

Fruit Tree (*Malus domestica*) Branch Biochar via Pyrolysis: Optimization of Temperature and Holding Time

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Discarded apple tree branches from Yantai, China, were pyrolyzed at 300 to 700 °C with holding times (0 to 120 min) to optimize biochar production parameters. Results showed that compared to 300 °C, increasing pyrolysis temperature from 400 to 700 °C (0 min holding) enhanced fixed carbon content 1.39 to 8.30%, carbon content by 16.31 to 28.84%, while reducing C/H ratios 18.29 to 66.33%. When holding time extended from 60 to 120 min across temperatures, fixed carbon content increased 0.31 to 2.56% below 600 °C but showed minimal gains at higher temperatures. Carbon content changes became negligible ($\leq 0.69\%$) above 500 °C with extended holding. Considering both quality and yield, 600 °C pyrolysis temperature with 60 min holding time was identified as the optimal condition for producing biochar with enhanced carbon stability and resource utilization efficiency. This study provided scientific support for converting fruit tree waste into functional carbon materials through controlled pyrolysis processes.

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INTRODUCTION

Fruit trees are important trees of economic value in China. Centering on fruit trees, an ecological industry embodying ecological, economic, and social benefits and integrating the primary, secondary, and tertiary industries is formed to enrich people, playing a crucial role in revitalizing rural areas and building a beautiful China. Since 2022, the global cultivated area of fruit trees has reached 64,859,300 hectares, with China accounting for 20.1% (13,009,530 hectares). In China, the cultivation area of apple trees, pear trees, hawthorn trees, and peach trees accounts for a relatively high proportion. The large-scale cultivation of these fruit trees not only creates significant economic benefits for the local areas but also improves the local ecological environment, contributing to the achievement of the ‘carbon neutrality’ goal. However, in the daily management of orchards, regular pruning and timely removal of old, sick, and dead trees are necessary to ensure the quality and yield of fruits (Brand and Jacinto 2020). According to statistics, in 2022, approximately 10,000 tons of fruit tree waste were generated in China (Liu *et al.* 2023). Due to the high moisture content, low energy quality, and high density of these fruit tree pruning wastes, it is difficult to directly replace coal, thus posing new challenges to the treatment of such wastes (Abdullah and Wu 2009; Vassilev *et al.* 2015).

Such waste is generally disposed of by incineration or burying, which may lead to air, water, and soil pollution. Nevertheless, these discarded substances are rich in organic matters like cellulose, hemicellulose, and lignin. When they are utilized in the processing of biomass for the production of energy, catalytic materials, and other products, they can effectively alleviate the dependence on fossil fuels, reduce the emission of greenhouse gases, and realize the high-efficient utilization of biomass resources. (Liu *et al.* 2023). Currently, converting fruit tree pruning waste into biochar for biomass energy utilization holds great potential (Yousaf *et al.* 2017). Biochar can be prepared with different methods, such as microwave pyrolysis, gasification pyrolysis, hydrothermal carbonization, and pyrolysis carbonization, showing different characteristics and application values. The biochar produced with the microwave pyrolysis method has uniform textures and rich pores. However, microwave-assisted pyrolysis requires stringent reaction conditions and the addition of catalysts. Especially, the biochar produced at a low power (300 W) has poor quality, while that produced at a high power costs high and has a low yield, which in turn affects its performance in specific applications. Gasification pyrolysis is the most common technique to produce syngas, such as carbon monoxide, hydrogen, and methane, from biomass feedstock (Gabhane *et al.* 2020; Yaashikaa *et al.* 2020). With this method, the biochar yield is low and the final conversion rate is only 20%. Moreover, issues, such as the treatment of tar by-products, prevention, and control of secondary pollution, and selection of catalyst types, all restrict the widespread adoption of the gasification pyrolysis method. Hydrothermal carbonization is a process in which liquid biomass is converted into biochar and other products using water as a medium under specific pressure and temperature conditions (ranging from 130 to 250 °C). The biochar produced via this method exhibits a notably lower oxygen-to-carbon (O/C) ratio, higher calorific value, enhanced grindability, and increased hydrophobicity. However, it also has lower carbon content and higher processing costs. Additionally, the stability of the biochar produced through this method is compromised due to the relatively low carbonization temperature (Zhu *et al.* 2019).

Pyrolysis carbonization is simple to operate and cost-effective. The biochar produced with this method has high calorific value, low cost, multiple applications, and can be produced continuously. As such, the pyrolysis carbonization method is extensively used in energy, environmental, and agricultural fields to produce various high-value-added carbon-based materials such as fuels, activated carbon, carbon-based fertilizers, and soil conditioners (Thakkar *et al.* 2016; Januszewicz *et al.* 2020; Li *et al.* 2020).

In the latest research on the pyrolysis of fruit tree branches into biochar, research by Xuele Li *et al.* (2023), systematically evaluated the feasibility of producing biochar from the pyrolysis of fruit tree pruning waste. They investigated the effects of seven tree species, different pruning parts, and temperatures on the pyrolysis process and the physicochemical properties of the biochar. The biochar produced from the pyrolysis of fruit tree pruning waste can serve as an ideal raw material for the production of high-value-added products. Research by Zheli Ding *et al.* (2021), reviewed the main technical methods for biochar preparation from tropical fruit trees, compared the structural characteristics of biochar products prepared under different pyrolysis temperatures, gasification conditions, microwave pyrolysis, and hydrothermal carbonization techniques, and elaborated on the applications of biochar in soil improvement, environmental remediation, energy utilization, and carbon neutrality. This provides scientific guidance and technical support for the preparation of biochar from tropical fruit tree waste and the application of biochar products. Meanwhile, studies have shown that the quality of biochar produced through pyrolysis

carbonization is markedly affected by pyrolysis process parameters, such as pyrolysis temperature, pyrolysis mode, and holding time (He *et al.* 2018). Research by Xinxing Zhou *et al.* (2021), demonstrated that at pyrolysis temperatures of 450 °C, 500 °C, and 550 °C, increasing the temperature could enhance the purity of inorganic oxides in biochar but decrease the biochar yield. However, Divyangkumar and Panwar (2025) argued that the optimal pyrolysis temperature range was between 500 °C and 600 °C. Within this range, the biochar achieved higher calorific value, higher fixed carbon content, and larger specific surface area. With the same carbonization process, the required pyrolysis temperatures vary, depending on the composition and physicochemical properties of the waste to be pyrolyzed (Angın 2012; Ibitoye *et al.* 2024). The following shows previous research findings regarding the impact of pyrolysis methods on the morphology, surface functional groups, pH value, and composition of biochar. Research by Ibitoye *et al.* (2024) proved that the biochar produced through slow pyrolysis at a low temperature had a higher yield and lower ash content than that produced through fast pyrolysis at a high temperature. Ganesan *et al.* (2025) drew similar conclusions, stating that the yield and pH value of biochar produced through rapid pyrolysis were lower than those of biochar produced through slow pyrolysis. The structure of biochar prepared by rapid pyrolysis tended to be less developed. However, as the temperature rose, the ash content increased under both pyrolysis conditions, while the fixed carbon content first increased and then fell and the H/C and O/C molar ratios gradually decreased in both cases. Liu *et al.* (2018) also found that low-temperature slow pyrolysis was beneficial for increasing biochar yield. However, in contrast to the findings of Ganesan *et al.* (2025), they posited that the pH value and degree of aromatization of the carbon product were consistently directly proportional to temperature. Holding time is a critical factor influencing the carbonization effect. Achieving a high biochar yield necessitates a low pyrolysis temperature combined with an extended holding time, which facilitates the re-polymerization of biomass components and allows for adequate reaction time. If the holding time is too short, the re-polymerization of biomass components may remain incomplete, resulting in a reduced biochar yield (Encinar *et al.* 1996; Park *et al.* 2008). Although holding time impacts the composition of liquid and gaseous products during carbonization, an extended holding time can enhance the development of micropores and macropores, increase pore size in the char, and improve the quality of the carbon product (Shaaban *et al.* 2014; Ghorbani *et al.* 2022). The influence of holding time on the carbonization effect is often mediated by parameters such as pyrolysis temperature and heating rate (Fassinou *et al.* 2009).

