

Thermophysical characterization of biochar from pyrolysis of orange waste

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Abstract: An increased interest in organic waste recovery results in thermal treatments for obtaining bio-oil, biochar, and gas. In this work, biochar was obtained from orange waste pyrolysis and submitted to inverse photopyroelectric technique, photoacoustic spectrum analysis, IR spectroscopy, and SEM analysis. The results indicate a thermal effusivity value close to that of water; this can be the result of organic matter. The most prominent bands were identified as carboxylic, carbonyl, lactone, phenol, lactol.

Keywords: Thermal effusivity, photoacoustic spectrum, calcination, Photothermal techniques.

I. INTRODUCTION

Nowadays, there is an increased interest in waste reduction and materials recovery. According to recent studies, in Mexico, 900 grams of waste per capita are produced daily, and out of this number, only 8% is recycled (Ibargüen Rodríguez et al., 2026). Several efforts have been made to reduce waste disposal and minimize pollution; in particular, thermal methods are the option of choice for organic waste treatment and energy recovery in the form of bio-oils, carbon, and gas (Lara Hernández et al., 2026).

The obtention of carbon has several advantages, such as waste reduction and lower emissions (Ee et al., 2025). On the other hand, in agriculture, carbon can be added to soil, giving a porous structure that acts as a scaffold for microbes and fungi and a water trap. Depending on the final pore size, carbon can retain nutrients and reduce leaching (Asingsamanunt et al., 2026; Muhammad et al., 2018; Villa-Parejo et al., 2025).

On the other hand, photothermal techniques have proved to be suitable methods for thermal characterization. Photothermal techniques rely on energy coming from a light beam being transformed into heat. When the light beam is frequency modulated, the produced heat takes the form of a thermal wave. Depending on the employed sensor, photothermal techniques can be used to gain knowledge about thermal, elastic, or even photothermal absorption. In this work, the thermal effusivity of carbon obtained from

residues from hydrothermal processes is reported.

II. METHODS

The starting materials are orange waste (peel and bagasse); the raw materials were obtained from local suppliers at Orizaba, Ver. Mexico (N 18° 51' 18.356" W 97° 6' 2.914").

A. Sample treatment

The orange waste was ground and placed into a bioreactor as described by Lara and colleagues (Lara Hernández et al., 2026). The solid fraction resulting from the hydrothermal process was pressed into pellets and submitted to pyrolysis. The obtained pellets were submitted to thermal analysis.

B. Thermal effusivity.

Thermal effusivity was measured using the so-called inverse photopyroelectric technique (IPPE) (Lara-Hernandez et al., 2018). IPPE consists of a modulated light beam focused on one side of a pyroelectric sensor, whereas the opposite side of the sensor is in thermal contact with the sample under analysis. The light is modulated in frequency; then, the beam energy is transferred to the sensor in the form of a thermal wave whose frequency is the same as the light beam. Under these experimental conditions, the pyroelectric sensor generates an electrical signal whose amplitude and phase are a function of frequency modulation, thermal properties of the sample, thermal properties of the sensor, and instrumental factors. At a specific frequency range, the pyroelectric signal can be modeled as:

$$\theta(\omega) = \frac{(1 - e^{\sigma_p l_p})(1+b) + (e^{\sigma_p l_p} - 1)(1-b)}{(g-1)e^{-\sigma_p l_p(1-b)} + (1+g)e^{\sigma_p l_p(1+b)}} \quad (1)$$

Being l_j the thickness of the j -th layer, $\sigma_j = (i+1)a_j$ the complex thermal diffusion coefficient, ω the light angular modulation frequency, $b = e_s/e_p$, $g = e_g/e_s$ effusivity ratios, and e_j the thermal effusivity of the j layer (surrounding gas, sample, pyroelectric sensor). The experimental setup is shown in Figure 1.

