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# Revalorization of Coffee Waste

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## Abstract

One of the household methods most used to prepare the coffee beverage is the coffee dripping method, which generates millions of tons of coffee waste (CW). Its disposition without control causes environmental matters due to the high consumption of oxygen during its decomposing process. However, the high availability, low cost, and chemical composition of CW (cellulose, hemicelluloses, lignin, ashes, protein, aliphatic acids, fats, and water) make them useful material for obtaining added-value products and bioenergy. In this chapter, the state of the art of different sustainable alternatives to revalorize CW is shown. CW has been successfully applied as an adsorbent for removing pollutants from wastewater and gas, a precursor for obtaining activated carbon, and a feedstock for producing energy and valuable products using mono-process extraction and biorefinery.

**Keywords:** spent coffee ground, adsorption, biorefinery, bioenergy, activated carbon, experimental design

## 1. Introduction

Today, coffee is the second worldwide traded commodity after the oil, and it is the second beverage most popular after water. The importance of the global coffee sector can be pointed out due to its presence in 80 countries employing approximately 100 million people [1]. In January 2020, the International Coffee Organization (ICO) estimated that coffee consumption would increase from 1.24 million bags to 169.34 million bags of coffee by the year 2019/2020 [2]. According to these data, there will be a high quantity of spent coffee grounds (SCGs) produced from coffee beverage preparation, which would be released as domestic or industrial trash and cause environmental matters. SCG is considered a toxic residue due to its content of polyphenols, tannins, and caffeine. It has been estimated that 1 ton of green coffee beans can generate 650 kg of SCG, and 1 kg of soluble coffee produced makes 2 kg of SCG wet [3, 4]. The high availability and low cost of SCG allow its revalorization for obtaining valuable products, such as chemical products, activated carbon, biodiesel, and bioenergy.

This chapter will briefly discuss the different ways to revalorize coffee waste. In the first part of this chapter, physicochemical properties are explained since they represent the first stage on SCG revalorization. In the second part, the use of coffee waste as an adsorbent for the removal of pollutants from liquids and gases is shown. The activated carbon produced from coffee waste and its utilization as an adsorbent to remove organic and inorganic pollutants is another topic explored. The recovery

of valuable compounds and energy using mono-process extraction and biorefinery from coffee waste will be reviewed. Finally, the experimental design methods to optimize the different processes of coffee waste revalorization are analyzed.

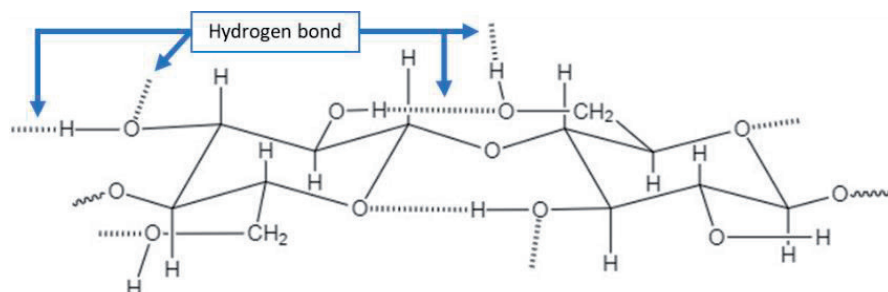
## 1.1 Physico-chemical properties of coffee waste

The biomass revalorization, such as coffee waste, depends primarily on their physicochemical properties, such as chemical composition, presence of extractable compounds, and diversity of functional groups. These properties are altered according to the type and plant variety; in the case of coffee, the most commonly used is the so-called Arabica coffee, so their main physicochemical characteristics were briefly analyzed.

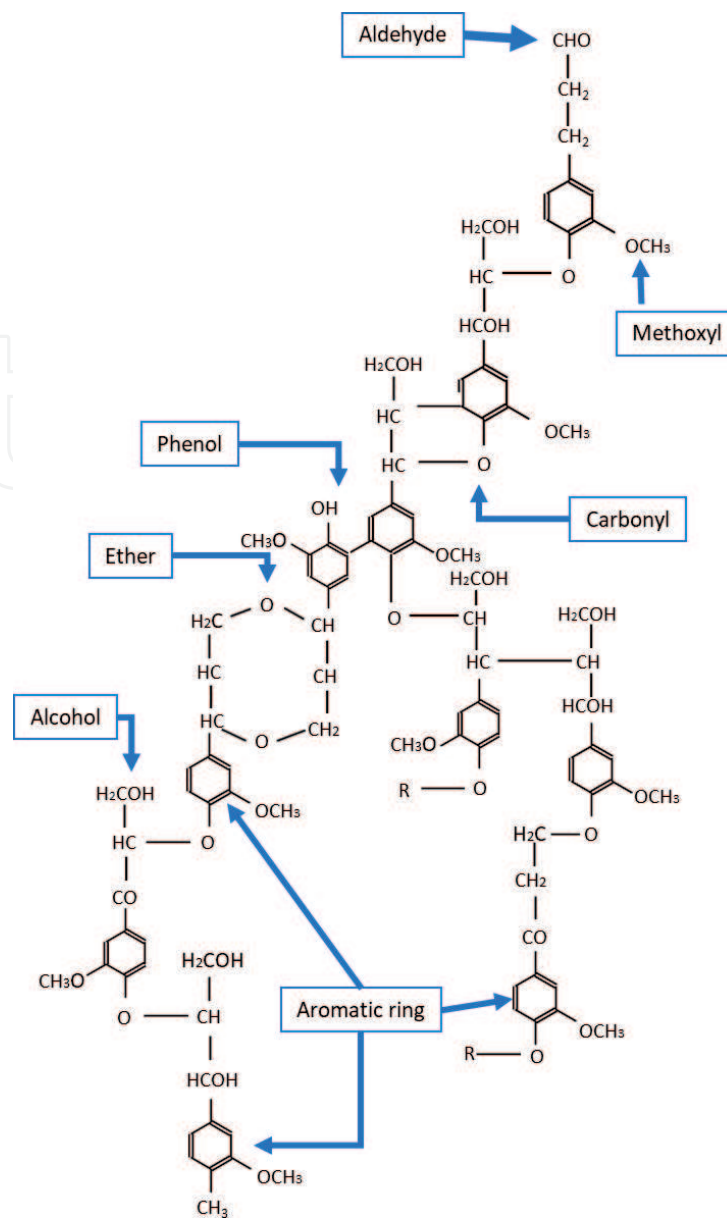
### 1.1.1 Chemical composition

Coffee waste, being lignocellulosic biomass, which is mainly composed of the essential life elements (C, H, O, and N), which are primarily forming cellulose (59.2–62.94 wt%), hemicellulose (5–10 wt%), and lignin (19.8–26.5 wt%) [5, 6]. Besides, these elements are present in the form of recoverable compounds, such as essential oils and flavonoids, among others. However, since this material has already been subjected to a hydrothermal extraction process, the presence of these compounds is usually low compared with lignocellulosic constituents (10 wt%) [6]. Moreover, this type of waste usually has some elements considered inorganic micronutrients such as calcium, magnesium, or sodium, but their concentrations are generally less than 5.0% dry weight [5–7].

The main component of plant biomass is cellulose, which is made up of linear chains of D-glucose linked by  $\beta$ -1,4 bonds, and it has a form of crystalline fibrillar aggregates, which are formed due to the hydrogen bonds among the HOS present in the D-glucose, as can be seen in **Figure 1**. On the other hand, hemicellulose forms an aggregate of simple sugars of different structures that are attached to cellulose microfibrils. Several authors had reported the presence of xylose, arabinose, galactose, and mannose in coffee residues. These types of molecules usually present cyclic structures of 5 or 6 constituents, being abundant in alcohol groups. However, their heterogeneity makes impossible the formation of crystalline arrangements [7, 8]. On the other hand, lignin, whose molecular representation is illustrated in **Figure 2**, is a biopolymer, not a polysaccharide, which is considered the most abundant in plant biomass. This biopolymer has a high structural diversity originated from the enzymatic dehydrogenation of coumaryl, coniferyl, and sinapyl alcohols and subsequent radical polymerization. This heterostructure provides properties such as hardness, resistance to microbial attacks, and oxidative stress, complicating its biodegradation [9].