In summary, different pyrolysis processes have vastly different impacts on the yield and quality of biochar, whereas pyrolysis temperature and holding time are the most important process variables affecting the pyrolysis carbonization process (Sun *et al.* 2017). Currently, the research on the process of directionally preparing biochar from orchard pruning waste is still in a relatively scarce state. Through a literature review, it has been found that in the research on biomass charcoal preparation in the past decade (2015 to 2025), the literature related to orchard pruning waste accounts for only 1.3% (n = 17/1308) of the total research volume, and most of them focus on the macro-level review of the resource utilization of the waste, lacking the research on the characteristics of biochar under specific temperatures and residence times. At the same time, most of the existing studies generally classify orchard waste as ‘wooden biomass’ and fail to conduct specific research on its complex components, resulting in a lack of data support for the optimization of key process parameters such as the pyrolysis temperature gradient (300 to 700 °C) and residence time (0 to 2 h). Therefore, pruned apple tree branches were selected in this study

to explore the dynamic changes in carbonization rate and the quality of carbonized products at different pyrolysis temperatures and holding times. The purpose is to provide a scientific reference for the resource utilization of orchard waste.

EXPERIMENTAL

Materials

In this experiment, the pruned fruit tree branches were collected from an apple orchard in Yantai City, Shandong Province, China. The variety of the fruit trees was Red Fuji (*Malus × domestica* Borkh.), and the trees were 15 years old. The main components of the branches are shown in Table 1. The branches were selected and cut to uniform length and diameter (15 cm × 2 cm) for easy placement in the muffle furnace chamber. They were then dried to constant weight at 45 °C for later use. The muffle furnace was provided by the Biomass and Circular Agriculture Experimental Center at the Yantai Institute of China Agricultural University, with a model number of RGQ1200-100, a controllable temperature range of 300 °C to 950 °C, and a heating rate of approximately 10 °C·min⁻¹.

Table 1. Main Composition of Fruit Tree Branches

Component	Cellulose	Hemicellulose	Lignin	Ash	Volatile	Fixed Carbon	C	H
Content (%)	45.69 ± 0.06	18.22 ± 0.06	22.37 ± 0.06	3.25 ± 0.06	68.66 ± 0.06	28.13 ± 0.06	44.8 ± 0.06	8.8 ± 0.06

Experimental Design

The branches were weighed and placed in the muffle furnace chamber, with pyrolysis temperatures set to 300, 400, 500, 600, and 700 °C and a heating rate of 10 °C·min⁻¹. High-purity nitrogen was used as the protective gas, with a gas flow rate of 1 L·min⁻¹. After reaching the pyrolysis temperatures, the holding times were set to 0, 3, 15, 45, 60, 90, and 120 min, respectively. Each experimental treatment with the same pyrolysis temperature and holding time was repeated thrice to reduce experimental error and ensure the accuracy and representativeness of the experimental data.

Sample Collection and Assay Methods

The extract, cellulose, holocellulose, and lignin contents in fruit trees were determined in accordance with Chinese national standards GB/T 2677.6 (1994), GB/T 2677.10 (1995), and GB/T 2677.8 (1994), respectively. The pH and electrical conductivity (EC) values were tested using a pH meter (Metrohm 691, Switzerland) and an EC meter (WTW inoLab Cond 7110, Germany), respectively. The carbon and hydrogen content in fruit tree branches were determined using a SDCHN 435 elemental analyzer (SDCHN435 Carbon & Hydrogen & Nitrogen analyzer, Changsha, China). The micromorphology of biochar was studied *via* an auto fine coater JEC-3000 FC (Tokyo, Japan) and scanned under an electron microscope (SEM, Tescan Mira4, Czech Republic) to obtain their SEM images. The obtained images were processed using the software ImageJ (National Institutes of Health, Bethesda, MD, USA). The pore size and porosity of the biochar were analyzed using the Analyze-ROI Manager in ImageJ.

Equations

After the branches were processed at the set pyrolysis temperatures and holding times, the pyrolysis products were taken out and treated as biochars prepared at different pyrolysis temperatures and holding times. Then, the biochars were allowed to cool in the air for 5 min, then placed in a drying oven to cool to room temperature, and weighed to calculate the biochar yield, as shown in Eq. 1:

$$Ym = \frac{m_{(end)}}{m_{(start)}} \times 100\% \quad (1)$$

In the formulas: Ym is the biochar yield under pyrolysis conditions, %; $m_{(end)}$ is the mass of the biochar after pyrolysis, g; $m_{(start)}$ is the mass of feedstock before pyrolysis, g.

A total of 1 g (accurate to 0.001 g) of biochar was weighed and placed into a porcelain crucible boat without placing a lid on it. After burning at 700 °C (± 20 °C) for 4 h, the biochar was taken out and allowed to cool in the air for 5 min, then placed in a drying oven to cool to room temperature. The product was then weighed for the calculation of ash content, as shown in Eq. 2:

$$A = \frac{m_2 - m_3}{m_1} \times 100\% \quad (2)$$

In the formulas: A is the ash content of the biochar, %; m_1 is the mass of the biochar before high-temperature treatment, g; m_2 is the mass of the crucible and ash after high-temperature treatment, g; m_3 is the mass of the empty crucible before high-temperature treatment, g.

A total of 1 g (accurate to 0.01 g) of biochar was weighed and uniformly placed at the bottom of a porcelain crucible boat with a lid placed on top. The crucible boat was then placed into a muffle furnace heated to 900 °C (± 10 °C) for 7 min. After that, the product was taken out and placed into a drying vessel for cooling to room temperature. The product was then weighed to calculate the content of volatile matter and fixed carbon, as expressed in Eqs. 3 and 4.

$$V = \frac{m_a - m_b}{m_c} \times 100\% \quad (3)$$

$$C = M - A - V \quad (4)$$

In the formulas: V is the volatile matter content of the biochar, %; m_a is the mass of the crucible and biochar before high-temperature treatment, g; m_b is the mass of the crucible and remaining substance after high-temperature treatment; m_c is the mass of the biochar before high-temperature treatment; C is the fixed carbon content of the biochar; and $M = 100\%$.

Data Statistics and Analysis

The experimental data were statistically analyzed using Microsoft Excel 2016 (Redmond, WA, USA) and SPSS 24.0 (SAS Inc., Cary, NC, USA). The least significant difference (LSD) method was adopted to analyze the significance of differences between groups. The experimental data are presented as "mean ± standard deviation". If $P < 0.05$, it indicates a significant difference; if $P > 0.05$, it denotes there is no significant difference.

