The relation between the characteristics of BC/HC and their potential application is shown in Figure 2.

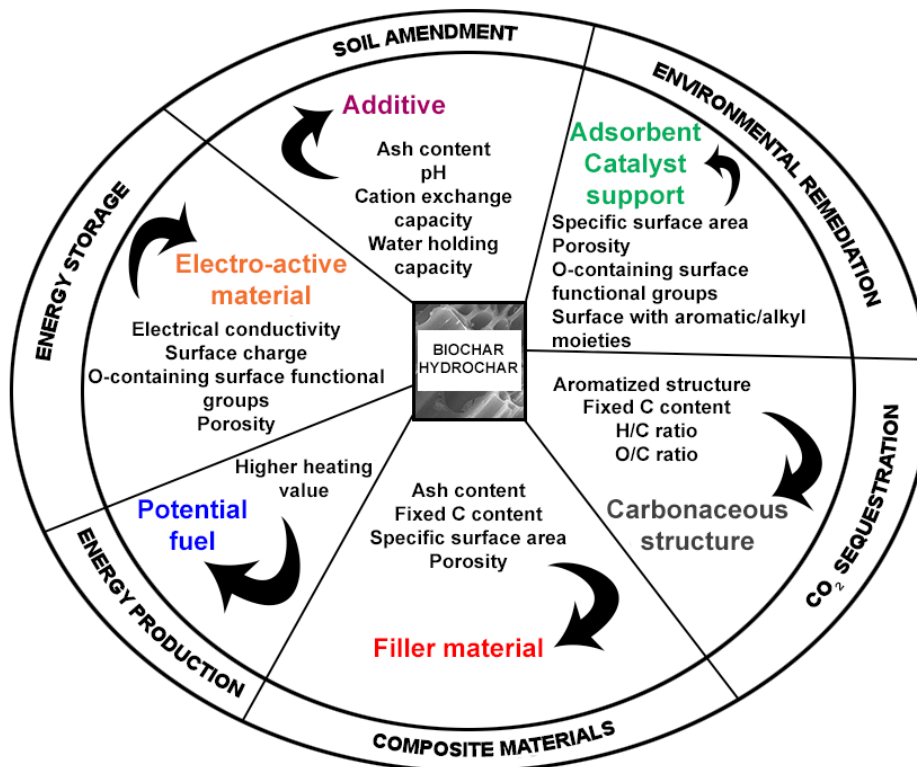


Figure 2. Characteristics of chars important to its potential applications.

The composition of char is highly dependent on the feedstock used for its synthesis, with lignocellulosic waste biomass being the most common. The amounts of cellulose, hemicellulose, and lignin present in the feedstock greatly influence the composition of the resulting char. Moisture (MO), ash, volatile matter (VM), and fixed carbon (FC) content in both BC and HC are mainly determined by the feedstock composition (Tables 2 and 3). Industrial waste-derived BCs have higher ash content compared to other feedstocks. The VM content can range from 24.9% to 78.3%, the ash content from 2.9% to 65.7%, and the FC content can be up to 54.8% in various feedstock-derived BCs (Table 2). HC is produced in an aqueous medium, and therefore, inorganic substances from the biomass are removed resulting in lower ash content, while pyrolysis-derived BC retains the minerals present in the feedstock [47]. Depending on the initial biomass, the largest part of HC represents VM (16.8–83.8%) and ash (0.1–82.0%), while FC ranges from 0.1 to 53.9% (Table 3). In general, the highest percentage of VM is present in HC obtained from algae, wood, and urban waste; while the highest yield of FC was measured in HC derived from wood waste biomass.

The reaction temperature and duration of the reaction have a significant influence on the physicochemical properties and yield of BC and HC. There is a correlation between the applied reaction temperature and HC yield and carbon conversion. When the synthesis takes place at low temperatures and with a short retention time, a high yield of char occurs, but a low carbon conversion is

achieved, resulting in chars with low heating value (HV) [48]. As the reaction temperature increases, the HV of the char increases. This happens because, at higher temperatures, cellulose and hemicellulose are broken down, leaving lignin, which has a higher heating value. HC has a higher heating value compared to BC, due to the presence of more volatile and oxygen-containing functional groups. During HTC, which is performed in an aqueous medium at milder temperatures, hemicellulose is degraded faster [49]. In contrast, BC produced via pyrolysis has a lower heating value due to the removal of oxidative functional groups during the process, resulting in a more carbon-rich material. The atomic ratio of H/C and O/C for HC is higher compared to those for BC [50].

