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Influence of Heating Curve and Phenolic Resin Concentration on Mechanical Properties of Wood-ceramics

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Wood ceramics represent a promising class of porous carbon materials offering potential solutions for sustainable air filtration due to their unique structural properties. This study systematically investigates the influence of heating curves and phenolic resin concentrations on the mechanical properties and microstructure of wood ceramics derived from wood powder and fibers. The specimens were impregnated with varying concentrations of phenolic resin and sintered under different heating strategies to optimize their structural integrity. Experimental results demonstrated that increasing the phenolic resin concentration significantly enhanced mechanically performance; specifically, raising the concentration from 30% to 50% resulted in an increase in compressive strength of up to 11 MPa. Scanning electron microscopy (SEM) analysis revealed that optimized sintering parameters facilitated the formation of a robust glassy carbon network, effectively bonding the wood reinforcement to the carbon matrix while maintaining a favorable pore structure. The findings indicate that optimizing the carbonization process and resin content is crucial for balancing the dimensional stability and mechanical strength of wood ceramics, thereby validating their feasibility as durable substrates for air filter elements.

Keywords: wood-ceramic, air filter, mechanical properties, microstructure.

1. INTRODUCTION

Particulate matter pollution derived from vehicle exhaust, industrial emissions [1, 2], and diverse combustion sources has emerged as a critical global environmental challenge that poses severe risks to human health and respiratory systems [3]. Consequently, the development of high-efficiency air filtration systems has garnered significant research attention [4]. Although traditional air filter media such as polymer nanofibers and glass fibers demonstrate effective filtration performance, they are often constrained by poor thermal stability, low mechanical rigidity, and non-biodegradability. These limitations restrict their utility in harsh environments such as high-temperature exhaust filtration and complicate post-use disposal [5]. Therefore, it is imperative to explore novel, sustainable, and robust porous materials capable of serving as durable air filter elements.

Wood ceramics have emerged as a promising class of eco-friendly porous materials [6] that offer potential solutions to these challenges. Fabricated by impregnating wood-based precursors such as wood powder or fibers with thermosetting resins, typically phenolic resin, followed by carbonization under a vacuum or inert atmosphere, wood ceramics inherit the natural porous structure of wood while acquiring the superior properties of glassy carbon [7]. Owing to their intrinsic properties, including high hardness, excellent friction characteristics, electromagnetic shielding capabilities, and corrosion resistance, wood ceramics have been widely investigated for various engineering applications [8, 9].

However, the deployment of wood ceramics as structural components for air filtration requires a delicate balance between porosity and mechanical strength [10]. The fabrication process is complex, particularly during the carbonization stage, where the thermal decomposition of wood components and the curing or carbonization of phenolic resin occur simultaneously alongside the release of volatile byproducts [11 – 13]. Improper heating rates or unoptimized resin concentrations can lead to excessive mass loss, shrinkage, macroscopic cracking, or structural collapse, thereby severely compromising the mechanical integrity of the final product [14 – 16]. While previous studies have focused on the tribological or electrical properties of wood ceramics, the systematic optimization of sintering parameters such as heating curves and resin content to enhance mechanical properties for filtration applications remains underexplored.

To address these issues, this study systematically investigates the influence of heating curves and phenolic resin concentrations on the mechanical properties and microstructure of wood ceramics derived from wood powder and fibers. By comparing different heating strategies, specifically focusing on the heating rate during the critical decomposition temperature range, and varying the phenolic resin impregnation concentration from 30% to 50%, this work aims to elucidate the relationship between fabrication parameters and the formation of the glassy carbon network. The findings of this research provide theoretical guidance and optimized processing parameters for fabricating high-strength wood ceramics, validating their feasibility as effective and durable candidates for sustainable air filtration applications. While the ultimate

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goal is air filtration, this study focuses on the critical prerequisite of mechanical stability during the sintering process.

