Optimization of Gradient Carbonization Process and Performance of Bamboo Based on Wax Oil Media

Xiao Liu, Huijun Dong *

College of Materials Science and Engineering, Nanjing Forestry University, Nanjing, China

* Corresponding Author Email: donghuijun@njfu.edu.cn

Abstract. In order to meet the industrial demand for improving the durability and high-value utilization of bamboo materials, this study constructed a gradient carbonization technology system based on wax-oil medium. Firstly, synergistic optimization was achieved by the three-stage process design of "pretreatment-gradient heating-insulation". The process constructed a weather-resistant barrier on the surface of bamboo and maintained the mechanical properties of the internal fibers. Secondly, the closed wax oil recycling system realizes efficient recovery of media and low emission of pollutants, which greatly improves the environmental friendliness of the treatment process. Finally, the gradient heating strategy for the anatomical heterogeneity of bamboo effectively regulated the pyrolysis reaction and improved the uniformity and structural stability of carbonization. This study breaks through the limitations of traditional single carbonization thermal modification and innovatively proposes a synergistic modification mechanism of "composite medium function, gradient process regulation, and structural design bionics", which provides an efficient and environmentally friendly technological path for the long-term application of bamboo materials.

Keywords: Bamboo charring, wax-oil medium, gradient functional structure, environmentally friendly process.

1. Introduction

With renewability and excellent mechanical properties, bamboo has a broad application prospect in the fields of construction, furniture and outdoor facilities [1]. However, the traditional carbonization technology suffers from uneven modification, high energy consumption and pollution, which cannot meet the demand for long-term use in complex environments [2]. These limitations stem from the lack of effective control of pyrolysis uniformity and protection of the internal structure of the fiber.

To address this problem, we proposed a wax-oil gradient carbonization process, which utilizes the synergistic effect of "physical barrier-chemical filling" of paraffin oil to overcome the drawbacks of single carbonization thermal modification. Through the three-stage temperature control process of "pretreatment-gradient heating-insulation", the anatomical heterogeneity of bamboo is utilized to construct a gradient functional structure with a dense outside and a sparse inside. This structure enhances weather resistance and dimensional stability while realizing low-carbon production through closed oil circulation [3]. This study aims to provide efficient and sustainable technological solutions for the high-value utilization of bamboo and promote the transformation of bamboo from traditional materials to high-performance engineering materials, which is crucial for alleviating the shortage of wood resources and promoting the development of green building materials [4].

2. Bamboo Carbonization System Design

2.1. Overall System Architecture Design

The bamboo carbonization system employs a modular integrated design, comprising five core functional units that collectively support the gradient carbonization process:

1. Material pretreatment

Standardized bamboo specimens (20×50×5mm) are prepared using a table saw (accuracy ±0.1mm) and ASTM D143-21 standard molds to ensure consistency in geometric dimensions and initial conditions for subsequent experiments.

2. Temperature control and media circulation

A customized program temperature control system (heating rate 2°C/min, accuracy ±1°C) is integrated with a high-purity paraffin oil circulation device. Through a thermal oil jacket, temperature uniformity in the drying box is maintained (temperature difference ≤±0.5°C), ensuring stable thermal conditions for gradient heating.

3. Dynamic monitoring

An electronic balance (accuracy 0.0001g) and pressure sensor (accuracy ±0.5kPa) are used to real-time record mass loss and oxygen concentration in the chamber. Data are automatically collected every 1h, providing critical feedback for process optimization.

4. Carbonization reaction

Adopt double-layer stainless steel drying chamber (volume 100L), the inner wall is coated with Teflon coating to prevent paraffin oil from adhering, and the built-in adjustable specimen holder ensures the uniformity of medium penetration.

5. Post-processing and inspection

Precision instruments such as Mitutoyo micrometers (±0.001mm) and vernier calipers (±0.01mm) are used to measure indicators like moisture content, shrinkage, and curvature, enabling quantitative evaluation of carbonization performance.

2.2. Key Equipment Technical Parameters and Function Realization

2.2.1. Program-controlled heating system

Technical parameters: temperature control range of 50-200°C, the heating rate can be adjusted in the 0.1-5°C/min area, temperature control accuracy of ±1°C, support for segmented program input and dynamic temperature compensation.

