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Biochar and Hydrochar in the Development and Application of Electrochemical Devices in the Sensing and Degradation of Target Compounds: A Mini-Review of the Recent Contributions of 2020-2023

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In this review, we present the versatility of biomass and its derivatives obtained through thermal conversion processes (biochar and hydrochar) for applications in the development of electrochemical devices. This work highlights the generation of carbonaceous materials through pyrolysis and hydrothermal carbonization, in addition to proposing strategies to modify their physicochemical properties through activation methodologies to enhance their application as electrode materials. The processes of monitoring and degrading chemical species are discussed based on the diversity of electrodes that can be obtained with these bioderived coals, according to recent contributions available in the literature (2020-2023), which show the wide applicability of these materials in the field of electrochemistry.

Keywords: biomass, pyrolysis, hydrothermal, electrodegradation, sensors

1. Introduction

In recent years, concerns about the environmental impact of waste disposal from agroindustry and everyday processes have been responsible for turning the scientific community's attention to the use of biomass and its derivatives. All organic matter that comes from animals or plants can be classified as biomass (Figure 1), and due to the diversity of obtaining sources, it has aroused interest for its application in the most varied sectors such as the generation of biofuels, biomaterials, agriculture, electrochemical devices and production of adsorbents, for example.[1-4]

Biomass derived from plants is mainly composed of hemicellulose, cellulose, and lignin, and their structure is composed of phenolic compounds, flavonoids, sugars, and alcohols, among others, which is one of the factors that justify the wide applicability of these materials. The conversion of residual biomass into value-added products is not only related to economic aspects that are accentuated by the reintroduction of these products in commercial circuits, but also due to environmental impacts, such as the reduction in the formation of by-products of biomass degradation, stable carbon (recalcitrant) generation, and useful materials for diverse applications.[5-7]

The thermal conversion of biomass, either hydrothermal or pyrolytic, has been an alternative for the recovery of these materials, as several compounds can be extracted through these processes. The gaseous portion obtained through pyrolysis, commonly known as biogas, is used mainly as an alternative for generating energy and chemical compounds of industrial interest. The liquid fraction, known as bio-oil, is a potential renewable fuel. Biochar (solid phase) is of great environmental importance because the structure formed can maintain the carbon that would be available in the atmosphere in the form of gases that cause the greenhouse effect in the soil for several years. In addition, the presence of functional groups and pores in

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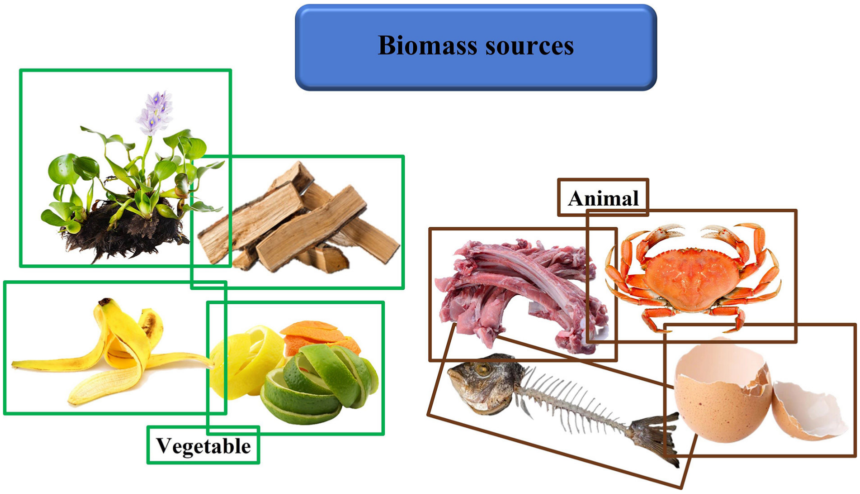

Figure 1. Representations of different biomass sources.

the structure of biochar is guaranteed by the degradation of lignocellulosic matter, which makes the material formed of great application in processes such as retention of water and nutrients in the soil and adsorption of contaminants.[5,8,9]

Analogously, the hydrothermal carbonization process is responsible for the formation of liquid, solid (hydrochar), and gaseous fractions. The phases obtained, as well as in pyrolysis, are mainly applied as fuel and in the formation of value-added chemical compounds, in the monitoring and remediation of soils and aquatic environments, in agriculture, and the composition of electrochemical devices. Despite the similarity in some of their applications, biochar (BC) and hydrochar (HC) have different physicochemical properties, such as chemical and structural compositions, owing to the different conditions used to obtain these materials.[10-12]

The biomass used as a precursor in thermal conversion processes directly influences the composition and physicochemical characteristics of the products obtained. Despite being mainly composed of cellulose (35-50%), hemicellulose (15-30%) and lignin (10-25%), these structures will have their percentage content varied from biomass to biomass and according to the stages of cultivation, resulting in structurally diverse materials being obtained through the conversion of different biomasses.[13] It is customary to select the thermal conversion method to be used based on the analyst's interests and biomass characteristics such as moisture content, where wetter biomasses (> 30%) tend to be submitted to hydrothermal treatment, aiming at eliminating the step of biomass drying and reducing the energy cost of the process.[14] Comparatively, biomasses face different conditions when subjected to pyrolysis (inert atmosphere and ambient pressure) or hydrothermal carbonization (presence of water and high pressures), which will result in different materials even when a common biomass is used, since the reactions through which it will pass will occur under different conditions.[15] Thus, these materials tend to present different functionalization, size, quantity and volume of pores, and effective surface area.

The literature presents review articles[16-20] on the application of biochar or hydrochar as electrode modifiers. The present work aims to summarize the recent advances (2020-2023) in obtaining electrodes using these materials and their applications in the processes of monitoring and degradation of organic and inorganic contaminants.