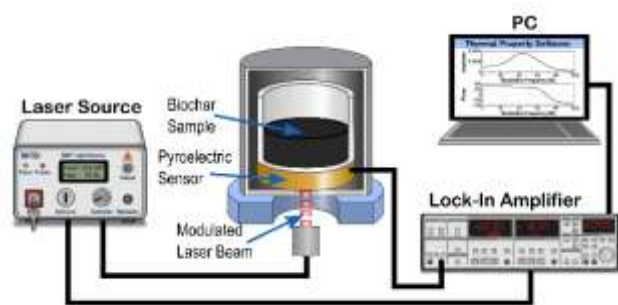


Figure 1. System control and monitoring design diagram.
Source: Authors.

When a frequency sweep is performed and the thermal properties of the pyroelectric sensor are known, the thermal effusivity of the sample can be obtained by fitting Equation 1 to the experimental data, either by fitting amplitude, phase, or both.

C. Photoacoustic spectrum

The photoacoustic spectrum was obtained by a light beam whose wavelength is varied and directed to the sample; the sample is heated, thus resulting in a sound wave produced at the boundary of the heated sample and the surrounding air. The sound wave was detected using an electret microphone and a lock-in amplifier as described elsewhere (Lara Hernández et al., 2026). Graphite charcoal was employed as a reference sample to normalize the sample spectrum in order to take into account the experimental conditions.

D. Vibrational Study by Infrared Spectroscopy

Fourier-transformed infrared (FTIR) spectra were obtained by scanning the powdered biochar sample from 400 to 4000 cm^{-1} using a Spectrum 3 spectrometer (Perkin Elmer, UK) with a single-bound diamond crystal Attenuated Total Reflectance (ATR) accessory. The sample was pressed onto the diamond probe and scanned at a resolution of 2 cm^{-1} .

E. Morphology and semiquantitative elemental analysis by Scanning Electron Microscopy

Morphology, composition, and elemental distribution were determined using an SEM system coupled with an EDS analyzer. These analyses were performed using a TESCAN Lyra 3 (Dual Beam FIB-SEM + STEM + EDX) microscope (Tescan, Brno, Czech Republic) equipped with a field emission gun (FEG) operating at 20 kV. The samples were coated with gold to prevent surface electron charging.

III. RESULTS

Figure 2 shows the pyroelectric experimental data and the corresponding fitting in a frequency range of 2 – 100 Hz. As a result of the corresponding fitting, the thermal effusivity gives a value of $1.536 \times 10^3 \pm 25.843 \text{ W} \cdot \text{s}^{1/2} \cdot \text{m}^{-2} \cdot \text{K}^{-1}$.

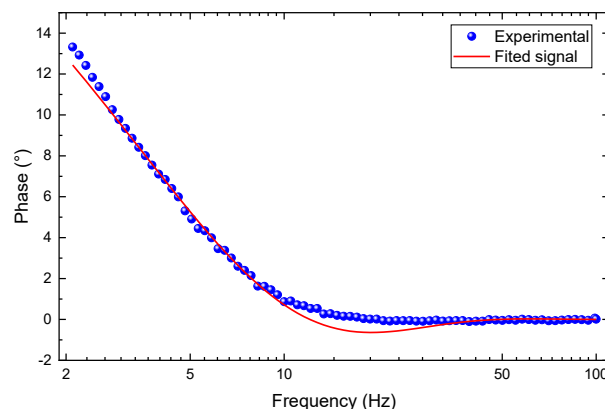


Figure 2. Frequency scan of the IPPE signal. Dots are the experimental points, and the solid line represents the best fit of equation 1 to the experimental points.

Figure 3 shows the photoacoustic spectra of biochar and the identified peaks.

