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## Turning Waste into Value: Comparative Synthesis of Activated Carbon from Non-Recyclable Polystyrene, Corn Stalks and Coffee Grounds

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### Abstract

This study investigates the synthesis of activated carbon (AC) from two distinct waste-derived feedstocks: coffee grounds (CG) and a mixture of non-recyclable polystyrene (PS) with corn stalks (CS) for the first time. The results of study show that the CG precursor applied the hydrothermal carbonization (HTC) at 240 °C for 5 h followed by KOH activation at 650°C, while the PS–CS mixture was subjected to pyrolysis at 500 °C for 1.5 h and activated at 750 °C. Interestingly, despite the milder treatment conditions, CG-derived carbon achieved a good BET surface area of 914.83 m<sup>2</sup> g<sup>−1</sup>, comparable to that of PS–CS-derived carbon (976.34 m<sup>2</sup> g<sup>−1</sup>). The superior pore development in the CG sample is attributed to its high oxygen and nitrogen content, which facilitates dehydration, decarboxylation, and the formation of micropores during HTC. In contrast, PS requires higher activation temperature due to its aromatic and oxygen-deficient nature, although co-pyrolysis with CS promotes synergistic gas evolution and pore widening. These findings highlight that feedstock composition strongly dictates process parameters and pore characteristics, providing insights for designing sustainable routes to produce high-performance activated carbon from diverse waste materials.

**Keywords:** Activated Carbon, Co-Pyrolysis, Corn stover, Polystyrene plastic, ground coffee, biomass

## 1. Introduction

In recent years, biomass-derived carbon materials have been given considerable attention owing to their low cost, environmental compatibility, and renewable nature, particularly when agricultural residues are utilized as precursors [1]. This sustainable approach not only reduces production costs compared to commercial activated carbon but also mitigates pollution and supports circular economy principles [2]. Many works have demonstrated the potential of various biomass sources for synthesizing high-performance porous carbons. For example, Xu et al [3] have studied the corn stalks by the pyrolysis with one-step and two-step chemical activation (KOH/NaOH) shows one-step KOH at 800 °C for 120 min gives richer pore structures and superior electrochemical behavior, illustrating how activation route/agent/temperature govern performance for agricultural residues. De Smedt et al [4] and co-workers have applied seaweed (macroalgae) and used molten-salt (eutectic  $\text{ZnCl}_2\text{-NaCl-KCl}$ ) for activation step that offers a lower-temperature route (400–600 °C) to mesopore-tailored carbons from brown/green seaweeds; best samples achieved  $\text{SSA} \approx 504 \text{ m}^2 \text{ g}^{-1}$  and high dye-adsorption capacities, underscoring eutectic-salt templating as a tunable strategy for diverse wet-biomass feedstocks. The study of Tian et al [5]. approaches using distinctive plant microstructures (e.g., bamboo-based industrial byproducts) have produced hierarchical nanoporous carbons with strong supercapacitor performance, illustrating how inherited tissue architecture can direct porosity and ion transport. Within this context, coffee grounds (CG)—a ubiquitous and inexpensive byproduct in coffee-producing nations—have emerged as a promising feedstock for porous carbon synthesis [6].

Simultaneously, agricultural residues like corn stalks (CS) represent abundant, cellulose-rich biomass suitable for thermochemical valorization into carbonaceous materials [7]. Co-pyrolysis of plastics and lignocellulosic biomass has emerged as a synergistic route for waste valorization, combining hydrogen-rich plastics with oxygen-rich biomass to enhance product yield and quality [8]. Polystyrene (PS) is among the most pervasive plastic pollutants in marine environments, accumulating in major oceanic gyres such as the Great Pacific Garbage Patch [9]. Compared with other polymers like polyethylene and PET, PS more readily fragments into microplastics which have been associated with oxidative stress, inflammation, and metabolic disorders in humans [10]. Moreover, carcinogenic styrene monomers and oligomers can leach from PS microplastics, intensifying health risks.

Regarding the trend of circular economics to turning waste into value, this work studies comparative synthesis of activated carbon from non-recyclable polystyrene, corn stalks, and coffee grounds for activated carbon production for the first time. Depending on the biomass resources as the waste materials the process to synthesis the activated carbon with high surface area will study in detail in this work. The result of this work is to contribute and develop an efficient process that transforms hazardous, low-value waste into functional carbon materials with enhanced surface area and adsorption potential, suitable for applications in environmental remediation and energy systems.

