Low-formaldehyde emission composite particleboard manufactured from waste chestnut bur

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Abstract
Chestnut bur is an agro-waste material generated in the chestnut production. It is a tannin-rich lignocellulosic material which might be a promising raw material for low-formaldehyde composite particleboard production when using urea–formaldehyde (UF) as bonding adhesive. In this study, the characteristics of chestnut bur were analyzed to assess its application value for composite panel. Five-type particleboards were manufactured from the mixture of chestnut bur/poplar particles with the weight ratios of 0/100, 25/75, 50/50, 75/25 and 100/0, UF resin was used as bonding adhesive. The effects of the addition amount of chestnut bur on the physical, mechanical properties and formaldehyde emission of particleboard were studied. The results showed as follows: (1) chestnut bur showed low cellulose, hemicelluloses and lignin contents, but high extractives and ash contents compared with poplar wood. Chestnut bur and poplar wood had the similar fiber morphology. (2) Composite board with relatively good performances could be manufactured with mix particles of chestnut bur and poplar wood. With the increasing of chestnut bur content, the mechanical properties of the composite board decreased, whereas the dimensional stability increased. (3) The total phenol content in the chestnut bur was as high as 13.79%. The phenolic substance in the chestnut burs could react with free formaldehyde. Hence, the free formaldehyde emission of particleboard was effectively reduced. In summary, waste chestnut bur is a suitable material which can not only be utilized as the base material, but also a natural free formaldehyde scavenger for composite particleboard production.

Keywords: Chestnut bur, Composite particleboard, Formaldehyde, Polyphenol

Introduction
Chestnut is a kind of nutritious edible nut fond by the people all over the world. It is produced from tree species of Castanea Mill, which is widely distributed in Europe, America and Asia. The Chinese chestnut, mainly Castanea mollissima, has been cultivated in China for more than 2000 years. In recent years, the total chestnut production increases rapidly in China, reaching 2.34 million tons in 2015 [1]. Chestnut bur is the barbed lignocellulosic husk on the outer layer of chestnut. With the increase of chestnut production, the chestnut bur currently amounts to approximately 2 million ton/year. However, chestnut burs are hardly utilized; most of them are abandoned or burned, which leads to a great threat to the local environment of chestnut producing areas.

Chestnut bur is a potential material for manufacturing bio-based composite panel, like particleboard and fiberboard [2]. In recent years, effective utilization of non-wood lignocellulosic materials and agro-wastes has been of great interest owing to a drastic fall in forest resources. Many of these materials such as bagasse, cotton stalk and rice straw have been proved to be suitable substitute materials in board production [3, 4].

In board manufacturing, urea–formaldehyde (UF) resin adhesive is most commonly used because of its excellent adhesion properties and low cost [5]. According to statistics, the amount of UF resin adhesive accounts for 91% of the amount of wood-based panel adhesives [6]. However,
UF-bonded boards have a problem of free formaldehyde release, which poses a serious threat to people's health. Many scholars are devoted to the research of reducing the formaldehyde emission of wood-based panels [7]. However, most of these methods have the problems of high cost or bringing secondary pollution [8].

In addition to containing a high proportion of woody fibers, the Chestnut bur is rich in polyphenols [9], which might react with the free formaldehyde when using UF resin as an adhesive for making board from chestnut bur, reducing the board formaldehyde emission to a low level. It was previously reported that polyphenol-rich lignocellulosic could reduce free formaldehyde emission. The free formaldehyde emission of particleboard prepared by adding 50% waste stone pine cones was 19.8% lower than that of whole wood particleboard [10]. The addition of mimosa barks had a positive effect on reducing the free formaldehyde emission of particleboard [11]. Light medium density fiberboard with low-formaldehyde emission was successfully prepared by adding chestnut shell powder [2]. However, making particleboard using chestnut burs as raw material was not studied until now.

As a polyphenol-rich non-wood lignocellulosic material, chestnut bur might not only be used as raw material to replace wood to make particleboard, but also can function as a formaldehyde scavenger to reduce the formaldehyde emission from the particleboards.

In this paper, the characteristics of chestnut bur material were studied. The particleboards were made of the mixture of chestnut bur particle and poplar particle with various weight ratios. The physical and mechanical properties and formaldehyde emission of particleboard were evaluated.

