Titre: Title:	Natural wax supported by microporous biochar to create a stable phase change material
Auteurs: Authors:	David Brassard, Adya Karthikeyan, & Jason Robert Tavares
Date:	2025
Type:	Article de revue / Article
Référence: Citation:	Brassard, D., Karthikeyan, A., & Tavares, J. R. (2025). Natural wax supported by microporous biochar to create a stable phase change material. The Canadian Journal of Chemical Engineering, 104(1), 44-51. https://doi.org/10.1002/cjce.70020

URL de PolyPublie: PolyPublie URL:	https://publications.polymtl.ca/66266/
Version:	Version officielle de l'éditeur / Published version Révisé par les pairs / Refereed
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Titre de la revue: Journal Title:	The Canadian Journal of Chemical Engineering (vol. 104, no. 1)
Maison d'édition: Publisher:	Wiley
URL officiel: Official URL:	https://doi.org/10.1002/cjce.70020
Mention légale: Legal notice:	

Received: 2 April 2025 | Revised: 10 June 2025 | Accepted: 11 June 2025

DOI: 10.1002/cjce.70020

RESEARCH ARTICLE

THE CANADIAN JOURNAL OF CHEMICAL ENGINEERING

Natural wax supported by microporous biochar to create a stable phase change material

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Funding information
Natural Sciences and Engineering Research Council of Canada, Grant/Award Number: RGPIN-2019-05378

Abstract

Energy storage, exploiting the latent heat of phase change materials, offers an efficient method to store and release heat. Initial developments of phase change materials and their containment targeted performance over environmental impact. This article presents a bio-sourced, fully compostable, and biodegradable composite phase change material made from hardwood charcoal and beeswax. This material combines effective heat transfer performance with a significant focus on sustainable end-of-life waste management, especially when compared to traditional methods that rely on synthetic or heavily modified bio-sourced phase change materials. We measured a beeswax uptake within charcoal's porous structure of up to 61.2%. This resulted in a composite with a latent heat enthalpy of 77.83 J g^{-1}, with cyclability tested up to 20 cycles without any reduction in performance. The process was scaled from millimetre-scale particles up to centimetre-scale particles with similar adsorption capacities. The Laplace–Young equation confirmed that adsorption of beeswax is mostly driven by a capillary pressure, of at least 2.2×10^4 N m^{-2}, caused by the naturally occurring porous structure of charcoal. These beeswax-loaded charcoal particles could find applications for heat recovery in HVAC systems or in packed bed heat storage systems.

KEYWORDS

beeswax, biosourced, charcoal, energy storage, phase change material

1 | INTRODUCTION

Phase change heat transfer is one of the most efficient methods of heat transfer, as it exploits latent heat, which is significantly higher than sensible heat transferred in single-phase mode. For example, the sensible heat required to raise the temperature of water from 0 to 100°C is 418 kJ kg^{-1}, while its latent heat of vaporization at 100°C is five times higher at 2260 kJ kg^{-1}. Researchers have tapped this interesting aspect of high-density heat transfer to design phase change materials (PCM) for passive heating and cooling applications.[1,2] PCMs are mainly classified based on their chemical nature as organic (e.g., paraffins, non-paraffins such as fatty acids, alcohols), inorganic (e.g., salt hydrates and metallic alloys), and eutectic mixtures of organic and inorganic materials. This class of thermal energy storage materials finds applications in electronics,[3,4] solar energy capture,[5,6] and construction materials[7,8] to name a few.

Research on PCMs has focused on various parameters such as thermal properties (e.g., specific heat, latent

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heat), stability for thermal cycling, commercial availability, cost, and encapsulation methods. However, with the concerns over climate change, the focus is now shifting to their environmental impact above other factors. Various naturally occurring materials such as stearic acid, coconut oil, animal fat, and beeswax have been studied in recent years. Duyar et al. studied lamb tail fat as a PCM by adding cyclophosphazene derivatives to improve its thermal properties.[9] Palmitic acid, a fatty acid found in soybean oil, is considered a good PCM for domestic applications given its calorific value.[10] Beeswax is studied as a PCM as it is a natural alternative to paraffin wax, since its melting range falls between 42.5 and 67.6°C.[11] In certain cases, to improve the thermal properties of a naturally occurring PCM, high thermal conductivity materials such as carbon nanotubes or graphene are incorporated,[12] but the biodegradability of such combinations has yet to be established.

