Use of activated charcoal as bio-adsorbent for treament of residual waters: a review

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ABSTRACT: Adsorption is gaining attention by becoming one of the most used technologies for the removal of contaminants from water. Adsorption with activated charcoal (AC) is an efficient method for treatment of effluents; the main advantage of AC production is the use of residues that would be inappropriate discarded. The objective of this work was to gather and organize the information available about the potential of using activated charcoal as a bio-adsorbent. Researches were conducted on scientific articles about the production of activated charcoal with adsorption characteristics for the removal of contaminants from residual waters. The efficiency of this technique is dependent on different parameters that affect the adsorption process, such as: pH of the solution, dye initial concentration, contact time, adsorbent amount, and temperature. The articles studied showed that the bio-adsorbent characteristics of charcoals are promising for the removal of pollutants from residual waters. The isotherm adsorption models developed by Langmuir, Freundlich, and Brunauer, Emmett, and Teller (BET) are often used to evaluate the adsorption capacity of activated charcoals.
Keywords: industrial effluents; contaminants; environmental management.

Emprego do carvão ativado como bioadsorvente para o tratamento de águas residuais: uma revisão

RESUMO: A adsorção vem ganhando destaque com uma das tecnologias mais empregadas na remoção de contaminantes em águas. No tratamento de efluentes, a adsorção com carvão ativado (CA) apresenta-se como um método eficiente. A principal vantagem da produção de CA é o aproveitamento de resíduos que seriam descartados de forma inadequada. O objetivo desta pesquisa é organizar algumas informações disponíveis com relação ao potencial do carvão ativado como bioadsorvente. Foram realizadas pesquisas em periódicos sobre produção de carvão ativado com características adsortivas na remoção de contaminantes em águas residuais. A eficácia dessa técnica sob diferentes parâmetros influencia no processo de adsorção, tais como: pH da solução, concentração inicial do corante, tempo de contato, quantidade do adsorvente e temperatura. Em todos os artigos estudados, as características dos carvões como bioadsorventes se mostraram promissores no processo para remoção de poluentes em águas residuais. Conclui-se que os modelos de isotermas de adsorção desenvolvidos por Langmuir, Freundlich e BET são frequentemente utilizados para avaliar a capacidade de adsorção dos carvões ativados.
Palavras-chave: efluentes industriais; contaminantes; gestão ambiental.

1. INTRODUCTION

The conservation of natural resources has become an important concern, mainly due to the neglect of humans who irresponsibly pollute and contaminate springs and change natural environments. The contamination of a water course is not always easily detected, since most contaminants present no visually identifiable evidences. Color is a parameter that raises attention due to the visual impact, and facilitates the identification of contamination.

Adsorption is gaining attention by becoming one of the most used technologies for the removal of contaminants from waters. It is a physical-chemical phenomenon based on the bound of components in gas or liquid phase to the surface of a material in solid phase. The components that connect to this surface are called adsorbates, and the material in solid phase that withhold them is called adsorbent.

The capacity of activated charcoal to remove different compounds in contaminated waters increased the interest and demand for this product. Its adsorption provides some advantages over classic methods of treatment of effluents, such as: low generation of residues, efficiency in the removal of substances, simple operation, easy recovery of metals, and the possibility of reuse the adsorbent.

Thus, the present study intended to highlight the potential of using activated charcoal with adsorption characteristics for the treatment of residual waters, through a literature review.

2. LITERATURE REVIEW
2.1. Activated charcoal

The first use of activated charcoal in the industrial sector occurred in the England, in 1794, as a discoloration agent in the sugar industry. This event marked the beginning of studies about activated charcoal on liquid phases. The first large-scale application on gas phases occurred in the mid XIX

century (1854); the mayor of London ordered the implementation of charcoal filters in all ventilation systems of sewers to remove unpleasant odors. In 1872, gas masks with charcoal filters were used in chemical industries to prevent inhalation of mercury vapors (BANDOSZ, 2006).

Theoretically, activated charcoal can be produced from any carbonaceous material. However, some characteristics, such as high fixed carbon content and low mineral content, should be considered in the choice of a precursor material. Products of plant origin are among the most interesting raw materials to obtain activated charcoal, mainly, lignocellulosic ones, which present cellulose, hemicellulose, lignin, and inorganic components as their main chemical components. Several agricultural wastes, including wood, plants, coconut shells, sugarcane bagasse, and fruit seeds, are among the products of plant origin that also present good properties for charcoal production (BOUCHELTA et al., 2008).