1.1. Hydrochar and biochar

Hydrothermal carbonization (Figure 2) is one of the thermal conversion processes of organic compounds and is responsible for the formation of structured materials derived from biomass subjected to temperature (100-250 °C) and self-generated pressure (2-22 MPa) conditions in the presence of aqueous solutions. Compared to the conventional pyrolysis method, hydrothermal carbonization has some advantages, such as reduced energy consumption owing to the absence of the biomass drying process and the use of milder temperatures for material formation.[19,21,22]

The properties of charcoal derived from this method depend on the precursor biomass, temperature, time, and pressure of the system used. The interaction of biomass with the solvent under these conditions results in hydrolysis, decarboxylation, dehydration, polymerization, aromatization, and carbonization reactions, which form the structure and functional groups of the products obtained. Among the characteristics of hydrochar, in some cases, the increase in the presence of acid functional groups, the presence of cavities and pores, and high surface area stand out, in addition to the considerable stability and

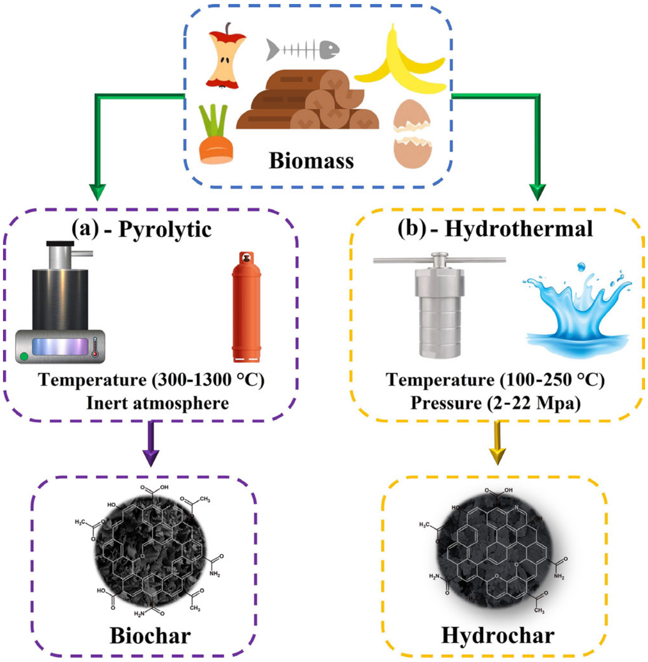

Figure 2. Schematic representation of different biomass thermal conversion processes to obtain biochar (a) and hydrochar (b).

degree of condensation of the structure, which is of great interest for construction of electrochemical devices, such as supercapacitors and sensors.[18,21,23]

The thermochemical degradation of biomass in a totally or partially inert environment, also known as pyrolysis (Figure 2), forms a porous solid material that is rich in carbon and functional groups on its surface, highlighting the high versatility of the material obtained for applications as adsorbents, agricultural remediators, electrochemical applications, and control of atmospheric contaminants. Biochar has physicochemical characteristics, such as adsorptive capacity, high specific surface area, microporosity, and ion exchange ability, which, as in hydrochar, are defined according to the temperature, chemical composition of the raw material, residence time, and behavior of the structures that make up the biomass to thermal degradation.[24-30]

During the pyrolysis of vegetal biomass, hemicellulose undergoes degradation in a temperature range between 220 and 380 °C, being the first structure to be decomposed. Then, the cellulose is degraded between 380 and 550 °C. Lignin, on the other hand, is highly thermally stable, which causes it to be slowly decomposed as the temperature increases and can be degraded up to about 950 °C.[13] In hydrothermal carbonization, the degradation of hemicellulose usually occurs from 180 °C, while cellulose and lignin start this process at 220 and 260 °C, respectively.[31] In this sense, the temperature to which the biomasses are subjected, and the duration of the thermal conversion process will directly impact the physicochemical properties of the products obtained, as they will affect the structures that will be degraded. The increase in residence time and temperature is responsible for privileging the formation of a charcoal with greater aromatic character and structural organization to the detriment of a lower functionalization of its surface.[14,28,30]

Based on the variability in the characteristics of the materials obtained by different processes, some studies have comparatively investigated the performance of hydrochar and biochar derived from the same biomass sources, in varied applications.[32-34] In addition, biomass treatment processes and/or the solid derivative obtained in thermal conversion are considered alternatives to enhance or guarantee the properties of interest in the formed material, contributing to the increase in the performance of the desired application.[24,35] Thus, literature has explored several strategies based on physical and/or chemical procedures to enable greater use of the properties of these materials.

1.2. Activation methods

The chemical and physical properties of bioderived charcoal can be optimized to obtain a more efficient product for the development of the intended processes. According to the literature,[20] some essential characteristics to enhance the performance are an increase in the size and number of pores, in addition to the incorporation of functional groups on the surface. Physical and chemical methods illustrated in Figure 3 can activate these materials.[16,35]

The most reported physical processes for optimizing the properties of bioderived materials use gas as an

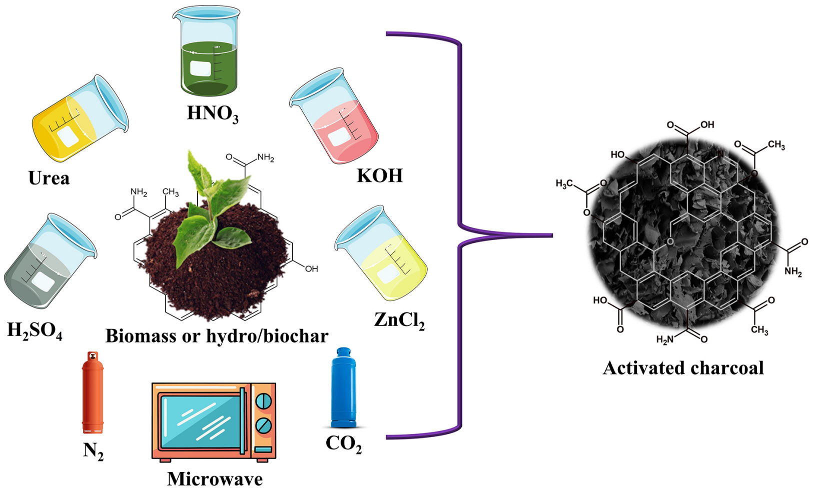

Figure 3. Representation of different possibilities for activation treatment of bioderived coals.

oxidizing agent with the material under heat treatment, which is commonly carried out under atmospheric air, water vapor, CO_2, or an inert gas such as N_2.[36,37] This method, also known as gas activation, aims to modify the structure and textural properties of biochar or hydrochar, mainly by opening the pores. In general, the energy expenditure for activation increases as the temperatures increase.[36] In addition, procedures involving treatment with ultrasound radiation to increase porosity, surface area, and microwave radiation, which minimize the energy used in biomass conversion, have also been applied to improve physicochemical properties and facilitate the acquisition of activated charcoal.[38-41]

Chemical modification can also be performed based on the application of the material. Thus, carbonaceous materials can be treated by exposing them to acidic solutions that promote the formation and/or increase in the number of pores, increase the surface area, and insert functional groups. Through the attack of the acid species (HNO_3, HCl or H_3PO_4), it is possible to reduce the size of the particles of the material, in addition to opening the pores caused by the elimination of the ashes present.[42-45] The use of other oxidizing agents, such as potassium permanganate, has also been reported, being responsible for generating an increase in pore volume, enhancing the adsorption capacity of dyes, for example.[46,47]

