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Impregnation of Norway spruce with low melting-point binary fatty acid as a phase-change material

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Abstract

Norway spruce was impregnated with a eutectic mixture of capric acid and stearic acid. The binary fatty acid was used as a bio-based phase-change material (PCM) for further applications with wood as a latent heat-storage material in building components. For this purpose, different concentrations of impregnation were evaluated. The weight-percentage gain of 124% was the highest for the pure fatty acid mixture, and the PCM-impregnated wood increased density from 470 kg/m³ (untreated wood) to 1045 kg/m³. Compressive strength and hardness were significantly elevated with higher content of fatty acids in the wood. Microscopy analysis confirmed the presence of the PCM within the wood cells. The treated wood also exhibited a larger time delay during heating and cooling due to the temperature shift in the phase transition. Depending on the concentration of the fatty acid in the wood, the latent heat during melting ranged from 50 to 125 J/g in the temperature from about 24°C to 30°C. These results suggest that the use of the capric acid and stearic acid mixture has a great potential to increase the heat-storage capacity of wood and reduce energy consumption for heating and cooling indoor spaces.

Abbreviations: CA-SA: Eutectic mixture of capric acid (83%) and stearic acid (17%); C10%: Wood specimen impregnated with acetone containing 10% PCM; C25%: Wood specimen impregnated with acetone containing 25% PCM; C50%: Wood specimen impregnated with acetone containing 50% PCM; C100%: Wood specimen impregnated with pure PCM; DSC: Differential scanning calorimetry; FT-IR: Fourier transform infrared spectroscopy; PCM: Phase change material; SD: Standard deviation; L: Longitudinal; R: Radial; T: Tangential; WPG: Weight percentage gain.

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1. Introduction

The global energy consumption is increasing significantly. The scientific forecasts predict that the energy consumption will increase by almost 50% by 2050. To meet the worldwide goals of combating the climate crisis, a multifaceted approach is required (Gi *et al.* 2018, IEA 2020). As the transition to renewable energy sources is very slow while energy consumption is increasing rapidly, other measures to reduce energy consumption are urgently needed. The residential sector in the EU is accounting for about 25% of final energy consumption whereby 65% of it is used for the space heating (Pérez-Lombard *et al.* 2008, Thomas and Rosenow 2019). Wood, a renewable building material, is known for its excellent mechanical properties and contributes to climate conservation. Trees absorb CO₂ from the air, which can then be stored long-term in wooden constructions. Wooden buildings can be lightweight structures, which is of great advantage, but it also means that the thermal mass of these buildings is low.

Storing thermal-energy for later use is a growing trend (Kurdi *et al.* 2021). In building constructions, thermal energy can be stored as a change of internal energy in the external walls in form of sensible heat, latent heat or both (Aditya *et al.* 2017). Latent heat-storage is one of the most promising and future-oriented method to increase the energy efficiency of the residential buildings (Kenisarin 2014). To achieve this, phase-change materials (PCMs) can be utilised. These materials have the potential to reduce peak temperatures, summer overheating, and decrease the cooling and heating energy consumption demands in buildings when applied properly (Kośny 2015).

Different types of PCMs such as solid–liquid, liquid–vapor, and solid–solid have been investigated, but for application in building constructions, mostly solid–liquid or solid–solid systems are chosen (Nazari *et al.* 2020). When the PCMs reach the phase transition temperature, they absorb large amounts of heat at an almost constant temperature. Then, when the

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ambient temperature falls, the PCMs solidify releasing stored energy. The application of PCMs in building materials has been widely studied in materials like concrete, masonry or gypsum boards (Kalnæs and Jelle 2015, Kenisarin and Mahkamov 2016). Besides the usage in construction sector, PCMs can be utilised in textiles (Sarier and Onder 2012), vehicles (Janowski and McCluskey 2014), devices in the space (Wu *et al.* 2013) or as storage for solar energy (Pandey *et al.* 2018, Tyagi *et al.* 2021, Al-Ahmed *et al.* 2021, Naveenkumar *et al.* 2022). In general, PCMs can generally be classified according to their origin in organic or inorganic materials (e.g. salt hydrates). The melting point of PCMs determines their suitability for different applications. For instance, materials with relatively low melting point, typically in the range of 20°C to 26°C, are most suitable for use in structural building components (Nazari *et al.* 2020). General requirements for PCMs, especially for organic materials, include high latent heat capacity, low volumetric alteration during the phase transition, repeatability of phase transition, thermal stability across multiple heating and cooling cycles, as well as chemical stability, non-corrosive, non-toxicity, non-flammability, cost-effectiveness and ease of availability (Sharma *et al.* 2009, Agyenim *et al.* 2010).

The relatively inexpensive fatty acids as representatives of organic compounds used as pure or binary and tertiary mixture are commonly implemented in various fields, which show great advantages and appropriate physical and chemical material characteristics. Melting points of widespread fatty acids are within 7°C to 71°C, and their heat of fusion range from 45 to 210 J/g (Shilei *et al.* 2006, Kenisarin 2014, Duquesne *et al.* 2021, Singh *et al.* 2020). Suitable melting point temperature between 20°C and 26°C can be achieved with a binary eutectic mixture. For example, PCM based on capric acid and stearic acid with a melting temperature of 24.68°C and a heat of fusion of 178.64 J/g can be prepared. According to the literature, the lowest possible melting temperature of these two fatty acids is achieved with a weight-percentage concentration of 83% or 84% capric acid and 17% or 16% of stearic acid, respectively (Sarı *et al.* 2008, Temiz *et al.* 2020).