The temperature used in the synthesis process has a significant impact on the physical and chemical properties of both BC and HC. BC produced at temperatures around 500–600 °C is mainly aromatic and has a graphite-like structure. In contrast, HC produced at lower temperatures about 200–250 °C contains residues more alkyl in nature [51]. BC synthesized at moderate temperatures may possess O and N functional groups on its surface, while BC produced at temperatures over 700 °C becomes hydrophobic and resistant to degradation due to the loss of almost all O and H in its structure [17]. Considering that it is produced at low temperatures, the greater presence of oxygen groups on the surface of HC contributes to its acidic character compared to BC, which is alkaline in nature. Additionally, BCs have an alkaline pH due to the high content of inorganic carbonates and ash. BCs

have a large SSA and pore volume [52, 53], which increases with increasing temperature. BC with high SSA (up to 1570 m²/g) is usually obtained from agro-waste and lignocellulose biomass (Table 2). The BC with extremely low SSA (0.59 m²/g) reported in the literature was groundnut char. The hydrochars derived from agrowaste are characterized by high SSA and pore volumes amounting up to 667 m²/g and 0.39 cm³/g, respectively (Table 3) [11]. The lowest SSA was recorded for HCs derived from industrial waste.

BC and HC were found to be effective adsorbents in water treatment. However, in order to improve their adsorption capacity and selectivity, these chars are often subjected to activation/functionalization steps. The characteristics of some BC/HC-based adsorbents after modification are shown in Table 4.

Activation can be achieved through physical or chemical treatments that increase the char's porosity and surface area, resulting in enhanced adsorption capacity. Physical activation (thermal or dry activation) involves heating the char in the presence of steam or CO₂, while chemical activation implies the utilization of chemical activation agents and mostly includes acid modification, alkaline modification, metal salts, and/or oxidizing agent modification [9]. Acid activation (with sulphuric acid, hydrochloric acid, nitric acid, oxalic acid, phosphorous acid, and citric acid) is necessary to modify the char surface by introducing acid functional groups and eliminating metallic impurities. Consequently, the obtained function-

alized char can selectively adsorb target contaminants from water. Alkaline activation (with sodium hydroxide and potassium hydroxide) can enhance the SSA of char and attach oxygenated functional groups to its surface. This type of modification results in a positive charge created on the surface of the char which in turn helps in the adsorption of negatively charged pollutants. For example, surface-modified human hair-derived BC, resulting in a high porosity with an SSA of 3015 m²/g, exhibited enhanced adsorption performance for various organic pollutants [89].

Impregnation of metals or metal oxides (such as ZnO, Fe, CuO, and Fe₃O₄) into the carbon structure can also improve its adsorption characteristics (Table 4). Furthermore, BC and HC in this form can be used as catalysts in advanced processes [58, 59, 90]. Metals or metal oxides (especially Fe) impregnated chars are used in heterogeneous Fenton/Fenton-like processes for water treatment [59, 65, 90, 94]. Metal oxide/char composites have found their application in photocatalytic processes [58], as well as in persulfate oxidation processes [43, 63]. Magnetic particles can be enriched by chars in order to effectively utilize them as adsorbents [91-93]. After treatment, they can be easily separated from water using an external magnetic field.

Table 2. Characterization of BC produced from various feedstocks (modified from [54, 55]).