Activation with alkaline solutions (KOH or NaOH) has been reported to yield HC and BC with mesoporous surfaces, higher specific surface area and pore volume, high aromaticity, and a more ordered structure.[48-51] Nitrogen sources (urea or ammonia) can be used to increase the interaction with other species and improve the electrical conductivity of the materials. This occurs because of the introduction of new energy levels to the structure of the material, resulting from the presence of free electrons of nitrogen atoms.[52-55] In addition, chemical treatments are performed using salts such as zinc chloride ($ZnCl_2$), which increases the surface area,[56,57] and other metals, which guarantee magnetic properties to charcoals.[58-60] The most common obtaining routes for these are pyrolysis, co-precipitation and calcination methods.[61] Magnetic biochar has active sites containing metals such as zinc, manganese, and iron, that enhance its surface interaction capacity and the chemical adsorption of the material. Also promoting the increase of electrical conductivity, facilitation of material removal using magnets and its ionic exchange capacity, while releasing soluble ions producing metallic precipitates.[62]

The use of hydrogen peroxide (H_2O_2) in the treatment of biochar plays a key role in enhancing oxygenated groups, which consequently increases the ion exchange capacity of these materials and promotes greater interaction between biochar and metals in adsorptive processes.[63] The literature presents results demonstrating that the application of H_2O_2 causes an increase in the pores of activated biochars by more than twice the pore diameter compared to pristine biochar. This effect is directly related to the triggered oxidative process that allows the clearance of pores caused by the presence of ash.[64]

The versatility of these bioderived coals has enabled the formation of composite materials by mixing them with other materials such as nanostructures, molecular recognition sites, oxides, among others (Figure 4).[26,65,66] In this way, synergistic effects are achieved by increasing the detectability and selectivity to analytes or by improving the catalytic efficiency of electrode processes.[65]

Thus, chemical or physical activation processes are responsible for enabling the application of these materials with high performance in adsorption systems or as electrochemical devices.

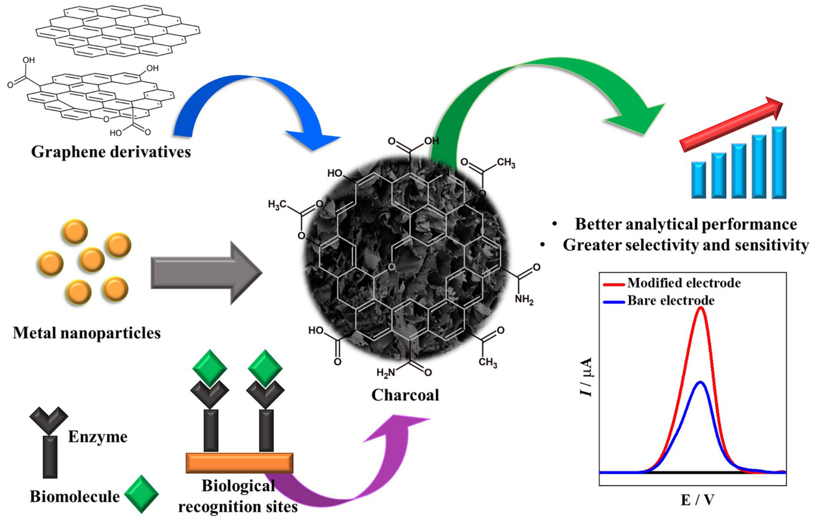

Figure 4. Schematic representation of the versatility of bioderived coals for composite formation and the effect on the properties of modified electrodes.

2. Electrochemical Devices

2.1. Electrochemical sensors

Electrodes act as electrochemical sensors and are of great interest for monitoring numerous species of interest. The versatility of the application of these devices is based mainly on the low cost associated with the analyses, which require cheaper equipment, and the use of a smaller amount of high-purity analytical reagents and solvents when compared to other sensing methods, such as chromatography and spectrometry. In addition to these advantages, it is possible to quickly obtain data with high sensitivity and selectivity, and figures of merit comparable to those obtained by more consolidated methods, thus arousing interest in the development and use of these devices.[26,67,68]

The electrodes have a variable composition and can be made of metals such as mercury, gold, silver, and platinum, carbon such as boron-doped diamond (BDD), glassy carbon (GCE), and carbon paste (CPE). In addition, it can be deposited on various substrates such as polyethylene, polypropylene, and cellulose. The structure of the electrodes is mainly related to the working potential window, the analytes to be evaluated, the integrity of the electrode material in the analysis medium and the selectivity, which allows them to be applied for the most diverse purposes.[69]

Modified electrodes can be obtained by immobilizing one or more species of interest on a base electrode that acts as a substrate, or by modifying the entire constitution. In this context, several techniques can be used to obtain modified electrodes, which will depend mainly on the type of electrode and the nature of the modifying agent.[70] Chemical adsorption can occur by immersing the electrode in a solution containing the modifier or by drop casting (Figure 5a) onto the electrode surface.[71] Modifications by electrochemical deposition (Figure 5b) are based on the application of current to the electrode immersed in a medium containing the modifier, which will be deposited on the surface of the electrode.[72,73] On the other hand, the formation of composites occurs when a modifying species is incorporated into the electrode, altering its entire composition, such as maceration (Figure 5c), often used to modify electrodes based on carbon paste.[26,67]

The choice of modifying agents is linked to the application of these sensors and aims to introduce materials that will enhance or generate new properties such as sensitivity, selectivity, electrical conductivity, surface area, adsorptive and/or complexing sites.[20,69] In this context, several studies have presented the optimization of the performance of electrochemical sensors guaranteed by modification with different biochars and hydrochars. Owing to their high sorption capacity, high porosity, and functionalized surface, these materials have been of great interest for the detection of organic and inorganic compounds in various matrices, enabling the development of optimized electrochemical methodologies that increase the interaction between the analytes to be studied and the sensor produced.[74]

Sant' Anna *et al.*[26] developed an electrochemical sensor based on water hyacinth biochar obtained at 400 °C (B) and reduced graphene oxide (rGO) for the detection of carbendazim (CBZ) in orange juice, lettuce leaves, drinking water, and wastewater. The developed sensor showed a limit of detection (LOD) of 2.3 nmol L^{-1} and recovery values in the range of 77.7-122.0%. The incorporation of biochar onto CPE provided an increase in signal intensity of more than 7 times the value obtained for the unmodified electrode. The authors attributed such improvement to the

