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DECAY RESISTANCE, CHEMICAL AND ANATOMICAL PROPERTIES OF SILICONE OIL MODIFIED FAST-GROWING PINE WOOD AGAINST WOOD-DESTROYING FUNGI

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Abstract:

Decay resistance, chemical and anatomical properties were determined for silicone oil heat treated fast-growing pine wood (Pinus massoniana L.) exposed to wood decaying fungi and compared with the untreated. The test samples were treated at 150^{o}C and 210^{o}C for 2h and 8h. Wood deterioration test was evaluated in the laboratory. Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), transmission light microscopy and scanning electron microscopy (SEM) were used to investigate the chemical changes after the treatment and exposure to white-rot and brown-rot fungi. The results showed that mass loss decreased with increasing treatment temperature and the highest decline in mass loss value was found at 210^{o}C for 8h, with 6.94% caused by white-rot and 7.03% caused by brown-rot fungi. The chemical components of the wood were significantly modified by silicone oil heat treatment. The anatomical structure revealed that silicone oil occupied the wood voids and penetrated the wood cell walls.

Key words*: decay resistance; Gloeophyllum trabeum; pine wood; silicone oil; white-rot fungus.*

INTRODUCTION

Pine wood [*Pinus massoniana* (Lamb) Hook] is one of the widely planted fast-growing tree species in southwest China, whose wood is gradually utilised in construction, furniture, and as raw materials for wood fiber. However, it has an inherent drawback, such as susceptibility to biological decay (i.e. low natural durability) restricting its potential wider applications and utilisation in sundry wood products and wooden structures etc.

The main approach to improve wood properties is to modify its cell wall constituents, e.g., cellulose, hemicellulose, lignin and extractives (Kaplan et al. 2018). In the past, chromate copper arsenate (CCA) and creosote were the traditional methods used to treat wood against biodegrading organisms. Still, their usage is strictly regulated because they are toxic and dangerous to the environment (Baar et al. 2021).

Over the last decades, there has been a renewed interest throughout the academic, industrial, and social sectors in wood modification. Wood modification involves changing the physical or chemical properties of wood to optimise its performance for exterior and interior applications (Militz 2020), resulting in a desired property enhancement during the service life of the modified wood (Hill 2007). The modification of wood has undergone significant evolution over time, resulting in the development of more innovative solutions that enhance the performance and durability of wood products (Mandraveli et al. 2024).

Thermal modification is one of the oldest methods used to modify wood properties such that at the end of the life cycle of the heat treated wood, it can be recycled without consequential environmental impact, to the contrary of chemically treated wood impregnated with biocidal active ingredients (Candelier et al. 2016). Oil is used in heat treatment as an alternative method of treating wood without chemical preservatives, and thermal modification in oil baths is an effective environmentally friendly, pollution-free, and sustainable wood modification method (Okon and Udoakpan 2019).

One of the known characteristics of heat treated wood is an improvement in decay resistance against biodeterioration agents like fungi. Oil heat treatment has been reported to be a very effective method in enhancing the resistance of wood against fungus (Lyon et al. 2007, Wahab et al. 2012, Umar et al. 2016). Kamdem *et al*. (2002) and Weiland and Guyonnet (2003) gave some reasons for the improvement in the

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durability of the treated wood including enhancement in the hydrophobic character of wood, production of extractives, modification of the wood polymers and degradation of hemicelluloses.

Therefore this research's overall aim was a comparative investigation of the resistance of untreated and silicone oil treated fast-growing pine wood to attack by brown and white-rot causing fungi. Furthermore, to gain a deeper understanding of the mechanisms behind the durability of the wood after treatment, we utilised analytical techniques such as Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), transmission light microscopy and scanning electron microscope (SEM). These methods were employed to examine changes in the chemical structure and microstructure of the modified pine wood. This study provides new theoretical and technical approaches for enhancing the durability of fast-growing trees.

MATERIAL AND METHODS

Pine wood (*Pinus massoniana* L.) was obtained from the Fujian Province in the People's Republic of China. The pine tree was 20 years old. From the butt end of the trunk, a radial board was cut and used for the preparation of 10mm × 10mm × 20mm (radial × tangential × longitudinal) samples with annual rings almost parallel to the tangential edges. The samples were conditioned in a chamber at 20 ± 2°C and 65 ± 5% relative humidity (RH) to a moisture content of about 12%. A total of 5 groups of 12 samples from sapwood were heat treated under defined conditions, and 1 group of 12 samples was left untreated (20°C).

The brown-rot fungus *Gloeophyllum trabeum* (Pers. ex. Fr.) Murr. (*G. trabeum*) and the white-rot fungus *Trametes versicolor* (L. ex Fr.) Qu'el. (*T. versicolor*) were obtained from the China Forestry Culture Collection Centre (Beijing, China). The fungi were cultivated on a culture medium of 3.7% potato dextrose agar (PDA) at 28°C and a relative humidity of 80% for 7 days before use.