Materials and methods

Materials

Chestnut burs were collected in Qianxi County, Hebei Province, China. Poplar particles were provided by Hengshun particleboard Co. Ltd., Henan Province, China. Urea–formaldehyde resin adhesive was provided by Senhua Wood Industry Co. Ltd., Hunan Province, China. The solid content of the adhesive was 53%, and the molar ratio of formaldehyde/urea was 1.05. NH₄Cl solution was used as a curing agent.

Analysis of raw materials

Chemical composition of chestnut bur and poplar

The poplar particles and chestnut burs that were ground to pass through 40-mesh screen and retained on 60-mesh screen were used for chemical composition analysis. The alcohol–benzene extractive, hot water extractive, Klason lignin, holocellulose, α-cellulose, and ash content of the samples were analyzed in accordance with the Chinese national standards [12–17] for fibrous raw material. The samples were extracted with a mixture of ethanol and benzene (1:2, v/v) and refluxed at 90 °C for 6 h to analyze alcohol–benzene extractive [12], and the samples were extracted with distilled water at 95–100 °C for 3 h to obtain hot water extractive [13]. The lignin content was detected by Klason method [14], and the holocellulose content was measured by using Wise method (the sample was treated with sodium chlorite to remove the lignin) [15]. The α-cellulose content was determined by extracting the holocellulose with 17.5% NaOH solution [16]. The ash content was obtained by burning the samples in a muffle furnace at 575 °C for 4 h [17]. All the above tests are in duplicate, the error of the two samples was no more than 0.2%.

Polyphenol content of chestnut bur and poplar

The ground particles of the chestnut bur and poplar that passed through a 20-mesh screen and retained on 80-mesh screen were used for the analysis of total phenol content. Standard solution is formulated using gallic acid. According to the standard curve of gallic acid, the regression equation is \( y = 5.34x + 0.0383, \quad R^2 = 0.9999 \).

5 g samples and 125 g of 60% ethanol solution were put into an Erlenmeyer flask, and extracted at 85 °C for 45 min. The above suspension was filtered through a 400-mesh gauze. The filtered solution was centrifuged at 10,000 rpm in a centrifuge for 5 min (VL-165B, Hunan Michael Experimental Apparatus Co. Ltd., Hunan, China). After centrifuging, the solution was diluted and subsequently treated with sodium carbonate and Folin–phenol solution, then measured with a spectrophotometer at 765 nm (722 s, Shanghai INESA Analytical Instrument Co. Ltd., Shanghai, China). Finally, the polyphenol content was calculated [18].

Pyrolysis-gas chromatography–mass spectrometry (Py-GC–MS) analysis

Chestnut burs that were ground to pass through 100-mesh screen were used for Py-GC–MS analysis. Separation of compounds was achieved by putting the sample into a pyrolysis port that was heated to 300 °C at a heating rate of 20 °C/ms, kept for 15 s (CDS5200). The pyrolysis products were analyzed by GC–MS and the temperature of the injection port and the separator was 280 °C. The analysis of chestnut bur was performed by GC (TRACE1310) with Agilent TR-5MS capillary column (0.25 mm × 30 m × 0.25 μm). The temperature was initially kept at 40 °C for 2 min, and then increased to 120 °C at a rate of 5 °C/min. Then the temperature was quickly raised to 230 °C at a heating rate of 10 °C/min and maintained for 10 min. With split mode, split ratio was 1:60 and split flow rate was 50 mL/min. The ion source...
of the mass spectrometer (MS) was EI (ISQ) with 280 °C. The MS was scanned at 70 eV in the 28–500 m/z range. Helium (99.999% purity) is used as the carrier gas with a constant flow rate of 1 mL/min.

Fiber morphology of chestnut bur and poplar
The samples of chestnut bur and poplar were boiled with distilled water in the separate test tubes until the samples were softened. 30% nitric acid and potassium chlorate were added for further softening. After rinsing with distilled water to neutral, the samples were separated into fibers by shaking. Finally, the fiber morphology of the raw material was observed under a microscope (Nikon H550S, Tokyo, Japan).

