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Characterization of 1-tetradecanol's performance as a phase change material in thermally modified ash wood

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Abstract

The use of phase change materials in thermally modified wood presents an innovative way to enhance the thermal properties of wood, contributing to energy efficiency and thermal comfort in various applications. However, careful consideration of the materials and methods used is essential to ensure the effectiveness and practicality of this technology. Phase change materials (PCM) can be integrated into thermally modified wood through various methods. One common method is impregnation, where the PCM is infused into the wood structure. Another method could be the application of PCM in coatings or finishes applied to the wood surface. In this study, 1-tetradecanol (TD) was used as a PCM and its effectiveness on thermally modified ash wood (TMA) was investigated. TMA has better thermal insulation properties, which are essential in energy storage applications to minimize heat loss. For this purpose, the leaching of TD from TMA was examined. Differential scanning calorimetry (DSC), thermogravimetric analyses (TGA), fourier transform infrared spectroscopy (FTIR) were examined. According to the results, there was a small amount of leakage in the leakage test. TGA analysis showed that TD/TMA left a lower residue of 16.30% at 800°C compared to the residue of 18.20% left by untreated thermally modified wood (TMA).

Keywords: Thermal energy storage, wood, vacuum impregnation, leakage test.

Termal olarak modifiye edilmiş dışbudak odunda faz değiştiren bir malzeme olarak 1-tetradekanolün performansı

Öz

Isıl işlemli odunda faz değiştiren malzemelerin kullanımı, odunun termal özelliklerini geliştirmek için yenilikçi bir yol sunarak çeşitli uygulamalarda enerji verimliliğine ve termal konfora katkıda bulunur. Bununla birlikte, kullanılan malzeme ve yöntemlerin dikkatli bir şekilde değerlendirilmesi, bu teknolojinin etkinliğini ve pratikliğini sağlamak için esastır. Faz değiştiren malzemeler (FDM), çeşitli yöntemlerle ısıl işlem uygulanmış oduna entegre edilebilir. Bunun için birçok yöntem vardır. Bu yöntemlerden biri FDM'nin oduna emprenyesidir. Diğer yöntem ise FDM'nin odun yüzeyinde üst yüzey işlemi veya cila olarak uygulanması. Bu çalışmada faz değiştiren malzeme olarak 1-tetradekanol (TD) kullanılmış ve termal olarak modifiye edilmiş dişbudak ağacı (TMA) üzerindeki etkinliği araştırılmıştır. TMA, enerji depolama uygulamalarında ısı kaybını en aza indirmek için gerekli olan daha iyi ısı yalıtım özelliklerine sahiptir. Bu amaçla, termal olarak modifiye edilmiş dişbudak odunundan (TMA) TD sızıntısı, sızıntı testi ile test edilmiştir. Örnekler diferansiyel taramalı kalorimetri (DSC), termogravimetrik analiz (TGA), fourier transform kızılötesi spektroskopisi (FTIR) ile incelendi. Sonuçlara göre sızıntı testinde az miktarda sızıntı vardı. TGA analizi, TD/TMA'nın, emprenyesiz ısıl işlemli onunda (TMA) 800°C'de bıraktığı %18.20'lik kalıntıya kıyasla %16.30'luk daha düşük bir kalıntı bıraktığını gösterdi.

Anahtar kelimeler: Termal enerji depolama, odun, vakum emprenyesi, sızıntı testi

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1 Introduction

Building-related sectors, responsible for approximately 40% of global energy consumption and 30% of CO_2 emissions, are the largest energy consumers (Najjar et al., 2019; Temiz et al. 2020; Amini et al. 2022). Hence, the advancement of eco-friendly buildings is crucial in shifting national energy frameworks towards sustainable energy sources, with the goal of decreasing reliance on fossil fuels and cutting CO_2 emissions. Numerous recent studies have explored and detailed the design and outlook of sustainable buildings, encompassing ideas such as net zero emission structures and plus energy homes (Lund, 2010; Li et al. 2023). Wood is regaining popularity as a sustainable construction material for large buildings due to its environmental benefits and strong physical properties. Despite historical concerns about fire safety, modern engineering has improved wood's viability, and several tall wood buildings around the world showcase its potential. The trend towards eco-friendly construction and advances in prefabrication techniques support wood's comeback. However, broader adoption of wood in construction requires updates in building codes to encourage better insulation and energy efficiency, as well as advancements in fire safety and connection technology. As the industry embraces digitalization and automation, role of wood in construction is set to grow, helping to reduce the environmental impact of new building (Wimmers, 2017). In recent years, many nations worldwide have observed a growing trend in the construction of multi-story timber buildings, thanks to advancements in engineering science and the technology behind building with wood (Žegarac Lescovar and Premrov, 2021; Antonini and Gaspari, 2022). Since ancient times, timber cladding has served as an accessible and economical means of weatherproofing. Presently, there is a resurgence of interest in timber cladding for its environmental benefits and natural aesthetics. There is considerable enthusiasm for employing timber cladding systems to enhance energy efficiency through the retrofitting and refurbishing of existing structures (Hill et al. 2022). Contemporary exterior wood coatings frequently utilize formulations such as water-based acrylic dispersions, water-borne alkyd emulsions, or high solids alkyd systems with low volatile organic compound (VOC) content, alongside traditional oil-based paints (Hill et al. 2022).