Activated charcoal is the name given to a group of charcoals that are characterized by having carbonaceous material of porous structure that presents a small quantity of heteroatoms bound to carbon atoms, mainly oxygen, and an internal surface that makes it capable of adsorbing molecules in liquid or gaseous phase (GORGULHO et al., 2008).

Commercial charcoals present a high surface area, high porosity, and variable surface chemical characteristics (presence of different functional groups, mainly oxygenated) with high reactivity degree. Variations in temperature and activation time affect the formation of chemical groups in the charcoal surface. The surface area, volume of pores, basicity, and adsorption capacity increase as the temperature and activation time are increased. However, it decreases charcoal yield and the mechanical resistance index (BORGES et al., 2016).

Charcoal can be activated by chemical or physical processes by using different compounds that, in general, increase the charcoal surface area through reactions throughout the material surface (MOHAN; PITTMAN JR, 2006).

The chemical activation consists of the impregnation of activator agents, such as phosphoric acid (H_3PO_4), to the material before carbonization. These agents provide the formation of cross connections, making the material less prone to volatilization when heated to high temperatures (Figure 1). The physical activation consists of reactions of charcoal with water or CO_2 vapors, or a mixture of these two gases after carbonization.

The most used chemical activators are: KOH (potassium hydroxide), K_2CO_3 (potassium carbonate), NaOH (sodium hydroxide), $NaCO_3$ (sodium carbonate), $MgCl_2$ (magnesium chloride), H_3PO_4 (phosphoric acid), $AlCl_3$ (aluminum chloride), and $ZnCl_2$ (zinc chloride).

KOH is very selective in the activation process, causing more localized reactions on the precursor and is more effective for materials with ordered structure. The use of H_3PO_4 as activator provides high surface areas to charcoals and causes physical and chemical modifications by penetrating the structures and partially dissolving the biomasses (WANG et al., 2010). Charcoals activated with sodium acetate present pronounced formation of micropores; moreover, their characteristics vary according to size, volume, and distribution of pores and presence of different functional groups in the charcoal surface (UTRILLA et al., 2011).

Activated charcoal are more porous than common charcoal (Figure 2).
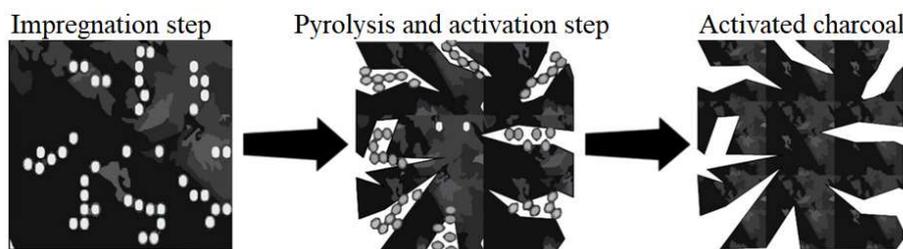
Figure 1. Representation of the chemical activation process. Source: Adapted from Pereira, 2010.
Figura 1. Esquema demonstrativo de como ocorre à ativação química. Fonte: Adaptado de Pereira, 2010.
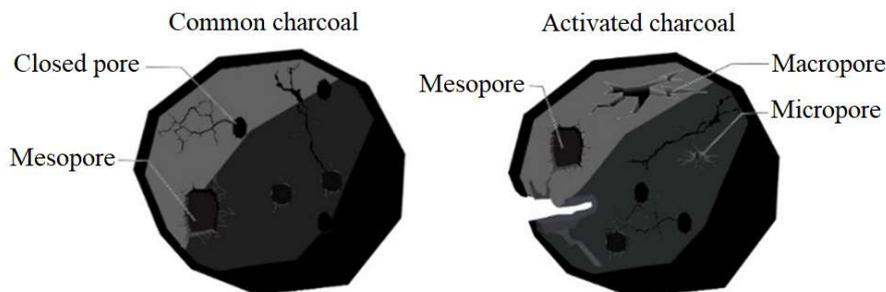
Figure 2. Representation of the porosity of common and activated charcoals. Source: Adapted from Souza, 2018.
Figura 2. Porosidade do carvão comum e ativado. Fonte: Adaptado de Souza, 2018.