PCM are often encapsulated in another material following a core-shell model or blended into a host material to form a composite.[13] Even though biodegradable PCMs are being explored, the encapsulation materials used to contain them are mostly synthetic polymers such as polyurethane or polyester, which are non-biodegradable and thus destined as landfill or incineration waste products at the ends of their life cycles.[14,15] Hence, in addition to employing a natural PCM, it is also essential to encapsulate it in a biodegradable or compostable material to ensure full product eco-friendliness. A few researchers have prepared PCM composites using biomaterials such as gelatinized potato starch,[16] chitosan blends, or agarose or cellulose derivatives[17] to house a polyethylene glycol PCM. However, end-of-life decomposition of these materials is not discussed, leaving the biodegradability question unanswered, and the mechanism of storage is underdeveloped.

In addition to being eco-friendly, the encapsulating material should be capable of storing a large volume of PCM. To store a high volume of PCM, for practical applications, the encapsulating material should not only be highly porous, but its pore size should be such that PCM leakage is prevented during phase transitions. Thus, in this work, we introduce a fully biodegradable[18–20] PCM-shell matrix using hardwood charcoal, a naturally occurring highly porous material as the housing material for beeswax, a natural phase change material.

Guidelines to achieve this storage can be obtained by estimating the capillary pressure of a PCM (e.g., beeswax) in the pore structure of the encapsulating matrix (e.g., hardwood charcoal) using the Laplace–Young equation.

$$\Delta p = \frac{2\gamma\cos\theta}{r}, \tag{1}$$

where Δp is the resulting capillary pressure, γ the beeswax's surface tension, θ is the contact angle, and r is the radius of the pores. The results from this calculation, reported below, suggest that the fine pore structure of charcoal makes it a suitable candidate to contain beeswax during repeated thermal cycles. An appropriate matrix and PCM combination is dependent on multiple factors such as melt viscosity, molecule size, pore size and surface affinity. It appears, based on our result, that hardwood charcoal in conjunction with beeswax threads the fine line where such a good match is present. To the authors' knowledge, this novel combination of beeswax within a fully biodegradable matrix was not previously reported in the scientific literature.

2 | METHODOLOGY

2.1 | Material

A bag of mixed hardwood charcoal lumps, sold under the brand name Master Chef, was bought locally from a retail store. A bag of filtered pure cosmetic grade yellow organic beeswax pellets, distributed under the brand name Mary Tylor Naturals, was bought from a generalist online retailer.

2.2 | PCM composite preparation

The charcoal pieces were first ground and then sieved to mesh sizes between 1 and 0.38 mm. Due to the fibrous, elongated aspect ratio of some of the ground charcoal particles, some larger particles were not filtered out. The resulting charcoal particles were mixed with beeswax pellets and heated in a furnace at 110°C in air. Alternatively, charcoal pellets were added to molten beeswax (at 110°C) to obtain similar results. The volume of beeswax in the beaker during infusion was maintained such as to completely submerge the charcoal under molten beeswax, allowing beeswax to be available in excess of the charcoal particles' sorption capacity. Infusion tests were performed both under and without vacuum. Superfluous beeswax on the outer surfaces of the charcoal particles was removed by placing and rolling the PCM composite dispersed on absorbent paper on a hot plate.

Scale-up tests were performed with a charcoal piece sanded to a rough prismatic shape with flat outer surfaces. In a furnace at 110°C, the charcoal piece was dipped in a small container holding molten beeswax with a depth of approximately 5 mm. Additional beeswax pellets were progressively added to maintain the immersion depth until pore saturation was reached. Pore saturation was defined as the moment when the beeswax level within the container did not drop anymore. The flat outer surfaces were wiped to remove any traces of excess beeswax.

