

A Novel Approach to Charcoal Fine Waste: Sustainable Use as Filling of Polymeric Matrices

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Abstract

This study aims to evaluate charcoal fines as potential reinforcing agents in biocomposites. Charcoal has both high carbon content and surface area depending on the manufacturing temperatures. Charcoal is a common residue in the coal industry that we propose using it to reinforce filling agents in several matrices in order to add value to this residue. This study investigated charcoal fines when using three pyrolysis temperatures (400, 600, and 800°C) to identify the most suitable charcoal to be used as raw materials in producing carbon biocomposites. We evaluated apparent density, porosity, morphology, and immediate chemical composition, and then performed a Fourier-transform infrared spectroscopy (FTIR) and an X-ray photoelectron spectroscopy (XPS). Charcoal fines produced at 800°C showed promising results as a polymeric matrix filling due to their higher porosity (81.08%), fixed carbon content (96.77%), and hydrophobicity.

Introduction

Studies have sought to develop new materials to meet current technological demands and industrial sectors with low environmental impact. Thus, science has proposed utilizing industrial waste as raw materials for other processes, promoting the use of resources and ensuring sustainability. Charcoal is a highly friable material; produced in copious quantities by the thermochemical conversion of plant biomass, resulting in enormous amounts of fines (> 25% of the production)¹. These residues usually lack adequate disposal, an problem for manufacturers, especially for manufacturers in developing countries, such as Brazil. We propose using these residues to develop new composite materials, such as reinforcing agents in polymeric, thermoplastic and/or thermorigid biocomposites used in various industrial applications. We base this hypothesis on its high carbon constitution, porosity, surface area^{2,3}, and the fact that we can control the pyrolysis process to obtain these specific properties. We aimed to evaluate the most appropriate parameters to develop new polymeric biocomposites in a matrix with charcoal fines.

Pyrolysis occurs in controlled amounts of oxygen at temperatures above 300°C, and factors such as permanence in reaction zones, final temperature, pressure, and heating rate influence it^{1,4}. Thus, we need to investigate the relevant parameters originating charcoal, especially the final temperature since it affects all important characteristics for the production of carbon biocomposites⁵.

Polymer production consists mostly of fossil fuels, severely impacting the environment due to the chemical, physical, and biological inertia of these materials². Currently, research aimed at producing new sustainable materials to circumvent this impact has intensified⁶. However, studies on the synthesis of polymeric materials with charcoal fine additives are still emergent. This material is an alternative to polymeric products from purely fossil origin, which could add value to this abundant and renewable biomass.

Proposed the first studies analyzing the potential of charcoal to produce polymeric biocomposites^{2,3,7}. They analyzed charcoal-filled composites and their thermal, mechanical, and electrical properties to

characterize their composition and behavior. These studies attest to the beneficial effects of adding charcoal to polymeric matrices to reinforce their properties. The abundant production of charcoal as an energy source and other applications may suit its potential use as an additive. We need studies assessing the use of charcoal fines as additives, since they are residues requiring reinsertion in the production chain to minimize their life cycle and add value to production. Charcoal depends on pyrolysis temperature which alter its characteristics for use as fillers of polymeric matrices. Managing pyrolysis temperatures so we can use fines in biocomposite production requires an urgent resolution for the development of new carbon products.

Charcoal fines resulting from pyrolysis of any biomass are an efficient alternative for producing sustainable composites since their carbon, porous, and hydrophobic structure allows greater interaction with polymer matrices³. This study investigates the physical and chemical properties of charcoal fines produced at different pyrolysis temperatures seeking good interactions with polymer resins in the production of carbon biocomposites.

Results And Discussion

E. saligna charcoal fines showed the following average values: basic density = 0.57 g.cm^{-3} ; extractives = 5.79%; lignin = 25.8%; ash = 0.15%, and porosity = 68.22%. These properties relate directly to pyrolysis products. Basic density and lignin content are relevant for charcoal production, increasing coal volumetric and gravimetric yields and favoring greater biocomposite manufacture^{8,9}. Yield also depends on pyrolysis variables, such as reaction atmosphere, heating rate, and, especially, final temperature (Fig. B – supplementary).

The results show that 400°C pyrolysis obtained the highest yield, 37.15%, whereas 800°C, the lowest, 27.42%; results inversely proportional to pyroligneous liquid yield. Fig. B shows non-condensable gas yields failed to significantly vary with increasing temperatures (supplementary material): 25.7, 23.27, and 22.04% in 400, 600 and 800°C, respectively.

To optimize production, greater charcoal yields favor biocomposite filling due to larger volumes of the material of interest, though its chemical and physical properties will effectively contribute to the synergy of the materials involved in producing carbon biocomposites¹⁰. A higher final pyrolysis temperature (600 to 800°C) increased in 7% the apparent density of charcoal (Fig. 1A).

This behavior is due to intense hydrogen output, an element capable of strong molecular bonds absorbing significant energy in pyrolysis above 500°C¹¹. This relates to the rearrangement of the chemical structure of biomass under heat, producing a graphitic structure that benefits the mechanical resistance of the material^{12,13}. By proposing charcoal as a reinforcing agent in polymeric matrices, we expect increased mechanical strength and developed density in the new material. We hope biocomposites filled with high-temperature charcoal will show higher strength, facilitating their use in structural applications.