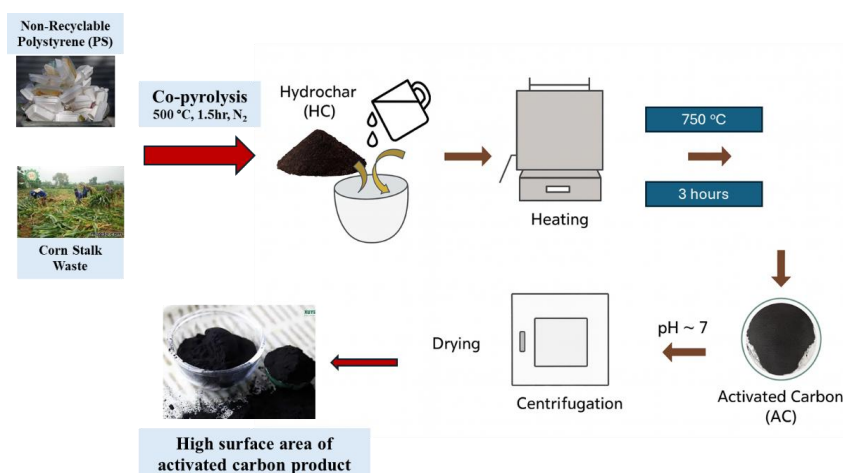
## 2. Materials and Methods

### 2.1. Materials

In this study, coffee grounds were collected from Dak Lak Province, Vietnam. The activating agent, potassium hydroxide (KOH, CAS: 1310-58-3, 85%), was obtained from Xilong Chemical Co., China. Hydrochloric acid (HCl, CAS: 7647-01-0, 36–38%) and acetone (C<sub>3</sub>H<sub>6</sub>O, CAS: 67-64-1, 99.0%) were also purchased from the same supplier. Double-distilled water (CAS: 7732-18-5) was sourced locally in Vietnam. All reagents were of analytical grade and used without further purification.

Polystyrene (PS) waste and corn stalk (CS) residues were collected from municipal and agricultural sources in Ho Chi Minh City, Vietnam. High-purity nitrogen gas (N<sub>2</sub>, 99.99%) supplied by a local distributor was used to maintain an inert atmosphere during the co-pyrolysis and activation processes.

### 2.2. Experiment of Co-pyrolysis process of PS and CS to produce activated carbon

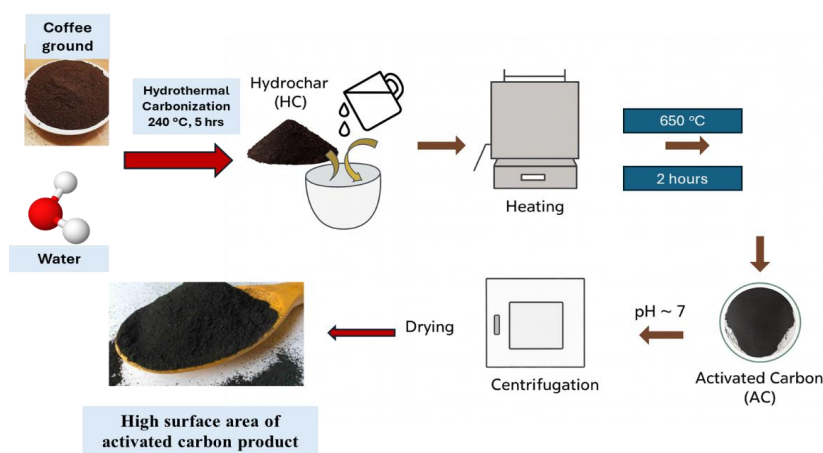


**Scheme 1.** Process of synthesis of Activated Carbon from Non-Recyclable Polystyrene and Corn Stalk Waste

The pre-treatment of the collected polystyrene (PS) waste was thoroughly washed with water, air-dried under sunlight for 24h, and cut them into uniform pieces (0.5 × 0.5 cm). Corn stalks (CS) were cleaned by removing leaves, flowers, and roots, retaining only the stalks, which were washed, chopped, oven-dried at 80 °C for 24 h, and sieved to a particle size of approximately 0.4 × 0.4 cm. The prepared PS and CS samples were stored in a desiccator until use. For co-pyrolysis, PS and CS were carried out the 1<sup>st</sup> step by mixing at a mass ratio of 1:2 (PS:CS) and heated in ceramic crucibles at 500 °C for 1.5 h under a nitrogen atmosphere to ensure inert conditions. After pyrolysis, the crucibles were cooled naturally to room