2.3 | Beeswax extraction tests

To assess the possibility of leaching during long term high temperature exposure, a desorption test was performed in a convection furnace heated at 110°C. The PCM particles were spread on clean aluminium foil. The thermal treatment was repeated on new aluminium foil as long as traces of wax could be detected on the surface of the aluminium foil.

2.4 | Characterization

Particle size analysis of charcoal was performed using a Mastersizer model 3000 (Malvern Panalytical Instruments Ltd). Two sets each of 30 particle size distribution measurements were obtained. The charcoal particles were dispersed in ethanol (95%) until the gauge obscuration reached 3%. Data points were fitted using Mie theory.

Experiments to validate favourable interfacial interaction of beeswax inside the porous structure of charcoal, with the Laplace–Young equation, were performed with an OCA-20 Contact Angle System from Dataphysics. The system operated at elevated temperature with a TEC 400 temperature controlled environmental chamber and a NHD Electrical needle heating device both set to 70°C. Beeswax was melted in the heated needle and then deposited on the surface of a heated charcoal piece. The experiment was repeated on multiple points at the surface of two samples to measure the effect of the orientation of the pore structure. On the first sample, the pores axes were normal to the test surface (normal direction); on the second sample, the pores axes were parallel (transverse direction). Thermal characterization of individual compounds and PCM composite particles was performed on a TA Instruments Q2000 differential scanning calorimeter (DSC) with Tzero hermetic pans at a scan rate of 2°C min^{-1} between 0 and 120°C for 1, 10, and 20 cycles. Accelerated thermal cycles were performed at 20°C min^{-1} over the same temperature range for the other cycles to reduce the test duration. The melt enthalpy and temperature were calculated during the cooling cycles from the area under the curve and the peak heat flow.

Scanning electron microscopy (SEM) images were obtained with a Hitachi TM3030Plus tabletop SEM at an acceleration voltage of 15 kV in mixed mode.

The average pore radius was estimated from SEM images using the ImageJ software package. An ellipse shape was used to measure the area of each individual cell in the image. The average pore area was calculated from all of the individual cells identified in the image. From the roughly circular shape of the pores, the average pore radius was estimated by calculating the radius of a disk of the average pore size area. Although it was applied to our material for other purposes, vacuum physisorption (often described as the Brunauer-Emmett-Teller