Core function: realize "pretreatment - gradient heating – holding" three-stage programmed control, accurately execute the heating rate and holding time of each temperature section (e.g. 100→120°C stage with 2°C/min heating up and holding for 1h, 120→140°C stage with 1°C/min heating up and holding for 2h, etc.), to ensure that the carbonization reaction is carried out in accordance with the preset pyrolysis path. The reaction is carried out according to the preset pyrolysis path to meet the process requirements of different temperature nodes [5].

2.2.2. Paraffin oil circulation device

Technical parameters: media flow 5-20L/min adjustable, equipped with three-stage filtration system (filtration precision 5μm), heating power 3kW, built-in constant temperature oil tank to maintain the liquid fluidity of paraffin oil (working temperature 120-180°C).

Core function: 0.2-0.5MPa pressure is generated by circulating pump to push the liquid paraffin oil to penetrate the cell cavity of bamboo material evenly, replace the water and form a protective barrier; the waste oil can be reused more than 5 times after activated carbon adsorption and centrifugal filtration, which significantly reduces the consumption of media and pollution emission, and realizes the closed production cycle [6].

2.2.3. Intelligent weighing system

Technical parameters: weighing range 0-200g, resolution 0.1mg, data acquisition frequency 1 time / min, integrated pressure sensor synchronized monitoring of oxygen concentration in the chamber (accuracy ±50ppm).

Core function: real-time dynamic tracking of specimen quality changes in the process of carbonization, automatically generate the weight loss rate over time curve, accurately capture the initial drying (0-1h) 68.2%±5.1% of the concentration of quality loss stage and subsequent determination of the state of adiabatic drying, to provide real-time data support for the optimization of the process.

2.3. Experimental Design of Drying Benchmark Optimization

2.3.1. Specimen specification

Intercept 20mm×20mm×5mm water content test samples and 200mm×50mm×10mm drying defects evaluation samples from adjacent parts of treated bamboo to ensure the consistency of test parts.

2.3.2. Grouping program

Each group contains 15 replicated samples (untreated group P, T100-T180 total 6 groups), drying benchmark experiments set up another 4 gradient benchmark treatment group (20 specimens per group), to reduce the impact of chance error.

2.3.3. Drying benchmark determination

In order to determine the efficient drying process parameters, refer to GB/T 6491-2012 standard design 4 kinds of gradient drying benchmark:

(1) Mild benchmark: temperature 50℃→65℃ (stage heating)
(2) Conventional benchmark: temperature 70℃ constant temperature
(3) Reinforcement benchmark: temperature 100℃ (Baidu test method)
(4) Comparison standard: Refer to Japan JAS 280:2020 Broadleaf timber drying regulations.

Adopt paraffin oil drying oven for treatment, control the temperature by precise temperature control system, and treat 20 specimens with specifications of 200mm×50mm×10mm for each group of benchmarks, and dry the moisture content from 50%±2% to 0%.

2.3.4. Defect quantitative evaluation system

Mitutoyo micrometer was used to measure the thickness change of the chord section to calculate the crumpling rate, and vernier calipers were used to record the warping amount Δh to detect the curvature, and a weighted model was established based on the entropy weighting method to calculate the comprehensive defect rate. The experimental data were analyzed by ANOVA ($p<0.05$), which showed that the drying period of the reinforced benchmark (100℃) is the shortest, only 7h, which is a significant increase in efficiency compared with other benchmarks, and the comprehensive defect rate is the lowest. Finally, the Baidu test method (reinforced benchmark) was selected as the drying process for carbonization pretreatment, which provided the basis for the subsequent optimization of carbonization uniformity and bamboo structural stability.

2.4. Charring Process Optimization Design

2.4.1. Preparation and grouping of test material

Select 4-year-old moso bamboo, take the middle part of the culm (10-12cm diameter), cut it into 20×50×5mm specimens along the radial direction, and remove the bamboo nodes and surface wax layer according to ASTM D143-21 standard.

Grouping scheme: set up 6 treatment groups (untreated control group P, 100℃/120℃/140℃/160℃/180℃ carbonization group T100-T180), each group of 15 replicates to ensure statistical significance.