RESULTS AND ANALYSIS

The Impacts on the pH and EC Values of Biochar

Compared to the pyrolysis temperature of 300 °C with the same holding time of 0 min, the pH values of biochars produced at pyrolysis temperatures of 400, 500, 600, and 700 °C were 7.63, 7.91, 8.35, and 8.95, respectively, indicating significant variations among the treatments. The pH values of biochars exhibited a distinct upward trend as the pyrolysis temperature and holding time increased. As observed in Table 2, the great increments in pH values occurred at 300 and 400 °C with a holding time of 0 min to 15 min, at 500 °C with a holding time of 15 min to 45 min, and at 600 and 700 °C with a holding time of 45 or 60 min, respectively. Over a holding time of 60 min, the pH values of biochars produced at pyrolysis temperatures of 400, 500, 600, and 700 °C were 8.43, 8.76, 9.53, and 9.73, respectively, that of the biochar produced at 300 °C. Within the holding time range from 60 to 120 min, the increment in the pH value gradually slowed down as the holding time extended. By the holding time of 120 min, the pH values of biochars produced at pyrolysis temperatures of 300, 400, 500, 600, and 700 °C were 8.01, 8.69, 9.22, 9.80, and 10.14, respectively, indicating that the change represents negligible divergence. Those of the biochar produced at the same temperatures with a holding time of 60 min. With the same holding time, large increments were seen in the EC of biochars produced within the temperature ranges of 300 to 400 °C, 400 to 500 °C, and 600 to 700 °C. Within the temperature range of 500 to 600 °C, the EC rose only about 0.1% with the same holding times of 0, 3, 15, 45, 60, 90, and 120 min, showing no marked differences.

Table 2. The pH and EC Values of Biochars

Category	Temperature (°C)	Holding Time (min)						
		0	3	15	45	60	90	120
pH	300	7.21 ^e ± 0.02	7.37 ^e ± 0.06	7.46 ^e ± 0.06	7.67 ^e ± 0.02	7.82 ^e ± 0.02	7.98 ^e ± 0.03	8.01 ^e ± 0.01
	400	7.63 ^d ± 0.03	7.77 ^d ± 0.03	8.01 ^d ± 0.01	8.34 ^d ± 0.04	8.43 ^d ± 0.02	8.60 ^d ± 0.01	8.69 ^d ± 0.02
	500	7.91 ^c ± 0.02	8.18 ^c ± 0.01	8.24 ^c ± 0.01	8.50 ^c ± 0.02	8.76 ^c ± 0.03	9.21 ^c ± 0.02	9.22 ^c ± 0.02
	600	8.35 ^b ± 0.02	8.45 ^b ± 0.04	8.85 ^b ± 0.05	9.25 ^b ± 0.02	9.53 ^b ± 0.02	9.63 ^b ± 0.04	9.80 ^b ± 0.05
	700	8.98 ^a ± 0.02	9.15 ^a ± 0.04	9.43 ^a ± 0.02	9.52 ^a ± 0.03	9.73 ^a ± 0.14	9.94 ^a ± 0.03	10.14 ^a ± 0.03
EC (μS/cm)	300	201 ^d ± 1.53	205 ^d ± 1.73	213 ^d ± 2.65	227 ^d ± 2.08	229 ^d ± 2.31	264 ^e ± 2.52	286 ^e ± 2.00
	400	245 ^c ± 1.53	256 ^c ± 2.31	271 ^c ± 0.58	268 ^c ± 1.29	292 ^c ± 1.00	324 ^d ± 2.89	345 ^d ± 3.21
	500	307 ^b ± 1.53	313 ^b ± 0.50	332 ^b ± 1.53	373 ^b ± 3.79	420 ^b ± 1.53	442 ^b ± 1.73	444 ^c ± 1.53
	600	309 ^b ± 1.53	318 ^b ± 0.53	339 ^b ± 1.53	389 ^{ab} ± 1.53	433 ^b ± 1.53	447 ^b ± 1.53	464 ^b ± 1.52
	700	393 ^a ± 1.53	394 ^a ± 2.00	407 ^a ± 3.51	415 ^a ± 3.00	442 ^a ± 1.53	456 ^a ± 2.00	468 ^a ± 2.00

Note: Different lowercase letters within the same column indicate significant differences ($P < 0.05$) among treatments at different pyrolysis temperatures with the same holding time

The Impact on the Components of Biochar

As the pyrolysis temperature rose and the holding time were extended, the ash content of biochar exhibited a rising trend (Table 3). Compared to the pyrolysis temperature of 300 °C with the same holding time of 0 min, the ash content of biochars produced at 400, 500, 600, and 700 °C increased 1.55 times, 2.57 times, 2.60 times, and 2.77 times, respectively, displaying notable differences among treatments.

Table 3. The Ash, Volatile, and Fixed Carbon Content of Biochars

Category	Temperature (°C)	Holding Time (min)						
		0	3	15	45	60	90	120
Ash (%)	300	5.77 ^e ± 0.02	6.80 ^e ± 0.02	9.18 ^e ± 0.01	9.81 ^e ± 0.01	11.07 ^e ± 0.05	11.72 ^e ± 0.02	11.92 ^e ± 0.01
	400	9.00 ^d ± 0.02	10.23 ^d ± 0.02	12.33 ^d ± 0.02	13.38 ^d ± 0.02	13.49 ^d ± 0.02	14.33 ^d ± 0.02	14.74 ^d ± 0.03
	500	14.87 ^c ± 0.02	15.08 ^c ± 0.02	15.45 ^c ± 0.03	15.59 ^c ± 0.02	15.72 ^c ± 0.02	16.23 ^c ± 0.04	16.46 ^c ± 0.02
	600	15.01 ^b ± 0.02	15.43 ^b ± 0.02	15.80 ^b ± 0.02	15.95 ^b ± 0.02	16.02 ^b ± 0.01	16.47 ^b ± 0.02	17.23 ^b ± 0.03
	700	16.01 ^a ± 0.03	17.48 ^a ± 0.02	17.42 ^a ± 0.02	17.54 ^a ± 0.01	17.64 ^a ± 0.04	17.75 ^a ± 0.01	18.93 ^a ± 0.05
Volatile (%)	300	22.52 ^a ± 0.02	22.00 ^a ± 0.03	18.81 ^a ± 0.06	17.56 ^a ± 0.04	15.57 ^a ± 0.03	14.08 ^a ± 0.03	12.75 ^a ± 0.03
	400	18.03 ^b ± 0.03	16.52 ^b ± 0.02	12.93 ^b ± 0.02	11.31 ^b ± 0.02	9.18 ^b ± 0.01	6.79 ^b ± 0.04	6.49 ^b ± 0.03
	500	9.54 ^c ± 0.02	8.84 ^c ± 0.01	8.41 ^c ± 0.01	6.77 ^c ± 0.03	6.44 ^c ± 0.02	5.49 ^c ± 0.34	5.16 ^c ± 0.04
	600	7.30 ^d ± 0.06	6.52 ^d ± 0.02	5.52 ^d ± 0.02	4.05 ^d ± 0.03	3.69 ^d ± 0.02	3.59 ^d ± 0.02	2.83 ^d ± 0.02
	700	6.33 ^e ± 0.02	5.25 ^e ± 0.02	4.04 ^e ± 0.03	3.90 ^e ± 0.02	3.21 ^e ± 0.02	2.13 ^e ± 0.02	1.72 ^e ± 0.02
Fixed Carbon (%)	300	71.71 ^d ± 0.03	71.90 ^e ± 0.04	72.01 ^e ± 0.06	72.62 ^e ± 0.03	73.36 ^d ± 0.07	74.20 ^e ± 0.03	75.33 ^e ± 0.02
	400	72.97 ^c ± 0.04	73.25 ^d ± 0.02	74.74 ^d ± 0.04	75.31 ^d ± 0.03	77.32 ^c ± 0.02	78.88 ^c ± 0.06	78.77 ^c ± 0.03
	500	73.59 ^b ± 0.01	74.09 ^c ± 0.01	76.14 ^c ± 0.02	77.64 ^c ± 0.04	77.84 ^b ± 0.02	78.28 ^d ± 0.33	78.38 ^d ± 0.02
	600	77.70 ^a ± 0.59	78.05 ^b ± 0.03	78.68 ^b ± 0.03	80.00 ^a ± 0.05	80.29 ^a ± 0.03	79.94 ^b ± 0.00	79.95 ^b ± 0.02
	700	77.66 ^a ± 0.01	77.93 ^a ± 0.00	78.54 ^a ± 0.03	79.56 ^b ± 0.03	79.45 ^a ± 0.05	79.32 ^a ± 0.02	79.24 ^a ± 0.06