**Figure 1.**  
Cellulose structure showing the hydrogen bonds.

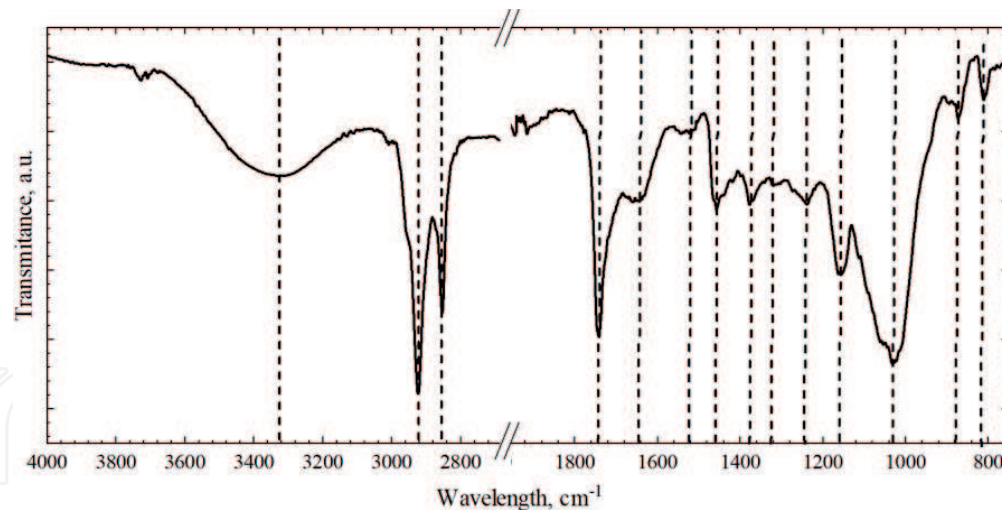


**Figure 2.**  
*Lignin chemical structure.*

### 1.1.2 Surface physicochemical properties

Given the structural diversity of the constituents of the coffee residue, a heterogeneous presence of functional groups on the surface of the material is expected, which will provide this biomass with unique characteristics. Cellulose and hemicellulose have functional groups of the alcohol type ( $R-OH$ ), which can favor the functionalization of these materials, for example, through esterification processes [9]. On the other hand, given its formation process, lignin as a macromolecule has phenolic and aliphatic hydroxyl groups, in addition to methoxyl, carbonyl, and aldehyde groups, among others [8]. The concentration of these groups will depend on the variety and class of the starting material. The structure of lignin is shown in **Figure 2**, and the functional groups mentioned above are indicated; it is important to highlight that lignin has aromatic rings capable of promoting interactions  $\pi-\pi^*$  with other compounds, which could allow the use of coffee waste as an adsorbent for organic compounds [10].

Among the various analytical techniques used to characterize solid materials is infrared spectroscopy with Fourier transform, which allows identifying surface functional groups simply and effectively. The infrared spectrum of coffee waste is presented in **Figure 3**. In it, the wavelengths at which the various vibrational modes



**Figure 3.**  
*FTIR spectrum of a sample of coffee waste.*

of the surface groups can be detected are indicated. The absorption bands found are similar to those reported by multiple authors for coffee residues of the Arabica variety [5, 6, 8]. In the spectrum, two absorption regions can be evidenced, the first one from 3800 to 2700  $\text{cm}^{-1}$ , finding signals around 3340  $\text{cm}^{-1}$  corresponding to the vibrations of the OH bonds present in the alcohol groups, followed by a doublet of bands at 2920 and 2860  $\text{cm}^{-1}$  of the CH interactions, present in all lignocellulosic structures. The second region, between 1900 and 750  $\text{cm}^{-1}$ , has a higher number of corresponding bands with links C=O of the carbonyl groups present in the aldehydes (1740  $\text{cm}^{-1}$ ); C=C of the double bonds of the aromatic structures of lignin (1640, 1525, and 1475  $\text{cm}^{-1}$ ); CH of the methyl and methylene groups of the polymer chains of the constituents (1440 and 1380  $\text{cm}^{-1}$ ); CO of the groups of the ester type (1320, 1240, and 1160  $\text{cm}^{-1}$ ) and alcohol (1030  $\text{cm}^{-1}$ ); and finally, the bands located at 870 and 810  $\text{cm}^{-1}$  are characteristic signs of substitutions in aromatic structures. Together these bands corroborate the polymeric nature of the coffee residue and make it possible to elucidate, at least qualitatively, the type of surface structures it possesses. The functional groups detected on the surface of the material are primarily acidic, which means that they are capable of yielding the proton and therefore can grant a negative charge density to the biomass surface depending on the pH of the medium. Volesky [11] and Ahsan et al. [12] reported that this type of functional group acts as active sites in the processes of pollutant removal. Several studies have quantified the presence of this type of active sites, indicating in a general way the predominance of phenolic, carbonyl, and carboxylic sites [5, 9, 13].

## 2. Pollutants removal by coffee waste

Coffee wastes in their several forms (e.g., coffee husks, coffee silverskin, coffee bean skins, and spent coffee grounds) have been used in the removal of inorganic and organic compounds from aqueous solutions at least for the last two decades. The first report about the use of coffee wastes for the removal of pollutants from wastewater was published in 2002 [14]. In this study, the authors evaluated several adsorbents, including coffee bean skins (CBSs), for the removal of copper and zinc ions from swine breeding wastewater. The copper removal efficiency of CBS was about 50%, whereas no zinc adsorption was obtained. However, no insight regarding the adsorption mechanism was provided. An attempt to elucidate the adsorption mechanism of metal ions was made by measuring the isotherms of lead adsorption



onto degreased and protein-denatured coffee grounds [15]. The amount of lead adsorption onto degreased coffee grounds did not exhibit significant change compared to that on coffee grounds. On the contrary, protein-denatured coffee grounds had an adverse effect on the amount of lead adsorbed. These results indicate that fat cannot adsorb lead ions, but proteins contained in coffee grounds are responsible for the removal of lead ions. Also, it was demonstrated that there is no dependence on the type of coffee beans (e.g., *C. robusta*, *C. arabica* from four different regions) in the adsorption of lead ions due to their similar protein content.

Untreated coffee husks (UCH) have been successfully used for the removal of several heavy metal ions such as chromium (Cr), copper (Cu), cadmium (Cd), and zinc (Zn). Oliveira et al. [16] reported maximum adsorption capacities of 7.5, 6.96, 6.85, and 5.56 mg/g for the adsorption of Cu, Cr, Cd, and Zn onto UCH, respectively. In this study, Boehm titration was used to determine the functional groups before and after the adsorption experiments. The authors observed a decrease in the quantity of functional groups due to heavy metals' adsorption. The results showed that all functional groups (carboxylic, lactonic, phenolic, and basic groups) were involved in the adsorption of heavy metal ions, with relative affinities as follows: Cu > Cr > Cd > Zn for basic groups; Zn > Cu > Cr > Cd for carboxylic groups; Cr > Zn > Cd > Cu for lactonic groups; and Cr > Zn > Cu > Cd for phenolic groups.

