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N-octadecane /bio-char composite: preparation, characterization and energy storage properties

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Abstract

Biochar (BC) is a carbonized material rich in functional groups that has many different uses such as soil reclamation, energy storage, anodic material in batteries and electromagnetic emission capture in buildings. The n-octadecane (OD) is a favourable organic phase change material (PCM) for thermal energy storage which can be applied in building energy storage materials due to its proper phase change temperature. In this study, BC was impregnated with the OD in the vacuum oven at 0.08 Mbar at 70 °C for 3h. The weight percentage gain was found to be 50% after impregnation. Leakage of OD from BC after impregnation was detected by leakage test. The physico-chemically characterize of thermally treated wood samples were examined by Fourier Transform Infrared Spectroscopy (FT-IR). Thermal degradation stability of the samples were analyzed by thermogravimetric analyses (TGA) and diffential scanning calorimetry (DSC) analysis. According to results, no leakage was observed after the leakage test in BC samples impregnated with OD. A significant amount of residue was evident in the BC after it was infused with OD indicating that its decomposition commenced only at markedly high temperatures according to TGA results. The FTIR spectrum doesn't show any extra absorbance peaks. According to obtained results, BC/OD exhibits favourable characteristics suitable for energy storage in buildings or similar applications.

Keywords: Biochar, n-octadecane, phase change materials

N-oktadekan / biyokömür kompoziti: hazırlanması, karakterizasyonu ve enerji depolama özellikleri

Öz

Biyokömür (BC) toprak ıslahı, enerji depolama, pillerde anodik malzeme olarak, binalarda elektromanyetik emisyon yakalama gibi birçok farklı kullanıma sahip, fonksiyonel gruplar açısından zengin, karbonize bir malzemedir. N-oktadekan (OD), uygun faz değişim sıcaklığı nedeniyle bina enerji depolama malzemelerinde uygulanabilen, termal enerji depolaması için uygun bir organik faz değişim malzemesidir. Bu çalışmada BC, OD ile vakumlu fırında 0,08 Mbar'da 70 °C'de 3 saat süreyle emprenye edildi. Emprenye sonrası ağırlık artışı %50 olarak bulunmuştur. Biyokömürden n-oktadekan sızıntısı, sızıntı testiyle tespit edildi. Isıl işlem görmüş ahşap numunelerinin fiziko-kimyasal olarak karakterizasyonu fourier transform kızılötesi spektroskopisi (FT-IR) ile incelenmiştir. Numunelerin termal bozunma stabilitesi, termogravimetrik analizler (TG) ve diferansiyel taramalı kalorimetri (DSC) ile analiz edildi. Sonuçlara göre OD emdirilmiş BC numunelerinde sızdırmazlık testi sonrasında herhangi bir sızıntı gözlenmemiştir. Elde edilen sonuçlara göre BC/OD, binalarda veya benzeri uygulamalarda enerji depolamaya uygun olumlu özellikler göstermektedir.

Anahtar kelimeler: Odun kömürü, n-oktadekan, faz değiştiren maddeler

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1 Introduction

The increased demand for energy-saving materials has arisen in response to the worldwide energy crisis (Dincer and Ezan 2018). Therefore, several types of materials have been researched that can contribute to energy conservation. First, aerogels, lightweight and porous, boast exceptional insulation properties and find applications in building insulation and windows (Wakel and Nasir 2023; Yue et al. 2022). Second, PCMs undergo phase transitions to absorb and release heat energy, regulating building temperatures and enhancing solar panel efficiency (Fan et al. 2023; Gencel et al. 2022; Sharaf and Huzayyin 2022). Third, thermoelectric materials convert heat into electricity recovering waste heat from industrial processes (Hasan et al. 2020). Fourth, smart windows adjust transparency and reflectivity to control sunlight influencing building temperature (Oh et al. 2021). Lastly, nanomaterials enhance the efficiency of solar cells, batteries and fuel cells. Among these, PCMs stand out as particularly promising for building applications (Cunha and Aguiar 2020; Wen et al. 2022; Ustaoğlu et al. 2021)