From an economic perspective and with the strong trend of using wood in building components, wood – a porous material – has a great potential for the incorporation of PCMs. Incorporating PCMs into lightweight timber construction can significantly reduce the energy required to maintain internal temperatures. As recent research has shown that there have been few attempts to impregnate PCMs into wood. For example, microencapsulated PCM was incorporated into red oak and sugar maple, resulting in a small weight gain of 5.4% ± 1.4% (Mathis *et al.* 2018). Another research group impregnated alkali-treated balsa wood with simple myristic acid as well as paraffin and polyethylene glycol, but the melting point was above 50°C for all materials (Liu, Wu, *et al.* 2021). Another group of researchers successfully impregnated untreated wood flour with single fatty acids as myristic acid, stearic acid as lauric acid (Liang *et al.* 2018). Pressure impregnation was used to treat Scots pine sapwood with a binary mixture of capric acid and stearic acid. The results showed a great increase in heat-storage capacity and a high retention value of $447.5 \pm 16.20 \text{ kg/m}^3$ (Temiz *et al.* 2020). An interesting investigation was conducted by Nazari *et al.* (2022) in which

Scots pine and European beech (sapwood) were impregnated with PCM based on coconut oil, increasing the weight by up to 95% for pine and 43% for beech. Furthermore, Palanti *et al.* (2022) examined the suitability of solid wood impregnated with PCM based on fatty acids for building construction, focusing on biodeterioration and degradation. As reported in the above-mentioned studies, it is possible to incorporate PCM into wood or wood flour in high quantities. However, as partially stated, fatty acids, when impregnated into porous materials, tend to leak when the temperature exceeds their melting point. Therefore, Harting *et al.* (2022) were able to significantly reduce the leakage of paraffinic PCM out of various wood species (spruce, oak, beech and poplar) by increasing viscosity by mixing thermoplastic powder.

The objective of this study was to investigate the effect of impregnating different concentrations (10%, 25%, 50%, and 100%) of fatty acid eutectic mixture on the physical and chemical properties of wood. The novelty of the study is in the selection of Norway spruce, a commonly used building material, for treatment and the examination of various concentrations. The preparation of the eutectic mixture of capric acid and stearic acid as a bio-based PCM allows for achieving a melting point that is well suitable for building constructions and helps to regulate indoor temperatures. The concentration of the PCM was lowered by dissolving it in acetone. The innovative application of acetone in dissolving fatty acids enables the reduction of the amount of PCM required for the impregnation process. Furthermore, the acetone may potentially facilitate the infiltration of PCM into the wood cell walls. Therefore, a volumetric change after impregnation and weight percentage gain were estimated. The distribution of the fatty acid mixture in the wood structure was visually confirmed using microscopic images. Mechanical tests and thermal analysis were conducted to determine the heat-storage performance. FT-IR analysis was performed to better understand the chemical alterations.

2. Materials and method

2.1. Materials

Sapwood of Norway spruce (*Picea abies* L.) sawn timber was used for this study. The boards were purchased in the local sawmill in Kuchl (Austria). The specimens were cut with different dimensions depending on the conducted analysis out of one trunk, which were free from any visible defects (knots, cracks, etc.). Capric acid (98%) was provided from Sigma-Aldrich (St. Louis, USA) and stearic acid (97%) was obtained from Acros Organics (Geel, Belgium). Acetone was received from VWR (Rue Carno, France).

2.2. Impregnations of the PCMs into wood

The impregnation was performed by using a eutectic mixture of capric acid and stearic acid (83% / 17% wt) as a PCM. The pre-weighed fatty acids were heated to 80°C and mixed for 30 min to obtain a homogeneous mixture. To achieve a lower weight-percentage gain (WPG) of impregnation, the ready eutectic mixture was solubilised in acetone at 40°C in concentrations of 10%, 25%, 50%, and 100%. The specimens were

further labelled C10%, C25%, C50%, and C100%, referring to the concentration of the fatty-acid mixture impregnated into the wood. Before impregnation, the wood was conditioned and had a moisture content of approx. 12%. Impregnation process began with vacuum (20 kPa) for 15 min. Then, the valve was opened and the liquid mixture was pulled into the impregnation chamber and the pressure was simultaneously increased to 600 kPa and kept for 2 h. To ensure low viscosity of the impregnating solution, the temperature in the chamber was kept at 40°C throughout the impregnation. After impregnation, the specimens were taken from the tank and excess fatty acid was gently wiped with paper towel. The specimens were placed in an oven at 60°C for 24 h to evaporate the acetone, and then measured and weighted. WPG was calculated according to Hill (2006) where M_m is the mass of modified wood and M_u is the oven dry-mass of unmodified wood. The dimensions of the specimens were taken in three directions after impregnation (D_m): radial (R), tangential (T), and longitudinal (L), and the cell wall bulking in every single direction (Bulking) was determined with the Equation 2, where D_u is the dimension of the specimens in one direction before the impregnation in dry state and D_m is the dimension after the impregnation.

$$WPG = [(M_m - M_u)/M_u] \quad (1)$$

$$Bulking = [(D_m - D_u)/D_u] \quad (2)$$

2.3. Light microscopy

To display the appearance of the fatty acid in the wood structure, the impregnated specimens were cut to smaller samples, and further analysed under microscope (Leica, Germany). To prepare the cross-section slices (thickness 20 μ m), the specimens were cut directly with sliding microtome (Leica, Germany) without any pre-treatment or softening. To stain the fatty acid in the wood, Oil Red O dissolved in isopropanol with a concentration of 0.5% was used. A minimal amount of the solution was applied to the specimens.