Pine wood was treated with silicone oil as the heating medium in a preheated Binder ED 53 (Tuttlingen, Germany) laboratory heating bath at 150°C and 210°C for 2 and 8h. The temperature and treatment time were selected based on previously reported heat treatment conditions. After the treatment, each treated sample was removed from the oil bath and cooled down in a desiccator. Then, the treated wood samples were exposed to fungi to determine the mass loss and some chemical properties.

Wood durability tests were conducted according to the Chinese Standard GB/T 13942.1-2009. The classification of wood decay resistance is presented in Table 1. The decay fungi were incubated at 28°C and 80% RH until the mycelium completely colonised the surface of PDA. Subsequently, the sterilised wood block samples from an autoclave were transferred to the fungal cultures after conditioning at 20 ± 2°C and 65 ± 5% RH and incubated for 12 weeks. All treated block samples were incubated at 28°C and 80% RH. After incubation, the mycelium was removed from the wood block, dried, and the mycelium-free wood blocks were weighed. The mass loss was calculated using equation (1). To determine the average mass loss (ML), 12 replicates were used for each treatment.

$$ML\ (\%) = \frac{m_1 - m_2}{m_1} \times 100 \qquad 1$$

where: m_1 = the initial weight before the decay resistance test
m_2 = the weight after 12 weeks of fungal incubation

Table 1

Decay resistance classification of wood

Decay-resistant class	Softwood mass loss (%)	Hardwood mass loss (%)
Highly durable (I)	0-10	0-10
Durable (II)	11-20	20-30
Moderately durable (III)	21-30	31-50
Non-durable (IV)	>30	> 50

For the chemical components analysis of untreated and treated wood samples exposed to fungi, the samples were chopped into small pieces and milled using a Wiley mill into a homogenous meal. The ground samples were sieved in a mesh of 0.25mm and 0.42mm (40 - 60 mesh) to get a standard particle dimension.

Changes in the chemical structure between untreated and treated wood samples were analysed using infrared spectroscopy. Fourier transform infrared (FTIR) spectra were obtained using Nicolet 380 FTIR spectrometer (Thermo Electron Instruments Co., Ltd., USA) between 3200 - 500cm^{-1}, with a resolution of 4cm^{-1}. Each sample was ground to powder, blended with KBr and then pressed into thin pellets. The pellets were then scanned to obtain FTIR spectra.

The crystalline structures of the untreated and treated wood samples were analysed using X-ray diffraction analysis. The X-ray diffraction measurement was performed with an X-ray diffractometer (Rigaku Miniflex600, Japan) using Cu Kα radiation with a monochromator, voltage 40kV, electric current 40mA, water flow 4.6 L/min and diffractograms range of 5 - 50^{o} 2θ with a scanning of 0.1^{o}/min.

Samples of untreated and treated wood used for anatomical imaging were microtomed using a rotating microtome HistoCore AUTOCUT (Leica, Wetzlar, Germany) after soaking the samples in water for several days until they became saturated and sank to soften the wood. Thin smooth sections were obtained on the radial surfaces of the wood and the thickness of the thin slices cut was 18µm. Staining was carried out by adding drops of safranin solution to the thin section in a petri dish. Samples were then placed on slides, covered with microscopic coverslip and oven-dried to remove air bubbles. The prepared surfaces were examined using a Zeiss Axioplan 2 Imaging reflected light microscope (Zeiss, Oberkochen, Germany) with an objective magnification of 10. The anatomic images were captured using the Nikon Digital Sight DS-U3 camera system (Nikon, Tokyo, Japan) in combination with the capture software NIS Elements Basic Research (Br) (Nikon Instruments Europe B.V., Amstelveen, Netherlands) with a magnification of 100x.

Untreated and treated wood sample morphologies were analysed using a scanning electron microscope (FEI QUANTA 200SEM). Air-dried samples were fixed onto aluminium stubs through carbon adhesive disks, and their fractured surface was observed with a low vacuum secondary electron detector using the accelerating voltage of 25.0kV. The samples were analysed at room temperature and an internal pressure of 0.50 torr.

The decay resistance of the untreated and treated wood was statistically analysed using R statistics version 4.3.1 (CoreTeam 2024). Means were analysed using one-way Analysis of Variance (ANOVA) with Tukey's multiple comparison test (TMCT) at $p < 0.05$.

