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*Article*

# One-Step CO<sub>2</sub> Assisted Pyrolysis as an Efficient Route for Producing Highly Porous Biochar from Spent Coffee Grounds

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## Abstract

Spent coffee grounds (SCG) are an abundant, carbon-rich residue suitable for converting into biochar through a thermochemical process. In this study, biochar was produced from SCG using a two-step pyrolysis-CO<sub>2</sub> activation process and a one-step CO<sub>2</sub> assisted pyrolysis method to compare their physicochemical and structural properties. Results showed that CO<sub>2</sub> activation significantly increases the porosity and surface area, from 9.8 m<sup>2</sup>/g for non-activated biochar (BCK) to 550.6 m<sup>2</sup>/g for BCK-CO<sub>2</sub> and 671.0 m<sup>2</sup>/g for SCG-CO<sub>2</sub>. FTIR and Boehm titration analyses confirmed a decrease in surface oxygenated groups after CO<sub>2</sub> treatment, and SEM-EDX and XRD analyses indicated a structural reorganization. The one-step CO<sub>2</sub> assisted pyrolysis produces biochar with a more uniform pore structure, a higher degree of carbonization, and better textural properties than the two-step method. Therefore, using CO<sub>2</sub> directly during pyrolysis can be an efficient and sustainable method to obtain highly porous biochar from spent coffee grounds, reduce energy demands, and make valuable organic waste.

**Keywords:** biochar; spent coffee grounds; one-step CO<sub>2</sub> activation; surface area; porous carbon; waste valorization

## 1. Introduction

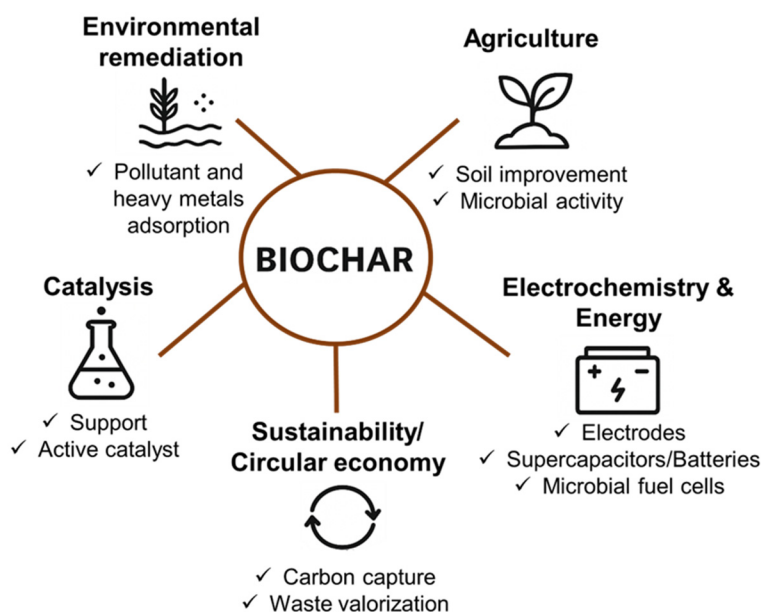
Biochar is a carbonaceous material obtained from biomass through thermochemical processes including pyrolysis, hydrothermal carbonization, and gasification [1,2]. Due to its unique physicochemical and structural properties, biochar is a versatile material used in environmental remediation, agriculture, catalysis, and electrochemical applications [3,4].

In environmental remediation, biochar improves soil and water quality by adsorbing heavy metals [5], organic pollutants [6,7], excess nutrients [8], and emerging contaminants [9]. It also improves soil properties by increasing porosity and water retention [10,11], promotes microbial activity and soil fertility [12–14]. In catalysis, biochar is used both as a support for metal nanoparticles, improving their dispersion, stability, and reusability [15,16], and as an active catalyst due to its surface functional groups [17]. This includes reactions such as biodiesel production [18] through transesterification [19], biomass conversion [20], and chemical transformations, including hydrolysis, isomerization, and dehydration [16]. In electrochemical applications [21], its high porosity and electrical conductivity make it a suitable electrode material for supercapacitors [22], batteries [23], electroanalytical sensors [24], and microbial fuel cells, promoting electron transfer and energy generation [25]. Biochar contributes to sustainability, enabling carbon capture and storage [26,27], waste valorization [28], and supporting circular economy strategies [29,30]. It is a stable medium for atmospheric carbon sequestration [31], is an efficient CO<sub>2</sub> adsorbent [32], and contributes to circular

and climate-resilient industries [33]. An overview of the main applications of biochar is illustrated briefly in Figure 1.