temperature, and the obtained hydrochar (HC) was collected for subsequent activation. The 2<sup>nd</sup> activation step was performed by mixing the HC with 5 M KOH at a mass ratio of 1:3 (HC:KOH). The mixture was heated in a muffle furnace at temperatures of 750 °C for 3 hours with a heating rate of 16.5 °C min<sup>-1</sup> under a nitrogen flow. After cooling to room temperature, the activated carbon (AC) was washed with 1 M HCl until neutral (pH 7), rinsed several times with deionized water and acetone, and oven-dried at 105 °C for 24 h (**Scheme 2**).

### 2.3. Experiment of fabrication from waste coffee grounds to produce activated carbon.



**Scheme 2.** Experiment of fabrication from waste coffee grounds to produce activated carbon (AC).

The process of fabrication from waste coffee grounds to produce activated carbon are carried out as: Prior to carbonization, the spent coffee grounds were sieved (2 × 2 mm) for uniformity, thoroughly washed with distilled water and acetone to remove impurities, and then dried at 105 °C for 24h. Hydrochar (HC) was produced via hydrothermal carbonization (HTC) as shown in Figure 1. Approximately 9 gram of dried coffee grounds were placed in a 100 mL Teflon-lined autoclave, with moisture adjusted to 80% using double-distilled water. The suspension was ultrasonicated for 30 min to ensure homogeneity before being heated at 240 °C for 5 h. After natural cooling, the obtained hydrochar was washed repeatedly with distilled water (four times) and acetone (twice), centrifuged, dried at 105 °C for 24 h, and stored in a desiccator for further use.

Activated carbon (AC) was synthesized from HC following the procedure illustrated in Figure 2. In a typical run, 2.5 g of HC was impregnated with 5 M KOH solution at a mass ratio of 1:1.5 (HC:KOH) and heated at 650 °C for 2 h under a

nitrogen atmosphere ( $16 \text{ L h}^{-1}$ ). After activation, the gas flow was reduced to  $12 \text{ L h}^{-1}$ , and the furnace was allowed to cool naturally. The resulting product was neutralized with HCl ( $\text{pH} \approx 7$ ), washed several times with distilled water and acetone, and oven-dried at  $105^\circ\text{C}$  for 24 h. The final AC samples were stored in a desiccator prior to characterization (Scheme 2).

#### 2.4. Characterization of biochar and activated carbon (AC)

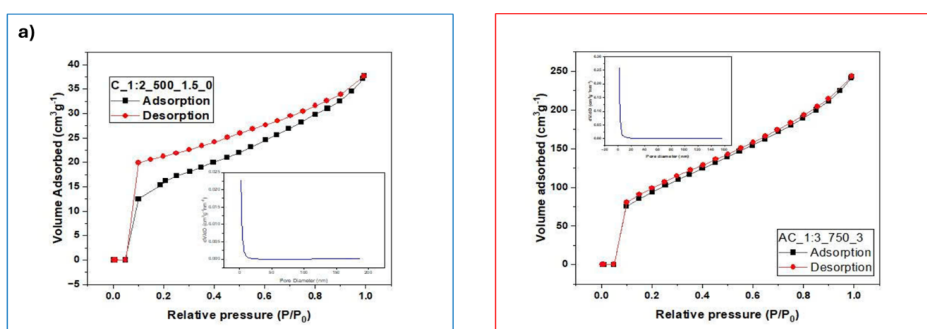
Nitrogen adsorption–desorption analyses were performed at 77 K using a BET Sorptometer (Porous Materials Inc., USA) to determine the textural properties of the samples. Specific surface area was calculated from the adsorption isotherms using the Brunauer–Emmett–Teller (BET) method, while pore volume and pore diameter were derived from the desorption branch according to the Barrett–Joyner–Halenda (BJH) model. Prior to measurement, samples were degassed under vacuum at  $150^\circ\text{C}$  for 8 h to remove adsorbed moisture and gases.