2.2. Characteristics of charcoal pores

Pore size distribution depends on the charcoal material type and activation method used. Considering the adsorption properties, the International Union of Pure & Applied Chemistry (IUPAC) established a classification according to

the material form and dimensions. Regarding the form, pores can be classified as open or closed; open pores are the holes in the external surface, represented by the letters B, C, D, and F in Figure 3. Pores that allow the flow of fluids are classified as transport pores, which may present branches (cage type)

that do not contribute to the transport. Closed pores are those consisted of an isolated hole. The charcoal porosity is dependent on the different sizes and forms of the pores, including their depth. This is an important aspect to evaluate the performance of activated charcoals (ZDRAVKOV et al., 2007).

All activated charcoals contain micro, meso, and macropores in their structures, with a considerably variation in relative proportion between them depending on the precursor and manufacturing process used. According to the IUPAC, charcoal pores are classified by their form and dimensions (Table 1).

Different parameters can be used to describe the adsorption capacity of activated charcoals (AC). This capacity can be evaluated using different compounds, such as iodine, methylene blue, and molasses, to measure their porosity (Figure 4) (DI BERNARDO; DANTAS, 2005).

The iodine number describes the microporosity expressed as quantity of iodine (weight) adsorbed by a given AC under specific conditions, usually related to adsorption (DABROWSKI et al., 2005).
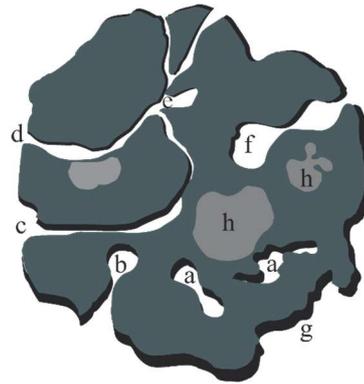
Figure 3. Representation of the different pore types: (a) closed, (b) bottleneck, (c) cylindrical, (d) tapered, (e) interconnected, (f) irregular, and (g) surface rugosity. Source: Adapted from Gimenez et al., 2014.
Figura 3. Representação dos diferentes tipos de poro: (a) fechado, (b) gargalo, (c) cilíndrico, (d) cônico, (e) interconectado, (f) irregular, e (g) rugosidade da superfície. Fonte: Adaptado de Gimenez et al., 2014.

Table 1. Classification of pores according to their diameter and function.
Tabela 1. Classificação dos poros de acordo com o diâmetro e função.

Tipo of pores	Dimensions	Characteristics
Primary micropores	Smaller than 0.8 nm	Contribute the most to a surface area that has high capacity to adsorb small molecules, such as gases and common solvents.
Secondary micropores	Between 0.8 and 0.2 nm	
Mesopores	Between 0.2 and 50 nm	Important for the adsorption of large molecules, such as dyes, and make most of the charcoal surface area impregnated with chemical products.
Macropores	Larger than 50 nm	Commonly without importance for adsorption, but serve as a transport pathway for gas molecules.

Fonte: IUPAC.
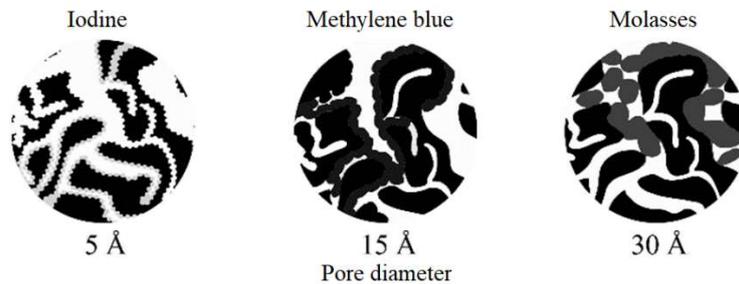
Figure 4. Adsorption of activated carbon. Source: Adapted from Agnicarbon.
Figura 4. Adsorção do carvão ativado. Fonte: Adaptado de Agnicarbon.

Methylene blue indicates the mesoporosity expressed as quantity of methylene blue (weight) adsorbed by a given AC under specific conditions. The methylene blue index indicates the capacity of AC to adsorb molecules with similar dimensions to methylene blue and is related to the surface area of pores larger than 1.5 nm (DI BERNARDO; DANTAS, 2005).

The number of molasses indicates the macroporosity; it is a discoloration index related to the capacity of AC to adsorb molecules of large molar masses. This discoloration index is measured in relation to a molasse solution and is expressed in percentage of discoloration relative to a standard carbon (DI BERNARDO; DANTAS, 2005).