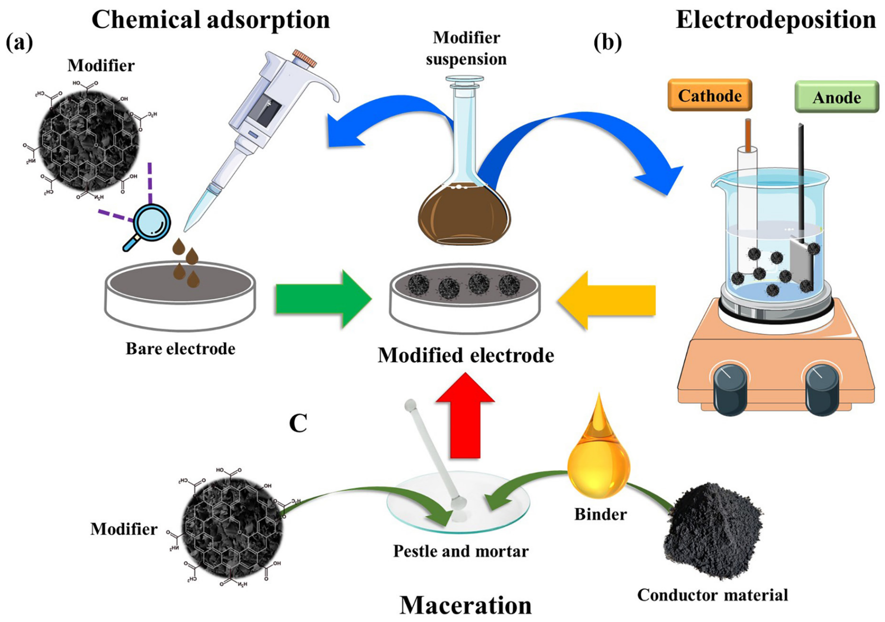

Figure 5. Schematic representation of the different construction process of the modified electrode.

interaction of the functional groups from biochar with CBZ and electrical conductivity of rGO which facilitated the charge transfer process. After activation of B with nitric acid solution (AB4), Sant' Anna *et al.*[75] obtained a composite electrode (rGO-AB4) and applied it to determine paraquat (PQ) in food samples with LOD of 0.02 μmol L^{-1} and recoveries between 87.7 and 103.8%. In addition, the electrode with AB4 showed an increase in the reduction peak of PQ by approximately 30% compared with that obtained with the precursor biochar (B), indicating that the activation process was essential for increasing the sensitivity of the electrode to the analyte. The reported increase was associated with synergistic interactions between rGO-AB4 and Paraquat guaranteed by the increase in the content of oxygenated and nitrogenated functional groups present in AB4.

Zhao and co-workers[76] prepared a composite with chitosan and charcoal derived from mung bean, from the hydrothermal treatment of biomass at 180 °C followed by the addition of KOH and water to form carbonaceous material. After the material was dried under vacuum, it was subjected to pyrolysis, and the resulting solid was treated with hydrochloric acid and subjected to a chitosan solution to obtain the modifier that was by drop casting deposited on a glassy carbon electrode. Carbendazim in apple and tomato juices were determined using the developed sensor, with recoveries between 98.8-103.2% and LOD of 20 nmol L^{-1}. Very similar to reported by Sant' Anna *et al.*,[26] in this case, the composite was responsible for the improvement of the obtained signal, which was associated with the composite of biochar and chitosan and its improved interaction with CBZ.

Sfragano *et al.*[77] reported the development of an electrochemical sensor based on woody biomass and sewage sludge. The biochar was produced by varying the temperature where the biomass was placed from room temperature to 850 °C. A portion of the biochar was washed with a commercial acid solution byproduct of the raw material gasification process (BioDea) and deionized water to obtain two modifiers: untreated (SSB-U) and treated (SSB-T). An increase in the surface area of SSB-T was observed and was related to the clearance of biochar pores when acid treated and washed. The modified carbon paste electrochemical sensors produced were used to detect (poly)phenolic compounds. The developed methodology showed different limits of detection for each electrode (SSB-U CPE and SSB-T CPE) and was evaluated for five analytes: hydroquinone (8 and 12 μmol L^{-1}), catechol (15 and 15 μmol L^{-1}), gallic acid (17 and 17 μmol L^{-1}), resorcinol (12 and 9 μmol L^{-1}) and vanillin (8 and 9 μmol L^{-1}). The resistance to charge transfer observed for SSB-U CPE (230 Ω) was lower than that of SSB-T CPE (367 Ω), which may be associated with the presence of inorganic species in the untreated material that act to facilitate electronic transfer. Both materials showed good LOD values for the evaluated species, highlighting the possibility of using biochar from urban sewage in the determination of these compounds.

Espro *et al.*[78] evaluated the application of hydrochar produced from orange peels via hydrothermal treatment at different temperatures (180, 210, 240, 270 and 300 °C). To obtain the desired electrode, hydrochar paste was printed on the surface of a screen-printed carbon electrode. The electrochemical sensor was developed for two different applications, one as a conductometric sensor for NO_2 detection with LOD of 50 ppb and for dopamine detection, which presented a LOD of 180 nmol L^{-1}. The electrode modified with HC produced at 300 ºC showed greater sensitivity for the determination of dopamine and NO_2. The increase in the temperature used in the process often generates an increase in the electrical conductivity of the material obtained, which, when applied to modify the working electrode, accelerates the transfer of electrons of the electrode. For both the applications, the material exhibited an efficient response.

A carbon paste electrode (CPE) was modified with biochar (700 °C) from babassu petiole and 1,2,3,4,8,9,10,11,15,16,17,18,22,23,24,25-hexadecafuoro-29*H*,31 phtalocyanine (CuHPc) for glyphosate determination. The CPE showed no electrochemical response to glyphosate since the pesticide molecule is not electroactive. In this sense, the signal was monitored from the variation in the CuHPc current intensity. When using BC in conjunction with CuHPc, a 100% increase in the electrochemical signal was observed by the modified sensor compared to the modified sensor with CuHPc alone. Such results are mainly linked to the high interaction between the functional groups of BC and CuHPc, which enhances the electrochemical response of the sensor to the analyte.[79]

Biochar from coffee husks (300 °C) was used to modify a carbon paste electrode that was applied for the simultaneous determination of Cd^{2+} and Pb^{2+}. Using spontaneous pre-concentration of the analytes, the CPE did not show appreciable oxidation signal, while the modified electrode obtained a 4- to 6-fold increase in the oxidation signal for Cd^{2+} and Pb^{2+}, respectively, when compared to the signal from the electrode modified with the precursor biomass. The thermal conversion process is responsible for improving characteristics such as electronic transfer and stability, in addition to causing structural changes like increased porosity and functionalization, which provides better adsorptive interactions with the analytes.[80]

A GCE was modified by drop casting using a hydrochar (180 °C) produced from acorn shells, which was treated with H_2SO_4 to obtain sulfonated HC microspheres and nanosheets (SCMN). The electrode modified with SCMN was used to determine glutathione, and showed greater sensitivity for the analyte, increasing the intensity and resolution of the analytical signal. The better performance of SCMN/GCE was attributed to the mesoporous nature of SCMN, in addition to the large surface area and high conductivity of the modifier material.[81]

Table 1 summarizes some recent publications on the use of bioderived carbons to modify electrodes and their application in the determination of various organic and inorganic compounds.