2.4. Compressive strength and hardness tests

Specimens for mechanical tests were conditioned in a climate chamber before testing at 20°C and at 65% relative air humidity. To analyse the influence of the fatty acids on the mechanical properties of wood, the compressive strength perpendicular to grain was measured. For each formulation, five specimens sized, 20 \times 20 \times 50 mm (R \times T \times L) were tested using a universal testing machine (Zwick and Roell 250). The Birnell hardness (HB) perpendicular to the grain was measured according to the standard DIN EN 1534:2000 (CEN/TC 175/WG2). A stainless-steel spherical indenter with a diameter of 10 mm was used.

2.5. FT-IR analysis

To analyse the chemical changes, attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectroscopy was conducted using a PerkinElmer Frontier FT-IR spectrometer

equipped with the ATR MIRacle accessory (PerkinElmer, Waltham, MA, USA). The specimens were cut with razor blades, and pieces from the inner part were taken for analysis. The measurement parameters were set at 32 scans with a resolution of 4 cm^{-1} in the wavenumber range between 4000 and 600 cm^{-1} . Each specimen was scanned in triplicate, and the mean value of these spectra was determined after correction of the baseline and area normalisation using the KnowItAll software (John Wiley and Sons, Inc., Hoboken, NJ, USA).

2.6. Differential scanning calorimetry

Differential scanning calorimetry (Mettler-Toledo DSC3) under a nitrogen atmosphere was used to determine the thermal properties of the fatty acid mixture and the impregnated wood. Samples of mass 20–30 mg were placed in enclosed aluminium crucible pans. The differential scanning calorimetry (DSC) tests were taken between 0°C and 50°C at a heating and cooling rate of 2°C/min. The heating-cooling cycle was repeated three times.

2.7. Thermal analysis

For thermal analysis, specimens of 80 \times 80 \times 20 mm were impregnated, and after evaporation of acetone, four specimens were joined together with polyurethane wood adhesives to form a panel of 160 \times 160 \times 20 mm. The experimental setup, as shown in Figure 1 was prepared. On both surfaces of the specimens, thin film thermocouples (type K) were installed. The temperature was measured to the nearest 0.1°C. For the heating phase, the heat source was set at 40°C for 12 h. For the cooling phase, the heat source was removed to allow the specimens to cool down to room temperature through the bottom surface. Using this setup, the time lag of the temperature on the top surface was measured.

3. Results and discussion

3.1. Material parameters of the impregnated wood

The weight percentage gain (WPG), retention, density, and volume increase of the specimens after impregnation and acetone evaporation are shown in Table I. Although spruce is generally considered as a difficult wood species to impregnate, the maximum WPG of 124% (SD 3.54) was achieved for specimens impregnated with pure fatty-acid mixture. The high WPG value can be attributed to the relatively small dimensions of the specimens, which led to a predominant longitudinal migration of the PCM into the wood.

With increasing acetone concentration in the PCM mixture, the WPG, retention, and density of the wood after impregnation decreased significantly. Furthermore, the data reveal that the use of acetone lowered the amount of PCM in the treated wood. The greatest gain in dimension is noticed in the C50% specimen, while the volume of the specimens without acetone changes the least. The increase in volume might be possible related to the penetration of the mixture of CA-SA and acetone into the cell walls. The penetration of the cell walls by impregnation of wood with fatty acid-based

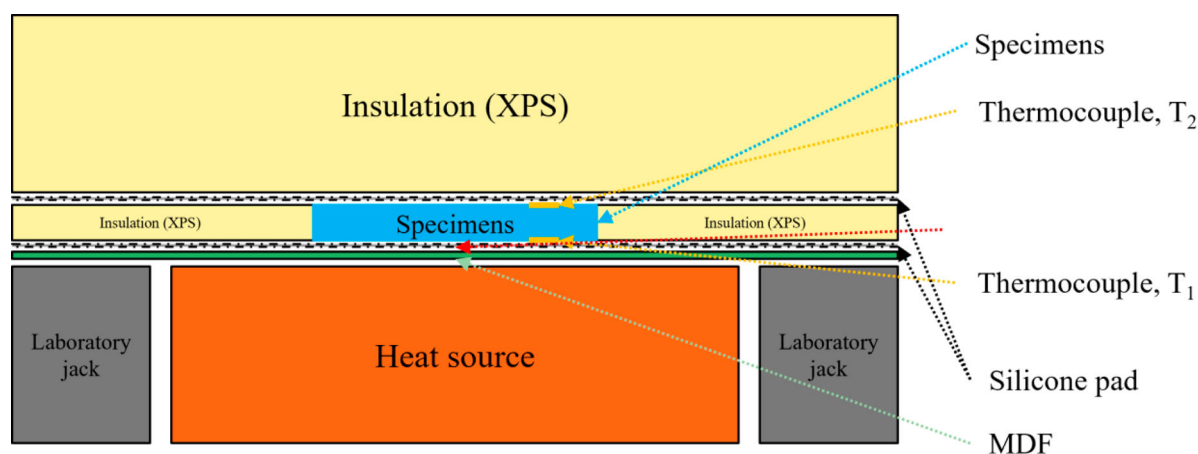


Figure 1. Test equipment for measuring the thermal properties of modified and untreated wood.