Note: Different lowercase letters within the same column indicate significant differences ($P < 0.05$) among treatments at different pyrolysis temperatures with the same holding time

The biochars produced within the pyrolysis temperature range of 300 to 500 °C experienced large increases in the ash content within the holding times of 0 to 3 min, 3 to 15 min, 15 to 45 min, and 45 to 60 min. Compared to a holding time of 0 min, the ash content of biochars produced at pyrolysis temperatures of 300, 400, and 500 °C, respectively, with a holding time of 60 min was 1.92 times, 1.50 times, and 1.07 times those with a holding time of 0 min. From 60 to 120 min, the ash content merely rose about 0% to 1% at pyrolysis temperatures of 300, 400, and 500 °C, indicating no significant differences. Under pyrolysis conditions of 600 and 700 °C, the ascending trend of ash content alleviated as the holding time extended. Statistical analysis demonstrated that the

ash content of biochar had a significantly positive correlation with both pyrolysis temperature and holding time. In contrast, the volatile matter content exhibited a negative correlation with the two factors. In comparison with the pyrolysis temperature of 300 °C with the same holding time of 0 min, the volatile matter content of biochars produced at 400, 500, 600, and 700 °C fell notably by 19.94%, 59.20%, 67.58%, and 71.89%, respectively. With the same holding time, the largest decreases in volatile matter content occurred within the pyrolysis temperature ranges of 300 to 400 °C and 400 to 500 °C. Within the range of 600 to 700 °C, the volatile matter content changed little, remaining between 3% and 7%.

Fixed carbon content, as an important indicator for evaluating the quality of biochar, has a strong positive correlation with both pyrolysis temperature and holding time. Compared to the pyrolysis temperature of 300 °C with the same holding time of 0 min, the fixed carbon content of biochars produced at 400, 500, 600, and 700 °C heightened greatly by 1.66%, 4.02%, 8.35%, and 8.30%, respectively. As listed in Table 2, the biochars produced at pyrolysis temperatures of 300, 400, 500, 600, and 700 °C underwent large rises in their fixed carbon content during the holding times of 40 to 60 min, 60 to 90 min, and 90 to 120 min, respectively. When the pyrolysis temperature was 600 °C, the fixed carbon content reached the highest over a holding time of 60 min. However, when the holding time extended to 90 and 120 min, this data decreased 0.4% and 0.5%, respectively, compared to that by 60 min.

The Impacts on the Elemental Content of Biochar

The C content of biochars showed an ascending trend with the increase in the pyrolysis temperature and holding time (Table 4). When the holding time was 0 min, the C content of biochars produced at pyrolysis temperatures of 400 °C, 500 °C, 600 °C, and 700 °C was 16.42%, 22.67%, 28.58%, and 28.84% higher, respectively, than that produced at 300 °C. By the holding time of 60 min, these data reached 1.14 times, 1.23 times, 1.25 times, and 1.28 times that at 300 °C, respectively. From the holding time of 60 min to 120 min, the biochar exhibited a slow increment in the C content. In contrast, the H content displayed a clear negative correlation with pyrolysis temperature and holding time. With a holding time of 0 min, the H content of biochars produced at 400 °C, 500 °C, 600 °C, and 700 °C was 4.87%, 19.83%, 34.79%, and 54.12%, respectively, lower than that produced at 300 °C. Among them, substantial decreases in the H content were observed when the pyrolysis temperature ranged from 400 °C to 500 °C and from 600 °C to 700 °C. When the pyrolysis temperature reached 700 °C, the H content remained between 1% and 2%. The H/C ratio also exhibited a negative correlation with pyrolysis temperature and holding time. This ratio was lower than 3% when the pyrolysis temperature was 600 °C with a holding time of 60 min.

The Impacts on the Biochar Yield of Fruit Tree Branches

Compared to the holding time of 0 min, the yield of biochars produced at pyrolysis temperatures of 300 °C, 400 °C, 500 °C, 600 °C, and 700 °C with a holding time of 120 min dropped 38.59%, 39.50%, 23.11%, 36.88%, and 25.66%, respectively (Fig. 1). Different pyrolysis temperatures with the same holding time also resulted in decreases in the biochar yield. Within the pyrolysis temperature ranges of 400 °C to 500 °C and 600 °C to 700 °C, the biochar yield fell greatly over holding times of 0 min, 3 min, 15 min, 45 min, and 60 min, by 24.11%, 29.58%, 28.58%, 26.66%, and 22.17%, respectively, as well as 17.66%, 20.41%, 18.64%, 19.41%, and 17.49%, respectively.