Feedstock ^a	Proximate analysis (%)				H/C	O/C	BET (m ² /g)	Pore diameter (nm)	Pore volume (cm ³ /g)	Ref.
	MO	VM	Ash	FC						
Animal waste										
Pig manure at 300°C	/	/	/	/	0.650	0.483	5.57	79.4	0.016	[53]
Pig manure at 600°C	/	/	/	/	0.521	0.426	9.15	48.5	0.021	
Pig manure at 900°C	/	/	/	/	0.380	0.186	16.7	25.7	0.035	
Agricultural waste										
Corn straws	6.17	71.2	6.52	16.1	0.130	0.99	/	/	/	[56]
Cellulose-enriched mesocarp of tangerine peels at 700°C	/	/	/	/	/	/	611	/	0.570	[52]
Cellulose-enriched mesocarp of tangerine peels at 800°C	/	/	/	/	/	/	855	/	0.710	
Cellulose-enriched mesocarp of tangerine peels at 900°C	/	/	/	/	/	/	922	/	0.680	
Tangerine peels at 900°C	/	/	/	/	/	/	246	/	0.130	
Bagasse powder at 800°C	/	/	/	/	0.036	0.345	646	2.92	0.300	[57]
Bamboo stakes	/	/	/	/	/	/	272	1.65	0.112	[58]
Walnut shells	/	/	/	/	/	/	113	5.62	0.150	[59]
Rubber seed at 500°C	4.95	24.9	20.2	54.8	0.524	0.329	5.00	21.5	0.026	[60]
Groundnut	4.08	35.3	15.6	45.0	1.350	/	0.59	/	/	[61]
Wood waste										
Coconut charcoal	/	64.2	2.90	17.9	0.120	/	612	/	0.670	[62]
Pine wood at 350-600°C	/	28.0	65.7	4.60	0.350	/	40.6	/	0.730	
90% pine; 10% fir wood	/	28.1	61.6	3.70	0.180	/	155	/	0.540	
Aged oak and hickory wood	/	74.1	25.4	nd	0.510	/	5.41	/	0.480	
Pinewood pellets	/	61.8	4.60	33.2	0.630	/	0.38	/	0.560	
Cypress sawdust	/	/	/	/	/	/	1570	2.35	0.920	[63]
Poplar sawdust at 900°C	/	/	/	/	/	/	405	1.91	0.194	[64]
Pine sawdust at 900°C	/	/	/	/	/	/	421	1.93	0.224	
Beech/oak sawdust	15.2	78.3	4.74	1.75	/	/	230	4.60	0.039	[8]
Urban waste										
Food waste	/	/	/	/	/	/	68.5	10.1	0.132	[65]

MO – moisture content, VM – volatile matter, FC – fixed carbon, / - not determined
^a temperature of pyrolytic conversion is given if reported in the cited literature source.

Table 3. Characteristics of HC derived from different feedstocks (modified from [14, 66]).

