Lignum Vitae Wood-Derived Composites for High Lubricating Performance

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Abstract: Natural wood is a clean and environmentally friendly material. However, the wood used for tribological applications has been gradually replaced due to the unsatisfactory lubricating performance. Herein, a novel process for achieving high lubricating performance wood-derived composites has been proposed. The Lignum vitae wood was chosen as a representative, where its lignin is firstly removed and then followed by polydimethylsiloxane (PDMS) vacuum infusion. The experimental results indicated that the unique soft-hard composite structure was formed by the wood fibers and the infused PDMS of the PDMS infused Lignum vitae-derived composites (PDMS-L). The PDMS fitted the rough peak of the copper ball through large deformation and the interlayer PDMS reinforced fibers provided strong support, thus the PDMS-L presented great lubricating performance. The friction coefficient and the wear depth decrease significantly after PDMS infusion. Compared to the original Lignum vitae, the maximum reduction in the friction coefficient and the wear depth of the samples after the PDMS infusion is 85.2% and 79%, respectively. This study shows the potential of wood for tribological applications and provides a new avenue toward lubricating performance improvement for wood-derived composites.

Key words: Lignum vitae wood-derived composites, lubricating performance, vacuum infusion, tribological applications

1 Introduction

Wood is widely accepted as an abundant and sustainable resource with porous structures consisting of hollow wood cells arranged in the natural growth direction [Fratzl and Weinkamer, 2007; Karakoc and Freunde, 2016]. The specific structure gives the wood a special strength-to-weight ratio [Jakob et al., 2022], a high degree of anisotropy [Berglund and Burgert, 2018; Deuerling et al., 2018; Eichhorn et al., 2001], and the porous structure also provides channels for the flow of the resin inside wood [Yin et al., 2016]. Wood has been widely used for the past few centuries, including furniture production, building, fuel and paper production [Yan et al., 2021; Wang et al., 2017]. Additionally, wood has also been used for tribological applications, such as the wooden sleeper and the wooden bearing [Friedrich et al., 2021; Sathre and Gorman, 2005]. However, the natural wood used for tribological applications has been gradually replaced by alloys or polymer matrix composites for many advanced applications due to the unsatisfactory lubricating performance. Unfortunately, polymer matrix composites are not particularly clean materials because polymer composites are usually difficult to degrade and some harmful substances are produced during the production. As a clean and environmental-friendly material, wood with high lubricating performance can be used for wide applications to replace alloy or polymer composite used as friction pairs, which is perfectly suitable for clean materials and environmental protection needs. It is of great significance to find a convenient approach to improve the lubrication performance of wood. However, the study focus on the lubricating performance improvement for the wood to amplify its application range is rarely found.

In order to improve the properties of wood for better applications, wood modification has become an important means to produce high performance wood-derived composites [Bekhta et al., 2018; Priadi and Hiziroglu, 2013]. Chemical modification is one of the most widely used modification methods. Leslaw used polymethylmethacrylate (PMMA) to modify the pine wood surface [Leslaw, 2016]. The results showed that the strength of the pine wood was significantly increased after surface modification. Additionally, the strength and the anisotropy of the pine wood could be controlled by adjusting the amount of the polymer. Bi et al. summarized the representative achievements of wood chemical modification and nanotechnology [Bi et al., 2021]. Various wood modification methods were analyzed and compared. Additionally, other modification methods are also used to better enhance the properties of wood. Song et al. moved the lignin of the wood, followed by hot-pressing to develop a dandified wood and the product presented excellent mechanical properties [Song et al., 2018].

The resin infusion is an excellent technology for modification that has been widely used to enhance the performance of the material [Sharma et al., 2022; Wang et al., 2017]. Due to the channels provided by the specific porous structure, wood presents a special potential to enhance the performance by infusion. Some achievements have already been made. Zhu et al. removed the lignin of wood to expand the channels inside the wood, followed by filling the epoxy resin into the wood to produce a new wood-derived composite [Zhu et al., 2016]. The results indicated that the wood-derived composite was highly transparent and presented better mechanical properties than the original wood. Xia et al. infused the epoxy resin into the Poplar [Xia et al., 2019]. Then the densification of wood-epoxy resin composite was densified by hot-pressing. The products presented unique mechanical properties, which were increased to the values of the alloy range.

Here, we suppose that it is feasible to manufacture a novel wood-derived composite through infusion to improve its lubricating performance. Based on our previous study, the lubricating performance of wood is highly influenced by its anatomical properties, including the anatomical structure and the extractives [Wu et al., 2022]. The Lignum vitae presents good lubricating performance due to its high content of extractives combined with the perfect mechanical properties given by the densest anatomical structure. In this case, the Lignum vitae has been chosen for further modification to produce the Lignum vitae-derived composites. Additionally, the PDMS, which has been widely used to produce medical equipment and artificial joints, is a material with good biocompatibility and is almost harmless to humans [Ariati et al., 2021]. In the present study, polydimethylsiloxane (PDMS) has been used for infusion because the main component of the extractives inside the Lignum vitae is silico-oxide compounds. The PDMS has been infused and solidified inside the Lignum vitae after removing the lignin. The microstructure, elements and the compressive strength of the Lignum vitae, the lignin removed Lignum vitae (L-removal) and the PDMS infused Lignum vitae-derived composites (PDMS-L) have been detailedly observed and tested to prove the successful infusion of PDMS, and to better understand the microstructure and the mechanical properties of the various kinds of samples. After that, the lubricating performance of the three kinds of samples has been investigated through a series of tribological experiments. The present study aims to provide a convenient approach to improve the lubrication performance of wood, and amplify its application range further.