Table 4. The C, H, and H/C Ratio of Biochars

Category	Temperature (°C)	Holding Time (min)						
		0	3	15	45	60	90	120
C (%)	300	66.82 ^d ± 0.07	67.44 ^e ± 0.04	69.02 ^e ± 0.11	70.43 ^e ± 0.09	71.22 ^e ± 0.10	72.44 ^e ± 0.12	73.04 ^e ± 0.09
	400	77.72 ^c ± 0.58	78.44 ^d ± 0.12	79.06 ^d ± 0.12	80.44 ^d ± 0.11	81.27 ^d ± 0.15	82.76 ^d ± 0.03	84.00 ^d ± 0.12
	500	81.97 ^b ± 0.08	82.83 ^c ± 0.09	84.02 ^c ± 0.11	85.38 ^c ± 0.07	86.45 ^c ± 0.04	87.27 ^c ± 0.06	88.69 ^c ± 0.05
	600	85.92 ^a ± 0.06	86.29 ^a ± 0.07	87.14 ^a ± 0.09	88.04 ^b ± 0.08	88.16 ^b ± 0.06	88.50 ^b ± 0.03	88.76 ^b ± 0.07
	700	86.09 ^a ± 0.08	86.47 ^b ± 0.03	88.50 ^b ± 0.04	88.77 ^a ± 0.06	89.25 ^a ± 0.02	89.33 ^a ± 0.04	89.93 ^a ± 0.10
H (%)	300	5.95 ^a ± 0.06	5.77 ^a ± 0.11	5.42 ^a ± 0.03	4.95 ^a ± 0.05	4.61 ^a ± 0.06	4.01 ^a ± 0.02	3.34 ^a ± 0.06
	400	5.66 ^b ± 0.07	5.06 ^b ± 0.07	4.86 ^b ± 0.08	4.07 ^b ± 0.06	3.80 ^b ± 0.05	3.56 ^b ± 0.07	3.07 ^b ± 0.07
	500	4.77 ^c ± 0.05	4.02 ^c ± 0.11	3.71 ^c ± 0.08	3.42 ^c ± 0.11	3.18 ^c ± 0.16	3.07 ^c ± 0.10	2.75 ^c ± 0.10
	600	3.88 ^d ± 0.06	3.51 ^d ± 0.12	3.15 ^d ± 0.07	2.92 ^d ± 0.05	2.29 ^d ± 0.11	2.09 ^d ± 0.06	2.08 ^d ± 0.07
	700	2.73 ^e ± 0.10	2.54 ^e ± 0.09	2.19 ^e ± 0.04	1.95 ^e ± 0.04	1.66 ^e ± 0.07	1.51 ^e ± 0.11	1.07 ^e ± 0.02
H/C (%)	300	8.91 ^a ± 0.09	8.56 ^a ± 0.15	7.85 ^a ± 0.03	7.03 ^a ± 0.08	6.48 ^a ± 0.07	5.54 ^a ± 0.02	4.57 ^a ± 0.07
	400	7.28 ^b ± 0.14	6.45 ^b ± 0.10	6.15 ^b ± 0.09	5.06 ^b ± 0.06	4.68 ^b ± 0.06	4.31 ^b ± 0.08	3.65 ^b ± 0.08
	500	5.82 ^c ± 0.07	4.86 ^c ± 0.13	4.42 ^c ± 0.10	4.00 ^c ± 0.12	3.68 ^c ± 0.18	3.52 ^c ± 0.11	3.10 ^c ± 0.11
	600	4.52 ^d ± 0.07	4.07 ^d ± 0.14	3.62 ^d ± 0.09	3.31 ^d ± 0.06	2.60 ^d ± 0.12	2.37 ^d ± 0.06	2.34 ^d ± 0.07
	700	3.17 ^e ± 0.11	2.94 ^e ± 0.11	2.48 ^e ± 0.04	2.20 ^e ± 0.04	1.86 ^e ± 0.08	1.69 ^e ± 0.12	1.19 ^e ± 0.02

Note: Different lowercase letters within the same column indicate significant differences ($P < 0.05$) among treatments at different pyrolysis temperatures with the same holding time

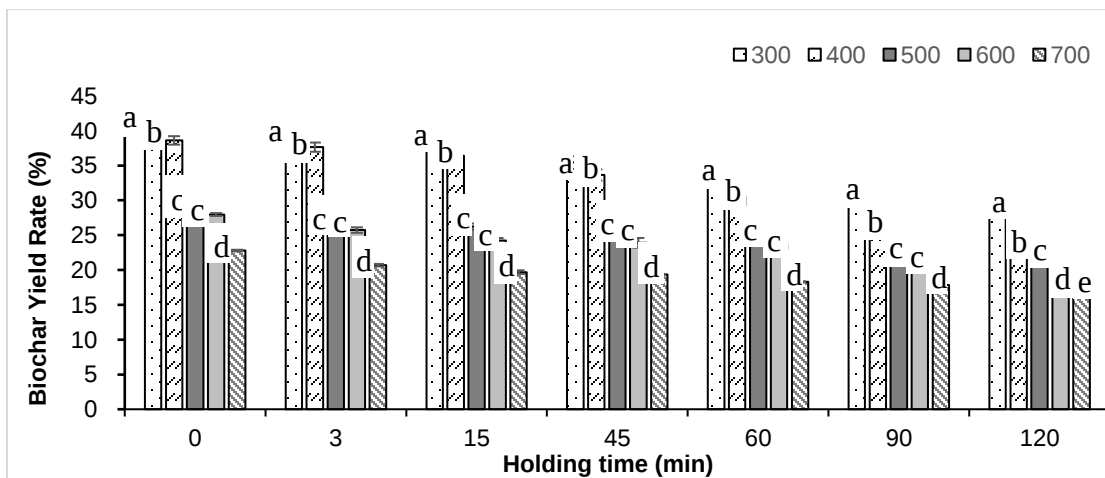


Fig. 1. Biochar yield of fruit tree branches processed with different pyrolysis temperatures and holding time; Note: Different lowercase letters above the bars indicate significant differences ($P < 0.05$) among treatments at different pyrolysis temperatures with the same holding time

These data reflected remarkable differences among treatments. However, when the pyrolysis temperature ranged between 500 °C and 600 °C and the holding time ranged between 0 min and 90 min, the biochar yield only decreased 5% to 6%, showing no marked differences among treatments.

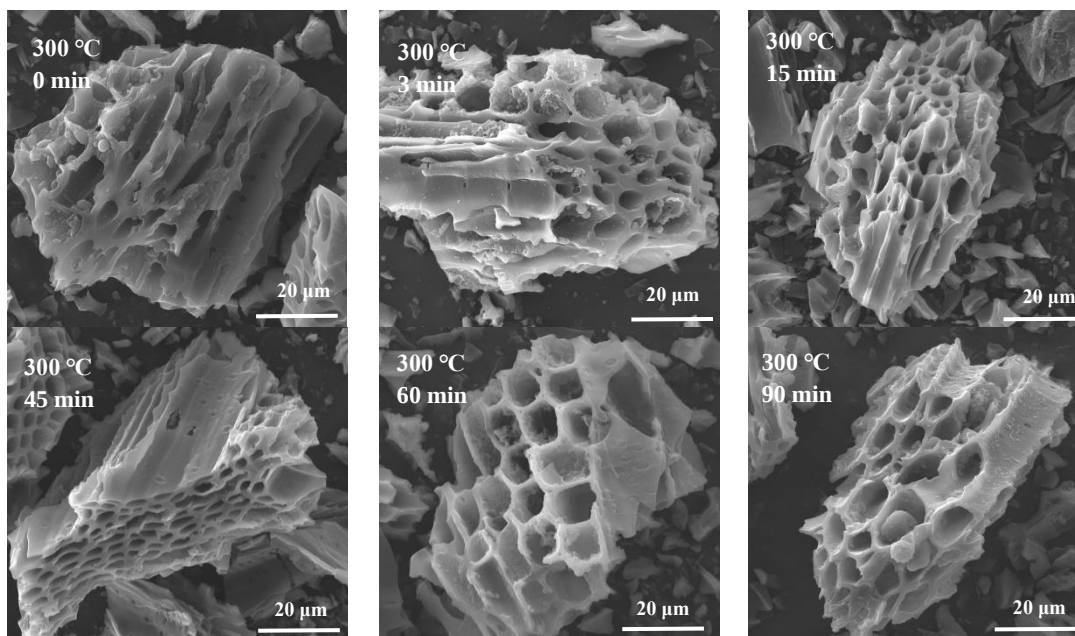
The Impacts on the Surface Morphological Characteristics of Biochar

Through a comprehensive analysis, 600 °C and 60 min were reckoned reasonable pyrolysis temperature and holding time for preparing biochar from fruit tree branches. To further verify the scientific validity of these carbonization process parameters, only the micromorphologies of biochar prepared at pyrolysis temperatures of 300 and 600 °C (Figs. 2 and 3) were characterized in this study. Then, the pore size and porosity of the prepared biochar were obtained based on SEM images and software analysis (Table 5).