Coffee silverskin (CS) is another relevant coffee waste evaluated for the removal of metal ions. CS is part of the outer layer of green coffee beans, which is generated during the roasting process, and it has no commercial value [17]. CS demonstrated similar adsorption efficiency of Ni and Zn when it was compared to SCG, while Cu ions were removed to a lesser extent by using CS. The authors attributed the higher performance of SCG to the higher content of lignocellulosic components. The maximum adsorption capacities on CS were 15.17, 9.58, and 1.43 mg/g, respectively, for Cu, Zn, and Ni ions.

Among the different forms of coffee wastes, spent coffee grounds (SCGs) collected from coffee shops or cafeterias have become one of the most popular coffee wastes studied for the removal of pollutants. Azouaou et al. [18] used them without treatment for the removal of Cd ions from aqueous solution. The authors reported an adsorption capacity of 15.65 mg/g and 120 min to achieve the adsorption equilibrium. Also, it was demonstrated that the particle size does not affect the removal of Cd, suggesting that intraparticle diffusion is not the rate-limiting step. Davila et al. investigated the adsorption mechanism of copper ions onto SCG [6]. They found that the amount of calcium ions and hydrogen ions, released from SCG carboxyl and hydroxyl groups during Cu adsorption, were similar to the amount of Cu ions adsorbed. Thus, the adsorption of Cu ions onto SCG was mainly due to ion exchange. The maximum adsorption capacity obtained was 14 mg/g. Similarly, Gomez-Gonzalez et al. [13] conducted the adsorption of Pb ions by SCG and examined the pH effect on the adsorption capacity. An increase in pH caused an increment of the adsorption capacity of Pb, and the maximum adsorption capacity reported was 22.9 mg/g at pH 5. On the other hand, Elsherif et al. [19] evaluated the removal of cobalt by SCG. The authors reported a maximum adsorption capacity of 243.9 mg/g.

Additionally, SCG has been used for the simultaneous removal of metal ions from aqueous solutions. In this regard, Futralan et al. [20] evaluated the performance of SCG for the simultaneous removal of Cu, Pb, and Zn from soil washing wastewater. The maximum removal efficiency obtained was 57.23, 68.73, and 84.55% for Pb, Cu, and Zn ions, respectively. The removal of mercury ions by SCG was reported by Mora Alvarez et al., and the maximum adsorption capacity was found to be 31.75 mg/g [21]. Two desorption agents were evaluated, nitric acid and chloride acid, where the latter presented better desorption of Hg ions. However, when SCG was subjected to one adsorption-desorption cycle, a loss of removal efficiency was observed, decreasing from 97 to 28% Hg removal. On the contrary, Kyzas [22]

demonstrated the strong reuse potential of SCG in the adsorption of Cu and Cr ions since only 10% of metal ion uptake was loss after 10 cycles of adsorption-desorption. Similarly, the adsorption capacity of Cu, Cd, and Pb ions by SCG remained the same during four adsorption-desorption cycles according to the report by Davila et al. [23]. In this study, SCG regeneration was carried out using citric acid, calcium chloride, and nitric acid as eluent agents. The trend of the desorption efficiency through four adsorption-desorption cycles was  $\text{HNO}_3 > \text{CaCl}_2 > \text{C}_6\text{H}_8\text{O}_7$ .

Although most applications of coffee waste have been made for the removal of inorganic pollutants from water, coffee waste also has demonstrated the potential for the removal of organic pollutants. For example, methylene blue (MB) was removed from aqueous solutions by UCH [24]. The results showed that above the point of zero charge of the UCH (approx. pH 4.5), there was no pH effect on the removal of MB. The maximum adsorption capacity of MB onto UCH was 55.3 mg/g. MB has been used as a model dye molecule to demonstrate the potential of an adsorbent for the removal of dyes from wastewater. The capability of coffee waste for the removal of organic pollutants is associated with the density of the oxygen-containing functional groups that increase the p-p interaction force between the coffee wastes and the organic molecules. Accordingly, Dai et al. [25] proposed an adsorption mechanism for tetracycline (typical bactericidal drug) onto SCG by pi-pi interaction between the aromatic ring of the tetracycline molecule (TC) and the aromatic functional groups of the SCG. The maximum adsorption capacity of TC onto SCG was found to be 64.89 mg/g. Also, the effect of ionic strength on TC adsorption was evaluated, where there was a competition for the adsorption sites, diminishing the adsorption capacity as the ionic strength was augmented.

It is noteworthy mentioning that most of the studies on pollutant removal by coffee wastes have been carried out in batch configuration. However, adsorption by continuous fixed bed systems are the common configuration used in industrial applications due to the high volume of pollutant-solution processed, operation simplicity, and higher mass transfer characteristics than batch systems. Despite that, only a few reports have been made on the use of coffee waste in fixed-bed columns. Utomo et al. [26] conducted column adsorption experiments for the removal of Cu, Zn, Cd, and Pb ions by SCG. The adsorption efficiencies were higher than 91% for all metal ions. Besides, the percentage of Cu ions adsorbed by a column packed with SCG was shown, where it can observe the breakthrough at 100 mL (30 min). A thorough study of the performance of Cd, Cu, and Pb ion removal in a fixed-bed column packed with SCG was presented by Davila et al. [23]. The effect of the process variables (e.g., flow rate, bed height) was evaluated, and the maximum breakthrough times of Cd, Cu, and Pb ions were 50, 160, and 220 min, respectively. Furthermore, the breakthrough curves were predicted well by using a mass transfer model that includes axial dispersion, external mass transfer resistance, and ion-exchange model to describe the equilibrium adsorption.

All the applications mentioned above of coffee wastes were about the removal of inorganic or organic pollutants from wastewater. Only one study has reported the use of coffee wastes for the removal of a gaseous pollutant [27]. In this study, a decrease in 43% on the ozone concentration in an ozone-filled chamber was achieved by using SCG, which was competitive to the performance of commercial activated carbon (about 56%).

### 3. Pollutants removal by modified coffee waste

High volumes of coffee waste with no commercial value are generated worldwide daily, causing an environmental burden. For this reason, several studies have been

conducted to reuse coffee wastes as adsorbents for the removal of several pollutants. Although untreated coffee wastes have demonstrated adsorption capacities similar or even higher than those obtained by commercial materials (e.g., activated carbon), recent studies have focused on the modification of coffee wastes to increase even further the removal efficiency. In this sense, Lafi et al. [28] modified the surface of commercial coffee waste with cationic surfactants, cetyltrimethylammonium bromide (CTAB) or cetylpyridinium chloride (CPC), to increase the affinity for the anionic dyes such as methyl orange (MO). The maximum adsorption capacity obtained for MO was 58.82 and 62.5 mg/g, onto CTAB-coffee waste and CPC-coffee waste, respectively. On the other hand, Cerino-Córdova et al. [29] modified the surface of SCG with citric acid to increase the amount of carboxylic groups. By doing that, the adsorption capacity of Pb and Cu ions was 3.2 and 8.1 times higher than those obtained by the unmodified SCG. Similarly, Botello-Gonzalez et al. [9] investigated the adsorption capacity of SCG modified with citric acid in the competitive adsorption of Pb and Cu ions. The maximum adsorption capacities of Pb and Cu ions were 130 and 45.4 mg/g, respectively. Additionally, the authors proposed a model based on ion exchange that takes into account the surface chemistry of the modified SCG interaction with the heavy metal ions in the liquid phase. In another study, SCG was acid activated with hydrochloric acid and examined for the removal of lead and fluoride ions [30]. The maximum adsorption capacities were 65.4 and 9.75 mg/g of Pb and F ions, respectively. Another acid activation of the surface groups of coffee waste was carried out with sulfuric acid [31]. The sulfonate coffee waste (CW-SO<sub>3</sub>H) was successfully used for the removal of bisphenol A (BPA) and sulfamethoxazole (SMX) from water. Highly negative surface charge was obtained after the incorporation of the sulfonic acid groups, increasing the interaction with the cationic pollutants. The maximum adsorption capacities were found to be 271 and 256 mg/g for BPA and SMX, respectively. Besides chemical modification of coffee waste, physical activation has been employed successfully for the removal of metal ions. For example, Delil et al. [32] conducted the reduction of the grain size of SCG by an ultrasonic process to increase the specific surface area. Also, the zeta potential of the activated SCG was more negative after the ultrasonic method, enhancing the adsorption of cadmium ions.

