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Effect of temperature on color changes and mechanical properties of poplar/bismuth oxide wood alloy during warm-press forming

Jin Yan¹, Liqiang Zhang^{1*}, Xianjun Li², Qingding Wu¹ and Jianan Liu¹

Abstract

Binderless poplar/bismuth oxide wood alloy is prepared using the warm-press forming technology. The effect of the forming temperatures on color changes and mechanical properties of the poplar/bismuth oxide wood alloy is studied. The results show that the surface color of the specimen gradually darkened as the forming temperature increased. There is the most obvious change from 140 to 160 °C. The CIE lightness color coordinate L^* and chroma coordinate b^* decrease with the increase of the forming temperature, while chroma coordinate a^* decreases initially, but later increases with treatment severity. The static bending strength (MOR), the elastic modulus (MOE) and the surface hardness (HV) increase first and then decrease with the increase of the forming temperature. The X-ray diffraction (XRD) analysis shows that the wood underwent carbonization at 180 °C, resulting in a decrease in the density and mechanical properties of poplar/bismuth oxide wood alloy, and a deepening of the surface color. The Fourier transform infrared spectroscopy (FTIR) analysis reveals that the pyrolysis of cellulose and hemicellulose, as well as the pyrolysis and condensation of lignin led to the color of poplar/bismuth oxide wood alloy deepening. The hydroxyl groups between the cellulose molecular chains are reduced and hydrogen bonds are formed at 140 °C, which improve the mechanical properties of poplar/bismuth oxide wood alloy. However, the massive degradation of hemicellulose weakens binding strength with cellulose and lignin at 160 °C above. It greatly reduces the mechanical properties of specimen.

Keywords: Poplar/bismuth oxide wood alloy, Warm-press forming, Color properties, Mechanical properties

Introduction

Wood is an environmentally friendly and renewable material. The inherent defects of wood, such as cracking and easy discoloration, directly affect its performance and limit the application of wood products [1]. Large amounts of residues are generated during the harvesting, logging, processing and utilization of wood. Therefore, the study of potential treatment methods to increase the added value of wood residues and improve the properties of wood is of great

importance for future applications. Warm-press forming is a new technology based on the theory of metal powder warm-press forming and wood material non-glue hot-press forming theory [2]. The bonding power of the glueless warm pressing technology is mainly due to the thermosetting properties of lignin [3]. This technology can improve the color and mechanical properties of wood products by changing the forming temperature, the forming pressure and the holding time. Previous studies showed that wood with proper heat treatment has higher mechanical properties. Kučerová et al. [4] investigated the effects of heat treatment temperature on the mechanical properties of fir wood. The results showed that the MOR and the MOE slightly increased in the initial stages of thermal

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treatment (100~150 °C). However, a severer treatment resulted in a reduction in their values. Van Dam et al. [3, 5] used coconut husks with high lignin content to produce high strength-high density binderless boards by hot pressing; they showed that the mechanical properties, water absorption and density of these boards were superior to those of particleboard and commercial medium-density fiberboard.

Another of the most significant changes related to thermal modification is the color of the wood. It is important for judging the performance of wood, especially for wood with decorative purposes [6]. Heat treatment technology can be used to deepen the color of the wood and reduce the color difference between the core and the edge of the wood, so that wood can exhibit the color of precious tree species [7, 8]. The usual way to obtain the desired color of wood is to use chemical dyes to react with the wood. However, the dyes used have some problems such as toxic nature, poor light resistance, poor biodegradability and low color fastness. The dyeing process requires a lot of water, and various penetrants and dyeing auxiliaries need to be added. Such a process will not only lead to waste of resources, but also cause water pollution. Therefore, it is difficult to promote the use on a large scale [9]. Yellow pigments based on bismuth compounds have some attractive color properties and high solar reflectance. It does not contain Pb, Cr, Cd, etc., in traditional yellow pigments, which is environmentally friendly [10]. Šulcová et al. [11] studied new environmentally friendly inorganic pigments based on Bi₂O₃ doped by metal ions and gave a direction for coloring ceramic glazes. In addition, bismuth oxide has useful chemical properties, such as high photocatalytic activity and stability. The photocatalytic activity of bismuth oxide comes from its ability to oxidize organic pollution and produce highly active substances, which are mostly used in the photocatalytic degradation of solar cells, optical coatings, optoelectronics and organic dyes [12].