Bioderived charcoals are valuable components for electroanalytical applications. Compared to conventional electrode modifiers such as metallic oxides and nanoparticles, they have low cost, greater biocompatibility, high stability and are easy to obtain. The versatility of combinations, sources, and processes for obtaining these carbonaceous materials associated with the presence of pores and functional groups, increases the possibility of analyte sorption on the surface of the electrodes, which is of interest for analytical strategies aimed at processes involving pre-concentration. In this sense, the determination of metallic ions such as Hg^{2+}, Cd^{2+} and Pb^{2+} has been quite evident given the high toxicity of these species, with electrodes based on charcoal being excellent for this purpose, as they generally involve less energy expenditure in the process with the use of spontaneous accumulation and lower values of potential for species reduction. Furthermore, in the indirect determination of non-electroactive species such as the pesticide glyphosate and some biomolecules, which are often carried out using redox probes and metal ions, charcoals contribute with their high adsorption and electron donor/acceptor activity that catalyze the reaction processes. The use of biochar and hydrochar in the modification of electrodes for sensing target compounds, aims not only to replace toxic materials such as mercury or that have expensive obtaining routes (e.g., platinum, gold, BDD and nanomaterials), but also to contribute synergistically to the better analytical performance of traditional electrodes and modifiers. Modification of the electrodes by maceration is more advantageous than drop casting, which despite being more used, usually involves long and successive steps, making the process more expensive and time consuming. Furthermore, after maceration, the electrode is ready for use being it easier to produce, apply and commercialize.

Associated with the concern about the urgency of monitoring the levels of some chemical species in a given environment, there is a need to remove these compounds to maintain the quality of these places. Therefore, researchers have focused on the development of materials and methods that allow the degradation of these polluting substances.

2.2. Electrodegradation of compounds

From the search for viable alternative processes for the elimination of persistent polluting compounds in the environment, the study of processes that present greater efficiency in the degradation of these compounds stands out, including advanced oxidative processes (AOPs), which are responsible for converting contaminants into less

Table 1. Precursor biomass, analytes, limits of detection, and samples evaluated by electrochemical sensors reported in the literature between 2020 and 2023

Biomass	Charcoal	Target compound	Limit of detection	Modification	Accumulation parameters	Samples	Reference
Corncob	BC	dibutyl phthalate	2.6 nmol L^{-1}	drop casting	N.F.	rice wine	82
Water hyacinth	BC	carbendazim	2.3 nmol L^{-1}	maceration	OCP 600 s	orange juice, lettuce leaves, drinking water and wastewater	26
Water hyacinth	BC	paraquat	20 nmol L^{-1}	maceration	OCP 300 s	coconut water, honey, lettuce, lemon, and wastewater	75
Pineapple leaves	BC	carbendazim	0.30 nmol L^{-1} 0.24 nmol L^{-1}	drop casting	OCP 600 s	cabbage, peach, apple, and lake water	83
Coffee grounds waste	HC	methyl parathion	9.19 nmol L^{-1}	drop casting	N.F.	soil	84
Babassu petiole	BC	glyphosate	0.02 µmol L^{-1}	maceration	N.F.	river water and orange juice	79
Paper waste pulp	HC	clenbuterol	3.03 pmol L^{-1}	drop casting	N.F.	human urine	85
Physalis alkekengi L. husks	BC	ascorbic acid	0.92 µmol L^{-1}	drop casting	N.F.	vitamin c chewable tablets	86
Lotus stem	BC	hydroquinone catechol nitrite	0.15 µmol L^{-1} 0.11 µmol L^{-1} 0.09 µmol L^{-1}	drop casting	N.F.	tap water	87
Rice flour	BC	hydroquinone catechol	0.047 µmol L^{-1} 0.037 µmol L^{-1}	drop casting	N.F.	N.F.	88
Seedling of white myoga ginger	BC	hydroquinone catechol	0.002 µmol L^{-1} 0.004 µmol L^{-1}	drop casting	N.F.	tap water	89
Bamboo	BC	methylparaben	0.05 µmol L^{-1}	drop casting	N.F.	hand sanitizer, mouthwash, deodorant, emollient for cuticles and moisturizer cream	90
Soulangeana sepals	BC	acetaminophen	1.0 nmol L^{-1}	drop casting	OCP 210 s	human serum	91
Soybean nodules	BC	ascorbic acid dopamine	1.90 µmol L^{-1} 3.18 µmol L^{-1}	maceration	N.F.	medical injection	92
Corn stalk	BC	*E. coli* O157:H7	Log 4 CFU mL^{-1}	drop casting	OCP 1800 s	N.F.	93
Sugarcane bagasse	BC	SARS-CoV-2	10 ng mL^{-1}	drop casting	OCP 900 s	human serum	94
Lotus seed pods	HC	hyperine	5 nmol L^{-1}	drop casting	OCP 180 s	*Hypericum perforatum*	95
Pine tree	BC	*Shigella dysenteriae* genes	9.14 fmol L^{-1}	drop casting	0 V 500 s	human plasma	96
Bamboo powder	BC	hydroquinone catechol	0.4 µmol L^{-1} 0.2 µmol L^{-1}	drop casting	N.F.	lake water	97
Orange peel	HC	dopamine	180 nmol L^{-1}	print	N.F.	N.F.	78
Lotus root	HC	hesperetin	11 nmol L^{-1}	drop casting	OCP 150 s	urine and *Buddleja officinalis* macim	98
Acorn shells	HC	glutathione	24.91 pmol L^{-1}	drop casting	N.F.	human blood serum	81
Sugarcane	BC	bisphenol A	3.18 nmol L^{-1}	drop casting	N.F.	ground water	99
Cigarette butt	HC	trandolapril	10.9 nmol L^{-1}	drop casting	N.F.	human serum	100

Table 1. Precursor biomass, analytes, limits of detection, and samples evaluated by electrochemical sensors reported in the literature between 2020 and 2023 (cont.)