2 Material and methods

2.1 Materials and chemicals

Lignum vitae was provided by a shipbuilding company in China. All the Lignum vitae were cut into wood blocks with a size of 20 mm × 10 mm × 15 mm (L×R×T) and polished before future processes. The chemicals used to remove the lignin in the Lignum vitae were sodium hydroxide (Analytical reagent) and sodium sulfite (Analytical reagent). The Polydimethylsiloxane (PDMS) used for infiltration was Sylgrad 184 (Dow Corning, USA). The solvent used for the solution was deionized water. The absolute ethyl alcohol was bought from Aladdin (Analytical reagent).

2.2 Lignin removal experiment

The solution contained NaOH (5 mol L^{-1}) and Na_2SO_3 (0.4 mol L^{-1}) was used for removing the lignin of the Lignum vitae [Song et al., 2018]. The Lignum vitae blocks were immersed in the boiling solution for 8 h to remove the lignin. After that, the Lignum vitae blocks were rinsed with deionized water for several times to remove the residual solution.

2.3 Manufacturing of Lignum Vitae-derived composites

PDMS solution was prepared by mixing the two components (Base component and Hardener) at a mass ratio of 10 to 1, followed by dissolving in the tetrahydrofuran (THF) at a mass ratio of 10 to 1 (PDMS component to THF). After the lignin was removed, the Lignum vitae blocks were placed in a special design container that fits their sizes and immersed into the solution, the solution was vacuumed and kept in the vacuum for a while to remove the gas in the Lignum vitae. After that, the vacuum was released, the PDMS, which was pushed by the atmospheric pressure, would fill into the Lignum vitae [Xia et al., 2019]. The process was repeated at least three times at a low temperature within two hours to prevent obvious changes in the viscosity of the PDMS solution. At last, the PDMS infusion Lignum vitae was placed in the oven at 60 ℃ for 8 h for solidification.

2.4 Microstructural and elements observation

The microstructure of the original Lignum vitae, the lignin removed Lignum vitae and the PDMS infused Lignum vitae were characterized by a scanning electron microscope (SEM) (Tescan, Czech). Energy disperses spectroscopy (EDS) (Oxford, UK) was used for the elemental analysis of samples' surfaces.

2.5 Compression testing

The wood blocks with a dimension of 20 mm × 10 mm × 15 mm (L×R×T) were used for compressive properties tests, and the blocks were compressed along the growth direction at a speed of 2 mm min^{-1}.

2.6 Tribological experiments

The tribological experiments were carried out with a reciprocating ball-on-disk Rtec tribometer (USA). The selection of experimental parameters was based on relevant studies [Liu et al., 2022; Mckenzie and Karpovich, 1968; Yin et al., 2016]. The dimension of the tested samples is 20 mm × 10 mm × 15 mm (L×R×T). The copper balls with a diameter of 10 mm were used as the contact-body and contacted with the samples at the L×T surface. The tribological experiments were performed by applying constant loads on the copper balls and moving the Lignum vitae blocks along the growth direction of the Lignum vitae at a constant speed. The experimental form was reciprocating friction while the applied loads were 5 N, 10 N, 15 N and 20 N, and the sliding distance and the frequency were 10 mm and 3 Hz, receptively. The experiments were carried out under dry frictional conditions for 30 min at room temperature. After the tribological experiments, the wear morphologies were observed by using the SEM and the wear track depth of the Lignum vitae blocks was measured by using a confocal laser scanning microscope (CLSM) (Keyence, Japan).

The friction coefficient was automatically collected by the Rtec tribometer during the tribological experiments. Due to the run-in period in the beginning, the collected data at the initial five minutes was abandoned, the average friction coefficient was calculated based on the collection date from 5 to 30 minutes of the experiments. After the wear track of the samples was observed by the CLSM, the average level changes were automatically calculated by the CLSM, and the wear depth was directly measured based on the average level changes line also by the CLSM.

3 Results and discussion

3.1 Characterization of samples

The microstructure, elements and compressive strength of the Lignum vitae, the lignin removed Lignum vitae (L-removal) and the PDMS infused Lignum vitae-derived composites are shown in Fig.1. The difference in the microstructure and the element between the surface layer and the interlayer of the PDMS-L is present in Fig.2.