Porosity is another relevant parameter for producing biocomposites. Charcoal is highly porous (up to 85% of its volume) and its pore sizes range from sub-nanometers to tens of micrometers, depending on raw materials and pyrolysis temperatures^{14,15}. Fig. 1B shows that higher temperatures result in a more porous charcoal – 800°C pyrolysis favored their formation. Studies state that the progressive removal of volatile materials from charcoal pores, increased connection of existing pores, and condensation of the remaining skeletal structure cause this behavior¹⁴⁻¹⁷. Their immediate chemical composition (Fig. 2) shows that higher pyrolysis temperatures increase the percentage of fixed carbon and, proportionally, the reduction of volatile materials.

Evaluating the immediate chemical composition of charcoal fines as a function of pyrolysis temperature is important for predicting biocomposite performance¹⁸. Studies discuss that materials with a higher carbon content provide greater mechanical strength, which can positively increase the resistance of the biocomposites produced^{19,20}. We observe a decrease in the ash content of coal produced at 800°C which may enable a greater interaction between polymeric matrices and charcoal fines due to its high fixed carbon and lower ash contents. We expect these characteristics to provide biocomposites with greater interaction and mechanical resistance. Confocal microscopy images show the increased porosity of charcoal at higher temperatures (Fig. 3).

We identify more pores, usually of a nonpolar nature, in the material produced at 800°C^{21,22}. These characteristics are relevant for using the material as reinforcing agents in biocomposites since they can promote greater adhesion to polymeric matrices, better wetting, and greater mechanical resistance by high impregnation and better spreadability. The literature indicates that a more porous structure allows polymeric resins to fuse and entangle with charcoal, creating a strong interfacial support between fillings and matrices, resulting in improved properties, especially mechanical resistance^{16,23,24}. However, polymer penetration into charcoal pores will depend on the viscosity of the polymer resin and pore size²³. We highlight that such aspects depend on particle dimensions of charcoal fines used as filler. Fig. 4 shows the SEM images of charcoal fines produced at different final temperatures.

Charcoal fines have a rough surface, due to pores collapsing and ash filling the porous system. Different temperatures did not produce significant morphological differences, which would require higher heating rates. Despite homogenization, we observe charcoal fines of varied sizes. Pyrolysis conditions influence surface morphology and the physical properties of charcoal fines. This evidences the need for a more rigorous preparation of the material prior to filling biocomposites, and further studies should investigate the nanometric scale in preparing this carbon raw material²⁵. Fig. 5 shows the FTIR spectra of charcoal fines produced at three pyrolysis temperatures and changes to their chemical structure.

The stretching vibration of the asymmetric OH group – referring to phenol, alcohol, carboxylic acid groups, and water – formed a 3663 cm⁻¹ broad peak. The 1671 and 1728 cm⁻¹ peaks refer to the vibration and elongation of the CO double bond, whereas the 1500 cm⁻¹ peaks, to the vibration in the double bond between aromatic carbons with olefins and aromatic structures. On the other hand, 1180 to

1297 cm⁻¹ peaks relate to elongating vibrations of the C-O connection. Fig. 5 shows that higher temperatures affected functional groups, changing coal structures. The formation of hydroxyl groups at 800°C favors the elevated polarity of the material, profoundly influencing charcoal and polymer matrix interaction in composites²⁶, essential for good mechanical properties^{27,28}. Moreover, the free hydroxyl groups in coal produced at 800°C allow greater interaction between materials, contributing to the generation of highly compatible composites^{2,3}.

We performed a XPS analysis to understand how the chemical changes to charcoal can contribute to resin interaction. Fig. 6 shows the deconvolution of the XPS spectra of coal fine samples.

Note the spectral region comprising the binding energies characteristic of C1s in the materials synthesized at 400 (Fig. 6A), 600 (Fig. 6B), and 800°C (Fig. 6C). Analysis of C1s spectra showed five peaks related to the different carbon species in charcoal fines. The most intense peak (EB= ~284.0 eV) corresponds to functional states of species with C=C or C-sp² bonds in aromatic/aliphatic carbons²⁹. Due to the nature of the atoms surrounding these species, the binding energy in this peak may undergo small variations due to changes in pyrolysis temperature, since heating affected the chemical composition of the surface³⁰.

Higher pyrolysis temperatures changed the structure and chemical nature of charcoal; attested by the increased intensity of peaks of aromatic/aliphatic groups in XPS spectra. As expected, heating develops more compact aromatic carbon structures and more intense spectral peaks. On the other hand, higher temperatures decrease the proportion of functional groups formed by carbon atoms and heteroatoms, especially oxygen, in the composition of charcoal fines³¹. Table 1 shows this aspect by comparing the mean relative percentages related to the contribution of each carbon species to the total composition of the material.

Table 1
Average percentage of functional states in C1s XPS spectra.

Sample	C=C, C-C, C-H (aromatic, aliphatic carbon)	C-O, C-O-C, C-OH (hydroxyl, ether, phenol)	C-O (ketone)	C=O (carbonyl)	O-C=O (carboxylic acid, ester)
CV 400	62.1 ± 1.9	19.3 ± 0.9	10.8 ± 0.7	3.9 ± 0.3	3.95 ± 0.08
CV 600	72.1 ± 1.7	12.7 ± 2.5	7.5 ± 0.5	3.4 ± 0.2	4.3 ± 0.1
CV 800	70.4 ± 1.8	12.5 ± 1.5	7.7 ± 0.8	3.8 ± 0.2	5.7 ± 0.2

Analyzing XPS spectra shows the majority presence of aromatic or aliphatic carbon species, and C-O/C-O-C/C-OH groups (ether, phenol, etc.), C-O (ketone), C=O (carbonyl) and O-C=O (carboxylic acid, ester),

located at 284.70 eV, 286.15 eV, 287.75 eV, and 288.80 eV binding energies, respectively^{30,31}. These functional carbon and oxygen groups comprise a smaller proportion of the composition, tending to decrease as temperatures rose from 400 to 600°C. However, they remained stable from 600°C upward since we observed no significant variations in percentage contributions as temperatures neared 800°C.