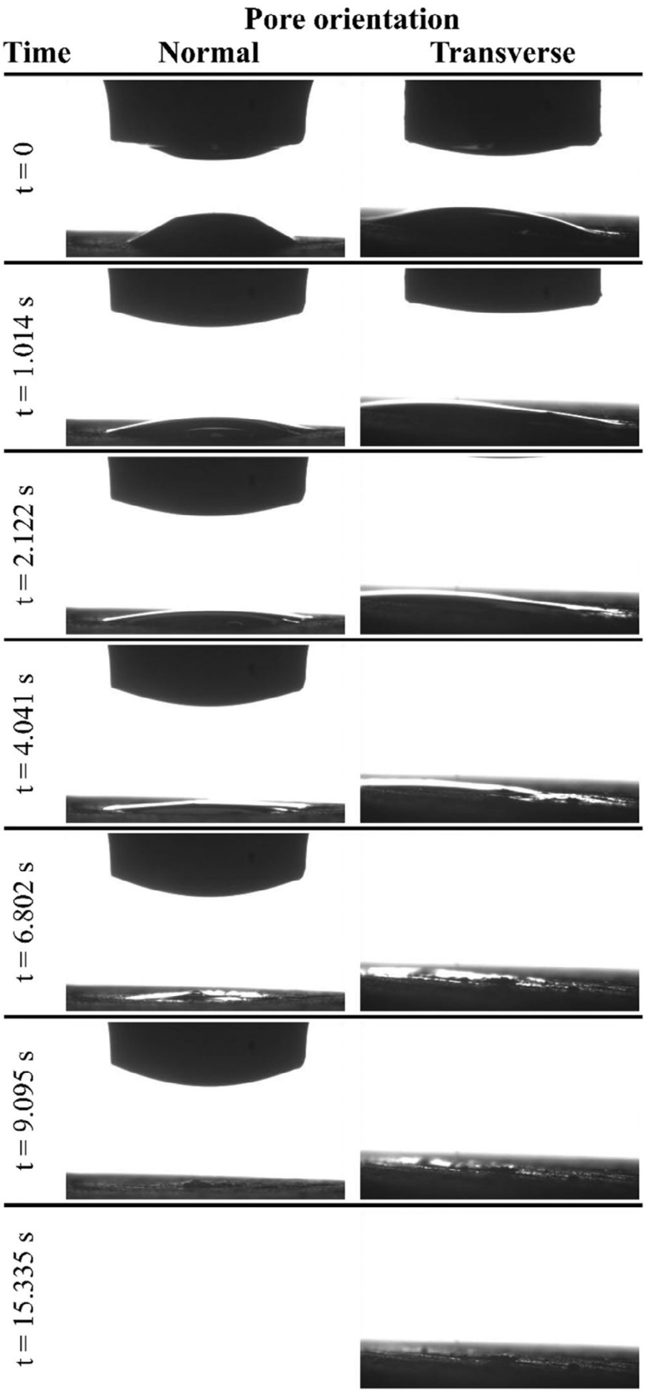

FIGURE 1 Timeline for complete adsorption of liquid beeswax in heated charcoal at 70°C.

or BET method), is limited to nanosized micropores, mesopores and macropores below 50 nm (as per the International Union of Pure and Applied Chemistry or IUPAC definition). It was not deemed an appropriate method to measure the large pore structure required for the PCM storage application in this work.

Thermal effusivity measurements were obtained using a modified transient plane source (MTPS) sensor from C-Therm. Tests at 55°C were performed in a temperature controlled environmental chamber. The measured thermal effusivity (E) were subsequently converted to thermal conductivity (λ) based on density (ρ) and specific heat (C_p) values with Equation (2).

$$\lambda = \frac{E}{\rho C_p}. \tag{2}$$

The density (ρ) value required for the calculation of thermal conductivity (λ) was obtained by dividing the mass of a charcoal piece by its volume. The mass was obtained with a laboratory analytic balance. The volume of the highly porous charcoal piece was obtained by 3D scanning through photogrammetry.

3 | RESULTS AND DISCUSSION

Experiments with molten beeswax and heated charcoal demonstrated a strong capillary adsorption. The complete adsorption of the beeswax within the pore structure occurred repeatedly in less than 10 s (Figure 1), with a slight variation depending on the orientation of the pore structure. A minimum of three tests per orientation were performed. As expected, adsorption was accelerated when beeswax was deposited on top of surfaces with normal orientation in regard to the pore structure, compared to faces presenting a transverse section of the pore structure. Further experiments were performed on ground