Fig. 1 highlights the structural and element characterization of the three kinds of samples. The selected Lignum vitae presents typical hard wood's porous structures. Many straight channels provided by various kinds of xylem cells including wood cells and vessels exist on the cross section of the Lignum vitae. The tangential section SEM image of the Lignum vitae also shows the small channels on the surface, which provides the possibility for the liquid to flow perpendicular to the growth direction. The channels provided by the wood fibers are surrounded by the cell wall, which consists of cellulose-reinforced hemicelluloses and lignin. After the lignin is removed, the structure with the various channels is still maintained, allowing the vacuum infusion of PDMS (Fig. 1, F and G). However, the linkages of the cellulose fibrils are cut off due to the removal of the lignin matrix, resulting in many gaps, which decreases the difficulty for the subsequent infusion. After PDMS is infused into the L-removal with vacuum assistance, the sample is cut and the middle central part has been observed. SEM images (Fig. 1, J and K) confirm the PDMS is indeed infused into the middle central part of the PDMS-L. On the cross section, all the channels are filled and bonded with the PDMS. The more obvious typical structure can be found on the tangential section, the vessels are clearly identified and are fully filled with PDMS. The wood cells are also easily identified and have been connected by the infused PDMS. Note that the original structure of Lignum vitae remains intact after PDMS infusion. The separated cellulose fibrils have been connected again by the infused PDMS with intermolecular forces. No obvious boundary is spotted between the PDMS and the cell walls of the various xylem cells.

Energy spectrum analysis has been done to examine the elements on the samples' surface. The main elements of the Lignum vitae and the L-removal are carbon and oxygen with a small amount of silicon (Fig. 1, D and H). Silicon is mainly derived from the silico-oxide compounds contained in the Lignum vitae. After PDMS infusion, the EDS results indicate that the main elements on the surface are carbon, oxygen and silicon (Fig. 1L). Silicon is used as the characteristic element and has been given special attention because it is one of the main elements contained in PDMS. According to the EDS results, silicon is dispersed uniformly on the surface of the Lignum vitae and the L-removal while not the PDMS-L, whose silicon seems more concentrated at the vessels due to the large channels (Fig. 1, D, H and L). Note that silicon weight percentage increases from 1.16% to 7.8% after PDMS infusion, which is a whopping 574.1% increase (Fig. 1M), confirming the existence of the PDMS. The high weight percentage of silicon combined with the structural characterization confirms that the PDMS has been successfully infused into the wood.

The compressive strength of the three kinds of samples has been tested. The compressive direction is along the growth direction of the wood. Fig. 1N indicates that the compressive strength decreased rapidly from 80 MPa to 25 MPa after lignin removal. It is because the wood can be regarded as a cellulose fibrils-reinforced material and the lignin acts as a basic to connect the cellulose fibrils by chemical bonds and hydrogen bond with strong bonds energy. After removing the lignin, the cellulose fibrils lack connection, causing a rapid decrease in compressive strength. After the PDMS infusion, the material's compressive strength increases slightly from 25 MPa to 34 MPa. It is mainly because the PDMS replaced the lignin to connect the cellulose fibrils again. However, the PDMS, whose mechanical properties are inferior, connects the fibers with low intermolecular forces, causing the compressive strength is not obviously increased.