In this paper, a mixture of poplar wood powder and bismuth oxide powder is used for warm-press forming. The effect of the forming temperature on the color properties and mechanical properties of the poplar/bismuth oxide wood alloy is studied. The chemical composition of the poplar/bismuth oxide wood alloy before and after warm-press forming is analyzed. The study method can provide a new, simple and environmentally friendly direction for wood coloring. These results can provide a reference for the subsequent research on the change law of the wood alloy color and mechanical properties during warm-press forming.

Material and method

Material

The material used in this study is the powder of naturally air-dried fast-growing poplar wood processing residues (Linyi City, Shandong Province, China). The particle size of poplar wood powder is less than 180 µm. The initial moisture content of the poplar wood powder is 8.7%. Commercially available nano-bismuth oxide with an average particle size ≤ 300 nm is used as an experimental auxiliary material.

Poplar/bismuth oxide wood alloy preparation

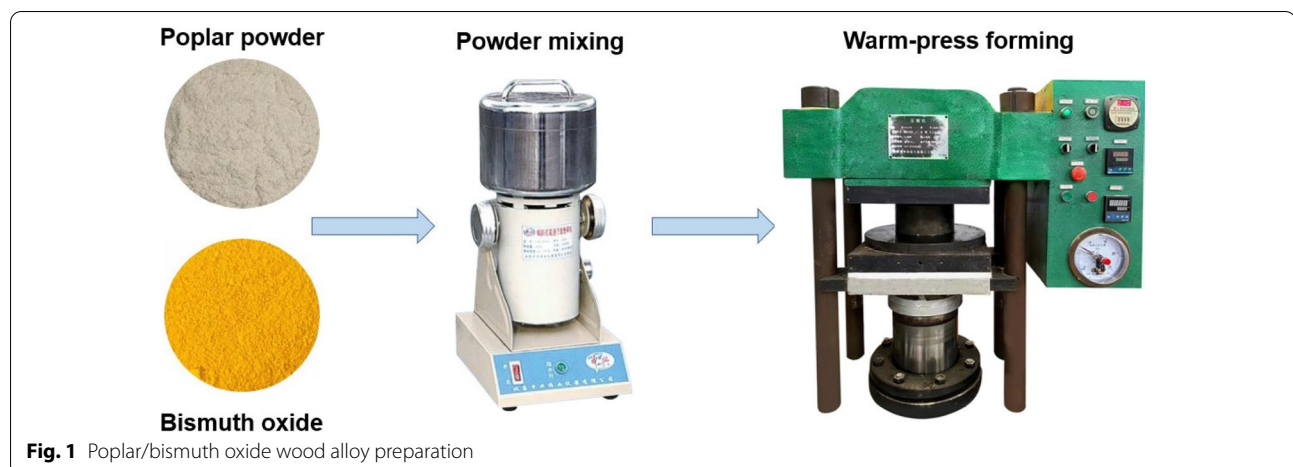
Poplar wood powder and bismuth oxide powder are put into the FW-400A tilt type high speed universal crusher (Zhongxing Weiye Instrument Co., Ltd, Beijing, China) according to the mass ratio of 9 to 1. The mixture is mechanically stirred at 26,000 rpm for 15 min to obtain the homogeneously mixed powder mixture with a total mass of 80 g. The mixed powder is compressed and formed in the HX-100 compression molding machine (Institute of Thermal Processing Engineering, Huazhong University of Science and Technology, Wuhan, China). The flowchart for the specific preparation of poplar/bismuth oxide wood alloy is shown in Fig. 1. The forming pressure is set to 80 MPa. The forming temperature is set to 100 °C, 120 °C, 140 °C, 160 °C, 180 °C and 200 °C, respectively. After the mold reaches the specified temperature, the compact is kept at this temperature and pressure for 30 min. The compact is taken out when the mold cools to room temperature. In addition, the pressure slowly disappears during the cooling process to reduce the rebound effect. The dimension (length × width × thickness) of the final specimen is 128 mm × 35 mm × 10 mm.

Density test

The mass of each specimen is determined with an electronic weighing scale (Guangzheng Medical Instrument Co., Ltd, Shanghai, China) and the volume is determined with electronic digital caliper (Hangzhou Delixi Group Co., Ltd). The length, width and thickness of the specimen are accurately measured three times at different positions and averaged. The specimen density is computed as the ratio of mass to volume (g/cm³).