PCM was reported by Nazari *et al.* (2022) as well. The penetration of the cell wall can lead to swelling of the wood cell walls. The accessibility of the substance into the wood increases with the higher polarity of the solvent, which has a high affinity to the macromolecular wood components of the cell wall, resulting in a swelling state that allows diffusion into the interior of the cell wall. Polar liquids penetrate the cell wall of the wood through bulk flow and cell wall diffusion, while non-polar liquids only penetrate the wood through bulk flow and filling of the wood lumens (Ahmed *et al.* 2017). Therefore, the stronger bulking of the specimens could be favoured by the use of the polar acetone solvent in the mixture.

3.2. Light microscopy

The penetration of the fatty acid mixture was analysed by light microscopy. Micrographs of transversal section of the impregnated wood are presented in Figure 2. The Oil Red O shows a reaction with the fatty acid in the wood, which allows visualisation of them in the wood matrix. It should be noted that the staining solution was based on isopropanol, which easily dissolves the fatty acid. However, only a small amount was applied, it still might cause local removal of the impregnate.

By increasing the PCM concentration in wood, higher penetration of the tracheids can be seen. The fatty acid mixture starts to appear, especially in the latewood for specimens C10%, and further by increasing the concentration, more and

more substance is visible in the earlywood. For the specimen C100%, almost full impregnation is achieved. The high penetration of the tracheids and fulfilling the lumens can be attributed to the relatively small specimen dimensions resulting in longitudinal impregnation direction. However, this observation was also made by other researchers (Ulvcrona *et al.* 2006) during the impregnation of Norway spruce with linseed oil. In which the specimens with low uptake had the lumen in earlywood only filled in some parts, while latewood lumens were always filled to a high degree. As a possible explanation for that, refer the authors, beside the low degree of pit aspiration in latewood, that this part of latewood may be mechanically weaker and by applying the high pressure during the impregnation (as it was also the case during this study) creates new penetration paths.

Another possible explanation for the low visibility of the PCM in the tracheids of the earlywood by specimens with low WPG could be that the relatively large diameter does not contribute to keeping high quantities of PCM, so that single PCM parts can be dissolved more easily with isopropanol during staining or even the PCM could leave the wood matrix during impregnation process. The differences between earlywood and latewood can be better seen in the micrographs from specimens C50% in Figure 3.

3.3. Mechanical tests

The results of the mechanical tests are shown in Table II. The average compressive strength perpendicular to the grain (radial) of the untreated wood was 7.80 (SD 1.36) N/mm².

Specimen C100% with the highest amount of fatty acid achieved a value of 23.16 (SD 2.66) N/mm², which was more than 195% higher than the value obtained for the untreated wood.

Furthermore, the measured hardness for the untreated specimen was 11.85 (SD 1.65) N/mm², which is similar to the values reported by Wagenführ 2000. As for perpendicular to grain compression strength, as expected there is a great increase in hardness with the increasing amount of fatty acid mixture in wood. Therefore, the increased wood hardness, as well as the compressive strength, is related to the filled cavities

Table I. WPG, retention, density, and wood bulking of the impregnated specimens with PCM. Standard deviations are reported in the brackets.

	WPG (%)	Retention (kg/m ³)	Density (kg/m ³)	Wood bulking	
				Radial (%)	Tangential (%)
untreated wood	–	–	469.50 (12.35)	–	–
C10%	14.25 (0.55)	66.53 (1.82)	533.72 (8.00)	1.66 (0.23)	2.56 (0.70)
C25%	37.47 (1.45)	174.01 (4.13)	638.67 (10.95)	1.91 (0.31)	3.51 (0.72)
C50%	72.97 (4.19)	336.85 (17.05)	798.64 (14.21)	2.49 (0.86)	3.92 (0.32)
C100%	124.11 (3.54)	578.45 (6.81)	1044.73 (2.45)	0.70 (0.20)	1.22 (0.31)