RESULTS AND DISCUSSION

Wood resistance against fungi attack

Silicone oil heat treatment of fast-growing pine wood reduced mass losses against wood-destroying fungi tests compared to untreated as shown in Figs. 1 and 2. The results of the decay resistance tests showed significant differences in mass loss of the treated pine wood exposed to white-rot fungus ($p<0.001$) (Fig. 1) and brown-rot fungus ($p<0.001$) (Fig. 2). The mass loss decreased with an increase in treatment temperature and a prolonged treatment time. According to the results in Figs. 1 and 2, silicone oil heat treated wood at 150^{o}C had a lower mass loss, resulting in a better decay-resistant class. The improved decay-resistant class of the treated wood was further verified at 210^{o}C, as this treatment received the best classification (decay-resistant class I, Table 1). The lowest mass loss was observed in treated wood samples exposed to white-rot (6.94%) and brown-rot (7.03%) fungi both at 210^{o}C for 8h, indicating higher resistance of the treated (decay-resistant class I, Table 1) against fungi attack and also indicating the effectiveness of silicone oil heat treatment in modifying the wood properties of pine wood. The positive effect of the silicone oil thermal modification on fast-growing pine wood resistance is related to the reduction in hemicellulose and the concomitant proportional increase in total lignin in the cell wall. Specifically, the treatment at 210^{o}C performed better than at 150^{o}C.

The white-rot fungus (62.14%) caused the greatest mass loss to the untreated pine wood compared to the attack by the brown-rot fungus (51.31%) which was classified as non-durable in Table 1. This result is not in line with the report by Brischke *et al*. (2024), the white-rot fungus (*T. versicolor*) caused a higher mass loss on black locust wood (hardwood), than the brown-rot fungus (*C. puteana*). In this study, white-rot fungus was generally more virulent than brown-rot fungus, but both test fungi caused mass loss well above the threshold of 20%. Hence, the test was valid according to EN 113-2 (2021). As shown in Figs. 1 and 2, silicone oil heat treatment increased the resistance of the fast-growing pine wood against attack by both fungi, consistent with previous findings (Esteves 2009, Kamdem *et al*. 2002).

Rousset *et al*. (2004) and Metsa¨-Kortelainen and Viitanen (2010) reported that thermal treated wood at high temperatures is more resistant to decay fungal attacks. Additionally, Momohara *et al*. (2003) reported that the resistance of timbers to fungal decay was enhanced by longer thermal treatment times and higher temperatures, which is consistent with the present findings.

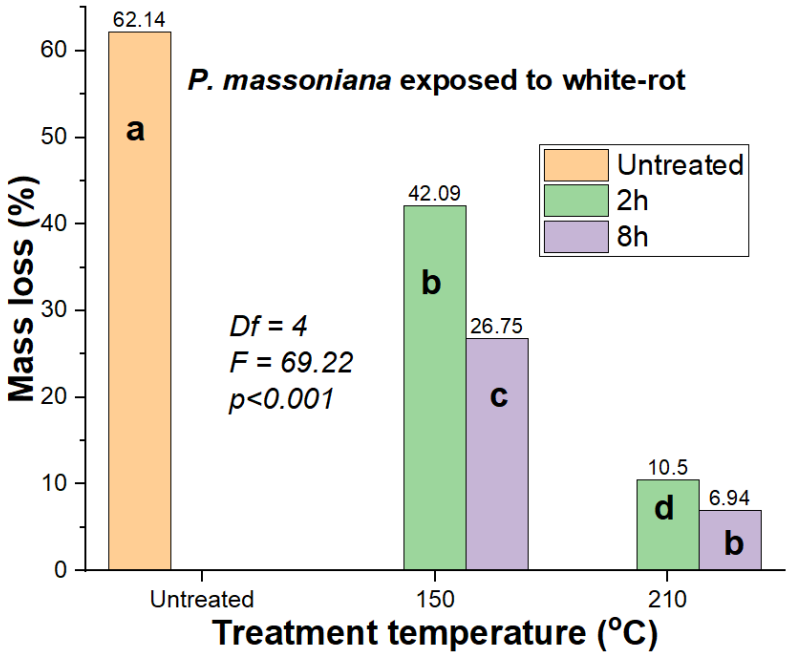

Fig. 1.
Mass loss of the untreated and silicone oil heat treated fast-growing pine wood exposed to white-rot fungus. The values represent the mean and mean within a column followed by the same letter are not significantly different (Tukey, p < 0.05).