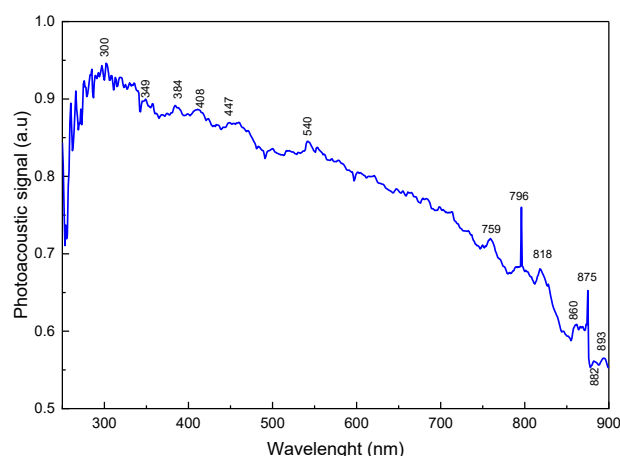


Figure 3. Photoacoustic amplitude spectrum of biocarbon sample.

Below 300 nm, the absorption spectra was assigned to an aromatic ring. In the 300-400 nm region, π/π^* transitions were observed from conjugated C=C double bonds in aromatic structures, and due to C=C (non-conjugated double bond) and C=O (carbonyl groups) (Albuquerque et al., 2013).

21 vibrational bands were found by infrared spectroscopy (Figure 4). The assigned bands are shown in Table 1.

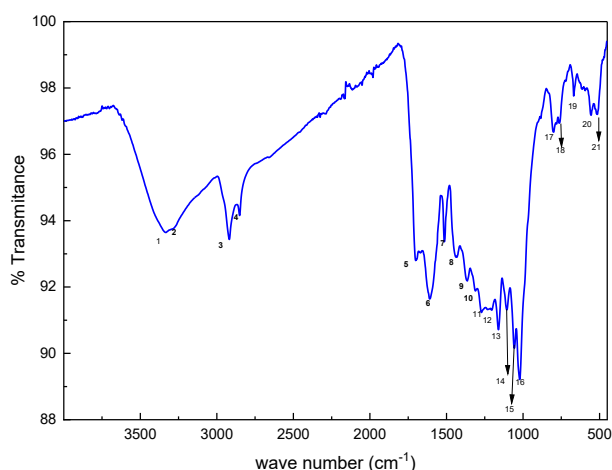


Figure 4. Infrared Spectra of biocarbon sample

Table 1. Assignment of vibrational bands for biochar from orange peel

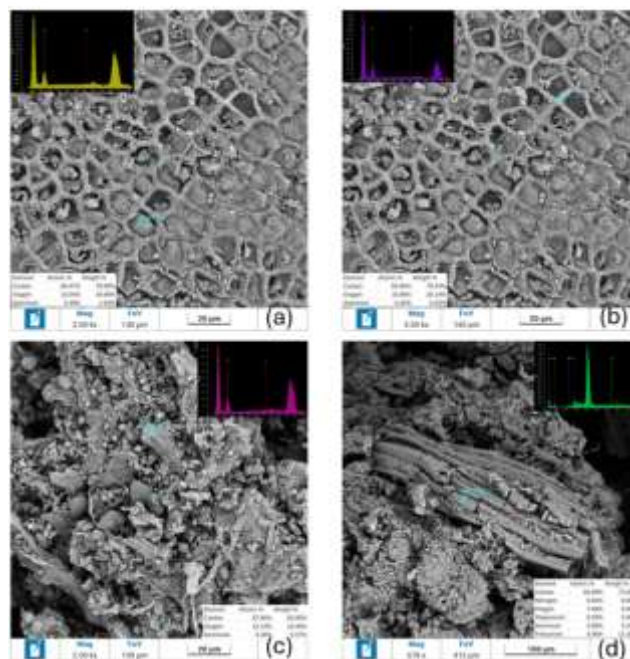
No	Vibrational Band (cm ⁻¹)	Assignment
1	3363	Bending vibrations of O–H functional groups (Altıkat et al., 2024)
2	3301	
3	2921	Carboxylic acids(Fanning & Vannice, 1993)
4	2854	
5	1704	
6	1612	Carboxyl-carbonate bonds
7	1513	C=C Stretching
8	1442	
9	1369	OH deformation of alcohols/phenols
10	1315	Lactones
11	1274	Carboxyl/carbonate bonds
12	1214	
13	1160	
14	1110	Alcohols
15	1058	
16	1024	Deformation vibrations of the C–H groups in methyl and methylene groups(Barroso-Bogeat et al., 2014; Fanning &
17	804	
18	767	

19	667	Vannice, 1993).
20	557	metal-oxygen and metal-hydroxyl vibrations(Zou et al., 2020).
21	514	

These vibrational bands are characteristic of the functional groups found in most carbonaceous surfaces such as carboxylic, carbonyl, lactone, phenol, lactol, anhydride, pyrone, ether, chromene, and quinone groups (Tomczyk et al., 2020).