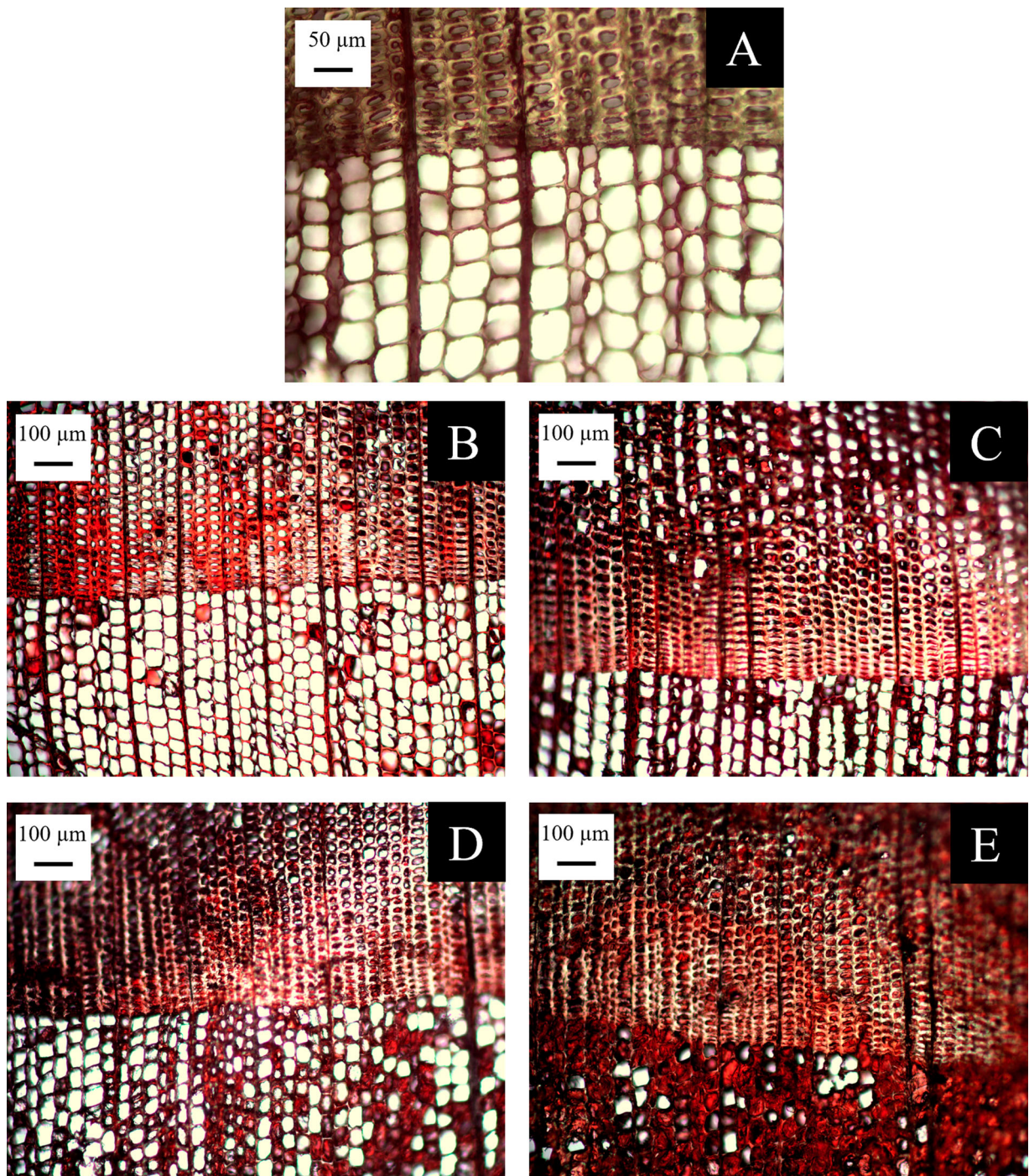


Figure 2. Micrographs of wood impregnated with PCM and stained with Oil Red O: (A) untreated wood, (B) C10%, (C) C25%, (D) C50%, (E) C100%.

of the wood structure with fatty acids and increased density (Temiz *et al.* 2020). The noticeably smaller increase in strength in both tests for C10%, C25%, and C50% specimen compared with C100% specimen may be caused by acetone. It has been mentioned by other authors such as Grabner *et al.* (2005) that the content of extractives in softwood may affect the transversal mechanical properties of the wood.

3.4. FTIR analysis of PCM impregnated wood

Figure 4 shows various FT-IR spectra of the CA-SA eutectic mixture, untreated wood, and the four wood specimens

impregnated with different concentrations of PCM. The C=O stretching vibration peak is observed at 1700cm^{-1} for the carbonyl group of carboxylic acid. The stretching vibration of the C–O appears at 1296cm^{-1} and at 1115cm^{-1} . Wagging of the CH_2 bonding is observed in region between 1268 and 1196cm^{-1} . The peak at 936cm^{-1} is due to the bending vibration of –COH. All of these signals come from a fatty acid mixture of capric acid and stearic acid, which as expected are not present in the spectrum of untreated wood (Filopoulou *et al.* 2021, Liu *et al.* 2021b, Nazari *et al.* 2021).

Figure 4 shows that the FT-IR spectra of the CA-SA mixture can be easily recognised in the impregnated specimens. As

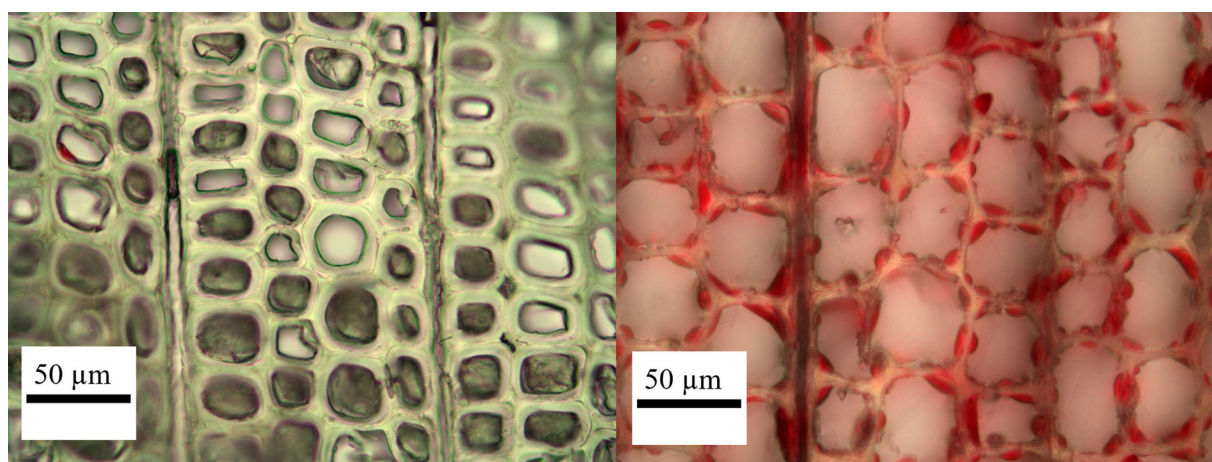


Figure 3. Micrographs of specimens C50%. Left the latewood section and right the earlywood section.