Biochar production from renewable biomass residues supports circular bioeconomy principles and enhances waste management strategies [34]. By converting organic waste into a valuable and stable carbon form, biochar keeps biomass out of landfills and incineration [35], sequesters carbon in soils, and reduces greenhouse gas emissions [36,37].

A wide variety of biomass feedstocks has been investigated for biochar production, including agricultural residues (e.g., crop stalks and straws [38], husks [39], sugarcane bagasse [40], and fruit or vegetable wastes [41]), seed-derived materials such as soy hulls [42] and sunflower residues [43], forestry byproducts including sawdust, bark, and branches [44].



**Figure 1.** Schematic representation of the main application of biochar.

With an estimated global production of 60 million tons annually [45], spent coffee grounds (SCG) are known for their high carbon and volatile matter content and are rich in bioactive compounds such as cellulose, lignin, proteins, lipids, and polyphenols [46,47]. When discarded in landfills, it contributes to methane emissions and environmental problems [48,49]. Converting SCG into activated biochar not only prevents these negative impacts but also creates valuable materials with environmental and technological applications.

The quality and performance of biochar depend on its surface area, pore structure, and surface functional groups [50,51]. Activation is essential in biochar production, as non-activated biochar exhibits limited surface area and low porosity [52,53]. Two main activation strategies are commonly employed: (i) physical activation using oxidizing gases such as  $\text{CO}_2$  or steam at high temperatures [53,54], and (ii) chemical activation using reagents such as acids ( $\text{HNO}_3$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ ,  $\text{H}_3\text{PO}_4$ , or organic acids like citric and oxalic acid [55,56]), alkalis ( $\text{KOH}$ ,  $\text{NaOH}$  [57,58]), metallic salts ( $\text{ZnCl}_2$ ,  $\text{FeCl}_3$  [59]), or oxidizers like  $\text{H}_2\text{O}_2$  [60,61]. While chemical activation generally produces biochar with higher surface areas and more developed porosity [62], it involves corrosive reagents that generate hazardous waste and are challenging to recover [63]. In contrast, physical activation is simpler, cleaner, and easier to scale up, since it depends on a controlled gas-solid reaction [64,65]. Carbon dioxide is favored in physical activation because it is economical [66], avoids harmful chemicals, and allows valorization of greenhouse gas, contributing to circular carbon management [67,68]. Physical activation with  $\text{CO}_2$  is therefore widely recognized as a greener alternative, producing high-porosity biochar, suitable for adsorption and catalytic applications [69,70].

CO<sub>2</sub> activation is generally carried out in a two-step process, where pyrolysis and activation are performed sequentially: the biomass is first pyrolyzed under an inert gas (e.g., N<sub>2</sub> or Ar) and then activated under a CO<sub>2</sub> flow at high temperatures [71]. Alternatively, one-step activation uses CO<sub>2</sub> as an activating agent, combining biomass pyrolysis and activation in a single process [72,73]. Despite its economic and environmental advantages, one-step CO<sub>2</sub> activation has received relatively limited scientific attention. Available studies indicate its potential to integrate pyrolysis and activation in a single step; however, further research is required to explore its applicability for diverse biomass and to optimize process parameters [66].

In this study, we present a comparative investigation of conventional two-step and integrated one-step CO<sub>2</sub> assisted pyrolysis to elucidate the effect of activation mode on the structural and textural properties of biochar and to better understand the CO<sub>2</sub> assisted carbon formation. Spent coffee grounds (SCG) were chosen as a representative feedstock due to their availability, uniform composition, and high carbon content. Using CO<sub>2</sub> as the activating agent not only promotes pore development but also provides a way to valorize a greenhouse gas. This work demonstrates that one-step CO<sub>2</sub> assisted pyrolysis can produce biochar with physicochemical properties comparable to or even superior to those obtained in the two-step process, reducing time and energy requirements, and offering a more efficient and sustainable route for biochar production.

## 2. Materials and Methods

### 2.1. Production of Biochar from Spent Coffee Grounds

Spent coffee grounds (SCG) collected from a coffee machine used in our workplace were dried for 4 h at 150 °C. The dried SCG was introduced into a tubular quartz reactor and subjected to pyrolysis in a Nabertherm RHTC 80-230/15 furnace at 850 °C for 2 h under an inert gas atmosphere. The resulting biochar was labeled as BCK. Then, this biochar was activated in a CO<sub>2</sub> flow of 200 mL min<sup>-1</sup> at 850 °C for 1.5 h, and the sample was denoted as BCK-CO<sub>2</sub>. The one-step CO<sub>2</sub> pyrolysis-activation was performed under identical conditions (850 °C, 2 h, CO<sub>2</sub> flow rate 200 mL min<sup>-1</sup>), and the biochar sample was named SCG-CO<sub>2</sub>.