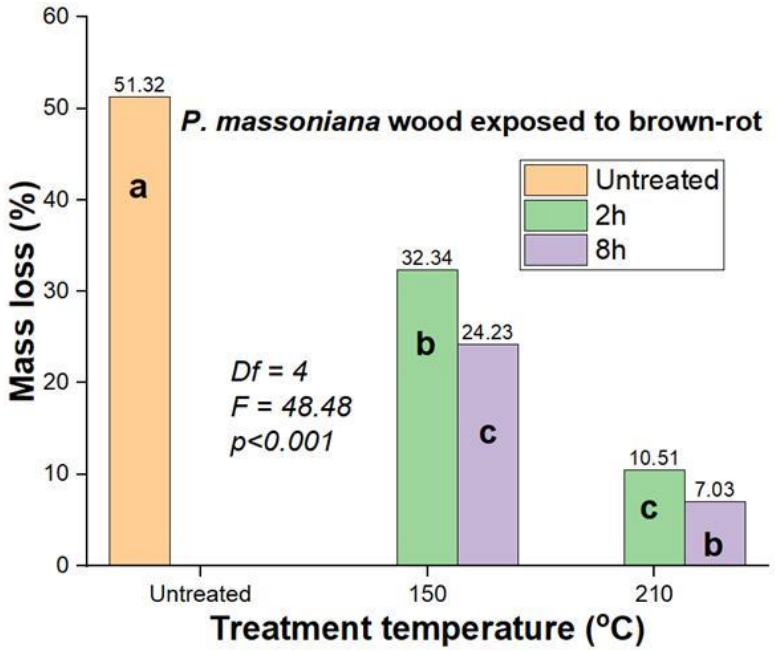

Fig. 2.
***Mass loss of the untreated and silicone oil heat treated fast-growing pine wood exposed to brown-rot fungus. The values represent the mean and mean within a column followed by the same letter are not significantly different* (Tukey, *p < 0.05*).**

The FTIR spectra of untreated and silicone oil heat treated fast-growing pine wood exposed to white and brown-rot fungi after 12 weeks of incubation are shown in Figs. 3 and 4 to investigate the structure changes in the wood constituents after treatment. Slight changes were observed in the spectra of the treated wood compared to the untreated, suggesting modification in the chemical components of the wood which does not promote fungal decay. C-H symmetric stretching vibration of methyl and methyl groups at various spectra characterises the peak at 2970cm^{-1}. The apparent displacement of the C-H band indicated that the cellulose structure was affected during heat treatment due to cellulose degradation in the cell wall of the wood (Okon et al. 2017). The peak at 1540cm^{-1} wavenumber was assigned to C=C stretching aromatic skeletal vibrations in lignin. The peak at 1012cm^{-1} band represents aromatic C-H in-plane deformation. The peak intensity decreases with increasing treatment temperature and time. The peak at 798cm^{-1} was assigned to C-H vibration within the guaiacyl lignin condensed unit and aromatic bending out of plane deformation of lignin, indicating a severe decrease of cellulose band and suggesting structural changes in the treated wood. Furthermore, treated wood showed a decline in the cellulose and hemicellulose contents and an increase in the lignin content, thus making the treated wood highly durable.

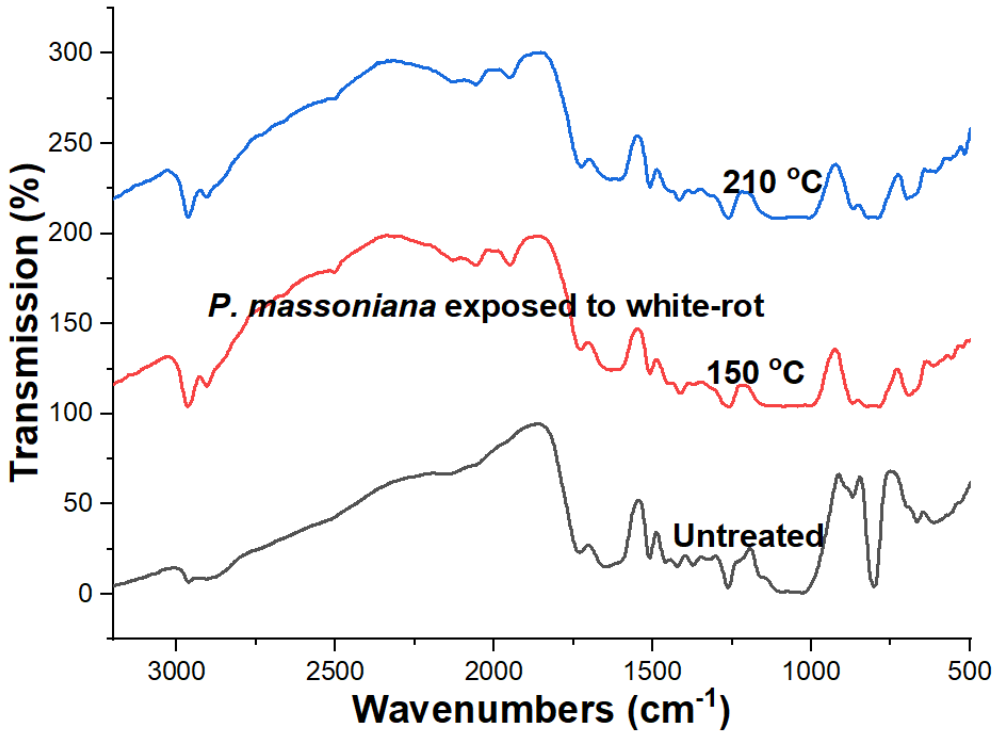

Fig. 3.
FTIR spectra of untreated and silicone oil heat treated Pinus massoniana wood exposed to white-rot fungi.