Feedstock ^a	Proximate analysis (%)			H/C	O/C	BET (m ² /g)	Pore diameter (nm)	Pore volume (cm ³ /g)	Ref.
	VM	Ash	FC						
Animal waste									
Poultry litter at 150-300°C	47.3-72.9	16.4-26.7	10.7-25.9	/	/	/	/	/	[67]
Dairy manure at 200-280°C	45.6-59.9	34.4-40.4	5.7-14.0	1.14-1.26	0.19-0.36	/	/	/	[68]
Agricultural waste									
Algae at 180-240°C	78.3-85.4	4.1-6.5	10.4-16.2	1.54-1.73	0.27-0.38	/	/	/	[69]
Corn stover at 180-260°C	47.2-78.8	2.38-3.36	18.6-49.7	0.76-1.33	0.19-0.68	/	/	/	[70]
Coconut husk at 140-200°C	53.5-59.8	0.1-0.6	39.6-46.2	/	/	/	/	/	[71]
Rice husk at 140-200°C	48.3-56.2	19.9-21.6	23.9-30.1	/	/	/	/	/	
Corn stover at 215-384°C	40.0-60.7	18.0-29.1	19.5-31.3	0.68-0.92	0.17-0.44	/	/	/	[72]
Rice husk	52.5	9.4	35.0	0.08	0.51	5.02	20.2	0.025	[73]
Rice husk at 180-250°C	42-63	23-32	13-26	0.08-0.13	0.81-0.36	/	/	/	[74]
Wheat straw at 180-260°C	60.1-84.6	1.6-2.6	9.1-36.0	0.09-0.14	0.35-0.90	/	/	/	[75]
Wheat straw at 160-240°C	62.5-80.6	2.2-5.3	16.7-34.2	0.09-0.12	0.49-0.91	/	/	/	[76]
Soybean straw	/	14.4-16.5	/	1.2	0.33	/	/	/	[77]
Walnut shell	55.5	0	42.4	0.10	/	642.6	1.08	0.35	[11]
Tea stalk	55.5	8.4	38.5	0.10	/	666.7	1.08	0.36	
Olive pomace	65.3	5.5	27.7	0.11	/	473.8	1.05	0.25	
Apricot seed	69.3	9.3	19.6	0.12	/	583.2	1.16	0.34	
Hazelnut husk	49.3	11.8	35.8	0.09	/	631.3	1.25	0.39	
Wood waste									
Waste wood	54.2-72.2	7.12-9.45	20.3-36.3	0.10-0.14	0.46-0.88	/	/	/	[78]
Wood sawdust	72.5	6.7	18.3	0.09	/	308.9	4.74	0.73	[11]
Wood meal at 180-260°C	57.6-83.6	<BDL	13.7-40.6	0.07-0.11	0.41-0.93	/	/	/	[79]
Eucalyptus wood at 180-240°C	47.9-79.1	0.10-0.20	20.8-51.9	/	/	/	/	/	[80]
Giant bamboo at 180-240°C	44.6-73.9	0.7-1.5	25.4-53.9	/	/	/	/	/	
Coffee wood at 180-240°C	53.1-77.6	0.8-1.1	21.3-46.0	/	/	/	/	/	
Pinewood at 180-240°C	65.3-83.8	1.69-1.88	14.5-33.0	0.09-0.13	0.40-0.82	11.8	/	/	[81]
Poplar wood at 180-240°C	57.0-76.2	7.51-11.07	16.3-31.9	0.09-0.13	0.49-0.86	7.12	/	/	[81]
Urban waste									
Waste rubber	61.2	6.6	32.2	0.1	0.01	/	/	/	[78]
Waste paper	47.2-70.9	13.2-31.2	15.9-21.6	0.07-0.15	0.67-1.33	/	/	/	
Waste food	45-59	4.2-4.6	36.4-50.3	0.1	0.3-0.4	/	/	/	
Organic fraction of municipal solid waste at 120-280°C	63.9-81.2	2.9-8.6	14.8-31.8	0.095-0.13	0.68-0.15	/	/	/	[82]
Waste textile at 230-280°C	73-77.2	0.44-0.64	22.3-26.4	0.07-0.09	0.57-0.70	/	/	/	[83]
Municipal solid waste pulp	40.2-65.7	24.1-46.1	11.1-20.3	0.09-0.13	0.36-0.97	/	/	/	[84]
Industrial waste									
Sewage sludge at 150-550°C	/	67.0-80.4	/	0.06-0.17	0.17-1.0	/	/	/	[85]
Sewage sludge at 190-250°C	52.4-55.2	44.5-47.5	0.1-0.3	1.65-2.28	0.17-0.57	8.8-31.0	/	/	[86]
Digested sewage sludge at 180-240°C	44.0-50.9	42.9-48.1	6.2-7.9	0.13-0.14	0.31-0.54	/	/	/	[87]
Sewage sludge (microwaved-assisted)	20.6	76.9	2.52	0.81	0.54	24.0	22.6	0.189	[19]
Organic Sludge at 180-240°C	34.1-44.3	48.6-57.9	7.2-9.3	1.58-1.76	0.33-0.45	26.5-35.4	32.8-39.2	0.248-0.303	[88]

MO – moisture content, VM – volatile matter, FC – fixed carbon, BDL – Below detection limit
^a temperature of hydrothermal conversion is given if reported in the cited literature source.

Table 4. Characteristics of different BC/HC-based composites.