2. EXPERIMENTAL DETAILS

The woodceramic preparation process involves phenolic resin impregnation of wood powder/wood fiber, drying and compression molding, carbonization and sintering, as shown in Fig. 1. Wood powder/wood fiber is impregnated with phenolic resin prepared (Grades 2130, Zhengzhou Hengtong Chemical Co., Henan,) in anhydrous ethanol for 4 days, as shown in Table 1.

Fig. 1. Wood-ceramic manufacturing process

The impregnated wood powder/wood fiber is dried in a forced-air oven, then placed into a ∅25 mm, 5 mm-thick mold, and sintered at 60 °C wood fibers in a 30 % phenolic resin solution prepared with anhydrous ethanol for 4 days. The impregnated wood powder/wood fibers are dried in a forced-air drying oven, then placed into a ∅25 mm, 5 mm-thick mold. After curing in a 60 °C forced-air drying oven for 12 hours, the mold is removed. The material is then sintered into wood ceramics in a vacuum tube furnace under a nitrogen atmosphere.

Table 1. Experimental design for phenolic resin impregnation ratios

No.	Wood flour: wood fiber (mass)	Phenolic resin (concentration)
1	1:0	30 %
2	1:0	40 %
3	1:0	50 %
4	0:1	30 %
5	0:1	40 %
6	0:1	50 %
7	1:1	30 %
8	1:1	40 %
9	1:1	50 %

Wood ceramic specimens were tested for flexural strength against compressive strength using a universal electronic testing machine (Instron 5967). The maximum load at fracture of the specimens was recorded and the flexural strength was calculated according to the standard formula. The wood-ceramic specimen sections were gold sprayed (Quorum Q150R) and placed under a scanning electron microscope (SEM, JSM-IT800) to observe the micro-morphology of the surface and fracture, the pore structure, the distribution of carbon phases, and the bonding of the wood fibers/wood flour to the carbon matrix.

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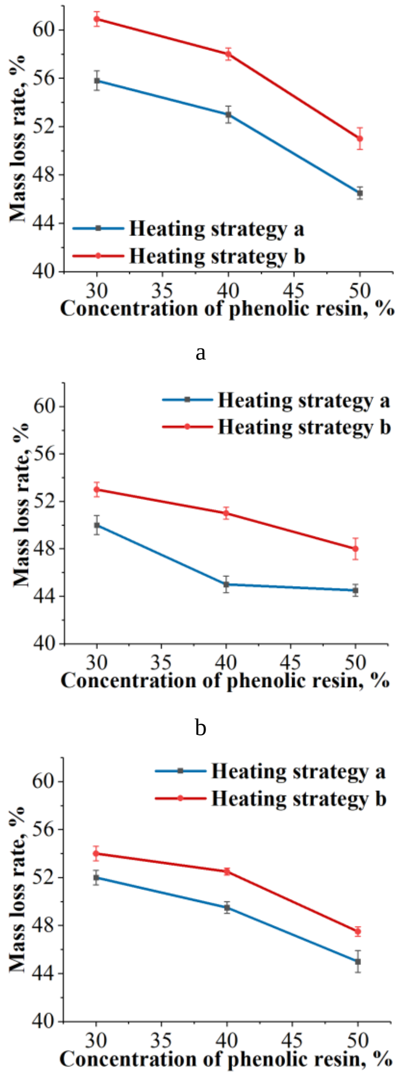
To investigate the effect of the heating rate on the preparation of wood ceramics during the sintering process. Two heating strategies were used based on the thermal properties of wood and phenolic resin. 5 °C/min was taken at both 0 ~ 150 °C. The heating strategy a took a heating rate of 1 °C/min before 600 °C and 5 °C/min at 600 ~ 800 °C. The heating strategy b takes a 5 °C/min heating rate before 600 °C and a 1°C/min heating rate at 600 ~ 800 °C. Both heating strategies were held for 100 min after the temperature reached 600 °C and 240 min after 800 °C.

3. RESULTS AND DISCUSSION

The mass loss rates of sintered specimens using wood flour with different phenolic resin concentration ratios under heating strategies a and b are shown in Fig. 2 a. The mass loss rate of sintered specimens using wood fiber with different phenolic resin concentration ratios under heating strategy a and b is shown in Fig. 2 b. The mass loss rate of sintered specimens using wood flour/wood fiber with different phenolic resin concentration ratios under elevated temperature strategy a and b is shown in Fig. 2 c.