Color properties test

The color parameters L^* , a^* and b^* of the specimens are analyzed by the CIE-L*a*b* system, in which L^* denotes the lightness axis [pure black (0) to pure white (100)], a^* denotes the green (− a) to red (+ a) axis, and b^* denotes the blue (− b) to yellow (+ b) axis, respectively. The



high-definition pictures of the specimens are obtained through the Ricoh 7502 high-definition printer scanner. 10 points are randomly selected as color measurement points in the specimen without defects and with uniform color. The values L^* , a^* and b^* are measured through Photoshop CC2019 and then the average values are calculated. The overall color difference (ΔE^*) is used to describe the overall color change of the specimen and calculated by using Eq. 1. The specimen with the forming temperature of 100 °C is used as the control group.

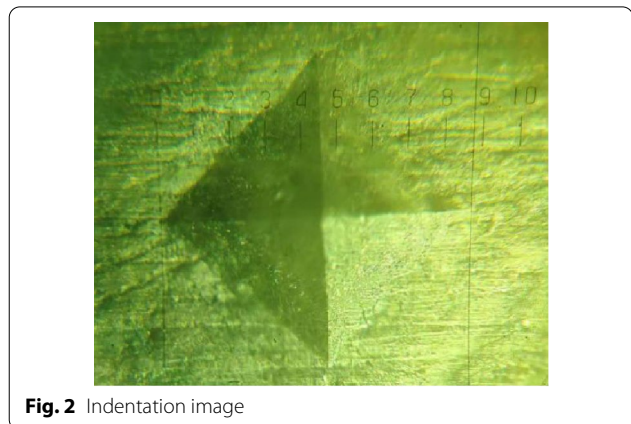
$$\Delta E^* = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2}, \quad (1)$$

where L_0^* , a_0^* and b_0^* are the color parameters of the control group, L^* , a^* and b^* are the color parameters of the experimental group.

Mechanical properties test

The values of MOR and the MOE are measured by using a WDW-10 microcomputer-controlled electronic universal testing machine (New Shijin Testing Machine Co., Ltd, Jinan, China). The test method adopts three-point bending with a span of 80 mm. Two parallel support rollers with the diameter of 15 mm and a loading roller with the diameter of 30 mm are used. The loading roller is loaded at a constant speed of 12 mm/min throughout the experiment.

The HV is measured by using an HV-5 small load Vickers hardness tester (Dechuan Testing Instrument Co., Ltd, Laizhou, China). The experimental force is set to 0.5 kgf and the holding time is set to 12 s. After unloading, the indentation on the surface of the specimen was observed and measured through a 40X objective lens. Figure 2 shows the image of the indentation observed in



the eyepiece. The surface hardness is calculated by using Eq. 2.

$$HV = \frac{1.8544P}{\left(\frac{d_1+d_2}{2C}\right)^2}, \quad (2)$$

where P is the experimental force (kgf), d_1 , d_2 are the diagonal length value of indentation image (mm), C is the magnification of objective lens, respectively.

Chemical composition test

The powder on the surface of the specimen is scraped off and fully dried for chemical composition analysis. The mixed powder before warm-press forming is selected as the control group, and the specimens with the forming temperatures of 100 °C, 140 °C, and 180 °C are selected as the experimental group. The D8 Advance X-ray diffractometer (Bruker Co., Ltd, German) is used to obtain the phases in the specimens. The functional groups in specimens are identified using

Bruker VERTEX 70 & ALPHA infrared spectrometer (Shimadzu Co., Ltd. Japan).

Results and discussion

Density change after warm-press forming

Figure 3 shows the effect of the forming temperature on the density of poplar/bismuth oxide wood alloy. It can be seen that the density of the specimens first increases slightly and then decreases. Compression leads to a decrease in the void volume of the wood and an increase in the amount of cell wall per unit volume, which leads to an increase in density [13]. There is a rapid decrease in density of the specimens at 160 °C above. This is due to the severe loss of moisture and loss of quality caused by high temperature. Boonstra et al. [14] believed that the main reason for the decrease in wood density after heat treatment is the degradation of hemicellulose and the evaporation of extracts.

Color changes after warm-press forming

The surface color of poplar/bismuth oxide wood alloy at different temperatures is shown in Fig. 4. It can be seen that the surface color of the specimens gradually darkens with the increase of the forming temperature. This darkening of the wood surface can be explained by the formation of hemicellulose degradation products, changes in extracts, and the formation of oxidation products such as quinones [2]. It can be seen in Fig. 4 that the surface color of the specimens changes most obviously in the temperature range of 140 ~ 160 °C.