### 2.2. Determination of Biochar Yield and Proximate Composition

The biochar yield was determined as the ratio between the mass of the obtained biochar and the initial dry mass of the biomass, expressed as a percentage. The ash content (ASH) of each biochar sample was measured according to the ASTM D3174 standard by combusting 1 g of biochar in an open crucible at 750 °C for 6 h in a muffle furnace (Nabertherm LE4/11/R6). The volatile matter content (VM) was determined following the ASTM D1762-84 procedure by combusting 1 g of biochar in a closed crucible at 950 °C for 10 min in a muffle furnace (Nabertherm LE4/11/R6), and calculating the mass loss corresponding to volatile components. The fixed carbon content (FC) was calculated by the difference between 100 and the sum of ASH and VM content using the following equation:

$$FC = 100 - (ASH + VM) \quad (1)$$

The thermal stability (TS) of biochar was estimated as the ratio between fixed carbon and the sum of fixed carbon and volatile matter, as shown in the equation:

$$TS = \frac{FC}{FC + VM} \quad (2)$$

### 2.3. Surface Characterization of Biochar

N<sub>2</sub> sorption analysis was performed at -196°C (BelSorp MaxX, Microtrac BEL Corporation, Japan), after sample preparation by degassing at 200°C under vacuum for 4 h. The standard BET method was used to evaluate the specific surface area (p/p<sub>0</sub> range of 0.025 – 0.25), total pore volume

was estimated at  $p/p_0 = 0.95$ , and pore size distribution was analyzed by the Barrett-Joyner-Halenda (BJH) method from the desorption branch.

Functional groups present in the biochar were identified by Fourier-transform infrared (FTIR) spectroscopy using a JASCO 6100 FT-IR spectrometer (JASCO International Co., Ltd., Tokyo, Japan) in the 4000 to 400  $\text{cm}^{-1}$  spectral domains with 4  $\text{cm}^{-1}$  resolution, using the KBr pellet technique. Each sample has been dispersed in about 300 mg of anhydrous KBr and mixed in an agate mortar; the mixtures were pressed into an evacuated die. The collection and analysis of spectral data was carried out using Jasco Spectra Manager v.2 software.

Additionally, the surface acidic and basic functional groups of the biochar were quantitatively determined by Boehm titration [74,75]. For the determination of acidic sites, 0.5 g of biochar was added to 50 mL of 0.1 M NaOH solution, while for basic sites, 0.5 g of biochar was added to 50 mL of 0.1 M HCl solution. The suspensions were shaken for 48 h at room temperature to ensure complete interaction between the solid and liquid phases. After equilibration, the samples were filtered, and aliquots of the filtrates were titrated: the remaining NaOH with 0.1 M HCl, and the remaining HCl with 0.1 M NaOH. The total amounts of acidic and basic groups were calculated from the volumes of titrant required to neutralize the remaining base or acid, respectively.

The surface characteristics and elemental composition of the biochar samples were analyzed by scanning electron microscopy (SEM) and EDS analysis, using a Hitachi SU8230 microscope, with an electron acceleration of 30 kV (Hitachi Ltd., Tokyo, Japan), equipped with an EDX spectrometer, operated with Aztec software version 5.1 (Oxford Analytics, Oxford, UK).

The structural order of the biochar samples was evaluated by X-ray diffraction (XRD). Measurements were performed at room temperature using a Rigaku SmartLab multipurpose diffractometer equipped with a 9 kW rotating anode and Cu  $K\alpha_1$  radiation ( $\lambda = 1.54056 \text{ \AA}$ ). Data acquisition was carried out with SmartLab Guidance software. Before analysis, the samples were finely ground in an agate mortar and pestle to ensure homogeneity and then loaded into the sample holder. Diffraction patterns were recorded over a  $2\theta$  range of 5–75° with a step size of 0.01°.

3. Results and Discussion

3.1. Determination of Biochar Yield and Proximate Composition

The biochar yield from spent coffee grounds (SCG) was 20%, in agreement with literature reports [76,77]. Subsequent CO<sub>2</sub> activation of the pre-formed biochar (BCK-CO<sub>2</sub>) resulted in a yield of 60% relative to the initial biochar, whereas one-step CO<sub>2</sub> assisted pyrolysis of SCG (SCG-CO<sub>2</sub>) produced a lower yield of 16%. This decrease reflects a higher reactivity of CO<sub>2</sub> and the simultaneous occurrence of devolatilization and gasification during the one-step process [66].