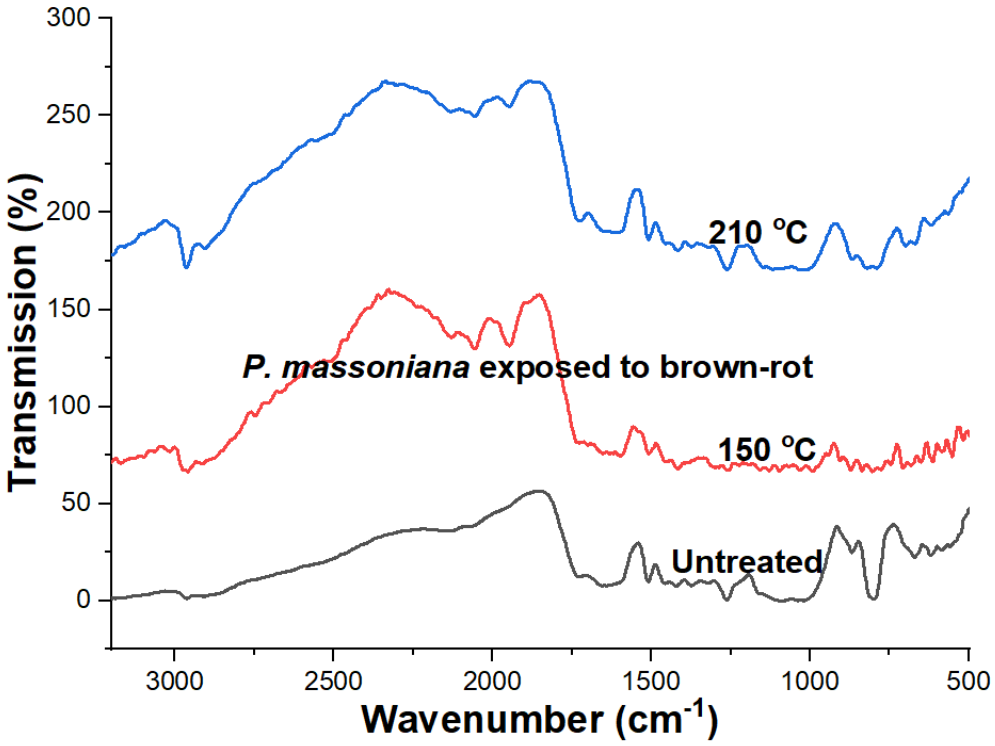

Fig. 4.
FTIR spectra of untreated and silicone oil heat treated Pinus massoniana wood exposed to brown-rot fungi.

Figs. 5 and 6 demonstrate the changes in the crystallinity of untreated and silicone oil heat treated fast-growing pine wood at different temperatures after exposure to decay fungal. The results show that heat treatment of the wood and exposure to fungal attack had a limited effect on the crystalline region and the diffraction position was stable. Though there was no significant alteration in the crystalline structure, the silicone oil heat treatment adjusted the cellulose crystallinity of the exposed wood. The diffractograms of the exposed wood to white and brown-rot fungi showed a cellulose I crystalline pattern with distinct peaks observed at 12.80°, 15.82° and 22.68° 2θ which corresponds to 101 and 002 of the crystallographic plane (Okon *et al*. 2017). The diffraction pattern of the exposed treated and untreated wood to fungal decay appears similar with a high-intensity peak at 22.68° 2θ corresponding to the increase in the relative crystallinity of the wood after treatment with silicone oil and exposure. The increase in cellulose crystallinity of the heat treated wood may originate from hemicellulose degradation, leading to the rise in the proportion of crystallisation. The bridging reaction in the amorphous region of cellulose contributed to a more orderly arrangement of microfibrils, shortening the molecular spacing and tending toward the crystalline region, which increased the possibility of hydrogen bond formation, thus increasing the degree of crystallinity. Increased crystallinity reduces the hygroscopicity and swelling of the cell walls, enhancing the durability of pine wood. Hastrup *et al*. (2012) reported that the relative crystallinity of pine wood exposed to white-rot fungi (*Irpex lacteus* and *Pycnoporus sanguineus*) increased after the exposure. They attributed the increase to hemicelluloses and easily available non-crystalline cellulose degradation during decay (Howell et al. 2009, Hastrup *et al*. 2012). Heat treatment above 200°C greatly degrades hemicelluloses and reduces the hygroscopicity of wood, thus preventing the fungal hyphae from feeding on carbohydrate products (Boonstra *et al*. 2007). Furthermore, severe degradation of hemicelluloses may lead to the formation of toxic compounds that can enhance fungal resistance (Kamdem *et al*. 2002, Dubey *et al*. 2012).

