

Application of Agricultural Waste from Main Brazilian Crops as Adsorbent for Wastewater Treatment

Aplicação de Resíduos Agrícolas das Principais Culturas Brasileiras Usados como Adsorventes para o Tratamento de Efluentes

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Adsorption is a powerful method for wastewater treatment. The adsorbent materials used are limited due to the high cost of production and for their difficult regenerability. This work aims to show the importance of reusing agricultural residues, transforming them into efficient adsorbent materials. Highlighting the growing interest in the subject through bibliometric analysis and punctuating the main preparation techniques with direction for specific characteristics desired in the materials, understood through the main techniques of physical and chemical characterization. It will also be shown the economic and quantitative relevance for Brazil of some agricultural crops of worldwide occurrence, as well as the potential of transforming their residues into adsorbent materials for the treatment of effluents. Finally, studies involving real effluents will be presented, corroborating the efficiency of these materials and the possibility of future industrial applications. This set of information presents the reader with a valuable amount of information about the real possibility of reusing agricultural solid waste as a powerful tool in the treatment of urban and industrial effluents, thus increasing reuse and attacking two important environmental problems.

Keywords: Bibliometric analysis; agricultural waste; low-cost adsorbents; wastewater treatment.

1. Introduction

Population growth, and the inherent need to meet the demands of a vast number of people, promotes the emergence of issues which endanger the delicate balance in ecological systems around the world. Among these problems, a spotlight must be placed upon the cumulative effect of man-made products, which, in large scale, is potentially devastating to environmental resources.¹ The finitude of natural and hydric resources, as well as the production solid, liquid and gas residues, manufactured through conventional production methods propel the search for efficient and sustainable technologies, and the development of research that improve processes and minimize environmental impacts.²

The rise in biomass uses as food, forage, bioplastic, biofuel, and bioenergy tends to significantly impact agricultural production, since the application of these products emerges as an alternative to natural resources demand. Such use has increased the portion agriculture occupies in the world economic scenario.³ Agricultural development is one of the most powerful tools against extreme poverty and hunger and might be the only way to feed an ever-growing population, which, according to estimates, will reach 9.7 billion people by 2050.⁴

Biomass comes from a wide variety of living organisms: animals, plants, microorganisms etc. Concerning agricultural biomass, an ample diversity of products derives from plant raw material processing and, consequently, the quantity of liquid, solid and gas residues produced is proportionally colossal. Among them, lignocellulosic solid waste, with high organic matter content and deriving out of human and animal food, is responsible for large environmental impacts: disposal of biomass, which takes place during harvest and processing, creates billions of tons of garbage annually. The requirement of a more productive and sustainable agriculture drives the search for new systems of production and processing, reusing or recycling raw material residues, linked to a circular economy concept and based upon reducing the waste disposal through a new chain of added value products.⁵

Brazil is one the world titans in the agribusiness and, in the last 40 years, has shown expressive increase in productivity. Nevertheless, such escalation depends on several parameters: population numbers; average income and consumption; technology innovation; land appreciation; preservation of natural resources; agricultural policies; environmental laws; regulation marks;

and international agreements.⁶ According to the Brazilian Institute of Geography and Statistics (IBGE), Brazil is a leading player in the production of a number of crops: cotton, peanut, rice, oat, banana, cocoa, coffee, sugarcane, cashew, maize, soy, sorghum and wheat. In 2022, a steep increase in tonnage harvested was observed in several of the abovementioned crops, from which the more pronounced were seen in coffee, cotton, sorghum, peanut, beans, wheat and maize, with yield raises of 9.7%; 15.0%; 21.0%; 27.0%; 27.0%; 27.3% and 37.4% respectively.^{4,7}

Accompanying the expressive food harvests, there is a proportional boost in the quantity of residues generated, and inadequately disposed. A rising tendency employed to mitigate this problem is the application of biomass as fertilizers, fuel material and adsorbents. According to the Food and Agriculture Organization of the United Nations (FAO), the world agroindustry production of residues reached 1.3 billion ton a year in the last decade, and one third of this was attached to wastage in the food industry.⁸

The usage of agriculture residues as adsorbents in wastewater treatment is becoming frequent due to the growing concern with the scarcity of hydric resources. This process takes advantage of the biomass capability of efficiently, and safely, removing pollutants via adsorption in fluids. They are not, however, the only adsorbents available; other materials, such as active carbon, zeolites and clays are also employed to the same end.⁹ Notwithstanding, plant biomass is a promising source for adsorbents due to the low cost, it is widely available and easy to obtain, in addition to possessing inherent interesting adsorptive properties, namely: high surface area, rugosity, morphology, and pore sizes.

Due to Brazilian monumental territorial extension, there is a massive diversity in biomass sources for researchers to study, among which it is imperative to highlight sawdust, husks, fruit seeds and bagasse, almond seed, sugarcane, grains, legume plants and mate.¹⁰ Agriculture residues in Brazil come from different sectors, due to the country's diversified economic matrix and ample extension. Therefore, in consonance with the production of goods and products, there is also the generation of residues. Using these otherwise discarded materials is an auspicious alternative to reduce natural resources extraction and pollutant accumulation. And, as a perk, this option increments the sustainability and economic value of the industry.¹¹

There are several methods employed in the treatment of liquid effluents. The most efficient techniques are: adsorption, membrane filtration, reverse osmosis, precipitation, electrochemical treatments, and ionic exchange.¹² Standing out of these processes, adsorption has become a method of choice to treat large volumes of effluent, due to the efficiency, sustainability, low cost, and feasibility it provides.¹³

Adsorption is a well-known, efficient and economic method used to remove impurities in liquid effluents. It is

based on a mass transfer phenomenon where molecules present in the fluid layer (adsorbate) adhere to a solid surface (adsorbent).¹⁴ Adsorption can occur through physisorption, chemisorption, ionic exchange or microprecipitation, due to electrostatic or chemical interactions which take place on the surface of the material, i.e., it is a surface adhesion phenomenon.¹⁵

Using natural adsorbents in wastewater treatment has shown interesting results in the removal of a wide range of water-soluble contaminants, even the ones known as persistent pollutants, which get this label due to their unusual resistance and recalcitrance to conventional techniques. Some examples that are part of this class are: micropollutants¹⁶; textile dyes¹⁷; oil spill¹⁸; pharmaceuticals¹⁹; heavy metals²⁰; and biocides²¹.

Bearing the information mentioned above in mind, the present work aims to demonstrate the potential applications of biomass derived from important Brazilian crops as sources of natural adsorbent materials used in the environment remediation and circular economy, through the transformation of agricultural residues into added value products. These adsorbents, that otherwise would be seen as waste, are employed in efficient and sustainable treatment of liquid effluents.

2. Agricultural Residues used as Adsorbents in Effluent Treatment: Bibliometric Analysis

A bibliometric analysis was carried out in October 2022, based on the search in the Scopus database of the terms adsorbent ("ADSORBENT"), agricultural residues ("AGRICULTURAL WASTE") and effluent treatment ("WASTEWATER TREATMENT") in the title, abstract and keywords from articles and reviews published in scientific journals written in English. Figure 1 presents the number of publications over time. The search returned 330 scientific articles, divided into 253 articles with original research data and 77 critical review articles, with a significant increase in production in the last six years.

The author who published the most on the topic was Vinod Kumar Gupta (six articles), from the University of Johannesburg, South Africa, followed by Alok Mittai (five articles), from the National Institute of Technology, India. The ten authors with the highest number of publications are shown in Figure 2a. The countries with the highest number of publications are India, China and Malaysia (Figure 2b) and the journals with the most articles are *Desalination and Water Treatment*, *Chemosphere* and *Bioresource Technology* (Figure 2c). Notably, this is a multidisciplinary topic that arouses interest in lines of research related to the areas of environment, energy, chemistry and engineering. The dominant areas of study, illustrated in Figure 2d, are environmental sciences (226), chemical engineering (92), chemistry (84), and engineering (64). However, it is noteworthy that the Scopus categories do not limit the

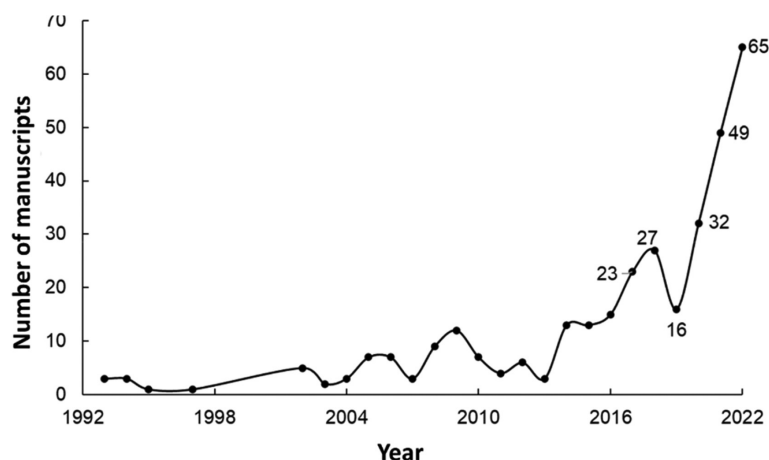


Figure 1. Publications containing the terms adsorbent, agricultural residues, and effluent treatment in the title, abstract and keywords: number of articles published per year.