Figure 5 shows the effect of the forming temperature on the color properties of poplar/bismuth oxide wood alloy. It can be seen that the lightness value L^* and the

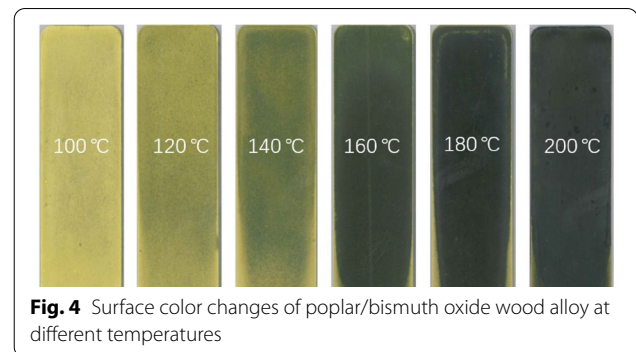


Fig. 4 Surface color changes of poplar/bismuth oxide wood alloy at different temperatures

chroma coordinate b^* are gradually decreasing with the increase of the forming temperature. When the forming temperature increases from 100 to 160 °C, the values of L^* and b^* decrease rapidly to 30 and 7, respectively. When the forming temperature increases from 160 to 200 °C, the values of L^* and b^* decrease slowly to 25 and 1, respectively. The change of the chroma coordinate a^* is relatively smooth. The a^* value reaches a minimum value of -9 at 140 °C and a maximum value of -3 at 200 °C. In general, the forming temperature has a significant influence on the values of L^* and b^* of the surface color and a slight influence on the value a^* .

Figure 6 shows the overall color difference ΔE^* of poplar/bismuth oxide wood alloy at different temperatures. It can be seen that the value ΔE^* increases at a rapid speed before 160 °C and increases at a slow speed after 160 °C. This conclusion is consistent with the color change rule of the specimens in Fig. 4. The maximum change in ΔE^* is 24.32 at approximately 140 to 160 °C. On the contrary, the change of the ΔE^* is the smallest value between 180 and 200 °C, and the change value is only 1.48. The results

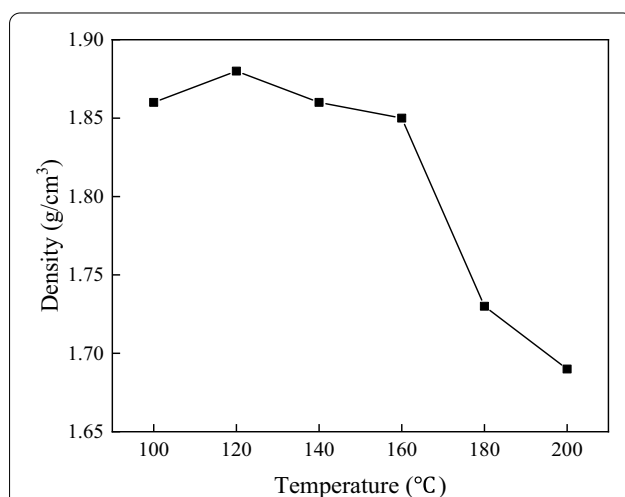


Fig. 3 Density of poplar/bismuth oxide wood alloy at different temperatures

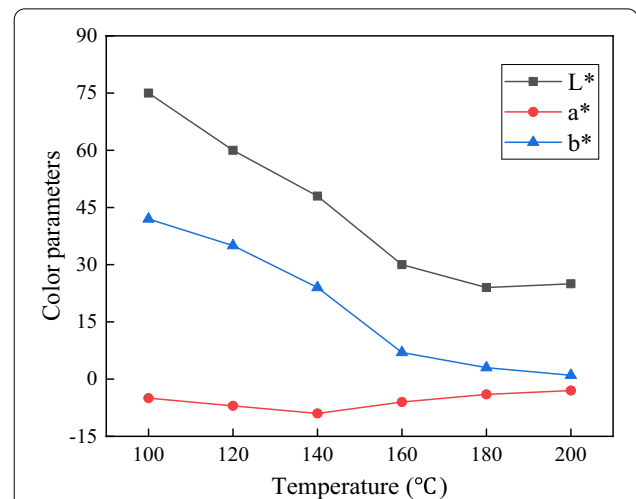
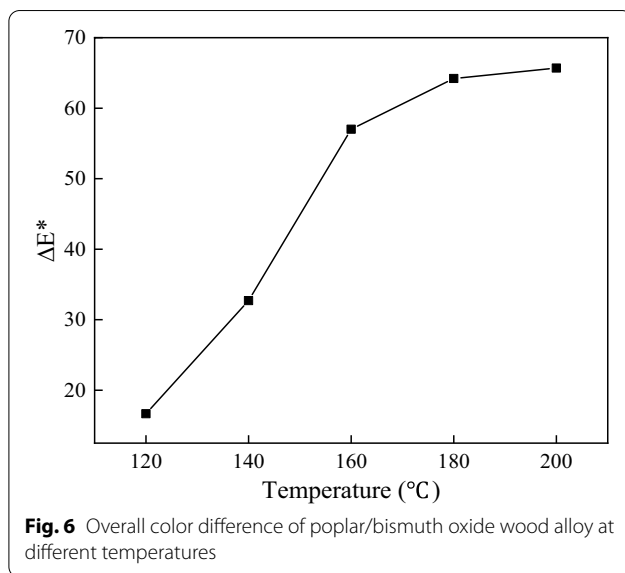