Thermal modification is one method employed to protect wood materials used in cladding. The process of heat treatment is a method for modifying wood that boosts its dimensional stability, resistance to water, and durability against biological threats, and it achieves this without relying on noxious chemicals (Yildiz et al. 2013; Temiz et al. 2013; Jirouš-Rajković and Miklečić, 2019). Thermal modification remains the leading process for producing modified wood in Europe in terms of volume, with thermally modified timber (TMT) seeing significant use in cladding applications (Hill et al. 2022; Herrera et al. 2018). While TMT is chosen for cladding because of its enhanced dimensional stability and decreased moisture absorption, it has also been observed to exhibit better performance in fire situations and increased resistance to mold (Hill et al. 2022).

Phase change materials (PCMs) are adept at absorbing and releasing substantial quantities of thermal energy through a phase transition process. They enhance energy efficiency in buildings and help to cut down on energy consumption derived from fossil fuels (Temiz et al. 2020). Wood and wood-based materials can serve as excellent matrices for PCMs due to wood properties such as inherent porous nature of wood, cost-effectiveness, abundant availability, non-toxicity, and high chemical stability. The interest in leveraging the wood framework as a support for PCM is increasing, with possibilities for integration into diverse wood forms including solid lumber, composite wood, delignified wood, and transparent wood (Can and Zigon, 2022; Hekimoğlu et al. 2021; Demirel, 2023).

Demirel (2023) conducted research on Scots pine samples that were thermally treated and then impregnated with Lauric Acid (LA) and Myristic Acid (MA) as a phase change material (PCM) using a vacuum process. The findings showed that the PCM significantly reduced water absorption and enhanced the dimensional stability of the wood samples. Additionally, the impregnation with LA-MA altered the mechanical properties of the thermally modified wood, which is notable because thermal modification usually diminishes mechanical properties.

The strategy of using thermally modified wood integrated with phase change materials (PCM) presents an innovative method to enhance the thermal characteristics of wood products. This approach offers energy-efficient solutions and elevates comfort in diverse applications. Ongoing research and advancements in this field have opened avenues for experimenting with novel PCM compositions, impregnation methods, and uses, aiming to boost thermal efficiency.

Due to its high melting point, 1-tetradekanol (TD) can be used particularly in building insulation, energy efficient technologies, passive solar space heating, saunas, and agricultural heating applications where temperature ranges beyond typical human comfort conditions are relevant. The key advantage of using high melting point PCMs in these applications is their ability to store thermal energy when it is available in excess and release it when needed. This leads to a more efficient use of energy, reduces the need for external energy sources, and helps in maintaining a more consistent temperature, which is vital in applications like underfloor heating, saunas, solar space heating, and greenhouse heating.

In this study, TD was used as a PCM and thermally modified ash wood (TMA) impregnated with 1-tetradecanol. TD is chosen for its suitable melting point and latent heat properties, which are crucial for efficient thermal energy storage. The aim of this study was to determine the characterization of thermally modified wood with TD to employ these wood materials in applications such as passive solar space heating and agricultural heating. The impregnation of thermally treated wood with 1-tetradecanol is not just a technical endeavor but a step towards creating more sustainable, energy-efficient, and environmentally friendly building materials that cater to the modern needs of energy conservation and climate control in architecture and construction.