According to Fig. 2, it can be found that the specimens sintered under heating strategy b have a higher mass loss rate with the same phenolic resin concentration. Compared with the heating strategy b, using a heating rate of 1 °C/min during the process of 150 °C ~ 600 °C can better enable the thermal decomposition reaction of wood material and phenolic resin, and the slow heating process is favorable for the discharge of small molecule decomposition products; at the same time, with the increase of phenolic resin concentration, the mass loss rate of the specimen gradually decreases, and it has the same performance under both heating strategies, but it can be found that, at a However, it can be found that in the 30 % ~ 40 % phenolic resin concentration section, the mass loss rate decreases much faster than that in the 40 % ~ 50 % section, which is due to the fact that with the increase of phenolic resin concentration, the content of phenolic resin infiltrated into the pores of the wood material increases, and the glassy carbon formed after the thermal decomposition has a high mass ratio, the char yield increases, and the mass loss rate decreases gradually; in the same temperature-raising strategy and the phenolic resin concentration, the mass loss rate of wood flour is 5.5 times higher than that of wood fiber, and the mass loss rate of wood flour is 5.0 times higher than that of wood fiber.

Under the same heating strategy and phenolic resin concentration, the mass loss rate of wood flour is about 5.7 % higher than that of wood fiber, which is due to the existence of wood fiber tubular cells and through holes in the impregnation process, which makes the phenolic resin penetrate into the interior of the wood fibers in a better way, and it has a higher impregnation rate compared with that of wood flour.



a

b

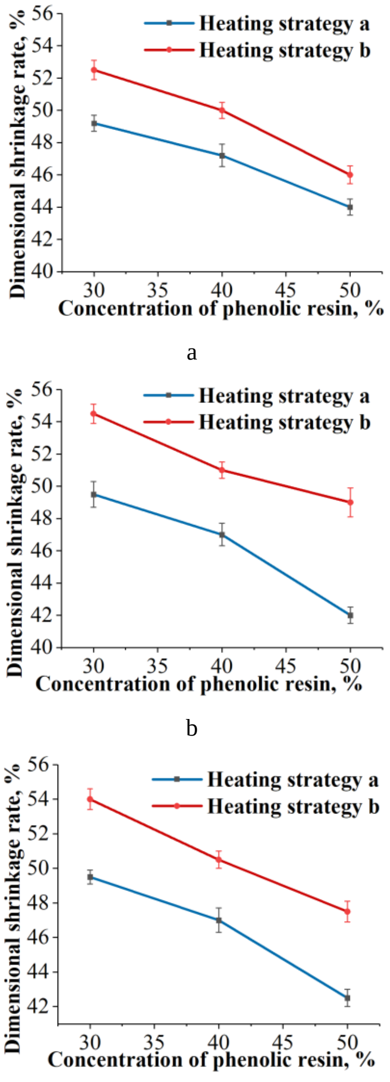
c

Fig. 2. The effect of different phenolic resin concentration ratios on the mass loss rate during sintering of the specimens

The thermal decomposition of phenolic resin into glassy carbon during sintering reduces the mass loss of wood fiber, which results in a higher mass loss rate for wood flour than for wood fiber.

The dimensional shrinkage of sintered specimens using wood flour with different phenolic resin concentration ratios under elevated temperature strategies a and b is shown in Fig. 3 a. The dimensional shrinkage of sintered specimens using wood fiber with different phenolic resin concentration ratios under heating strategies a and b is shown in Fig. 3 b. The dimensional shrinkage of specimens sintered under heating strategies a and b using wood flour/wood fiber with different phenolic resin concentration ratios is shown in Fig. 3 c.