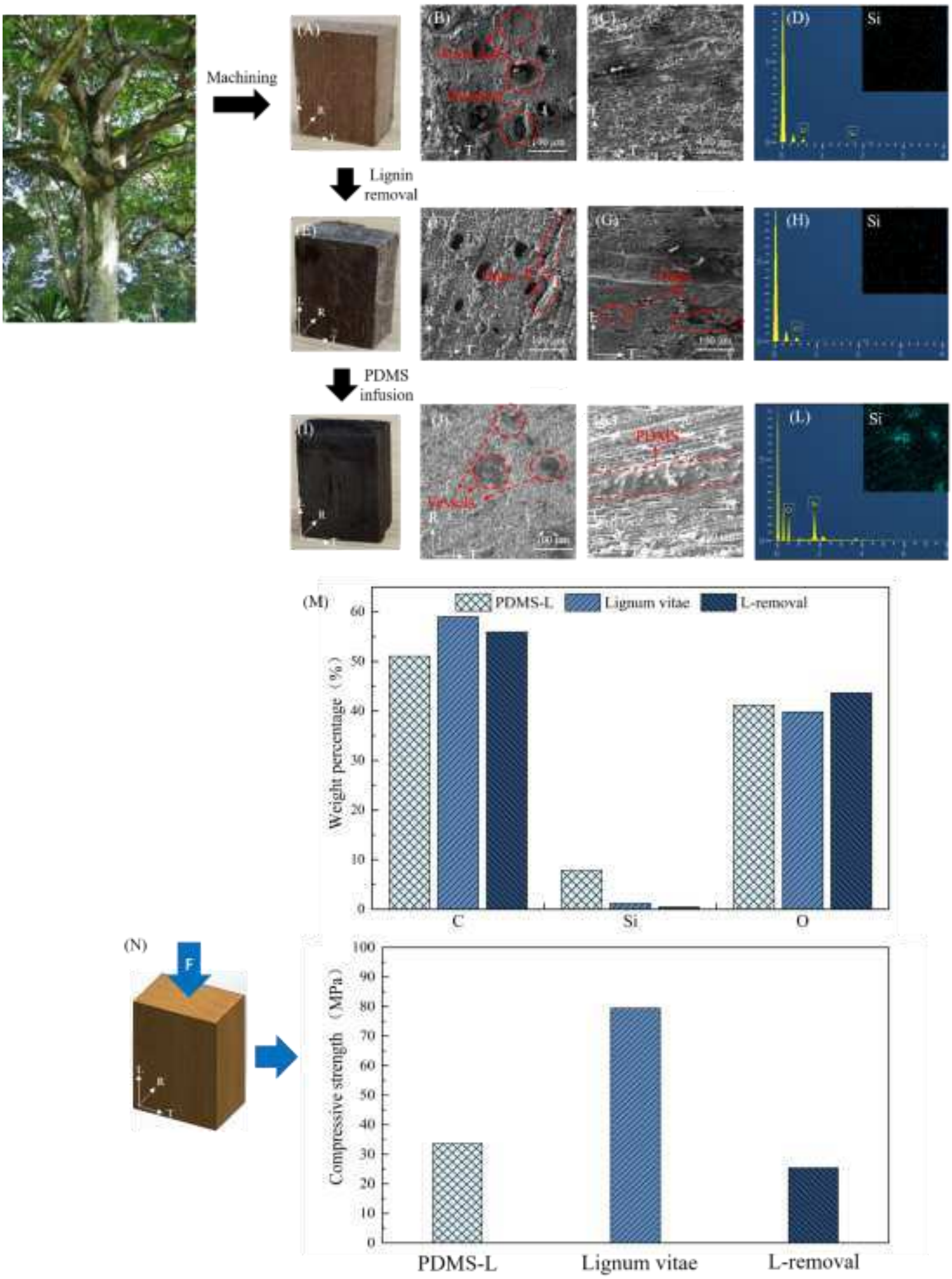

Fig. 1. Characterization of various kinds of wood. (A), (E) and (I) are the Lignum vitae, the L-removal and the PDMS-L's middle central part, respectively. (B), (F) and (J) are the cross section observed by SEM of the Lignum vitae, the L-removal and the PDMS-L's middle central part, respectively. (C), (G) and (K) are the tangential section observed by SEM of the Lignum vitae, the L-removal and the PDMS-L's middle central part, respectively. (D), (H) and (L) are the EDS results of the Lignum

vitae, the L-removal and the PDMS-L's middle central part, respectively. (M) The weight percentage of elements. (N) The compressive strength tests.

We also investigated the surface layer of the PDMS-L samples. According to the SEM images, the surface layer and the interlayer structure present a certain difference. The original structure of the Lignum vitae, including the various kinds of xylem cells and the channels, disappears on the surface layer. Once the surface layer has been cut, the unique porous structure of wood exists again. On the cross section, the various xylem cells, which cannot be identified on the surface layer, can be distinguished on the interlayer (Fig. 2, B and C). On the tangential section, the difference is more obvious. SEM image of the surface layer presents a smooth surface without any original typical structure while the wood cells and the vessels are easily distinguished in SEM image of the interlayer (Fig. 2, D and E). It is worth mentioning that the distribution of silicon of the surface layer and the interlayer is also quite different. Silicon uniformly distributes on the surface layer. On the contrary, most silicon concentratedly distributes in the vessels while a few uniformly distribute in the wood cells on the interlayer. The weight percentage of silicon on the surface layer is 128.5% higher than that on the interlayer. Taking the above differences into consideration, it is reasonable to get the conclusion that a thin PDMS film layer adhered to the surface layer of the PDMS-L (Fig. 2G).