Composites	Proximate analysis (%)				H/C	O/C	BET (m ² /g)	Pore diameter (nm)	Pore volume (cm ³ /g)	Ref.
	MO	VM	Ash	FC						
BC-based composites										
Modified BC derived from human hair	/	/	/	/	/	/	3015	2.00	1.51	[89]
Bagasse powder BC at 800°C	/	/	/	/	0.036	0.345	645.9	2.92	0.30	[57]
Post-deashed BC from Bagasse powder with HCl at 800°C	/	/	/	/	0.028	0.215	511.7	3.28	0.17	
Pre-deashing BC derived from Bagasse powder with HCl at 800 C	/	/	/	/	0.027	0.193	519.3	2.84	0.28	
Fe/BC composite (BC derived from waste coffee grounds at 500°C)	/	/	23.6	/	0.430	0.069	11.5	12.5	0.011	[94]
Fe/BC composite (at 600°C)	/	/	19.5	/	0.264	0.044	383.2	5.00	0.168	
Fe/BC composite (at 700°C)	/	/	18.0	/	0.175	0.040	431.7	4.70	0.186	
BC derived from Bamboo stakes	/	/	/	/	/	/	272.3	1.65	0.112	[58]
ZnO/BC derived from Bamboo stakes	/	/	/	/	/	/	316.1	3.87	0.306	
BC derived from Walnut shells	/	/	/	/	/	/	113.2	5.62	0.150	[59]
Fe/Cu oxide modified BC derived from Walnut shells	/	/	/	/	/	/	312.3	4.83	0.340	
Fe ₃ O ₄ /BC composite (BC derived from coconut shell at 500°C)	/	/	/	/	0.034	/	239.9	2.88	0.173	[90]
HC-based composites										
Sewage sludge at 190-250°C	52.4–55.2	44.5–47.5	0.1–0.3	1.65–2.28	0.17–0.57	8.8–31.0	/	/	/	[86]
KOH-modified HC derived from Sewage sludge at 190-250°	45.3–51.1	48.8–52.1	0.1–2.6	1.57–1.67	0.2–0.4	0.29–13.4	/	/	/	
Sewage sludge (microwaved-assisted)	20.6	76.9	2.52	0.81	0.54	24.0	22.6	0.189		[19]
CaO-modified HC from sewage sludge (microwaved-assisted)	20.2	78.3	1.12	0.69	0.54	21.8	10.1	0.076		
H ₃ PO ₄ -modified HC from sewage sludge (microwaved-assisted)	16.8	82.0	1.48	<0.32	0.42	11.4	19.8	0.076		

MO – moisture content, VM - volatile matter, FC - fixed carbon

ADSORPTION MECHANISM OF CECs REMOVAL USING BC/HC-BASED MATERIALS

The heterogeneous structure of BC and HC allows them to remove organic pollutants from water through various adsorption mechanisms, including physical adsorption, precipitation and complexation, and pore filling [95]. The adsorption efficiency is influenced by several factors, such as the pH, ionic strength, and polarity of the organic pollutant.

Electrostatic interactions primarily occur between ionisable organic compounds and the positively charged surface of BC. The efficiency of adsorption using electrostatic interactions is influenced by the pH and ionic strength of the water matrices [96].

Adsorption of organic contaminants onto char using the *pore filling* mechanism depends on several factors, including the total volume of micropores and mesopores of the char, the type and nature of the char, as well as the polarity of the organic pollutant. High efficiency of the process is achieved when adsorbents with a low content of volatile matter are applied and used to treat low-polluted samples [97].

The adsorption of organic contaminants by the *partitioning mechanism* takes place by diffusion of adsorbate into the pores of the non-carbonized part of the char and interaction with it. This mechanism is more efficient using char with a high content of volatile substances and in the cases treating effluents with a high concentration of organic pollutants [98].

Adsorption of aromatic compounds usually takes place via the *electron donor/acceptor interaction* mechanism on chars that have a structure similar to graphene, which is affected by the char production temperature. In the case of char synthesized at temperatures <500 °C,

the π aromatic char system behaves like an electron acceptor, while if it was prepared at a temperature >500 °C, the char behaves like a donor [99, 100]. The presence of functional groups (e.g. hydroxyl, carbonyl, carboxyl, and amine) on the surface of chars increases the adsorption affinity of organic contaminants onto char.

The adsorption mechanism by hydrophobic interactions is characteristic of the hydrophobic and neutral organic compounds due to their highly hydrophobic nature. Chars that have been prepared at high temperatures show a greater affinity for pollutant binding through hydrophobic interactions. At high synthesis temperatures, there is a loss of functional groups containing oxygen and hydrogen, which makes char more aromatic and hydrophobic, and therefore more suitable for removing hydrophobic organic pollutants [101].

In general, the adsorption of pollutants from water matrices by BC/HC depends on the characteristics of both pollutants and adsorbents and process conditions, involving various mechanisms. Pore filling, hydrogen bonding, and π - π interactions with the surface of the char are the possible mechanisms for antibiotics removal [115, 116]. The adsorption of endocrine-disrupting chemicals, in addition to the mechanism of π - π interactions, hydrophobic interactions and hydrogen bonding, also includes electrostatic interactions [124].

Factors affecting the efficiency of organic pollutants removal

The efficiency of removing pollutants from water using BC/HC as adsorbents depends on a lot of factors. In addition to the properties of the chars and the nature of the organic pollutants, the operating reaction conditions of the process have a great influence on the efficiency,

such as adsorbent content, pH, temperature, presence of other constituents of water (e.g. ions) [9, 102-104].

