

Properties of Oak Wood Incorporating Microencapsulated Phase Change Material

Ahmet Can,^{a,*} Mehmet Emin Ergun,^{a,b,*} and İsmail Özlüsoylu^a

Microencapsulated phase change materials (MPCMs) incorporated into oak wood *via* vacuum impregnation have shown promise as thermal energy storage (TES) materials. Physical and chemical properties of MPCMs and resulting Phase Change Energy Storage Wood (PCESW) were analyzed. Scanning electron microscopy and particle size analyses revealed similar particle sizes, while X-ray diffraction (XRD) and Fourier transform infrared spectra confirmed crystal phase and chemical structure. Thermal gravimetric analysis (TGA) and differential scanning calorimetry determined thermal properties, including phase change temperature, enthalpy, thermal stability, and conductivity. The MPCMs exhibited a phase change enthalpy of 146.0 J/g and temperature of 35.0 °C, with excellent thermal stability. The FTIR, XRD, and TGA analyses showed unchanged chemical structure, crystallinity ratios, and decomposition in two stages, respectively. The PCESW exhibited a latent heat storage of 3.02 J/g at 25.4 °C. Decay tests demonstrated noticeably reduced weight loss (1.22% and 1.55%) for MPCMW samples treated with *Trametes versicolor* and *Coniophora puteana*, compared to unleached control samples (19.7% and 20.8%). These findings indicate the high efficiency and potential of PCESW as a thermal energy storage material.

DOI: 10.15376/biores.18.3.6068-6085

Keywords: Oak wood; Microencapsulated phase change materials; Impregnation; Energy storage

Contact information: a: Department of Forest Industry Engineering, Bartın University, 74100 Bartın, Turkey; b: Department of Forestry, Akseki Vocational School Alaaddin Keykubat University, 07630 Antalya, Turkey; *Corresponding author: acan@bartin.edu.tr; mehmet.ergun@alanya.edu.tr

INTRODUCTION

The demand for energy continues to rise due to the growth in population and the rapid development of technology. About 40% of the energy consumed is used for buildings, and people need to heat or cool the environment where they live to ensure their thermal comfort. The use of insulation materials in buildings saves energy by decreasing the need for ventilation, heating, and air conditioning systems, which reduces the use of natural resources for energy supply. Energy storage systems are necessary to provide the necessary thermal comfort for a longer time by consuming less energy. Phase-changing materials (PCMs) are substances that are capable of storing and releasing large amounts of energy as they change between solid and liquid states. When incorporated into wood, PCMs can help to regulate the temperature and moisture content of the wood, which can improve its dimensional stability, decay resistance, and durability. The microencapsulation process is used to prevent unwanted infiltration problems and volume changes that occur during the use of phase-changing materials and to extend the service life (Cao *et al.* 2017). Microencapsulated phase change materials (MPCM) can be employed in many fields

because of their capacity to absorb and release energy when it is necessary (Giro-Paloma *et al.* 2016). The use of MPCM in Portland cement concrete (PCC) and geopolymer concrete (GPC) resulted in lower thermal conductivity and higher thermal energy storage, while the specific heat capacity of concrete remained practically stable (Cao *et al.* 2017). The integration of PCMs into building fabrics has been investigated as a potential technology for minimizing energy consumption in buildings. The use of phase change materials in building products can store significant amounts of thermal energy without large structural mass associated with sensible heat storage (Li and Fang 2010).

Microencapsulation is a preservation and release technique that involves the encapsulation of different components within a shell or coating of microscopic size. This process involves coating small particles, liquids, or gases with a layer of coating or matrix. Microencapsulation typically refers to the encapsulation of particles that range in size from 100 nanometers to 1 mm. The purpose of microencapsulation can vary, but some of the most common goals include protecting the encapsulated material from degradation or external factors, modifying the release properties of the encapsulated material, or controlling the interaction of the encapsulated material with its environment. The applications of microencapsulation can be found in various fields, including food and beverage, pharmaceuticals, cosmetics, and materials science (Sobel *et al.* 2014). The shell material is usually insoluble and unreactive towards the core, constituting approximately 80% of the microcapsule's weight. Microencapsulation shells are commonly composed of a variety of materials, including proteins, gums, sugars, natural and modified polysaccharides, lipids, synthetic polymers, or waxes (Gibbs *et al.* 1999). Microencapsulation is widely applied in various fields such as the pharmaceutical industry, perfume, coating, and pesticide. Recently, microencapsulation has been used for different purposes on wood-based materials. The microencapsulation technique is used in wood-plastic composites (Wang *et al.* 2017), in solid wood (Can *et al.* 2021), in coatings (Han *et al.* 2022), and on plates (Lubis *et al.* 2020). In studies carried out, very different substances, such as ammonium polyphosphate (Wang *et al.* 2016), urea (Liu *et al.* 2021), wood particle (Ye *et al.* 2018), and neem tree (*Azadirachta indica* A. Juss) extract have been used (Liu and Xu 2019) as the core material in microencapsulation. It has been determined that the antifungal properties of wood-based materials are increased (Miri Tari *et al.* 2022), mechanical properties are improved (Pan *et al.* 2022), their resistance to combustion is increased (Wang *et al.* 2016), the release of free formaldehyde is decreased (Duan *et al.* 2015), and aging resistance is increased (Huang *et al.* 2022) in various cases.

When MPCM are impregnated into wood, the resulting material can possess improved thermal insulation properties. The MPCM-impregnated wood can absorb and store heat energy in the form of latent heat during a phase change and release it later when the temperature drops. This can help maintain a stable temperature inside a building, reducing the need for heating and cooling. There are different methods for impregnating MPCM into wood, such as vacuum impregnation or pressure impregnation. The choice of method will depend on various factors, including the desired properties of the final product, the amount and distribution of MPCM within the wood, and the cost and feasibility of the process. The MPCM-impregnated wood possesses unique properties compared to traditional wood. It has increased thermal conductivity, lower thermal diffusivity, increased thermal storage capacity, reduced heat loss, and improved thermal insulation. Such properties make it suitable for use in construction and building, especially in passive solar systems, where it can save energy and costs.

The study investigated the potential benefits of using MPCMs to improve the properties of oak wood. The specific Thermoball-28 used in the study was impregnated into oak wood using the vacuum method. The results of the study showed that the incorporation of microencapsulated PCMs into oak wood improved its dimensional stability, decay resistance, and durability. The study also found that the use of microencapsulated PCMs was more effective than other wood preservation methods, such as impregnation with boron compounds or heat treatment. Therefore, the results of the study provide valuable insight into the potential benefits of using microencapsulated PCMs to improve the properties of oak wood and how they compare to other wood preservation methods.

EXPERIMENTAL

Materials

For the purpose of this study, oak (*Quercus robur*) was purchased from Sülekler Forest Products (Bursa/Turkey). Sapwood of oak was used as the matrix material and its average density was 0.652 g/cm³. The MPCM emulsion (solid content: 38.8%) was provided by Insilico in South Korea. Based on the information published by the company, the wall material of MPCM was formed by polyoxymethylenemelamine. Styrene maleic anhydride monomethyl-maleate polymer was used as the surfactant. The specifications of the MPCM are presented in Table 1.

Table 1. Some Specifications of the Used MPCM (Thermoball 28)

Thermoball 28	
pH	6.05
Mean particle size	4.27 µm
Specific gravity	0.994
Viscosity (24°C)	187.8 cPs
Heat capacity	122.55 J/g
Appearance	White slurry

The Thermoball 28 MicroPCM emulsions were used to impregnate the oak wood specimens through the vacuum method. To prepare the impregnation emulsions, the purchased MicroPCM emulsion was utilized. Each group consisted of ten wood samples, which were placed in a beaker and 500 mL of MPCM emulsion was added. The oak wood was subjected to a vacuum for 30 min to eliminate the gas and ethanol inside the wood, allowing the solution to fill the internal space of the wood once the vacuum was released. Afterward, the impregnated wood samples were cleaned and left to dry for 96 h at 30 °C.