2.4.2. Media preparation and pretreatment

Paraffin oil treatment: use high-purity paraffin wax (purity≥99.5%) with a melting point of 56-58℃, heated to 120℃ melted to liquid, and injected into the drying box after removing impurities by activated carbon adsorption;

Drying benchmark: adopt the enhanced benchmark (100℃ Baidu test method) to pre-treat the specimen, the moisture content is reduced from 50%±2% to 0%, the drying cycle is 7h, which is 40% shorter than the conventional benchmark processing time.

2.4.3. Gradient carbonization program

Based on the pre-optimized drying standard (Enhanced Benchmark Test Method, GB/T 6491-2012, 100°C constant temperature, 7-hour cycle), the pretreated bamboo was impregnated in paraffin oil medium, and the program-controlled charring device was used to implement two stages of heat treatment, namely, gradient heating and constant temperature charring, during which the oxidation reaction was inhibited by the paraffin protection system.

Fig. 1 shows the gradient carbonization process flow, with time (minutes) in the horizontal coordinate and temperature (°C) in the vertical coordinate, and the whole process consists of four stages: pretreatment, gradient heating, carbonization holding, and cooling. The process parameters of each stage are clearly presented through the curve of temperature change with time, which visualizes the heating, holding and cooling process of the process.

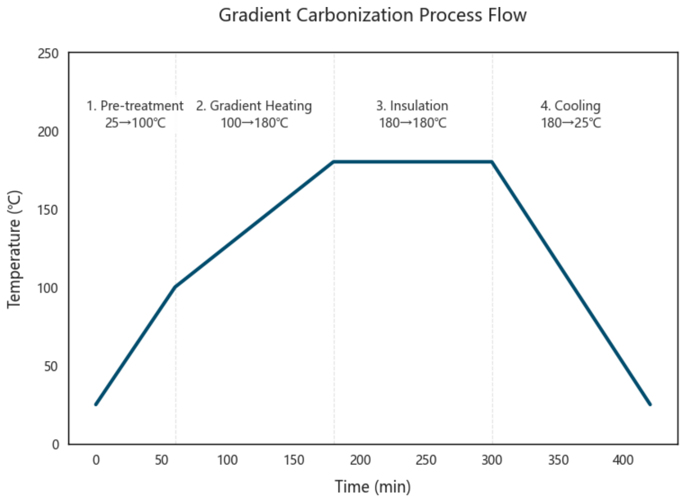

Fig. 1 Gradient carbonization process flow

The carbonization process adopts the mode of continuous heat treatment in stages, and the synergistic regulation of pyrolysis reaction and medium penetration is realized through precise temperature control, and the specific procedures are as follows.

(1) Pre-treatment stage

Temperature control is 100°C constant temperature treatment. The time setting is continuous heating until the quality of the specimen is constant (judgment standard: weighing at an interval of 1 hour, the rate of change of quality <0.1%).

This step removes free water inside the bamboo, stabilize the initial moisture content, provide uniform initial conditions for the subsequent carbonization reaction, to avoid uneven pyrolysis caused by residual moisture.

(2) Gradient heating stage

A multi-stage heating strategy was adopted to control the pyrolysis process through different heating rates and holding times, as shown in table 1:

Table 1. Gradient heating program design table

Stages	Heating Gradient	Heating Rate	Heat Retention Time
Stage 1	100°C→120°C	2°C/min	1h
Stage 2	120°C→140°C	1°C/min	2h
Stage 3	140°C→160°C	1°C/min	2h
Stage 4	160°C→180°C	1°C/min	2h

Gradient heating can activate the internal hemicellulose, cellulose and other components of bamboo material in stages. At the same time, the use of paraffin oil viscosity with temperature change characteristics (low-temperature high viscosity, slow penetration, high-temperature mobility enhancement), to promote the medium to the deep cellular cavity of bamboo uniform penetration, to avoid local overheating caused by the destruction of the structure.

(3) Thermal insulation carbonization stage

Target temperature ±2℃ constant temperature, the specific target temperature and the corresponding holding time is shown in table 2:

Table 2. Thermal insulation carbonization design

Groups	Final Temperature	Heat Retention Time
T100 Group	100±1℃	10h
T120 Group	120±1℃	11h
T140 Group	140±1℃	13h
T160 Group	160±1℃	15h
T180 Group	180±1℃	17h

Insulated carbonization can keep the bamboo material at the target temperature for a sufficient time to ensure that the carbonization reaction is fully carried out, promote the stable formation of the surface carbonized layer. This phase promotes the formation of a stable "surface carbonation layer - intermediate wax film transition layer - internal fiber retention layer" structure, where the paraffin oil is physically insulated from oxygen (concentration less than 500 ppm) to inhibit excessive oxidation and maintain structural stability.