### Table 1 Distribution of particle size of chestnut bur and poplar based on mesh analysis

| Materials    | Mesh no. |
|--------------|----------|
|              | > 20     | 20–40 | 40–80 | < 80    |
| Chestnut bur | 30       | 50    | 16    | 4       |
| Poplar       | 70.7     | 15.1  | 4.5   | 9.7     |

### Preparation and performance testing of particleboard

**Manufacture of low-formaldehyde chestnut bur/poplar composite particleboard**
Chestnut burs were adjusted to a moisture content of approximately 60% and crushed by a knife-ring flaker (PZ8, Pallmann, Germany). The particles of chestnut bur and poplar that cannot pass through the 10-mesh screen were removed. Then the particles were dried to a moisture content of 5% at 80 °C in a laboratory oven. Table 1 shows the size composition of chestnut bur and poplar particles based on mesh analysis by screening test. Figure 1 shows the morphology of chestnut bur and poplar particles.

The dimensions of the boards were 300 × 300 × 8 mm, and the target board density was 0.7 g/cm³. The weights of particles with the weight ratios of chestnut bur/poplar at 0/100, 25/75, 50/50, 75/25 and 100/0 were measured, respectively. UF resin adhesive (with 1% NH₄Cl solution) was then sprayed to the mixed particles at a resin content of 12% based on the oven-dried weight of the particles. Mat forming was done using a forming box. The mats were pressed at 160 °C for 5 min, with the highest pressing pressure of 3–4 MPa. The thickness of the particleboard was controlled by 8 mm-thick distance bars. Figure 2 shows the preparation route of low-formaldehyde chestnut bur composite particleboard. Four boards of each type were prepared, 2 for physical and mechanical properties test and 2 for free formaldehyde emission test.

**Evaluation of physical and mechanical properties of particleboard**
Prior to the evaluation of the mechanical and physical properties, the boards were conditioned at ambient conditions for about 1 week, reaching a moisture content of 5%-7%. The properties of the particleboards were then
evaluated according to the Chinese national standard GB/T4897-2015 for particleboard [19].

The modulus of rupture (MOR) test and the modulus of elasticity (MOE) test were conducted on four 200 × 50 × 8 mm specimens cut from each board, by a three-point bending test over an effective span of 160 mm at a loading rate of 5 mm/min. Four test specimens of 50 × 50 × 8 mm were prepared from each sample board for the internal bonding (IB) strength test at a loading rate of 5 mm/min (MWW-100, Naier Testing Machine Co. Ltd., Jinan, China). Four test specimens of 50 × 50 × 8 mm were prepared from each sample board for the purpose of thickness swell (TS) and water absorption (WA) test (2 h immersion in 20 °C water).

### Evaluation of free formaldehyde emission from particleboard

The free formaldehyde emission of particleboard is tested according to the Chinese national standard GB/T17657-2013 [20], using desiccator method. First, the particleboards were conditioned at the relative humidity of 65% and temperature of 20 °C for one week. The particleboards were then sawn into test specimens of 150 × 50 mm. Ten specimens were placed in a desiccator (with a container containing 300 ml distilled water for free formaldehyde absorption) and sealed for 24 h at 20 °C. The formaldehyde adsorption solution was subjected to a color reaction, and the absorbance was measured at 412 nm. Finally, the formaldehyde concentration was calculated according to the formaldehyde standard curve. The regression equation of the standard curve of formaldehyde is: $y = 7.6025x + 0.011$, $R^2 = 0.9996$.

### Statistical analysis

The average values with standard deviation of all data were used for analysis using SPSS. Duncan’s mean separation tests were applied. The significant differences were set to $p < 0.05$.

### Results and discussion

#### Characteristic of raw materials

**Chemical composition**

The chemical composition of chestnut bur is shown in Table 2. Alcohol–benzene extractive, hot water extractive and ash content of chestnut bur were much higher than that of poplar wood. The alcohol–benzene extractive and hot water extractive of chestnut bur were 10.9 and 29.3%, respectively, which were 2.4 and 3.8 times of the poplar wood, respectively. The cellulose, hemicelluloses and lignin content of chestnut bur showed low values. The holocellulose (including cellulose and hemicelluloses) and lignin content of chestnut bur showed low values. The holocellulose (including cellulose and hemicelluloses) and lignin content of chestnut bur was only 52.87 and 18.3%, respectively, which was 31.4 and 32.0% lower than that of poplar wood, respectively. Holocellulose is the main component of wood cell wall. The strength of wood is due to the cellulose and in part to the hemicelluloses, and lower holocellulose content might directly affect the cell structure of wood, thereby reducing the mechanical properties of wood [21]. Lignin plays a role in connecting cells and imparting rigidity of the cell.

