Biomass derived carbon aerogel as an ultrastable skeleton of form-stable phase change materials for efficient thermal energy storage

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Abstract
Direct conversion of biomass to carbon aerogel provides a promising approach to developing supporting material for phase change materials (PCMs). In present work, carrot and pumpkin derived carbon aerogels (CCA and PCA) were fabricated via a hydrothermal and post-sintering process. N2 adsorption-desorption isotherms were used to evaluate the specific surface area and pore distribution of the carbon aerogels. It showed that the carrot carbon aerogel sintered at 800 °C (CCA800) possessed a specific surface area of 33.80 m²·g⁻¹, which is separately 168%, 165%, and 287% higher than that of carrot carbon aerogel sintered at 1000 °C (CCA1000, 12.59 m²·g⁻¹), pumpkin carbon aerogel sintered at 800 °C (PCA800, 12.77 m²·g⁻¹), and pumpkin carbon aerogel sintered at 1000 °C (PCA1000, 8.74 m²·g⁻¹). Owing to its porosity, the carbon aerogels with a high loading content of palmitic acid (PA)/thiol-ene resin (TE) composite as a PCM without leakage. A thermal conductivity enhancement of 60.5% was achieved by 50PA/TE@PCA1000 compared with 50PA/TE, and DSC result showed its latent heat of 88.26 J/g. The excellent properties of PCMs composites lead to a promising application foreground.

1. Introduction
The growing greenhouse gas emissions and resource constraints are driving all fields of human-being to improve the utilization of energy [1–3]. The thermal energy storage system usually charges at a peak time and release thermal energy in off-peak time. In that case, the energy storage systems will consume less energy during peak time and decrease greenhouse gas emissions, such as building heat preservation, solar collectors, and ice-storage air conditioning system [4–7]. Latent heat storage based on the phase change materials (PCMs) plays an important role among the thermal energy storage systems owing to their high density of energy storage and stable chemical structure [8]. Additionally, organic PCMs (paraffin, fatty acid and its derivatives, etc) have received much attention over inorganic PCMs (molten salts, crystalline hydrated salts, metals and their alloys, etc) because of the competitive pricing, less undercooling, and outstanding reversibility [8–11]. Among various organic PCMs, palmitic acid (PA) has gained remarkable attention due to its relatively high latent heat density, non-toxic, and chemical stability [12–14].

To store and release the latent thermal energy, lots kinds of resins are used to encapsulate the organic PCMs and prevent its leakage, such as high density polyethylene, silicone rubber, and polyethylene terephthalate [15–17]. However, these methods will increase the thermal resistance of PCMs. Recently, impregnating PCMs into inert porous supporting materials is a promising practice, to maintain a solid-state even the temperature is above the melting transition temperature of the PCMs [15, 18–21]. Carbon aerogel (CA) has aroused wide attention due to its large specific surface area, high porosity and good thermal conductivity [7, 22–24], which can effectively eliminates the thermal resistance and leakage of PCMs. However, the methods of preparation of the porous supporting materials used in these investigations were complex, and the existing composite PCMs
consisted of PCMs and porous supporting materials cannot perfectly prevent the PCMs from leaking during the phase transition process [25, 26].

The objective of this study is to prepare a biomass-derived supporting material for form-stable PCMs with low leakage rate and good thermal conductivity, and make the form-stable PCMs one of the candidates of thermal management system to be used in electronic fields. In present work, carrot and pumpkin derived carbon aerogels (CCA and PCA) were employed as supporting materials to prepare form-stable PCMs and improve the thermal conductivity of PA. The cooperation between carbon aerogels and thiol-ene resin (TE) led to perfectly preventing the PCMs from leaking during the phase transition process. The resin can easily be impregnated in carbon aerogels owing to its low viscosity, and it also has a suitable curing temperature. The involvement of carbon aerogels led to outstanding improvement in thermal conductivity of PA/TE composite and amended the leakage phenomenon of the composite because of high thermal conductivity and porosity of carbon aerogels. Meanwhile, the PCMs composite with pumpkin-derived carbon aerogel (sintering temperature of 1000 °C) achieved the highest thermal conductivity. This study may expand the applications of form-stable PCMs as thermal management systems.

2. Experimental

2.1. Materials
Fresh pumpkin and carrot were bought from local markets. 1,3-Divinyltetramethyldisiloxane (purity 98%) was purchased from acr GmbH (Karlsruhe, Germany). 2,2’-(Ethylenedioxy) diethanethiol (purity 95%) was purchased from Sigma Aldrich Company (Mexico). Dicumyl peroxide (purity 98%) was purchased from Sigma Aldrich Company (Italy). 1,1,1-Trimethylopropane(3-mercaptopropionat) was purchased from Bruno Bock Chemische Fabrik GmbH & Co. KG (Marschacht, Germany). Ethanol and palmitic acid (PA) were obtained from Aladdin agent corporation. The agents used without further purification.