CO<sub>2</sub> activation led to a slight increase in ash content (from 7.7% in BCK to 8.4% in BCK-CO<sub>2</sub> and 9.6% in SCG-CO<sub>2</sub>), due to the concentration of inorganic residues [78]. Volatile matter (VM) also increased progressively (11.9%, 14.4%, and 17.3%, respectively), showing the formation of a more porous and reactive carbon structure. This behavior is attributed to the gasification process, which promotes the release of light organic fragments during heating [79,80]. The increase in VM correlates with the observed increase in surface area and pore volume of the activated samples (Table 1). In contrast, fixed carbon decreased from 80.4% in BCK to 77.2% in BCK-CO<sub>2</sub> and 73.1% in SCG-CO<sub>2</sub>. A similar trend is observed for thermal stability, which decreases from 0.87 in BCK to 0.84 in BCK-CO<sub>2</sub> and 0.80 in SCG-CO<sub>2</sub>.

Table 1. Proximate composition and surface characteristics of the biochar samples.

| Sample | Proximate composition |        |        |    | Textural characterisation                         |                                                  |                     | Surface acid-base properties |                         |
|--------|-----------------------|--------|--------|----|---------------------------------------------------|--------------------------------------------------|---------------------|------------------------------|-------------------------|
|        | ASH (%)               | VM (%) | FC (%) | TC | S <sub>BET</sub> <sup>a</sup> (m <sup>2</sup> /g) | V <sub>p</sub> <sup>b</sup> (cm <sup>3</sup> /g) | d <sub>p</sub> (nm) | Total acidity (mmol/g)       | Total basicity (mmol/g) |

|                     |     |      |      |      |        |       |     |     |     |
|---------------------|-----|------|------|------|--------|-------|-----|-----|-----|
| BCK                 | 7.7 | 11.9 | 80.4 | 0.87 | 9.75   | 0.005 | -   | 4.6 | 2.2 |
| BCK-CO <sub>2</sub> | 8.4 | 14.4 | 77.2 | 0.84 | 550.59 | 0.291 | 4   | 3.2 | 1.9 |
| SCG-CO <sub>2</sub> | 9.6 | 17.3 | 73.1 | 0.80 | 671.04 | 0.331 | 3.6 | 3.2 | 1.9 |

3.2. Surface Characterization of Biochar

The N<sub>2</sub> adsorption-desorption isotherms and corresponding BJH pore size distributions of the biochar samples are presented in Figure 2, while the main textural parameters are summarized in Table 1. The non-activated biochar (BCK) shows a very low specific surface area (9.8 m<sup>2</sup>/g) and negligible pore volume (0.005 cm<sup>3</sup>/g), indicating that the porosity generated during pyrolysis at 850 °C was limited. After CO<sub>2</sub> activation, a remarkable increase in surface area and pore volume was observed, reaching 550.6 m<sup>2</sup>/g and 0.291 cm<sup>3</sup>/g for BCK-CO<sub>2</sub> and 671.0 m<sup>2</sup>/g and 0.331 cm<sup>3</sup>/g for SCG-CO<sub>2</sub>, respectively. The isotherms of the activated biochar display a typical type IV(a) shape according to the IUPAC classification [81], with an evident H4-type hysteresis loop, which is characteristic of mesoporous materials containing both micro- and mesopores formed during the activation process. The sharp increase in nitrogen uptake at low relative pressures ( $P/P_0 < 0.1$ ) indicates the presence of micropores, while the hysteresis loop at higher relative pressures ( $0.4 < P/P_0 < 0.9$ ) is associated with capillary condensation in mesopores. The pore size distribution curves (Figure 2, right) confirm the predominance of narrow mesopores centered around 3.6-4 nm, in agreement with the BJH mean pore diameters. The slightly higher specific surface area of SCG-CO<sub>2</sub> compared to BCK-CO<sub>2</sub> suggests that the one-step process was more effective in developing the pore structure than the two-step pyrolysis-activation of preformed biochar.