The total phenol content in the chestnut bur was as high as 13.79% and the total phenol content in poplar was 0.98%. It was reported that natural polyphenols can replace phenol to react with formaldehyde for phenol–formaldehyde adhesive preparation [9]. This suggests that polyphenol in the chestnut bur might react with the free formaldehyde during the manufacture of UF-bonded composite panels to lower the formaldehyde emission.

**Py-GC–MS analysis**

From 10 to 40 min, the total ion chromatography (TIC) characteristic map obtained by pyrolysis GC–MS analysis of the chestnut bur is shown in Fig. 3. Table 3 shows the phenolic components in the chestnut bur. In the ortho and para positions of the phenolic hydroxyl group of these phenolic substances, there are reactive sites to which other chemical groups can be attached.

**Fiber morphology**

The fiber morphology of the material is shown in Fig. 4. The fiber length, diameter and length/diameter ratio are 1.06 mm, 17.51 µm, and 60.54, respectively, which are similar to that of poplar and within the size range of hardwood fiber (Table 4). Fiber size is critical during the preparation of particleboard as it affects the board quality [23, 24]. Chestnut bur is suitable for the manufacture of particleboard.

**Physical and mechanical properties**

**Bending strength**

Figure 5a shows the relationship between the weight ratio of chestnut bur/poplar and the MOR of the particleboard. As the content of chestnut bur increased from 0 to 100%,
the MOR values of the particleboards showed a downward trend. The MOR showed significant differences ($p<0.05$) at different chestnut/poplar contents. When the chestnut contents were 0, 25, 50, 75 and 100%, the MOR were 23.9, 22.8, 14.5, 13.3 and 5.2 MPa, respectively. It was found from these data that the MOR was considerably lower when using pure chestnut bur for making particleboard, however, the MOR increased rapidly when just adding small amount (25%) of poplar particles. The composite particleboard with 75/25 of chestnut/poplar weight ratio had MOR higher than the requirement (11.0 MPa) of GB/T4897-2015 [19] for P2-type particleboard (furniture grade particleboard used under dry condition). The MOR of particleboard (Fig. 5b) showed the same trend as MOR. When the chestnut bur content was 75%, the MOE of particleboard exceeded the minimum requirement (1800 MPa) of GB/T4897-2015 [19] for P2-type particleboard.

The MOR and MOE of particleboards largely depend on the characteristics of the raw materials. In general, the bending strength of bio-based composite boards is highly dependent on the contents of cellulose and hemicelluloses in the material. High-strength composite boards should be made from materials with high holocellulose content. In this study, although the chestnut bur had the similar fiber geometry as that of poplar, it had low hemicellulose but high extractives content which resulted in poor bending strength of the composite panels. The bending strength of chestnut particleboard could be improved by mixing small portion of poplar particles into the chestnut bur particles. Our experiment indicated that only 25% addition of poplar particles showed effective improvement to the MOR and MOE of particle boards.

**Internal bond strength**

The relationship between the weight ratio of chestnut bur/poplar and the IB of the particleboard is shown in Fig. 6. The IB values decreased with increasing of the chestnut bur content. The IB showed significant differences between the composite particleboards with different chestnut bur/poplar ratio. When the chestnut bur contents increased from 0 to 50%, and 100%, the IB value of the particleboard decreased from 1.24 to 0.84 and 0.61 MPa, which reduced 32.3 and 50.8%, respectively. Although the addition of chestnut bur had a negative effect on IB, the composite boards still recorded relatively high IB values, even the lowest IB of the particleboard (with 100% chestnut bur content) far surpassed the requirement of GB/T4897-2015 [19] for P1-type particleboard (general purpose particleboard used under dry condition) (0.28 MPa) and P2-type particleboard (0.40 MPa).