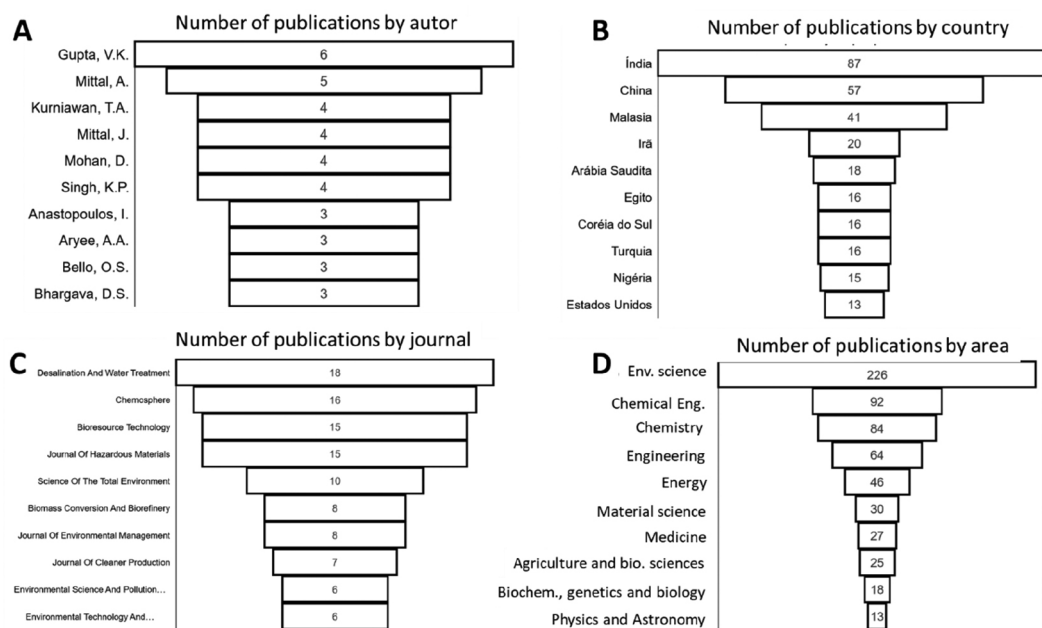


Figure 2. Bibliometric analysis of publications containing the terms adsorbent, agricultural residues and effluent treatment in the title, abstract and keywords: number of articles published by author (a), by country (b), by journal (c) and by study area (d).

articles to just one area, meaning that a publication can be related to several areas of knowledge.

Keyword analysis has gained special attention recently, as it can help to understand the evolution of studies in a given area and analyze the trend of the themes explored. Publications on the topic began in the 1990s, with eight articles published. The first three articles were published in 1993, in the same issue of the journal *Water Research*.^{22,23,24} The works investigated the potential for phosphate removal by activated carbon from tamarind nutshell, a low-cost and abundantly available material. The following year, three studies were published using other residues, namely activated charcoal from peanut shells to remove cadmium in effluents²⁵, coconut fiber to treat dyeing effluents²⁶ and pine bark pretreated with acidified formaldehyde solution to remove heavy metals Zn^{2+} , Cu^{2+} and Pb^{2+} .²⁷ Other articles

published in the 1990s were critical reviews on the use of soybean and cottonseed husk by-products, rice straw and sugarcane bagasse as adsorbents for metal ions in aqueous solutions²⁸ and different adsorbent materials used in the treatment of industrial pollutants, including low-cost residues.²⁹ In this decade, only seven keywords appeared in five articles or more, as shown in Figure 3.

In the 2000s, 55 articles were published, with 74 keywords presented in Figure 4. In this decade, in addition to critical reviews and experimental research, a wide range of agro-industrial residues began to be explored, with attractive determinants of low cost and High Availability. The most cited articles in this decade investigated the use of chitosan, biomass, bagasse, coconut husks, peanuts, almonds, wheat, soybeans, almonds, apricot seeds, cherry and ash, among other agricultural residues.³⁰⁻³⁵



Figure 6. Keywords used by the authors in articles about adsorbents made from agricultural waste for use in effluent treatment in the years 2021 and 2022.

3.1. Production methods

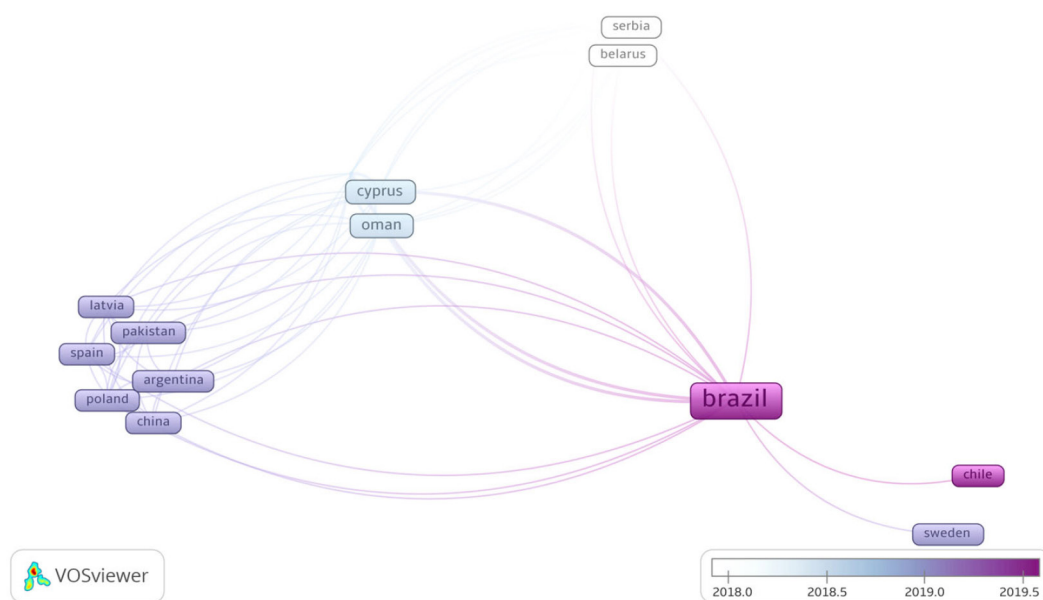
Affording materials from natural sources to manufacture adsorbents might be difficult due to the lack of selectivity towards pollutant specificities, which exhibit surface

Table 1. Articles on adsorbents made from agricultural residues for use in effluent treatment most cited by other authors

Title	Reference	Year	Journal	Citations
<i>Non-conventional low-cost adsorbents for dye removal: A review</i>	32	2006	<i>Bioresource Technology</i>	3508
<i>Application of low-cost adsorbents for dye removal - A review</i>	33	2009	<i>Journal of Environmental Management</i>	2851
<i>Adsorption of methylene blue on low-cost adsorbents: A review</i>	35	2010	<i>Journal of Hazardous Materials</i>	2248
<i>Single- and multi-component adsorption of cadmium and zinc using activated carbon derived from bagasse - An agricultural waste</i>	30	2002	<i>Water Research</i>	1024
<i>Adsorption of heavy metals on conventional and nanostructured materials for wastewater treatment purposes: A review</i>	41	2018	<i>Ecotoxicology and Environmental Safety</i>	866

Table 2. Articles on adsorbents made from agricultural waste for use in effluent treatment published by Brazilian authors

Title	Reference	Year	Journal	Citations
<i>Removal of Methylene Blue from an Aqueous Medium Using Atemoya Peel as a Low-cost Adsorbent</i>	48	2021	<i>Water, Air, and Soil Pollution</i>	2
<i>Investigation of Citrus reticulata peels as an efficient and low-cost adsorbent for the removal of safranin orange dye</i>	49	2021	<i>Environmental Technology (United Kingdom)</i>	9
<i>Potential of agricultural and agroindustrial wastes as adsorbent materials of toxic heavy metals: A review</i>	50	2020	<i>Desalination and Water Treatment</i>	6
<i>Agricultural biomass/waste as adsorbents for toxic metal decontamination of aqueous solutions</i>	51	2019	<i>Journal of Molecular Liquids</i>	96
<i>Evaluation of two different carriers in the biodegradation process of an azo dye 09 Engineering 0907 Environmental Engineering</i>	16	2019	<i>Journal of Environmental Health Science and Engineering</i>	3
<i>Efficient mercury removal from wastewater by pistachio wood wastes-derived activated carbon prepared by chemical activation using a novel activating agent</i>	52	2018	<i>Journal of Environmental Management</i>	81

**Figure 7.** Collaboration network between countries in articles on adsorbents made from agricultural residues for use in effluent treatment published by Brazilian authors.

functionalities of diverse types of molecules.⁵³ This practice, however, has expanded, mainly because several biomass-derived adsorbents present competitive adsorptive capacities, when compared to synthetic commercial products, because they are readily available, are morphologically advantageous and usually exempt pre-treatments, making their use not only practical but highly cost-effective.⁵⁴

Thus, depending on the source of biomass, proposed use and number of pollutants to be removed, these materials can be directly employed in their natural forms,^{55,56} or subjected to physical-chemical pre-treatments.⁵⁷ An example is active carbon, the most common adsorbent, which still presents difficulties in industrial applications in the wastewater treatment due to the inflated cost of the synthesis. On the other hand, production of biochar poses as an economically feasible alternative, especially considering the low emissions of GHG.⁵⁸ Taking that into account and bearing in mind the scenario of the main Brazilian agribusiness cultures suitable for adsorbent production, henceforth we present the central processes used in the manufacture of adsorbents:

3.1.1. Pyrolysis

Currently, pyrolysis is one of the most widespread methods in the literature⁵⁹ due to the versatility in transforming diverse lignocellulosic biomass through thermal decomposition using temperature gradient under inert atmosphere, producing: a carbon-rich residual solid rich named biochar; an organic liquid product made of water, acids, alcohols, aldehydes, ketones, esters, phenols and aromatic compounds known as bio-oil; and gas mixture formed by moisture, H₂, CO, CO₂, CH₄, which can also be used as energy sources.⁶⁰

Several authors have analysed pyrolysis process parameters to produce biochar, including temperature and decomposition time.⁶¹⁻⁶³ In general terms, temperature determines the degree of decomposition and carbonaceous content of the final charcoal, bio-oil and biogas products, and its raise may lead to higher contents in inorganic matter from the original raw material, and the formation of ashes.⁶⁰ Pyrolytic reactions are divided in: primary, generating products through the heat of biomass; secondary, which originate from the conversion of primary products; and tertiary, coming from the degradation of the last step products.⁶⁴ In addition, temperature directly influences the overall process cost, indication that, although low temperature might not provide a top-notch biochar, when too much heat is applied the reaction yield tends to be reduced and that restrains large scale production.⁶⁵

Time of pyrolysis, in turn, sorts it in fast, when there is a short gas residence time (few seconds), which essentially produces bio-oil, and slow, aiming to form biochar, where the lignocellulosic biomass is processed for hours.⁶⁶ Under temperatures ranging from 200 and 600 °C, it is possible to decompose raw material raising the degree of carbonization and pH of carbon content, while removing hydrogen, oxygen, nitrogen, and sulphate.⁶⁷