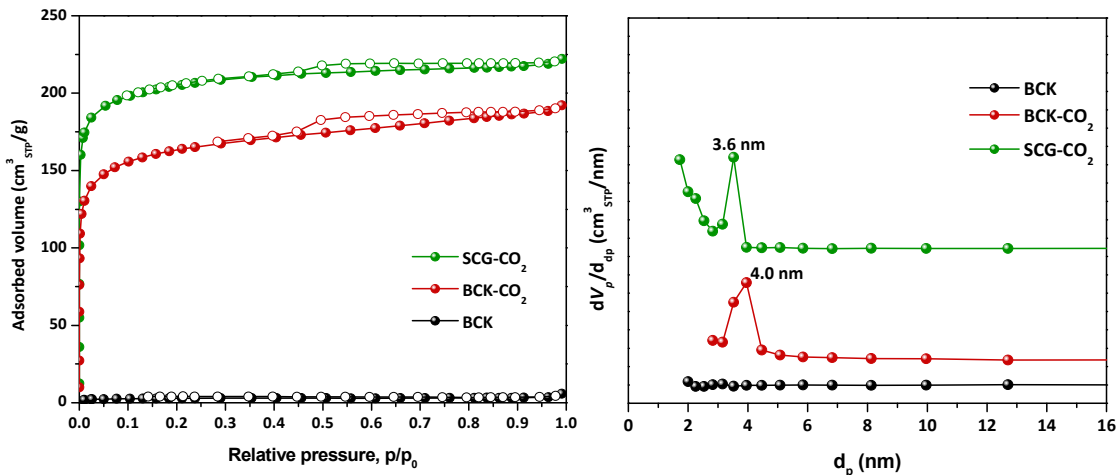
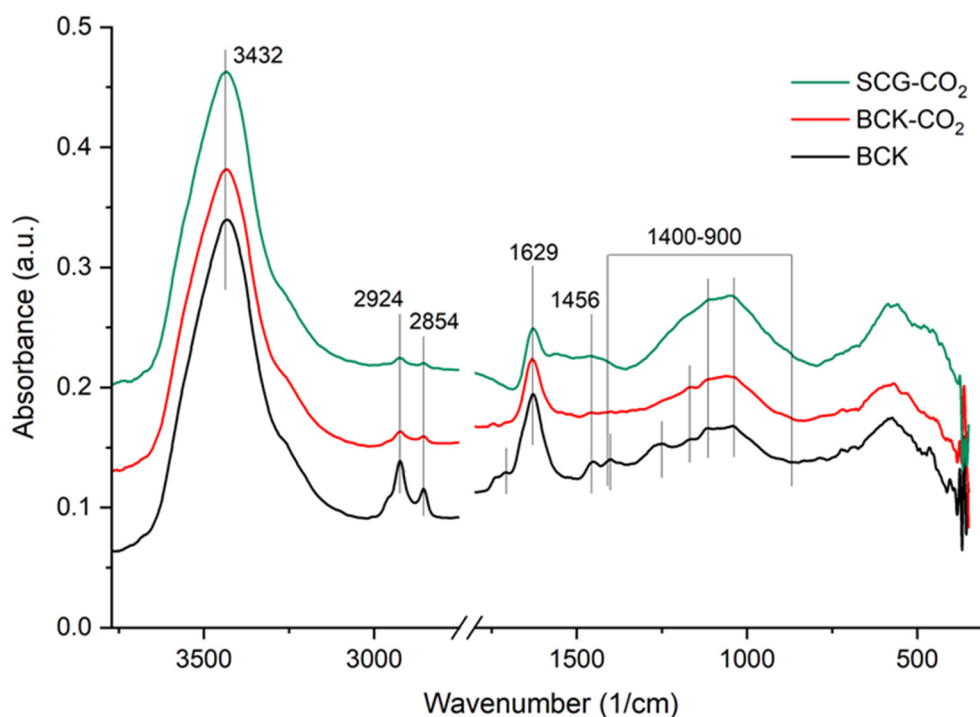


Figure 2. N<sub>2</sub> adsorption-desorption isotherms (a) and pore size distribution (b) for biochar samples.

The FTIR spectra of the biochar samples, shown in Figure 3, exhibit a broad and intense absorption band in the 3600–3000 cm<sup>-1</sup> region, with a maximum at 3432 cm<sup>-1</sup>, corresponding to the stretching vibrations of –OH and N–H groups from water, alcohols and/or phenols, carbohydrates, carboxylic structures, and amino acids [82,83]. The bands at 2924 and 2854 cm<sup>-1</sup> are attributed to the asymmetric and symmetric stretching vibrations of aliphatic CH<sub>3</sub> and CH<sub>2</sub> groups [82,84]. The weak band at 1711 cm<sup>-1</sup> is assigned to amide C=O vibrations, while the medium-intensity band at 1629 cm<sup>-1</sup> is associated with –O–H and C=C vibrations [83]. The absorption at 1456 cm<sup>-1</sup> corresponds to aromatic C=C stretching and asymmetric stretching of COO– groups [85,86].

The spectral region between 1400 and 900 cm<sup>-1</sup> is characterized by bands related to C–O, C–H, and C=C stretching, bending vibrations of C–O–H, C–O–C, and C–C–H groups, as well as stretching vibrations of C–C, C–O, and C–O–H in saccharides [82,86]. Compared with BCK, the spectra of BCK-CO<sub>2</sub> and SCG-CO<sub>2</sub> show decreased intensity and broadening of the resolved bands, indicating an enhancement of the amorphous character of the biochar after CO<sub>2</sub> treatment. Furthermore,

comparison between BCK-CO<sub>2</sub> and SCG-CO<sub>2</sub> reveals a decrease in the intensity of the 1629 cm<sup>-1</sup> band and an increase in the intensity of the broad band in the 1400–900 cm<sup>-1</sup> region.