2.2. Preparation of carbon aerogel
The scheme of preparation of carbon aerogels is shown in figure 1. Firstly, the rind, seeds, and soft pulp from pumpkin (the rind from carrot) were removed. Then the flesh of the pumpkin (or carrot) was cut into an appropriate shape and dimension, and then placed into a Teflon-lined stainless steel autoclave. The autoclave was heated at 180 °C for 10 h. The pumpkin (carrot) hydrogels were immersed in hot water (around 60 °C) and ethanol for 2 days, respectively, to remove soluble impurities. The pumpkin aerogel (or carrot aerogel) was obtained via lyophilisation process. Finally, the pumpkin aerogels (or carrot aerogel) were fully converted to pumpkin carbon aerogel (or carrot carbon aerogel) through sintering in a tube furnace at 800 °C or 1000 °C for 90 min in argon atmosphere. The products were marked as PCA800, PCA1000, CCA800 and CCA1000 according to the sintering temperature of carbon aerogel.
2.3. Preparation of form-stable phase change materials

Firstly, 10 g 1,3-Divinyltetramethyldisiloxane, 5.872 g 2,2′-(Ethylenedioxy)diethanethiol, and 5.706 g 1,1,1-Trimethylolpropane (3-mercaptopropionat) were added into a beaker, they were treated by ultrasonic at 80 °C till it became a homogeneous solution. Secondly, 21.578 g PA (50 wt%) and 0.647 g dicumyl peroxide were added into the beaker and treated by ultrasonic for another 30 min. The previous mixture was named as PA/TE solution. Then the PA/TE solution was poured into a preheated Teflon mold and cured at 120 °C for 4 h, palmitic acid was sealed in thiol-ene thermosetting resin after curing and this product was labelled as 50PA/TE. Meanwhile, the CCA (or PCA) and PA were put into a beaker and then they were placed in a vacuum oven, and heated to 120 °C for 30 min after holding the pressure at 0.1 bar for 10 min. The samples were marked as PA@CCA800, PA@CCA1000, PA@PCA800, and PA@PCA1000. Finally, the cooled PA/TE solution and CCA (or PCA) were put into a beaker and placed in a vacuum oven, and then heated to 120 °C for 4 h after holding the pressure at 0.1 bar for 10 min. The samples were separately labelled as 50PA/TE@CCA800, 50PA/TE@CCA1000, 50PA/TE@PCA800, and 50PA/TE@PCA1000. The scheme of preparation of 50PA/TE@CA composites is shown in figure 2.

2.4. Characterization

X-ray diffraction (XRD, Ricoh, D/max2550VB/PC) was used to characterize the crystalline structure of the carbon aerogel (CA). Laser Micro-Raman Spectrometer (Renishaw, Invia reflex) was used to examine the graphitization of the carbon aerogel. The morphologies of the as-synthesized carrot aerogels, pumpkin aerogel and their corresponding carbon aerogels were determined by scanning electron microscopy (SEM, Hitachi, S4800). The specific surface area and pore distribution were measured by N₂ absorption-desorption isotherms (Micromeritics ASAP2020 Analyser). DSC analysis was performed on a differential scanning calorimeter (DSC, TA Instrument, DSC Q-2000). Thermal conductivity (λ) was calculated by equation (1):

\[ \lambda = \alpha C_p \rho \]  

where \( \alpha \), \( C_p \) and \( \rho \) are the thermal diffusivity, specific heat capacity and density of specimen, respectively. \( \alpha \) of samples (12.7 mm × 12.7 mm × 1 mm) at 25 °C were measured by laser flash method utilizing a NETZSCH LFA 447 NanoFlash instrument. \( C_p \) was determined by a TA Instrument Q-2000 differential scanning calorimetry. The infrared camera (Optris, PI 400) was used to measure the temperature curves of the samples.

3. Results and discussion

3.1. Morphology of carbon aerogels

As shown in figures 3(a) and (d), the SEM images of carrot aerogel and pumpkin aerogel present an interconnnected 3D networks that are mainly composes of a typical layer structure. The interlayer distance is
estimated to be in the range of 5–10 μm, the oriented hierarchical 3D structure was formed by the growth of ice crystals from the bottom to the top, and the pumpkin flesh and carrot flesh were discharged to the boundary between the ice crystals during the freezing-casting process. Besides, aerogels show a dark brown colour (figure 1), indicating that low-temperature hydrothermal treatment cannot fully convert the tissue of pumpkin or carrot to carbon. Further sintering treatment of pumpkin aerogel and carrot aerogel at 800 °C or 1000 °C under an argon atmosphere leads to the evaporation of volatile organic species and the formation of PCA and CCA (figure 1). Figures 3(b), (d), (e) and (f) show the micro-structure of CCA800, CCA1000, PCA800 and PCA1000, respectively. Compared with carrot aerogel and pumpkin aerogel, the carbon aerogels show similar interconnected structure. However, a large number of pores appeared on the carbon nanosheets and formed a porous structure, and the higher sintering temperature further causes damages on the porous structure, which leads to lower densities of CCA1000 and PCA1000 than that of CCA800 and PCA800, respectively.