Table 5. The Impact of Pyrolysis Temperature on the Pore Structure of Biochar Based on SEM Images

Temperature (°C)	Category	Holding Time (min)						
		0	3	15	45	60	90	120
300	Porosity (%)	35.33 ^b	41.33 ^b	43.00 ^b	51.32 ^b	55.33 ^b	56.37 ^b	60.67 ^a
	Pore Size Range (μm)	0.2 to 15.2	0.2 to 15.8	0.2 to 15.4	0.4 to 15.1	0.7 to 15.8	1.2 to 15.5	1.6 to 16.1
	Average Pore Size (μm)	4.96 ^b	5.23 ^b	5.29 ^b	6.66 ^a	6.71 ^a	7.73 ^a	8.73 ^b
600	Porosity (%)	40.67 ^a	43.67 ^a	46.67 ^a	56.33 ^a	65.00 ^a	61.00 ^a	51.00 ^b
	Pore Size Range (μm)	1.0 to 8.2	1.3 to 10.0	1.5 to 11.6	1.6 to 12.7	3.7 to 84.4	1.9 to 39.9	1.7 to 32.4
	Average Pore Size (μm)	5.02 ^a	5.32 ^a	6.18 ^a	6.89 ^a	13.23 ^b	9.68 ^b	6.32 ^a

Note: Different lowercase letters within the same column indicate significant differences ($P < 0.05$) among treatments at different pyrolysis temperatures with the same holding time.



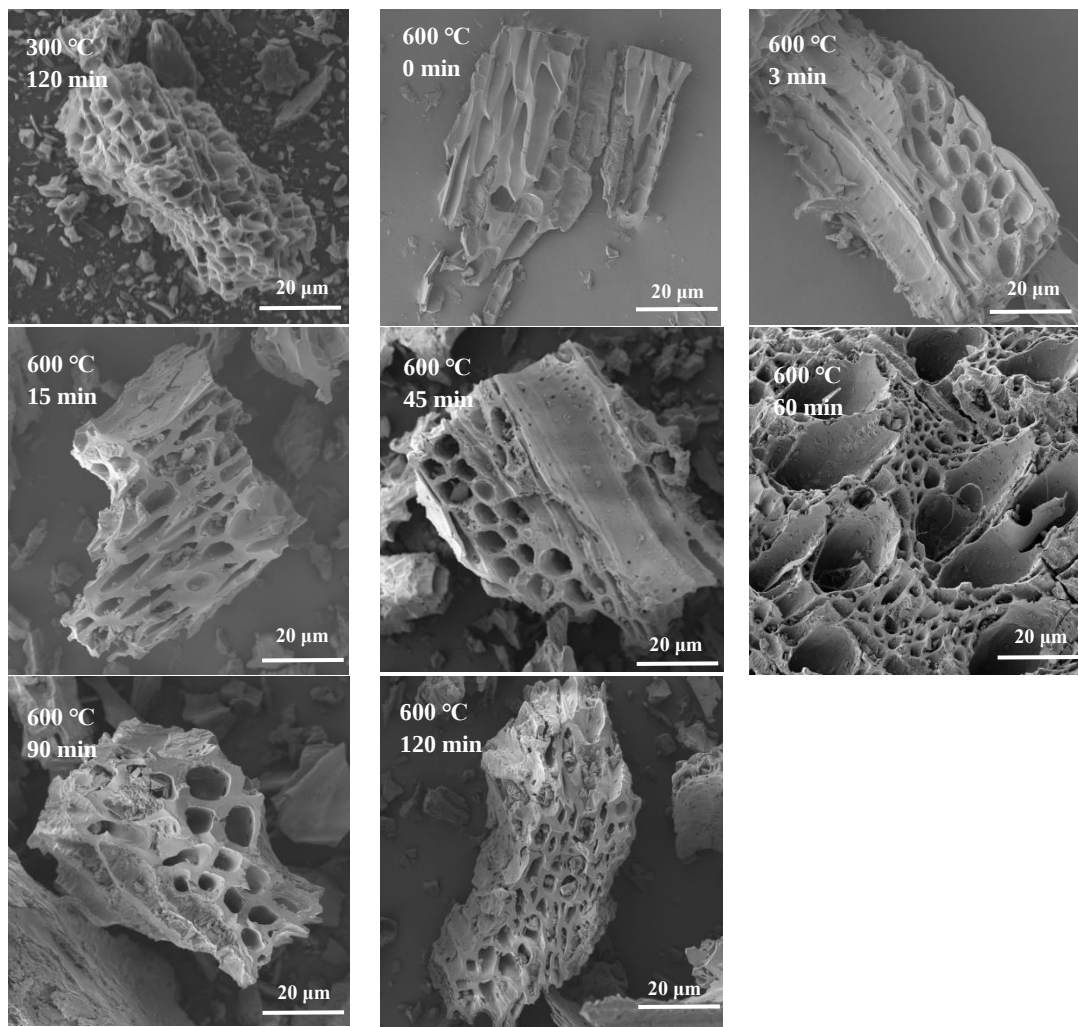


Fig. 2. SEM Images of the biochars prepared at pyrolysis temperatures of 300 °C and 600 °C
 Note: The scale bar represents micrometer-level dimensions (5000× magnification)

At the low pyrolysis temperature of 300 °C, the porosity of biochars produced with holding times of 15 min, 45 min, 60 min, 90 min, and 120 min increased 16.98%, 21.71%, 48.29%, 56.61%, 59.55%, and 71.72%, respectively, compared to that produced with a holding time of 0 min. Similar to the change trend of porosity, the minimum, maximum, and average pore sizes of biochars prepared at 300 °C were significantly in a positive correlation with the holding time. By the holding time of 120 min, these three pore sizes of biochars were enlarged by 8.01 times, 1.06 times, and 1.76 times, respectively, those of biochars produced with a holding time of 0 min. Under the high-temperature pyrolysis condition of 600 °C, the surface morphological characteristic data of biochars increased first and then declined as the holding time extended. By the holding time of 60 min, the porosity, as well as the minimum, maximum, and average pore sizes, of biochars peaked, reaching 1.60 times, 1.60 times, 10.29 times, and 2.64 times, respectively, those of biochars produced with a holding time of 0 min.

Figure 3 illustrates that the biochars prepared at 600 °C with a holding time of 60 min exhibited small, round pores with regular shapes and uniform distribution. However, as revealed through a comparison of data in Table 5, the porosity obtained at the pyrolysis temperature of 600 °C with a holding time of 60 min was 8.19% higher than that obtained

at 300 °C with a holding time of 120 min; the range of average pore size was enlarged by about 5.03 times, with a 51.49% increment in the average pore size. Therefore, 600 °C (pyrolysis temperature) and 60 min (holding time) were more favorable for the formation of the microstructure of biochars compared to 300 °C and 120 min.