**Figure 3.** FTIR spectra of biochar samples.

The surface acid and basic functional groups of the biochar samples, determined by Boehm titration, are summarized in Table 1. The non-activated biochar (BCK) shows a total acidic group of 4.6 mmol/g and a total basic group of 2.2 mmol/g. After CO<sub>2</sub> activation, both concentrations of acidic and basic groups decreased to 3.2 mmol/g and 1.9 mmol/g, respectively, for BCK-CO<sub>2</sub> and SCG-CO<sub>2</sub>. Since both pyrolysis and activation were performed at the same temperature (850 °C), this decrease cannot be attributed to thermal degradation of surface functionalities, but rather to gas–solid reactions between CO<sub>2</sub> and the carbon matrix [87]. The reaction ( $C + CO_2 \rightarrow 2CO$ ) consumes less stable oxygenated groups (e.g., carboxylic, lactonic, phenolic), “cleaning” the biochar surface and generating new pores [88]. Therefore, the SCG-CO<sub>2</sub> sample shows higher surface areas and pore volumes, despite a lower total concentration of surface functional groups.

This interpretation is supported by the FTIR spectra of the biochar samples (Figure 3). Compared with BCK, both BCK-CO<sub>2</sub> and SCG-CO<sub>2</sub> show a decrease in intensity and broadening of the absorption bands, suggesting a lower abundance of oxygen-containing surface groups. The attenuation of the band at 1629 cm<sup>-1</sup> (–O–H and C=C vibrations) and the relative strengthening of the broad band in the 1400–900 cm<sup>-1</sup> region (C–O and C–C stretching in aromatic and saccharide structures) confirm the partial removal or transformation of surface oxygen groups observed in the Boehm titration results. Together, the FTIR and Boehm analyses indicate that CO<sub>2</sub> activation increases porosity and modifies surface chemistry, producing biochar with higher surface area and improved textural properties, but with a reduced number of reactive acidic and basic sites.

The surface morphology and elemental composition of the spent coffee grounds (SCG) and the corresponding biochar samples were analyzed by scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM–EDX), as shown in Figure 4. A representative SEM image of raw SCG was included for comparison, illustrating the morphological transformation of the biomass after pyrolysis and activation. The raw SCG show a compact and irregular structure with partially collapsed cell walls and limited visible porosity, typical of lignocellulosic residues [89]. After pyrolysis at 850 °C (BCK), the structure became more rigid and carbonaceous, with the formation of the pores due to the decomposition and volatilization of organic components. Both activated samples

(BCK-CO<sub>2</sub> and SCG-CO<sub>2</sub>) show highly developed and interconnected pore networks with a characteristic honeycomb texture, in contrast to the dense morphology of the raw SCG. This confirms that CO<sub>2</sub> treatment effectively promotes pore formation through gasification reactions ( $C + CO_2 \rightarrow 2CO$ ), which gradually open up the carbon framework and generate new micro- and mesopores [90]. SCG-CO<sub>2</sub> exhibited a more open and uniformly porous structure than BCK-CO<sub>2</sub>, suggesting that simultaneous pyrolysis and activation facilitate a more homogeneous pore system. These morphological observations are consistent with the BET results, which showed higher specific surface area and pore volume for SCG-CO<sub>2</sub>.