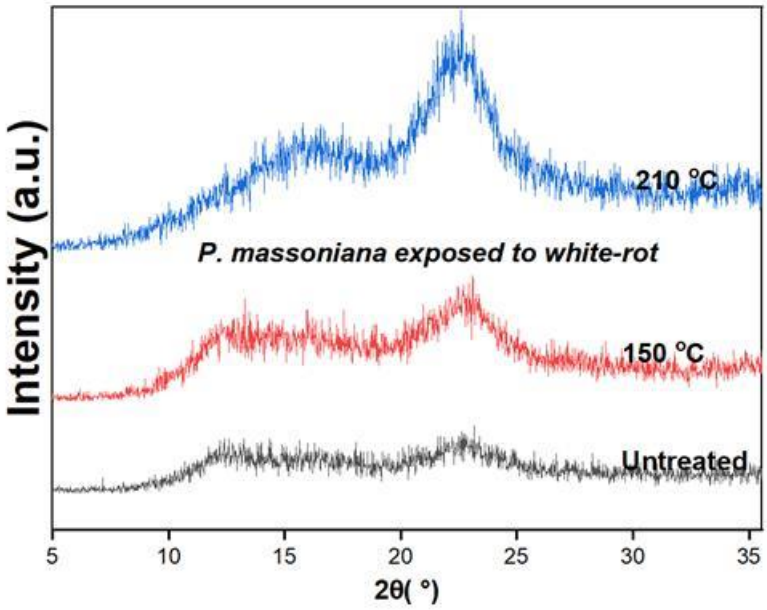

Fig. 5.
X-ray diffraction patterns of untreated and silicone oil heat treated Pinus massoniana wood exposed to white-rot fungi.

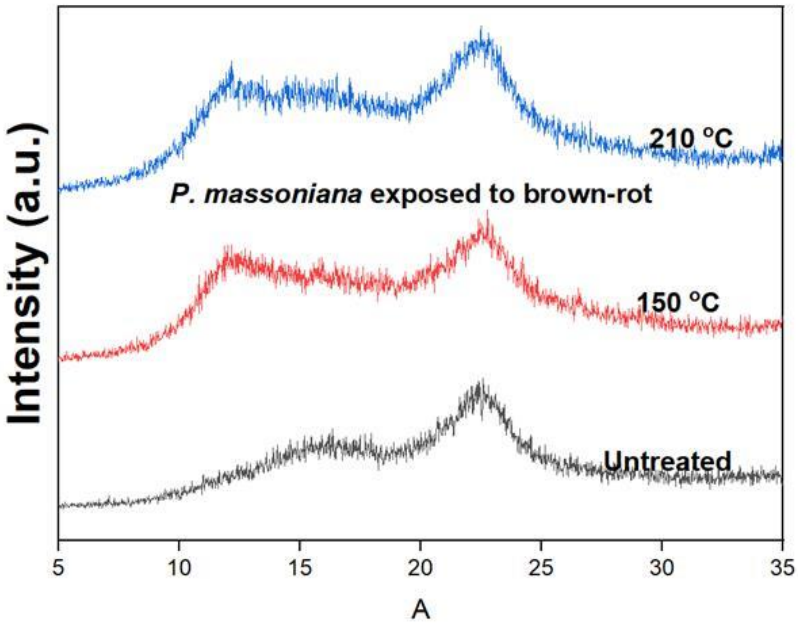

Fig. 6.
X-ray diffraction patterns of untreated and silicone oil heat treated Pinus massoniana wood exposed to brown-rot fungi.

The microstructure and morphologies of the untreated and silicone oil heat treated fast-growing pine wood at different temperatures and subsequent exposure to decay fungal were investigated (Fig. 7a to 7e). Fig. 7a to 7c show the anatomical images of the radial section of untreated and silicone oil heat treated pine wood samples. As shown in Fig. 7a, the wood anatomical features were visible on the untreated samples. Though slight changes were observed in the anatomical properties of the treated samples, this was attributed to the effect of the treatment. The anatomical structure of the wood appears not to be damaged by

the impact of the treatment and there are no signs of cracks and distortion of the vessels after silicone oil heat treatment at 150°C and 210°C for 8h compared to the untreated (Fig. 7b and c). The shape of the wood vessels was not deformed after the treatment, demonstrating that the anatomical structures of the wood were less affected during silicone oil heat treatment. The colour of the wood samples was observed to become darkened (Fig. 7b and c) after silicone oil heat treatment at 150°C and 210°C respectively, this was attributed to hemicelluloses degradation. Exposure of the untreated pine wood to white-rot fungus showed that the wood samples were completely covered by mycelium (Fig. 7d), indicating its susceptibility to fungal attack. On the other hand, mycelium found it difficult to grow on the silicone oil heat treated samples (Fig. 7e), the treated wood samples were resistant to fungal attack.