Surface morphology was characterized using Scanning Electron Microscopy (SEM). In this technique, an electron beam interacts with the sample surface, generating backscattered and secondary electron signals that are processed to produce high-resolution images, enabling visualization of surface texture and pore structure at the nanometer scale.

### 3. Results and discussion

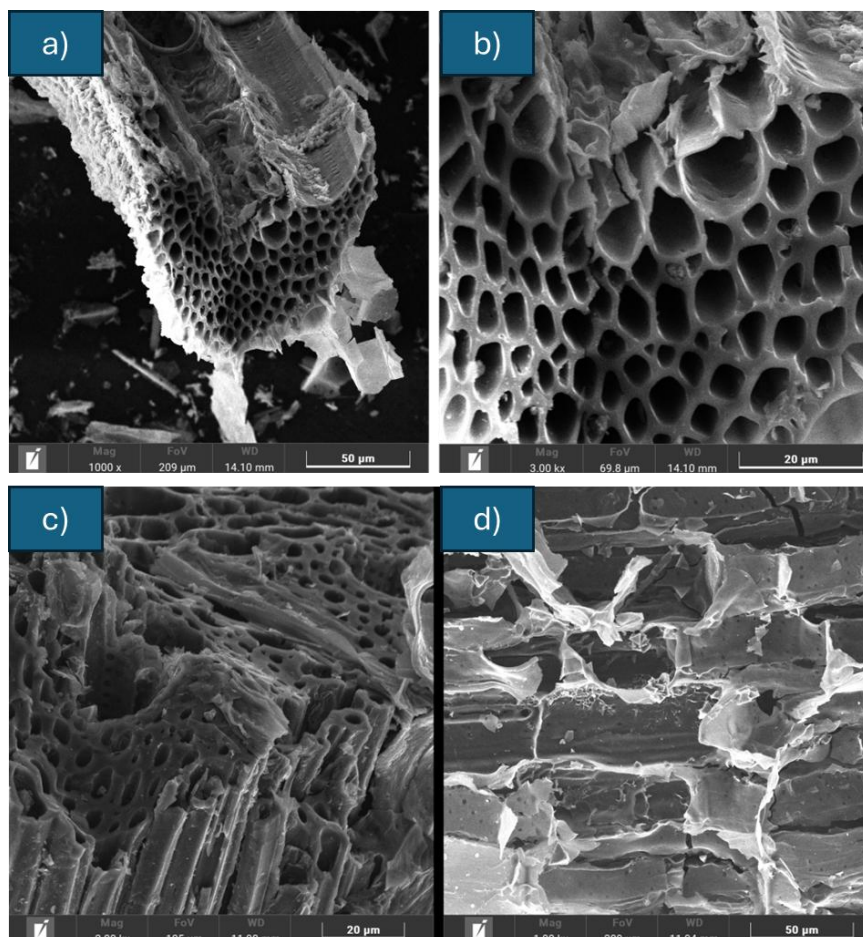
#### 3.1. The properties of activated carbon are produced from non-recyclable polystyrene (PS) and corn stalk (CS) waste.

Following the co-pyrolysis of PS waste and CS, the properties of as-synthesized biochar (BC) were measured. BET surface area and pore size are key indicators in the conversion of biomass into carbon materials. The surface area and pore size of the activated carbon were determined by nitrogen adsorption–desorption experiments using the Brunauer–Emmett–Teller (BET) method. **Figure 1** presents the nitrogen adsorption–desorption isotherms of the synthesized biochar.



**Figure 1** The BET surface area and pore size of the (a) hydrochar (HC) samples and (b) activated carbon (AC) of co-pyrolysis of PS and CS