2. Material ve Method

2.1 Material

Ash wood (*Fraxinus excelsior* L.) samples subjected to thermal treatment were provided by Novawood, a heat treatment company located in Bolu, Turkey. The heat treatment process comprised three key phases: preparation for heat, the heat treatment itself, and cooling-conditioning. The initial stage involved two steps. The first step commenced at 25 °C and progressed to 120 ºC over 14 hours for ash wood. The second step started at 120 °C, reaching 212 ◦C within 13 hours. Ash wood exposed to a 2 hours heat treatment at 212 °C. Following the heat treatment, the wood materials exposed to a cooling process, consisting of cooling and conditioning stages. The cooling phase lasted 14 hours, cooling the wood materials to 120 °C. Subsequently, a 7-hour conditioning stage followed. At a temperature of 60 °C, the wood materials were removed from the boiler, marking the conclusion of the heat treatment procedure.

TMA was first fragmented into smaller pieces and then pulverized into a powder. The wood samples, which were cut into small pieces, were ground in an IKA MF 10 Basic

microfine grinder drive. Subsequently, the wood powder exposed to sieving with a mesh number 40 sieve to eliminate fine particles. Wood powder (4 gr) filtered through a 40 mesh sieve was used.

The TD was purchased from Sigma- Aldrich Company. It was used as the phase change material for thermal energy storage. The chemical formula of TD is $C_{14}H_{30}O$ (assay ≤100.0; melting point: 36 °C; molecular weight: 214.39).

2.2 Method

2.2.1 Impregnation process

The TD was melted by heating at 80 °C that was higher than its melting temperature. Moisture content of wood particles are 12%. TD/ TMA were prepared 50% (w/w). Milled TMA was impregnated with the TD in the vacuum oven at 0.08 Mbar at 70 °C for 3h. The mixtures were mixed every 1 h (vacuum was stopped and restarted) to ensure homogeneous mixing. This process was conducted considering the study of Hekimoğlu et al (2023). The weigt percantage gain (WPG) was calculated using Eq. 1.

$$WPG\ (\%) = \frac{W2-W1}{W1} \times 100 \quad (1)$$

Where, W_2 is the weight after impregnation, W_1 is the weight before impregnation.

2.2.2 Leakage test

The TD/TMA mixture was pressed after impregnation with Panavıse 502 Precision PanaPress (Figure 1) and subjected to the leakage test. The TD/TMA composite was positioned on filter paper and subsequently placed inside an oven set at 50 °C for a duration of 30 minutes. This test was conducted considering the study of Hekimoğlu et al. (2023).The amount of leakage remaining on the filter paper was calculated using Eq. 2.

$$Leak\ rate\ (\%) = ((W_2 - W_1)/\ W_1) \times 100 \quad (2)$$

Where, W_1 is the weight of filter paper before test; W_2 is the weight of filter paper after test.

Figure 1. Pressing of TD/TMA after impregnation.

2.2.3 Characterizations

FTIR spectroscopy was utilized to identify the chemical structures of the substances and to explore the physicochemical interactions between them, using a Perkin Elmer Frontier Model from the USA.Spectra were captured at a resolution of 4 cm^{-1} in the range of 4000 to 500 cm^{-1}

DSC was utilized to ascertain the enthalpy of melting and solidification along with the phase transition temperatures of the TD/TMA composite. The DSC analysis was performed using a Hitachi DSC 7020 model at a rate of 3°C/min in a nitrogen environment.

Thermal stability of the samples was assessed using a thermogravimetric analyzer (SDT Q600 TA Instrument). Samples weighing between 5 and 10 mg were placed in a platinum pan and heated under a nitrogen atmosphere. The heating rate was set at 10°C/min across a temperature from 30°C to 600°C.

3 Results and Discussion

3.1 Leakage test results

Leakage test for PCMs is a critical assessment to determine the containment reliability of the PCM during its phase transition, especially when it shifts from a solid to a liquid state. The objective is to ensure that the material does not escape from its encapsulation or containment system, which is essential for practical applications like thermal energy storage, where the PCM must remain contained over numerous cycles of melting and solidifying. The weight percantage gain was found 48%. The image of TD/TMA subjected to leakage test is given in Figure 2.

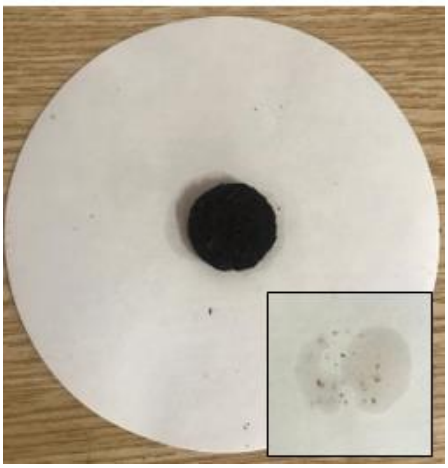

Figure 2. The leakage test results of TD/TMA after 1.cycle

The leakage rate (%) was determined along 4 cycles of heating/cooling (Table 1). After the 3th cycle, the leakage amounts reached similar values. That's why only the first 3 cycle values are given.