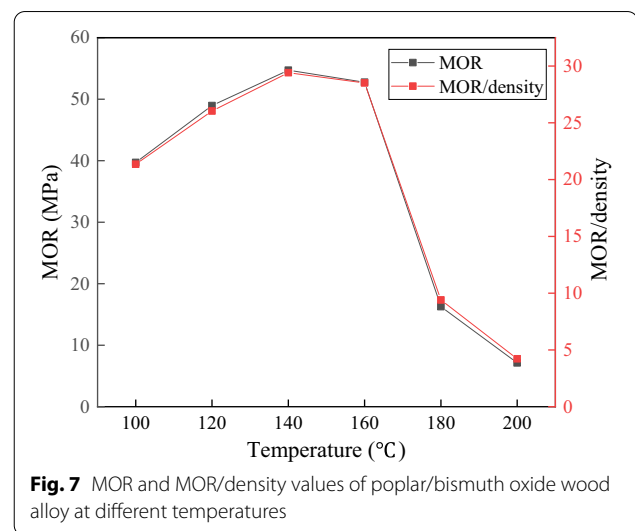
Fig. 5 Color properties of poplar/bismuth oxide wood alloy at different temperatures



show that the forming temperature has a significant influence on the surface color of poplar/bismuth oxide wood alloy during warm-press forming. The ΔE^* value is most sensitive to the forming temperature range of 140 to 160 °C.

Mechanical properties change after warm-press forming

The mechanical properties of poplar/bismuth oxide wood alloy after warm pressing are measured and summarized in Table 1. Figure 7 shows the change in MOR and MOR/density of poplar/bismuth oxide wood alloy at different temperatures. The MOR and MOR/density values increase first and then decrease with the increase of the forming temperature, and reach the maximum value at 140 °C. The MOR and MOR/density values increase by 37.74% and 37.75%, respectively, compared to 100 °C. This is because a proper increase of temperature can increase the plasticity of the wood, making the specimens more compact and increasing its strength. The MOR and MOR/density values of the specimens decrease rapidly when the forming temperature exceeds 160 °C. The increase in the forming temperature intensifies the

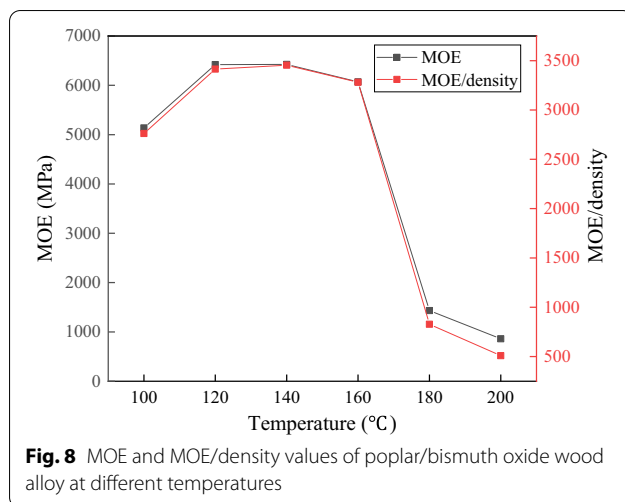


massive degradation of hemicellulose, which leads to the loss of hemicellulose binding in the cell wall and weakens the binding strength with cellulose and lignin. In addition, the rapid degradation of hemicellulose produces a large amount of acetic acid, which catalyzes the degradation reaction of cellulose. At this stage, the main chemical composition of the wood has undergone significant changes, which results in a dramatic change in the MOR of poplar/bismuth oxide wood alloy [15, 16].