Using this methodology, many cultures are being assessed to generate porous structures to be used in contaminated water treatment. Interest in corn cob, due to the porous macroscopic structure and shortage of industrial applications, directed mainly to animal food, stimulate the search for sustainable adsorbents. Authors subjected the previously washed and dried cobs to pyrolysis at 550 °C under N₂ atmosphere for 2 hours, yielding a biochar that, although presenting relatively low surface area (8.99 m²/g), was able to adsorb up to 95% of heavy metals (lead and cadmium) at concentrations of 0.95 to 1.95 mg/mL.⁶⁸

Synthesized biochar from coffee grounds using pyrolysis at different temperatures (400, 600 and 800 °C) for 4 hours, obtaining an adsorbent material capable to quantitatively remove perfluorooctane sulfonate (PFOS).⁶⁹ Produced biochar through slow pyrolysis, 2 h, at 450 °C, from agriculture biomass – soy, corn and rice straw – to remove the herbicide atrazine, which is extensively used in pest control. The authors obtained materials bearing surface areas ranging from 17.5 and 25.8 m²/g, pore volumes from 0.08 to 1.19 cm³/g and alkaline pH, reaching adsorptive capacities up to 3 mg/g of pollutant.⁷⁰

Produced biochar from sugarcane bagasse, through slow pyrolysis (400 and 600 °C), to adsorb phosphate from an aqueous solution. The materials showed specific surface areas of 1.1028 and 1.8623 m²/g and reach 15 mg/g of adsorptive capacity. Authors added chitosan to the bagasse biochar surface (1:1), amounting to removal values of 20 mg/g with twice the specific surface area.⁷¹

Wheat is another popular culture regarding the application of pyrolysis to develop biochar for adsorption processes. Authors made biochar using slow pyrolysis (300 to 600 °C) and applied it to removal of heavy metal in contaminated water, achieving removals of 3.56 and 5.85 mg/g of mercury. The best results were accomplished using lower temperatures.⁷² Researchers studied the production of biochar, also by slow pyrolysis, to remove Cd (II), using five different plant raw materials: corn straw, cotton, wheat, rice and poplar. Authors obtained high surface materials under a carbonization temperature of 600 °C, varying from 37.7 m²/g with poplar biochar to 183.3 m²/g with wheat biochar, reaching adsorptive capacities of up to 500 mmol/kg.⁷³

3.1.2. Hydrothermal synthesis

Recent hydrothermal processes used to synthesize biochar, or hydrochar as they are known, have fomented interest of various researchers on the grounds of being a low-cost and environmentally friendly technology, since it does not release hazardous gas in the atmosphere.^{74,75} In this method, carbonaceous raw material is applied to a sealed reactor, which is heated to temperatures varying from 150 to 350 °C for up to 12 hours under 10 MPa of pressure (Román *et al.*, 2016)⁷⁶. Heating and pressure within the system generate a series of reactions, e.g., hydrolysis, dehydration, decarboxylation, demethanization and aromatization,⁷⁷ which, compared to conventional pyrolysis, yield materials

with higher carbon and oxygen content, presence of well-defined pores and acidic pH, at the range of 3 to 5.^{78,79}

The hydrothermal process is mainly influenced by the nature of the carbonized biomass, water content and temperature. High water content may accelerate carbonization, low temperature promotes the formation of biochar and steep heat favours the production of liquid and gas.⁸⁰

A great deal of industrial crops residues has already been used as raw materials for biochar hydrothermal synthesis, by evaluation of the combustion effects in the wheat straw biochar synthesis and observed that as temperature and pressure rise, hemicellulose content gradually diminishes, especially from 180 °C. For cellulose, as a result of the crystal structure, degradation does not start until 220 °C, whereas lignin content rose from this temperature forth, and also, starting at this temperature up to 260 °C, carbon microspheres begin to form on what was left of the biomass.⁸¹ In a study showing the application of hydrothermal synthesis to coconut peat and husk, egg shells, rice husk and lemon at 200 °C for 20 hours, authors managed to assemble highly porous adsorbents, with surface areas between 2.14 m²/g for coconut peat, to 21.8 m²/g for its shell. Compared to materials in their natural form, there was a significant increment, since their specific surface areas ranged from 0.09 to 1.23.⁸²

Authors compared the efficiency of sugarcane bagasse *in natura* and transformed in hydrochar in recovering phosphate from aqueous medium. The charcoal was synthesized at 230 °C for 1 hour, and the authors observed that carbonization opened pores on the structures which increase the phosphate removal by the 30% increase in removal related to *in natura*.⁸³ Less common residues were also evaluated, cassava husk hydrochar, for instance, showed a removal of the dye rhodamine B reaching 96%.⁷⁵ A collection of the most employed biomasses will be shown later on this work.

3.1.3. Treatments and activations

To achieve the necessary attributes that allow high adsorption yields in aqueous systems, materials require treatments aiming to improve both structural and functional properties.⁸⁴ This type of process, commonly known as pre-treatments or structural activation, removes unorganized carbonaceous structures, exposes lignin moieties to react with the activator, then interact with the porous walls, enhancing, in the first step, the mesoporosity of the material and in the second, its macroporosity.⁸⁵

Acid treatments are applied to the crude biomass when one focus on functional alterations in hydroxyl groups, esters and carboxylic acids which compose the natural raw material, because the low pH will reduce lignin content and break bonds between cellulose and hemicellulose, also lowering crystallinity until the material becomes amorphous.⁸⁶ The resulting effect in general is an enhancement in surface area and pore diameter, as observed authors who treated rice husk with H₃PO₄ and used it to

adsorb Cu (II). Microscopy images identified a significant pore enlargement, and infrared spectroscopy showed the presence of intense –COOH and –OH stretches, suggesting the exposure of the cellulose nucleus. These changes lead to 17.03 mg/g of adsorption capacity and 89% removal.⁶⁷

The aforementioned treatments, when applied to charcoal and biochar are named activating, as employed by authors Piriya *et al.* (2021), who manufactured biochar from coconut shells through slow pyrolysis, and promoted a sequential acidic activation with H₂SO₄ e H₃PO₄, leading to a porosity enhancement from 19.19 to 49.02%. The authors deduced this improvement is due to the spike in number of carboxyl, phenol and hydroxyl groups.⁸⁷

When the alkaline approach is the choice, natural carbonaceous materials have their morphological structure altered through lysis of lignocellulose, hemicellulose hydrolysis, lignin depolymerization and loss of fatty acids, which usually cover cellulose fibres, exposing their hydroxyl-rich core and increasing the adsorptive capacity of high-rugosity materials, enlarging the size of the pores and favouring electrostatic interaction between adsorbent and adsorbate.^{88,89}

Sugarcane bagasse was chemically treated with NaOH to degrade lignin and expose cellulosic content and used it to adsorb the herbicide glyphosate from aqueous medium. The authors observed that the procedure heightened adsorptive capacity by nearly 35% and the rate of adsorption by over 60%, attesting the feasibility of the chemical process.⁹⁰

Alkaline treatment (NaOH) was used on wheat straw biochar and compared it to acidic activations with HCl and HNO₃ to the same material. The alkaline treated material surpassed its acidic counterpart performance thanks to its large surface area and hydrophobicity, reaching removals of ofloxacin, tetracyclin and bisphenol-A as high as 95%. Electrostatic and hydrophobic interaction and filling of pores mechanisms are responsible for these results.⁹¹ Following this thread, other researchers developed rice husk biochar using KOH and obtained an adsorbent with a surface area of 1300 m²/g, corroborating the influence of the alkaline activator to the efficiency of the biochar.⁹²

Another way of chemical activation is the application of saline solutions (ZnCl₂, FeCl₂ etc) in order to reduce the temperature of thermal activation, focusing on the production of mesoporous biochar with high surface area.⁹³

Thermal activations are prevalent amongst natural biomasses and their biochar, given that they only require the material be subjected to heat under oxidating atmosphere, which causes the enhancement of physical, chemical and mechanical properties, due mainly to the process conditions and the character of precursor biomass, being capable of altering the surface composition, water content and even granulometry.^{94,95} For biochar, activating processes demand exposure to temperatures up to 700 °C for hours to achieve the desired surface modifications.⁹⁶

Biochar adsorbent was produced from wheat biomass using, amongst other techniques, thermal activation at

900 °C, reaching six-fold increase in surface area and pore volume.⁹⁷ An adsorbent from coffee grounds was synthesized through a thermal treatment aiming to adsorb diesel oil. The authors observed that heating the grounds to 300 °C lead to increments in specific surface area, hydrophobicity, thermal stability, crystallinity, and adsorptive capacity to diesel of circa 50%.⁹⁸

3.1.4. Natural Biomass

In face of the cost involved in conditioning, production and activation processes of natural adsorbents, many authors suggest the application of biomass as adsorbents without any previous treatments.^{2,99} Besides the low cost linked to elevated availability, good selectivity to a number of pollutants, natural biomasses, especially those without inherent added value, become competitive related to commercial adsorbents.¹⁰⁰

Residues from important agricultural cultures, cotton stalk among them were used to test their biosorbent abilities, without any pre-treatment, towards removal of heavy metals from water media, with positive results from lead and copper.¹⁰¹ Recently, solely washed soybean husks were used to remove plantation field impurities, and subsequent drying and milling. The material was applied to remove reactive blue dye, with adsorptive potential of 53.33 mg/g and removal of 88%.¹⁰²

Another recurrent Brazilian native residue, the cassava bagasse, was evaluated without pre-treatment, in the removal of scarlet red dye from water. Data showed a material with low surface area micropores (3.01 m²/g), which, nevertheless, was able of removing 84% of the dye under acidic conditions with approximately 25 mg/g of adsorptive potential, putting this biomass among the promising sources for adsorbents.¹⁰³

Sugarcane bagasse was also tested as adsorbent *in natura*, varying only the granulometry of the material. The structures obtained presented similar adsorptive capacities to methylene blue dye, close to 4 mg/g, regardless the initial concentration, reaching equilibrium between 5 and 7 hours. In this study, the authors indicate that sugarcane bagasse could be in a range of 20 to 45 mesh, with no significative difference in adsorption, hence, with a minute cost because of the reduction in processing times.¹⁰⁴

Although the large-scale application seems yet far in the future, popularization of biosorbents *in natura* have augmented, largely because several agriculture residues have demonstrated potential adsorptive characteristics, added to the sustainable and environmental appeal that the correct use of this sort of material promotes.¹⁰⁵