Table 1. The leakage rate (%) of TD/TMA

	1.cycle	**2.cycle**	**3.cycle**
Leakage rate (%)	0.95	1.86	0.91

According to the leakage test result, a small amount of tetradecane leaked onto the filter paper. This shows the ability of TMA to retain tetradecane in the liquid phase. During the leakage test, it was observed that a minor quantity of TD permeated through the containment material, evidenced by its presence on the filter paper. This suggests that while the containment largely maintains structural integrity, there are pathways through which the TD can migrate in its liquid state.

3.2. FTIR results

The FTIR spectra of TD, TD/TMA and TMA are shown in Figure 3.

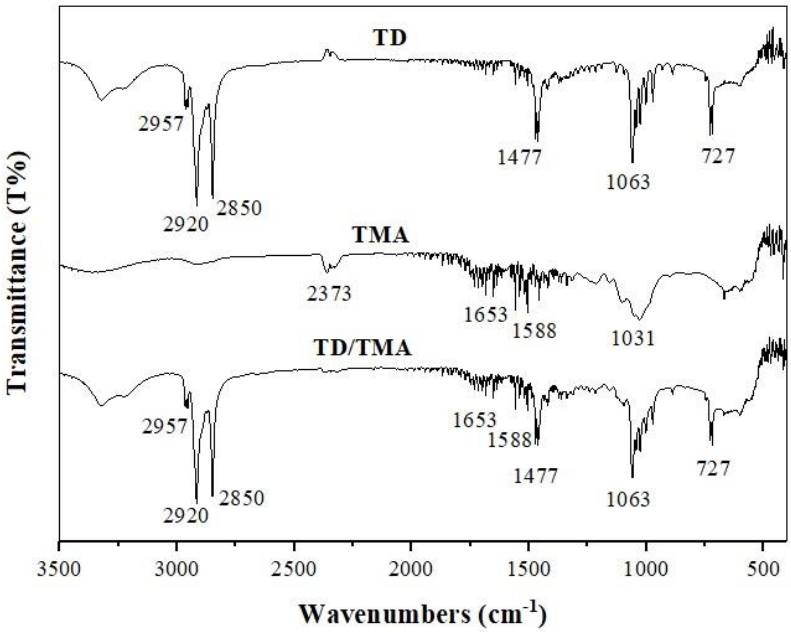

Figure 3. The FTIR spectra of the TD, TD/TMA and TMA

The spectrum of TD shows characteristic peaks at 2957 cm^{-1} and 2850 cm^{-1}, which are typical of the C-H stretching vibrations in the aliphatic hydrocarbon chains. The peak at 1477 cm^{-1} corresponds to the bending vibrations of C-H, and the peaks around 1063 cm^{-1} and 727 cm^{-1} could be attributed to C-O stretching and C-H rocking vibrations, respectively, which are consistent with the alcohol functional group in TD.

The TMA spectrum lacks the prominent peaks seen in the TD spectrum, indicating the absence of long aliphatic chains or alcohol functional groups in the untreated wood. Instead, the TMA shows broader and less defined peaks, which might indicate the presence of various oxygen-containing functional groups, such as those found in lignin and cellulose, which are common in wood.

The spectrum for the TD/TMA composite shows the characteristic peaks of TD, suggesting that the TD has been successfully impregnated into the thermally modified wood. The retention of distinct peaks for TD in the composite indicates that the compound is physically present within the wood structure without significant chemical modification.

When comparing the spectra, it is evident that the composite (TD/TMA) retains the chemical signature of TD, while also displaying the underlying absorbance pattern of the thermally modified wood. This suggests that there is no chemical reaction between the TMA

and TD, as no new peaks have emerged, and all major peaks from both components are present in the composite spectrum.

3.3 DSC results

The DSC thermograms for TD and TD/TMA are shown in Figure 4. Thermal properties of the TD and TD/TMA, including entalpy and peak temperature for melting and solidifiying processes are summarized in Table 2.