Figure 8 shows the effect of the forming temperature on the MOE and MOE/density of poplar/bismuth oxide wood alloy. The MOE and MOE/density values of the specimens initially increase gradually with the increase of the forming temperature and reach the maximum value at 140 °C. This may be due to the decrease in water content [15, 17]. However, when the forming temperature exceeds 160 °C, the MOE and MOE/density values decrease with a rapid velocity. Compared with the forming temperature of 160 °C, the MOE value decreases by 76.39% and the MOR/density value decreases by 74.75% at 180 °C. This is because the degradation of the cell wall has a greater impact on the MOE than the decrease in the moisture content. The decrease in MOE caused by cell

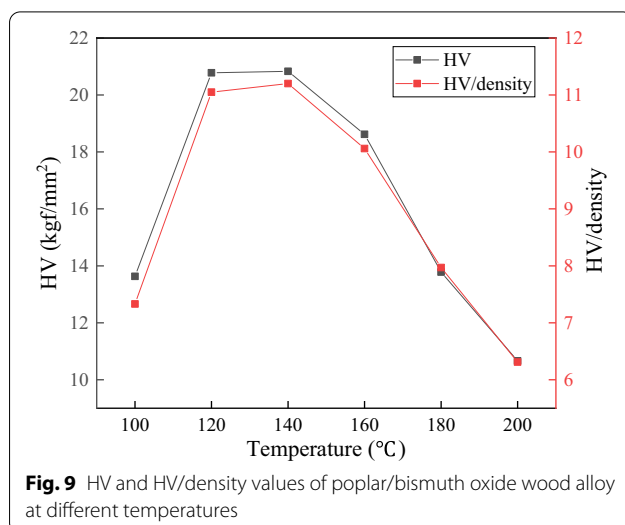
Table 1 Specific data for mechanical properties and mechanical properties/density

Temperature (°C)	Density (g/cm ³)	MOR (MPa)	MOR/ density	MOE (MPa)	MOE/ density	HV (Kgf/mm ²)	HV/ density
100	1.86	39.72	21.35	5136	2761.29	13.63	7.33
120	1.88	48.95	26.04	6420	3414.89	20.78	11.05
140	1.86	54.71	29.41	6424	3453.76	20.83	11.20
160	1.85	52.75	28.51	6069	3280.54	18.62	10.06
180	1.73	16.27	9.40	1433	828.32	13.78	7.97
200	1.69	7.13	4.22	861	509.47	10.66	6.31



wall degradation cannot compensate for the increase in MOE caused by the decrease in water content [17].

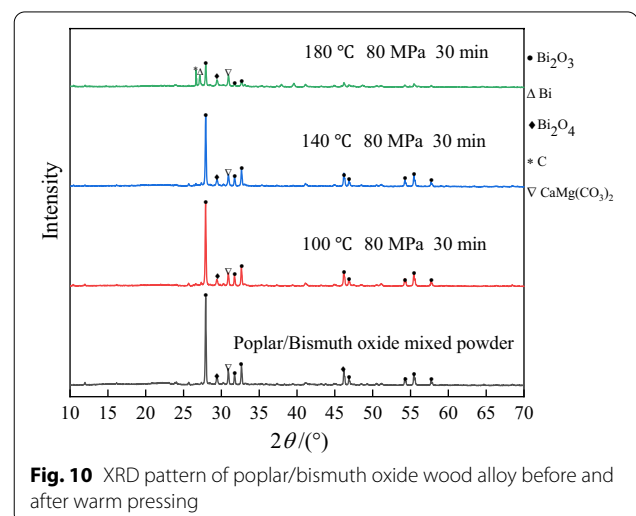
As shown in Fig. 9, the HV and HV/density values of poplar/bismuth oxide wood alloy initially increases and then decreases with the increase of the forming temperature. This is in accordance with the conclusion of Pelit and Yorulmaz [18]. And the HV and HV/density values reach the maximum at 140 °C, which are 52.82% and 52.80% higher than those at 100 °C, respectively. This may be due to the increase in cellulose crystallinity [17]. The hemicellulose in the wood cell wall is the first to undergo partial structural reorganization or degradation due to its poor thermal stability. The hydroxyl groups in the molecular chains are shed. The loss of water in the amorphous region of cellulose and the close proximity of neighboring cellulose surfaces result in a tighter arrangement of cellulose molecular chains. The hydroxyl groups



between cellulose molecular chains undergo a "bridging" reaction to form hydrogen bonds (O–H...O), which leads to an increase in the crystallinity of cellulose molecules [19, 20]. However, the HV and HV/density values of the specimens gradually decrease with further increase in the forming temperature, which could be attributed to the density loss due to excessive temperature and thermal decomposition of the composites, such as fracture and fragmentation of the wood cell wall [18, 21].