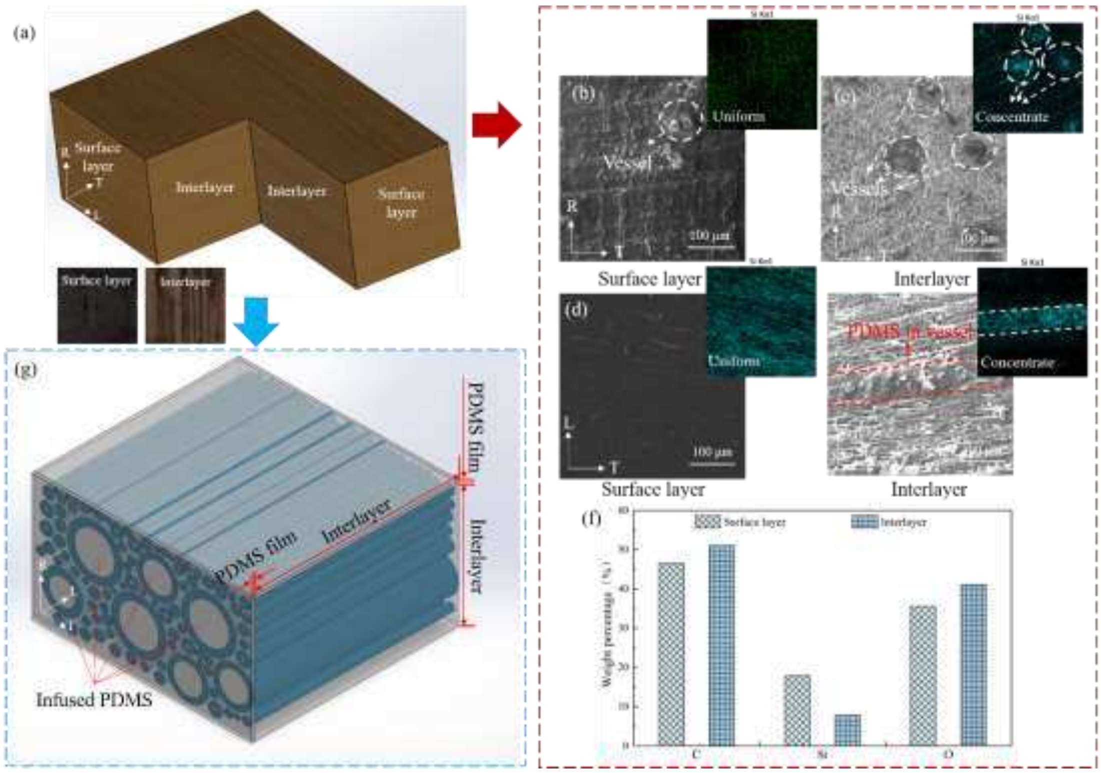

Fig. 2. Structural and elements characterization of the PDMS-L. (A) The model for the surface layer and the interlayer. (B) and (C) are the cross section of the surface layer and the interlayer, respectively. (D) and (E) are the tangential section of the surface layer and the interlayer, respectively. (F) The weight percentage of elements of the surface layer and the interlayer. (G) the model for the PDMS-L description.

3.2 Tribological experimental results

Fig. 3 illustrates the tribological experiments and the friction coefficient of the three kinds of samples. The friction coefficient of PDMS-L is significantly lower than the other two kinds of samples, which is 78.1%, 82.3%, 85.2% and 75.5% lower than the Lignum vitae at 5 N, 10 N, 15 N and 20 N, respectively (Fig. 3B). The friction coefficients of the PDMS-L are 0.13, 0.10, 0.08 and 0.12 at 5 N, 10 N, 15 N, 20 N, which are pretty low values under dry frictional conditions. We also investigated the operational effectiveness of the PDMS-L along with the whole experiment. The friction coefficient of the PDMS-L at 20 N increases slowly and smoothly from around 0.06 to 0.12 at the initial stage of the experiment and remains stable with minor fluctuations around 20 minutes since the experiment started (Fig. 3C). It is reasonable to get the conclusion that the lubricating performance of the PDMS-L is guaranteed throughout the whole experiment. The wear track of the samples after tribological experiments has been observed. According to the wear morphologies, the wear track is cambered and some wear particles accumulate at the edge of the wear track, resulting in the edge heightening of the wear track (Fig. 4, A, to C). The level of the PDMS-L changes quite slightly compared with the other two kinds of samples. Additionally, the wear track depth, which is obtained by measuring the outline of the wear track, has been used to evaluate the wear resistance of the samples (Fig. 4, D and E). Among the three kinds of samples, the PDMS-L presents the best wear resistance, the wear depth decreases obviously after PDMS infusion. The wear depth of the PDMS-L is 45.5%, 59.0%, 70.2% and 79.0% lower than the Lignum vitae at 5 N, 10 N, 15 N and 20 N, respectively (Fig. 4E). The above results indicate that the lubricating performance of the PDMS-L has been significantly improved compared with Lignum vitae.