The IB value depends on the bonding strength between particles, which is affected by the characteristics of particles. The extractive content is one of the most important factors. In the previous studies, it was reported that extractives in the raw materials affected the curing of adhesive when manufacturing composite panels, thus resulting in the inferior bonding strength in the board [10]. Stone pine cone contains significant amounts of ethanol/toluene extractives (29.2%). Decreasing of IB value with the increasing of pine cone particle content in the
| Number | Time (min) | Chemical composition          | Molecular formula | Structural formula |
|--------|------------|-------------------------------|-------------------|-------------------|
| 1      | 14.71      | Phenol                        | C₆H₆O             |                   |
| 2      | 17.69      | 2-methyl-phenol               | C₇H₈O             |                   |
| 3      | 18.73      | p-cresol                      | C₇H₈O             |                   |
| 4      | 19.10      | 2-methoxy-phenol              | C₇H₈O₂            |                   |
| 5      | 24.95      | 2-methoxy-5-methyl-phenol     | C₈H₁₀O₂            |                   |
| 6      | 28.60      | 4-ethyl-2-methoxy-phenol      | C₉H₁₂O₂            |                   |
| 7      | 30.42      | 2-methoxy-4-vinyl-phenol      | C₉H₁₀O₂            |                   |
| 8      | 32.46      | 2,6-dimethoxy-phenol          | C₈H₁₀O₃            |                   |
| 9      | 35         | 2-methoxy-4-(1-propenyl)-(z)-phenol | C₁₀H₁₂O₂       |                   |
| 10     | 36.44      | trans-isoeugenol              | C₁₀H₁₂O₂           |                   |
panels could be attributed to higher contents of extractives in the cone [10, 25]. In the chemical composition analysis of this study, the extractive content of chestnut bur was much higher than that of poplar wood, which explained the reduction of IB values when the chestnut bur content increased in the composite boards. In addition, the outer surface of the chestnut bur is covered with a layer of wax-like substance, which might lead to poor wettability of adhesive to the particle surfaces, and further cause inferior particle–particle bonding.

**Dimensional stability**

Figure 7a, b shows the effect of weight ratio of chestnut bur/poplar on the 2hTS and WA values of particleboards. The 2hTS and 2hWA of particleboards decreased with the increasing content of chestnut bur. When the chestnut bur content increased from 0 to 100%, the TS decreased from 13.5 to 6.5%, while the WA decreased from 54.3 to 31.1%, this indicated that the dimension stability of the panels was significantly improved by adding chestnut bur.

### Table 4 Fiber dimensions of chestnut bur and poplar

| Materials     | Fiber length (mm) (um) | Fiber diameter (um) | L/D         |
|---------------|------------------------|---------------------|-------------|
| Chestnut bur  | 1.06 (0.36)            | 17.51 (3.37)        | 60.54 (22.53)|
| Poplar        | 1.00 (0.17)            | 21.15 (3.60)        | 47.28 (9.41)|
| Softwood [22] | 2.25–4.28              | 25.6–56.0           | 55.4–129.4  |
| Hardwood[22]  | 0.47–2.92              | 14.4–30.0           | 20.9–91.6   |

The results are given as averages and standard deviations (in parentheses) from the mean values of 100 randomly chosen fiber samples. L/D, length/diameter ratio of each fiber sample.

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![Fig. 4](image-url)  
*Fig. 4* Fiber morphology of materials. **a** Fiber morphology of poplar. **b** Fiber morphology of chestnut bur

![Fig. 5](image-url)  
*Fig. 5* Bending strength of composite particleboard. **a** The effect of the weight ratio of chestnut bur/poplar on the MOR of particleboard. **b** The effect of the weight ratio of chestnut bur/poplar on the MOE of particleboard. Error bars indicate standard deviations. Different letters in the figure indicate that means are significantly different ($p < 0.05$).
The chestnut bur had high extractive content. The alcohol–benzene extractive was 10.9% which was 2.4 times of the poplar wood. The hydrophobic substance like resin, fat and wax in the alcohol–benzene extractive could contribute to low TS and WA values. The previous research also found the extractive has a positive effect on the water resistance of the board. Buyuksari et al. proved that the TS and WA of composite particleboards significantly decreased by adding waste stone pine cones which contained higher extractives [10]. The waxy layer on the surface of chestnut bur also retarded the liquid water penetration. In addition, the phenolic substances in the chestnut bur may undergo addition reaction with formaldehyde in the UF resin to form hydroxymethyl phenol and further formed stable phenolic resin by condensation reaction, which might be one of the reasons for dimensional stability improvement.