Note in the XPS spectra (Fig. 6) that the energy of the C-C/C-H group dominates the charcoal composition, intensifying as temperatures increased, whereas oxygen functional groups showed an inversely proportional trend. We can classify the functional groups on the surface of charcoal fines with carbon chains, such as the C-C/C-H group, as hydrophobic; and those containing oxygen, such as C-O/C-O-C/C-OH, C-O, C=O and O-C=O, as hydrophilic³¹. The hydrophobic surface of charcoal fines improved with higher temperatures, attested by the increased contribution of C-C/C-H species dominating the material composition.

Studies point out that greater hydrophobicity ensures greater polymer compatibility^{28,32,33}. More hydrophobic materials result in better polymer filling and compatibility, evenly incorporating charcoal particles into polymer matrices and improving the tensile strength and flexion of biocomposites²². Thus, charcoal fines could reinforce polymer matrices due to their hydrophobicity which can provide resistance to biocomposites.

Methods

Biomass characterization and charcoal production

Chips from 10-year old *Eucalyptus saligna* trees were used as raw materials for charcoal fines (Fig. A - supplementary). Basic density³⁴, chemical composition of extractives, lignin³⁵, hemicelluloses³⁶, ash content³⁷, and porosity³⁸ were measured.

Then, the material was pyrolyzed. Wood dried in a 103±200°C oven was charred in a reactor inside a muffle whose inert atmosphere was obtained by the continuous insertion of nitrogen gas (3 ml.min⁻¹). Chips were pyrolyzed at 400, 600 and 800°C, at a heating rate of 10°C.min⁻¹, beginning at 30°C until they reached their final temperature, in which they remained for 120 min of conditioning. After each process, the charcoal mass was measured, and the pyroligneous liquid obtained by gas condensation was evaluated for its mass and volume. Thus, the gravimetric charcoal, pyroligneous liquid, and non-condensable gas yields were estimated in relation to the initial wood chip mass via Equations 1, 2, and 3.

$$CY = \left(\frac{C_m}{D_m} \right) \times 100 \quad (1)$$

$$PLY = \left(\frac{PL_m}{D_m} \right) \times 100 \quad (2)$$

$$\text{NCGY} = 100 - (\text{CY} + \text{PLY}) \quad (3)$$

Where: CY = charcoal yield (%); Cm = charcoal mass (g); Dm = dry wood mass (g); PLY = pyrolytic liquid yield (%); PLm = pyrolytic liquid mass (g); NCGY = non-condensable gas yield (%).

Charcoal chips were ground in an MA-500 ball mill for three hours. Fine were homogenized through a 250-mesh granulometry sieve (0.056 mm) (Fig. A – supplementary), according to the conditions proposed in this study. This will simulate the fines obtained in the industrial operations.

Charcoal fine characterization

Apparent density was estimated by dividing the sample mass by its volume ($\text{Apd} = \text{mass}/\text{volume}$, in $\text{g}\cdot\text{cm}^{-3}$), and porosity³⁸; whereas volatile material, ash, and fixed carbon³⁹.

Visual analysis of charcoal microstructures

This analysis was performed in two steps. First with the aid of the Olympus LEXT Confocal Microscope – 3D Measuring L. Microscope 4000 to assess degradation or maintenance of solid charcoal in chips prior to maceration. Images were obtained via an objective 50x magnification lens (0.95 numerical aperture; with a 0.26 mm × 0.26 mm field of view, and about 0.25 μm sampling distance) and an objective 100x magnification lens (0.95 numerical aperture, with a 0.13 mm × 0.13 mm working field of view, and a 0.13 μm sampling distance). 405 nm light beam wavelength. The OLS4000 2.1 software was used to treat the images. Then, charcoal fines were analyzed (250 mesh - 0.056 mm). Fixing the material in a metal support with carbon tape and metallized with gold in the Balzers Union SCD 030 system ensured the precise scanning of secondary electrons during microscopy via a JSM-IT200 scanning electron microscope (Tokyo, Japan) operating at 10Kv. SEM images were obtained using proprietary JEOL software.

Fourier-transform infrared spectroscopy (FTIR)

To characterize the aromatic structures (functional groups and chemical bonds) of charcoal fines, a spectroscopy analysis was performed in the Fourier-transform infrared region (FTIR).

Charcoal fine X-ray photoelectron spectroscopy

A K-Alpha spectrometer (Thermo Scientific) from the National Nanotechnology Laboratory (LNNano) at the National Center for Research in Energy and Materials (CNPEM, Brazil) was used for X-ray photoelectron spectroscopy (XPS) analyses of charcoal fine surface energy. A monochromatic Al K α (1486.6 eV) X-ray source of 300 W was used at a 30° take-off angle to the sample surface.

Measurements were taken in a 9 × 9 mm² area under a 5 × 10⁻¹⁰ mbar high vacuum at room temperature. Spectra were obtained in bond energies between 0 and 1150 eV via three scans with 160 eV passing energy and 1 eV resolution.