2.3. Isotherms

The adsorption capacity of a material can be quantitatively evaluated by the isotherms, which show the balance between the fluid phase concentration and the accumulated adsorbate concentration in adsorbent particles,

under hot conditions. Thus, isotherms can be represented by mathematical expressions that correlate the amount adsorbed to the pressure or concentration in a specific temperature. The shape of these isotherms is related to the porosity of the solid adsorbent (LOPES et al., 2002).

Adsorption isotherms in liquid phase are represented by curves of solute concentration in solid phase as a function of the solute concentration in fluid phase in a specific temperature. Isotherms are the most convenient factor to determine the adsorption balance and the theoretical treatment. Therefore, isotherms are the first experimental information used to choose the most appropriate charcoal for a specific application.

The aspect of isotherms is the first experimental tool to determine the type of interaction between the adsorbate and adsorbent, which can be classified in five classes, as proposed by Brunauer, Emmett, and Teller (BET). Adsorption isotherms are curves built by using equilibrium data (q_e = adsorption capacity in equilibrium; C_e = equilibrium

concentration of the solute in liquid phase) at constant temperature. Isotherms are useful and important by providing estimates of the thermodynamic parameters of the process.

The types of isotherms (Figure 5) are related to the type of pores involved. Isotherms type I are associated to micropore adsorptions; isotherms type II are related to adsorption in non-porous systems; isotherms type IV denote desorption not coinciding with adsorption, resulting in presence of a fine capillarity; isotherms type VI denote a gradual adsorption of a multilayer and are related to adsorptions in non-porous and uniform surfaces; and isotherms type III and V are related to weak interactions in systems that present macro and mesopores.
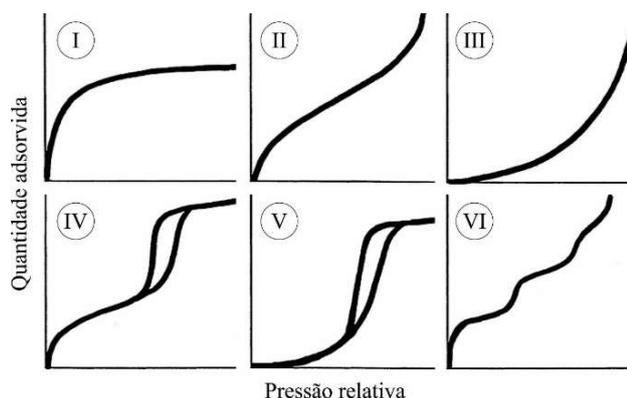
Figure 5. Main isotherm types. Source: Adapted from IUPAC.
Figura 5. Principais tipos de isotermas. Fonte: Adaptado da IUPAC.

Adsorption isotherms enable the determination of parameters related to the balance of the process, such as constant of adsorption and maximum amount of adsorbate that the material can retain in the adsorbent surface. Isotherms present several forms, providing important information on the adsorption mechanisms and the nature of forces between the adsorbent and the solute.

Researches commonly evaluate the capacity of activated charcoals to retain organic compounds through adsorption isotherms in the liquid phase, relative pressure (P/P_0), and amount of gas adsorbed.

Several mathematical models describe the correlation between the amount adsorbed per unit of weight of adsorbent and the adsorbate concentration in water. The most used models are Langmuir, Freundlich, and Brunauer-Emmett-Teller (BET), due to their potential to predict the maximum adsorption capacity of solids and describe the dynamics of the experimental data.

2.4. Langmuir isotherm

The first theoretical equation correlating the amount of gas adsorbed to the equilibrium pressure was proposed by Langmuir. The Langmuir isotherm is a characteristic of microporous solids that have relatively small external surfaces. The interpretation of the different adsorption models is carried out by considering the binding energy. The equation predicts the adsorption capacity of a saturated monolayer under high concentrations of solute (KUMAR; SIVANESAN, 2006).

The Langmuir model estimates the adsorption capacity of charcoals and the type of adsorbate-adsorbent interactions (Equation 1). In this model, the attraction between the

adsorbate and adsorbent surfaces is mainly based on electrostatic or Van Der Waals forces, and occurs in monolayers.

$$Q_e = \frac{q_{max} \cdot k_L \cdot C_e}{(1 + K_L \cdot C_e)} \tag{01}$$

where: Q_e is the amount adsorbed in the equilibrium, expressed as mg g[-1]; $q_{máx}$ is the maximum sorption capacity, expressed as mg g[-1]; K_L is the sorption energy constant, expressed as L mg[-1]; and C_e _e is the concentration of ions in equilibrium, expressed as mg L[-1].