the amount of PCM in the wood increases, the absorption peaks of the fatty acid in the modified wood specimens become higher. Detection of absorptions bands of CA-SA in wood-impregnated specimens indicate the successful wood impregnation but there is no sign of any new bond that could be created between the fatty acid and the wood matrix. As already mentioned by other authors, it can be concluded, based on the FT-IR analysis that only physical interaction occurs between wood and fatty acids (Liang *et al.* 2018, Temiz *et al.* 2020).

3.5. Differential scanning calorimetry

Figure 5 shows the DSC curves obtained for capric acid and stearic acid mixture and the wood impregnated with the PCM in different concentrations. The melting temperature and the latent heat during melting can be taken from Table III. A significant reduction of the latent heat in comparison to the pure fatty acid can be noticed according to the mass changes of PCM in the composite. During the heating and cooling of the pure fatty acid mixture, only one clear peak is observed. The mixture starts to melt at about 25.3°C and reaches the fully melted state at around 35.9°C. However, during cooling of the material, the peak is shifted and material starts to solidifying at around 25.1°C and reaches the fully solid state at around 16.8°C. Similar shifts in melting and solidification temperatures of the fatty acid mixture were noted by Du *et al.* (2021) as well.

Table II. Compressive strength and hardness of the specimens impregnated with PCM and untreated wood.

	Compressive strength parallel to grain (N/mm ²)		Hardness perpendicular to grain (N/mm ²)	
untreated wood	7.80	(1.36)	11.85	(1.65)
C10%	9.40	(1.07)	12.34	(0.90)
C25%	11.23	(1.37)	15.01	(1.82)
C50%	13.37	(1.85)	17.43	(2.74)
C100%	23.16	(2.66)	31.05	(2.79)

Note: *Standard deviations are reported in brackets.

As already reported by Cabeza *et al.* (2015), the main issue with the DSC measurement is, that it is not well suited for heterogeneous samples impregnated with PCMs. Especially due to the very little amount (few mg) needed for the analysis which is difficult in preparation for a representative sample. As a result, the values obtained may not accurately reflect the performance of the PCM-impregnated wood when tested on a larger scale.

The PCM incorporated into wood shows slight alternations compared to pure fatty acid, particularly in the shifting of onset, endset and peak temperatures. This observation has been described as well by Nazari *et al.* (2022) for incorporating fatty acid into beech and pine, which can be possibly related to a shift in the crystalline point to a lower temperature. The study conducted by Temiz *et al.* (2020) showed that impregnation of Scots pine with the capric acid and stearic acid eutectic mixture resulted in a lower melting enthalpy of 94 J/g compared to the C100% specimen, which is related to a lower retention rate of the PCM. Another research group, Amini *et al.* (2022) analysed different concentrations of capric acid impregnated into Scots pine sapwood. The highest achieved retention was about 267 kg/m³ with an enthalpy of 70.5 J/g. The retention is slightly lower than that of the specimen C50%, therefore the enthalpy is proportionally lower. Moreover, the specimen C50% has a comparable latent heat to wood flour impregnated with the capric acid and stearic acid mixture (83% and 17%), which has a melting latent heat of 88.5 J/g (Sari *et al.* 2020).

3.6. Thermal analysis of PCM impregnated specimens

Figure 6 illustrates the results of the thermal analysis of untreated and CA-SA-impregnated specimens at concentrations of 10%, 25%, 50%, and 100%. As expected, when heating the untreated wood, which only stores sensible heat, the temperature rises continuously until reaching the maximum value and then remains constant. In the modified specimens, except for C10%, a distinct shift in temperature is observed at the point when the melting temperature of the PCM is reached.

This change in temperature rise is attributed to the phase change of the fatty acid from solid to liquid or liquid to solid during the heating and cooling, respectively. The PCM

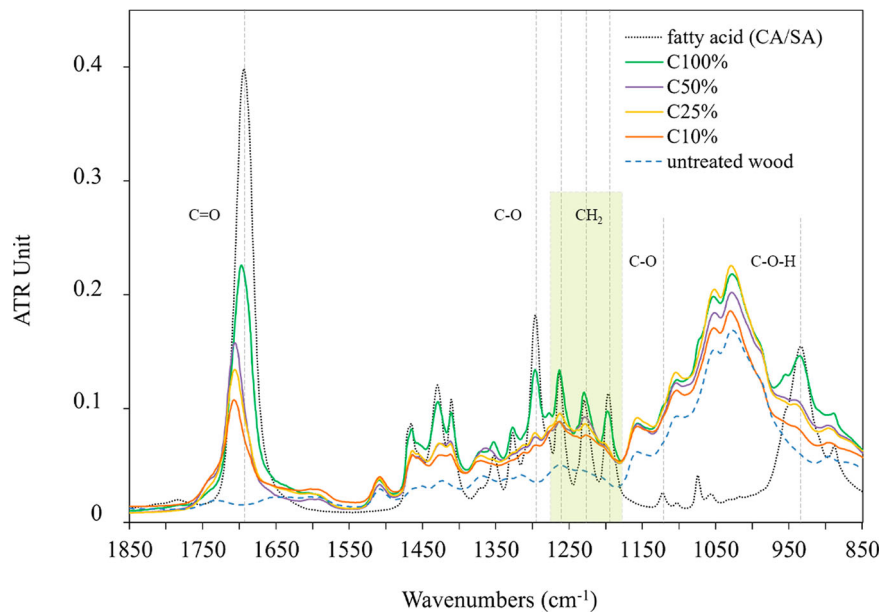


Figure 4. FT-IR spectra of fatty acid mixture, untreated wood and wood impregnated with PCM.