Data analysis

An entirely randomized experimental design with a dependent variable (pyrolysis temperature) and five replications was used for data analyses. Data were initially subjected to normality (Shapiro Wilk) and Levene's (variance homoscedasticity) tests, and then to Pearson's correlations and the Regression Analysis F-test. To estimate R^2 coefficients, the t-test was used. When necessary, models were adjusted to improve standard error values and the coefficient of determination. All tests had a 95% confidence interval.

Conclusion

Charcoal fines at 800°C show good carbonic, porous, and hydrophobic results, making them a potential material for biocomposite production which would allow a greater interaction and higher compatibilization with polymer matrices. The search for innovations and use of polymeric biocomposites is increasing, and this study shows another possible use for these materials. We suggest future studies seeking optimizing the pyrolysis yields or their speed (flash pyrolysis) in order to obtain charcoal for polymeric matrices.

Practical and policy implications and future perspectives

Composites are technologically strategic materials with many applications in biomedicine, engineering, architecture, and dentistry, among others. However, non-biodegradable petroleum-derived materials comprise several of these biocomponents. Research has widely investigated biocomposite development to offer a sustainable appeal to production systems since they are strategic, efficient, economically viable, and environmentally correct products which add value to charcoal residues. Currently, this waste lacks an adequate disposal or use due to its substantial amounts and the absence of government and social awareness. We believe no technical or scientific study have examined the practical potential of charcoal fines as reinforcement in polymeric matrices. Using this residue in polymeric biocomposites can reduce costs, dependence on fossil sources (plastics and other petroleum products) and, consequently, mitigate greenhouse gas emissions.

Our results have practical and political implications, showing the potential benefits of reusing residual charcoal fines to produce biocomposites worldwide. The actions that can be developed based on our proposal favor the so-called industrial symbiosis, where waste from one sector becomes resources for the generation of products from another sector, contributing to resource efficiency and circular economy⁴⁰. It is important to note that this paper presents an innovative approach towards sustainability by including the concept of industrial symbiosis that is a part of the emerging field of industrial ecology demands resolute attention to the flow of materials and energy through local and regional economies. Industrial symbiosis engages traditionally separate industries in a collective approach to competitive advantage involving physical exchange of materials, energy, water, and/or by-products⁴¹. The keys to industrial symbiosis are collaboration and the synergistic possibilities offered by geographic proximity. Moreover, Industrial symbiosis⁴² describes how a network of diverse organizations can foster eco-innovation and

long-term culture change, create and share mutually profitable transactions—and improve business and technical processes. Thus, this perfectly complement the technical assessment of the biomass when processed under different conditions with the utilization of waste from one industry to be used by another industry to add value.

Our proposal contemplates two of the Sustainable Development Goals developed with the participation of world leaders and international non-governmental organizations. These are Goal 9 which aims to “Build resilient infrastructure, promote inclusive and sustainable industrialization and foster innovation”, Goal 12 which aims to “Ensure sustainable production and consumption patterns”. The Brazilian National Solid Waste Policy (Law 12305/2010) provides for this action, encouraging waste reuse and valorization^{43,44}. Brazil still needs to develop sectoral plans to reduce such waste accumulation, but national development planning or regulation still ignore most of these policies and instruments. Moreover, we should mention this study may impact society since biocomposites manufactured with charcoal fine-filling will benefit small and medium producers of this raw material. This study assists public management and companies in planning future goals towards charcoal waste which will generate and develop new materials for various industrial sectors, such as civil construction, automotive, biomedical, among others. Supplementary Figure C presents a graphical abstract of the present research.

Declarations

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Author contributions statement

F.M.D and G.F.M.C conducted the experiments; F.M.D, G.F.M.C, A.F.D.J data analysis and writing of the original draft; A.F.D.J acquisition of financing; M.P.O, L.P.R.P, D.P, M.G.J, M.G.A, D.S actively helped from the critical correlated areas of the research. All authors reviewed the manuscript.

Competing interests

The authors declare no conflicting interests.