#### 4. Pollutant removal by coffee waste composites

Composite adsorbents with coffee waste (CWC) have been synthesized and examined for the removal of pollutants from aqueous solutions. In this regard, some studies have evaluated the encapsulation of coffee wastes in polysaccharides such as calcium alginate (CA) and chitosan (Cs). For instance, spent coffee grounds were encapsulated by using CA to increase the adsorption capacity of Ni, Cd, and Cu [33, 34]. The results showed high adsorption capacities and faster adsorption rates than CA beads alone. In another study, coffee wastes were mixed with Cs and poly(vinyl alcohol) (PVA) to enhance the adsorption capacity of pharmaceuticals [35]. The addition of coffee wastes to the matrix of Cs-PVA allowed an increase in the adsorption of metamizole (MET), acetylsalicylic acid (ASA), acetaminophen (ACE), and caffeine (CAF) as compared to the virgin material.

On the other hand, coffee waste composites with magnetic properties have been synthesized to facilitate the removal of the adsorbent from the liquid media. In this sense, magnetic coffee waste composite prepared from Fe<sub>3</sub>O<sub>4</sub>, PVA, and alkaline pretreated SCG was evaluated for the removal of Pb ions from aqueous solutions [36]. The maximum adsorption capacity of Fe<sub>3</sub>O<sub>4</sub>/PVA/APSCG of Pb ions was reported as 57 mg/g. Similarly, a magnetic coffee waste composite was prepared by using SCG and Fe<sub>3</sub>O<sub>4</sub>, without PVA as a cross-linking agent [37]. The maximum



adsorption capacity of Pb ions was found to be 41.15 mg/g when a 2% loading of Fe<sub>3</sub>O<sub>4</sub> nanoparticles was used. A further increase in the Fe loading decreased the removal of Pb ions due to the agglomeration of Fe<sub>3</sub>O<sub>4</sub> on SCG.

Other types of coffee waste composites studied for the removal of pollutants from aqueous solutions are those obtained from the combination of clay or siliceous materials with coffee waste. In this regard, limestone combined with SCG was synthesized for the removal of both anionic and cationic dyes (methylene blue and orange II, respectively) [38]. The maximum removal percentage for methylene blue (MB) and orange II (OR II) was 100 and 85% at pH 8 and 2, respectively. However, in competitive adsorption experiments, the presence of MB causes a reduction in the removal of OR II from 85 to 60%. Another coffee waste composite reported as a heavy metal scavenger is composed of coffee wastes and attapulgite clay (SCG-AC) [39]. The maximum adsorption capacity of Pb ions was reported to be 4.45 mg/g.

## 5. Solid coffee waste as a precursor to activated carbon

The use of lignocellulosic waste to obtain valuable products has been proved to be an critical ecological strategy because these wastes are widely available. Therefore, these wastes represent a pollution problem in the water, soil, and air. A pyrolysis process can be used to obtain some valuable products, such as biofuels and activated carbon, among other useful products. Activated carbon is widely used as an adsorbent material to remove pollutants from aqueous solutions and to capture CO<sub>2</sub> or H<sub>2</sub>S in the gas phase. Thus, by using lignocellulosic waste, it is possible to prevent soil, water, and air pollution and to apply activated carbons in tertiary treatment of wastewater.

### 5.1 Pyrolysis process

There are many sources to obtain agricultural waste, for instance: barley husks, coconut shells, sawdust, and spent coffee grounds, among others. These wastes have different percentages of cellulose, hemicellulose, and lignin. Today, agricultural wastes are readily available and are released to the environment or used for other proposes, such as livestock feed. The content of fixed carbon in these wastes and their abundance has led several researchers to investigate the use of these wastes as precursors to produce activated carbon, which can be used as adsorbent material to remove pollutants from aqueous solutions.

The pyrolysis process is useful to obtain some valuable products from lignocellulosic biomass. Pyrolysis means the thermal decomposition of lignocellulosic biomass under an inert atmosphere, for instance: nitrogen, argon, steam, and carbon dioxide, among others. The products of the pyrolysis process include biochar, biofuel, and volatile compounds. To determine the appropriate temperature range to carry out this process, a thermogravimetric analysis is required. Thus, the process is usually performed within temperature ranges from 400 to 600°C and from 700 to 1200°C for chemical and physical activations, respectively. During the pyrolysis process, the biomass loses humidity between 100 and 200°C. At temperatures higher than 200°C, cellulose, hemicellulose, and lignin contents are decomposed at different temperature ranges, besides volatile compounds are released, which content condensable vapors (phenol and aromatics, among others), and light hydrocarbon compounds.

The pyrolysis mechanism of lignin is more complex than that of cellulose and hemicellulose. During the lignin decomposition, there are primary reactions in the range of 200–400°C and secondary reactions at temperatures higher than 400°C [40]. On the other hand, at temperatures of 200–400°C, hemicellulose is broken down [41], and cellulose can be decomposed in a temperature range of

315–400°C [42]. All these decomposition processes lead to polymerize pyrolytic products to develop activated carbons.

## 5.2 Biochar activation

The biochar obtained in the pyrolysis process can be subjected to an activation process, which is a method useful to develop the physical and textural properties of the adsorbent material, such as total pore volume, surface area, and porosity. Besides, the activation process widens the pore diameter from nanopores to mesopores and macropores. This improves the internal diffusion of the pollutants inside the adsorbent particle. The activation of carbon can be carried out by physical or chemical activation. Chemical activation can be performed at a temperature range of 400–700°C by using inorganic compounds. On the other hand, the temperature range for physical activation with steam or CO<sub>2</sub> is from 700 to 1200°C, which means more power consumption.

## 5.3 SCG activated carbon

Commercial activated carbon (CAC) is a useful material to remove pollutants from aqueous solutions. However, CAC can be expensive; for this reason, some researchers have studied several materials to produce activated carbon from lignocellulosic wastes by pyrolysis such as coconut shell, corncob, carnauba pall and fine nut, sawdust, and candied chestnut [43–47]. Given the lignocellulosic structural nature of solid coffee residues, carbon content is predominant compared to other constituent elements. This, along with the abundance of the residue, makes it an optimal material as a precursor in the synthesis of activated carbon [48, 49].

Several researchers have reported the use of coffee waste to produce activated carbon. **Table 1** shows the activation conditions to produce SCG activated carbon by chemical or physical activation. SCGs were chemically activated by KHO, ZnCl<sub>2</sub>, H<sub>3</sub>PO<sub>4</sub>, or H<sub>2</sub>SO<sub>4</sub> or physically activated. The activation temperatures were between 400 and 800°C, and it is important to mention that chemical activation allows low temperatures for the pyrolysis process, instead of physical activation. In most cases, the yield and pore size were reported. A high pore diameter is an important parameter because it allows for the internal diffusion of pollutants inside the adsorbent particle, enhancing the adsorption capacities. According to the data shown in **Table 1**, SCG is a viable option to produce activated carbon because the obtained SCG carbon has a high surface area and a reasonable pore width, which are relevant parameters to carry out an adsorption process to remove pollutants from aqueous solutions.