Thermal energy storage (TES) is regarded as an innovative method for storing energy by utilizing latent heat PCM. These materials are thoroughly studied for potential use in textiles (Sarier and Onder 2007) and electronics to provide thermal protection for microelectronics (Tan and Tso. 2004). The primary benefit of employing these materials in buildings is their ability to align energy supply and demand, leading to a reduction in energy costs. TES materials that are based on PCM are favoured over sensible heat storage and thermochemical heat storage technologies due to their high heat storage density, smaller temperature fluctuations and nearly isothermal phase change processes (Zalba et al. 2003).

Over the last few decades, researchers have explored different PCMs like paraffin waxes (such as n-octadecane and n-hexadecane), fatty acids, salt hydrates and combinations thereof aiming to create effective latent heat TES materials suitable for diverse applications. Paraffin waxes are particularly favoured for incorporation into building materials with their inherent benefits (Zalba et al. 2003; Yuan et al. 2014; Farid et al. 2004). Nevertheless, the utilization of these materials in construction has been restricted primarily due to two key issues: low thermal conductivity and the occurrence of liquid leakage during their phase change processes.

Solid-liquid PCMs are broadly categorized into paraffins, hydrated salts, fatty acids, fatty alcohols, organic esters and certain polymers (Liang et al. 2018; Can, 2023). The predominant challenges encountered in their practical implementation are the leakage of molten PCM and low thermal conductivity (Ma et al. 2019; Demirel 2023; Amini et al. 2023). Despite considerable efforts to address these issues, it remains a formidable task to create PCMs with the desired operating temperature, high latent heat storage (LHS) capacity, relatively elevated thermal conductivity, excellent cycling thermal and chemical stability and cost-effectiveness (Cheng et al. 2017). One of the most favored approaches for effectively addressing these challenges involves integrating PCMs with porous, lightweight and economical carrier materials (Jeon et al. 2019; Sari et al. 2020; Can and Žigon 2022).

The thermal conductivity of many form-stable composite PCMs is poor in thermal conductivity property leading to the incorporation of carbon-based fillers with enhanced thermal conductivity such as graphene, graphene oxide, carbon nanotubes and carbon nanofibers (Atinafu et al. 2020). Despite this improvement, their widespread use in TES systems is hindered by significant drawbacks like high cost and issues related to agglomeration.

Biochars derived from bio-waste are viable options to substitute the carbon fillers mentioned earlier as they can be produced in a more cost-effective and environmentally friendly manner (Khadiran et al., 2015a; Hekimoğlu et al., 2021). Consequently, they are employed to address leakage challenges and improve the thermal conductivity of PCMs due to their favourable surface textural properties (Gu et al., 2019; Hussein et al., 2015; Hekimoğlu et al., 2023).

PCMs are favoured for solar passive TES applications in a solid-liquid state. Nevertheless, their TES potential is significantly limited by low thermal conductivity and the challenge of molten PCM leakage. In this study, environmentally friendly, abundant and cheap BC was used to prevent the leakage problem, and the use of OD as a PCM was examined.

2. Material ve Method

2.1 Material

The OD was purchased from Sigma-Aldrich Company. It was used as the PCM for TES. The chemical formula of OD is $C_{18}H_{38}$; assay ≥99.0; density 0.78 g/cm^3. It is within the alkane group and has a melting temperature of 28 °C. BC (density: 0.30-0.41 g/cm^3; purity: 95%; particle size < 100 nm) was purchased from Nanografi Company (Turkey).