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Figures

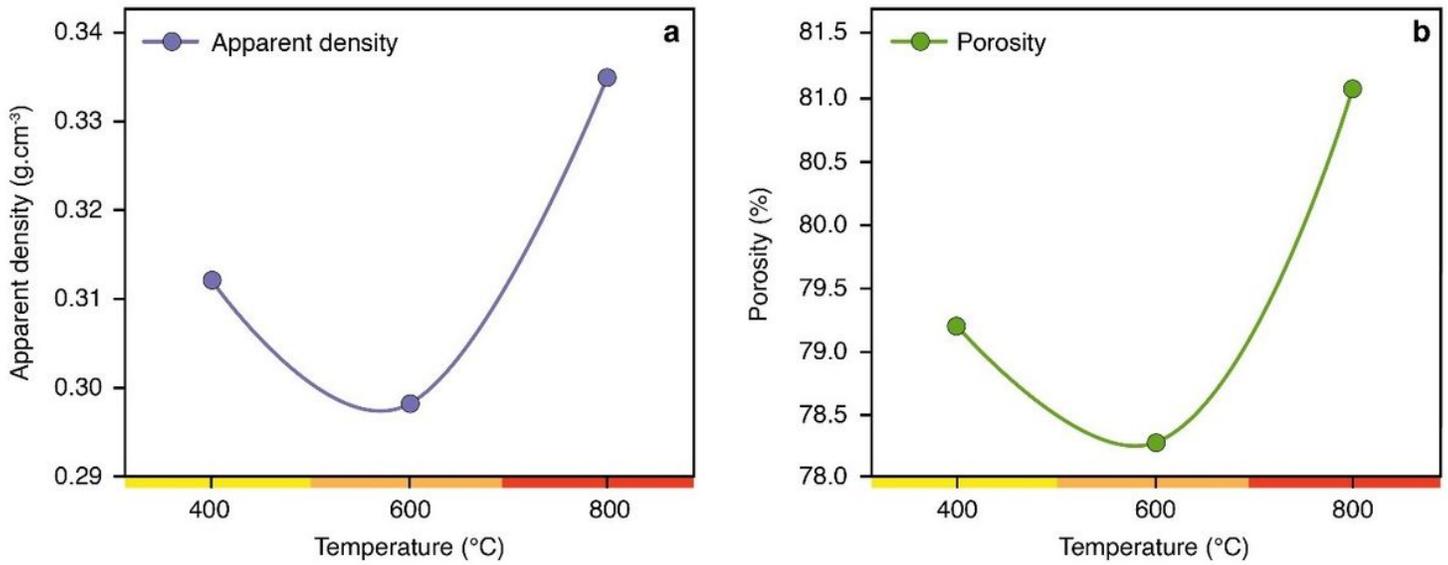


Figure 1

(A) Apparent density and (B) porosity of charcoal fines produced at different pyrolysis temperatures. Source: The authors (2022).

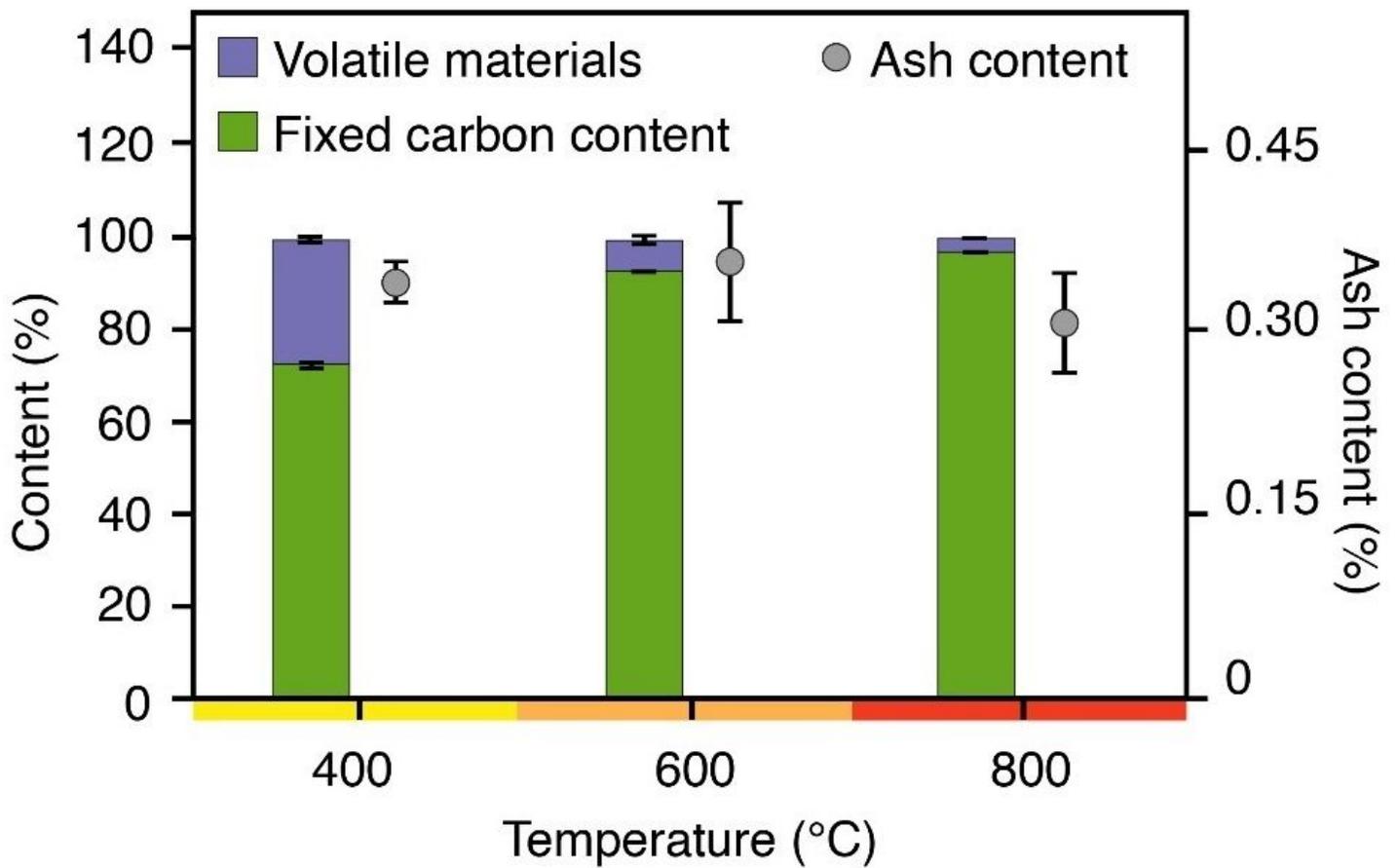


Figure 2

Immediate chemical composition of charcoal fines. Source: The authors (2022). Where: FC = fixed carbon content; VM = volatile material content; AS = ash content.