Figure 1 (a) shows BET analysis of the hydrochar (HC) has a specific surface area of  $58.74 \text{ m}^2 \cdot \text{g}^{-1}$  and good morphology with uniform the structure and pore size. Furthermore, Figure 1 indicates that the biochar exhibits predominantly mesoporous characteristics, with pore diameters ranging from  $\sim 5$  to  $180 \text{ nm}$ . At low relative pressures ( $P/P_0 < 0.1$ ), an initial steep increase is observed, indicating the presence of a significant amount of micropores in resulting the BET surface area of as synthesized biochar that obtained from the co-pyrolysis process. Following the activation of HC to synthesis the activated carbon (AC), the physicochemical properties of AC were investigated in Figure 1 (b). The BET surface area analysis revealed that the resulting activated carbon (AC) possesses an excellent specific surface area up to  $914.83 \text{ m}^2 \cdot \text{g}^{-1}$ . The most promising feature is the sharp, nearly vertical increase in nitrogen adsorption at very low relative pressures ( $P/P_0 < 0.1$ ), suggesting nitrogen filling micropores smaller than  $\sim 3 \text{ nm}$ . These results can understand that an extensively developed microporous network. Furthermore, following this initial steep rise, the isotherm levels off and continues to much increase with a slight hysteresis loop, indicating the presence of mesopores, though they constitute only a minor fraction relative to the micropores. Moreover, a pronounced, sharp peak below  $3 \text{ nm}$  pore diameter confirms that the material's structure is dominated by uniform micropores. These results can be indicated clearly by SEM images on Figure 2. The hydrochar has porous morphology with clear pore side (cross section view) mophorlogy (Fig. 2 (a-b)). Furthermore, after activating carbon by KOH, we can observe the more porous morphology and more pore side of the activated carbon (AC) (Fig. 2(c-d)). The results can suggest that the that the co-pyrolysis of PS and CS process that discovered in this works is significant process with high BET surface area and uniform pore side of as-synthesized activated carbon compared to other previous works. Its results could be contributed due to developed technological process is appropriate and promising, successfully synthesizing activated carbon with superior properties from two materials with distinct characteristics, namely PS plastic and CS biomass that can be produced the value products and applied to wide applications.

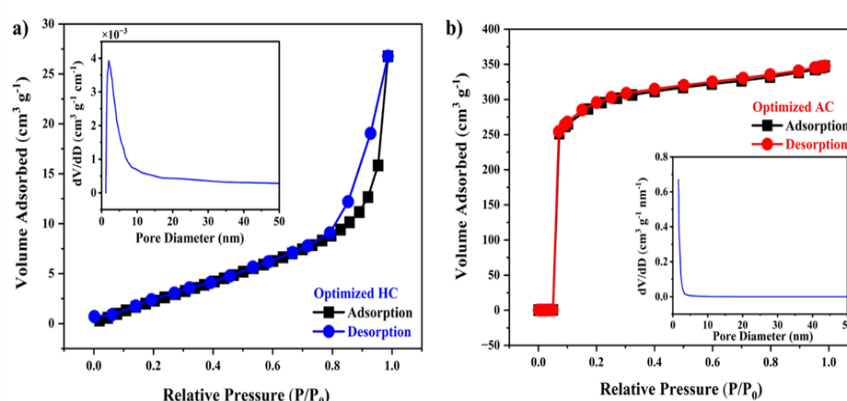


**Figure 2** SEM images of the (a-b) hydrochar (HC) samples and (c-d) activated carbon (AC) of co-pyrolysis of PS and CS

### 3.2. The properties of activated carbon produced from ground coffee

The nitrogen adsorption-desorption isotherms and textural parameters of the hydrochar (HC) and activated carbon (AC) samples produced from ground coffee are shown in **Figure 3.3**. It is interesting to find that a significant increase in surface area, pore diameter, and total pore volume was observed after carbonization and subsequent activation of the coffee ground precursor. The specific surface area of the hydrochar was determined to be  $23.06 \text{ m}^2 \text{ g}^{-1}$ , which increased markedly to  $976.34 \text{ m}^2 \text{ g}^{-1}$  after KOH activation. This enhancement is attributed to the strong redox reactions between KOH and the carbon matrix, which enlarge and develop the pre-existing pore structure of the hydrochar. Consequently, both the average pore diameter and pore volume increased from 2.01 nm to 2.19 nm and from 0.047

$\text{cm}^3 \text{g}^{-1}$  to  $0.535 \text{ cm}^3 \text{g}^{-1}$ , respectively, indicating a mesoporous structure (2–50 nm). This observation can be explained by using KOH as the activating reagent is very promising because of its lower activation temperature and higher yields, and well-defined micropore size distribution and ultrahigh specific surface area [11]. When the activated temperature is lower than  $1000^\circ\text{C}$  and an activator is used, part of the carbon microcrystals would react with the activator to inhibit the arrangement of the ordered structure [12], enhancing the overall textural properties of the activated carbon.