According to Fig. 3, it can be found that under the same phenolic resin concentration, the sintered specimens under heating strategy b have higher dimensional shrinkage, which is due to the fact that the faster the heating rate is during the heating process from 150°C to 600°C, the faster the thermal decomposition of phenolic resin can't be carried out completely and then it enters into thermal decomposition at higher temperature, so the amount of glassy carbon produced is lower than that generated under the low heating rate, and the dimensional shrinkage becomes higher and the dimensional stability deteriorates.



a

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Fig. 3. The effect of different phenolic resin concentration ratios on the dimensional shrinkage rate during sintering of the specimens

The amount of glassy carbon produced is lower than the amount of glassy carbon produced under a low temperature rise rate, so the dimensional shrinkage rate becomes high and the dimensional stability becomes poor; under the same temperature rise strategy, the larger the concentration of phenolic resin, the smaller the dimensional shrinkage rate. The glassy carbon generated by thermal decomposition of phenolic resin has high strength and acts as a skeleton to support the internal structure of wood ceramics. The more phenolic resin content, the more stable the dimensional structure.

The wood-ceramic engine air filter element will be subjected to a certain load exerted by the gas in the process of use, through the test of the physical and mechanical properties of the wood-ceramic engine air filter element to characterize its ability to withstand the load, which can provide a certain basis for the reliability of its work. Experiments were conducted to analyze the effects of heating strategy a, heating strategy b and different concentrations of phenolic resin on the flexural strength of the three wood ceramics. The experimental results are shown in Fig. 4.

Fig. 4 shows the effects of heating strategy a, heating strategy b and different concentrations of phenolic resin on the flexural strength of the three wood-ceramic components.

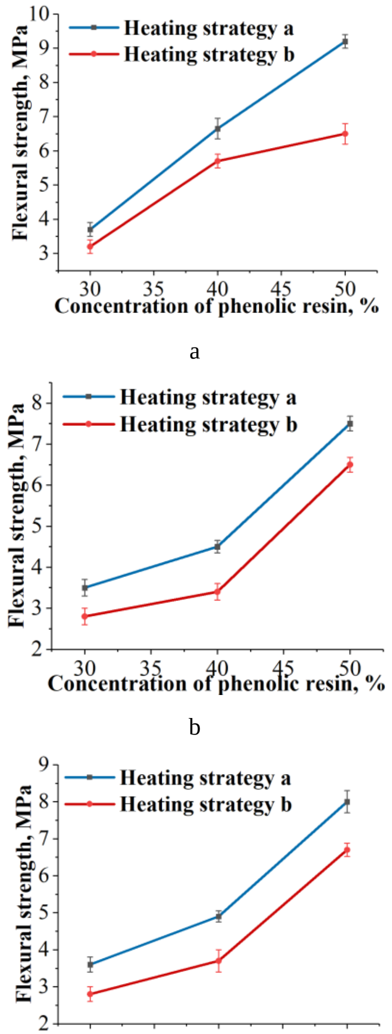

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Fig. 4. The effect of phenolic resin concentration on the flexural strength of wood ceramic: a – wood powder ceramic structure; b – wood fiber ceramic structure; c – wood powder/fiber ceramic

It can be seen that the fracture strength of the wood-ceramic engine air filter element sintered by heating strategy a is significantly better than that of heating strategy b, with a difference of up to 2 MPa. During the sintering and carbonization process, the carbon aromatic ring structure starts to form when the temperature reaches 550 °C to 600 °C. The heating rate of heating strategy a is relatively gentle, and as the temperature increases, the carbon polycyclic ring structure starts to form. Due to the gentle heating rate of heating strategy a, the number of carbon atoms in the carbon polycyclic structure increases with increasing temperature, the arrangement between atoms is more regular, and the graphite microcrystals are more organized, with higher flexural strength.

The flexural strength of the wood ceramics increased with the increase of phenolic resin concentration at the same heating strategy, and the wood powder ceramics prepared by both heating strategies had high flexural strength, and the wood fiber ceramics had the lowest flexural strength. The glassy carbon generated by the phenolic resin has high hardness, and the amorphous carbon generated with the wood material can be fused and coexist together at high temperatures, and the fusion interface gradually disappears with the increase of temperature, and the two different forms of carbon are combined to enhance the strength of the chemical bond. Wood flour, due to its excellent physical properties, is better able to fuse with the glassy carbon produced by phenolic resins, and the strength of the chemical bond formed is higher than that of wood fiber.