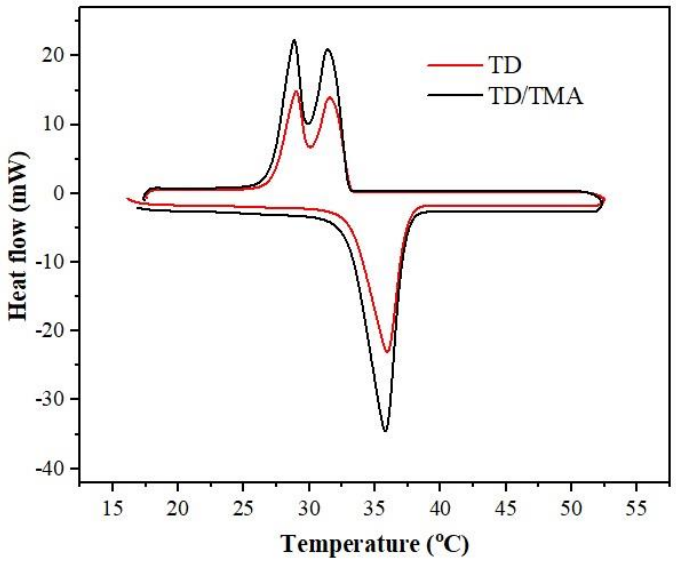

Figure 4. DSC thermograms of TD and TD/TMA

Table 2. DSC values of the TD and TD/TMA

	Melting		**Solidifiying**	
	T_{peak} (°C)	ΔH (J/g)	T_{peak} (°C)	ΔH (J/g)
TD	33.2	208.4	33.07	207.6
TD/TMA	33.3	103.1	33.10	102.9

Both TD and TD/TMA showed very similar melting and solidification points (TD: 33.2°C and 33.07°C; TD/TMA: 33.3°C and 33.10°C respectively). This indicates that the thermal modification of the wood and the addition of TD do not significantly alter the phase change temperature of TD.

There is a noticeable difference in the melting and solidification enthalpies between pure TD and the composite. The pure TD shows higher enthalpy changes (208.4 J/g for melting and 207.6 J/g for solidification) compared to the composite (103.1 J/g for melting and 102.9 J/g for solidification). This reduction in the enthalpy change suggested that the interaction with the thermally treated wood affected the energy required for the phase transition of TD. Ayaz et al. (2023) investigated TD as PCM with functionalized multi-walled carbon nanotubes for high-density thermal energy storage. TGA data for TD yielded similar results.

The similar phase change temperatures imply that the composite material could be used in the same applications as pure TD in terms of operating temperature range (Tripathi et al. 2023). However, the lower enthalpy values for the composite suggest that it would store less thermal energy per unit mass compared to pure TD.

The DSC graph shows that the thermal behavior of the composite closely tracks that of the pure TD, suggesting that the thermal properties of TD are preserved in the composite. This is an important consideration for applications where the phase change properties of the material are critical.

3.4 TGA results

The weight loss as a function of time and temperature was measured in the TGA, as shown in Figure 5. Table 3 lists the temperatures at which weight losses (at 10, 20, 30, 40, 45%) were produced and the amount of residue.