The content of adsorbent, as well as the initial content of contaminants, affects the adsorption efficiency. The degree of pollutants removal from water increases with increasing adsorbent content, until the optimal value of the adsorbent dosage is reached. By increasing the content beyond the optimal value no further improvements in pollutant removal can be achieved [102]. This tendency could be justified by the optimal utilization of active adsorption sites [105]. The efficiency of the adsorption process decreases as the initial pollutant content increases [103] and as the activation centres on the surface of the adsorbent become occupied by the pollutant molecules [106]. The removal efficiency of organic contaminants from water by adsorption is significantly affected by the initial pH of the solution due to certain charge characteristics of the adsorbent surface determined by the protonation and deprotonating of oxygen functional groups [107]. Furthermore, the pH of the solution also affects the ionization level of the pollutants [108]. At a pH below the PZC, the surface of the adsorbent carries a net positive charge, and therefore, it attracts and adsorbs negatively charged anions. Conversely, when the pH is above the PZC it is more favourable to adsorb positively charged ions [109]. This phenomenon is due to the interaction between the charged functional groups present on the surface of the adsorbent and the charged groups of the pollutants in the solution.

The effect of temperature on the adsorption capacity of char can vary depending on the specific pollutant and the characteristics of the char. Increasing the temperature can enhance the adsorption capacity of BC for certain organic pollutants, such as p-nitrophenol and diclofenac, through an endothermic process [104, 110]. This is because an increase in temperature can lead to an increase in the kinetic energy of the molecules, allowing for a more efficient adsorption process. However, for other pollutants and types of char, increasing the temperature may have an exothermic effect, which can reduce the adsorption capacity [111, 112].

Examples of CECs removal from water using BC/HC-based materials as adsorbents

BC and HC have a high affinity for the adsorption of organic pollutants. Tables 5 and 6 show summarized literature data on the effectiveness of BC, HC and their composites in removing organic pollutants, such as antibiotics, pesticides, etc. from water matrices.

Antibiotics have an important function in medication; however, their presence in the environment is undesirable due to their negative impacts on health and the environment. Antibiotics (such as tetracycline, sulfamethoxazole, cefotaxime, sulfadiazine, etc.) can be effectively removed from water matrices using BC and HC as adsorbents [113-116]. In order to improve their efficiency as adsorbents, it is necessary to perform their activation using acids or alkaline [113].

Utilizing KOH-activated BC, high adsorption capacities of tetracycline, oxytetracycline and chlortetracycline have been achieved amounting to 476.19 mg/g, 407.5 mg/g and 555.6 mg/g, respectively [113]. It is known that the adsorption capacities of BC increase at higher temperatures of the carbonization process [117]. On the other hand, mesoporous HC derived from potatoes through the soft-template method showed a maximum adsorption capacity of tetracycline of 238.7 mg/g [115]. The authors attributed this success to adsorption mechanisms: pore filling, hydrogen bonding and n- π interaction.

NaOH-activated HC has been reported to effectively remove sulfamethoxazole. The authors emphasized that the pore filling, charge-assisted strong hydrogen bonding, and π - π interactions with the surface of HC are the possible mechanisms [116]. Moreover, using NaOH-modified BC derived from *Anthriscus sylvestris* pharmaceuticals such as diclofenac and cephalexin could also be effectively removed (adsorption capacities were 392.9 mg/g and 724.5 mg/g, respectively) [118].

The removal of pesticides (e.g. atrazine, simazine, etc.) from water by using char-based sorbents was also investigated [119-121]. Pyrochar has shown a high adsorption capacity for atrazine and simazine with values of 1158 mg/kg and 243 mg/kg, respectively [119]. KOH-activated HC derived from sewage sludge showed up to 64% removal of terbuthylazine from water [120]. Utilization of unmodified HCs derived from different feedstocks as adsorbents for removing 2,4-dichlorophenoxy acetic was reported, having adsorption capacities up to 88, 90 and 89 mg/g, respectively [121].

Endocrine disrupting chemicals, EDCs (such as bisphenol A) are contaminants that can impact the endocrine system and subsequently impair the development and fertility of animals and humans. These components can be effectively removed from water using HC as an adsorbent [122, 123]. Adsorption of EDCs by HC depends on the characteristics of both pollutants and HCs, and process conditions, involving various mechanisms, such as electrostatic interactions, π - π interactions, hydrophobic interactions and hydrogen bonding [124].