2.2 Method

2.2.1 Impregnation process

BC was dried in an oven at 100 ˚C for 7 h to remove moisture. The OD was melted by heating at 70 °C which was higher than its melting temperature. OD/BC was prepared 50% (w/w). BC was impregnated with the OD in the vacuum oven at 0.08 Mbar at 70 °C for 3h. The mixtures were mixed every 1 h to ensure homogeneous mixing. The wight percantege gain (WPG) of the BC was found to be approximately 50% after impregnation.

2.2.2 Leakage test

This test is referenced from some previous articles (Hekimoglu et al., 2023; Amini et al., 2023). Filter paper Whatman No:2 was used for the PCM leakage test. The OD/BC was placed on the filter paper and then it was placed in an oven at 50 °C for 30 min. The leakage was determined visually by seeing the leak of OD on the filter paper.

2.2.3 Fourier-transform infrared, diffential scanning calorimetry and thermogravimetric analyses

FTIR analyses were employed to determine the chemical structures of the components and the physicochemical interactions among them (Perkin Elmer Frontier Model, the USA). The spectrums were recorded with 4 cm^{-1} resolution between 4000 and 500 cm^{-1}.

DSC was employed for determining the melting and solidification enthalpy values with phase change temperatures of the OD/BC. DSC was conducted at a heating/cooling rate of 3°C/min under nitrogen atmosphere in Hitachi DSC 7020 model DSC instrument. These measurements were repeated for three times and the mean deviation value was determined as ±0.13ºC and ± 1.24 J/g for the melting and solidification temperatures and latent heat values of the samples

A thermogravimetric analyzer (SDT Q600 TA Instrument) was used in determining the thermal stability of the samples. The samples (5-10 mg) were put in platinum pan under nitrogen at a heating rate of 10ºC/min above a temperature range of 30ºC to 600ºC.

3 Results and Discussion

3.1 Leakege test results

The leakage test for a PCM like OD in BC is an essential evaluation to ensure the material's suitability for practical applications particularly in thermal energy storage systems. The test is designed to determine whether the PCM leaks out of its containment material when it transitions from solid to liquid and vice versa. The image of BC/OD powders subjected to leakage test is given in Figure 1.

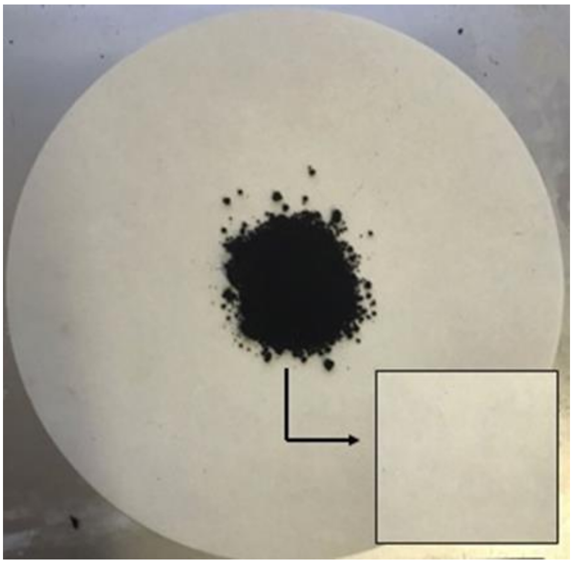

Figure 1. The leakage test results of BC/OD

No leakage was observed after the leakage test in BC samples impregnated with OD. As seen in the figure, no leakeage is observed in BC/OD powders with OD mass content of 50 wt%. The WPG of the BC was found to be approximately 50% after impregnation. This outcome suggests the effective penetration of OD into the porous structure of BC. Khadiran et al (2015b) obtained similar results in their study and suggested that the porous network of BC may offer extensive capillary and adsorption properties that effectively prevent the leakage of molten OD. The leaching test results aligned well with the findings from the DSC analysis. When the amount of leakage is minimal, DSC results will show stable thermal properties. In our study, DSC results of BC/OD showed more stable thermal properties. A composite featuring a greater loading of OD would be more advantageous for applications involving TES materials. In their study, the leakage test results agreed well with the findings from the DSC analysis. They stated that a composite containing higher OD loading would be more advantageous for applications involving thermal energy storage materials.