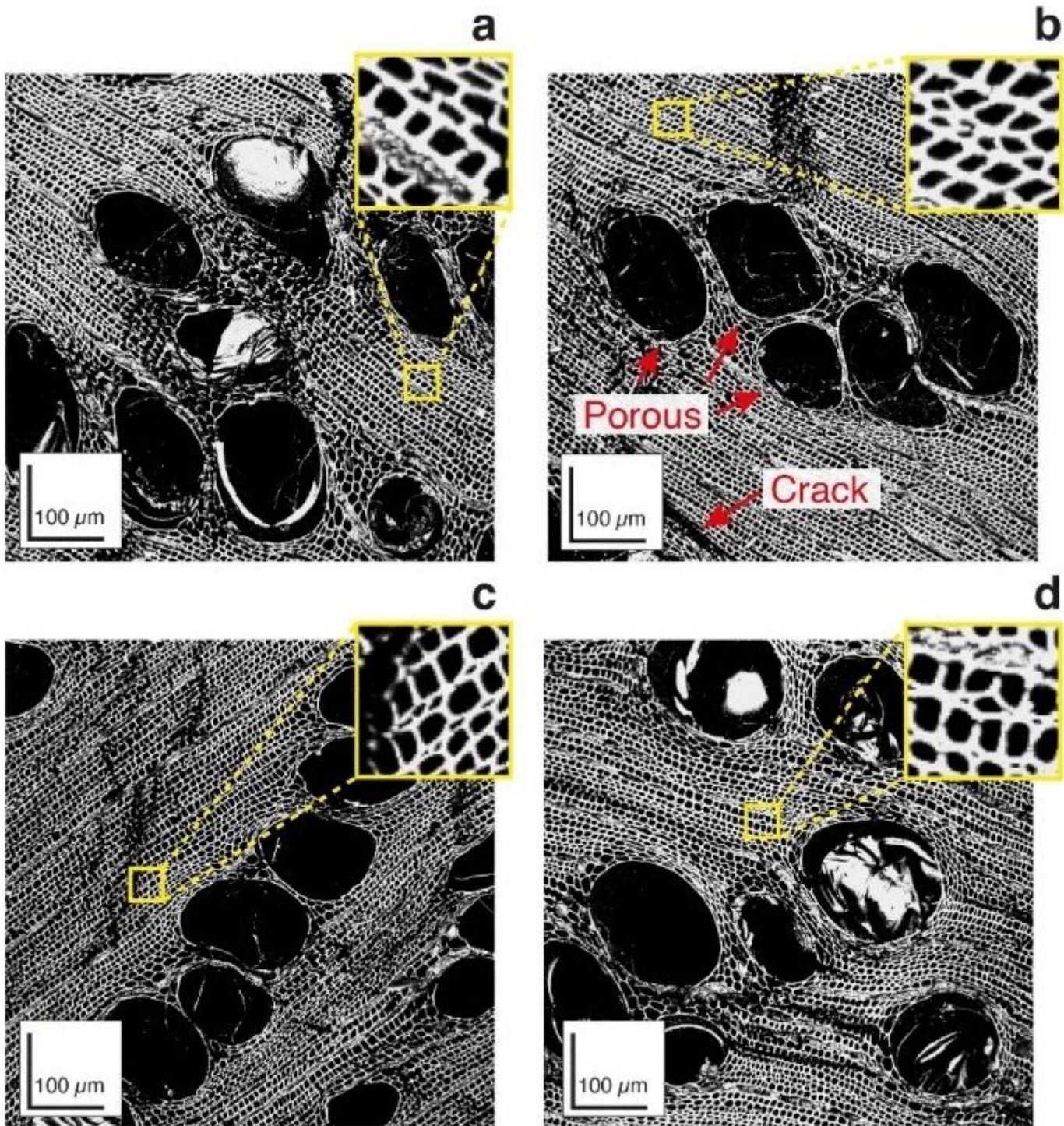


Figure 3

Confocal microscopy of the anatomical transverse structure of charcoal produced at 400°C (A and B), 600°C (C), and 800°C (D). Source: The authors (2022).