3.2. Characterization of natural adsorbents

In order to assure properties befitting the application of an adsorbent, it is necessary to subject the pre-treated materials, or yet, the natural biomass to characterization measurements, to seek the identification of structural and

functional elements required to attest that the material might be an adsorbent,¹⁰⁶ and to what kind of pollutant they would efficiently interact with. The nature of these interactions rises great interest in the field, since the mechanistical explanations are responsible for extrapolating the use of these products to other contaminated water scenarios, including multiple component matrices of pollutants.¹⁰⁵

On the subject of adsorbents of natural origin from agricultural crops, analyses might show characteristic elements pertinent to this class, and, depending on the treatment that the biomass received, one can observe specific parameters that will help to identify if said treatment was the driving motor to arrive at these characteristics. Among the frequently methods applied in material characterization, it is possible to highlight the most relevant:

3.2.1. Scanning Electron Microscopy (SEM)

This is technique features among the most widely explored to access solid surface, morphology and shape, and, to adsorption, it allows the elucidation of the adsorbent structures in terms of particle shape and size, presence and organization of pores and rugosity, i.e., elements that contribute to filling and adhesion of particles in the surface.²

For agriculture-derived materials in their natural forms, it is possible to stablish structural common patterns of interstitial layers, fibre arrangements, high rugosity amorphous granules, and pores distributed over several sizes.¹⁰⁷ Authors have compared scanning electron micrographs of corn straw before (Figure 8a) and after activation with HCl (Figure 8b). The acid promoted a surface decomposition, fomenting the opening of cavities with an average diameter of 10 µm, which is auspicious for adsorption processes.¹⁰⁸

Jawad *et al.* (2020)¹⁰⁹ also used SEM to observe the structural alterations to corn straw biomass caused by acidic activation. However, in addition to identifying the pore opening after activation (Figure 9a), they noted, in the micrographs of the material after the adsorptive process (Figure 9b), a complete closure of the pores, suggesting a complete material covering and an elevated morphological influence in the adsorption.

Other authors have evaluated surface properties of natural biomasses using SEM, as done by Frollini *et al.* (2013),¹¹⁰ who analysed structures of sugarcane bagasse, showing this material has a surface organized in hemicellulose bundles with a cellulosic interior arranged in hollow tubular structures with variable diameters. Chen *et al.* (2013) identified accumulated lamellar structures form the walls of wheat and maize straw, with a capillary framework within the lamellas that might actively contribute to adsorption.¹¹¹

In the form of biochar, authors have indicated that such materials tend to form different rugosity degrees. When subjected to activation processes, there are usually hemicellulose residues clustered within the pores, accumulation gradients of residues, and larger specific surface areas and wider pores.⁸³

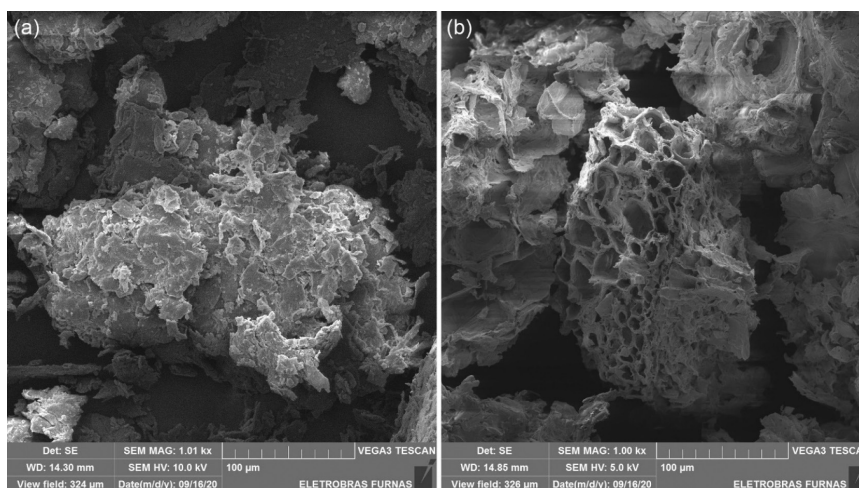


Figure 8. (a) SEM of corn straw without pre-treatments; (b) SEM of corn straw treated with HCl.¹⁰⁸

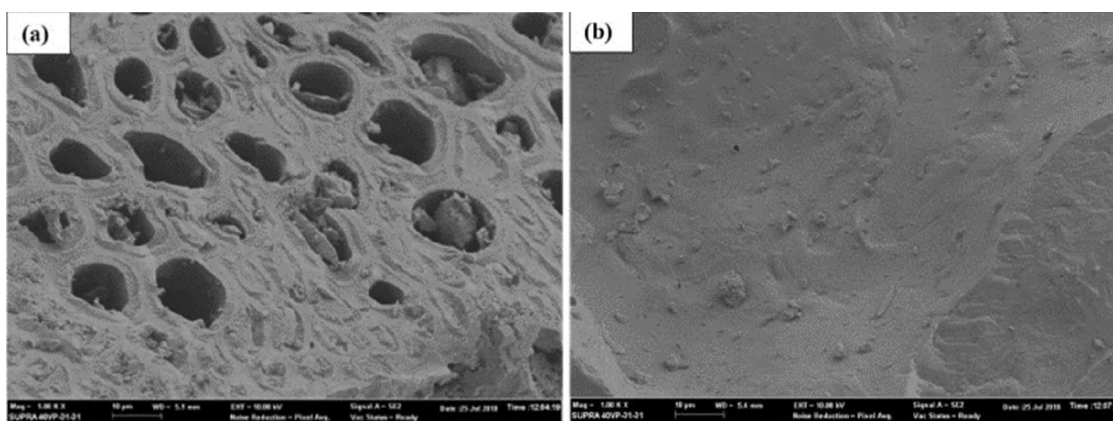


Figure 9. (a) SEM of corn straw treated with H_3PO_4 ; (b) SEM of corn straw treated with H_3PO_4 covered with methylene blue dye after the adsorptive process.¹⁰⁹

3.2.2. Fourier Transform Infrared Spectroscopy (FTIR)

From the molecular vibration response after being blasted with radiation in the infrared frequency, it is possible to obtain a functional groups signature of the adsorbent surface, aiding to identify likely mechanisms of interaction between materials and pollutants. According to Furlan *et al.* (2008), who evaluated IR spectra of corn, wheat and soybean straws, in natural biomass there is nearly a standard spectrum to be expected, constituted of OH stretches ($3100\text{--}3500\text{ cm}^{-1}$) from moisture and interstitial water content, C=O signals around 1633 cm^{-1} , O-C-O at 1400 cm^{-1} and C-O-C at 1045 cm^{-1} , representing the cellulosic structure.¹¹²

Golveia *et al.* (2021) evaluated corn stalk and identified OH stretches at $3000\text{--}3700\text{ cm}^{-1}$ and C-C-C bends at $1100\text{--}850\text{ cm}^{-1}$, proving the cellulosic nature of the material and suggesting favourable interactions with polar pollutants.¹⁰⁸ Kamsonlian *et al.* (2012) showed similar functional groups in banana peels.¹¹³

Thawornchaisit *et al.* (2021) worked with raw sugarcane bagasse, and biochar from the same biomass, and show the existence of hydroxyl groups from moisture and Csp³-H stretches, suggesting the presence of aliphatic compounds.

Carbonyl groups, pointing out to hemicellulose, appeared only in the crude bagasse, which is expected in face of the thermal degradation that takes place during carbonization. At the $1100\text{ to }1500\text{ cm}^{-1}$ range, where cellulosic and aromatic signals can be found, only biochar showed vibrational signals, confirming the exposure of cellulosic core of the material.⁸³

Mariana *et al.* (2018) used FTIR analyses of coffee bean residues *in natura* and after acid activation with HCl. The authors identified Csp³-H groups at 2905 cm^{-1} , OH and carboxylic acids with a large band between $3550\text{ and }2400\text{ cm}^{-1}$ and C=O from esters at 1755 cm^{-1} . Prominent C=O and COOH stretches suggest the acid treatment degraded the hemicellulose, exposing lignin and cellulose.¹¹⁴ Similarly, Guo *et al.* (2016) observed that when wheat straw is treated with an alkali (e.g., NaOH) C-O stretches were augmented, leading to an increase in Cu (II) adsorption through interaction with ether groups.¹¹⁵

In the same field, Souza *et al.* (2021) also evaluated FTIR spectra of biochar from açai seeds. The biochar samples were activated via acid (H_3PO_4) or alkaline (NaOH) processes. The result suggested that both treatments remove vibrational bands at $1773, 1685, 1570\text{ and }143\text{ cm}^{-1}$, which represent

the decomposition of oxygenated moieties within cellulose and hemicellulose. FTIR data also indicated the presence of acids, phenols, ethers and esters, once again pointing to an exposure of the cellulosic matrix post-treatment. In addition, functional groups bearing phosphorous were also present in the H_3PO_4 product, e.g., the $\text{P}=\text{O}$ stretch at 1261 cm^{-1} . The authors have shown that treated açai biochar has a predominantly acidic surface, favouring the adsorption of pollutants with an alkaline character.¹¹⁶

3.2.3. X-Ray Diffraction (XRD)

According to Anirudham and Unnithan (2007), X-ray diffractogram of natural adsorbents of cellulosic character, exemplified by their studies with coconut fibres, essentially correspond the presence of scattered angles at 21.2° , 38.2° e 43.5° , representing an orderly array of cellulose molecules.¹¹⁷

Similar patterns were observed by Ajala *et al.* (2022) when they evaluated sugarcane bagasse both *in natura* and chemically treated as adsorbents. Characteristic peaks were measured at 14° , 21° and 28° , with a higher intensity for the 21° signal, indicating the unfolding of the cellulosic structure in a crystalline material.¹¹⁸ Harripersadth *et al.* (2020), studying the same biomass, encountered peaks at the 22° range referents to native cellulose with plane (002) present. The authors, however, demonstrate that the presence of lignin, an amorphous material bound to cellulose, reduces the intensity, or withdraw the sharp form of peaks of the biomass.¹¹⁹ Jawad *et al.* (2018) evaluated banana peel and detected peaks at 24° (002) and 42° (101), indicating this biomass presents an analogous cellulosic crystalline structure.¹²⁰