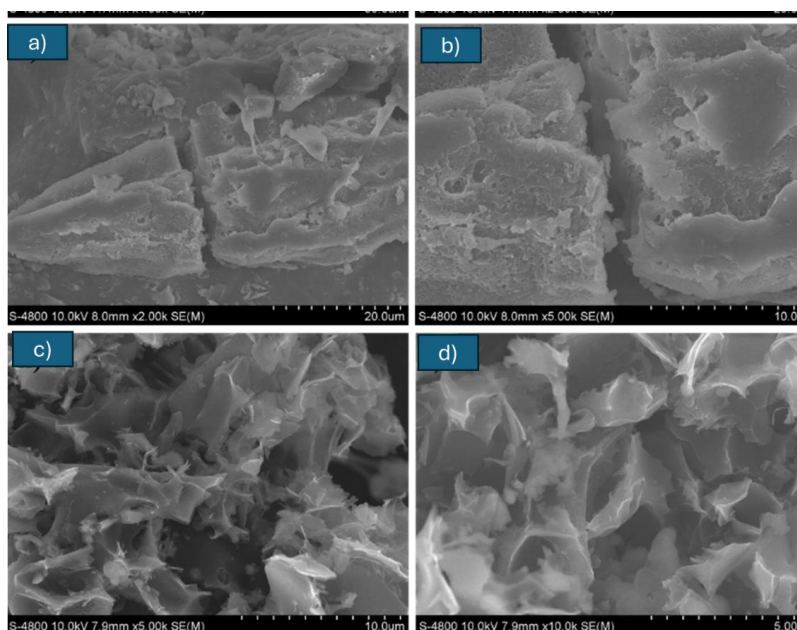


**Figure 3** The BET surface area and pore size of the (a) hydrochar (HC) samples and (b) activated carbon (AC) produced from ground coffee

The surface morphologies of the hydrochar (HC) and activated carbon (AC) samples are illustrated in Figure 3.4. The SEM images clearly demonstrate the morphological evolution of the material following hydrothermal carbonization and subsequent KOH activation. As shown in Figure 3.4 (a–b), the hydrochar exhibits a roughened and irregular surface with the formation of initial pore structures. This morphological change can be attributed to the partial degradation of organic components in the spent coffee grounds—mainly cellulose, hemicellulose, and lignin—during the hydrothermal process [11]. The decomposition of these biopolymers leads to the release of volatile compounds and the creation of microvoids, which initiate the formation of a primary porous framework [13]. After chemical activation with KOH, the surface of the HC undergoes substantial modification, resulting in a highly porous and expanded structure (Figure 3.4 (c–d)). During the pyrolysis of biomass, dehydration leads to water loss, while the release of volatile components from the carbon matrix facilitates the formation of biochar’s porous structure and the development of initial pore networks. The resulting biochar exhibits a wide distribution of pore sizes, ranging from nanometers to several ten micrometers. Furthermore, moderate temperatures ( $650^\circ\text{C}$ ) of activation process are suitable for the development of the pore structure that is corresponding to previous works [14]. These reactions effectively remove

disordered carbon and enlarge existing pores, transforming the relatively dense HC into a well-developed mesoporous carbon network.

The resulting AC surface shows a significantly increased roughness and porosity compared to the hydrochar precursor, consistent with the dramatic rise in BET surface area and pore volume reported earlier. This enhanced micro–mesoporous architecture improves adsorption capacity and ion transport, making the activated carbon highly suitable for energy storage and environmental applications [15]