Biomass	Charcoal	Target compound	Limit of detection	Modification	Accumulation parameters	Samples	Reference
Castor cake	BC	caffeic acid	30.9 nmol L^{-1}	maceration	OCP 300 s	wine	101
Lotus stem	BC	glyphosate	76.9 nmol L^{-1}	drop casting	N.F.	apple juice and milk	102
Soulangeana sepal	BC	riboflavin	0.2 nmol L^{-1}	drop casting	OCP 210 s	milk	103
Babassu	BC	Cd^{2+} Pb^{2+}	110 nmol L^{-1} 56 nmol L^{-1}	drop casting	–0.9 V 120 s	river water	104
Magnolia grandiflora	BC	Hg^{2+} Pb^{2+}	> 4.98 pmol L^{-1} 4.83 pmol L^{-1}	drop casting	–0.7 V 210 s	lake water and paddy water	105
Coffee grounds	BC	Pb^{2+}	4.5 nmol L^{-1}	maceration	OCP 300 s	gunshot residue and hair dye	106
Peach wood	BC	Pb^{2+}	96.5 pmol L^{-}	drop casting	–1.2 V 300 s	tap water	107
Litsea cubeba shell	BC	Pb^{2+}	4.83 pmol L^{-1}	drop casting	–0.7 V 270 s	paddy water	71
Cinnamomum camphoras branches	BC	Cd^{2+} Pb^{2+} Hg^{2+}	0.036 nmol L^{-1} 0.003 nmol L^{-1} 0.011 nmol L^{-1}	maceration	–1.3 V 420 s	tap water and lake water	108
Litsea cubeba shell	BC	Hg^{2+}	0.15 nmol L^{-1}	drop casting	OCP 90 s	paddy water and apple	109
Babassu petiole	BC	Cd^{2+} Pb^{2+}	0.11 µmol L^{-1} 0.056 µmol L^{-1}	drop casting	–0.9 V 120 s	river water	104
Coffee husks	BC	Cd^{2+} Pb^{2+}	15.0 nmol L^{-1} 1.00 nmol L^{-1}	maceration	OCP 600 s	drinking water, ground water, and river water	80
Walnut shell	BC	Cd^{2+} Pb^{2+} Cu^{2+} Hg^{2+}	0.086 nmol L^{-1} 0.175 nmol L^{-1} 0.246 nmol L^{-1} 0.383 nmol L^{-1}	maceration	–1.1 V 150 s	water and soil	110
Oak	BC	Cd^{2+} Pb^{2+} Hg^{2+}	0.090 µmol L^{-1} 0.366 µmol L^{-1} 0.489 µmol L^{-1}	maceration	–1.2 V 250 s	lake water and cucumber	111
Spirogyra	BC	nitrite	8.29 µmol L^{-1}	drop casting	N.F.	N.F.	112
Eggshell membrane	BC	nitrite	0.63 µmol L^{-1}	drop casting	N.F.	tap water, mineral water, and sausage	113
Orange peel	HC	sulfites nitrites	2.08 µmol L^{-1} 43 µmol L^{-1}	drop casting	N.F.	mineral water and wine	114
Tangerine peel	BC	nitrite	0.32 µmol L^{-1}	drop casting	N.F.	soil, spinach and mustard	115

BC: biochar; N.F.: not found; OCP: open circuit potential; HC: hydrochar; CFU: colony forming unit.

toxic chemical species.[116] Techniques[117-122] such as ozone oxidation, electrochemical oxidation, Fenton oxidation, photolysis, and photocatalysis are discussed in the literature with applications, mainly, for the treatment of effluents, and there are studies that use combinations between the techniques to achieve the best efficiency of the process.

AOPs are known to be robust and non-selective processes,[123] which involve the generation of oxidizing radicals capable of reacting with organic compounds to produce carbon dioxide, water, and inorganic ions.[124] According to Wang *et al.*,[125] the electrochemical process can occur via two main pathways (Figure 6), in which the oxidation/reduction of the species occurs through direct charge transfer between the electrodes and the species. In contrast, the indirect route, consists of electrochemical processes occurring through redox mediators generated in the environment, such as hydroxyl radicals ($^{\bullet}OH$) and reacts with species.[126]

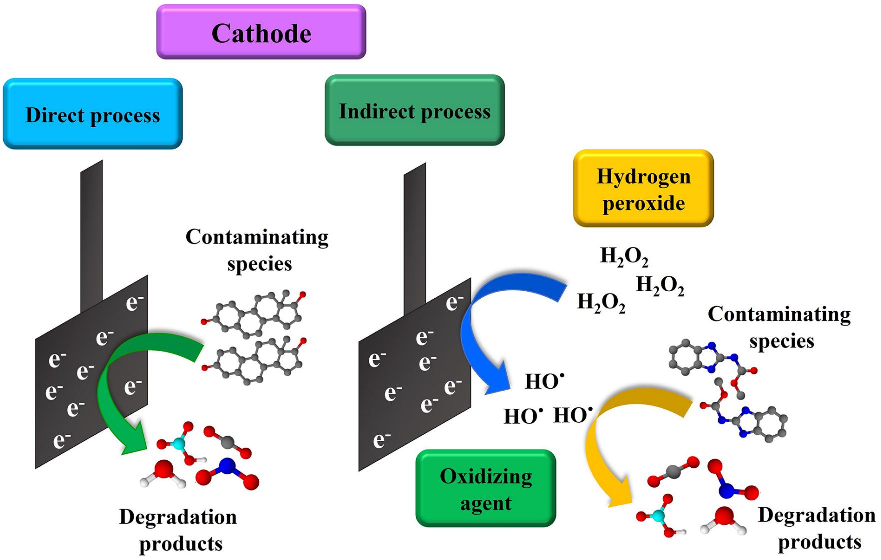

Figure 6. Schematic representation of the electrochemical degradation processes of contaminating species.