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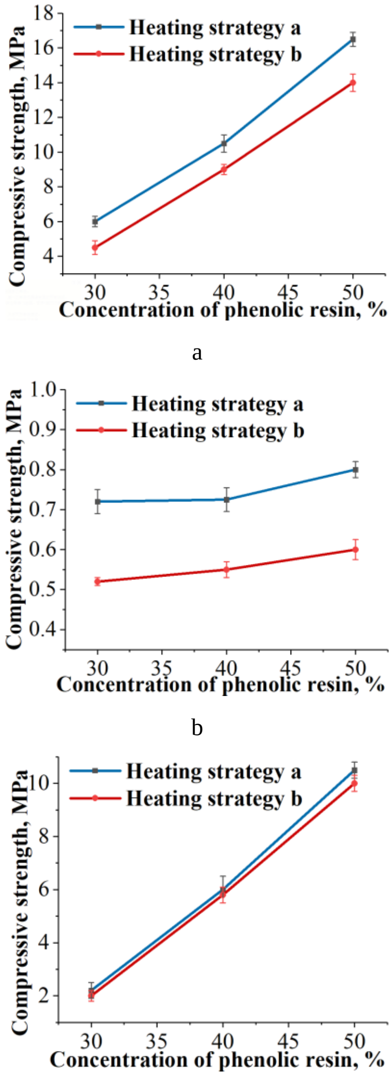
Different heating strategies have a certain effect on the compressive strength of wood ceramics, and the effect is more prominent for wood fiber ceramics. The increase of phenolic resin concentration can significantly improve the compressive strength of wood ceramics. When the phenolic resin concentration is increased from 30 % to 50 %, the compressive strength is increased by up to 11 MPa, but the effect on improving the compressive strength of wood fiber ceramics is not obvious.

To contextualize this performance, traditional cellulose-based filter papers typically exhibit tensile and compressive strengths in the range of 2 – 5 MPa, as reported by Hervy et al. [17] and Lin et al. [18]. Therefore, the wood ceramics developed here offer superior structural integrity. However, it should be noted that our values are lower than the 15 – 20 MPa reported for metal-doped wood ceramics by Zeng et al. [19]. Nevertheless, the metal-free composition of our material provides distinct advantages in terms of weight reduction and eco-friendly disposal.

As shown in Fig. 6 for the planar electron microscope images of wood powder ceramics, wood fiber ceramics, wood powder/wood fiber ceramics, it can be seen that all three ceramics have a more obvious glassy carbon continuous phase, the continuous phase is most prominent in the wood fiber ceramics, and the stacking morphology of the wood powder ceramics is a granular stacking.



a

b

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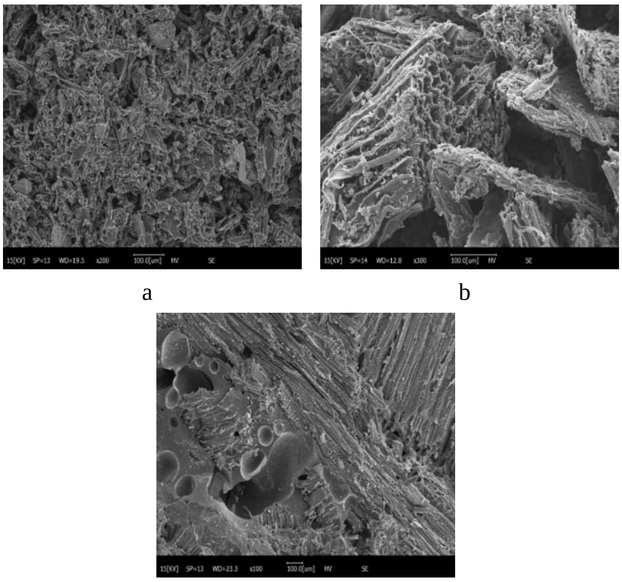
Fig. 5. The effect of phenolic resin concentration on the compressive strength of wood ceramic: a – wood powder ceramic structure; b – wood fiber ceramic structure; c – wood powder/fiber ceramic