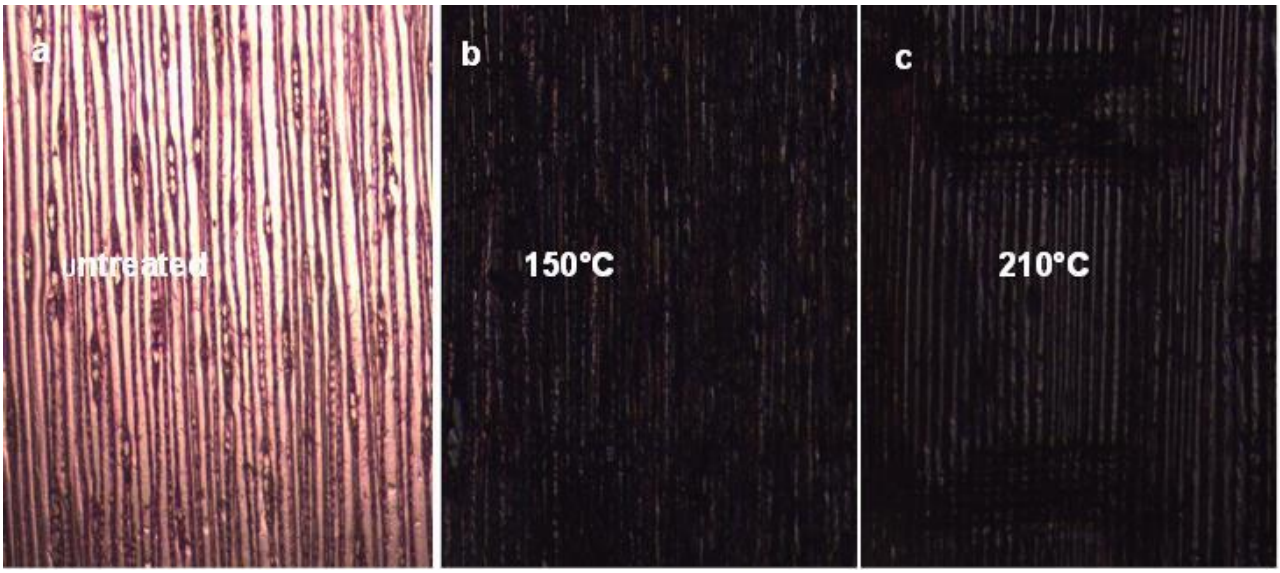

Fig. 7.
Radial section anatomic images: (a) untreated fast-growing pine, (b) silicone oil treated at 150°C/ 2h, (c) silicone oil treated at 210°C/ 8h.

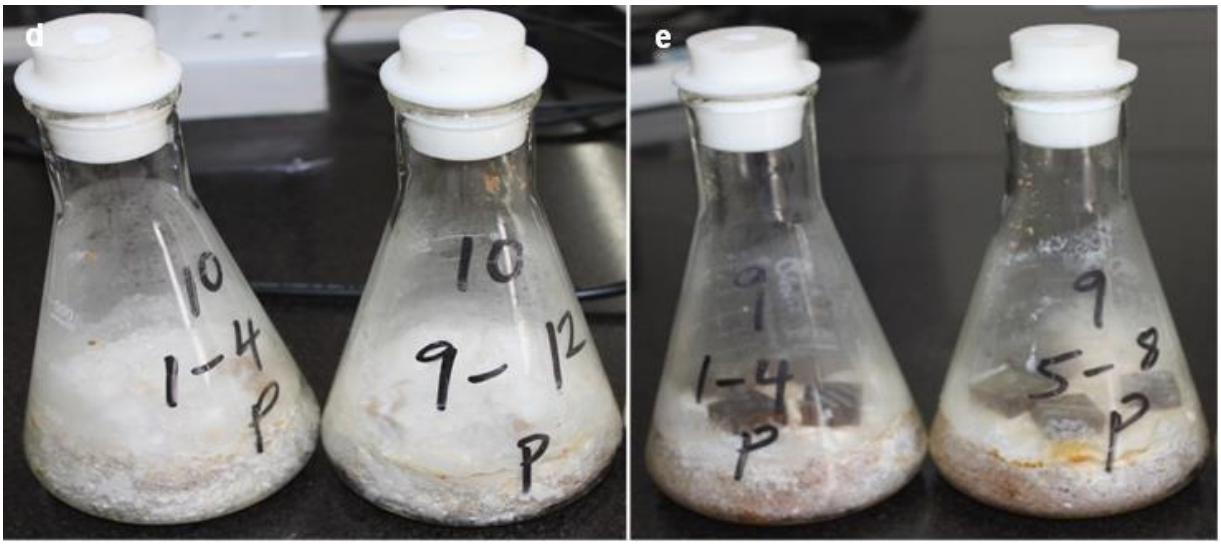

Fig. 7.
Decay resistance test of pine wood: (d) untreated representative was more susceptible to fungal attack, (e) silicone oil heat treated samples exposed to white-rot fungus according to GB/T 13942.1-2009 standard.