The absence of leakage may be due to the sorption and diffusion mechanisms of OD into the BC. The surface area and pore volume of BC can facilitate the diffusion of OD into its internal structure, thus it can reduce the risk of leakage. Moreover, the thermal conductivity and heat capacity of BC could play a role in distributing the thermal energy evenly preventing localized melting that could lead to leakage.

3.2. FTIR results

The FTIR spectra of OD/Biochar are shown in Figure 2.

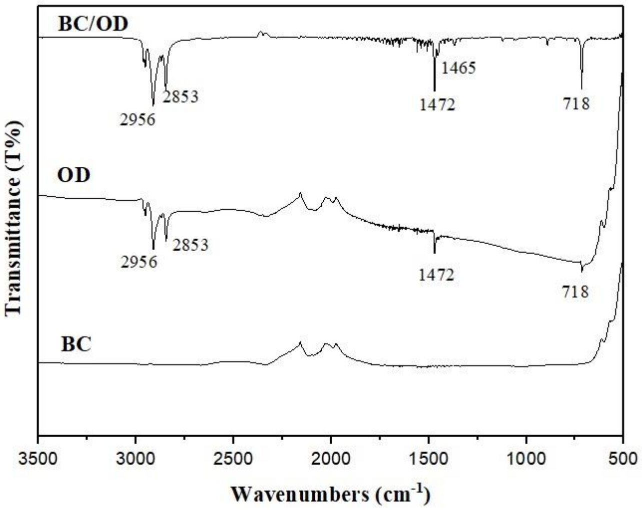

Figure 2. The FTIR spectra of the BC/OD, OD and BC

The peaks at 2956 and 2853 cm^{-1} represent the stretching bands of C−H in OD which are indicative of the long hydrocarbon chains present in OD. These peaks are present in both the OD and BC/OD samples but are absent in the BC sample, which is expected, since BC is primarily composed of aromatic carbon structures and does not contain long aliphatic chains.

The peaks at 1465 cm^{-1} and 1472 cm^{-1} can be attributed to the bending vibrations of C-H bonds in both the OD and BC/OD samples. The similarity of the peaks in this region for OD and BC/OD suggests that the octodecane in the composite retains its chemical identity without significant alteration.

The BC spectrum does not show significant peaks in the region typical for aliphatic C-H stretches suggesting that the material is composed mainly of aromatic or graphitic carbon structures. However, the lack of well-defined peaks in the aromatic C=C stretch region (around 1500-1600 cm^{-1}) could indicate that the structure of the BC is highly amorphous or that the aromatic domains are small.

The BC/OD spectrum combines features from both OD and BC, with the aliphatic C-H stretch peaks indicative of octodecane and the lack of distinct aromatic peaks indicative of the BC component. The presence of both sets of peaks suggests that the composite contains both components without significant chemical interaction that would change the fundamental vibrational modes of either component.

There are no new absorption bands evident in the FTIR spectrum. Consequently, the combination of OD with BC occurred through a physical process rather than involving any chemical reaction.

3.3 DSC results

The DSC thermograms for OD and BC/OD are shown in Figure 3. Thermal properties of the OD and BC/OD, including entalpy and peak temperature for melting and solidifiying processes are summarized in Table 1.