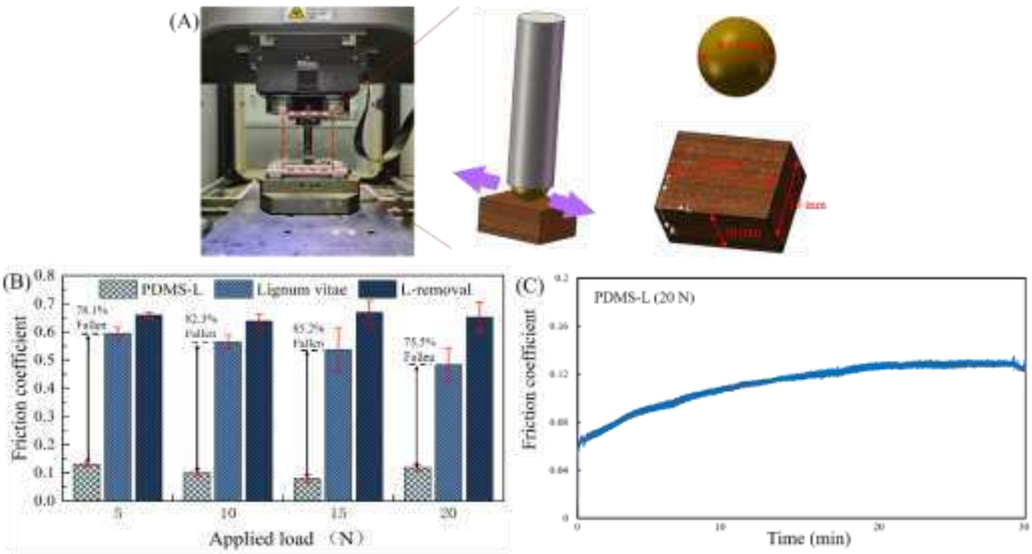

Fig. 3. The scheme of the tribological experiments and the friction coefficient of the samples. (A) The schematic for the tribometer and the friction pairs. (B) The friction coefficient of the three kinds of samples. (C) The real-time friction coefficient of the PDMS-L at 20 N.

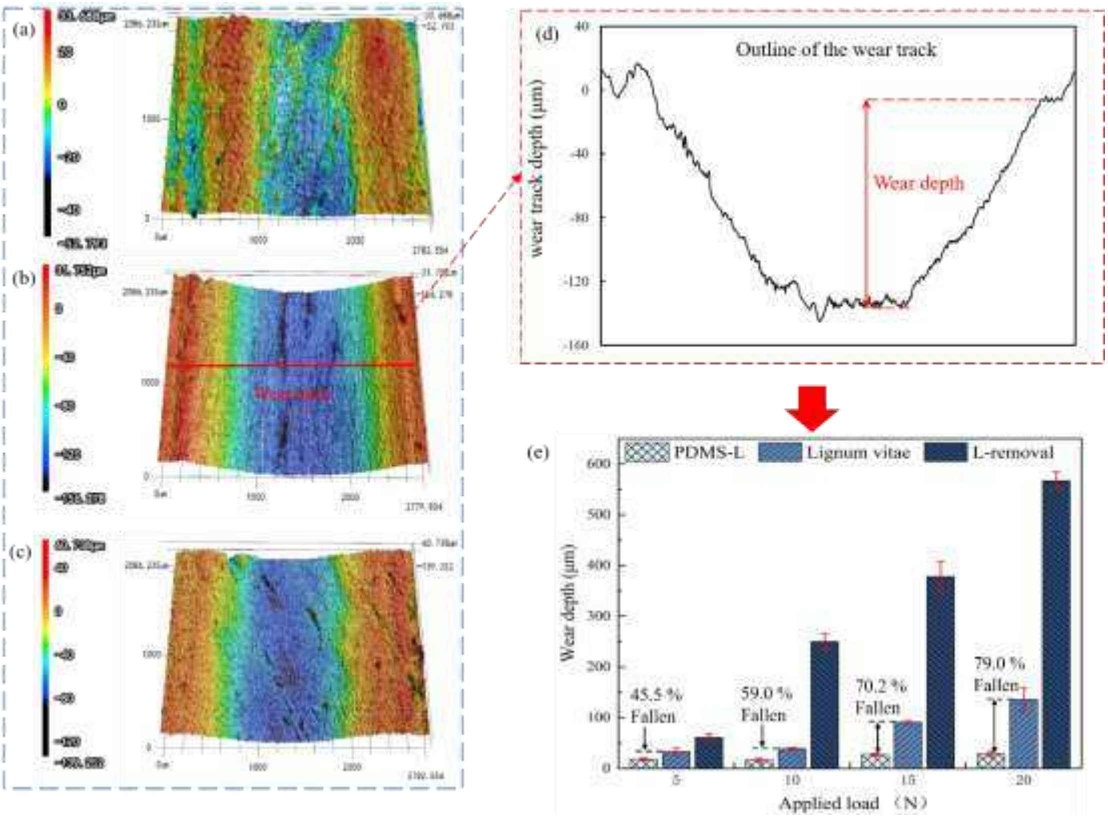

Fig. 4. The wear morphologies and wear track depth of the samples.
(A), (B) and (C) are the three dimensional wear tracks of the PDMS-L, the Lignum vitae and the L-removal at 20 N, respectively. (D) The wear track measured by CLSM. (E) The wear depth of three kinds of samples.

3.3 Wear mechanisms

The wear morphologies of the samples at 20 N have been investigated by using SEM to better understand their wear mechanisms. According to the wear morphologies, the wear of the Lignum vitae, as we mentioned in our previous study, initiates from the crushing of the hollow wood fibers followed by pulling out and tearing the destroyed wood fibers along the parallel direction (Fig. 5B) [Wu et al., 2022]. After lignin is removed, the wear of the L-removal is similar to the Lignum vitae but the wear becomes serious, many torn fibers can be found on the surface of the L-removal after experiments (Fig. 5C). It is mainly because of the cellulose fibrils' linkage is released after the lignin is removed, which makes the fibers easily to be destroyed.