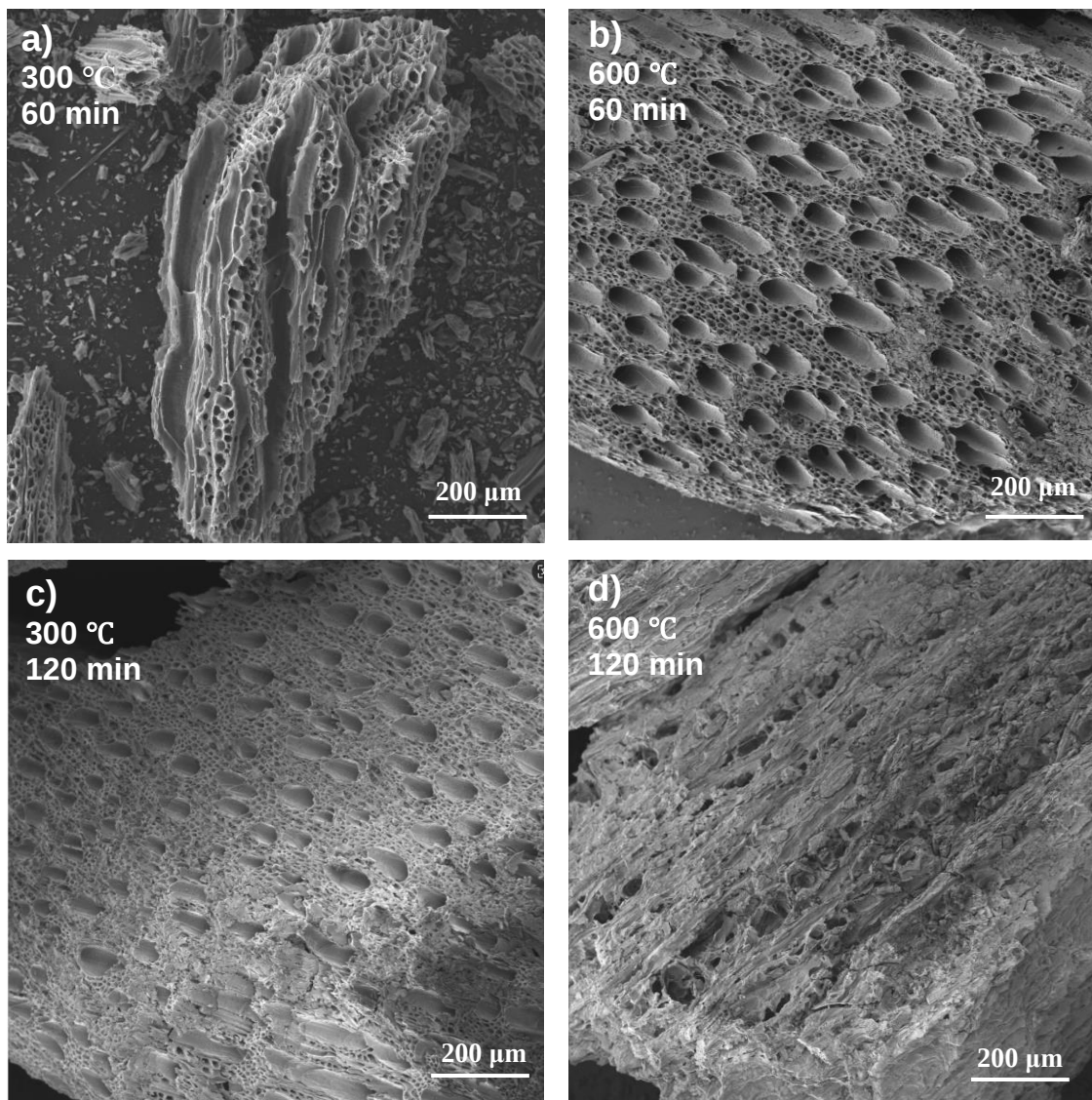


Fig. 3. SEM images of the biochars prepared at pyrolysis temperatures of 300 °C and 600 °C with holding times of 60 min and 120 min

Note: The scale bar represents micrometer-level dimensions (500× magnification)

DISCUSSION

The research results revealed that the pH value of biochar increased with the increases in the pyrolysis temperature and holding time. The primary reason was the decomposition of acidic functional groups, such as carboxyl and phenolic hydroxyl groups, and the volatilization of organic acids from fruit tree branches at different pyrolysis carbonization temperatures. This finding was consistent with the research conclusions of

Singh *et al.* 2022. As the pyrolysis temperature and holding time increased, the increase in the pH value of biochar was marked during the holding time of 0 min to 60 min but slowed down during 90 min to 120 min. This was mainly due to the rapid decomposition of cellulose and lignin, as well as volatilization of the biochar, within the holding time of 0 min to 60 min. Meanwhile, alkaline mineral elements, such as K, Ca, and Mg, were enriched in the ash in the form of oxides or carbonates, leading to a rapid increase in the pH value. Moreover, the biochar surface had rich oxygen-containing functional groups. As the pyrolysis temperature increased, the number of acidic oxygen-containing functional groups on the biochar surface decreased significantly, while the number of alkaline oxygen-containing functional groups continued to increase (Sahoo *et al.* 2021; Bi *et al.* 2023). After a long holding time from 90 min to 120 min, the volatilized gas and liquid products in the biochar lessened, and decomposable substances in the biochar were almost exhausted, resulting in a slow increase in the pH value (Yu *et al.* 2022).

Data have indicated that pyrolysis increased the EC of biochars. This was because high pyrolysis temperature promoted the decomposition of organics, leading to an increase in the carbon content of biochar, which in turn enhanced the biochar's EC (Chen *et al.* 2025). With the same holding time, the biochar underwent a large increase in its EC within the temperature ranges of 300 to 400 °C, 400 to 500 °C, and 600 to 700 °C. The EC showed a significantly positive linear correlation with the pyrolysis temperature and holding time. This result evidenced that many volatile matters and incompletely carbonized components remained in the biochar when the pyrolysis temperature was low. As the temperature rose, soluble salts in the organic matter were pyrolyzed continuously (Chen *et al.* 2025). However, when the pyrolysis temperature ranged from 500 °C to 600 °C, the electrical conductivity (EC) exhibited a less pronounced upward trend. This phenomenon may be attributed to the decomposition and volatilization of mineral elements, as well as excessive carbonization within this temperature range, which leads to the loss of certain functional groups. As the holding time was extended, these reactions became more complete or ceased, resulting in a less significant increase in the EC of biochar (Altıkat *et al.* 2024). Conversely, when the pyrolysis temperature increased to 700 °C, the EC of biochar significantly heightened again. This result indicates that at a pyrolysis temperature of 700 °C, the confinement of the specific surface area was disrupted, allowing more soluble salts within the organic matter to be pyrolyzed continuously (Zhang *et al.* 2015). The composition of biochar varied with the carbonization temperature and holding time. As the pyrolysis temperature and holding time increased, the biochar experienced the dehydration of hydroxyl groups and the thermal decomposition of lignin and cellulose structures. This led to increments in the proportions of minerals and other inorganic components, and consequently, an increase in the ash content (Zhang *et al.* 2022). The increase in the ash content of biochar indicated an enhancement in the determining factors that affected the pH value (Tomczyk *et al.* 2020). This finding was verified in the experiment of this study. The volatile matter content of biochar exhibited a negative correlation with pyrolysis temperature and holding time. This suggested that high temperatures caused unstable carbon-containing substances to undergo continuous reactions and release in gaseous form, promoting the breakage and recombination of chemical bonds in organic functional groups. Meanwhile, extending the holding time allowed more volatile components to be decomposed and released, thus reducing the volatile matter content in biochar (Zhou *et al.* 2021). Within the pyrolysis temperature of 600 to 700 °C, the volatile matter content remained within the range of 3% to 7% as the holding time extended. This implied that compared to pyrolysis temperature, holding time had a minor impact on the volatile matter