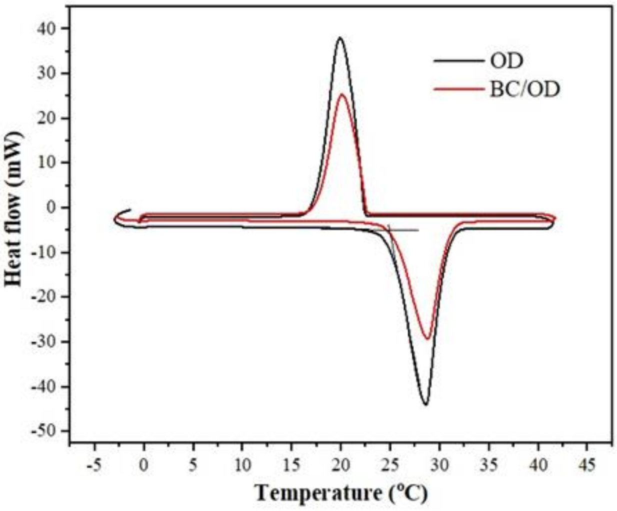

Figure 3. DSC thermograms of OD and BC/OD

Table 1. DSC values of the OD and BC/OD

	Melting		Solidifiying	
	T_{peak} (˚C)	ΔH (J/g)	T_{peak} (˚C)	ΔH (J/g)
OD	25.11	215.1	22.77	213.4
BC/OD	25.32	106.4	22.79	105.2

The melting temperature of pure octodecane is slightly lower (25.11 °C) compared to the biochar/octodecane composite (25.32 °C). The increase in melting temperature for the composite suggests that the presence of BC may influence the thermal stability of octodecane, possibly due to interactions at the interface between biochar and octodecane or due to a change in the crystalline structure when BC is added.

The enthalpy change (ΔH) for melting is significantly higher for pure octodecane (215.1 J/g) than for the composite (106.4 J/g). This substantial decrease in ΔH for the BC/OD composite indicates that less energy is required for the phase transition which may be due to the BC disrupting the crystalline lattice of the octodecane leading to a lower latent heat of fusion.

The solidifying temperatures of OD and BC/OD are very close (22.77 °C for OD and 22.79 °C for BC/OD) it suggests that BC has a minimal effect on the initiation of solidification. However, the ΔH is also about half for solidification for the composite similar to the melting behaviour (105.2 J/g for BC/OD) compared to pure octodecane (213.4 J/g for OD) which further supports the notion that the composite requires less energy for the phase change.

Hekimoğlu et al. studied active carbon/expanded graphite hybrid for the development of nanodecane-based composite. Pure nanodecane demonstrates its solid-to-solid transition peak at 23.36 °C during the heating cycle with an enthalpy change of 36.12 J/g and at 17.73 °C during the cooling cycle with an enthalpy change of 35.93 J/g.

Lee et al., (2017) investigated the thermal enhancement of n-octadecane/porous nano carbon-based materials (OPNCs) utilizing a three-step filtered vacuum impregnation technique. They employed n-octadecane as the phase change material (PCMs) alongside a variety of carbon-based supports such as C-300, C-500, Activated Carbon (AC), Expanded Graphite (EG) and Exfoliated Graphite Nanoplatelets (xGnP) that are all derived from identical raw materials. Differential Scanning Calorimetry (DSC) analysis was conducted which measured the latent heat capacities of OPNCs ranging between 220 J/g and 393 J/g to assess the thermal efficiency of OPNCs.

3.4 TGA results

The weight loss as a function of time and temperature was measured in the TGA as shown in Figure 4. Table 2 lists the temperatures ,at which weight losses (at 10, 20, 30, 40, 45%) were produced, and the amount of residue.

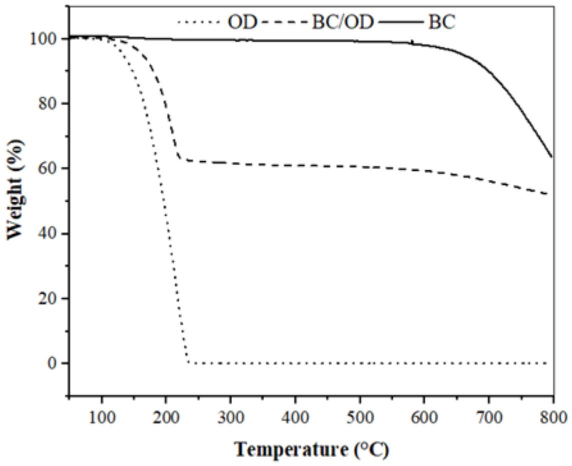

Figure 4. TGA curves of the OD, BC/OD and OD

TGA graph for OD, BC and a BC/OD provides valuable data on the thermal stability and decomposition characteristics of these materials.