In the wood fiber and wood flour/wood fiber in the micrographs can be clearly observed in the tube cell structure of the wood material, a part of the phenolic resin is filled in the distribution of complex inter-fiber spaces, which enhances the structural strength of the inter-fibers and improves the mechanical properties of the cell wall. The amorphous carbon generated by the wood material after high-temperature carbonization is gray, the glassy carbon generated by phenolic resin is white, and the pores appear black in the micrograph. In the carbonization process, because the shrinkage rate of wood material after carbonization is larger than that of phenolic resin, the spacing of wood material increases after carbonization, and the pores have a tendency to increase. Although quantitative permeability tests are reserved for future work, SEM analysis (Fig. 6) qualitatively indicates that samples with 30 % resin retain a higher volume of open pores compared to the 50 % group.



a

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Fig. 6. SEM images of wood ceramic specimens: a – wood powder ceramic structure; b – wood fiber ceramic structure; c – interface bonding between carbon matrix and reinforcement

This suggests a trade-off where higher resin content improves strength but may reduce permeability, a factor that must be optimized for filtration efficiency.

In the micrograph, the fine precipitation phase generated by the carbonization of phenolic resin can be observed, and the phenomenon of fracture of the wood material did not occur. The cell wall of the wood material was strengthened by the phenolic resin, and the organization structure was more stable and relatively regular. Due to the fluidity of the phenolic resin when cured by heat, the distribution of the phenolic resin was not uniform after curing, resulting in a relatively dense surface near the bottom part of the material when sintered. The wood powder ceramic material has small internal pores and a relatively dense structure, and the material shows topological homogeneity as a whole. The wood fiber ceramics have better porosity compared to the other two ceramics, the tubular cell structure is well preserved after high-temperature sintering, the glassy carbon improves the strength of the structure, and the amorphous carbon and the glassy carbon are able to form a better lattice structure.

SEM analysis revealed that optimized sintering parameters facilitated the formation of a robust glassy carbon network. This result validates our initial expectation that a controlled heating rate is essential to prevent structural collapse during the rapid volatilization phase. Consistent with the pyrolysis kinetics described by Lu et al. [20], the optimized heating curve allowed for the gradual release of volatiles, thereby maintaining a favorable pore structure and bonding the wood reinforcement effectively to the carbon matrix.

4. CONCLUSIONS

In this study, wood-based ceramic air filter elements for engines were prepared and investigated at the micro-level.

Analysis of mass loss rate and dimensional shrinkage of the prepared wood-based ceramics showed that as the phenolic resin content increased, the dimensional stability of the samples improved. The mass loss rate was primarily influenced by the heating strategy, phenolic resin concentration, and wood material morphology. The mass loss rate of wood powder ceramics was higher than that of wood fiber ceramics. Wood-based ceramic materials exhibited good flexural performance. Experiments revealed that the choice of heating strategy during high-temperature sintering had a certain impact on the flexural performance of the ceramics. Increasing the phenolic resin concentration enhanced the flexural strength of the wood-based ceramics. Due to their excellent physical properties, wood powder blended more effectively with the glassy carbon generated from phenolic resin, resulting in stronger chemical bonds compared to wood fiber. An increase in phenolic resin concentration significantly improved the compressive strength of the wood-based ceramics. Increasing the phenolic resin concentration from 30 % to 50 % resulted in a compressive strength increase of up to 11 MPa, the structural feasibility of these materials as durable substrates for air filter elements. This research advances the field of sustainable filtration by demonstrating that low-cost wood waste can be converted into high-performance structural filters through precise kinetic control of the carbonization process. Consequently, this work opens a new avenue for applying wood ceramics in high-temperature air purification, addressing the critical need for robust, biodegradable alternatives to synthetic fiber filters.

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