The use of coffee extract residue to produce ethanol and activated carbon was conducted and studied by Fotouhi et al. [46]. The coffee solid residue was chemically activated by using H<sub>3</sub>PO<sub>4</sub> at 600°C and physically activated with steam at 700°C. The produced adsorbent showed a pore volume range from 0.22 to 0.59 cm<sup>3</sup>/g and a surface area from 233 to 696 m<sup>2</sup>/g. Diaz de León et al. [50] reported the use of SCG to produce activated carbon by chemical activation with ZnCl<sub>2</sub>. An experimental design was carried out varying three factors: temperature (450–600°C), activation time (40–120 min), and impregnation ratio mass of ZnCl<sub>2</sub>: the mass of spent coffee ground (0.5:1.5). The optimal conditions reported were 600°C, 40 min of activation time, and 1.5 g ZnCl<sub>2</sub>/g SCG. At these conditions, a surface area of 1280 m<sup>2</sup>/g, a yield of 26%, and a total pore volume of 0.77 cm<sup>3</sup>/g were reported. The adsorbent obtained was used to remove phenol from aqueous solutions at pH 7, and maximal adsorption capacity of 160.52 mg of phenol/g was reached.

Surface area (m <sup>2</sup> /g)	Total pore volume (cm <sup>3</sup> /g)	Pore width (nm)	Pyrolysis temperature (°C)	Activation agent	Flow	Yield (%)	Ref.
1040.3	0.635	—	700–900	KOH	Ar	—	[48]
1039	0.481	4.7	500	ZnCl <sub>2</sub> Steam	N <sub>2</sub>	40% 20%	[49]
1280	0.77	3	600	ZnCl <sub>2</sub>	N <sub>2</sub>	26	[50]
831	0.44		400, 450, and 500	ZnCl <sub>2</sub>	Air	15.99 22.95%	[51]
1121	0.954	1–3	800	ZnCl <sub>2</sub>	N <sub>2</sub>	—	[52]
2785	1.36	1.051	400 and 700	KOH	N <sub>2</sub>	11–16%	[53]
1778	0.657	—	800	KOH	N <sub>2</sub>	—	[54]
146.1	0.0705	1.6	600	H <sub>2</sub> SO <sub>4</sub>	N <sub>2</sub>	42.77– 51.85%	[55]
1082	0.51	3.0	600 and 700	KOH or CO <sub>2</sub>	N <sub>2</sub>	23–29%	[56]
889 (ZnCl <sub>2</sub> )	0.765 (ZnCl <sub>2</sub> )	3.44 (ZnCl <sub>2</sub> )	600	ZnCl <sub>2</sub> or H <sub>3</sub> PO <sub>4</sub>	N <sub>2</sub>	—	[57]
1003 (HPO <sub>3</sub> )	0.618 (HPO <sub>3</sub> )	2.44 (HPO <sub>3</sub> )					

**Table 1.**

Reports of chemical activation conditions to produce activated carbon from coffee wastes.

According to the data shown in **Table 1**, SCG can be considered an excellent precursor to produce activated carbon. The large surface area achieved in SCG activated carbon could be used to remove inorganic and organic compounds from aqueous solutions.

### 5.3.1 Removal organic and inorganic pollutants by activated carbon-spent coffee grounds

Activated carbon is a material in which carbon is forming disordered graphite plates, in whose peripheries there is a wide diversity of functional groups, which gives it unique physico-chemical properties. Additionally, this material usually has raised surface areas, generally greater than 1000 m<sup>2</sup>/g, which develops through various oxidation reactions [58]. Given these characteristics, this material is typically used in numerous applications, excelling in the removal of organic and inorganic compounds present in the gas and liquid phase.

Waste coffee grounds were used to produce activated carbon by KOH under Ar atmosphere at three temperatures (700, 800, and 900°C), the adsorbent material was tested to adsorb CH<sub>4</sub> and H<sub>2</sub>. The activated carbon at 900°C showed a CH<sub>4</sub> adsorption capacity of 1.96 mmol/g at 273 K and 100 kPa. However, at 3000 kPa, the highest adsorption capacity was reported to be 4.2 mmol/g. The three adsorbents materials (700, 800, and 900°C) were also tried to adsorb H<sub>2</sub> at 77 K and 100 kPa, achieving the highest adsorption capacity of 1.75 wt% [48]. Activated carbon from waste SCG as the precursor was physically activated by CO<sub>2</sub> or steam at high temperatures (700–900°C) and chemically activated by ZnCl<sub>2</sub>, KOH, and H<sub>3</sub>PO<sub>4</sub> at 450 and 600°C. Nevertheless, in this work, only the raw material, the activated carbons by ZnCl<sub>2</sub> or steam, was tested to adsorb Bisphenol-A. The removal of Bisphenol-A was found to be 98, 12, and 0% for carbon activated by ZnCl<sub>2</sub>, raw material, and carbon activated by steam, respectively. These results were compared



with a commercial activated carbon, which showed a Bisphenol-A removal of 93%. The poor adsorption performance of the SCG carbon activated by steam is due to the low surface area reported for this material ( $4 \text{ m}^2/\text{g}$ ) [46].

SCG was used as a precursor to prepare activated carbon by chemical activation with  $\text{ZnCl}_2$  at three impregnation ratios, at room temperature, and during 8, 12, and 24 h. The adsorption of  $\text{Cu(II)}$  was conducted using this activated carbon. The experimental data were fitted by using the Langmuir, Freundlich, and Elovich isotherms; the maximum adsorption capacity (Langmuir) was  $285.71 \text{ mg/g}$ , and the maximum  $\text{Cu(II)}$  removal reported was 18% at 100 rpm, and the pH solution value was not reported [51].

The production of SCG-based activated carbon was carried out at three impregnation ratios, g  $\text{ZnCl}_2/\text{g}$  precursor (1:0.5, 1:1, and 1:2); the impregnated precursor was carbonized under  $\text{N}_2$  atmosphere at  $800^\circ\text{C}$  during 60 min, and this material was tested for  $\text{H}_2\text{S}$  separation. The SCG activated carbon was used to study  $\text{H}_2\text{S}$  dynamic breakthrough capacity passing a dilute flow of  $\text{H}_2\text{S}$ -Air (1000 ppm, 80% humidity) through a fixed bed. The adsorbents activated at impregnation ratios of 1:2 and 1:1 showed the lowest ( $18.2 \text{ mg/g}$ ) and the highest breakthrough capacity ( $127 \text{ mg/g}$ ), respectively [51].

Chemical activation of SCG was performed with KOH using 2:1 and 4:1 impregnation ratio (KOH: precursor) to produce activated carbons. The carbonization process was carried out at  $400$  or  $700^\circ\text{C}$  under  $\text{N}_2$  flow for 2 h. These activated carbons were tested to adsorb  $\text{CO}_2$  at  $0$ ,  $25$ , and  $50^\circ\text{C}$  and  $0$ – $10$  bars. The highest adsorption capacity obtained was  $6.8 \text{ mmol/g}$  at 1 bar and  $0^\circ\text{C}$ , when the activated carbon was produced at  $700^\circ\text{C}$  and an impregnation ratio of 4:1. However, when the carbonization process was performed at 10 bars, the highest uptake achieved was  $23.26 \text{ mmol/g}$  [53].

SCG microporous activated carbon (using potassium hydroxide as activation agent) was synthesized and characterized. The precursor was pyrolyzed using 1:9, 1:18, and 1:36 mmol KOH:g SCG of impregnation ratios and under  $\text{N}_2$  flow at  $800^\circ\text{C}$  during 1 h. SCG activated carbons were used to adsorb phenol and methylene blue. The equilibrium was attained within 100 and 360 min for phenol and methylene blue, respectively. The maximal adsorption capacity, based on Langmuir isotherm, for phenol and methylene blue was 3008 and 1058 mmol/g, respectively [54].