XRD analysis

Figure 10 displays the powder XRD diffractograms of poplar/bismuth oxide wood alloy before and after warm pressing. It can be seen that the mixed powder before warm-press forming, as well as the specimens at 100 °C and 140 °C all show a sharp and strong Bi_2O_3 diffraction peak at a 2θ value of 27.93°. However, when the forming temperature is 180 °C, the Bi_2O_3 diffraction peak at 27.93° decreases significantly, and the Bi_2O_3 diffraction peaks at 46.19°, 46.84°, 54.25°, 55.44° and 57.71° basically disappear. Moreover, the diffraction peak of C appears at 26.65° and the diffraction peak of Bi appears at 27.14°. It shows that that the specimens are carbonized at a forming temperature of 180 °C and that Bi is formed by the reaction of C with Bi_2O_3 . The carbonization of the poplar/bismuth oxide wood alloy leads to a decrease in the density and mechanical properties of specimens, and a deepening of the surface color. And the formation of Bi may be the reason for the rise of the chroma coordinate a^* . The diffraction peak with 2θ value of 29.43° in the XRD pattern is the diffraction peak of Bi_2O_4 . And this diffraction peak remains basically constant with the increase of the forming temperature. The diffraction peak at 30.9° is probably from $\text{CaMg}(\text{CO}_3)_2$ in poplar ash. It's peak remains essentially stable in this temperature



range, indicating that the decomposition temperature of $\text{CaMg}(\text{CO}_3)_2$ is higher than 180 °C.

FTIR analysis

The FTIR spectroscopy is employed to investigate the chemical bonding of poplar/bismuth oxide wood alloy in the range of 400~4000 cm^{-1} . The FTIR spectra of poplar/bismuth oxide wood alloy before and after warm pressing are shown in Fig. 11. The characteristic bands in FTIR spectra of poplar/bismuth oxide wood alloy are summarized in Table 2. The peak at 464 cm^{-1} is attributed to Bi–O stretching vibration. This phenomenon is consistent with the data reported by Irmawati et al. [22]. The absorption peak at 1456 cm^{-1} is attributed to methyl C–H bending vibration. And the absorption peak at 1427 cm^{-1} corresponds to benzene ring skeleton structure combined with C–H bond vibration. The apparent

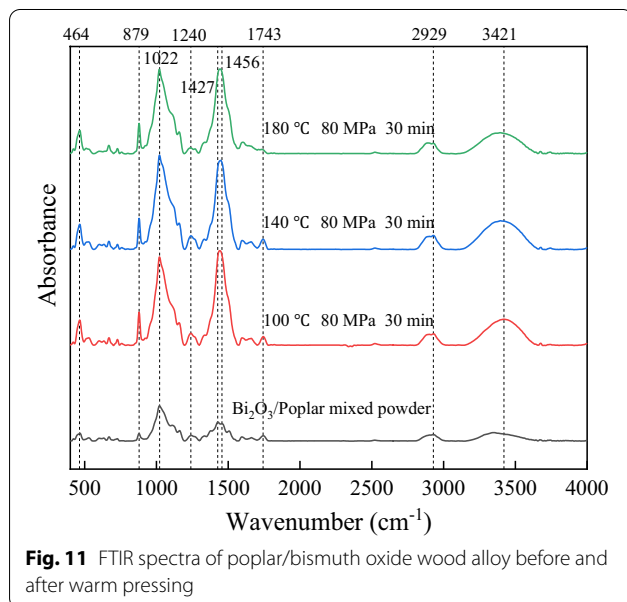


Fig. 11 FTIR spectra of poplar/bismuth oxide wood alloy before and after warm pressing

change from bimodal to shoulder peaks indicates the softening and plasticization of lignin. The side chain break of benzene ring makes the benzene ring skeleton relatively increased [23].

Hemicellulose-related bands in the spectra are observed at approximately 1743 cm^{-1} and 1240 cm^{-1} . The peak at 1743 cm^{-1} represents the linkage of the acetyl group with the ester group or carboxyl group. There is a significant decrease as the forming temperature increases, indicating the degradation of hemicellulose. The acetyl group on the hemicellulose is cleaved to produce acetic acid, which reduces the number of carbonyl C=O bands. In addition, acetic acid can further catalyze the pyrolysis of polysaccharides and the condensation and degradation of lignin structures, contributing to wood discoloration. Phenolic compounds generated by the pyrolysis of hemicellulose with chromogenic groups can also cause changes in wood color. The absorption peak near the C–O–C stretching vibration (1240 cm^{-1}) also decreases, which further confirms that the hemicellulose is substantially degraded during warm-press forming [15, 24, 25]. The massive degradation of hemicellulose weakens the binding strength with cellulose and lignin. It reduces the mechanical properties of specimens [15].