Table 5. BC-based adsorbents applied for the removal of organic pollutants from wastewater.

BC-based adsorbent	Pollutant	Removal efficiency (%) or adsorption capacity (mg/g)	Ref.
Pharmaceuticals			
BC derived from oil palm fiber	Acetaminophen	7.30 mg/g	[125]
	Cephalexin	7.90 mg/g	
	valsartan	23.8 mg/g	
Cobalt-gadolinium modified BC	Ciprofloxacin	99.5% (6 h)	[42]
	Tetracycline	99.2% (6 h)	
Sulfamic acid modified BC	Tetracycline	412.9 mg/g	[126]
H ₂ SO ₄ or KOH activated BC derived from rice husk	Tetracycline	93.1% (180 min)	[127]
H ₃ PO ₄ modified BC derived from rice straw and swine manure	Tetracycline	167.5 mg/g	[128]
BC derived from cellulose-enriched mesocarp of tangerine peels at 900°C	Tetracycline	80% (10 min)	[52]
BC derived from bagasse powder at 800°C	Tetracycline	57.59 - 64.31 mg/g	[57]
Iron oxide/BC composite	Ofloxacin	96% (5 h)	[102]
H ₃ PO ₄ modified BC derived from bamboo	Sulfamethoxazole	88.1 mg/g	[129]
	Sulfathiazole	237 mg/g	
	Sulfamethazine	65.7 mg/g	
	Sulfamethoxazole	99.5 mg/g	[130]
	Sulfamethoxazole	49.8% (30 min)	[131]
Activated BC derived from cotton shell	Sulfadiazine	≈95% (1400 min)	[132]
NaOH modified BC derived from <i>Anthriscus sylvestris</i>	Diclofenac	392.9 mg/g	[119]
	Cephalexin	724.5 mg/g	
Agrochemicals			
P-doped BC	Triazine	>96% (20 min)	[133]
Sawdust-coal BC	Epoxiconazole	97% (400 min)	[134]
Grape pomace derived BC	Cymoxanil pesticide	161 mg/g	[135]
KOH activated magnetic BC from loofah sponge	Imidacloprid	738 mg/g	[92]
Reduced graphene oxide/BC composite derived from corn straw	Atrazine	67.5 mg/g	[137]
Phenols			
BC derived from waste wood biomass	Phenol	50% (120 min)	[8]
Activated BC derived from corn cob	Phenol	66.1%	[137]
Sewage sludge derived BC	2,4-Dichlorophenol	100%	[138]
CO ₂ activated BC	Phenol	50–60% (60 min)	[139]
	Chlorophenol		
BC derived from <i>S. boveanum</i> macroalgae	2,4-Dimethylphenol	17.0 mg/g	[140]
Endocrine-disrupting compounds			
Magnetic CuZnFe ₂ O ₄ /BC composite	Bisphenol A	101.5 mg/g	[130]
BC derived from human hair at 700-900°C	Bisphenol A	1162-1229 mg/g 51-65% (30 min)	[89]
Graphene oxide/magnetic BC composite	17 β -estradiol	46.2 mg/g	[141]
Iron/graphite BC composite	17 β -estradiol	100% (45 min)	[43]

Table 6. HC-based adsorbents applied for the removal of organic pollutants from wastewater.

HC-based adsorbent	Pollutant	Removal efficiency (%) or adsorption capacity (mg/g)	Ref.
Pharmaceuticals			
Calcium alginate/activated HC	Acetaminophen	batch test 165.9 mg/g	[142]
		fixed-bed 127.0 mg/g	
KOH activated HC derived from brewer's spent grain	Acetaminophen	318.0 mg/g	[143]
Acid functionalised HC derived from loquat cores waste	Diclofenac	96%	[144]
	Antipyrine	76%	
	Prednisolone	80%	
Modified HC derived from rice husk	Phenanthrene	41.0 mg/g	[91]
Magnetic HC derived from sludge	Tetracycline	145.0 mg/g	[93]
	Ciprofloxacin	74.2 mg/g	
Batatas derived HC	Tetracycline	238.7 mg/g	[115]
Iron-loaded HC derived from sludge	Tetracycline	104.8 mg/g	[145]
	Doxycycline	129.0 mg/g	
Calcined spherical HC	Ibuprofen	95.6 mg/g	[146]
HC derived from olive oil wastes	Triclosan	98%	[147]
	Ibuprofen	43%	
	Diclofenac	64%	
Agrochemicals			
Magnetic HC derived from pomegranate waste	2,4-dichlorophenoxyacetic acid	101.1 mg/g	[148]
KOH/melamine coactivated HC derived from corn straw	atrazine	216.5 mg/g	[149]
Phenols			
Biowaste derived HC	Guaiacol	100%	[150]
	Phenol	100%	
	Resorcinol	100%	
	Vanillyl alcohol	61%	
Endocrine-disrupting compounds			
N-doped HC derived from <i>Ulva prolifera</i>	Bisphenol A	33.3 mg/g	[151]