The influence of the impregnation ratio of  $\text{H}_2\text{SO}_4$  over SCG granular activated carbon to treat leachate was studied. Six samples of leachate with the following chemical and biological parameters were treated: COD ( $1010$ – $1815 \text{ mg/L}$ ),  $\text{BOD}_5$  ( $184$ – $338 \text{ mg/L}$ ),  $\text{NH}_4\text{-N}$  ( $2208$ – $2780 \text{ mg/L}$ ), iron ( $4.25$ – $4.73 \text{ mg/L}$ ), and  $\text{PO}_4\text{-P}$  ( $220$ – $284 \text{ mg/L}$ ). However, in this research, only the removal percentage of iron and  $\text{PO}_4\text{-P}$  was found to be 77 and 84%, respectively, when impregnation ratios of 2.5 and 0.5 were used [55].

SCG obtained from a trademark coffee (Nespresso®) was used as a precursor to produce activated carbon. KOH was used as an activating agent at four impregnation ratios. The chemical activation process was carried out at 873 K, and physical activation with  $\text{CO}_2$  was carried out at 973 K. These materials were used to capture  $\text{CO}_2$ , which is a byproduct of the combustion process. However, in this study, a pure  $\text{CO}_2$  flow was tested at 298 K and 101 kPa. The  $\text{CO}_2$  adsorption capacities of the adsorbents activated by using chemical activation and physical activation were 3 and  $2.3 \text{ mmol/g}$ , respectively [56].

SCG carbon activated with phosphoric acid and zinc chloride was used to adsorb  $\text{Pb(II)}$  and  $\text{Cd(II)}$ . The precursor was mixed with  $\text{ZnCl}_2$  or  $\text{H}_3\text{PO}_4$  at chemical agent/coffee residue mass ratios of 25, 50, 75, and 100% at  $85^\circ\text{C}$  for 7 h. When the precursor was activated with  $\text{H}_3\text{PO}_4$  (50% impregnation ratio), the maximal adsorption capacities, based on Langmuir isotherm, were as follows:  $89.28 \text{ mg Pb(II)/g}$  and  $46.95 \text{ mg Cd(II)/g}$ . With  $\text{ZnCl}_2$  (75% impregnation ratio) as the activation agent, the maximal adsorption capacities were  $63.29 \text{ mg Pb(II)/g}$  and  $37.04 \text{ mg Cd(II)/g}$  [57].



The use of activated carbon derived from SCG was recently reported, and the activation procedure was carried out with  $\text{ZnCl}_2$ . To optimize the activated carbon production, an experimental design was performed; the independent factors were temperature (450 and 600°C), activation time (40 and 120 min), and impregnation ratio (0.5 and 1.5 g  $\text{ZnCl}_2$ /g SCG), and the experimental responses were surface area, yield, and hardness. The optimal conditions were impregnation ratios of 1.5, 600°C, and 40 min. At these conditions, the experimental responses were surface area 1279.96  $\text{m}^2/\text{g}$ , yield 26%, and hardness 76.77%. The activated carbon produced at these conditions was used to adsorb phenol from aqueous solutions, based on Langmuir isotherm, the maximum adsorption capacity was 160.52 mg/g, and the equilibrium was attained less than 150 min [50].

## 6. Mono-process extraction, bioenergy, and biorefinery

The circular economy demands the efficient utilization of resources in the production systems and the long-term material use by recycling or remanufacturing [59]. This concept can be correctly applied to the product obtained from biomass processing, such as coffee waste. This material could be a feedstock for a mono-process extraction, bioenergy production, and biorefining. The first stage of the process design is determining the composition of SCG, which has been shown to be highly dependent on coffee varieties [60–62]. The range of the biochemical composition values obtained is shown in **Table 2**. It is important to consider that SCG has a high quantity of organic compounds such as polyphenols, polysaccharides, amino acids, fatty acids, and minerals.

### 6.1 Valuable chemical compound recovery by mono-process extraction

These techniques use chemicals to extract valuable organic compounds (lipids, polysaccharides, phenolics, tannins, and caffeine), and it could be assisted by ultrasound, enzyme, or microwave. These chemical compounds can be useful to obtain high added value products: biodiesel, cosmetics, food additives, pharmaceuticals, packing materials, and adhesives. These techniques are divided into conventional (Soxhlet extraction, maceration, and hydrodistillation) and nonconventional techniques (supercritical fluid extraction, enzyme-assisted extraction, ultrasound-assisted extraction, microwave-assisted extraction, pulsed electric-assisted extraction, and pressurized liquid extraction). The factors studied in the SCG mono-process extraction have been the coffee varieties, solvent, time, pressure, and temperature. The Soxhlet extraction process has several disadvantages, such as low productivity, high solvent consumption, and high extraction time [63]. Ultimately, the main goal of nonconventional methods is to decrease the utilization of synthetic and organic chemicals and operational condition and to improve the yield and quality of extract, which makes them environmentally friendly compared to conventional techniques [64]. Mono-process extraction has been used in SCG for oil, caffeine, phenolic compounds, polysaccharides, and tannin recovering.

#### 6.1.1 Oil recovery

The oil content in SCG is highly dependent on the coffee variety (**Table 2**). It has been demonstrated that oil extracted from SCG could be used in biodiesel production. Solvent extraction and supercritical fluid extraction with  $\text{CO}_2$  and ethanol as a solvent

Biochemical composition	
Compound	Concentration (wt%db*)
Lipids	6.7–19
Carbohydrates	14.1–72.4
Proteins	4.3–17
Mannose	21.2–47
Galactose	25–30
Glucose	19–24
Arabinose	3.8–6
Caffeine	0.96–7.9
Oil	10–20 wt%
Ultimate analysis	
Element	ww%db*
C	52.1–53
H	6.8–7.03
N	1.71–3.47
S	0.1
O	34.7–38.1
Proximate analysis	
	ww%db*
Moisture	11.5–61
Volatile	79.5
Ash	0.68–2.2
Fixed carbon	8.2

\*db: Dry base.

**Table 2.**  
 Composition, ultimate, and proximate analysis of SCG.

have been the methods used for oil recovery. The solvent type (ethanol anhydrous, ethanol hydrous, hexane, and methanol), g SCG/g solvent ratio (20.3–23.8 g/g), extraction time (19.5–30.4 min), and temperature (30°C) were studied. The oil yield obtained varied from 7.5 to 14.7 w/w% d.b. The best oil yield obtained (14.7 w/w% d.b.) was using 22.5 g SCG/g hexane, 30°C, and 30.4 min of extraction time [60, 63, 65].

### 6.1.2 Caffeine recovery

Caffeine is an alkaloid, which is the coffee chemical compound most recognized in the world. The content of caffeine in coffee beans is higher than SCG; however, a high quantity remains in SCG. The Soxhlet extraction, ultrasound-assisted extraction, membrane technology, and pressurized liquid membrane with ethanol and water have been the methods used for caffeine recovery. The range of caffeine yield was similar for the different methods, falling in the range from 0.734 to 43 mg/g db [60, 61]. However, the pressurized liquid extraction (PLE) has the advantage of decreasing solvent use and operating time, being an oxygen and light-free environment process.

### 6.1.3 Phenolic compound recovery

SCG has a high content of phenolic compounds (caffeoylquinic, feruloylquinic, p-coumaroylquinic, ferulic, and quinic acids). These have anticancer, antidiabetic, antioxidant, antiviral, antiallergen, antimicrobial, and antifatigue activities. Additionally, these chemical compounds could be incorporated into skincare products. Different methods, such as subcritical water, ultrasound-assisted, pressurized liquid extractions, and supercritical fluid extraction with CO<sub>2</sub>, have been used for phenolic compound recovery [61, 62]. The experimental results showed a range of phenolic compound recovery from 19 to 273.4 mg GAE/g. The results demonstrated that the ethanol extraction method with oil extraction by hexane pretreatment was the best process, followed by the autohydrolysis process (273.4 mg GAE/g). The optimal experimental conditions were 5 ml ethanol/g SCG and ambient temperature [60, 61].