Characterization of the MPCM and MPCM Impregnated Wood (MPCMW)

Particle size analysis was performed using a Malvern Panalytical Mastersizer 3000 equipment that uses laser diffraction technology. A Fourier transform infrared (FTIR) spectrometer was used to make measurements for the chemical structure studies of MPCM, wood, and MPCMW. Solid wood samples were used for the measurements, which were performed using 32 scans with a resolution of 4 cm⁻¹ and a wavelength range of 700 to 4000 cm⁻¹. A Carl Zeiss Gemini 500 scanning electron microscope was used to take measurements of the morphology of the wood samples. Prior to measurement, gold was

applied to the wood samples. Differential scanning calorimetry (DSC) analysis was used to ascertain the thermal characteristics of wood samples. For the measurements, a Pyris 1 DSC, Perkin-Elmer instrument was employed. The experiments were performed in an atmosphere of argon gas at temperatures between 10 and 90 °C with a 5 °C/min cooling and heating rate.

The efficiency of heat utilization by MPCM on modified wood can be evaluated using the coefficient of thermal efficiency X%, which is determined for both the melting ($X_m\%$) and crystallization ($X_c\%$) processes using Eq. 1,

$$Xm/c\% = \frac{\Delta H(MPCMW)}{\Delta H(MPCM)} * 100\% \quad (1)$$

where $\Delta H(MPCMW)$ is the enthalpy of melting/crystallizing of MPCM on a modified wood and $\Delta H(MPCM)$ is the enthalpy of melting/crystallizing of MPCM powder.

To evaluate the thermal stability of the samples, a PerkinElmer Pyris™ 1 thermogravimetric analyzer was employed. The analysis was conducted under a nitrogen atmosphere, in the temperature range of 20 to 600 °C, at a scanning rate of 10 °C min⁻¹. The crystal structure of the microcapsules was discovered using X-ray diffraction (XRD) investigation. CuK irradiation ($\lambda = 1.5405 \text{ \AA}$) at a rate of 1 °/min was used to perform analyses in the range of 10 to 70 2 θ° .

Statistical Analysis

A descriptive analysis was developed (mean and standard deviation) for water absorption and decay test. ANOVA was applied to verify the effect of treatment with the silver nanoparticles. Duncan's test was set at 99% confidence level to determine the statistical difference between the means.

RESULTS AND DISCUSSION

Characterization of MicroPCM

The morphology, particle size distribution, FTIR, XRD, DSC, and thermal gravimetric analysis (TGA) curves of the produced MicroPCMs are given in Fig. 1. Figure 1a displays the morphology of MPCM, which exhibited mostly spherical shapes with smooth surfaces. The diameter of the MPCM particles ranged from 0.0 to 20.0 μm , with an average diameter of 2.5 μm . These results suggest that MPCM particles are small enough to penetrate the wood vessels, as depicted in Fig. 1b. The chemical structure of MPCM, measured by FTIR, showed that there was no chemical bond between core material and the shell material, but rather a physical bond. Various methods for impregnating MPCM into wood include vacuum infusion of the polymer material used, which is also observed in the capsule material. As can be seen in the XRD result in Fig. 1d, the MPCM characteristic peak appeared at 19.21°. The MPCM displayed distinct endothermic and exothermic behavior during temperature changes, as evidenced by the DSC and TG curves depicted in Fig. 1e and f. The melting process of MPCM began at 20 °C and ended at 35 °C, while the freezing process started at 10 °C and ended at 22.9 °C. These results suggest that the phase change temperature range of MPCM is suitable for maintaining human body comfort. Additionally, the enthalpies of melting and freezing processes were measured as 146.0 and 147.6 J g⁻¹, respectively, which indicates exceptionally high energy storage capacity.

The TG and DTG curves of MPCM in Fig. 1f demonstrate a considerable reduction in weight between 200 and 410 °C, indicating the decomposition of the MPCM core material. Additionally, the DTG curve shows a minor weight loss stage at 400 to 410 °C, which is associated with the decomposition of the wall materials. Moreover, the MPCM showed excellent thermal stability at room temperature with only a 4.1% weight loss at temperatures below 100 °C.