As for the PDMS-L, more wear morphologies and a wear mechanism diagram are used to explain the wear mechanism more reasonably (Fig. 5, A and D). The wear mechanism diagram summarized the lubricating performance improvement mechanism of the PMDS-L (Note that the copper ball is omitted in parts of the images). The PDMS-L sample is covered by a thin PDMS film, which is compressed by the copper ball at first, followed by wear and destruction as the experiment continues. During the process, friction and wear mainly occur at the interface of the PDMS film, the top soft PDMS film bears the applied load. Large deformation happens for the PDMS film to completely fit the copper ball's rough peaks on the surface. Additionally, the interlayer, which is formed by the PDMS reinforced fibers, provides strong support. Due to the unique soft-hard composite structure, the friction coefficient of the PDMS-L is pretty low at the beginning. The wear of the PDMS-L in this process is mainly the tear of the PDMS film. However, the wear resistance of PDMS is relatively poor. The PDMS film, which is at the top of the surface layer, has been gradually destroyed as the experiment goes on and the inner PDMS reinforced wood fibers are gradually exposed. This section is a transitional process in which the destroyed area of the PDMS film gradually increases and the amount of the exposed wood fibers gradually increases. During this process, the friction coefficient of the PDMS-L increases slowly and smoothly from around 0.06 to 0.12 as the experiment goes on due to the PDMS film being destroyed gradually. Note that different from the original Lignum vitae, the wood fibers of the PDMS-L are filled with PDMS, resulting in the deformation of the wood fibers rather than crushing. Thus the main wear of the PDMS-L in this process is the tear of the PDMS film combined with the stretching and tear of the cell walls of the wood fibers along the parallel direction. As the experiment continues, once the cell walls of the wood fibers are destroyed, the inner PDMS is exposed. The exposed PDMS plays the same role as PDMS film to fit the copper ball's rough peaks through deformation, where the bottom PDMS reinforced fibers provide strong support for the exposed PDMS. Such a unique soft-hard composite structure significantly improves the lubricating performance of the PDMS-L. Once the exposed PDMS is destroyed, the PDMS inside the bottom wood fibers will expose again after the cell wall is destroyed. The large amount of wood fibers ensures this wear cycle, thus the friction coefficient of the

PDMS-L becomes stable with little fluctuation and the lubricating performance of the PDMS-L is guaranteed throughout its service life.

There is an issue that deserves consideration, both the friction coefficient and the wear depth of the L-removal are higher compared with the original Lignum vitae. Based on the wear mechanism, the deformation resistance of the L-removal decreases due to the linkages of the cellulose fibrils being cut off after lignin is removed, resulting in the extreme decrease in the wear resistance (Fig. 1N). Lots of fibers are torn and exist on the surface of the L-removal, some of which will enter the friction pairs, resulting in the increase of the friction coefficient (Fig. 5C). Additionally, the effect of applied loads on the lubricating performance of the PDMS-L has been summarized based on the wear mechanism. Due to the increase in the contact area and, consequently, the increase in the friction force with the increase in the applied load, resulting in more PDMS are destroyed. Due to the tight contact of the copper balls and the samples, the broken bits of PDMS may not be discharged in time and exist in the rubbing pairs, which will reduce the lubricating performance of the samples. However, when the applied loads are 5 N, 10 N and 15 N, the broken bits of PDMS are few and have a limited effect on the friction properties of PDMS-L. Thus the friction coefficient of the PDMS-L decreases with the increase in the applied load from 5 N to 15 N. When the applied load is 20 N, a large area of the PDMS film is destroyed and a large amount of broken bits of PDMS are produced. These broken bits roll in the rubbing pairs and destroy the contact face as the copper ball slides, causing the increase in the friction coefficient. The above is also the reason for the friction coefficient reduction percentages of PDMS-L compared with the Lignum vitae increase with the applied load increases from 5 N to 15 N, and then decreases from 15 N to 20 N. Differing from the wood fibers of the PDMS-L bear the load through the elastic deformation of the infused PDMS, the wood fibers of the original Lignum vitae bear the load through irreversible plastic deformation. Increasing in the applied load will significantly increase the amount of the crushed wood fibers, resulting in the rapid growth of the Lignum vitae's wear depth. However, the elastic deformation of the infused PDMS makes the PDMS-L less sensitive the applied load than the Lignum vitae, resulting in the continuous increase in the wear depth reduction percentages of PDMS-L compared with the Lignum vitae with the applied load increase from 5 N to 20 N.