Biochar adsorbents are usually formed of amorphous structures and their diffractogram do not show well-defined peaks, they show though broad bands in the 2θ region between 22 and 25° ,¹⁰⁹ due to the remains of cellulose not degraded during the thermal process. A similar pattern was assessed by Papala *et al.* (2021),¹²¹ who produced biochar from rice husk and identified a large reflexion peak at $2\theta = 23^\circ$ (002), related to the biomass *in natura*, which formed a sharper and closed peak ($2\theta = 23^\circ$ (002), suggesting the degradation effect the biochar production brings about, becoming more amorphous due to the lignin residual content. Karami *et al.* (2022) identified $2\theta = 22-28^\circ$ and $2\theta = 42^\circ$ for biochar from corn stalks, suggesting an amorphous carbon structure formed by $\text{C}_6\text{-C}_{60}$ e graphite befitting the scorching temperature applied to producing this biochar.⁹⁷

3.2.4. Thermal analysis

Thermogravimetry, (TG) with Derivative Thermogravimetric analysis (DTG), and Differential Scanning Calorimetry (DSC) are the most prevalent thermal analyses. Golveia *et al.* (2021), working with corn stalk residues, observed, using thermogravimetry, that lignocellulosic materials tend to have three mass loss

stages. TG and DTG both indicate that the mass loss at 100°C is mainly associated with free water enclosed in the crop-derived material. Hemicellulose decomposes at around 275°C and cellulose, which presents higher heat resistance, starts decomposing at 300°C . Through DSC analysis, Golveia *et al.* (2021) also observed an exothermic spike that start at 350°C and peaks at 450°C .¹⁰⁸

Vieira *et al.* (2012) analysed natural and thermally activated (calcination) rice husks as adsorbents. TG/DTG measurements suggested a mass loss of 6% between 48 and 90°C , indicating the presence of volatiles and water in the system, and another loss of 54% at 350°C – degradation of hemicellulose and most of cellulose. Finally, at approximately 600°C , lignin starts decomposing until there is nothing but ashes. Calcinated samples showed mass losses of only 2.5% from 25 to 150°C , referent to the remaining free water – which was expected since the volatiles and most of the water were removed during thermal treatment – and another 22% mass reduction due to interstitial water and then carbonization. DSC analyses of rice husk showed energy absorptions at 77°C and 300°C , implying the evaporation of volatile and non-volatile organic compounds, respectively.¹²²

Other farming cultures also exhibited similar thermal behaviours. Awokoya *et al.* (2016) evaluated the cassava husk structure, noticing 5% of mass loss until 100°C , but a surprising 80% loss from 300 to 390°C , due to the degradation of carboxylic compounds.¹²³ And Verma *et al.* (2021), working with coconut shell residues, likewise observed an 80% mass loss from 170 and 415°C , denoting in both cases that lignocellulosic composition of the material determines the rate of mass loss within the cited ranges. In Verma and co-workers' (2021) case, the adsorbent lost 3% of hemicellulose, 54% of cellulose and 30% of lignin from 170 to 415°C . And, as expected, hemicellulose degraded first, followed by cellulose and, lastly, lignin decomposed.¹²⁴

3.2.5. Adsorption-desorption of Nitrogen at 77 K

Adsorption-desorption of N_2 at 77 K helps to identify the porous character of a system, as well as to calculate the surface area and porous volume and diameter, alongside with BET and BJH techniques.⁷³ Regarding adsorbents from agricultural biomass, Bavaresco *et al.* (2021) observed that corn straw bears a low surface specific area when in the natural form, $2.662\text{ m}^2/\text{g}$, and has a mesoporous structure with total pore volume of $0.0066\text{ cm}^3/\text{g}$. After thermal activation via calcination, on the other hand, the authors showed both surface area ($109.4\text{ m}^2/\text{g}$) and porous volume ($0.075\text{ cm}^3/\text{g}$) enhancements, becoming more competitive against commercially available active carbons.¹⁰⁷ Surface areas and porous volume in biochar are inversely proportional to the lignin content in the precursor biomass, corroborating the previous data that show the deterioration of lignin during activation or carbonization might improve the adsorbent character of agrarian residual material. Table 3 shows a collection of surface areas of different crops and their biochar applied to adsorptive processes.⁷³

Table 3. Surface area and porous volume of adsorbent materials from agrarian origin

Culture	Surface Area (m ² /g)	Pore Volume (cm ³ /g)	Reference
Soybean Husk	< 1	-	125
Soybean Biochar	1425-1620	0.638-0.751	126
Corn Stalk	1.22	-	127
Corn Stalk Biochar	407	0.155	128
Coffee Grounds	0.19	-	129
Coffee Grounds Biochar	388.4 - 987.9	0.528-0.984	130
Rice Husk	1.4	-	122
Rice Husk Biochar	13.6	0.0076	131
Cassava Husk	2.051	0.002233	132
Cassava Stem Biochar	6.882 - 9.4964	0.00237 - 0.00247	133
Beans	179.1	0.036	134
Bean Biochar	-	-	-
Banana Peel	3.089	0.004977	132
Banana Peel Biochar	1.2 - 7.5	-	135
Wheat Straw	4.6 - 5.9	0.745	136
Wheat Straw Biochar	15.8	-	137
Coconut Fibre	38.84	0.436	138
Coconut Fibre Biochar	331.86-811.46	-	139
Açaí	-	-	-
Açaí Seed Biochar	1.94 - 491.90	0.003 - 0.315	140
Cocoa Husk	0.021	-	141
Cocoa Husk Biochar	118 - 502	0.09-0.50	142
Manga Peel	13.455	0.055	143
Manga Peel Biochar	2.09 - 5.74	0.00183 - 0.00366	144

The data shown in Table 1 are a sign of the viability of adsorptive application of natural biomass from agricultural origin, considering their porous structures, especially biochar. These products are competitive facing commercial active carbons, since they have specific surface areas at the range of 1500-2000 m²/g and porous volumes around 0.7 cm³/g,¹⁴⁵ making the materials not only economically viable related to the cost of traditional active carbons, but also readily available.

4. Adsorbents Produced using Important Crops from Brazilian Agribusiness

Agribusiness market moves billions of dollars a year – Brazilian 2021 harvest alone generated a revenue of R\$ 651.75 bi (IBGE, 2021)¹⁴⁶ – besides creating jobs and strengthening the economy, especially in the poorest regions of the country. Out of this number, 83.4% are owed to ten cultures: soy, wheat, sugarcane, coffee, cotton, rice, orange, cassava, beans and banana. Besides these, based on the 2021 harvest data (Table 4), wheat, coconut, açaí, mango and cocoa cultures are prominent amongst the national crops.¹⁴⁷

Planted areas and quantities harvested of the cultures highlighted above (Table 4) draw attention to a factor that largely impacts the economy and, withal, the environment: the number of solid residues generated during and after harvesting these vegetables.

Taking soybean as an example, despite the absence of continuous data collection on the tonnage of residues produced every year, one can use the harvest index

(HI = mass of grain produced over total dry mass of the plant) to suggest the quantity of “garbage” left at the end of the season. In a recent work, Jiang and co-workers (2019) found HI values between 0.14 and 0.60, averaged at 0.42 for soybeans cultivated in the Northeast of China. If Brazilian agriculture yields a similar index, the bulk of solid waste here would be around 321 million tons.¹⁴⁸

This alarming number supply of material should not simply be discarded in the environment. For some important cultures, the wastes are already reused within the agroindustry itself. Sugarcane, for instance, is widely used as source for bioenergy and biomaterials.¹⁴⁹⁻¹⁵¹ There are yet abundant biomass that goes to waste in the agrarian sector. This plant-based biomass, constituted mainly of lignocellulose and hemicellulose as already discussed, may be sustainably utilized to develop high-value products, among which, feature the biosorbents.¹⁵²

The following topics discuss the major types of adsorbents produced using some of the most important Brazilian agricultural crops – soy, corn, coffee, rice, cassava, beans, banana, wheat, coconut, açaí, coca and manga – and their application in wastewater treatment.

Table 4. Numbers of planted area, quantity harvested (in tons) and revenue of some of the main Brazilian agricultural crops during the 2021 harvest

Culture	Planted Area (Hectare)	Harvest (Tons)	Revenue (Millions Reais)
Soy	39,185,745	134,934,935	341,747,600
Corn	19,587,069	88,461,943	116,396,867
Coffee	1,836,741	2,993,780	34,896,546
Rice	1,689,189	11,660,603	19,146,736
Cassava	1,205,829	18,098,115	12,702,124
Beans	2,613,086	2,899,864	12,049,373
Banana	453,273	6,811,374	9,998,070
Wheat	2,750,264	7,874,525	10,998,648
Coconut	186,392	1,638,573*	1,299,188
Açaí	208,111	1,485,113	5,305,523
Cocoa	600,789	302,157	3,973,400
Mango	76,061	1,505,372	1,953,638

Source: IBGE - <https://www.ibge.gov.br/explica/roducao-agropecuaria/>. *Quantity of fruit

4.1. Soy

Soy is the second Brazilian agrarian commodity in terms of quantity of residues generated, surpassed only by sugarcane.¹⁵³ Grain husk and the straw are the main source for adsorbent materials obtained from this culture. The straw, a lignocellulosic material originating at the stalk, is abundant and unexpensive, but, due to their low-nutritional value, it is frequently discarded or burnt. The husk, on the other hand, corresponding to 8-10% of the total mass of grain, is a by-product of plant processing, especially for oleochemistry industry.¹⁵⁴

Soy straw has a surface teeming with pores of various sizes, with opening diameters ranging from nanometres to micrometres. This allows, for adsorbents produced with this biomass, the adsorption of molecules, being organic or inorganic (e.g., metals) in different regions of the material.¹⁵⁴

Pyrolysis stands out as a method to produce adsorbents from soy straw. Several examples of soy biochar can be found in the literature being used to treat effluents containing textile dyes,¹⁵⁵ basic and anionic compounds¹⁵⁶ and metallic cations, such as chromium¹⁵⁷ and cadmium¹⁵⁸. Products from this plant matrix are not limited to biochar, straw – both *in natura* and chemically treated – have been used in the treatment of effluents rich in Cu²⁺¹⁵⁴ and the active carbon – activated with ZnCl₂ – of the same source was efficient in the treatment of coloured effluents.¹⁵⁹