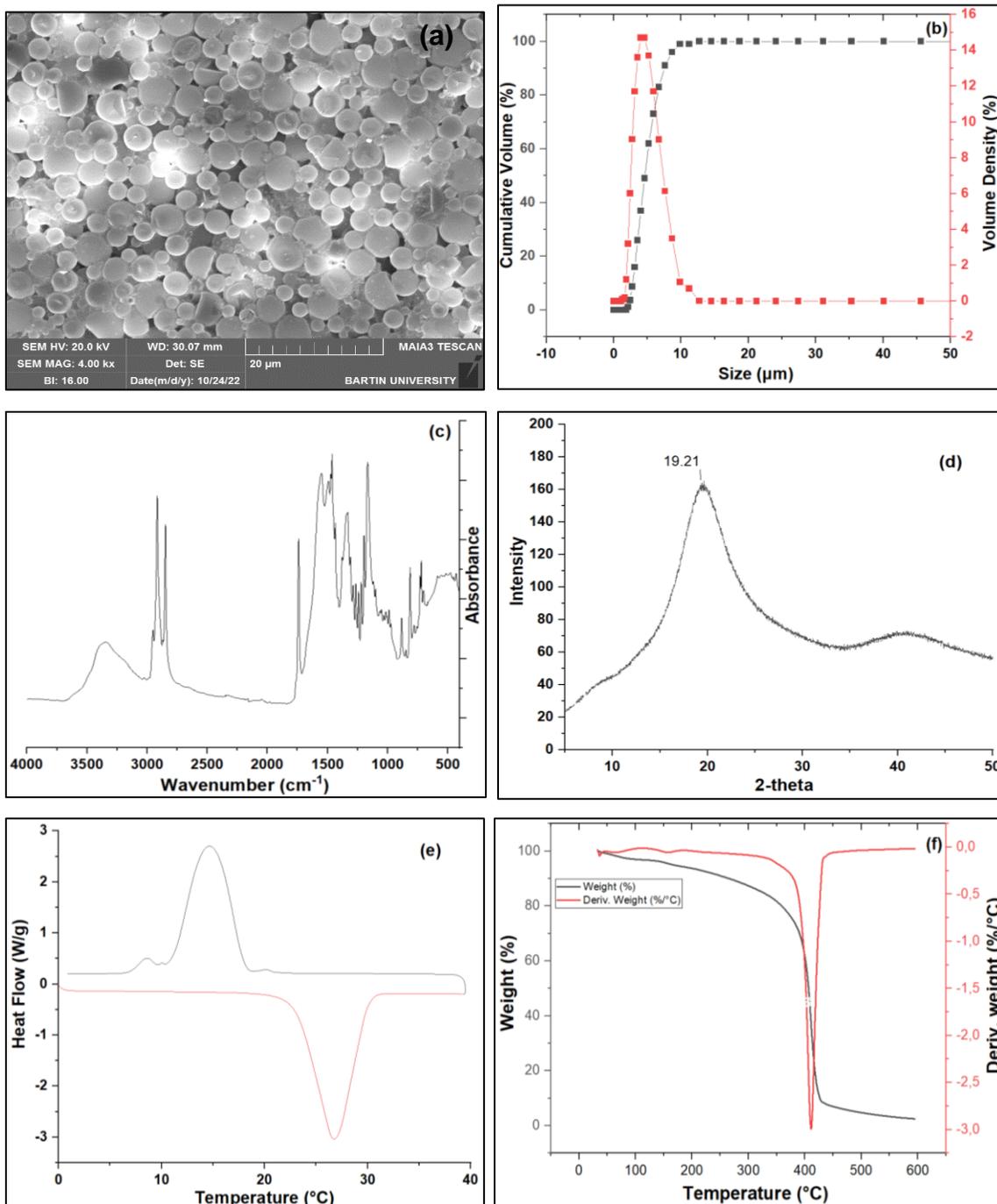


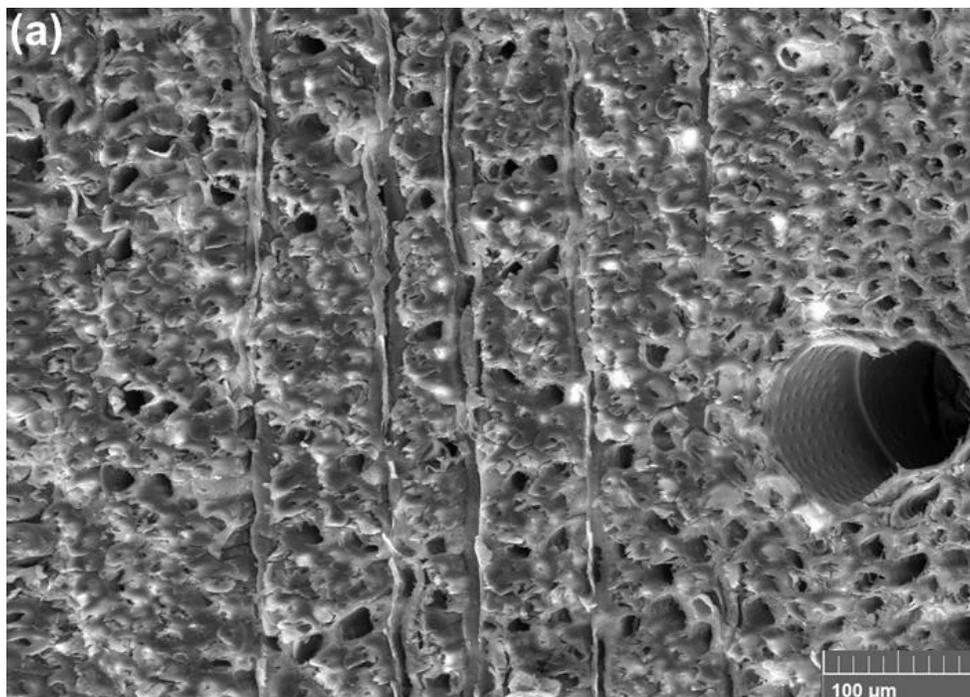
Fig. 1. Morphology (a), particle size distribution (b), FTIR (c), DSC (d), XRD (e), and TGA curves (f) of MicroPCM

Characterization of PCMW

The samples of oak wood impregnated with MicroPCM and untreated are shown in Fig. 2. The distribution of MicroPCM (MPCM) in wood can have a noticeable impact on its thermal insulation properties. There are a few different methods that can be used to distribute the MPCM within the wood, each with their own advantages and disadvantages. One method is to use vacuum impregnation to introduce the MPCM into the wood.

In this method, the wood is placed in a sealed chamber and a vacuum is applied to the chamber. The MPCM is then introduced into the chamber, and it is drawn into the wood due to the pressure differential between the inside and outside of the wood cells. The choice of distribution method will depend on the desired properties of the final product, such as the amount and distribution of the MPCM within the wood, as well as the cost and feasibility of the process. The method of vacuum impregnation tends to produce more homogenous and consistent distribution of the MPCM in the wood, while the pressure impregnation tends to produce more concentrated MPCM in the outer layers of the wood.

The thermal energy storage properties of MicroPCM, wood, and PCMW were analyzed using DSC analysis to determine their freezing temperature, melting temperature, and latent heat storage capacity. Figure 3 shows the DSC thermograms of these materials, while Table 2 presents the stored and released energy during their respective phase transitions.



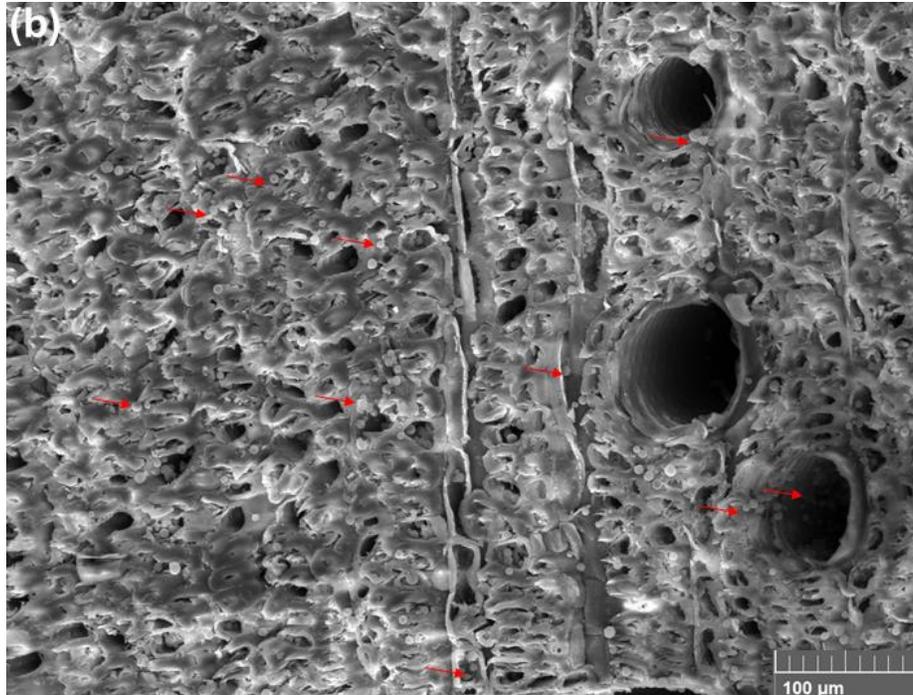


Fig. 2. SEM images of MicroPCM in wood

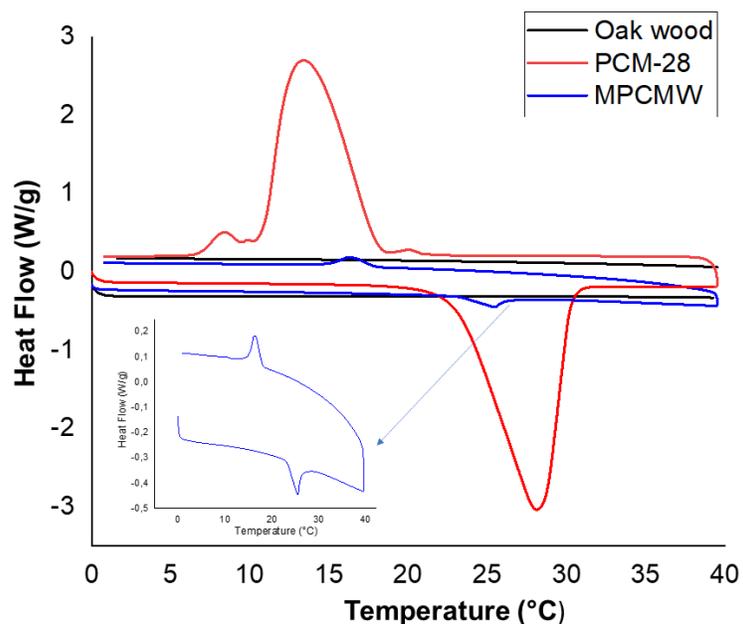


Fig. 3. Melting and solidifying DSC curves of the Oak wood, PCM-28, and MPCMW

The melting and solidifying temperatures (initial temperatures) were 23.1 and 18.0 °C for MicroPCM, and 23.2 and 18.0 °C for PCMW, respectively. It was observed that on the solidifying curve, the phase change temperature shifted by 14.7 °C for MicroPCM and 16.4 °C for PCMW. The observed shift in initial temperatures suggests the presence of supercooling, which may be more pronounced in smaller DSC samples. However, impregnating the wood with PCM *via* microencapsulation decreased the supercooling effect by 1.8 °C. The latent melting temperatures calculated based on the area below the

peak on the DSC curve were 146 J/g for MicroPCM and 3.0 J/g for PCMW. The latent solidification temperatures were 148 J/g for MicroPCM and 4.13 J/g for PCMW. It was determined that more hidden energy could be stored by MicroPCM impregnation to the wood.