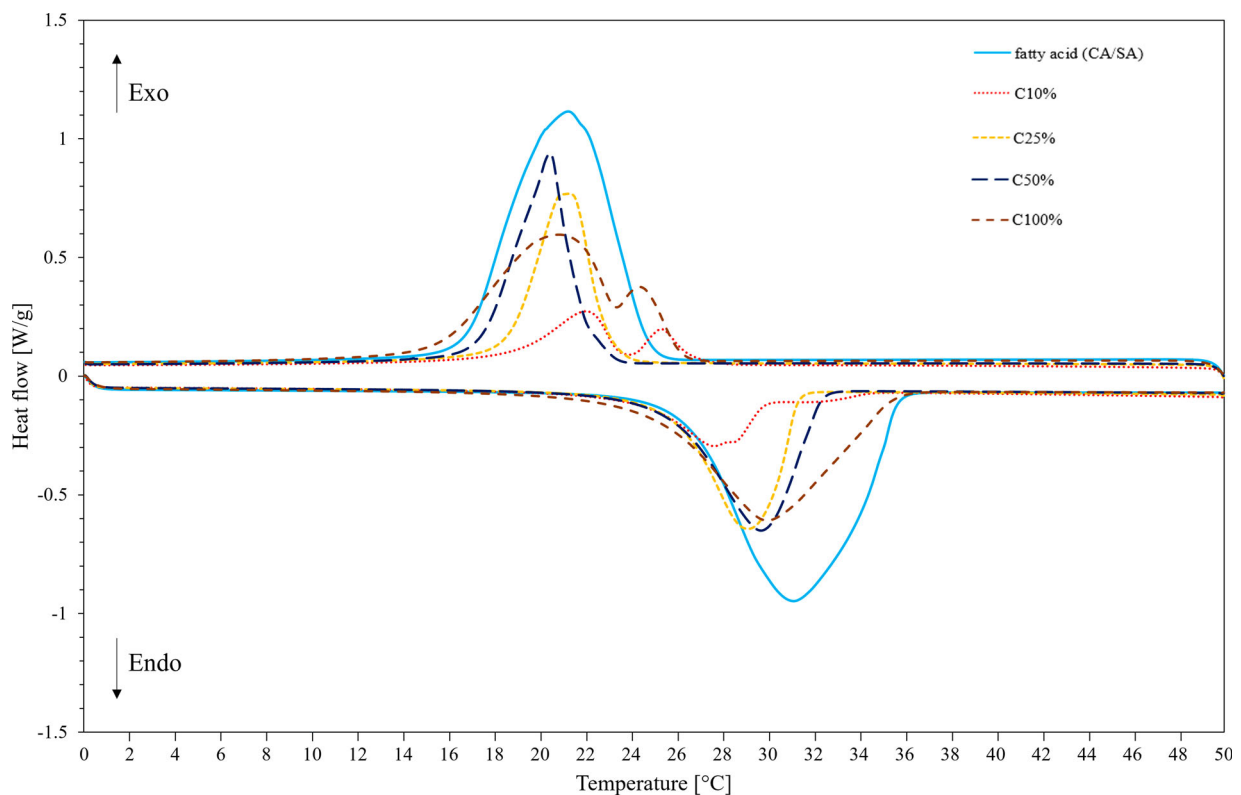


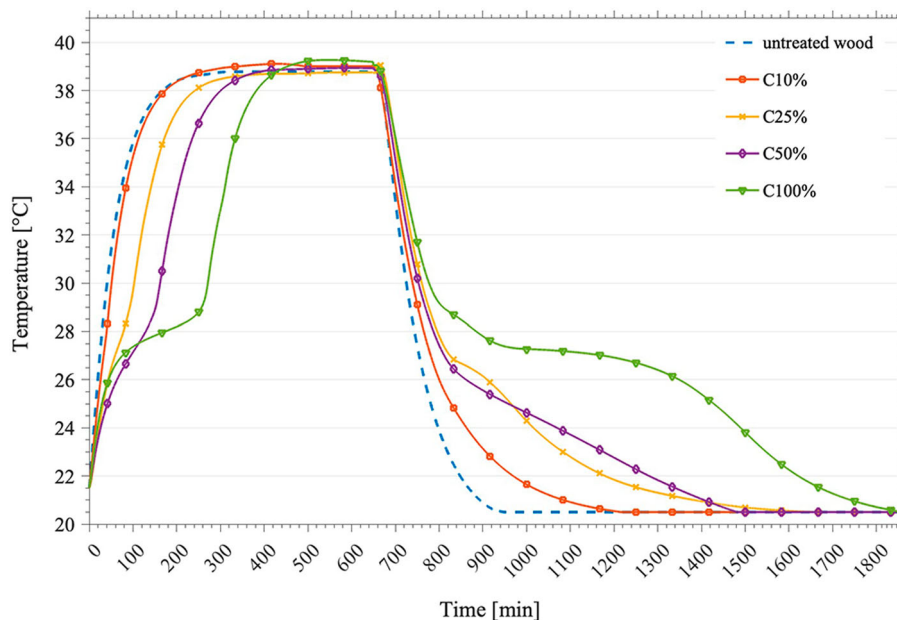
Figure 5. DSC curves of fatty acid mixture and wood impregnated with PCM.