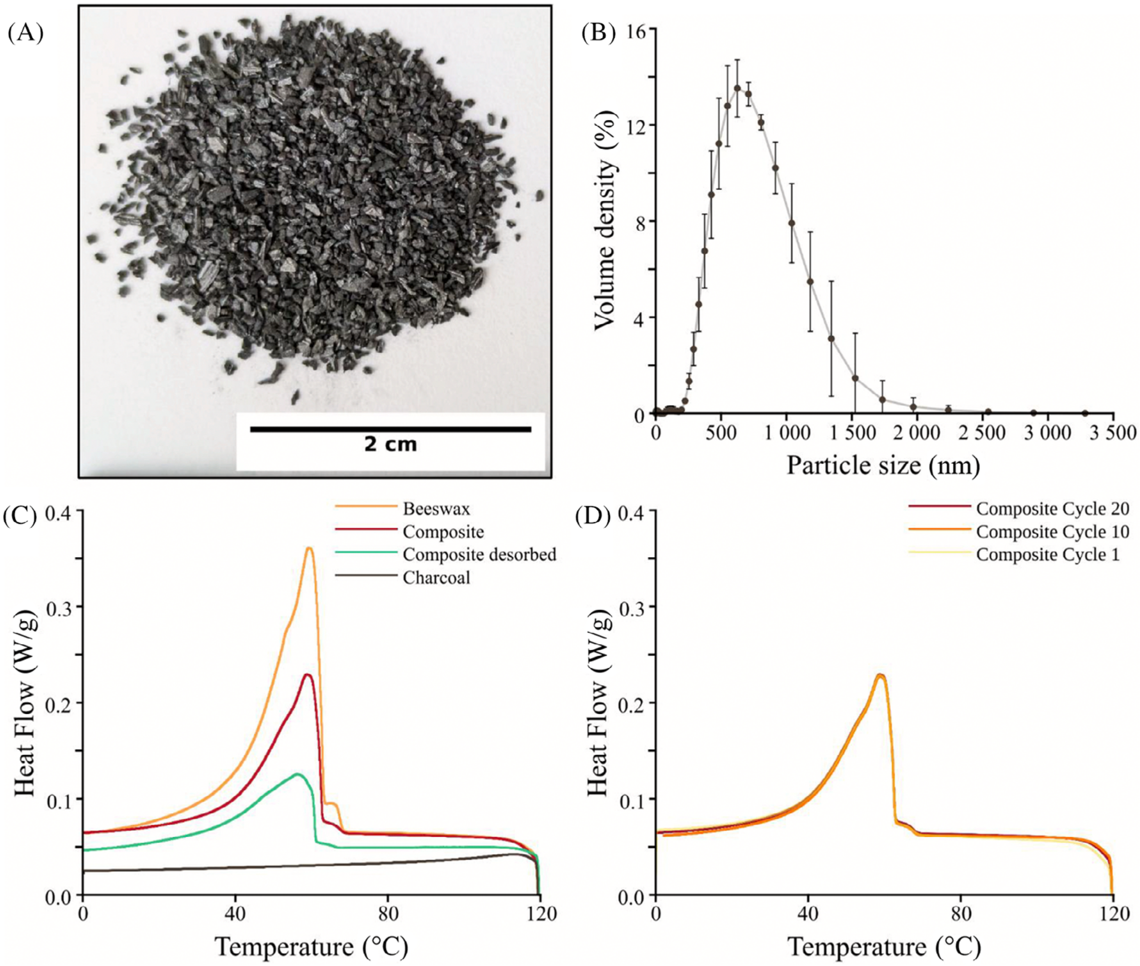

FIGURE 2 (A) Photograph of charcoal particles after grinding; (B) the resulting particle size distribution of ground charcoal; (C) the thermal properties of the composite and its constituents as obtained by a differential scanning calorimeter (DSC); and (D) the thermal stability of the composite after 1, 10, and 20 heating and cooling cycles.

charcoal with an average particle size of 0.68 mm (Figure 2A,B). The porous nature of the charcoal samples limits the applicability of the contact angle method, which was developed for flat non-porous surfaces, and as such, only general trends should be assessed from this result.[21]

SEM confirmed that the beeswax is adsorbed within the pore structure of the charcoal instead of only on the surface (Figure 3). The surface of charcoal particles after grinding is covered with small grinding residues (Figure 3A). Scanning images into the pores (Figure 3B,C), we found that beeswax seeped into the pore structure. In addition, the mass uptake of beeswax was similar for ground charcoal particles (Table 1) and centimetre-sized samples. This result support the hypothesis that beeswax is absorbed in the pore structure throughout the bulk of charcoal particles.

The comparison of thermal performance between particles infused under vacuum or atmospheric pressure did not present notable deviations (Figure 4), which implies that the capillary force was the main driving force for the sorption of beeswax within the pores of charcoal.