Table 2. DSC Data of the PCM-28, MPCMW, and Coefficient of Thermal Efficiency of Melting (X_m) and Crystallization (X_c)

	Melting			Solidifying			X_m	X_c
	Onset Temperature (°C)	Peak Temperature (°C)	Latent Heat	Onset Temperature (°C)	Peak Temperature (°C)	Latent Heat		
PCM	23.07	26.73	146.0	18.02	14.67	147.57		
PCMW	23.21	25.40	3.02	17.97	16.42	4.13	2.81	2.07

Microencapsulated PCM has been used together with different building materials such as plaster (Gencil *et al.* 2022), wood (Wang *et al.* 2020), concrete (Jayalath *et al.* 2016), and polyurethane sandwich panels (Naikwadi *et al.* 2021). It was found that by the impregnation of microencapsulated PCM into maple wood, the energy storage capacity increased by 7.1% (Mathis *et al.* 2018). In contrast, in a wood-based floor application, with the addition of PCM to the microcapsule by 5%, the latent heating and cooling temperatures increased similarly to the current study, and these temperatures were measured as 3.28 and 3.23 J/g, respectively (Jeong *et al.* 2012). The obtained results show that applying MicroPCM to wood provides better thermal regulation, and leads to the absorption and release of more energy during the melting and solidification process. Overall, the heat storage capacity of wood can vary depending on the specific characteristics of the wood and the conditions under which it is stored, but it generally increases as the density, thermal conductivity, and moisture content decreases. The thermal energy storage capability of wood can be improved by delignifying the wood and using different materials for microencapsulation, as shown in different studies (Lin *et al.* 2022, 2023).

The TG and DTG curves of the oak wood, PCM-28, and MPCMW are given in Fig. 4a and 4b, respectively.

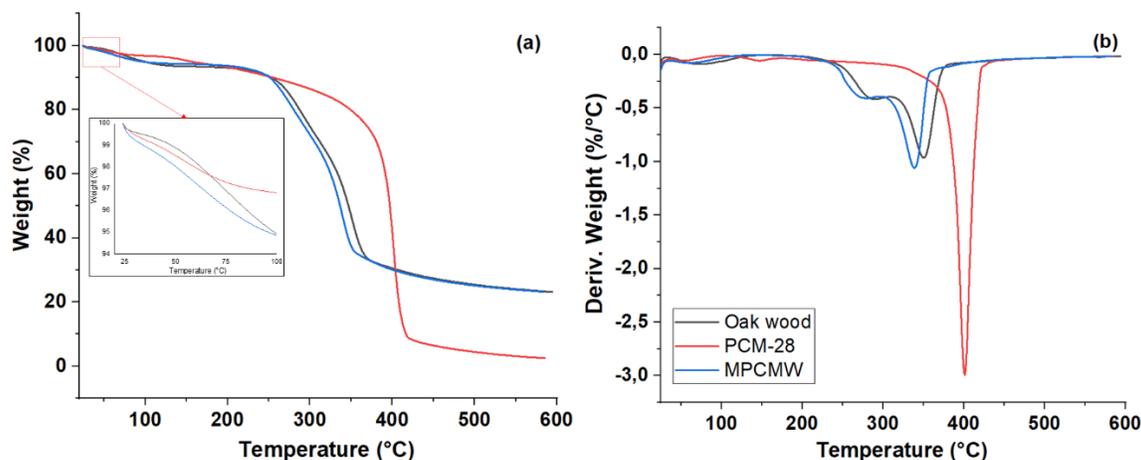


Fig. 4. TGA (a) and DTG (b) curves of the oak wood, PCM-28, and MPCMW

It can be seen that the degradations of wood and PCM-28 exhibited only one step between 100 to 350 °C, and 200 to 410 °C, respectively. The thermal decomposition of MPCMW took place in two steps. In the first step, the TGA thermograms indicate that MPCMW first showed weight losses between 200 to 280 °C. Second, the MPCMW showed decompositions between 280 to 340 °C. It is clear that the thermal resistant temperatures of PCM-28 were much higher than those of the oak wood and MPCMW. Melamine modification can indeed improve the heat resistance of microcapsules. This is because melamine, a nitrogen-rich organic compound with a triazine ring, can form strong and stable bonds with the polymer chains that make up the microcapsule shell. These bonds increase the rigidity of the polymer chains and decrease their mobility, making the shell more resistant to thermal motion and heat. The triazine ring in melamine has a high degree of symmetry and a planar structure, which enhances its ability to interact with the polymer chains and reinforce them. Additionally, the nitrogen atoms in the triazine ring have lone pairs of electrons that can form hydrogen bonds with the polymer chains, further strengthening the interaction between the two.

The XRD analyses were performed to examine the effect of PCM, wood, and PCMW on crystal structures, and the obtained diffraction patterns are shown in Fig. 5. Crystallinity is a measure of the degree to which the cellulose molecules in wood are organized into ordered, crystalline structures. In general, wood species with higher crystallinity have higher mechanical properties, such as greater strength and stiffness, than those with lower crystallinity. Oak wood is known for its high crystallinity. The cellulose molecules in oak wood are highly ordered, with a high degree of molecular alignment and regular packing, which gives it its characteristic strength and durability. The cellulose in oak wood is made up of long chains of glucose molecules that are linked together in a regular and orderly pattern, giving it a high degree of crystallinity. The crystallinity of oak wood can vary depending on the specific conditions under which the tree grew, such as the species of oak, the growing conditions, and the age of the tree. Oak wood that is grown in a slow-growth environment or under stress, such as drought, tend to have a higher crystallinity compared to specimens from wood grown in ideal conditions (Gawron *et al.* 2012). The crystallinity of oak wood can be measured using a number of techniques, such as X-ray diffraction, infrared spectroscopy, and solid-state NMR spectroscopy. These techniques can provide information about the degree of crystallinity and the organization of the cellulose molecules within the wood. In summary, oak wood is known for its high crystallinity, giving it strong and durable properties. Crystallinity can vary depending on the specific conditions under which the tree grew, but it is generally considered a high crystalline wood (Geffert *et al.* 2019; Kubovský *et al.* 2020).