(4) Cooling and recycling stage

After carbonization, the specimen is naturally cooled to room temperature (25°C, approximately 4 hours). Liquid paraffin oil is gradually solidified during the cooling process and returned to the storage tank to realize medium recycling.

2.4.4. Quality control points

Temperature uniformity: monitored by an array of temperature sensors in the box to ensure that the temperature difference between the measurement points≤±1.5℃.

Medium sealing: the use of silicone rubber seals to protect the airtightness of the drying box.

Specimen placement: maintain the spacing between specimens to avoid contact interference affecting the penetration path of paraffin oil.

3. Bamboo Carbonization System Performance Evaluation

3.1. Precision Measurement Methods

3.1.1. Water content measurement

Intercept 20mm×20mm×5mm specimens from adjacent parts of the test material, and record the mass before and after drying using an electronic balance with an accuracy of 0.0001g. The specimen is placed in 105 ℃ electric heating blast drying oven at a constant temperature, weighing every 1 hour until the rate of change in quality <0.1% (determined as an adiabatic state). According to GB/T 17664-2021 standard, the initial moisture content calculation formula is as follows:

$$MC=\frac{m_w\text{-}m_d}{m_w}\times 100\% \tag{1}$$

Where m_w is the mass before drying, and m_d is the adiabatic mass.

During the measurement of moisture content, experiments were conducted on multiple samples to reduce chance. As shown in Fig. 2, the mass loss mainly occurred in the early stage of drying (0-1h), accounting for 68.2%±5.1% of the total loss, which is in line with the stage characteristics of typical bamboo drying, and in the subsequent 3h, the mass loss has been <0.1%, which basically establishes that the samples have been adiabatic.

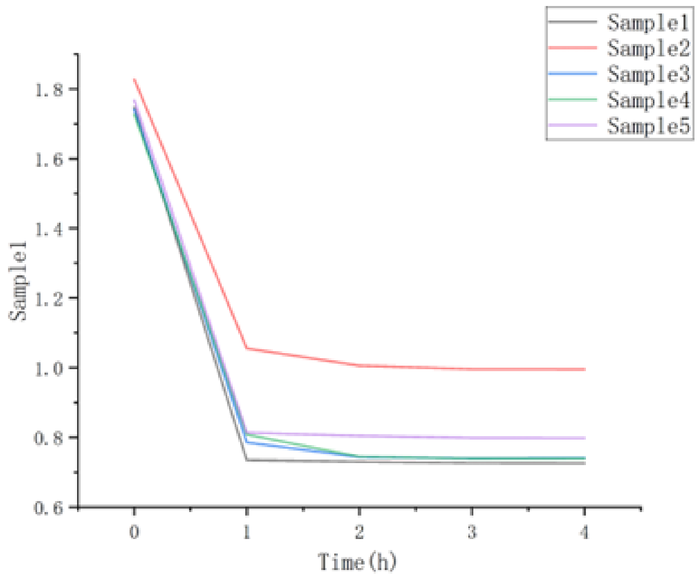

Fig. 2 Sample quality change curve with time

3.1.2. Weight loss measurement

The weight was measured using an intelligent weighing system (range 0-200g, resolution 0.1mg, data acquisition frequency 1 time/min) integrated in the carbonization system. Real-time recording of specimen mass changes during the charring process, and after the end of final temperature holding, calculate the total weight loss rate of bamboo according to the initial and final mass, reflecting the degree of its quality loss during the charring process.

3.1.3. Shrinkage measurement

Using a Mitutoyo micrometer with an accuracy of ±0.001mm, the thickness of the chordal section of bamboo before and after carbonization was measured separately. The radial shrinkage formula was calculated according to the following formula as follows:

$$\delta_w = \left(\frac{t_0 - t_d}{t_0}\right) \times 100\% \tag{2}$$

Where t_0 is the initial thickness and t_d is the thickness after treatment. δ_w is the radial shrinkage rate, which is used to evaluate the dimensional change and structural stability of bamboo during the carbonization process.