Electrochemical degradation has been constantly applied in the remediation of environmental problems caused by the disposal of contaminating species without proper treatment, mainly because of its great operational ease.[127-131] This process can be defined directly by the transfer of charge between the electrode surface and the pollutants or indirectly by the interaction with the reactive oxygen species formed from the oxidation reactions of water in electrochemical systems.[132]

The choice of material that constitutes the electrodes is a fundamental aspect of the efficiency of the degradation process.[133] Because the good performance of the electrode guarantees the transformation of contaminating species, it is necessary to pay attention to its composition. Thus, BC and HC applications in the development and application of modified electrodes have been carried out for the degradation of polluting compounds. Reports in the literature indicate that both carbonaceous materials are efficient as catalysts for redox reactions, in addition to having different structures that favor this application.[134]

Bioderived carbons have been successfully applied in processes of removal and degradation of target compounds. This occurs because these materials have surface characteristics that benefit the interaction and electronic transfer between electrodes and chemical species.[135] Recently, BC and HC are being used as electrode materials, since their high surface area is ideal for the adsorption of compounds, allowing the degradation to occur directly on the electrode surface. In addition, surface functionalization, given the presence of heteroatoms such as sulfur, phosphorus, oxygen and nitrogen in the carbonaceous structure, acts as an electron donor/acceptor, influencing degradation processes that occur indirectly with the formation of oxidizing agents being facilitated.[27,65] Furthermore, strategies to increase the electrical conductivity of these coals have been explored through the incorporation of metallic species or optimizations during the thermal conversion process, resulting in materials with greater catalytic efficiency.[135]

The mechanisms behind the electrodegradation processes of inorganic or organic species are closely related to the reaction medium in which the target compounds are inserted. In this sense, when the system presents substantial target adsorption on the electrode surface, degradation occurs through direct electronic transfer between electrode and analyte. In other cases, the electrode supplies the species present in the medium, such as H_2O_2 and persulfate ($S_2O_8^{2-}$), with electrons for the formation of oxidizing radicals that will act in the conversion and mineralization of the compounds. It may also be possible for both processes to occur concurrently.[65,136]

Zhao *et al.*[73] reported the fabrication of a biochar-based electrode that is efficient in the reductive degradation of 4-chlorophenol in effluents. Carbonaceous material was produced by subjecting the sludge to a temperature of 1000 °C for 2 h. To obtain the electrode, the pyrolyzed solid was mixed with carbon black and polytetrafluoroethylene and dissolved in *N*-methylpyrrolidone. The electrode was repeatedly immersed in nickel aerosol and dried in an oven at 105 °C in a palladium solution to form the composite. The decomposition of 4-chlorophenol (4-CP) was performed in an electrochemical cell using a titanium sheet as the anode, and the developed methodology promoted the removal of 98.9% of the studied analyte. Compared to the others evaluated, the composite electrode containing the sludge biochar was the most efficient in the degradation of the target compound, in addition to being reusable. The authors attributed the result obtained to the increase in the conductive area and the interaction interface of the electrode with 4-CP, promoting the degradation of part of the analyte directly on the surface of the electrode, even though the indirect process is the predominant one.

Cao *et al.*[137] evaluated the electrochemical degradation of 2,4-dichlorophenol (2,4-DCP) using corn stalks hydrochar to obtain a modified electrode. The biomass was then subjected to hydrothermal processing at 240 °C for 4 h. To compare the performance, biochar was synthesized using corn stalk as the raw material, subjected to a gradual temperature increase up to 900 °C, and maintained for 4 h. The methodology developed promoted adsorptive removal of 40.1% (BC) and 36.5% (HC). After 180 min of electrolysis, there was an increase in the removal achieved by hydrochar (65.4%), whereas biochar suffered saturation (ca. 60%), which may be mainly linked to the different structures formed during the two thermal conversion processes. Despite presenting a higher performance in the removal via adsorption of 2,4-DCP, the electrode modified with BC undergoes a rapid stability of its degradation capacity, while the one obtained with HC increases. The characterization of the materials indicate a greater surface area for the BC, which justifies the increase in adsorption, but the surface of the HC is more functionalized due to the lower temperature used in the conversion process. These oxygenated groups act as electron donors and increase the degradation efficiency of 2,4-DCP, using a material obtained with less energy expenditure.

Zhang *et al.*[27] reported the development of an electrode aimed at the electrochemical reduction of nitrate ions. The orange peel biomass mixed with $FeCl_3$ solution for 3 h was subjected to hydrothermal treatment at 150 °C for 6 h. The hydrochar obtained was subjected to a reducing agent ($NaBH_4$) under intense agitation to form of Fe_3O_4. Hydrochar was mixed with copper foam with a binder and a lithium-based reagent, which, after drying, was subjected to calcination for 2 h at 500 °C to form the electrode material. The materials were deposited on a copper foam electrode and comparatively evaluated in the reduction of nitrate through the electrogeneration of oxidizing radicals. The results showed that the hydrochar was responsible for increasing the stability of the electrodes, suggesting a greater interaction between Fe_3O_4 and HC that act to increase the catalytic efficiency of the process. The constructed methodology promoted the removal of approximately 98.9% of nitrate.

Wang *et al.*[138] constructed a three-dimensional electrolysis system using biochar as a particulate electrode for the decomplexation and degradation of Cu-1-hydroxyethylidene-1,1-diphosphonic acid. They mixed commercial biochar derived from almond shells with an acidic solution for 24 h. Up to 90.7% of copper was removed using a graphite sheet anode and cathode, with biochar as catalyst under ideal electrolysis conditions. In addition to the adsorptive effect related to the surface area and the interaction of biochar with Cu^{II} ions, there was a significant participation of the functional oxygenated groups of the BC in the electrolysis process of the compound through its electron donating activity.

Geng *et al.*[139] developed an electrode based on bamboo chopsticks biochar obtained at 600 °C for 2 h. A modified electrode was used as the cathode, and a graphite counter electrode was used to remove nitrate from groundwater. The BC obtained at 600 ºC showed an increase in the surface area and in the amount of micropores compared to those obtained at lower temperatures. The developed method presented an efficiency for nitrate reduction of 75.8% after 4 h of retention. Such results were only possible because of the combination of the specific surface area and electrical conductivity of the biochar applied as an electrode material, that contributed to the adsorption and degradation of nitrate.