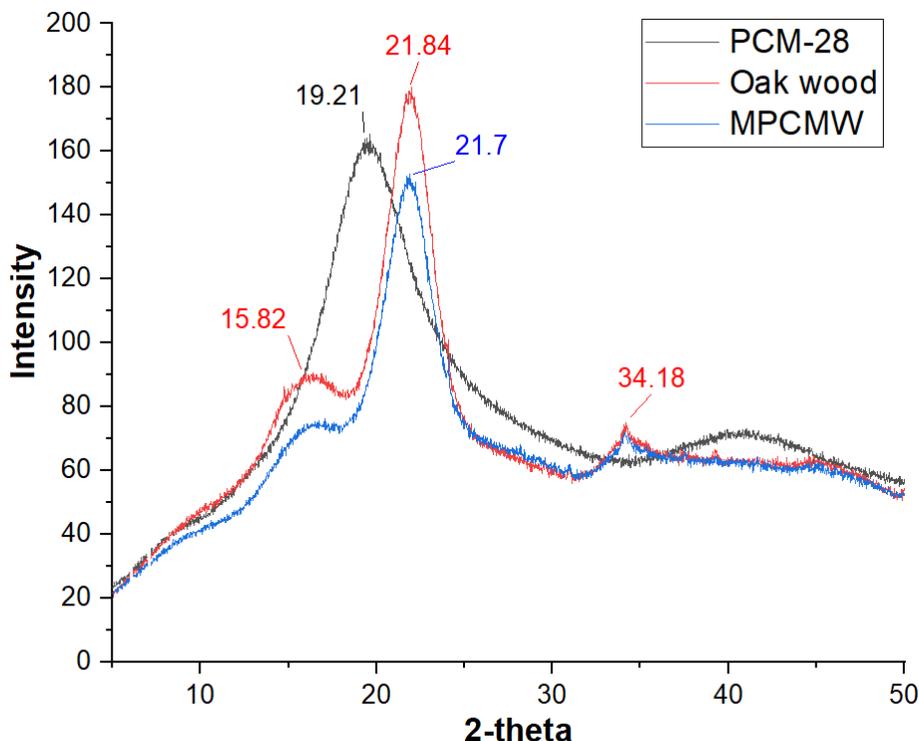


Fig. 5. XRD curves of the oak wood, PCM-28, and MPCMW

The cellulose on the wall of oak wood had a high-density diffraction band at 2θ equal to 21.84° , while it had low-density diffraction bands at 15.82° and 34.18° ; this revealed a typical diffraction graph. The obtained peaks showed the expected crystal structure of cellulose in oak wood. It was observed that the XRD results of different studies conducted with oak wood (Broda and Popescu 2019) were similar to the XRD results of the current study. It was determined that the MPCMW peaks (2θ) were 15.82° , 21.7° , and 34.18° . In contrast, it was determined that PCM-impregnated wood had all the peaks found in non-impregnated wood. In addition, the 2θ data showed that after impregnation of oak wood with PCM, there was no change, although the peak densities decreased slightly. These findings showed that PCM provided physical adhesion to the wood with its impregnation or did not cause any chemical changes in the structure of the wood. Sari *et al.* (2022) stated that there was a decrease in the XRD peaks obtained in the study they conducted (Sari *et al.* 2022). The similar study showed that prepared and characterized sodium acetate trihydrate/ethylene glycol (SAT/EG) composite PCM using XRD. The XRD patterns of the composite PCM showed no peaks disappearing or newly emerging, but only lower peak intensities than those of pure EG and pure SAT. They explained that this was due to the low loading of SAT in the composite PCM (10 wt%), which made the diffraction peaks of SAT less obvious in the XRD patterns. They also suggested that increasing the loading of SAT would enhance the peak intensities of SAT in the composite PCM (Gu *et al.* 2016).

The FTIR spectra of the control wood, PCM, and PCMW are shown in Fig. 6. When looking at the FTIR peaks of the control wood, it can be said that the peak around 3400 cm^{-1} indicates the presence of OH groups in the wood. C-H stresses are observed around 2900 cm^{-1} . Peaks between 1600 and 1800 cm^{-1} indicate the presence of xylan, unconjugated hemicellulose, and conjugated lignin C=O groups (Can and Žigon 2022). The C-H

deformation peaks in lignin and carbohydrates are observed between 1400 and 1300 cm^{-1} . Peaks between 1300 and 700 cm^{-1} indicate syringyl rings, C-O vibrations caused by syringyl derivatives, C-H and C-O-C vibrations in cellulose, and C-O vibrations in cellulose and hemicellulose (Ghavidel *et al.* 2021).

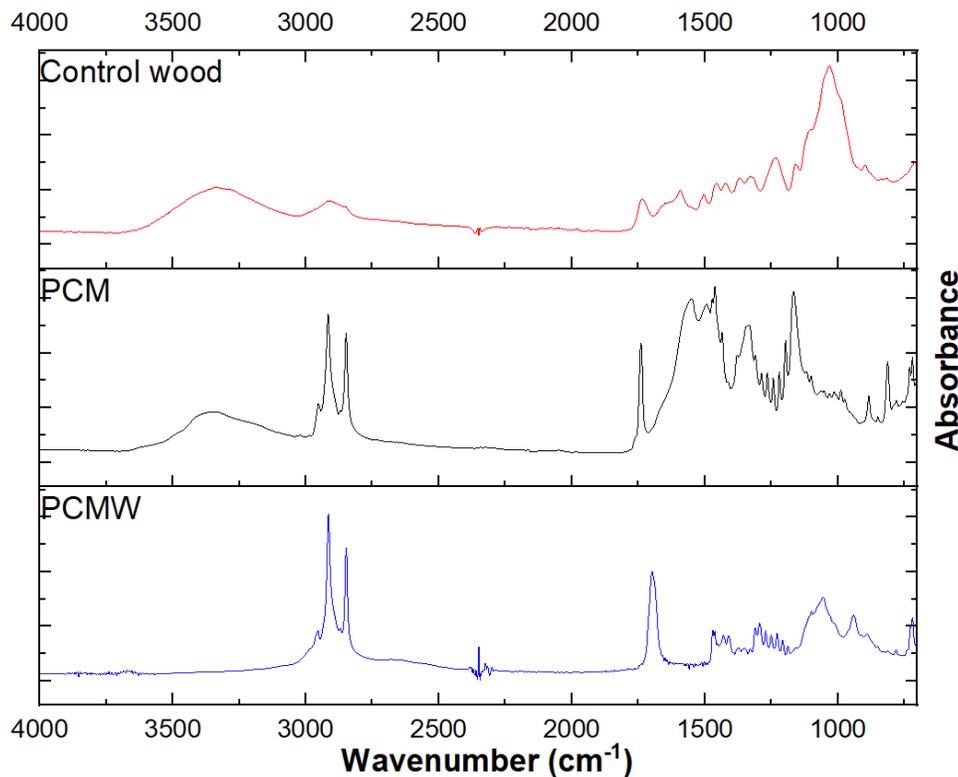


Fig. 6. FTIR spectra recorded for the oak wood (control), PCM-28, and PCMW samples

Impregnating oak wood samples with PCM caused alterations in their chemical structure. The chemical structure of the PCM is reflected in the wood samples, with peaks detected at 2916 and 2849 cm^{-1} , particularly in the wood samples. Due to the aldehyde content in PCM, 1739 cm^{-1} C=O stress was observed (Vilà Ramirez *et al.* 2009); a similar peak was also observed in the PCMW sample. In addition, peaks originating from oak wood were seen around 1700 cm^{-1} , while the peak at 1739 cm^{-1} was observed to expand in PCMW. Additionally, it was observed that a significant decrease occurred in the intensity of the 1462 and 1165 cm^{-1} peaks, which are also powerful in PCM, after impregnation to oak wood. Figure 7 shows the water absorption (%) and tangential swelling (%) values of MicroPCM impregnated by oak wood between 20 min and 96 h of soaking time.