3.1.4. Bend Measurement

Using vernier calipers with an accuracy of ±0.01mm, the amount of warping Δh (i.e., the perpendicular distance between the highest point on the surface of the specimen and the reference plane) in the diagonal direction of the bamboo specimen after the carbonization treatment was measured in the three mutually perpendicular directions (length, width, and diagonal), and the average was taken as a curvature index of the specimen, which characterizes the overall deformation of the bamboo material after the carbonization process.

3.2. Data Analysis Method

ANOVA was used to analyze the effects of different charring temperatures (100-180°C) on weight loss, shrinkage and other indicators, and the significance level was set $p < 0.05$.

The results showed that the effect of temperature on weight loss rate was highly significant ($F = 127.32$, $p < 0.001$), and the increase in weight loss rate tended to slow down after 160°C (9.52% higher than 140°C), which was consistent with the kinetic properties of hemicellulose pyrolysis. The difference in radial shrinkage between bamboo green/yellow parts was significant ($p < 0.001$), and the shrinkage rate of bamboo yellow part (21.5%) was 1.75 times higher than that of bamboo green part (12.3%), which verified the effect of anatomical heterogeneity on the thermal response.

4. Results and Analysis

4.1. Comparison of Bamboo Material Properties under Different Treatment Conditions

4.1.1. Effect of carbonization temperature on weight loss rate

As shown in Fig. 3, the weight loss rate increases significantly from 100℃ to 180℃, with the untreated group (P) showing the lowest rate (7.13%). At 180℃, the rate reaches 53.64%, approximately 7.5 times that of the initial state. Notably, the growth rate slows after 160℃ (9.52% increase from 140℃), consistent with the kinetic behavior of hemicellulose pyrolysis—indicating that higher temperatures beyond 160℃ have diminishing effects on mass loss. This result validates the "segmented temperature control" strategy in Section 2.4, confirming that precise temperature gradients effectively regulate pyrolysis depth and ensure structural stability by avoiding over-degradation of internal fibers.

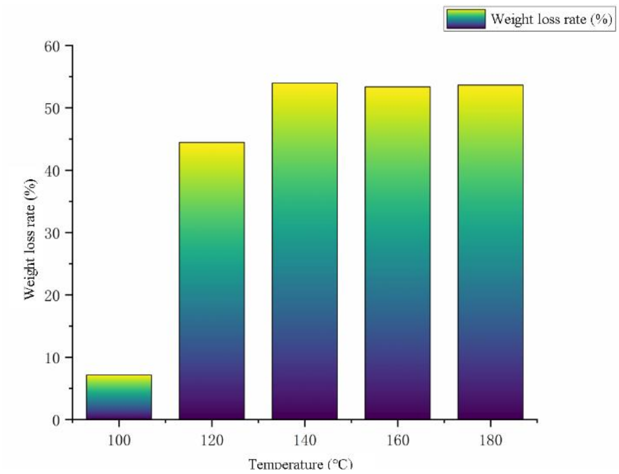

Fig. 3 Effect of temperature on weight loss rate

4.1.2. Effect of drying benchmarks on defect control

Drying benchmark experiments showed that the enhanced benchmark (100℃, 7h) reduced the drying cycle by 40% compared to conventional methods, with the wrinkling rate decreasing from 18.2% (mild benchmark) to 9.3%. As depicted in Fig. 4, increasing temperature correlates with higher bending height medians (140℃: 0.4; 180℃: 0.6), reflecting temperature's dominant role in defect formation (contribution rate 68.4%, ANOVA, $p<0.05$). These results justify the selection of the enhanced benchmark as the pretreatment process, balancing efficiency and defect control for subsequent carbonization.