2.5. Freundlich isotherm

The Freundlich isotherm does not clearly indicates a physical interpretation of real phenomena. However, this empirical correlation presents a general characteristic and usually shows good results for modeling adsorption processes in liquid or gas phase (WAWRZKIEWICZ et al., 2015). It considers that the adsorption occurs in a heterogeneous surface and is based on the existence of a structure in several layers, predicting an exponential distribution of several adsorption sites with different energies, and that the adsorption energy decreases because of the adsorption; therefore, the Freundlich equation can be applied to non-uniform surfaces.

The Freundlich model (Equation 2) provides a representation of adsorption equilibrium of only one solute. The equation of this model indicates that the adsorption energy decreases logarithmically as the surface become covered by the solute, which differentiates it from the Langmuir equation.

$$Q_e = k_F \cdot C_e^{1/n} \tag{02}$$

where: Q_e is the amount adsorbed in the equilibrium, expressed as mg g[-1]); K_F is the sorption capacity (mg g[-1]) (L mg[-1]) $1/_n$; C_e is the adsorbate concentration in equilibrium, expressed as mg L[-1]; K_F and $1/_n$ are Freundlich empirical parameters, which depend on several experimental factors and are related to the adsorbent adsorption capacity and the adsorption intensity, respectively; the exponent $1/_n$ indicates whether the isotherm is favorable; $1/_n$ values within the interval $0.1 < 1/_n < 1$ denote favorable sorption conditions: the closer the $1/_n$ value is to 1, the most favorable is the process.

2.6. Brunauer-Emmett-Teller (BET) equation

In 1938, Brunauer, Emmett, and Teller proposed a theory for the adsorption phenomenon considering the same adsorption mechanisms of the Langmuir theory. However, they introduced some hypotheses, which admit the possibility that the layer has the capacity to produce adsorption sites, generating the deposition of one layer on the other.

The BET equation was developed to correlate the values obtained from adsorption isotherms to specific areas (Equation 3).

$$Q_e = \frac{q_m \, b \, C_e}{(C_s + C_e)\,[1 + (b-1)C_e/C_S]} \tag{03}$$

where: Q_e and q_m have the same meaning presented in the Langmuir model, i.e., the amount adsorbed per unit of weight of the solvent (mg g[-1]) and maximum adsorption capacity (mg g[-1]); b is the saturation in all layers; C_e is the concentration of equilibrium in the fluid; C_s is the concentration of solute in the saturation of all layers.

The constants of Langmuir and BET with negative values

present no physical sense, since they do not fit to the hypotheses considered in the model studied.

2.7. Methylene blue

The chemical structures of dyes present several complex organic molecules and aromatic molecular structures, making them more stable and resistant to degradation. There are more than 1,000 commercial dyes available, which usually have synthetic origin and complex aromatic molecular structures, making them more stable and resistant to biodegradation (WAWRZKIEWICZ et al., 2015).

Methylene blue is a water-soluble cationic dye used to compose models in oxidation reactions by presenting adsorption within visible wavelengths ($\lambda_{máx}$ = 665 nm), high solubility, and similar properties to textile dyes. This material has been used to compose models for organic contaminants, oxidation reactions, and for the characterization of adsorbents regarding capacity to adsorb large molecules.

AC has excellent adsorption properties due to its high surface area and porous structure, and is the most used adsorbent for the removal of dyes. However, its high cost has promoted a search for cheaper adsorbent materials that do not require many previous treatments, such as natural materials, agricultural and industrial residues, and domestic residues, including fruit residues.

Several low-cost adsorbent materials, and effective and relatively easy techniques for the treatment of residual waters, have been studied, such as sisal fiber (DIZBAY-ONAT; VAIDYA; LUNGUC, 2017), fabric residues (NOWROUZI et al., 2017), solid residues with lignin (HAO et al., 2017), sunflower seed bran (MORALI; DEMIRAL; ŞENSÖZ, 2018), acrylic fiber residues (NAEEM et al., 2018), coffee residues (POBLETE et al., 2017), fruit processing residues (SELVARAJU; ABUBAKAR, 2017), and residues of *Nigella sativa* L. (ABDEL-GHANI et al., 2017).

2.8. Adsorption tests of activated charcoal

Several methods have been studied and used to treat different types of effluents to meet the emerging standards and legislations for environmental protection. The technologies used include adsorption because it is an efficient, highly selective, and economically viable process.