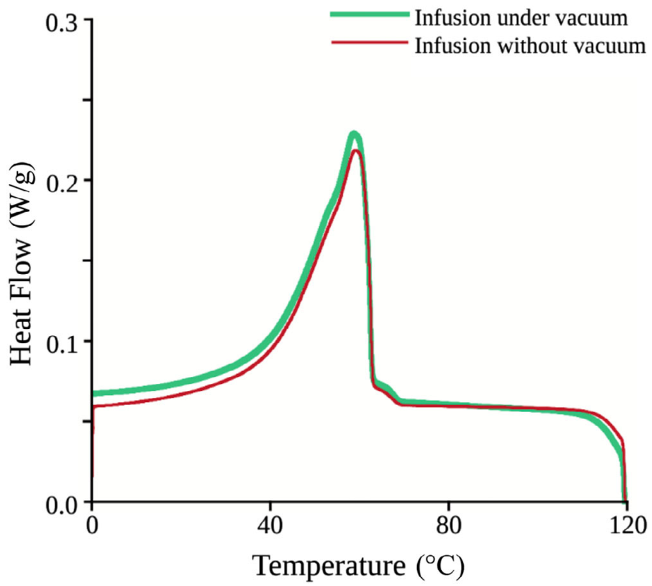

FIGURE 4 Thermal characteristics of composite infused under and without vacuum at its melting peak as obtained by a differential scanning calorimeter (DSC).

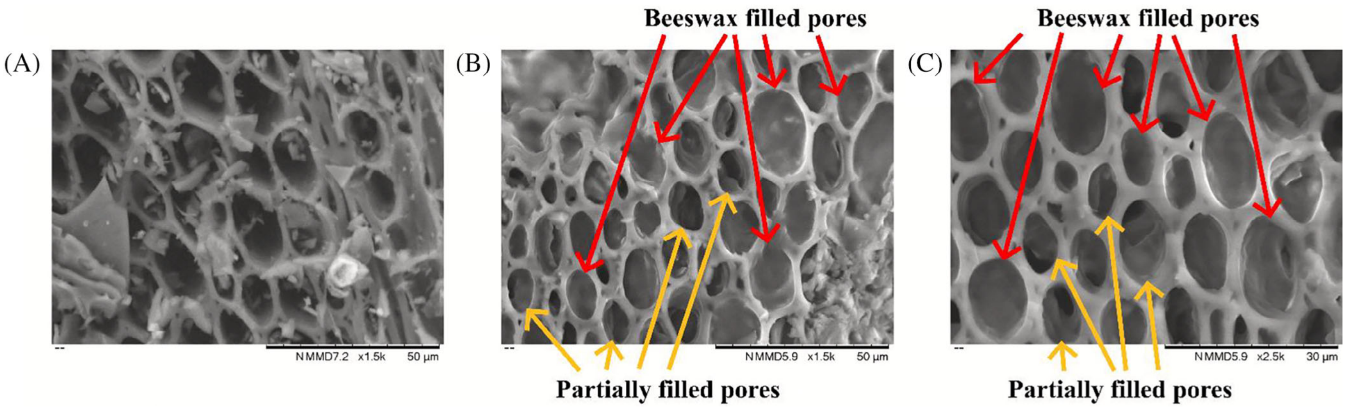

FIGURE 3 SEM images highlighting the pore structure of charcoal (A) with empty pores after grinding and (B and C) highlighting the presence of beeswax within the pore structure after beeswax infusion.

TABLE 1 Thermal properties of beeswax, the composite, and the composite after desorption.