### 6.1.4 Polysaccharides recovery

The polysaccharides in SCG present different structures, such as galactomannans, arabinogalactans, and cellulose, which are used as dietary fiber ingredient in functional food. These compounds have immunostimulatory, antimicrobial, and antioxidant activities. Furthermore, they have good thermal stability properties. Various methods for polysaccharide purification from SCG have been utilized successfully, such as extraction with chemical agents (potassium hydroxide and sulfuric acid), subcritical water hydrolysis, autohydrolysis, and microwave superheated water extraction methods. The polysaccharides extracted from SCG varied from 22 to 61.9 w/w% d.b., and several studies have demonstrated that the yield increases when the coffee is roasted [5, 62, 65–68]. The best method of polysaccharide extraction (61.9 w/w% d.b.) was the microwave superheated water extraction, with the following experimental conditions: 1 g SCG/10 ml of water, 2 min of extraction time, and 200°C.

### 6.1.5 Tannin recovery

Tannins are low-cost natural biopolymers that could serve as biosorbents and prepare as adhesives. The extraction of tannins from SCG has been carried out by Soxhlet extraction with 5% of sodium hydroxide. The best tannin extraction yield was 21.02 mg tannins/g d.b. at 8.2 g SCG/g NaOH, 30 min of extraction time, and 100°C [69].

## 6.2 Energy recovery from SCG

The chemical composition of SCG makes them a viable material to use them as feedstock to produce biodiesel, bio-oil, syngas, and energy via a combustion process.

### 6.2.1 SCG pellets for energy production

The combustion is the process used for obtained energy from SCG due to its calorific value. The SCG can be used after oil and lipid extraction processes. Some studies have been carried out to increase the calorific value of SCG. These wastes have been blended with other materials such as sawdust, beechwood, and glycerol. The solid fuels obtained have a range of heating values from 18.27 to 24.913 MJ/kg [70–73]. SCG calorific values are higher than other types of biomass, and it could be considered a viable fuel to cover the needs of thermal energy of the coffee industry [72].

### 6.2.2 Biodiesel production

Today, the world needs to change the fossil fuel dependence to renewable energy, as it is the case for biodiesel, which has less hydrocarbon, CO<sub>2</sub>, and particle emission than conventional diesel [61]. New bioresources for biodiesel production are being explored, and SCG can be a viable alternative due to its high lipid containing 6–27.8% w/wt [61, 74, 75]. The biodiesel can be produced by transesterification of lipid and oil extracts. It is important to point out that biodiesel yield could be improved when catalysts and ultrasound-assisted processes are employed. The range of biodiesel yield obtained in different studies varied from 16.73 to 100% [63, 76, 77].

### 6.2.3 Bio-oil production

The main goal of this process is converting SCG into bio-oil. Fast pyrolysis, hydrothermal liquefaction in hot-compressed water, and co-liquefaction in sub-critical water have been tested. It is important to point out that in the pyrolysis process, bio-oil, water, biochar, and syngas are produced. The bio-oil yield obtained for these methods varied from 36 to 61.8 wt% of bio-oil [60, 69, 78–83]. The fast pyrolysis has been the method with the best bio-oil yield.

### 6.2.4 Biosyngas production

The SCG could be used to generate power and heat. This process is well known as cogeneration or combined heat and power process. It is used to satisfy the energy needs of industrial plants. The energy and power are generated by SCG gasification at moderate pressure (0.3–0.5 bar), temperature above 650°C, and using oxidants such as air, steam, and carbon dioxide. The gas produced of this reaction is named syngas or producer biogas, which contains methane, carbon dioxide, carbon monoxide, and hydrogen. The syngas can be burnt in a fuel cell or a conventional combustion engine [61, 84].

## 6.3 Biorefinery

Biomass revalorization via the conversion into value-added products and fuel is the main goal of a biorefinery, which is considered a sustainable process. The productivity maximization of intermediates and products is reached when an optimal sequence of multifunctional processes is integrated into the biorefinery. Then, the economics of waste revalorization is enhanced. The biorefinery uses several techniques and treatment methods for biomass conversion such as fermentation, extraction, hydrolysis, transesterification, and pyrolysis. It is important to point out that biological processes could also be used (fermentation, anaerobic digestion, etc.). A biorefinery could use the separation processes and unit operations of a petrochemical complex [3]. However, a biorefinery is highly dependent on biomass composition, availability, and the economic value of bioproducts obtained [3, 60, 69].

A biorefinery could be an efficient method for obtaining valuable products from SCG due to its elemental composition; chemical composition (oil content, fatty acid, carbohydrates, carbonaceous and nitrogen compounds, etc.); low cost; high availability; and calorific value. The SCG could produce several value-added products (biosorbent, green composite, antioxidants, polyols, carotenoids, polyphenols, polyhydroxyalkanoates, polyurethane foam, Chlorogenic acid, tannins, activated carbon, PHA, caffeine, etc.) and bioenergy (biogas, biodiesel, and bio-oil). Attabani et al. proposed a biorefinery process using SCG as feedstock for obtaining biofuel, bioethanol, biogas, bio-oil, H<sub>2</sub>, biodiesel, fuel pellets, biochar, polymers,



compost, adsorbent, bioactive compounds, and pharmaceutical products [72]. The production of xylitol, activated carbon, phenolic acid, lactic acid, and heat using brewer's spent grain as the feedstock of a biorefinery [60, 85]. The process sustainability of biorefinery was demonstrated, thanks to the economic margin (62.25%), the potential environmental impact (0.012 PEI/kg products), and the carbon footprint (0.96 kg CO<sub>2</sub>-e/kg of BSG).

## 7. Experimental design to process optimization

Response surface methodology (RSM) is a methodology used to improve process via very few essays, reducing cost and time. The RSM uses statistical and experimental design tools to obtain an optimal response, which is useful for making the right decision. The process performance is very complex due to numerous parameters that affect their behavior. RSM allows built process behavior maps based on mathematical models containing the significant parameters to achieve the maximum, target or minimum process performance.

The optimization of complex processes locates the best experimental conditions at which the process presents the minimum or maximum performance (yield, efficiency, etc.). The use of experimental design for optimizing processes has several advantages: less treatment time, low cost, and efficient use of resources, such as materials, equipment, and workforce. Besides, it uses tools of numerical regression to fit the data to mathematical models to predict values on the region of studied factor levels.

### 7.1 Use of experimental design on coffee waste

The experimental design has been used to optimize the extraction conditions of coffee parchment waste (CP) [86], antioxidant phenolic compounds from coffee silverskin (CS) [8], total phenolic compound and caffeine from SCG [86], coffee oil from SCG [87], the removal conditions of free fatty acid of SCG [88], the conditions to reducing sugar from SCG [89], organic acids [90] and alcohol production from coffee waste [91], and the conditions for the quantification of heavy metals (Cd(II) and Pb(II)), where a carbon-paste electrode modified with SCG was used as a working electrode [92].

#### 7.1.1 Type of experimental designs

The experimental design tools most used are the central composite design, the Box-Behnken design, and the Plackett-Burman design.