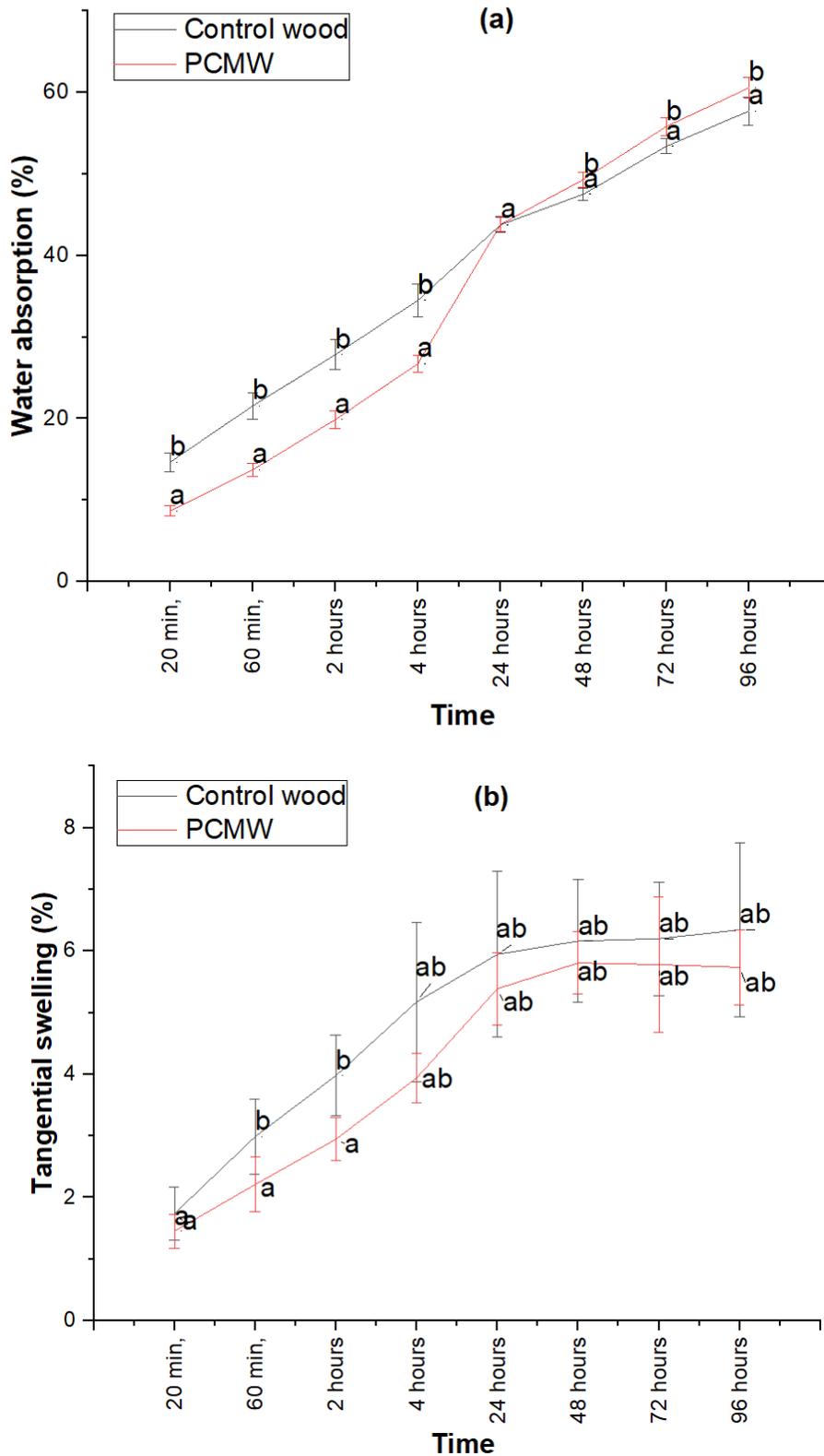


Fig. 7. Water absorption (%) (a) and tangential swelling (%) (b) of wood and MPCMW

It is observed that the water absorption amount of oak wood impregnated with MPCMW was less in the first 24 h compared to oak wood not impregnated with MPCMW. In

addition, samples impregnated with PCM absorbed water higher than non-impregnated wood at the end of 96 h. It has been stated that this may be because the control sample has a higher moisture content because of the hysteresis event occurring in the wood (Mathis *et al.* 2018). The water absorption of oak wood impregnated with PCMW was lower than that of non-impregnated wood in the first 24 h, but higher at the end of 96 h. This may be explained by the fact that the PCM filled the wood pores and reduced the initial water uptake, but it also increased the moisture content of the wood due to its hygroscopic nature (Temiz *et al.* 2020). The PCM also prevented the wood from drying out quickly, resulting in a higher water absorption at the end of the test period.

As the moisture content of the wood increases, the relative humidity also increases. However, when the moisture content decreases, the relative humidity does not decrease. Therefore, different moisture contents can be obtained at the same relative humidity (Mendis *et al.* 2023). The hysteresis event can affect the water absorption capacity of the wood. Maybe the control sample has a higher moisture content due to a hysteresis effect in wood samples impregnated with PCM (Can *et al.* 2023). A similar trend was observed by Nazari *et al.* who impregnated pine wood with octadecane as PCM and found that the impregnated samples had lower water absorption than the untreated samples after 6 hours, but higher after 96 hours. They attributed this to the hysteresis effect of wood and the leakage of PCM during phase change (Nazari *et al.* 2022). The mean WA values of the control and test groups at different times were examined statistically, it was determined that there was a significant difference ($p < 0.05$) for all periods except 24 h for water absorption.

Wood samples treated with MicroPCM exhibited a decrease in swelling compared to untreated samples. MicroPCM is insoluble in water and effectively resists leakage, limiting water absorption by accumulating within the wood cells and lumen. The interaction between MicroPCM and wood can hinder the formation of hydrogen bonds between water and wood molecules, ultimately reducing water absorption (Xu *et al.* 2020). Additionally, MicroPCM can prevent prolonged water-wood contact, which can lead to higher levels of swelling (Amini *et al.* 2022).

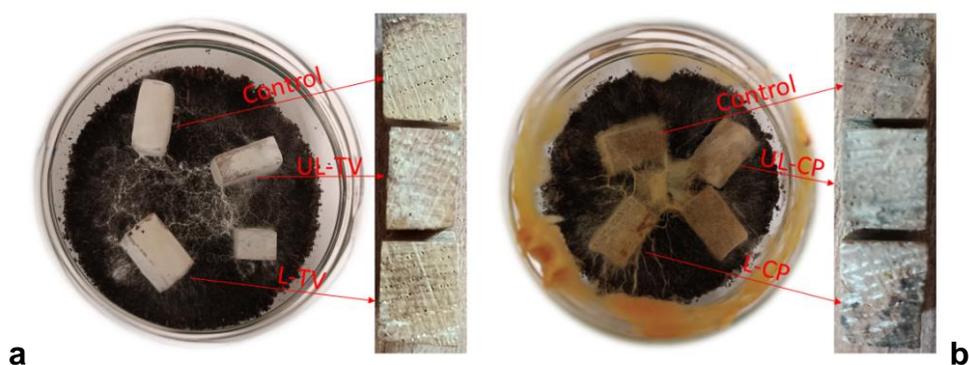


Fig. 8. Samples after decay test: *Trametes versicolor* (a) and *Coniophora puteana* (b) (UL: Unleached samples, L: leached samples)

As shown in Fig. 8, the control and leached samples were completely covered by fungi, and the lumen spaces were visible in the cross-section of the wood in the leached samples. This indicates that the capsules moved away from the wood samples. Furthermore, deformations were observed in both the control and leached samples. In the

Duncan test results, the unleached samples were evaluated among themselves, and the leached samples were evaluated among themselves. When the tests samples were evaluated among themselves, it was seen that unleached samples were in the same homogeneity group. Likewise, the leached samples were within their own homogeneity group.