Material	Cycle	T_m (°C)	Melting enthalpy (J g^{-1})	Mass fraction of beeswax (%)
Beeswax	1	59.0	145.7	
	2	59.4	145.3	
	Average	59.2	145.5	100
Composite	1	59.1	77.74	
	10	59.1	77.91	
	20	59.2	77.85	
	Average	59.1	77.83	53.5
Composite after desorption	1	56.3	48.83	
	10	56.4	48.63	
	20	56.4	48.86	
	Average	56.4	48.77	33.5

To estimate the capillary pressure of the charcoal–beeswax system, an average pore radius of 4.8 μm was calculated based on all the pores visible in Figure 3C and a surface tension of 3.3×10^{-2} N m^{-1} for beeswax was obtained from the literature.[22] Determining the beeswax contact angle on porous charcoal was not straightforward. Contact angle measurements were extracted from the initial adsorption experiments. Following the strong interaction between both materials, the maximum contact angle measured right after beeswax is deposited on the surface was 38° and it quickly dropped to 0° after complete adsorption. The capillary pressure for contact angles of 38° and 0° were calculated respectively to be 1.1×10^4 N m^{-2} and 1.4×10^4 N m^{-2}. This high value supports our observation that beeswax is strongly adsorbed within the charcoal pore structure and that this adsorbed beeswax should not significantly leach during thermal cycles.

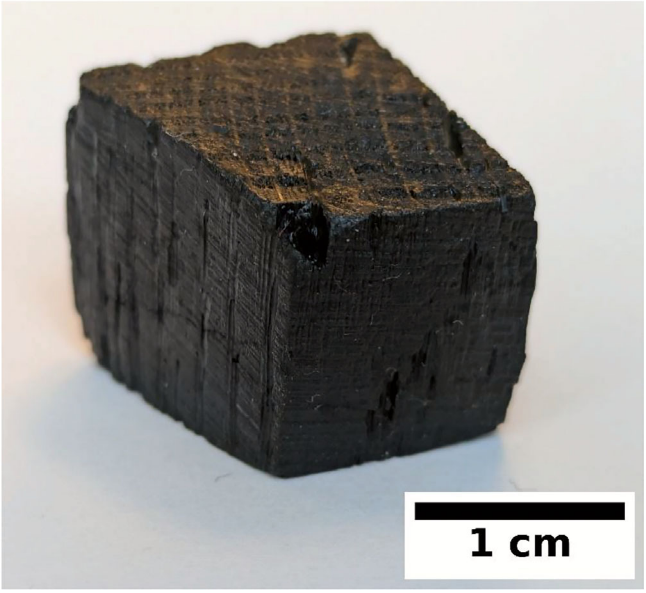

FIGURE 5 Centimetre-size beeswax infused charcoal composite piece used for the scale-up validation experiment.

The thermal behaviour of charcoal, beeswax, and the composite particles are presented in Figure 2C and in Table 1. Results of the cycling experiments, included in the testing procedure of the composite particles, demonstrated that the composite material maintains its thermal characteristics well over 20 heating and cooling cycles without any visible loss in performance (Figure 2D and Table 1). Experiments were not extended beyond 20 cycles due to the high reproducibility of the thermal behaviour between cycles 1, 10, and 20. Based on the melting enthalpy of beeswax, the mass fraction of beeswax absorbed within the composite sample can be estimated at 53.5%.

Following a leaching test, the particles presented repeatable thermal behaviour but a reduced beeswax mass fraction of 33.5% and a slightly lower melting temperature (Table 1). This reduction of the melting temperature is indicative of a preferential leaching of some of the higher melting temperature compounds of beeswax with regard to the remaining compounds. It should be noted that the leaching tests are performed at 110°C which is well outside the expected operating range for beeswax PCM. Operating under milder conditions should reduce leaching.

To confirm that the production of charcoal and beeswax based composite PCM at a larger scale is possible, depending on the application, a prismatic piece of charcoal with flat outer surfaces was partially dipped in molten beeswax in a furnace (Figure 5). The pore structure readily adsorbed the beeswax through capillary forces.

TABLE 2 Thermal conductivity measurements for beeswax, charcoal, and the composite.