Soybean husk is, however, underused. Despite the high quantity of polysaccharides, especially cellulose, only around 10% of the husk is destined to animal nutrition, a great deal of the overplus ends being discarded or incinerated.¹⁶⁰ This plant possesses interesting characteristics – dilation capacity, porosity, surface area – that concomitantly favours the adsorption of molecules both hydrophobic and hydrophilic.¹⁶¹

The main effluents treated with soybean husk adsorbents originated from the pharmaceutical and, principally, textile industries. Recent studies have demonstrated the efficacy

of this biomass when applied to the remediation of residual waters contaminated with hormones,¹⁶² non-steroidal anti-inflammatories¹⁶⁰ and antibiotics.¹⁶³

Dyes are also important pollutants, prevalent in diverse industries. Beyond the obvious role in textile factories, they are widely explored in the production of plastics, paper, medicines, and cosmetics, to cite only a few, which consume enormous volumes of water, generating vast quantities of coloured effluents.¹⁶⁴ Soybean husk products are being extensively studied as suitable agents to address this issue. The list of dyes treated is ample, but we can point out the use of this biomass to adsorb direct red 80 and 81, acid blue 92 and acid red 14,¹⁶⁵ safranin T, Remazol brilliant blue and direct violet 51,¹⁶⁶ reactive yellow 145 and reactive blue 21,¹⁶¹ methylene blue¹⁶⁴ and reactive red 4B.¹⁶⁷

4.2. Corn

With a revenue of over 116 billion reais in the 2021 harvest,¹⁴⁶ corn ranks second among Brazilian cultures that generate wealth in the country, surpassed only by soy. A cornerstone for this result is the impressive adaptability of this crop to the most diverse climate conditions: maize is cultivated from south to north, in both humid and arid regions.¹⁶⁸

Residues assembled from different corn parts are employed in the production of adsorbents, the more fruitful are the: cob, stalk, straw and bract. The active carbon manufactured from corn cob proved to be a versatile material, it was suitable for removing metals from water in lab-scale tests,^{169,170} in addition to being successfully used to treat effluents containing amino acids¹⁷¹ and dyes.¹⁷² The cob is also a source for nanocomposites which demonstrated high adsorptive capacity for metals, such as lead¹⁷³ and cadmium.¹⁷⁴

Corn stalk is another waste product of the maize industry produced in exuberant amount. It is constituted mainly of cellulose, hemicellulose, lignin and SiO₂,¹⁷⁵ which makes

it attractive as a source of adsorbents. Products made from the stalk have been applied to the remediation of metal solutions, both *in natura*, to adsorb Pb (II),¹⁷⁶ and modified with amines, to remove Cr (VI)¹⁷⁷ and Cd (II).¹⁷⁸ Biochar derived of the same biomass presented remarkable results in remediation of coloured effluents¹⁷⁹ and ionic liquids.¹⁷⁵

Likewise, corn straw is also a rich biomass source commonly incinerated and, generally underused.¹⁸⁰ Straw structure is irregular and porous, with large surface area, favouring the physical adsorption of pollutants. Its chemical composition is rich in carbohydrates, lignin and proteins, with an elevated ion coordination capacity.¹¹¹ The combination of these characteristics boosts the efficiency in the adsorption of organic – green malachite dye,¹⁸⁰ ethers and benzene¹⁸¹ and atrazine¹⁸² – and inorganic compounds – cations such as Cr³⁺ and Cr⁶⁺.¹¹¹

4.3. Coffee

Coffee is one of the most consumed beverages in the world. The culture, which has a central role in Brazilian history and politics for the past three centuries, continues to be lucrative to farmers, raising almost 35 billion reais in the 2021 harvest.¹⁴⁶ Nonetheless, with the rise of worldwide production, there a proportional increase in residues. The main remnant products of the coffee industry are the grounds, silverskin and husk.¹⁸³

Coffee grounds is a small-sized particulate residue, high in moisture and organic content, mildly acidic, obtained after preparing coffee.¹⁸³ Colossal amounts of grounds are discarded per year, the residue mass corresponds to approximately 65% of grain.¹⁸⁴

Adsorbents synthesized using coffee grounds as source material are applied to the remediation of a diverse group of effluent, since long-established contaminants, such as pharmaceuticals¹⁸⁴⁻¹⁸⁶ and metallic cations¹⁸⁷ to complex mixture of organic remains, found in mine leachates¹⁸⁸ and landfills.¹⁸⁹

Silverskin is a by-product of the coffee beans roasting. This fine membrane which covers the grain comes off during the heating process and is the only by-product originated at this stage of the productive chain. Its structure is predominantly constituted of food fibre – rich in carbohydrates –, proteins and a few minerals. The inadequate disposal of this biomass causes serious environmental problems: silverskin has a high content of methylxanthines, polyphenols and tannins, which renders an elevated phytotoxicity.^{183,190}

The use of silverskin as adsorbent is recent, but it has increasingly attracted attention in the last decade. Ismail and co-workers (2017) have shown that silverskin nanoparticles are able to remediate used cooking oil.¹⁹¹ And Pozo *et al.* (2021) used the biomass-derived biochar to treat coloured effluents.¹⁹⁰ The *in natura* product also presented high adsorptive capacity, when employed to remove Cu²⁺, Zn²⁺, Ni²⁺¹⁹² and Cr³⁺ e Cr⁶⁺¹⁹³ from contaminated water.

Coffee husk, constituted of the external dry skin, pulp and parchment, is a major residue that remains after harvesting and processing of coffee, and makes up nearly 1/4 of the grain mass.¹⁹⁴ Husk-derived biochar is particular attractive. To obtain a good yield in the production of the biocharcoal it is crucial to collect a biomass rich in cellulose, lignins and ashes, these components represent 84% of the husk.¹⁹⁵

Active carbon produced using coffee husk has been extensively explored to treat coloured effluents, containing, e.g., methylene blue,^{194,196} methyl violet and pararosaniline¹⁹⁷ and malachite green.¹⁹⁸ This sor of adsorbent is also able of removing ions from residual waters¹⁹⁹ and pharmaceuticals from biological samples.²⁰⁰

4.4. Rice

Brazilian production of rice in 2021 surpassed 11.6 million tons.¹⁴⁶ During the processing of this gigantic quantity of grains, the rice husk is commonly discarded. This product, which represents around 1/5 of its mass,²⁰¹ is comprised of around 20% of SiO₂,²⁰² making it a promising candidate as source for adsorbent materials.

This significant amount of silicon dioxide enables the synthesis of a type of adsorbent that is not usually produced from organic matter. Silica derived from rice, with and without the addition of iron, was used to remediate coloured effluents.²⁰³ For the remediation of pharmaceuticals, Pham and co-workers (2021) synthesized hybrid nanoparticles (SiO₂ and CeO₂) and used them to remove amoxicillin from residual waters.²⁰⁴

Rice husk is largely employed to adsorb metals from water solutions. Materials manufacture from this part of the grain showed to be effective to adsorb Cu²⁺, Al³⁺, Ni²⁺ and Zn²⁺ and Pb²⁺, Cu²⁺, Cd²⁺, in addition to binary mixtures of these cations.^{201,205} The versatility of this biomass is not limited to inorganic ions: hydrolysed hush, straw and active carbon from rice are excellent adsorbents when applied to organic compounds, such as 2-nitrophenol,²⁰⁶ the antibiotics azithromycin and erythromycin²⁰⁷ and the analgesic paracetamol.²⁰⁰

4.5. Cassava

Cassava plan is a tubercle native of South America which is part of the diet of around 700 million people worldwide, especially in the developing countries. Brazilian harvest represents 10% of the global production.⁵ Plantations are distributed for all states and the cultivation takes place at both massive estates and at small farms.

The culture yields colossal quantities of solid residues, principally throughout root processing. Among the generated wate, cassava peel has a notorious position, it constitutes 3 to 5% of the root weight,²⁰⁸ which, considering the 2021 harvest,¹⁴⁶ leads to estimate ranging from 543 to 905 ton of peel produced only in Brazilian land.

The use of casava residues as raw material for adsorbents is novel, there are only a few groups working with this biomass. Despite that, the performance of the products already produced from the peel and the root bagasse foresees an auspicious future in the field, especially regarding the application in the treatment of effluents containing metallic cations, such as Cd^{2+} , Pb^{2+} , Cr^{3+} and Ni^{2+} .²⁰⁸⁻²¹⁰

4.6. Beans

Beans are farmed in all five regions of the country. Brazil is the largest producer in the world, the 2021 harvest yielded over 12 billion reais.¹⁴⁶ Paraná, Minas Gerais and Bahia states have the most massive farms and produce circa of 50% of beans reaped in the national territory.²¹¹

After reaping and threshing the grain, around 60% of the plant becomes waste, and Brazil is responsible for 5.7 million tons of beans per year.²¹² In an attempt to reduce the impact of the waste, and further obtain products with significant added value using materials that otherwise would be garbage, the bean pod and husk have been surveyed as precursors to the synthesis of adsorbents.

The number of studies reporting the use of this biomass *in natura* is modest, most likely because the crude material presents low adsorptive capacity.²¹² Yet, Bayomie and co-workers (2020) showed that, under ultrasound assisted stirring, bean husk biomass in the natural state can remediate coloured effluents.²¹³ Besides that, the biochar, manufacture using both pod and husk, was successfully utilized to adsorb organic compounds, such as naphthalene and the dyes methyl orange, methylene blue, Congo red and malachite green.^{214,215}

A fascinating alternative to amplify the biomass adsorptive capacity is the collection of chemical treatments previously discussed. Sá and co-workers (2022) showed that the acid activation of the bean pod with HNO_3 lead to a product with considerable affinity to fluorescein, a property which only aroused after the chemical treatment.²¹² For the removal of inorganic compounds, Raulino and co-workers (2018) described a 45% increase in the removal rate of Cu^{2+} , Zn^{2+} , Ni^{2+} , Pb^{2+} and Cd^{2+} , after the pod was subjected to an alkaline treatment followed by the addition of organic acids.²¹⁶

4.7. Wheat

Wheat is one of the most consumed cereals in world. The culture represents 8% of all vegetable reaped on the planet and, with 800 million tons harvested, ranks fourth in worldwide production, behind sugarcane, corn and rice.⁸ Wheat sales yielded nearly 11 billion reais in the 2021 Brazilian harvest.¹⁴⁶ The husk, straw and bran are main residues gathered during wheat harvesting and processing.^{217,218}

The wastes of the wheat culture, quite similarly to the biomasses discussed so far, are comprised mainly of

cellulose, hemicellulose, lignin and other carbohydrates.²¹⁸ Therefore, the methods used to prepare adsorbents detailed in the previous section also apply to this biomass.