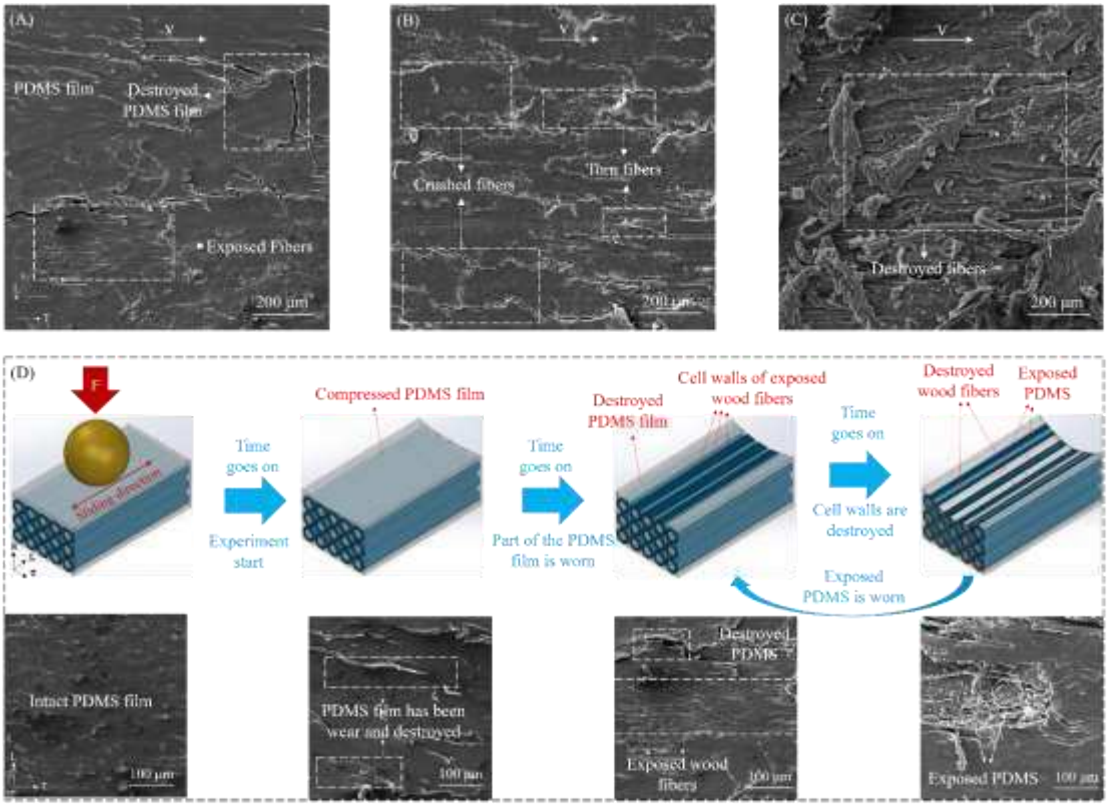

Fig. 5. The wear morphologies taken by SEM and the wear mechanism of the PDMS-L. (A), (B) and (C) are the wear morphologies of the PDMS-L, the Lignum vitae and the L-removal at 20 N, respectively.

(D) The wear mechanism of the PDMS-L.

4 Conclusion

In order to improve the lubricating performance of wood for wide applications, a new Lignum vitae wood-derived composite is developed through a convenient and effective infusion process. The lignin of the Lignum vitae is removed at first, then followed by means of vacuum-assisted PDMS infusion. After that, a series of experiments have been done to detailedly analyze the characterization and the lubricating performance of the Lignum vitae, the L-removal and the PDMS-L. There are some conclusions drawn as follows.

(1) The microstructure and EDS observation results prove that the PDMS has

successfully infused into the Lignum vitae. After PDMS infusion, the wood fibers, which are separated during the lignin removal processing, have been connected again and a thin PDMS film adheres to the surface layer of the material.

(2) The compressive strength decreases rapidly after lignin is removed and increases slightly after PDMS infusion.

(3) According to the tribological experimental results, the friction coefficient of the PDMS-L is pretty low, which is 78.1%, 82.3%, 85.2% and 75.5% lower than the Lignum vitae at 5 N, 10 N, 15 N and 20 N, respectively. The wear depth of the PDMS-L is 45.5%, 59.0%, 70.2% and 79.0% lower than the Lignum vitae at 5 N, 10 N, 15 N and 20 N, respectively.

(4) The PDMS infusion perfectly improves the lubricating performance of the PDMS-L by forming the unique soft-hard composite structure. The PDMS can fit the rough peak of the copper ball through large deformation and the interlayer PDMS reinforced fibers can provide strong support

Overall, the design and development of the wood-derived composite provide a new route for the excellent tribological application of natural wood. However, the long-term lubricating performance of the samples hasn't been tested in our study. We will do long time experiments and also try to improve the lubricating performance of other kinds wood by using the same methods in the future.

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CRediT authorship contribution statement:

Zumin Wu: Conceptualization, Investigation, Methodology, Data curation, Writing–original draft. **Zhiwei Guo**: Supervision, Funding acquisition, Writing–review and editing. **Chengqing Yuan**: Supervision, Funding acquisition, Writing–review and editing. **Qiren Huang**: Investigation. **Chun Wang**: Writing–review and editing.

Hongyuan Zhao: Writing–review and editing.

Declaration of competing interest:

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data and materials availability:

Raw/processed data will be made available upon request.

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