The radial section morphologies of untreated and treated wood were observed using a scanning electron microscope (SEM) (Fig. 8). The natural porous structure of the untreated sample and the large vessels were easily observed (Fig. 8a) and there was a slight shrinkage and deformation in the cell wall. For the treated wood samples in Figs. 8b and c, the volume of void spaces in the samples was considerably reduced after heat treatment with silicone oil, indicating that the silicone oil used for the treatment of the

wood penetrated the cell walls of the wood and adhered to the vessels as evidenced by SEM images. There was an even distribution of silicone oil in the internal structure of the treated wood which protected the microstructure of the wood with a relatively flat, smooth surface and clear cell wall. It can be observed, that the microstructure of the treated wood remained relatively stable, with a high degree of surface smoothness. The natural pores of the wood structure were filled with silicone oil, thus conferring resistance against fungal attack in the treated wood, which reduced weight loss as reported in this study. Furthermore, Silicone oil was able to act as a physical barrier (seal) with a biocidal effect, these explain the reduction in weight loss of the treated wood at 150^{o}C and 210^{o}C compared to the untreated. Furthermore, the arrows indicate that most vessels in the treated wood were filled with silicone oil when the samples were treated at 150^{o}C and 210^{o}C respectively (Figs. 8b and c). Silicone oil deposited in the large vessels formed a uniform film covering the walls, aggregating in wood lumens (Fig. 8c). Improvement in the durability of the wood could be attributed to the blocking effect of the silicone oil after heat treatment. Earlier research reported that most impregnations undergo polymerisation of monomer within the wood cell walls that may cause a certain level of swelling of the wood substrate (Deka and Saikia 2000).

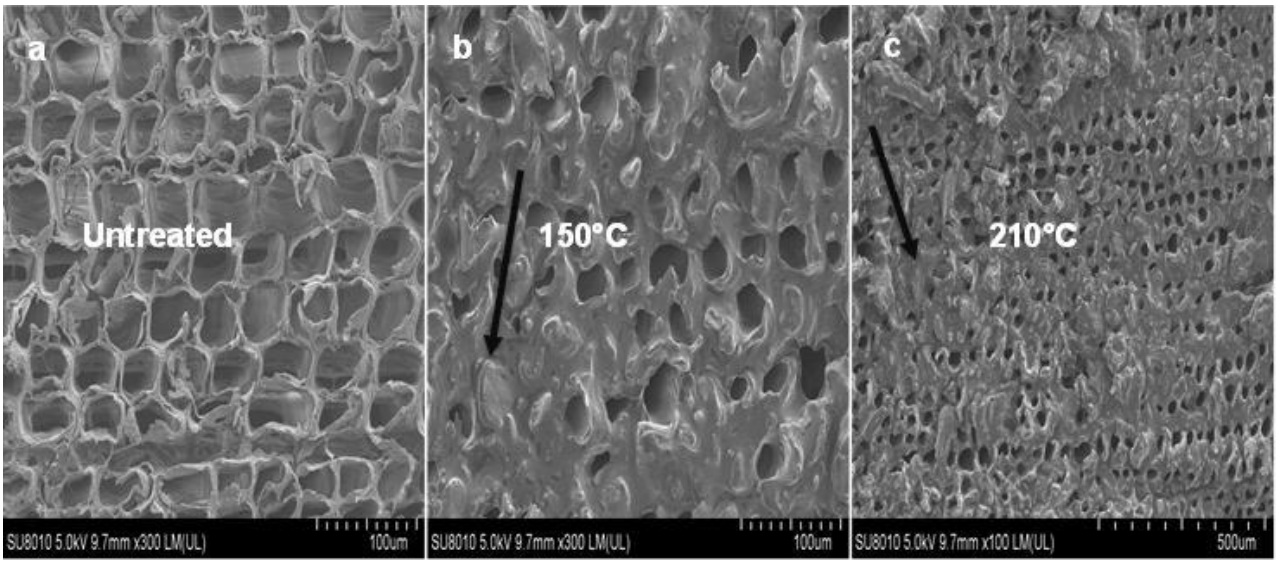

Fig. 8.
Radial section scanning electron micrograph (SEM) images: (a) untreated, (b) silicone oil treated at 150^{o}C/ 2h, (c) silicone oil treated at 210^{o}C/ 8h.

CONCLUSION

This study showed that the mass loss of all silicone oil heat treated fast-growing pine wood significantly decreased compared with the untreated samples, and the mass loss generally decreased with increasing heat treatment temperature. After exposure to both fungi, the lowest mass loss was recorded at 210^{o}C for 8h, 6.94% for white-rot and 7.03% for brown-rot fungi. The attack by the white-rot fungus was more virulent than the brown-rot fungus, silicone oil modified wood at 210^{o}C was classified as very durable (decay-resistant class I) against the white-rot and brown-rot fungi. FTIR spectra of the silicone oil treated wood showed slight changes, indicating modification of the chemical components such as cellulose, hemicellulose and lignin contents, which hinders fungal growth and makes the treated wood highly durable. XRD indicated a noticeable increase in the crystallinity of the silicone oil treated wood after exposure to decay fungal, thus enhancing the decay resistance of the wood. The impact of the treatment did not damage the wood's anatomical structure and there were no signs of cracks or distortion to the wood vessels due to silicone oil heat treatment of the wood at 150^{o}C and 210^{o}C for 8h. The SEM results revealed that silicone oil penetrated the wood cell walls and occupied the wood voids, thus improving the decay resistance of the treated wood against decay fungi. Improvement in the durability of the treated wood is attributed to the blocking effect of the silicone oil acting as a biocide against fungal growth.

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