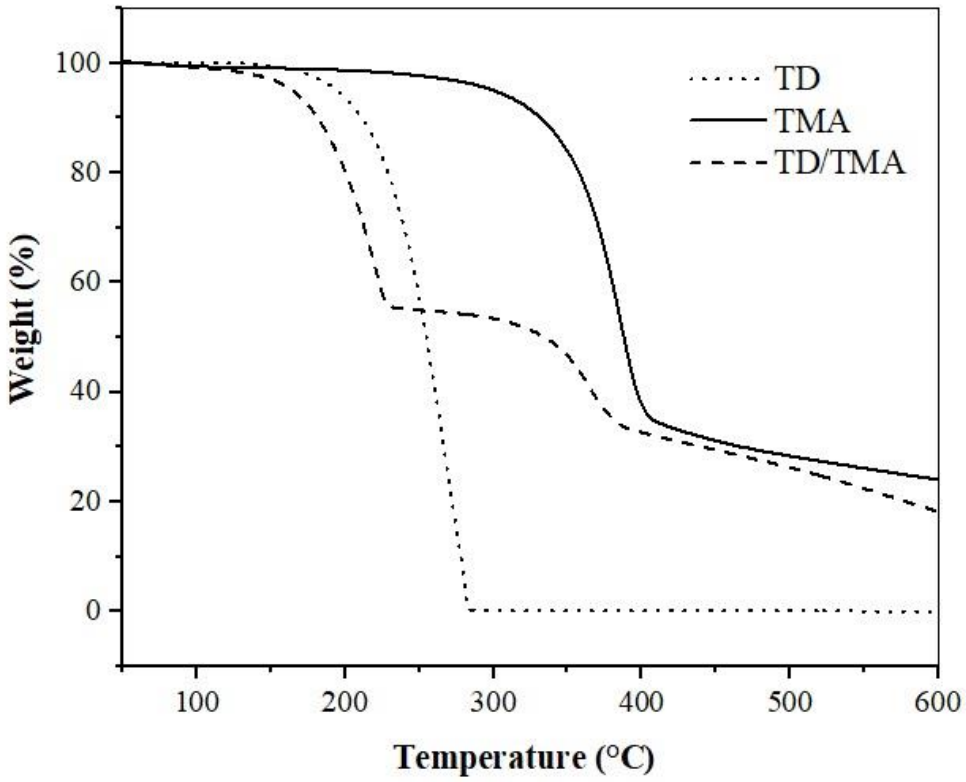

Figure 5. TGA curves of the TD, TD/TMA and TMA

Table 3. TGA data of the TD, TD/TMA and TMA

	TG/°C				
	10%	30%	50%	75%	Residue (%)
TD	211.11	239.71	254.65	269.03	0.00
TD/TMA	181.11	212.92	333.64	516.37	16.30
TMA	326.46	370.9	387.9	567.7	18.20

TMA shows a high degree of thermal stability up to 326.46°C, which was where it begins to lose 10% of its weight. It continued to degrade gradually, with a 50% weight loss at 387.9°C, and leaved a residue of 18.20% at the end of the TGA run. This indicated that TMA retained much of its structure until a relatively high temperature, which is characteristic of the thermal modification process that enhances the resistance to thermal degradation of wood.

The composite begined to lose weight at a significantly lower temperature (181.11°C for 10% weight loss) compared to TMA alone. This early weight loss is likely due to the volatilization or degradation of TD, which has a lower thermal stability than TMA. The composite continued to degrade, with a 50% weight loss observed at 333.64°C, which is significantly lower than TMA alone. The residue left at the end of the TGA run for the composite is notably higher (16.30%), likely due to the combined residue from TMA and the non-volatile components of TD.

The TGA curves showed that the presence of TD in the composite lowers the initial degradation temperature, reflecting the thermal properties of TD. The weight loss pattern suggests that the TD is released from the composite at lower temperatures, followed by the degradation of the thermally modified wood component at higher temperatures.

The differences in thermal degradation profiles between TMA and TD/TMA suggested that the composite material may have applications where controlled release of TD is desired at lower temperatures, while still benefiting from the enhanced thermal properties of TMA at higher temperatures. The high residue content in the composite could be advantageous in applications requiring high carbon content materials. It is also important to potential applications of these materials in the context of their thermal degradation properties, such as in building materials, thermal energy storage systems, or other areas where thermally stable or PCMs are utilized.

The temperatures at which wood components begin to decompose are as follows. Hemicellulose starts to degrade at the lowest temperatures among the major components of wood, typically beginning around 150°C to 260°C. The degradation of cellulose usually starts at temperatures above 260°C and progresses rapidly beyond 350°C. Lignin is the most thermally stable component of wood. Its degradation begins at around 280°C but continues over a wide range of temperatures, even beyond 500°C (Rowel, 2005).

4 Conclusion

In this study, TMA was treated with TD as a PCM. Since the melting point of TD (36 C) is high, the resulting product is especially targeted for use in places such as underfloor heating systems, saunas, solar space heating and greenhouse heating. The data and recommendations obtained as a result of the experiments are listed below.

- The leakage of TD was minimal. However, to better bond PCM to wood, additional processes can be performed in the impregnation, the rate of TD can be reduced or a combination with PCM with a lower melting point can be created.
- The resulting DSC data can be used to discuss the suitability of the TD/TMA composite for thermal energy storage and its advantages, such containment of the material during phase change. Additionally, the reduction in enthalpy of the composite can be further investigated to understand the interaction between TD and thermally treated wood at the microscopic level, which may reveal insights into the structure-property relationships of the composite.
- The TGA analysis indicates that the elevated residue levels in TD/TMA could be beneficial for uses that demand materials rich in carbon content. Moreover, the

significance of these materials lies in their possible applications relating to their thermal degradation characteristics. This includes their use in building materials, thermal energy storage systems, and various other domains where materials that are thermally robust or capable of phase transitions are required.

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Author Contributions
Gaye Köse Demirel: Conceptualization (Developing research ideas and objectives), Data curation, Formal Analysis, Funding acquisition, Investigation, Methodology, Resources, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing.

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