incorporated into the wood begins to melt at approximately 25°C and continues until about 29°C. During cooling, there is a visible solidification of PCM for specimen C100%, which begins shortly at 29°C, and then at 26.5°C, a further noticeable change in cooling rate occurs. Similar behaviour was detected by DSC analysis, however, the end of the temperature shift is reached at the point where the specimen reached the peak temperature during the DSC analysis. The temperature

transition for specimens C50% and C25% is visible at 26.5°C, indicating solidification. The specimen C10% does not show the expected behaviour for a PCM as the temperature decreases constantly without any significant change in speed. It should also be noted that the heating time is shorter than the cooling time. This is due to the setup used for measurement. A hot plate was used for the heating phase, which creates forced convection, while for the cooling phase, the

Table III. Melting and solidification temperatures as well as heat of fusion of the PCM impregnated in wood, determined by DSC.

	Heating				Cooling				Heat of fusion	
	(°C)				(°C)				(J/g)	
	Onset	Endset	1 st peak	2nd peak	Onset	Endset	1 st peak	2nd peak	Heating	Cooling
C10%	24.3	34.5	27.6	32.2	26.7	16.3	25.3	22.0	49.7	46.1
C25%	24.8	31.4	29.1		23.9	16.7	21.3		75.84	75.4
C50%	24.8	32.1	29.9		22.9	16.0	20.4		84.8	85.0
C100%	23.5	35.2	29.9		27.0	16.0	24.5	20.8	125.0	122.3
fatty acid (CA-SA)	25.3	35.9	30.9		25.1	16.8	21.2		178.9	171.7

**Figure 6.** Thermal analysis of heating and cooling the specimens impregnated with PCM.

heat source was removed, and natural convection sink the temperature. Therefore, the concentrated heat (energy) cannot be released as quickly as with the forced convection. The time required for the temperature to rise from 21.5°C to 38.5°C was 213 min. Heating the specimen C10% took almost the same amount of time. However, during cooling, the specimen C10% does not show the typical behaviour of a change in aggregate state, nevertheless, it took about 150 min longer than the untreated wood to reach the temperature of 21.5°C which corresponds to an increase of 74% compared to the untreated specimen. This means that even this small amount of CA-SA contributes to the increase in time lag. In the same temperature range for the heating phase, the time delay was 91, 128, and 194 min for specimens C25%, C50% and C100% respectively. It means that the highest increase in time delay is for specimen C100%. Even more time delay was measured during the cooling phase, which was 376, 468, and 794 min for specimens C25%, C50% and C100%, respectively. The maximum cooling time was measured for specimen C100%, which is an increase in 392% compared to untreated wood. As expected, due to the high latent heat of CA-SA, the time lag for both heating and cooling increases with the increasing content of PCM in the wood, indicating that the heat storage capacity of impregnated wood can be greatly increased through the incorporation of fatty acids.

4. Conclusion

In this study, a binary fatty acid mixture of capric acid and stearic acid was prepared and successfully pressure impregnated into Norway spruce sapwood specimens at different concentrations. The highest WPG of 124% was achieved for the pure fatty acid mixture, and it successfully decreased as the concentration of the fatty acid in the mixture was lowered with acetone. Moreover, by dissolving the phase change material (PCM) in acetone, the amount of PCM needed for impregnation in the production process can be reduced.

- The light microscope revealed successful impregnation of the PCM into the wood matrix. The filling of the wood matrix can be explained by the relatively small specimen dimensions, which resulted in impregnation along the longitudinal direction.
- Compressive strength perpendicular to the grain and hardness were greatly increased by impregnation, filling the hollow structure of the wood and increasing its density.
- Chemical characterisation with FT-IR confirmed successful impregnation with the fatty acids, but no covalent bonding was detected, indicating no chemical interaction between the fatty acids and the wood.
- Simple thermal analysis showed a significant increase in the heat-storage capacity of PCM incorporated into wood.

The liquefying and solidification of the fatty acid in the wood could be easily identified due to the large shift in temperature delay during heating and cooling. The DSC analysis confirms the trend of the thermal analysis. The results show a slight shift in the onset and endset temperatures compared to the pure fatty acid mixture.

In conclusion, CA-SA impregnated into wood showed great potential to increase the thermal mass of the wood. However, further analysis of the detailed wood morphology after impregnation and more comprehensive thermal analyses is needed. Furthermore, attention should be given to the potential leakage of PCM from the wood.

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Disclosure statement

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Author contributions

Conceptualization: J.G.; Methodology: J.G.; Formal analysis: J.G.; Investigation: J.G, P.M.; Writing - original draft preparation: J.G., T.S.; Writing - review and editing: J.G., P.Z., T.S.; Funding acquisition: A.P., T.S.; Resources: A.P., T.S.; Supervision: P.Z., T.S.

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