Pasieczna-Patkowska et al. have compared infrared and photoacoustic spectra of biochar. In this study was found that bands between 875 to 760 cm⁻¹ correspond to aromatic C-H out-of-plane deformation, band at ~720 cm⁻¹ indicates the presence of long chain alkanes (CH₂ wagging).(Pasieczna-Patkowska & Madej, 2018; Yang & Simms, 1995)

On the other hand, several sections of biochar were used to study its morphology and semiquantitative composition (Figure 5).



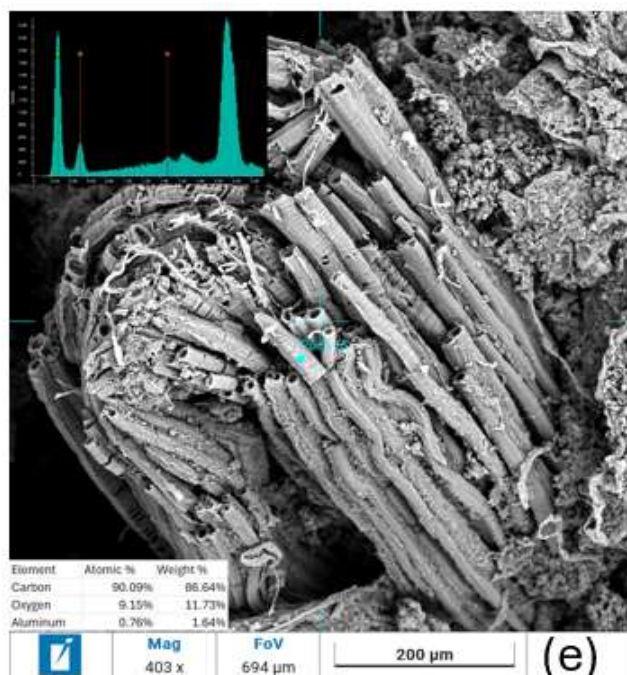


Figure 5. Micrograph of biocarbon from orange waste pyrolysis (a), (b) y (c) 2000x, (d) 678x, (e) 403x

Figures 5a and 5b show a honeycomb characteristic of biochar (Patwa et al., 2021). This morphology has been attributed to the pyrolysis of lignin, cellulose, and hemicellulose (Das et al., 2015). However, due to the complex structure of orange waste, different morphologies were obtained by the pyrolysis reaction. Figure 5c shows compact and porous structures. Figures 5d and 5e evidenced a morphology like coiled rods. There was a presence of C, O, Al, N, Mg, and K in the biochar. Carbon was the principal element in the sample (86.19%), followed by Oxygen (12.21%) and Aluminium (0.42%); these data were obtained from the average of five measured biochar sections. Only one shows nitrogen, magnesium, and potassium, probably due to a high content of minerals associated with the structure of the pristine biomass.

CONCLUSION

Thermal effusivity, in addition to the photoacoustic spectrum, gives a first insight into the thermal behavior of the carbon sample obtained from orange waste. As can be seen from the thermal effusivity of the sample, the obtained values are close to those of water; this can be related to the organic origin of the sample.

Infrared spectroscopy showed vibrational bands associated with functional groups like lactones, aromatic rings, and carboxylic acids on the biochar surface.

The morphology found in this study was attributed to the presence of lignin, cellulose, and hemicellulose of the complex biomass used to obtain the biochar. Coiled rods, honeycomb, globular, and compact structures were identified. The biochar is composed principally of C (86.19%), O (12.21%), and Al (0.42%).

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