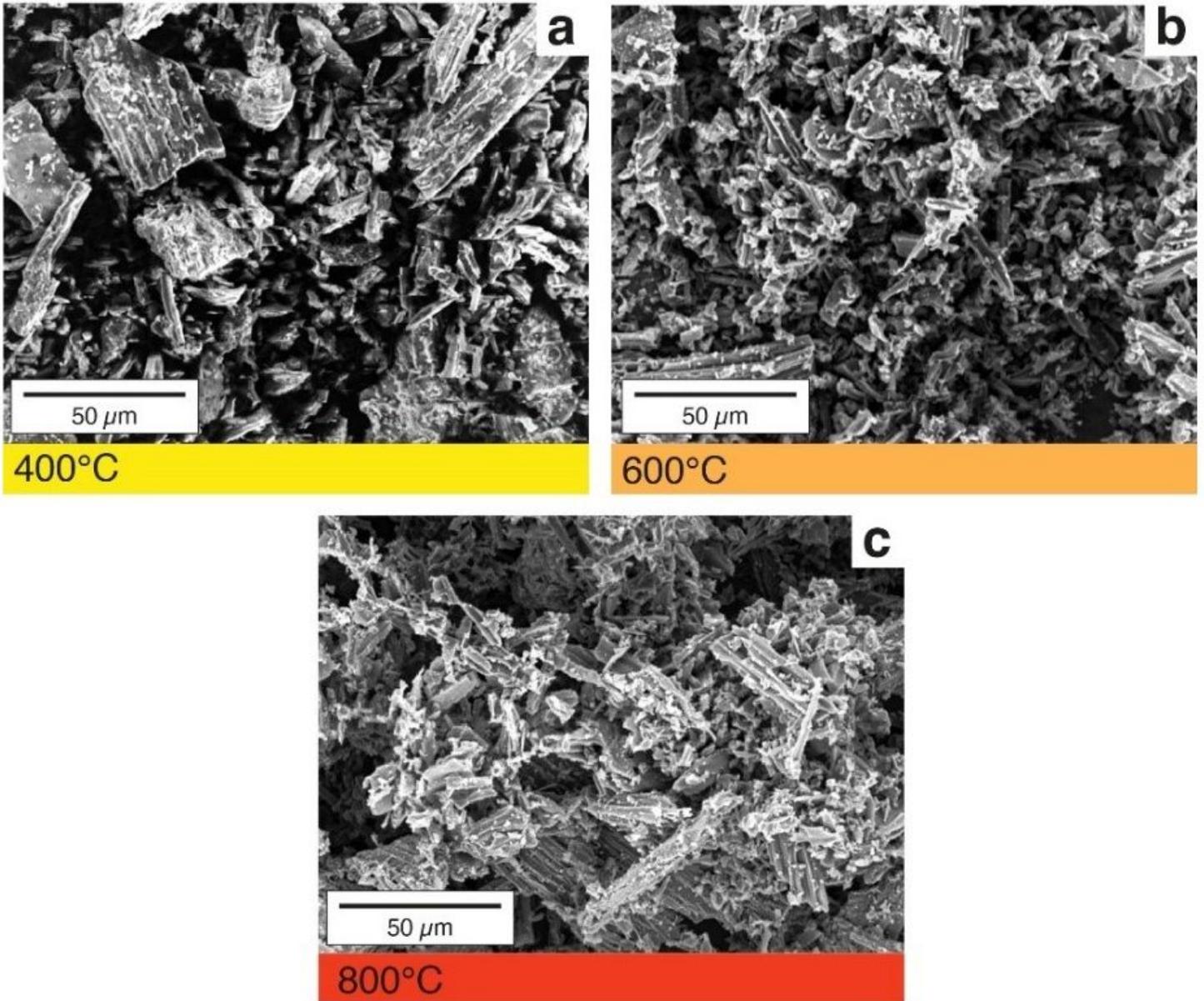


Figure 4

Micrographs obtained by scanning electron microscopy of charcoal fine samples synthesized at 400 (a), 600 (b), and 800°C (c) in x300 magnification. Source: The authors (2022).