The absorption peaks of C–H stretching vibration in the methyl and methylene groups is found in 2929 cm^{-1} . The functional groups of this absorption peaks are abundant in cellulose [24]. The intensity of the characteristic peak at 2929 cm^{-1} is less affected by the temperature change, indicating that the cellulose is more stable during the warm pressing.

The main chemical components of wood (cellulose, hemicelluloses, lignin) contain large amounts of free hydroxyl groups. The hydroxyl groups may form intramolecular hydrogen bond and intermolecular hydrogen bond [19, 26]. The intramolecular hydrogen bonding does not change the position and shape of the spectral band, but only increases the intensity of the absorption

Table 2 The characteristic bands in FTIR spectra of poplar/bismuth oxide composites

Wavenumber (cm^{-1})	Assignments
464	Bi–O stretching vibration
879	Cellulose β -glycosidic bond
1022	C–O stretching vibration of primary alcohols, saturated fatty ethers
1240	C–O–C stretching vibration
1427	Benzene ring skeleton structure combined with C–H bond vibration
1456	Methyl C–H bending vibration
1743	C–O stretching vibration of hemicellulose
2929	C–H stretching vibration in the methyl and methylene groups
3421	Hydroxy O–H stretching vibration

peak. The absorbance at 3421 cm^{-1} arising from the hydroxyl groups. The peak at about 3421 cm^{-1} in the spectra $100\text{ }^{\circ}\text{C}$ to $140\text{ }^{\circ}\text{C}$ is strengthened gradually. This result is the hydroxyl groups form intramolecular hydrogen bonds. The formation of hydrogen bonds leads to increased crystallinity, which facilitates the improvement of the mechanical properties of the poplar/bismuth oxide wood alloy. As shown in Fig. 10, when the temperature is $180\text{ }^{\circ}\text{C}$, the intensity of hydroxyl absorption at 3421 cm^{-1} decreases. This may be due to the fact that the high forming temperature leads to a reaction of the free hydroxyl groups between the cellulose molecules in the wood, removing a water molecule and forming an ether bond, so the number of free hydroxyl groups is greatly reduced [19]. The ether bond structure formed in cellulose reduces the crystallinity of cellulose, which reduces the mechanical properties of the poplar/bismuth oxide wood alloy [15].

Conclusions

The forming temperature plays an important role in the darkening of poplar/bismuth oxide wood alloy during the warm-press forming. The carbonization of the wood deepens the surface color of poplar/bismuth oxide wood alloy. The pyrolysis of cellulose and hemicellulose, as well as the pyrolysis and condensation of lignin are responsible for darker color. The lightness L^* and the chroma coordinate b^* decrease with the increase of the forming temperature, while chroma coordinate a^* decreases initially, but later increases with treatment severity. The effect of the forming temperature on the L^* and b^* is greater than the effect on the a^* . The overall color difference ΔE^* increases with the increase of the forming temperature. The color parameters are most sensitive to temperature in the range of $140\text{--}160\text{ }^{\circ}\text{C}$.

Appropriate increase of the forming temperature can make a positive contribution to improve the mechanical properties of poplar/bismuth oxide wood alloy. The hydroxyl groups between the cellulose molecular chains form hydrogen bonds at $140\text{ }^{\circ}\text{C}$, which improves the MOR, MOE and HV values of poplar/bismuth oxide wood alloy. However, the massive degradation of hemicellulose leads to weakened binding strength with cellulose and lignin at $160\text{ }^{\circ}\text{C}$ above. The change trend is opposite to that at $140\text{ }^{\circ}\text{C}$ when the forming temperature is above $160\text{ }^{\circ}\text{C}$. And the carbonization of wood at $180\text{ }^{\circ}\text{C}$ also results in a decrease in the density and mechanical properties of specimens.

Abbreviations

MOR: Static bending strength; MOE: Elastic modulus; HV: Surface hardness; XRD: X-ray diffraction; FTIR: Fourier transform infrared spectroscopy; L^* :

Lightness axis [pure black (0) to pure white (100)]; a^* : Green ($-a$) to red ($+a$) axis; b^* : Blue ($-b$) to yellow ($+b$) axis; ΔE^* : Overall color difference.

Author contributions

YJ contributed to the analysis of the results and writing of this manuscript. ZLQ contributed to the review of this manuscript and supervised the entire work. All authors have participated sufficiently in designing the experiments and discussing the results. All authors read and approved the final manuscript.

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Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests

The authors declare that they have no competing interests.

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