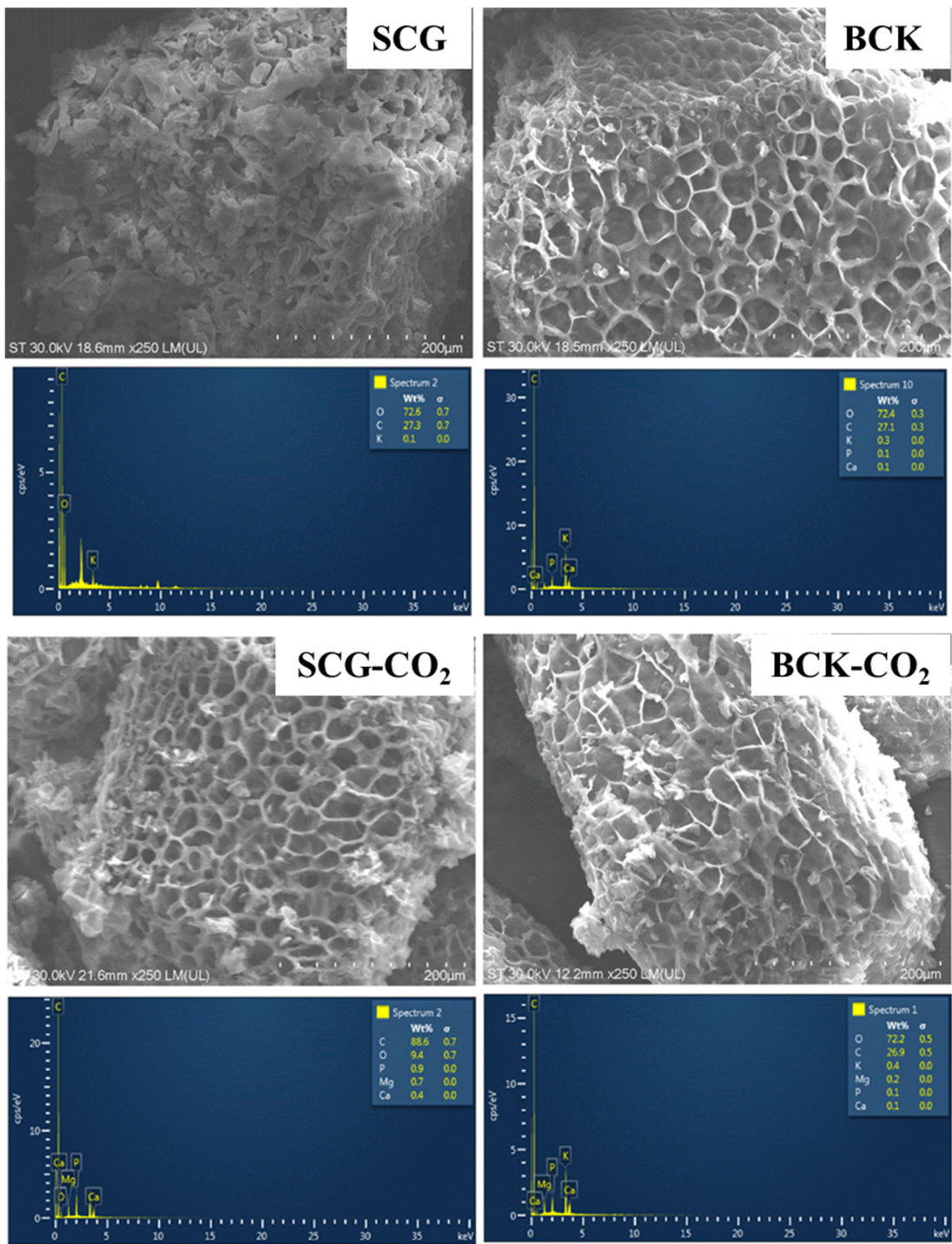
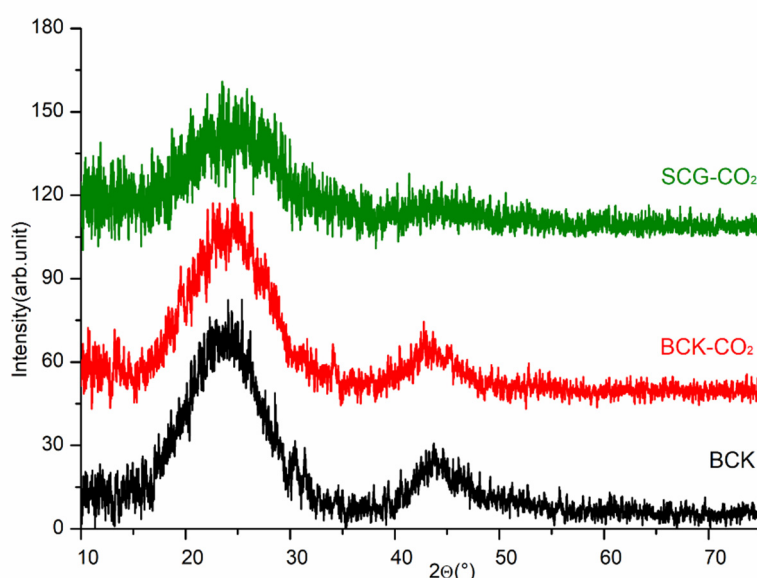


Figure 4. SEM micrographs and corresponding EDX spectra.

The EDX spectra show that carbon and oxygen are the main elements in all samples. Small amounts of inorganic components such as K, Mg, Ca, and P are also identified, originating from the natural minerals in the coffee biomass. After activation, the relative content of these elements increased slightly, due to the concentration of residual ash as some of the carbon was removed during gasification.