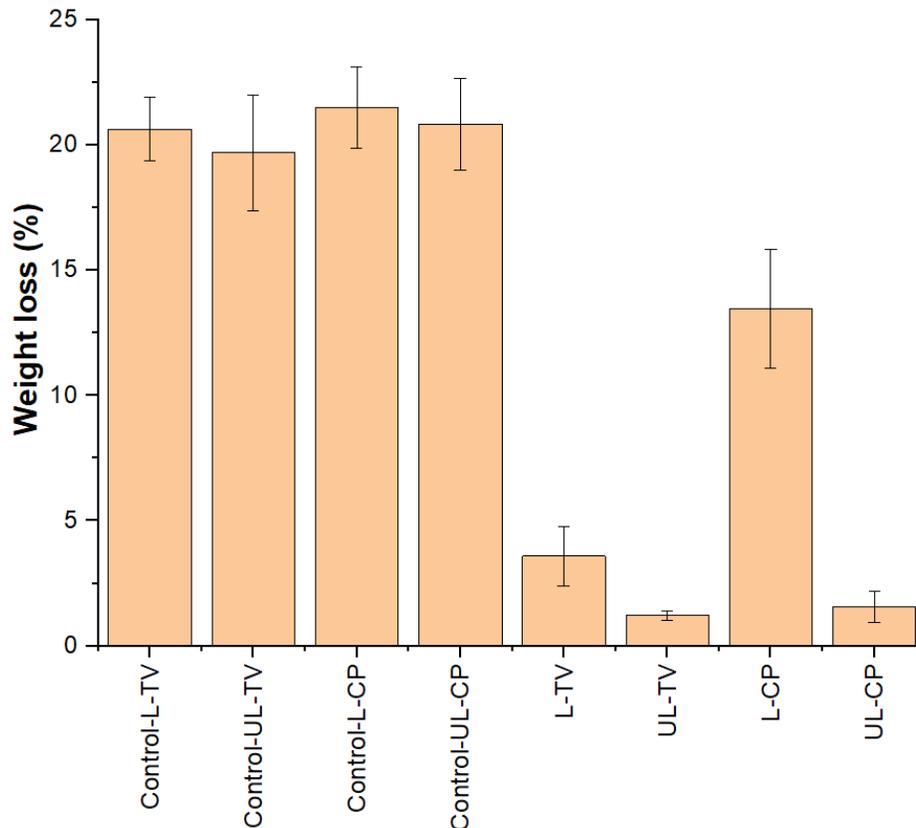


Fig. 9. Weight loss of wood samples after decay tests (^aThe letters indicate Duncan's homogeneity groups in the column)

The average weight losses (%) of samples are given in Fig. 9. The weight loss of the virulence controls subjected to decay fungi confirmed that the decay test was valid in accordance with EN 113 (1996) and that the testing environment was favorable for the growth of fungi. According to the EN 113 (1996) test, the average weight loss of the impregnated samples must be less than 3% of their initial dry weight for a possible wood preservation method to provide the appropriate decay resistance. Unleached samples exhibited less than 3% weight loss in both *T. versicolor* and *Coniophora puteana* fungus. Leached and unleached samples produced similar weight losses. Weight loss of 20% occurred in the samples exposed to the *T. versicolor* fungus, and around 21% in the samples exposed to the *C. puteana* fungus. Weight loss in the test samples showed a remarkable decrease compared to the control. Weight loss occurred 3.58%, and 1.22% in leached and unleached samples exposed to *T. versicolor* fungus, 13.44% and 1.55% in leached and unleached samples exposed to *C. puteana* fungus, respectively. Similar results were obtained (Can and Žigon 2022). Spruce (*Picea orientalis* (L.) Peterm.) sapwood samples

impregnated with 100% concentration of n-heptadecane obtained weight loss from 1% to 10% for white-rot fungus and from 1% to 8% for brown-rot fungus.

CONCLUSIONS

1. The differential scanning calorimetry (DSC) results demonstrated that the energy storage and release capabilities of the MicroPCM-impregnated wood were markedly improved compared to the control samples. Furthermore, the phase change temperatures were appropriate for building applications.
2. In scanning electron microscopy (SEM) images, it was observed that MicroPCM was distributed homogeneously in wood cells.
3. At the end of 600 °C, MicroPCM lost almost all its weight, while the weight loss of impregnated samples and control samples was approximately 75%.
4. As a result of the Fourier transform infrared (FTIR) and X-ray diffraction (XRD) analyses, it was determined that no changes occurred in the chemical structure and crystallinity ratios of PCMW.
5. Because MicroPCM reduced the contact of water with wood, the amount of tangential swelling of wood decreased.
6. The application of MicroPCM to oak wood has the potential to create a thermal-regulating building material.

REFERENCES CITED

- Amini, M. H. M., Temiz, A., Hekimoğlu, G., Demirel, G. K., and Sarı, A. (2022). "Properties of Scots pine wood impregnated with capric acid for potential energy saving building material," *Holzforschung* 76(8), 744–753. DOI: 10.1515/hf-2022-0007
- Broda, M., and Popescu, C.-M. (2019). "Natural decay of archaeological oak wood versus artificial degradation processes — An FT-IR spectroscopy and X-ray diffraction study," *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy* 209, 280-287. DOI: 10.1016/j.saa.2018.10.057
- Can, A., Erkan, G., Duran, H., and Sivrikaya, H. (2021). "Microencapsulated diammonium hydrogen phosphate (DAHP) with a polyurethane shell: Characterization and its properties in wood," *European Journal of Wood and Wood Products* 79(6), 1405–1417. DOI: 10.1007/s00107-021-01717-y
- Can, A., Lee, S. H., Antov, P., and Abd Ghani, M. A. (2023). "Phase-change-material-impregnated wood for potential energy-saving building materials," *Forests* 14(3), 514. DOI: 10.3390/f14030514
- Can, A., and Žigon, J. (2022). "n-Heptadecane-impregnated wood as a potential material for energy-saving buildings," *Forests* 13(12), 2137. DOI: 10.3390/f13122137
- Cao, V. D., Pilehvar, S., Salas-Bringas, C., Szczotok, A. M., Rodriguez, J. F., Carmona, M., Al-Manasir, N., and Kjøniksen, A.-L. (2017). "Microencapsulated phase change materials for enhancing the thermal performance of Portland cement concrete and

- geopolymer concrete for passive building applications,” *Energy Conversion and Management* 133, 56-66. DOI: 10.1016/j.enconman.2016.11.061
- Duan, H., Qiu, T., Guo, L., Ye, J., and Li, X. (2015). “The microcapsule-type formaldehyde scavenger: The preparation and the application in urea-formaldehyde adhesives,” *Journal of Hazardous Materials* 293, 46–53. DOI: 10.1016/j.jhazmat.2015.03.037
- Gawron, J., Szczęśna, M., Zielenkiewicz, T., and Gołofit, T. (2012). “Cellulose crystallinity index examination in oak wood originated from antique woodwork,” *Drewno: Prace Naukowe, Doniesienia, Komunikaty* 55(188), 109-114.
- Geffert, A., Výbohov, E., and Geffertov, J. (2019). “Changes in the chemical composition of oak wood due to steaming,” *Acta Facultatis Xylogologiae Zvolen* 61(1), 19-29. DOI: 10.17423/afx.2019.61.1.02
- Gencil, O., Hekimoglu, G., Sari, A., Ustaoglu, A., Subasi, S., Marasli, M., Erdogmus, E., and Memon, S. A. (2022). “Glass fiber reinforced gypsum composites with microencapsulated PCM as novel building thermal energy storage material,” *Construction and Building Materials* 340, article 127788. DOI: 10.1016/j.conbuildmat.2022.127788
- Ghavidel, A., Bak, M., Hofmann, T., Vasilache, V., and Sandu, I. (2021). “Evaluation of some wood-water relations and chemometric characteristics of recent oak and archaeological oak wood (*Quercus robur*) with archaeometric value,” *Journal of Cultural Heritage*, 51, 21-28. DOI: 10.1016/j.culher.2021.06.011
- Gibbs, B. F., Kermasha, S., Alli, I., and Mulligan, C. N. (1999). “Encapsulation in the food industry: A review,” *International Journal of Food Sciences and Nutrition* 50(3), 213-224. DOI: 10.1080/096374899101256
- Giro-Paloma, J., Martnez, M., Cabeza, L. F., and Fernandez, A. I. (2016). “Types, methods, techniques, and applications for microencapsulated phase change materials (MPCM): A review,” *Renewable and Sustainable Energy Reviews* 53, 1059-1075. DOI: 10.1016/j.rser.2015.09.040
- Gu, X., Qin, S., Wu, X., Li, Y., and Liu, Y. (2016). “Preparation and thermal characterization of sodium acetate trihydrate/expanded graphite composite phase change material,” *Journal of Thermal Analysis and Calorimetry* 125(2), 831-838. DOI: 10.1007/s10973-016-5444-4
- Han, Y., Yan, X., and Tao, Y. (2022). “Effect of number of impregnations of *Microberlinla* sp. with microcapsule emulsion on the performance of self-repairing coatings on wood surfaces,” *Coatings* 12(7), 989. DOI: 10.3390/coatings12070989
- Huang, N., Yan, X., and Han, Y. (2022). “Preparation of melamine-formaldehyde resin/rice husk powder coated epoxy resin microcapsules and effects of different microcapsule contents on the properties of waterborne coatings on *Tilia europaea* surface,” *Coatings* 12(8), article 1213. DOI: 10.3390/coatings12081213
- Jayalath, A., San Nicolas, R., Sofi, M., Shanks, R., Ngo, T., Aye, L., and Mendis, P. (2016). “Properties of cementitious mortar and concrete containing micro-encapsulated phase change materials,” *Construction and Building Materials* 120, 408-417. DOI: 10.1016/j.conbuildmat.2016.05.116
- Jeong, S.-G., Jeon, J., Seo, J., Lee, J.-H., and Kim, S. (2012). “Performance evaluation of the microencapsulated PCM for wood-based flooring application,” *Energy Conversion and Management*, IREC 2011, The International Renewable Energy Congress, 64, 516-521. DOI: 10.1016/j.enconman.2012.03.007