Wheat husk, after acid activation with perchloric acid, showed remarkable adsorption properties to remediate coloured effluents.^{219,220} The wheat straw and bran *in natura* originated products able to adsorb lead²²¹ and chromium.^{111,222} The biochar produced with these biomasses also shows high adsorptive capacity towards metallic cations.^{223,224}

Table 5 summarizes the different types of adsorbents produced using biomass residues accumulated after harvesting and processing of the main cultures of the Brazilian agribusiness.

5. Application in Real Wastewater Treatment and Perspectives

A great deal of new adsorbents is emerging in the last years, with special attention to reuse waste. Among the valuable wastes that can be used as adsorbents, agricultural residues have shown huge potential due its diversity, amount generated and specially regard to the features these natural materials. During the preparation of this work it was possible to notice that besides the high number of different materials been studied, still existing a lack of works applying these materials on real wastewater. Therefore, the authors want to point out the necessity to amplify the use o agricultural base materials and consequently stimulate the transference of the technology from lab scale to industrial applications. For this, it will be shown a group of results using the agricultural cultures presented above which were applied to real contaminated water.

For instance, in order to verify the efficiency of the soy waste biomass as adsorbent to remove heavy metals from water, Bulgariu and Bulgariu (2018) tested batch system and industrial wastewater from a printing company. The removal of $\text{Pb}(\text{II})$, $\text{Cu}(\text{II})$ and $\text{Ni}(\text{II})$ ions was investigated and an economical study was carried out. The authors have achieved a high removal potential and a lower cost compared with the precipitation method usually applied by the company.²²⁵

Additionally, Ma and co-workers (2019) transformed corn straw in a highly efficient porous adsorbent used to remove chromium (VI) from water. As can be seen on Figure 10, the thermal activation has transformed the waste material promoting the porous and revealing functional groups on the surface of the material that have chemical activity to attract and trap the pollutant studied. Figure 10. Preparation of the adsorbent and suggestion mechanism for chromium (VI) removal. It was observed a removal efficiency of 97.90%, even in presence of Fe^{2+} , Ni^{2+} and Cu^{2+} , no change was observed in the adsorption efficiency. These results indicated that although the existence of Fe^{2+} exhibits a competitive effect, the porous carbon adsorbent still presented excellent removal of Cr (VI) from electroplating wastewater.²²⁶

Table 5. Adsorbents produced using solid residues from the main Brazilian agricultural crops and the types of effluents they remediate

Culture	Part of the plant	Type of adsorbent	Effluents/residues treated	References
Soy	Straw	<i>In natura</i> and Chemically activated Biochar/Active carbon	Cu ²⁺	154
			Methyl violet Ammonia, nitrate and phosphate	155
			Cr ³⁺	156
			Cd ²⁺	157
			Brilliant red K-2BP	158
	Husk	<i>In natura</i> and Chemically activated		159
			Hormones	162
			Non-steroidal anti-inflammatories	160
			Antibiotics	163
			Dyes	165
Corn	Cob	Chemically activated Biochar/Active carbon	Pb ²⁺	166
			Cd ²⁺	173
			Cr ⁶⁺	174
			Amino acids	169
			Dyes	171
	Stalk	<i>In natura</i> Chemically activated Biochar/Active carbon	Cu ²⁺ e Ni ²⁺	172
			Pb ²⁺	176
			Cr ⁶⁺	177
			Cd ²⁺	178
			Dyes	179
Coffee	Straw	<i>In natura</i> Chemically activated Biochar/Active carbon	Ionic liquids	175
			Cr ³⁺ e Cr ⁶⁺	111
			Dyes	180
			Methyl-t-butyl ether and benzene	181
			Atrazine	182
	Grounds	<i>In natura</i> Biochar/Active carbon Chemically activated	Mine leachate	188
			Cr ⁶⁺	187
			Tetracycline	186
			Balofloxacin	184
			Landfill leachate	189
Rice	Silverskin	<i>In natura</i> Biochar/Active carbon Chemically activated	AAS, paracetamol e caffeine	185
			Cu ²⁺ , Zn ²⁺ , Ni ²⁺	192
			Cr ³⁺ and Cr ⁶⁺	193
			Dyes	190
			Used cooking oil	191
	Husk	Biochar/Active carbon Chemically activated	Dyes	194
			NH ₄ ⁺	197
			Paracetamol	199
			Dyes	200
				198
Beans	Husk	Chemically activated Biochar/Active carbon Modified silica	Cu ²⁺ , Al ³⁺ , Ni ²⁺ and Zn ²⁺	201
			Pb ²⁺ , Cu ²⁺ , Cd ²⁺	205
			2-nitrophenol	206
			Paracetamol	200
			Azithromycin, Erythromycin	207
	Pod	Chemically activated Biochar/Active carbon	Dyes	203
			Amoxicillin	204
			Cd ²⁺ , Pb ²⁺ , Cr ³⁺	208
			Ni ²⁺	209
				210
Wheat	Husk	<i>In natura</i> Biochar/Active carbon	Fluorescein	212
			Cu ²⁺ , Zn ²⁺ , Ni ²⁺ , Pb ²⁺ , Cd ²⁺	216
			Naphthalene	214
			Dyes	213
			Dyes	215
	Straw	<i>In natura</i> Biochar/Charcoal	Dyes	219
				220
			Cr ³⁺ and Cr ⁶⁺	111
			Ni ²⁺	223
			Pb ²⁺	221
Cassava	Bran	<i>In natura</i> Biochar/Active carbon	Cr ⁶⁺	222
			Cr ⁶⁺	222
			Cr ⁶⁺	224

In this way, Lima and co-authors (2017) used ultrasound modified corn straw to remove malachite green removal from synthetic and real effluents. The application in real effluents composed of dye mixtures and inorganic compounds. The authors concluded that the ultrasound treatment turned the adsorbent in a more attractive material to remove the studied pollutants, by comparison with the raw material. The raw material and ultrasound modified adsorbent achieved removal percentages of 77 and 81%, respectively. Nevertheless, the most attractive result was found when the adsorbent was applied to treat textile effluents with a mixture of dyes and inorganic compounds, in which the color removal of 92% was achieved.¹⁸⁰

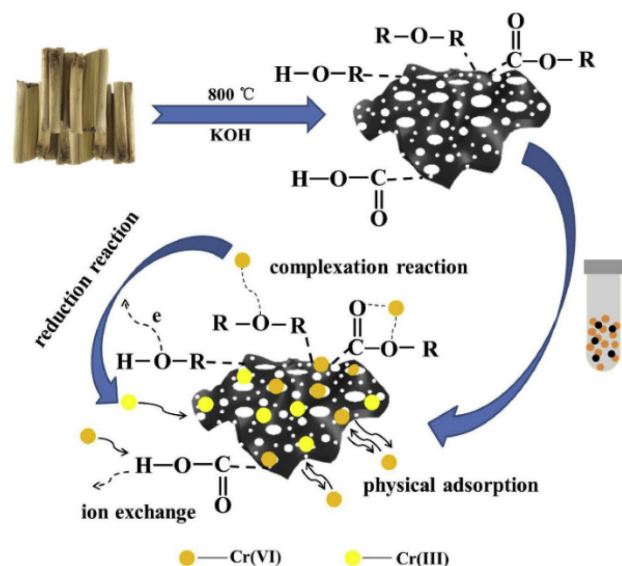


Figure 10. Preparation of the adsorbent and suggestion mechanism for chromium (VI) removal.²²⁶

Additionally, Pathak and co-workers (2015) have applied rice husk ad adsorbent to treat dairy wastewater. Using an adsorbent dosage of 5 g/L, the authors achieved 92.5% removal at 30 °C, indicating a cost-effective process since it is cheaply available raw material. Thereby, the authors pointed that the use of the raw material without previous treatment could increase the COD load of the wastewater, because the silica on the outer and impurities (fats and waxes) on the inner surfaces cause improper binding between the active sites and molecules. Therefore, activation must be conducted to ensure highly efficiency of the adsorbent.²²⁷

Using spent coffee grounds activated carbon, Yuan and Ferraz (2020) applied the produced adsorbent to remove organic matter and color from synthetic and real landfill leachate. The authors activated the biomass with phosphoric acid at different ratios, the material impregnated using the lowest acid ratio and pyrolysis temperature of 500 °C presented higher surface area. The activated carbon produced achieved a COD removal of 40 mg per gram of adsorbent, the material showed excelente performance

when real landfill leachate was used, reaching >90% of color and COD removal.²²⁸ Follow this line, coffee husk was used to produce biochar by pyrolysis process without any activation process and used to remove ammonium in water and wastewater. The pyrolysis was carried out at low temperatures of 350 °C in a short time of 1 h. The adsorbent presented a relatively high adsorption capacity after 6 h with maximum 2.8 mg N/g biochar. The ammonium removal by coffee husk biochar could be reached the efficiency of 43% at 25 °C, initial ammonium concentration of 50 mg/L, and pH of 7. These findings are relevant to highlight that a worldwid culture as coffee can significantly improve the water and wastewater treatment in a social and economical way, since the coffee husk biochar produced from low temperature pyrolysis could substitute high-priced commercial adsorbent to treat ammonium from different water and wastewater sources.²²⁹