Conclusion

BC and HC are carbon materials produced from biomass either via pyrolysis or hydrothermal processes, representing eco-friendly and cost-effective adsorbents for the removal of emerging contaminants from different water matrices. By choosing the raw material, as well as by optimizing the reaction conditions of the carbonization process (pyrolysis, HTC, etc.), it is possible to influence the obtaining of HC/BC with certain characteristics suitable for removing target pollutants from water. The removal mechanism (e.g. electrostatic interaction, pore filling, partitioning, electron donor/acceptor interaction, hydrophobic interactions) that will take place depends on the characteristics of the adsorbent itself, as well as the type of pollutant. If biowaste is used for BC and HC production, it has an additional value in reducing the environmental problems linked to waste disposal. BC and HC could be also considered as a way of removing carbon from the geochemical carbon cycle, particularly if the spent char-based adsorbent could be incorporated into other materials. Further research is needed in order to fully understand the particularities of each adsorption process. Factors, such as the type of adsorbent used, activation methods, target contaminants, and process conditions can lead to different efficiencies. Additionally, in real-life scenarios, the presence of multiple emerging contaminants may have a synergistic effect on the adsorption process. Therefore, it is also important to conduct comprehensive research in this direction.

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Izvod

BIOČAĐ I HIDROČAĐ KAO ADSORBENTI ZA UKLANJANJE EMERGENTNIH ZAGAĐUJUĆIH MATERIJALA IZ OTPADNIH VODA

Aleksandra Adamović¹, Mirjana Petronijević², Sanja Panić², Dragan Cvetković¹, Igor Antić², Zoran Petrović³, Nataša Đurišić-Mladenović²

¹Univerzitet u Nišu, Tehnološki fakultet, Leskovac, Srbija

²Univerzitet u Novom Sadu, Tehnološki fakultet Novi Sad, Novi Sad, Srbija

³Univerzitet u Istočnom Sarajevu, Tehnološki fakultet, Zvornik, Republika Srpska, Bosna i Hercegovina

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Poslednjih decenija ugljenični materijali, kao što su biočađ (BC) i hidročađ (HC), se intenzivno koriste kao isplativi i ekološki prihvatljivi adsorbenti u tretmanu vode. Specifične fizičko-hemijske karakteristike uglja omogućavaju efikasno uklanjanje širokog spektra zagađujućih materija iz vode, uključujući emergentne kontaminante (npr. farmaceutski aktivna jedinjenja, agrohemijske i jedinjenja koja ometaju rad endokrinog sistema). BC i HC se mogu sintetisati iz različitih biljnih materijala, čak i iz otpadne biomase, što ih čini jeftinim materijalima. U stvari, primena otpadne biomase za proizvodnju ugljeva može se označiti kao obećavajući put ka postizanju ciljeva održivog razvoja u vezi sa upravljanjem čvrstim otpadom. Izbor sirovine i optimizacija reakcionih uslova procesa karbonizacije (piroliza, hidrotermalna karbonizacija, itd.) utiču na performanse materijala na bazi ugljenika koji dovode do proizvodnje sa željenim karakteristikama pogodnim za uklanjanje određene grupe jedinjenja. Cilj ovog preglednog rada je da pruži pregled trenutnog statusa istraživanja u vezi sa primenom BC i HC kao adsorbentata u uklanjanju emergentnih polutanata (CEC) iz otpadnih voda. Biće obrađena i fizičko-hemijska svojstva supstrata, vrste sinteze, tehnike karakterizacije, kao i mehanizam i uticaj parametara procesa u uklanjanju CEC-ova iz otpadnih voda.

Ključne reči: biočađ, hidročađ, adsorpcija, emergentni polutanti, tretman vode