- Kubovský, I., Kačíková, D., and Kačík, F. (2020). "Structural changes of oak wood main components caused by thermal modification," *Polymers* 12(2), 485. DOI: 10.3390/polym12020485
- Li, H., and Fang, G.-Y. (2010). "Experimental investigation on the characteristics of polyethylene glycol/cement composites as thermal energy storage materials," *Chemical Engineering & Technology* 33(10), 1650-1654. DOI: 10.1002/ceat.201000102
- Lin, X., Chen, X., Weng, L., Hu, D., Qiu, C., Liu, P., Zhang, Y., Fan, M., Sun, W., and Guo, X. (2022). "In-situ copper ion reduction and micro encapsulation of wood-based composite PCM with effective anisotropic thermal conductivity and energy storage," *Solar Energy Materials and Solar Cells* 242, article 111762. DOI: 10.1016/j.solmat.2022.111762
- Lin, X., Qiu, C., Wang, K., Zhang, Y., Wan, C., Fan, M., Wu, Y., Sun, W., and Guo, X. (2023). "Biomimetic bone tissue structure: An ultrastrong thermal energy storage wood," *Chemical Engineering Journal* 457, article 141351. DOI: 10.1016/j.cej.2023.141351
- Liu, L., and Xu, G. (2019). "Preparation and stability of microcapsule wood preservative from neem extract," *BioResources* 14(2), 3352-3363.
- Liu, Y., Qin, X., Zhu, X., Wu, L., Xu, Y., Huang, K., Huang, J., and Chen, Y. (2021). "Microencapsulation of formaldehyde scavenger agent and its application to veneered panels," *European Journal of Wood and Wood Products* 79(3), 579-588. DOI: 10.1007/s00107-021-01656-8
- Lubis, M. A. R., Park, B.-D., and Lee, S.-M. (2020). "Microencapsulation of polymeric isocyanate for the modification of urea-formaldehyde resins," *International Journal of Adhesion and Adhesives* 100, article 102599. DOI: 10.1016/j.ijadhadh.2020.102599
- Mathis, D., Blanchet, P., Landry, V., and Lagièrre, P. (2018). "Impregnation of wood with microencapsulated bio-based phase change materials for high thermal mass engineered wood flooring," *Applied Sciences* 8(12), article 2696. DOI: 10.3390/app8122696
- Mendis, M. S., Ishani, P. A. U., and Halwatura, R. U. (2023). "Impacts of chemical modification of wood on water absorption: a review," *Journal of the Indian Academy of Wood Science* 20(1), 73-88. DOI: 10.1007/s13196-023-00309-y
- Miri Tari, S. M., Tarmian, A., and Azadfallah, M. (2022). "Improving fungal decay resistance of solvent and waterborne polyurethane-coated wood by free and microencapsulated thyme essential oil," *Journal of Coatings Technology and Research* 19(3), 959-966. DOI: 10.1007/s11998-021-00573-y
- Naikwadi, A. T., Samui, A. B., and Mahanwar, P. A. (2021). "Fabrication and experimental investigation of microencapsulated eutectic phase change material-integrated polyurethane sandwich tin panel composite for thermal energy storage in buildings," *International Journal of Energy Research* 45(15), 20783-20794. DOI: 10.1002/er.7138
- Nazari, M., Jebrane, M., and Terziev, N. (2022). "Solid wood impregnated with a bio-based phase change material for low temperature energy storage in building application," *Journal of Thermal Analysis and Calorimetry* 147(19), 10677-10692. DOI: 10.1007/s10973-022-11285-9

- Pan, P., Yan, X., and Zhao, W. (2022). "Effect of coating process of photochromic and thermochromic composite microcapsules on coating properties for basswood," *Coatings* 12(9), article 1246. DOI: 10.3390/coatings12091246
- Sarı, A., Hekimoğlu, G., Karabayır, Y., Sharma, R. K., Arslanoğlu, H., Gencel, O., and Tyagi, V. V. (2022). "Capric-stearic acid mixture impregnated carbonized waste sugar beet pulp as leak-resistive composite phase change material with effective thermal conductivity and thermal energy storage performance," *Energy* 247, article 123501. DOI: 10.1016/j.energy.2022.123501
- Sobel, R., Versic, R., and Gaonkar, A. G. (2014). "Chapter 1 - Introduction to microencapsulation and controlled delivery in foods," in: *Microencapsulation in the Food Industry*, A. G. Gaonkar, N. Vasisht, A. R. Khare, and R. Sobel (eds.), Academic Press, San Diego, pp. 3-12. DOI:10.1016/B978-0-12-404568-2.00001-7
- Temiz, A., Hekimoğlu, G., Köse Demirel, G., Sarı, A., and Mohamad Amini, M. H. (2020). "Phase change material impregnated wood for passive thermal management of timber buildings," *International Journal of Energy Research*, 44(13), 10495-10505. DOI: 10.1002/er.5679
- Vilà Ramirez, N., Sanchez-Soto, M., Illescas, S., and Gordillo, A. (2009). "Thermal degradation of polyoxymethylene evaluated with FTIR and spectrophotometry," *Polymer-Plastics Technology and Engineering* 48(4), 470-477. DOI: 10.1080/03602550902725472
- Wang, W., Cao, H., Liu, J., Jia, S., Ma, L., Guo, X., and Sun, W. (2020). "A thermal energy storage composite by incorporating microencapsulated phase change material into wood," *RSC Advances* 10(14), 8097-8103. DOI: 10.1039/C9RA09549G
- Wang, W., Peng, Y., Chen, H., Gao, Q., Li, J., and Zhang, W. (2017). "Surface microencapsulated ammonium polyphosphate with beta-cyclodextrin and its application in wood-flour/polypropylene composites," *Polymer Composites* 38(10), 2312-2320. DOI: 10.1002/pc.23813
- Wang, W., Zhang, S., Wang, F., Yan, Y., Li, J., and Zhang, W. (2016). "Effect of microencapsulated ammonium polyphosphate on flame retardancy and mechanical properties of wood flour/polypropylene composites," *Polymer Composites* 37(3), 666-673. DOI: 10.1002/pc.23223
- Xu, J., Yang, T., Xu, X., Guo, X., and Cao, J. (2020). "Processing solid wood into a composite phase change material for thermal energy storage by introducing silica-stabilized polyethylene glycol," *Composites Part A: Applied Science and Manufacturing* 139, article 106098. DOI: 10.1016/j.compositesa.2020.106098
- Ye, X., Wang, H., Wu, Z., Zhou, H., and Tian, X. (2018). "The functional features and interface design of wood/polypropylene composites based on microencapsulated wood particles via adopting in situ emulsion polymerization," *Polymer Composites* 39(2), 427-436. DOI: 10.1002/pc.23952

Article submitted: June 18, 2023; Peer review completed: July 8, 2023; Revised version received and accepted: July 17, 2023; Published: July 21, 2023.

DOI: 10.15376/biores.18.3.6068-6085