The capacity of biochars derived from agricultural wastes to remove Cd(II) and Cu(II) from aqueous solution and contaminated mine water was evaluated by Bandara and co-workers (2020). The authors produced biochar from different agricultural sources, for instance, poultry litter; lucerne shoot; vetch shoot; canola shoot; wheat straws; and sugar-gum wood. Afterwards, it was demonstrated that poultry litter-derived biochar removed Cd(II) and Cu(II) from mine water up to the levels recommended by the World Health Organisation. The experiments showed that precipitation with carbonate and phosphate, complexation with $-\text{OH}$ and $-\text{COOH}$ groups and electrostatic interaction with O-containing surface functional groups were the main mechanisms involved in the removal, as represented in Figure 11. This remarks can drive the biomass selection to produce the biochar addressed to maximise remediation of multi-metals in contaminated water.²³⁰

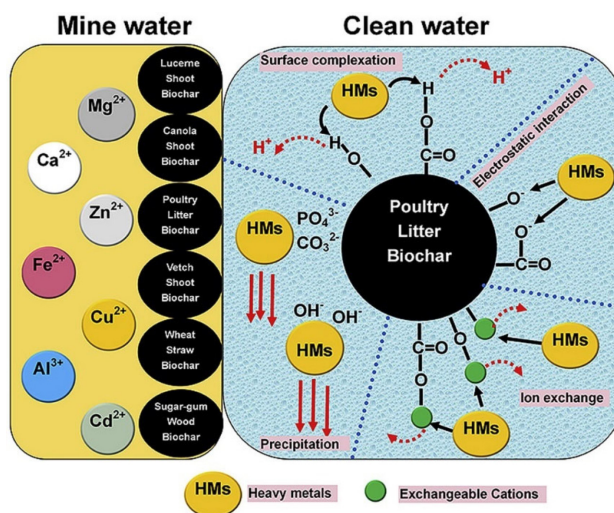


Figure 11. Different mechanisms involved on metals removal from water and wastewater.²³⁰

Talking about mechanism, it was determined that cassava waste consists of ligands such as hydroxyl, sulfur, cyano,

and amino which could bind heavy metal ions. Abia and co-authors (2003) conducted a study establishing the optimal concentration of thioglycolic acid for the removal of Cd^{2+} , Cu^{2+} , and Zn^{2+} ions by cassava waste. The increase on the concentration of the modifying agent (thioglycolic acid) has elevated the presence of sulfhydryl groups, which raised the adsorption capacity of cassava waste. The optimized adsorptions were achieved in less than 30 min. The order of maximum adsorption capacity among the three heavy metal ions was as follows: $\text{Zn}^{2+} > \text{Cu}^{2+} > \text{Cd}^{2+}$, and the adsorption capacities of modified cassava waste were reported to be 647.48 mg Cd/g and 559.74 mg Zn/g, comparing to only 86.68 mg Cd/g and 55.82 mg Zn/g when using raw cassava waste.^{231,232}

Also trying to find a great adsorbent material from agricultural sources to remove heavy metals from water, Oyewo and co-workers (2016) transformed banana peel in a nanosorbent for the removal of radioactive minerals from real mine water. The functional groups responsible for the banana peels capability to coordinate and remove metal ions (uranium and thorium) were identified at absorption bands of 1730 cm^{-1} (carboxylic groups) and 889 cm^{-1} (amine groups) via FTIR analysis. The authors found a maximum adsorption capacity of 27.1 mg g^{-1} , 34.13 mg g^{-1} for uranium and 45.5 mg g^{-1} , 10.10 mg g^{-1} for thorium in synthetic and real mine water, respectively. These results indicated a prospective adsorbent material for the removal of radioactive substances from aqueous solution and also from real mine water, always observing the target pollutant to apply a more suitable material.^{233,234}

In a review discussing the conversion of banana waste into adsorbents materials and its applications to remove various types of water-soluble pollutants, it was possible to observe several advantages of use this agricultural waste. Such features are low cost, widely availability, and protecting environment by preventing methane/ CO_2 gas formation due to unsafe dumping in wetlands (aerobic and anaerobic decomposition of banana tree waste can produce methane gas) or burning (CO_2). It was possible to observe that modifications on the adsorbent can improve its surface area and expose some functional groups that can improve the adsorption capacity of different pollutant types. It was also possible to highlight that there is a gap in research of the adsorption mechanism involving banana-derived adsorbents and water pollutants, on the other hand, as expressed on Figure 12, banana tree can provide several parts that can be used as adsorbent and were already tested to remove metals, dyes, pesticides, radionuclides, inorganic anions and a huge variety of organic pollutants. Nevertheless, the authors have concluded that, besides the mechanistic studies, cost studies must be conducted to stimulate the industrial use of nonconventional adsorbents. The use of biomass-derived adsorbents in wastewater industries are strongly recommended owing to cheaper and readily available, user-friendly technology, and environmentally sustainable operations.²³⁴



Figure 12. Different parts of banana tree applied to remove pollutants from water and wastewater.²³⁴

Another highly important culture with great potential to have their wastes transformed into adsorbents are beans. Etorki and co-workers (2014) used Fava Beans as low cost adsorbent material for removal of $\text{Pb}(\text{II})$, $\text{Cd}(\text{II})$ and $\text{Zn}(\text{II})$ ions from aqueous solutions. The authors have investigated the use of the adsorbent to treat real taken from batteries factory. The authors found that the best conditions to remove $\text{Pb}(\text{II})$, where $\text{pH} = 3.0$, contact time was 1:30 h and adsorbent concentration was 0.5 g .²³⁵

As a prominent culture, already known and widespread in several places around the world, the Açai also had its residues studied and transformed into adsorbent materials. Waste from the açai processing industry, for added value concerns, was used as biomass for energy production, additionally, the biochar resulting from this process was activated with pure NaOH and applied as adsorbent to remove Methylene Blue and reactive dyes from raw textile effluents. Relevant modifications were observed on the adsorbent surface after its activation. A increase in its specific surface area from 1.94 to $491.90\text{ m}^2\text{ g}^{-1}$ have led to an expressive rise in its adsorption capacity for Methylene Blue from 33.73 to 93.23 mg g^{-1} . Activated carbon from açai was also studied on the adsorption of raw textile wastewater, achieving a reduction of 84.62% in the Biochemical Oxygen Demand, these remarks are a special motivation to research and investments on the use of agricultural waste for environmental remediation, since the waste here have been used not only to generate energy but to help in real wastewater purification.²³⁵

Finally, wastes from coconut trees and its applications. As already pointed, coconut trees are on top level of importance for several reasons, their large presence in many countries on the world and especially the huge amount of these trees in the Brazilian coast. Coconut has highly economical impact and social relevance, however their waste is still worth of study and used in many different ways. For instance, Yasdi and co-workers (2021), have pointed out the use of coconut shell-based activated carbon to treat

real wastewater from kitchen restaurant by reducing the Biochemical Oxygen Demand (BOD). The authors have activated the adsorbent using acid and base. The results showed that the best activator for coconut shell carbon was H_3PO_4 3 M. The adsorption process's optimum pH was at pH 3 with an adsorption percentage of 88.626% and contact time of 10 minutes.²³⁶

Additionally, Nandeshwar and co-workers (2016) have used coconut shells carbonized at 500° C and activated with HCl, HNO_3 , H_2SO_4 as adsorbent to remove iron from real industrial wastewater samples from villages near Nag River (India). Their results showed a high increase on the adsorption capacity after the activation process by all acids used, probably by the elevation of the surface area from (9–11 cm^2/g) to (HCl, 754 cm^2/g ; HNO_3 , 542 cm^2/g ; H_2SO_4 , 511 cm^2/g). The adsorption experiments provided a corroboration to the effect of activation, in case of activated carbons prepared from coconut shells, the material activated by (HNO_3) gave the highest iron removal for all samples (A, 85%; B, 77%; C, 82%; D, 76%; E, 72%). The respective iron removals for the case the material activated by (HCl) were also high (A, 70%; B, 75%; C, 81%; D, 75%; E, 68%), but when sulfuric acid was used for activation the removals were slightly lower (A, 68%; B, 72%; C, 76%; D, 74%; E, 66%). In comparison with the respective removals for non-activated material, the iron removal was quite low (A, 36%; B, 38%; C, 57%; D, 54%; E, 57%). This pointedly demonstrates the effect of activation on the adsorption capability of the adsorbents prepared materials prepared from agricultural wastes.²³⁷

With the points highlighted in this topic and in the previous ones, it is possible to understand the valuable possibility of the application of agricultural residues in the treatment of effluents acting as adsorbents. It is undeniable that there is a large gap between the various studies on a laboratory scale and the transfer of the application to real treatment systems. This gap is evidenced by the low number of studies involving treatment of real effluents on bench scale, on pilot scale and the transformation of these materials into commercial product for industrial application. The points shown aim to highlight Brazil with its great agricultural capacity, which in turn generates a large amount of solid waste from these activities. The use of residues in the preparation of adsorbent materials, which can be considered as value-added materials, aims to solve two environmental problems in parallel. The destination of the large amount of solid waste that still does not have proper application and disposal, and the large amount of liquid waste that can be treated using low-cost adsorbents. The production of these adsorbent materials lead to a resolution to the current problem of the adsorption phase in the treatment of effluents, where the highest associated cost is related to the price and quantity of the adsorbent materials used.

6. Concluding Remarks

Currently, it is mandatory to search for economically viable alternatives that aim to reduce energy expenditure and the raw material. In the field of liquid waste, population growth points to exponential industrial growth. This inevitable reality enhances the search for more effective waste treatment methods, to the point of exacerbating reuse, reducing the search for primary sources. In this field, the work described here sought to shed light on the diversity of agricultural crops present in a country of continental dimensions like Brazil, which produces a large amount of food and consequently generates a large amount of solid and liquid waste. In addition, it is worth noting that such agricultural crops of high relevance in Brazil are also found scattered throughout the world. The work showed the economic importance of these crops, highlighting the amount produced and the associated value, as well as incorporating this amount and this importance with environmental issues. The growing interest in the use of natural materials in studies involving effluent treatment via adsorption was shown through a bibliometric analysis. Then, details of how these adsorbent materials can be prepared and directed to greater adsorption efficiency were discussed, when the characteristics of each material and each synthesis variable are known by appropriate techniques for characterizing structures and surfaces. Finally, the gap between laboratory-scale studies and the end of the production chain, which is the commercial and industrial application of these low-cost adsorbent materials from agricultural waste, was highlighted. It was evident that the use of these materials can solve the problem of solid waste that needs to be disposed of properly and liquid waste that needs to be treated so that the water is returned to water bodies and reused.

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