Since no water repellent was added during board manufacturing, only the 100% chestnut bur content particleboard could meet the 2hTS requirement of GB/T4897-2015 [19] for P2-type particleboard (≤ 8%).

**Formaldehyde emission**

Figure 8 shows the relationship between the weight ratio of chestnut bur/poplar and the free formaldehyde emission of the particleboard. With the increase of the content of chestnut bur, the free formaldehyde emission of the particleboard reduced significantly. When the chestnut bur content increased from 0 to 100%, the free formaldehyde emission of the particleboard decreased from 0.794 mg/L to 0.370 mg/L, 53.4% reduction. All types of particleboards showed significant differences \( p < 0.05 \) in free formaldehyde emission value from each other. When the content of chestnut bur attained 75% or more, the free formaldehyde emission of particleboard can conform to the \( F^{***} \) standard of Japanese JIS (0.5 mg/L). Therefore, the particleboard can be upgraded from the original \( F^{**} \) level to \( F^{***} \) by mixing chestnut bur.

During the preparation of phenol–formaldehyde resin, the phenolic hydroxyl group of the phenol is electron-donating under the combined influence of inductive effect and conjugated effect. The electron cloud density of the ortho and para positions of the phenolic hydroxyl...
group is greater than that of the meta position, and the addition reaction of the ortho and para positions is prone to occur. Formaldehyde can react with phenol on these positions of the benzene ring to form hydroxymethyl substitution [26]. The reaction equation for the reaction of phenol with formaldehyde is as follows:

\[
\text{OH} + \text{H}_{2}\text{C} = \text{O} \rightarrow \text{OH} - \text{CH}_{2}\text{OH} + \text{H}_{2}\text{O}
\]

In this study, the chestnut burs contained much higher total phenol content than that of poplar (Table 2), and Fig. 3 and Table 3 also show that there are 10 kinds of phenolic substances in the chestnut bur. The ortho and para positions of the phenolic hydroxyl group of these substances are the reactive sites to which formaldehyde can be attached just like preparation of phenol–formaldehyde resin. During the hot pressing of the composite board, these phenolic substances might react with the free formaldehyde in the UF resin adhesive, which reduced the free formaldehyde of composite board, therefore, the free formaldehyde emission of the composite board decreases with the increase of the amount of chestnut bur particles.

Compared with the composite boards made from other tannin-rich agro-waste materials [2, 10], the board prepared from chestnut burs had higher mechanical properties and more effective in formaldehyde reduction. In this study, by adding 50% chestnut bur content, the composite board recorded: MOR 14.5, MOE 1924.5 and IB 0.84 MPa. The free formaldehyde emission value was 0.516 mg/L, 35.0% lower than that of 100% poplar wood particleboard.

Conclusions

Low-formaldehyde emission composite particleboards were successfully manufactured by using chestnut bur and poplar wood as raw materials. With the increase of the chestnut bur content, the free formaldehyde emission of the composite particleboards reduced significantly, and the dimensional stability (2 h TS and 2 h WA) of the boards improved. However, the MOR, MOE and IB of the composite boards were decreased. When the chestnut bur content in the composite board was not higher than 75%, the mechanical properties of particleboards could meet the requirement of Chinese national standard GB/T18497-2015 [19] for P2-type particleboard. Chestnut bur can be used as an alternative raw material for low-formaldehyde composite particleboards production.

Abbreviations

UF: Urea–formaldehyde; TIC: Total ion chromatography; Py-GC–MS: Pyrolysis–gas chromatography–mass spectrometry; GC: Gas chromatography; MS: Mass spectrometer; MOR: Modulus of rupture; MOE: Modulus of elasticity; IB: Internal bonding; TS: Thickness swell; WA: Water absorption.

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Authors’ contributions

JL: carried out the tests, data analysis, writing the manuscript, and figure preparation. JW: composite board preparation and properties test, and data analysis. JX: designed the research, draft manuscript, prepared figures, and supervision. 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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