Biochar (200 °C) from blue algae (ABC) was used to build an electrochemical system for the degradation of organic pollutants from the electronics industry. The cathode and anode were filled with ABC and ABC modified with iron and/or nickel and applied in an electro-Fenton type process. After evaluating the performance of the different electrodes, a greater removal efficiency was observed when the modified Ni-Fe-ABC was used (85.1%), which was attributed to the synergistic and catalytic effect of the interaction of iron and nickel with the biochar to generation of radicals responsible for the degradation process.[140]

Sandwich-like electrocatalysts based on carbon sheets (CS) were obtained from the pyrolysis of corn starch (800 °C) and loaded with cerium-doped SnO_2 nanoparticles, through the co-precipitation method ($Sn_xCe_zO_y$/CS). The application of these electrocatalysts demonstrated a high performance in the direct electrodegradation of tetracycline. By varying the molar composition of the compounds, $Sn_{0.75}Ce_{0.25}O_y$/CS greater catalytic activity was observed with removal of 95% of tetracycline occurring in 120 min, and more than 90% of the total organic matter present being mineralized in 480 min. The high electrocatalytic performance observed was associated with the synergistic effect of the composite containing biochar, which increased the mass transfer effect, the adsorptive capacity, and the ease of passage of electrons from the modified electrode.[141]

Hollow spheres of cerium dioxide (CeO_2) were prepared and loaded onto porous carbon obtained by pyrolysis of skimmed cotton (CSC) at 500 °C. Being applied in the indirect degradation of phenol, the removal efficiency reached 97.6% in 120 min, and the CSC/CeO_2 cathode showed excellent stability even after being used 20 times with a reduction in removal of only 5.4%. The CSC/CeO_2 composite material showed high catalytic efficiency in the formation of oxidizing agents and phenol degradation, being superior to many works mentioned in the literature. These results were associated with the synergistic effect of the interaction between the cerium oxide hollow spheres and the biochar obtained from cotton.[142]

Rice straw was used as a precursor for conductive biochar obtained at 900 °C, which was applied to obtain a modified electrode used to remove cadmium. Simulated wastewater polluted with cadmium was treated by electrodeposition with a removal rate of 76.60% in 240 min.[143] The authors concluded that biochar acts by increasing the area of interaction with the analyte, as well as helping in the structural stability and electronic transfer of the electrode, promoting the achievement of a low-cost method for the treatment of water polluted by heavy metal.[143]

Table 2 summarizes some studies in the literature that apply HC or BC in the degradation and removal of organic and inorganic contaminants through AOPs.

As in the field of electroanalysis, the application of BC and HC in the development of electrodes for the electrochemical removal of species is still a recent field of study, which is reflected in the number of articles published on the subject. The notable predominance of the use of BC compared to HC may be mainly related to the different properties observed for the materials, such as the smaller surface area of HC resulting from the reactions that occur during its formation, even though it has a lower cost to obtain. The functionalized surface of these materials contributes to greater interaction of the electrodes, as well as facilitating the generation of oxidizing agents that will help in the degradation of the target compounds, increasing the performance of the process. On the other hand, the oxidizing agents generated during electrodegradation can cause damage to the structure of the electrodes, which reduces usability. Despite this, the use of HC and BC is responsible for

Table 2. Precursor biomass, analytes, and removal efficiencies of electrodegradation processes using bioderived coals reported in the literature between 2020 and 2023

Biomass	Charcoal	Target compound	Removal efficiency / %	Pathway	Reference
Corn straw	BC	2,4-dinitrotoluene	100	indirect	144
Iris sibirica L. roots	BC	2,4-dichlorophenol	98	indirect	145
Sewage sludge	BC	methyl orange tetracycline	98 88	indirect	146
Rice straw	BC	cadmium	76	direct	143
Skimmed cotton	BC	phenol	97	indirect	142
Corn starch	BC	tetracycline	95	direct	141
Chlorella	BC	borberine hydrochloride	91	indirect	147
Sludge	BC	sulfamethazine	98	indirect	148
Kapok fiber	BC	sulfamethoxazole	97	indirect	149
Sugarcane bagasse	BC	4-chlorophenol	99	indirect	150
Sludge	BC	4-chlorophenol	99	indirect	73
Coconut shell	BC	2,4,6-trichlorophenol	ca. 55	indirect	151
Blue algae	BC	mono-2-ethylhexyl phthalate	85	indirect	140
Almond shells	BC	EDTA	97	indirect	152
Sludge	BC	methyl orange	94	indirect	153
Algal powder	BC	sulfadiazine	99	indirect	154
Corn stalk	BC	nitrobenzene	99	direct	155
Bamboo	BC	coking wastewater	92	direct	156
Corn stalk	HC	2,4-dichlorophenol	76	indirect	137
Orange peel	HC	nitrate	99	direct	27
Bamboo	BC	nitrate	76	direct	139
Almond shells	BC	Cu-1-hydroxyethylidene-1,1-diphosphonic acid	91	indirect	138

BC: biochar; HC: hydrochar; EDTA: ethylenediaminetetraacetic acid.

increasing the structural integrity, being environmentally friendly and reducing the cost of obtaining electrodes, which makes it even more viable for application in environmental remediation compared to traditional procedures and electrodes.

3. Conclusions

The excellent properties of biomass thermal conversion products, such as biochar and hydrochar, have led to their widespread application in the most diverse sectors. The field of development of electrode materials based on these derivatives has been widely explored because these materials are considerable sustainable and have a low production cost. This contributes to the valorization of biomass, through the generation of several value-added materials. Since the possibility of modifying these carbonaceous materials emphasized their versatility and reinforces their application, numerous advances in the field of monitoring and degradation of organic and inorganic compounds have been achieved, mainly by the potentialization of electrodes when composed of these materials used as modifiers. However, the need for pre- or post-treatment of materials using high temperatures and reagents to improve their properties makes the production process more expensive and generates waste. This highlights the need to develop single-step methodologies that use low-cost and bioavailable reagents.

4. Novelties and Challenges

The development of biochar and hydrochar and their applications as components in electrodes represents the union of new and promising areas of research. In this sense, the insertion of these materials in electrochemical processes has contributed to the advancement of more efficient and environmentally friendly electrochemical technologies, in addition to converting potentially toxic materials such as sludge and sewage into usable substrates. Associated with this is the low cost and relative operational ease of the synthesis and application procedures, when compared to chromatographic methods and the production of nanomaterials. The versatility of these coals is evidenced by the different possible combinations with other chemical species, resulting in synergistic interactions that are responsible for the greater selectivity, sensitivity and efficiency of the modified electrodes. However, there are still challenges to be overcome in relation to the electrochemical compatibility of carbonaceous materials, since several processes are commonly used to optimize the composition, especially when hydrothermal carbonization is the chosen route. In addition, the use of biomass from animals as the source of these coals, such as bones, fat, feathers, and eggshells, represents a gap still little explored by the electrochemical area. Advanced oxidative processes employing electrochemical techniques associated with coals have proven to be an excellent proposal, as the exploitation of their catalytic and adsorptive properties may be the key to reducing the use of expensive and potentially toxic materials such as some nanomaterials, for example. Thus, the use of biochar and hydrochar in electrochemical devices has the potential to reconcile more sustainable and efficient solutions for monitoring and removing compounds of interest.

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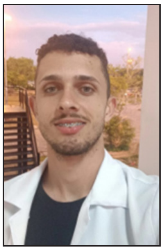

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