content (Yu *et al.* 2022). Fixed carbon content is an important indicator of the stability of biochar. As pyrolysis temperature and holding time increased, the fixed carbon content first increased and then decreased. Within the pyrolysis temperature range of 300 to 600 °C, the increase in fixed carbon content was pronounced. Especially at 600 °C, the fixed carbon content of biochar reached as high as 80.3% over a holding time of 60 min. The main reason was that the biochar produced with this pyrolysis process had the lowest volatile matter content (Papa *et al.* 2024). As the pyrolysis temperature rose and the holding time extended, once the pyrolysis temperature ascended to 700 °C, fixed carbon may experience further decomposition or oxidation, a decrease in the fixed carbon content of biochar was seen due to structural collapse caused by the intense decomposition and volatilization of mineral elements, along with excessive carbonization (Altıkat *et al.* 2024).

The C content of biochar heightened greatly with the pyrolysis temperature, indicating that high pyrolysis temperatures drove the decomposition of organics in biomass, allowing more carbon to be retained in solid form and form a stable carbon structure (Zhang *et al.* 2015). Studies have shown that extending the holding time would promote the accumulation of C (Murtaza *et al.* 2024). However, in this study, this promoting effect of holding time was not marked when the holding time exceeded 60 min. Instead, the C content showed a downward trend. The reason might be that the biochar carbonization had become mature gradually and thus was less affected by the holding time, restricting the increase in the C content (Wang *et al.* 2020).

The H content of biochar decreased with increased in pyrolysis temperature and holding time. This was because of the dehydrogenation and deoxygenation reactions of volatile components in the biomass under high-temperature conditions. Moreover, the increases in pyrolysis temperature and holding time intensified the decomposition and escape of H-containing compounds (Liu *et al.* 2018). H/C, as an important indicator for elemental analysis of polymers, is used to determine the aromatic structure and composition of polymers (Cantrell *et al.* 2012; Do and Nguyen 2024). In this study, the H/C ratio fell with increments in both pyrolysis temperature and holding time. This reflected that high pyrolysis temperatures enhanced the release of volatile matters and the removal of carboxylic acid functional groups, thus facilitating the breakage of weak chemical bonds and the formation of condensation products (Liu *et al.* 2018). This study further uncovered that extending the holding time could further reduce the H/C ratio of biochar, indicating an important impact of holding time on the chemical structure of biochar. With the increase in pyrolysis temperature, biochar displayed increasing aromatization degree, decreasing polarity, and more stable properties (Do and Nguyen 2024).

The yield of biochar decreased progressively with increasing pyrolysis temperature and holding time, which can be attributed to the volatilization of moisture and organic compounds present in the biomass feedstock. Significant reductions in biochar yield were observed across various pyrolysis temperatures and holding times. This finding indicated that major chemical reactions taking place at this stage involved the parallel or sequential decomposition of macromolecules such as cellulose, hemicellulose, and lignin. These reactions resulted in the formation of small-molecule gases and large-molecule condensable volatile matters (Januszewicz *et al.* 2020). When the temperature reached 500 and 600 °C, radical fragments generated from bond breakage recombined, forming very large molecules and coke; the carbonization of lignin, cellulose, and hemicellulose in the branches was nearly completed, with decreasing liquid and gaseous products; the biochar yield tended to stabilize (Xiong *et al.* 2019). When the temperature rose to 700 °C, more

substances were decomposed into oil and gasified, inhibiting the biochar formation, thus demonstrating a decrease in the biochar yield (Altıkat *et al.* 2024).

The biochars produced at a low pyrolysis temperature (300 °C) with holding times from 0 min to 120 min showed unceasing growth in both the porosity and average pore size. This was because, at low temperatures, a longer holding time could promote sufficient polymerization and condensation reactions, thereby contributing to the formation of a more developed pore structure (Cárdenas-Aguilar *et al.* 2024). However, biochars produced at a high temperature (600 °C) peaked both in the porosity and average pore size as the holding time extended up to 60 min. When the holding time went beyond 60 min, the biochars began to show a decline in the porosity, as well as the minimum, maximum, and average pore sizes (Table 5). By the holding time of 120 min, local collapse appeared on the biochar surface (Fig. 3), but the porosity and average pore size were still higher than those of biochar produced at 300 °C (Table 5). The reason might be that at a high temperature, a small carbon network plane would form inside the biochar with the rise in the pyrolysis temperature (to 600 °C); the carbon structure would be rearranged as the holding time extended (Devi *et al.* 2021; Rabiee Abyaneh *et al.* 2024), forming a large-molecule condensed aromatic carbon network plane. This enhanced the graphitization of biochars formed at high pyrolysis temperatures, showing a developed pore structure (Suresh Babu *et al.* 2024). Yet when the holding time was excessively long, volatile matters in the biochar might escape completely, causing the pore structure to lose support and collapse. Alternatively, the ash might become soften or even melt at high temperatures. With the extension of holding time, the molten ash might block the pores already formed in the biochar, leading to a collapse of the pore structure (Tang *et al.* 2017).

CONCLUSIONS

1. The biochars prepared at 400, 500, and 600 °C, compared to 300 °C with the same holding time of 0 min, achieved an increase of 1.39%, 2.25%, and 7.96%, respectively, in the fixed carbon content; the C content increased 16.31%, 22.67%, and 28.58%, respectively; the C/H ratio decreased 18.29%, 34.68%, and 49.27%, respectively. These data suggested that the biochar quality was improved as the pyrolysis temperature increased within the range of 300 °C to 600 °C. Compared with 600 °C, under the pyrolysis temperature of 700 °C, the fixed carbon of biochar showed a downward trend, and the yield of biochar also decreased by about 3%. Therefore, in the range of 600 to 700 °C, the quality of biochar decreased with the increase of pyrolysis temperature.
2. As the pyrolysis temperature increased (to 300, 400, 500, 600, and 700 °C), the biochars prepared with a holding time of 120 min, compared with 60 min, increased 2.56%, 2.35%, 2.30%, 1.02%, and 0.31%, respectively, in its fixed carbon content; the C content elevated by 2.69%, 1.88%, 0.69%, 0.42%, and -0.20%, respectively. This demonstrated that as the pyrolysis temperature rose, the effect of extending the holding time on improving the biochar quality gradually diminished.
3. Biochars produced at a pyrolysis temperature of 600 °C with a holding time of 60 min demonstrated the highest fixed carbon content (80.29%), high carbon content (89.2%), H/C ratio (2.60), and yield (23.7%), and uniform pore structure, meeting the requirements for optimal biochar characteristics. Therefore, 600 °C (pyrolysis

temperature) and 60 min (holding time) were determined as the ideal pyrolysis process parameters for preparing biochar from fruit tree branches.

4. At 300 °C low-temperature pyrolysis, extending the holding time within ≤ 120 min significantly enhances polymerization and condensation reactions, promoting biochar pore development and increasing porosity and average pore diameter. At 600 °C high-temperature pyrolysis, a 60 min holding time optimizes biochar porosity and average pore diameter, indicating an ideal balance between pyrolysis and pore evolution.

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