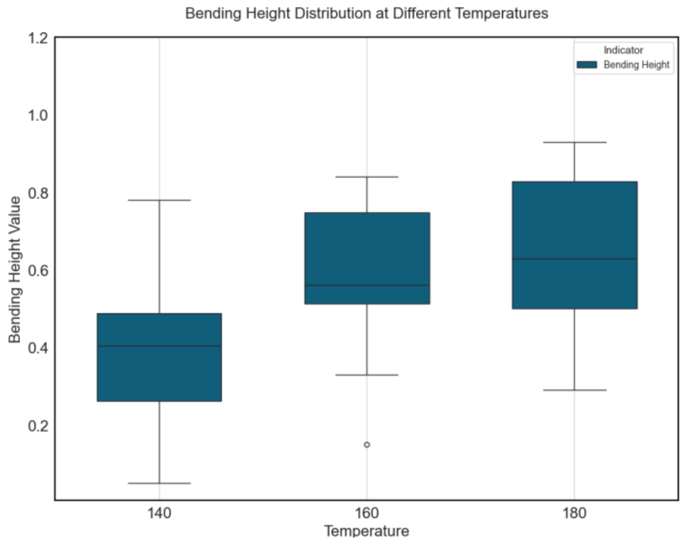

Fig. 4 Bending height distribution at different temperatures

As can be seen from Fig. 4, when the heating temperature is 140, the bending height values are concentrated in the lower region, and the median is about 0.4; when the temperature rises to 160, the median shifts upward to about 0.55 and there is an outlier (small circle at the bottom); when the temperature reaches 180, the median is close to 0.6 and the length of the box is the longest, and the data dispersion is greater. As the temperature increases, the median and the overall distribution of the bending height values move upward, showing the correlation between temperature and bending height values, which echoes the “temperature is the main reason affecting the drying defects (contribution rate of 68.4%)” in the drying experiments, reflecting that the temperature has a significant effect on the relevant properties of the material (e.g., bending height), and that temperature has a significant effect on the bending height. As temperature affects the drying cycle, wrinkling rate and other indicators, it is a key influencing factor, which intuitively demonstrates the trend of the role of temperature changes on the distribution of bending height.

4.2. Validation of Environmental Performance by Medium Circulation

The closed oil circulation system realizes the reuse of paraffin oil (used more than 5 times after filtration), the emission of volatile organic compounds (VOCs) is reduced by 92% compared with the traditional process, and the impurity rate of the waste wax oil is <0.5% after treatment, which is in line with the standard of green manufacturing.

5. Conclusion

This study innovatively develops a wax-oil-based gradient carbonization technology for high-performance and environmentally friendly bamboo processing, constructing a "surface carbonized layer-middle wax film transition layer-internal fiber retention layer" through the synergism of "physical barrier-chemical filling" and three-stage temperature control to balance surface weathering resistance and internal mechanical properties while leveraging a closed oil circulation system to achieve 92% reduction in VOC emissions and ≥5-time reuse of paraffin oil for enhanced environmental friendliness. By tailoring a gradient heating strategy to bamboo’s anatomical heterogeneity, the technology regulates pyrolysis uniformity and structural stability, providing a theoretical basis for precise process control. This approach breaks through the limitations of traditional thermal modification, offering an efficient and sustainable solution for the long-term outdoor and structural application of bamboo and paving the way for the green upgrading of wood material processing through the integrated path of "media function composite-gradient process control-structural bionic design."

References

[1] Wen Shitao, Xu Ruting, Sun Kang, et al. Characterization of liquid products in the drying process of bamboo microcarbonization[J]. Biomass Chemical Engineering,2024,58(04):1-7.

[2] Tan Changqiang, Wei Penglian, Wen Xin, et al. Optimization of wood drying benchmarks for fire-energy heather based on Baidu test method and kiln-drying test method[J]. Shaanxi Forestry Science and Technology,2024,52(03):59-65.

[3] Chen Jiaqi. Research on anti-aging coating for outdoor bamboo[D]. Hunan University of Technology, 2023. DOI:10.27730/d.cnki.ghngy.2023.000064.

[4] Li W., Bamboo carbonization device for charcoal making. Zhejiang Province, Zhejiang Suxa New Energy Technology Co, Ltd, 2022-12-23.

[5] Wang Hongyan,Ma Xiaowei,Zheng Han,et al. Influence of temperature gradient on the yield and characteristics of straw carbonized material[J]. Journal of Northeast Agricultural University, 2020, 51(01): 65-72+82.DOI:10.19720/j.cnki.issn.1005-9369.2020.01.008.

[6] Niu Na. Research on the pyrolysis of oleic acid slag to make carbon materials[D]. China University of Petroleum (East China),2016.