Material (temperature)	**Thermal effusivity** **($W\ s^{1/2}\ m^{-2}\ K^{-1}$)**		**Thermal conductivity** **($W\ m^{-1}\ K^{-1}$)**	
Orientation	**Average**	**St. dev.**	**Average**	**St. dev.**
Beeswax (23°C)	833.7	3.8	1.49	0.014
Charcoal (23°C)				
Axial	207.1	3.8	0.098	0.004
Transverse	164.5	2.1	0.062	0.002
Composite (23°C)				
Axial	340.5	3.0	0.237	0.004
Transverse	323.0	1.1	0.213	0.001
Composite (55°C)				
Axial	433.5	1.7	0.384	0.003
Transverse	408.4	3.5	0.342	0.006

Additional beeswax was added until the pore structure reached saturation. The initial and final masses were recorded to be 3.33 and 5.37 g, respectively. The beeswax mass fraction was thus calculated to be ~61.2%, slightly higher than the mass fraction absorbed by the ground particles. This variation can be attributed to natural variations in the pore structure.

As expected, filling most of the pores of charcoal with beeswax instead of air improved its apparent thermal conductivity (Table 2), making the composite more interesting for heat storage applications, with a notable 60% increase within the melting zone at 55°C. The deviation from the mixing rules observed in the thermal conductivity results implies incomplete filling of the pores and residual air within the structure, preventing a higher increase of the thermal conductivity.

The granular nature of charcoal pellets or ground charcoal particles could allow for applications for heat recovery where air is flowed through a bed of PCM composite with minimal pressure drop.[23–25] Following Ergun's equation, describing the pressure drop of a packed bed column, the size of the PCM pellets could be tailored as to choose between a faster heat recovery with smaller particles or a lower pressure drop with larger pellets. The common nature and low cost of the materials, as well as the ease of manufacturing of the PCM base makes them interesting for large scale applications.

4 | CONCLUSION

This article proposes the concept of a low cost, biosourced, biodegradable, and scalable system for heat storage with a PCM and a porous absorbent. This concept is demonstrated with a novel combination of natural beeswax as the PCM and hardwood charcoal as the porous adsorbent. Adsorption of molten beeswax within the charcoal structure is driven by the strong capillary pressure induced by the naturally occurring porous structure of charcoal. Instead of aiming for maximum performance, this system targets a balanced approach with minimal environmental impact and easy end-of-life disposal. This solution has been demonstrated both with sub-millimetre particles and centimetre-scale pellets. Heat storage solutions with air flowing through a bed of pellets or particles PCM composite can be envisioned as a possible direct application of this development. Future work will have to focus on a technology demonstrator and on the biodegradability of the composite system. In addition, further experiments will be needed to assess the effect variations between batches of charcoal, including the impact of original wood or biomass type, and deviations over extended cycles.

AUTHOR CONTRIBUTIONS

David Brassard: Conceptualization; investigation; writing – original draft; methodology; validation; visualization; writing – review and editing; formal analysis; data curation; resources; project administration. **Adya Karthikeyan:** Writing – original draft; conceptualization; investigation; methodology; validation; writing – review and editing; formal analysis; resources; project administration. **Jason R. Tavares:** Funding acquisition; supervision; project administration; writing – review and editing.

FUNDING INFORMATION

This work was supported by the Natural Sciences and Engineering Research Council of Canada [grant number RGPIN-2019-05378].

CONFLICT OF INTEREST STATEMENT

The authors have no conflicts of interest to declare that are relevant to the content of this article.

PEER REVIEW

The peer review history for this article is available at https://www.webofscience.com/api/gateway/wos/peer-review/10.1002/cjce.70020.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are openly available in Dataset for the article 'Natural Wax Supported by Microporous Biochar to Create a Stable Phase Change Material' at https://borealisdata.ca/dataset.xhtml?persistentId=doi:10.5683/SP3/E93E3U, reference number https://doi.org/10.5683/SP3/E93E3U.

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How to cite this article: D. Brassard, A. Karthikeyan, J. R. Tavares, *Can. J. Chem. Eng.* **2026**, *104*(1), 44. https://doi.org/10.1002/cjce.70020