The SEM-EDX results are consistent with the trends observed in the BET and FTIR analyses, showing that CO<sub>2</sub> activation leads to a clear structural reorganization and an increase in carbon content at the biochar surface. These changes give a highly porous material suitable for adsorption and catalytic applications.

The X-ray diffraction (XRD) patterns of the biochar samples (BCK, BCK-CO<sub>2</sub>, and SCG-CO<sub>2</sub>) are shown in Figure 5. All samples show two broad diffraction peaks around  $2\theta \approx 23-26^\circ$  and  $43-44^\circ$ , corresponding to the (002) and (100) planes associated with turbostratic carbon [91]. The wide and low-intensity peaks suggest that the carbon structure is mostly amorphous, with only limited ordering of graphene-like layers, a common characteristic of biochar obtained from lignocellulosic biomass [92].



**Figure 5.** X-ray diffraction (XRD) patterns of the biochar samples.

The (002) reflection corresponds to the stacking of aromatic layers, while the peak intensity reflects the crystallite size, showing the degree of order present in the graphitic structure of the biochar [93]. The broad and weak feature at  $43-44^\circ$  corresponds to highly defective porous carbons characterized by disordered stacking of graphitic layers [91]. Besides the main peaks, a few weaker crystalline signals were also observed. In the BCK sample, small peaks appeared at around  $2\theta \approx 30-31^\circ$  and  $56^\circ$ , whereas BCK-CO<sub>2</sub> displayed a peak near  $34^\circ$ . These are most likely due to residual inorganic minerals, such as potassium or calcium compounds, left from the original coffee biomass [94].

The XRD results show that all samples are predominantly composed of disordered, amorphous carbon with a small graphitic structure. Variations in peak shape and intensity indicate that CO<sub>2</sub> activation increases structural disorder and promotes the formation of a more defect carbon network, in agreement with the enhanced porosity observed in the BET analysis.

To provide a comparative perspective, Table 2 highlights the key differences between the two-step pyrolysis - CO<sub>2</sub> activation and the one-step CO<sub>2</sub> assisted pyrolysis employed in this study. Although the one-step process is not novel, our results emphasize its practical advantages over the traditional method. By integrating pyrolysis and activation in a single CO<sub>2</sub> atmosphere step, the process eliminates the need for reheating, reduces energy consumption, and allows continuous CO<sub>2</sub>

interaction throughout pyrolysis. Despite its simplicity, the one-step process generates biochar with highly developed porosity and surface characteristics, indicating that CO<sub>2</sub> serves efficiently as both a reaction medium and an activating agent.

**Table 2.** Comparison between two-step pyrolysis - CO<sub>2</sub> activation and one-step CO<sub>2</sub> assisted pyrolysis.

| Parameter                   | Two-step pyrolysis - CO <sub>2</sub> activation                 | One-step CO <sub>2</sub> assisted pyrolysis               |
|-----------------------------|-----------------------------------------------------------------|-----------------------------------------------------------|
| Process design              | Pyrolysis under inert gas + separate CO <sub>2</sub> activation | Integrated pyrolysis and activation under CO <sub>2</sub> |
| Number of thermal stages    | 2                                                               | 1                                                         |
| Energy demand               | High (requires reheating)                                       | Lower (single heating step)                               |
| CO <sub>2</sub> utilization | Only during activation                                          | During the entire thermal process                         |
| Time efficiency             | Longer (two distinct stages)                                    | Shorter (one-step process)                                |
| Environmental footprint     | Higher (greater energy input)                                   | Reduced (lower energy, CO <sub>2</sub> valorization)      |
| Textural properties         | Relative high surface area                                      | Superior                                                  |

Based on these results, the one-step CO<sub>2</sub> assisted pyrolysis process can be regarded as an efficient and sustainable alternative for biochar activation, providing comparable structural and textural properties while reducing process complexity and energy consumption.

4. Conclusion

The comparative evaluation of two-step pyrolysis - CO<sub>2</sub> activation and one-step CO<sub>2</sub> assisted pyrolysis of spent coffee grounds shows that both methods produce porous biochar. However, the one-step process yielded materials with higher surface area, pore volume, and carbon content, due to the simultaneous pyrolysis and activation in a CO<sub>2</sub> atmosphere. CO<sub>2</sub> treatment increased porosity through gasification reactions and facilitated the removal of unstable oxygenated surface groups, resulting in a more amorphous and more defect carbon network. Overall, these results demonstrate that direct CO<sub>2</sub> assisted pyrolysis is a promising, efficient, and environmentally friendly strategy for converting coffee waste into valuable biochar suitable for adsorption and catalytic applications.

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**Data Availability Statement:** The data presented in this study are available on request from the corresponding author.

**Conflicts of Interest:** The authors declare no conflicts of interest.

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