Box-Behnken experimental design was used by Mirón-Mérida et al. [86] to maximize the extract yield, total phenolic content, antioxidant activity, and caffeine content on CP simultaneously. The effects of three parameters on the responses were studied: liquid/solid ratio (10, 30, and 50), extraction temperature (45, 60, and 75°C), and ethanol percentage (50, 75, and 100%). The maximum extract yield of 2.36% was achieved at 75°C with 66.76% ethanol as a solvent and with 50 of liquid/solid ratio. The maximum caffeine extracted was 1.513 g caffeine kg<sup>-1</sup> CP at 74.35°C and 69.64% ethanol with 33.47 of liquid/solid ratio. The highest total phenolic content of 2.84037 g gallic acid kg<sup>-1</sup> CP was obtained at 14.33 liquid/solid ratio, 70.74% ethanol, and 75°C. For the maximum extraction of 12.69 μmol Trolox g<sup>-1</sup>, CP of antioxidant activity was attained at liquid/solid ratio of 50, temperature of 75°C, and ethanol of 59.47%. Finally, the optimal extraction conditions were established at 75°C with 41 liquid/solid ratio using 70% of aqueous ethanol as solvent.

Ballesteros et al. [8] used a 23 face-centered central composite design to maximize the extraction of antioxidant phenolic compounds and oxidant activity from CS. The effects of ethanol concentration (20 and 90%), solvent/solid ratio (10 and 40 ml/g), and extraction time (90 and 30 min) were studied on the two responses. The highest phenolic compounds of 13 mg gallic acid equivalents/g CS, with the maximum antioxidant activity of 18.24  $\mu\text{mol}$  Trolox equivalents/g CS and 0.83 mmol Fe(II)/g CS, were achieved at 60% ethanol as solvent, a ratio of 35 ml/g CS dry matter, during 30 min at 60–65°C [8].

Shang et al. [87] developed a two-stage experimental statistical analysis to optimize extraction conditions for total phenolics (mg/g) and caffeine (mg/g) from SCG. First, the process parameter was screened through a Plackett-Burman experiment design to identify the significant parameters of the pressurized liquid extraction method that affect the extraction efficiency, using six parameters at two levels: temperature (80 and 160°C), the concentration of ethanol in water (25 and 75%), extraction time (5 and 20 min), pressure (500 and 2500 psi), sample loading weight (0.5 and 2.5 g), and flush (20 and 100%). The most critical parameters affecting total phenolics and caffeine extraction were temperature and sample loading weight, at 95°C and 0.8 g, respectively. In the second optimization stage, a second-order central composite experimental design, employing the two significant parameters, was used to maximize the total phenolics and caffeine. The highest total phenolic compounds of 22.91 mg/g and caffeine extraction of 9.66 mg/g were achieved with 0.8 g sample loading weight at 195°C.

Pichai and Krit [88] applied response surface methodology to optimize the effects on the coffee oil yield for the solvent extraction process of the ratio of DSCG-hexane (1:8–1:22 g/g) and extraction time (6–34 min). According to the optimal conditions of 1:22.5 g/g mass ratio of DSCG-to-hexane and 30.4 min of extraction time under the 30°C of room temperature, the highest coffee oil yield estimated (14.75 wt%) and experimental (14.68 wt%) was reached.

Mueanmas et al. [89] used a central composite design to investigate the effect on the FFA removal percentage of the mole ratio (5–15) of MeOH-free fatty acid (FFA), the quantity of catalyst (5–15 wt%), the reaction temperature (50–70°C), and the reaction time (30–120 min). The maximum predicted (95.06%) and experimental (93.88%) of FFA removal was attained at 9.1:1 mol ratio of MeOH/FFA with 11.7 wt% of catalyst and 97.2 min of reaction time at 65°C.

Ravindran et al. [90] proposed a central composite design to maximize the reducing sugar yield of SCG, after enzymatic saccharification of pretreated biomass and ultrasound-assisted potassium permanganate oxidation. The effects of five parameters on the responses were studied: 77.08 FPU/mL of cellulase (biomass loading 1–5 g/50 ml), 72.23 U/mL of hemicellulase (biomass loading 0.3–1.5 ml/50 ml), pH (4.8–6.6), and incubation time (24–120 h). A maximum reducing sugar yield of 35.64 mg/mL of reaction volume was estimated with a high biomass loading of 5 g/50 mL, 1.5 mL/50 mL of cellulase, 0.37 mL/50 mL of hemicellulase, pH 6.7, and a low incubation time of 24 h. The experimental values obtained using the optimized parameters are in the range of total reducing sugar of  $35.15 \pm 0.2$  mg/mL.

Montoya et al. [91] developed a Plackett-Burman design to evaluate the effect of the parameters on H<sub>2</sub>, organic acids, and alcohol production from coffee waste. The coffee waste was pretreated using a consortium of bacteria and fungi (indigenous from coffee waste) with hydrolytic and fermentation activity in a hydrothermal reactor. The parameters of pH (4.0–7.0), temperature (30–50°C), agitation (0–180 rpm), headspace (50–70%), percentage of bioaugmentation (without microbial consortium to 20%), the concentration of coffee pulp and husk (2–6 g/L), coffee processing wastewater (7–30 g COD/L), and yeast extract (0–2 g/L) were studied. Under the optimum conditions of 30°C, 180 rpm, 50% headspace, without

bioaugmentation, 2 g/L pulp and husk coffee, 30 gCOD/L coffee processing wastewater, and 2 g/L yeast extract, estimated production of 82 ml H<sub>2</sub> was achieved.

Finally, Estrada-Aldrete et al. [92] applied a central composite design to optimize the quantification of two heavy metals (Cd(II) and Pb(II)) at trace levels using a paste carbon electrode of spent coffee grounds, which was chemically modified by citric acid. The metal quantification was carried out by differential pulse anodic stripping voltammetry technique. The electrodeposition potential (−1200, −950, and −700 mV) and accumulation time (30, 75, and 120 s) were employed as design parameters. The optimal conditions to achieve the maximum Pb(II) anodic peak current of  $2.09 \times 10^{-4}$  A were −1200 mV electrodeposition potential and 120 s accumulation time. The maximum Cd(II) anodic peak current of  $1.385 \times 10^{-3}$  A obtained at −1155 mV potential and 76 s time.

## 8. Conclusion

Coffee waste is widely available, and while it is being disposed of as domestic or industrial garbage, it represents a vital source to obtain valuable products and energy. Physico-chemical properties of coffee waste allow their revalorization in various applications, highlighting as a feedstock of biorefinery, due to the presence of useful chemical compounds; as a raw material in the synthesis of activated carbon, given the predominance of carbon; or applied directly as a biosorbent in pollutant removal from gas or liquid, thanks to its surface characteristics. The implementation of environmentally friendly processes based on coffee waste requires a deepening knowledge of the physico-chemical properties.

Coffee wastes are low-cost adsorbents for the removal of organic and inorganic pollutants from aqueous solutions in batch systems. However, more studies are needed to fully characterize the performance of coffee waste in continuous systems as fixed-bed columns to scale-up the process. Since coffee waste was found to be efficient in the removal of ozone, it is expected that future studies will focus on the application of coffee wastes in the removal of gaseous pollutants.

SCG activated carbon could be used in the adsorption process for removing organic and inorganic pollutants from aqueous solutions. According to recent literature analyzed, the activated carbon or biochar obtained from SCG shows excellent properties to be used as adsorbent materials, such as high surface area, wide pore, and total pore volume. Most of researchers have used an electric furnace to perform the carbonization process, which requires high power consumption; this represents an environmental liability because this production process leads to air pollution by greenhouse gases. Thus, it is necessary to increase the studies of the use of microwaves in the carbonization process. This technology requires a low time to perform the carbonization. Therefore, a low power consumption is needed.

An experimental design is a powerful tool to optimize systems where the mathematical relationships between the parameters and the process performance are unknown. Some attempts have been made to use them on the processing of coffee. However, it is necessary to use them to obtain optimal conditions for the recovery of valuable compounds on mono-process extraction before the implementation of a biorefinery.

Experimental design methodology could help to obtain a sustainable process not only in the revalorization of coffee waste but also in all the stages of coffee processing.

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