OD exhibits a single-stage weight loss which is typical for pure hydrocarbons. This weight loss corresponds to the melting and subsequent vaporization of the octodecane as the temperature increases and it leaves no residue indicating complete volatilization.

Table 2. TGA data of the OD, BC/OD and OD

	TG/°C					
	10%	20%	30%	40%	45%	Residue (%)
BC	698.68	742.52	776.25	-	-	63.92
BC/OD	179.61	198.07	210.41	559.07	725.34	51.79

BC/OD shows a multi-stage weight loss. The initial weight loss up to about 200°C can be attributed to the loss of octodecane. The fact that the BC/OD curve follows the OD curve initially suggests that the presence of BC does not significantly affect the initial thermal decomposition of octodecane. However, beyond this point, the curve deviates, and the weight loss proceeds at a slower rate compared to pure octodecane suggesting that the BC has a stabilizing effect on the remaining octodecane in the composite or that the BC itself is decomposing at a slower rate.

The BC curve shows that BC is thermally stable over a wide temperature range and begins to decompose significantly only at high temperatures. This is consistent with the nature of BC which is a carbon-rich material and generally exhibits high thermal stability.

In terms of residue percentage, biochar leaves a substantial amount of residue (63.92%) which is characteristic of carbonaceous materials that do not completely decompose until very high temperatures. The BC/OD composite leaves a smaller percentage of residue (51.79%) compared to pure BC which suggests that a portion of the BC has been consumed or transformed during the thermal process potentially due to interactions with the octodecane.

Khadiran et al. (2015b) studied on the preparation of a shape-stabilized n-octadecane/AC nanocomposite, intended for thermal energy storage (TES) in building applications, employing a singular impregnation technique. The TES materials devised during this research were examined using a variety of methods including FTIR, RAMAN, DSC, FESEM, nitrogen adsorption-desorption and leaching tests. Thermograms from TGA/DTG of the unaltered n-octadecane showed a single degradation phase from 125.0 °C to 174.3 °C attributed to the vaporization of n-octadecane. Conversely, the AC sample exhibited no signs of decomposition so it suggests that the AC, selected for this study, possesses thermal stability and is suitable as a structural support material.

Chen et al. (2012) utilized activated carbon (AC) to fabricate shape-stabilized composites of lauric acid and AC. Findings showed that lauric acid readily infiltrated the porous structure of the AC resulting in improved thermal conductivity within the composites.

4 Conclusion

As a result of this study, the following outputs were obtained.

- Weight percentage gain was found to be 50% as a result of the impregnation of BC with OD as a PCM by vacuum process.
- No leakage was observed in BC/OD which was subjected to leakage testing at 50 °C for 3 hours. This shows that OD is effectively retained in the porous BC.
- No additional absorbance peaks are present in the FTIR spectrum so it indicates that the integration of OD into BC was achieved via a physical method rather than through chemical interactions.
- Regarding the residue percentage, BC retains a considerable portion of its amount of residue, a trait typical of carbon-based substances that fully decompose at extremely elevated temperatures. As a result, a notable residue was observed in the BC that had been combined with OD which only began to break down at significantly high temperatures.
- BC/OD provide promising property for use in energy storage applications in buildings or other similar applications. BC/OD appears to be highly appropriate for thermal energy storage objectives by taking into account the entire range of DSC data.

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None.

Author Contributions

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

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Conflict of interest statement
The author declare no conflict of interest.

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