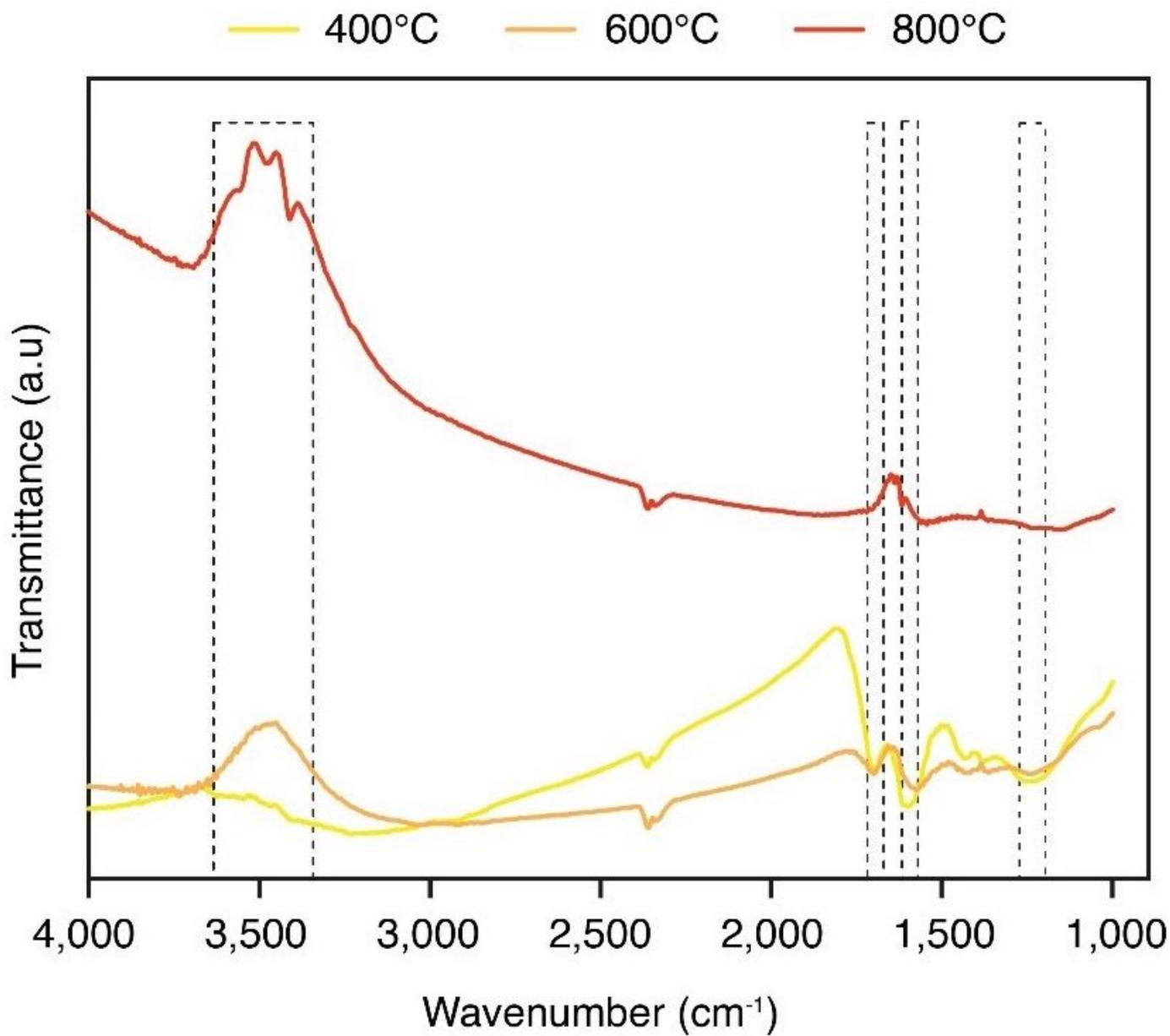


Figure 5

FTIR spectra of charcoal fines produced at different pyrolysis temperatures. Source: The authors (2022).

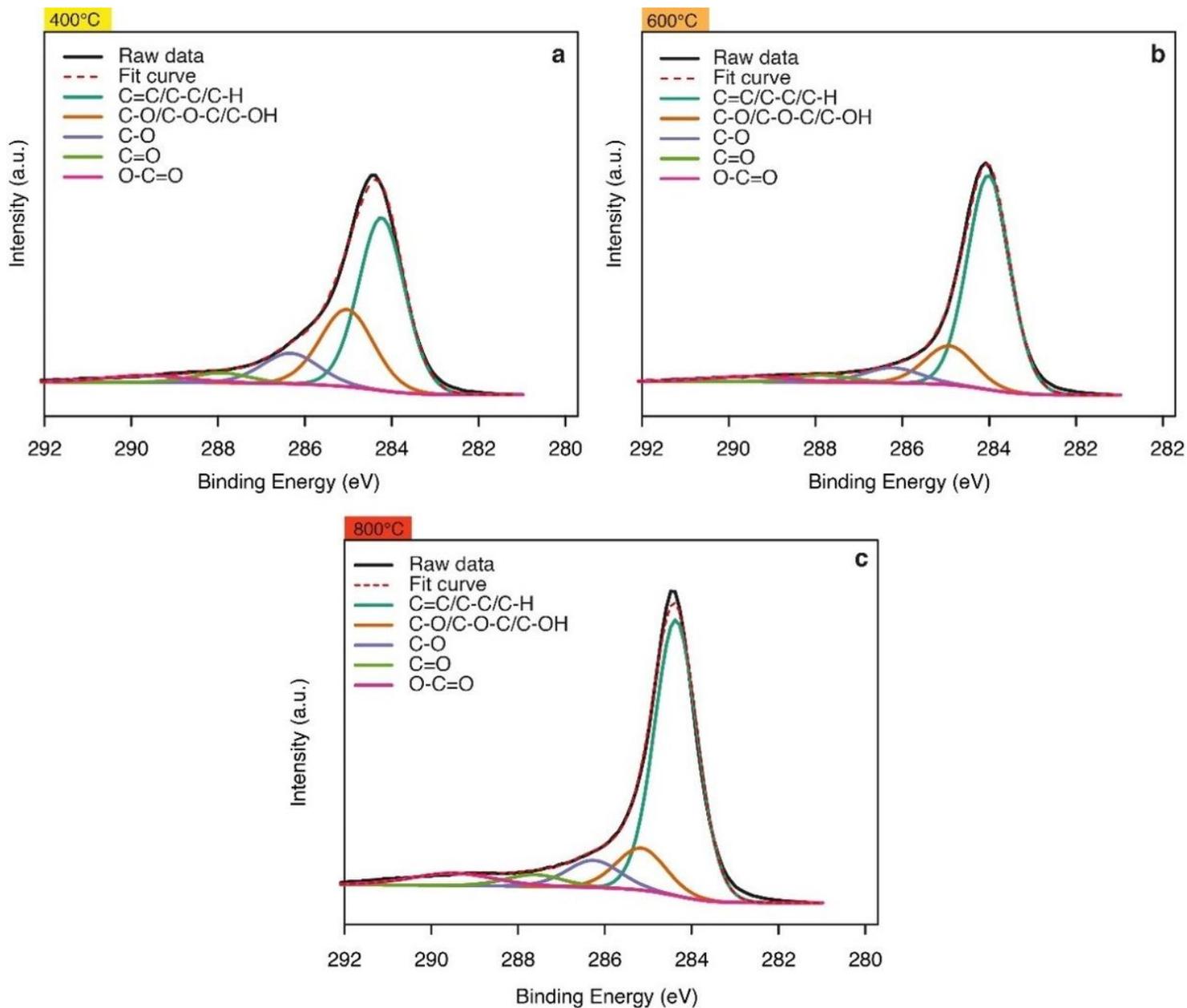


Figure 6

X-ray photoelectron spectroscopy (XPS) of charcoals at temperatures of 400 (A), 600 (B), and 800°C (C). Source: The authors (2022).

Supplementary Files

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