**Figure 4** SEM images of the hydrochar (HC) and (c-d) activated carbon (AC) from the ground coffee

### 3.3 Comparison of the activated carbon produce from non-recyclable polystyrene, corn stalks and coffee grounds.

Table 3.1 compares the synthesis and activation conditions of hydrochar (HC) and activated carbon (AC) obtained from coffee grounds (CG) and the co-pyrolysis of polystyrene (PS) with corn stalks (CS). Despite both samples being activated with KOH, notable differences in surface area and processing conditions are observed, the difference of parameter of process may be due to the inherent chemical composition of the feedstocks. Spent coffee grounds are rich in oxygenated functional groups, lipids, proteins, and carbohydrates, which readily undergo dehydration, decarboxylation, and aromatization during hydrothermal carbonization (HTC) [11]. The HTC process at 240 °C for 5 h effectively promotes the formation of hydrochar with abundant oxygen-containing surface groups that facilitate pore formation during subsequent KOH activation at 650 °C. These

oxygen-rich compounds enhance the reactivity between KOH and carbon, resulting in significant pore development and a high BET surface area of  $914.83 \text{ m}^2 \text{ g}^{-1}$ , even under relatively mild activation conditions [16].

**Table 1** Comparison the process to synthesis the activated carbon (AC) obtained from coffee grounds (CG) and the co-pyrolysis of polystyrene (PS) with corn stalks (CS).

| No. | Feedstock     | The method to produce HC method, (Temperature, time) | Activated Temperature (°C) | Activated Time (h) | Char/Activator ratio( w/w) | Activation solvent | Activation method | BET surfaces area ( $\text{m}^2 \text{ g}^{-1}$ ) |
|-----|---------------|------------------------------------------------------|----------------------------|--------------------|----------------------------|--------------------|-------------------|---------------------------------------------------|
| 1   | Coffee ground | Hydrothermal (240 °C, 5h)                            | 650                        | 2                  | 1:1.5                      | KOH                | Pyrolysis         | 914.83                                            |
| 2   | PS and CS     | Pyrolysis (500 °C, 1.5h)                             | 750                        | 3                  | 1:3                        | KOH                | Pyrolysis         | 976.34                                            |

In contrast, the PS/CS composite requires more severe pyrolysis and activation conditions (500 °C carbonization for 1.5 h and 750 °C activation for 3 h). This difference observation from the distinct composition of PS, which is a highly aromatic, hydrogen-rich polymer with negligible oxygen content. In which, PS exhibits low porosity because of its dense hydrocarbon structure and lack of oxygen-based gasification reactions [11]. However, when combined with oxygen-rich corn stalk biomass as CS, synergistic effects occur—hydrogen radicals from PS facilitate the thermal decomposition of cellulose and lignin in CS, while oxygenated volatiles from CS promote the formation of new pores in the PS-derived carbon matrix [17]. As a result, the co-pyrolyzed sample achieved a slightly higher surface area ( $976.34 \text{ m}^2 \text{ g}^{-1}$ ) due to the enhanced pore widening and gas release during activation.

In the other hand, the CG-derived carbon exhibits efficient pore development at lower carbonization and activation temperatures, suggesting its high intrinsic reactivity and suitability for low-energy carbon synthesis routes. In contrast, the PS–CS composite requires higher energy input but benefits from synergistic chemical interactions that improve porosity and carbon yield. These results confirm that the feedstock composition-especially oxygen and volatile content-plays a crucial role in determining the optimal processing parameters and final textural properties of biomass- and polymer-derived activated carbons [18]

#### 4. Conclusions

The comparative analysis between SCG- and PS–CS-derived activated carbons were studied in this work. The result reveals that feedstock composition plays a decisive role in determining the optimal carbonization and activation conditions.

CG, being rich in oxygenated compounds and nitrogenous species, enables the formation of a highly porous carbon structure under relatively mild HTC and activation conditions, achieving a BET surface area of  $914.83 \text{ m}^2 \text{ g}^{-1}$ . Conversely, the PS–CS mixture required more severe pyrolysis ( $500^\circ\text{C}$ ) and activation ( $750^\circ\text{C}$  for 3 h) to reach a slightly higher surface area of  $976.34 \text{ m}^2 \text{ g}^{-1}$ . This improvement arises from synergistic effects between hydrogen-rich PS and oxygen-rich CS, which enhance volatile release and pore expansion. These results of work indicate that both waste systems demonstrate strong potential for the sustainable production of porous carbon materials. However, CG offers a lower-energy, more environmentally friendly pathway, while PS–CS co-pyrolysis maximizes surface development at the expense of higher energy input. The obtained results of this study will open further research focus on balancing activation severity and precursor chemistry to achieve scalable, energy-efficient production of high-surface-area carbon suitable for adsorption, catalysis, and energy storage and environment applications.

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