Adsorption is one of the most used technique for the removal of contaminants from water. It is a physical-chemical phenomenon based on the bound of components in gas or liquid phase to the surface of a material in solid phase. The components that connect to this surface are called adsorbates; the material, in solid phase, that withhold them is called adsorbent. Desorption is the removal of molecules from sites of the surface, which is an interesting subject for the regeneration of adsorbents.

This technique provides significant advantages, including low cost and availability, profitability, easy operation, and efficiency when compared to the conventional methods. The efficiency of the adsorption process can be affected mainly by the size of the surface area and amount (weight) of the adsorbent. Low-cost adsorbents have presenting promising benefits for commercial purposes and for the development of adsorption processes. This denotes a need for deeper studies with tests of treatments of industrial effluents using these materials (BAZRAFSHAN et al., 2016).

The adsorption technique with the use of activated charcoals is economically viable and presents simple application for the removal of contaminants. The advantages of activated charcoals over other conventional treatments are: the low need for area (approximately 25% to 50% of the area needed for a biological system); higher operational flexibility; and lower sensitivity to daytime variations (AMIN, 2008).

Activated charcoal is one of the most used adsorbents for the removal of contaminants from liquid mediums because of its high capacity to capture molecules by chemical interactions, and its high removal rates due to its large specific surface area. The application of this material to effluent treatments is related to decreases in organic material, mainly regarding species that alter the color of contaminated mediums.

The pH of the liquid phase is an important variable for the control of the adsorption process because it affects the nature of the charge on the surface of the adsorbent and the adsorbate speciation. Thus, the efficiency of the process is strongly affected by the pH of the medium; when the pH is below the zero-point-charge pH of the adsorbent, the surface is positively charged, enabling electrostatic interactions between the surface and the free fluoride (VALENÇA et al., 2017).

Dye adsorption processes are dependent on pH and ionic effects. Jung et al. (2016) found maximum adsorption capacities for activated charcoals produced from calcium alginate/spent-coffee-grounds (CA-SCG), with pH values of 3.0 and 11.0 for 665.9 and 986.8 mg g^{-1} at 30 °C, respectively. The tests showed that CA-SCG have potential for reuse, presenting removal efficiencies higher than 80%, even after seven consecutive cycles.

The effect of pH may vary according to the biomaterial, type of dye, and charcoal granulometry and activation used. Some studies on the removal of methylene blue were carried out by Azharul Islam et al. (2017), confirming its efficiency.

Costa; Furmanski; Dominguini (2015) evaluated the production of activated charcoal from nut shells, and physical and chemical activations, and found that the material produced has potential to remove methylene blue from liquid solution and that the treatment with ZnCl$_2$ increases the adsorption capacity from 68 to 104 mg g^{-1}. They also point out that the Langmuir isotherm model presents good correlation and adjustment to the results under the conditions evaluated.

Activated charcoal is one of the most studied adsorbents used for treatment of residual waters and removal of phenolic compounds (BAZRAFSHAN et al., 2016). Zarei et al. (2013) evaluated the efficiency of activated charcoal from *Moringa peregrina* peels for the removal of phenol from liquid solutions and found that it is a low-cost adsorbent that presents high performance for removal of phenol from liquid solutions.

Borges et al. (2016) used wood residues (*Eremanthus* sp.) to produce activated charcoals in briquette form (AC-B), using a physical activation process and different temperatures and activation times, and found good adsorption of phenol (73%) and methylene blue (23%) at concentrations of 1,000 mg L^{-1}, using tests in batches. They also found that the isotherms of the best AC-B showed maximum adsorption capacities of 16.1 mg g^{-1} (methylene blue) and 98.20 mg g^{-1} (phenol).

Leite et al. (2017) produced activated charcoal from *Casuarina equisetifolia* cones and found satisfactory adsorption capacity at the environment temperature, reaching 52.3 mg g^{-1} within 10 minutes of contact using 1.0 g of activated charcoal disperse in 500 mL of an oily synthetic effluent.

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They present similar results to those found for commercial activated charcoals, and the adsorption capacity of the activated charcoal produced was higher than that obtained with the commercial product under the same operational conditions.

3. CONCLUSIONS

The articles studied showed that the bio-adsorbent characteristics of charcoals are promising for the removal of pollutants from residual waters. The isotherm adsorption models developed by Langmuir, Freundlich, and Brunauer, Emmett, and Teller (BET) are often used to evaluate the adsorption capacity of activated charcoals. The literature review showed that several studies systematically detail the adsorption and removal of residues using activated charcoal.

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