As shown in figure 4, specific surface area and pore distribution of the as-prepared carbon aerogels are characterized by N2 absorption-desorption isotherms. The Brunauer–Emmett–Teller (BET) specific surface area of CCA800, CCA1000, PCA800, and PCA1000 are 33.80, 12.59, 12.77, and 8.74 m² g⁻¹, respectively. The specific surface area of carbon aerogels with sintering temperature of 800 °C higher than that of carbon aerogels with sintering temperature of 1000 °C because the high temperature destroyed the porous structure of carbon aerogels. As shown in figure 4(b), the carbon aerogels with a low sintering temperature show more micropore and mesopore than the carbon aerogels with a high sintering temperature. The specific surface area of CA also
influenced by the micro-structure of raw materials, and the specific surface areas of CA derived from different raw materials were listed in Table 1.

![Table 1. The specific surface area of CA originated from different raw materials.](image)

| Samples     | Raw materials          | Specific surface area, m²·g⁻¹ | References |
|-------------|------------------------|-------------------------------|------------|
| Cotton2400  | Absorbent cotton       | 1.4                           | [25]       |
| CM-650      | Luffa acutangula       | 147.3                         | [27]       |
| KGA-1       | Konjac glucomannan     | 873                           | [28]       |
| CCA800      | Carrot                 | 33.80                         | This work  |
| PCA800      | Pumpkin                | 12.77                         | This work  |

The thermogravimetric curves of aerogel and carbon aerogels are shown in figure 5. The aerogel decomposed from about 200 to 700 °C, and the weight loss of carrot aerogel was 64%. The TGA curves illustrated that the flesh of pumpkin and carrot were fully converted to carbon aerogel after sintering treatment. All of the carbon aerogels show a small mass loss during the TGA analysis due to the decomposition when the temperature higher than 650 °C and the adsorption of water in the micropores and mesopores of carbon aerogels.

XRD patterns of the carrot-derived and pumpkin-derived carbon aerogels are shown in figure 6(a). In the case of carbon aerogel, two broad peaks located at 24° and 44° were observed, suggesting a poor ordering of graphene nanosheets along their stacking direction. Meanwhile, it is observed from the XRD patterns that with the increase of sintering temperature, the obtained carbon aerogels present enhanced graphitization, as...
indicated by the higher intensity of the spectra of CCA1000 and PCA1000 than that of CCA800 and PCA800, respectively. Raman spectra of these four samples were also acquired to further confirm the graphitization of the samples, as shown in figure 6 (b). The peaks of D-band and G-band located at 1330 and 1590 cm\(^{-1}\), respectively. Here D-band is related to the A\(_{1g}\) vibration mode of the disordered carbon, while G-band represents the E\(_{2g}\) vibration mode of the ordered graphitic carbon \[25\]. The defect extent of carbon materials can be directly reflected in the intensity ratio of D band and G band \((I_D/I_G)\). The lower \(I_D/I_G\) values of CCA1000 and PCA1000 indicate that the higher sintering temperature resulted in the higher extent of graphitization of the carbon aerogels.

### 3.2. Thermal properties and shape stability properties of PCM composites

High latent heat is a critical property for a PCM, and the thermal energy storage capacity of the PCMs depends on the latent heat. In this work, the latent heat of the PCMs were examined by using DSC instrument. Figure 7 shows the DSC curves of pure PA and composite PCMs. There are several kinds of shapes of DSC curves, but all of the DSC curves of PCMs are broader than that of pure PA except PA@PCA800 and PA@PCA1000, which indicates the introduction of CCA and thiol-ene broaden the temperature range of the phase transition process. The corresponding thermal performances of pure PA and composite PCMs are listed in table 2. For PA, the melting point and melting enthalpies are 62.8 °C and 197.78 J g\(^{-1}\), respectively. The melting temperature of PA@CA and 50PA/TE@CA are located in the range of 62.8 ± 2 °C, indicating that the thiol-ene resin and carbon aerogel have a slight influence on phase change temperature.

The thermal degradation of PA, thiol-ene resin, 50PA/TE, and 50PA/TE@CA are shown in figure 8. The temperature of 5% mass loss of pristine PA and thiol-ene resin are located at 213.5 and 313.8 °C, respectively. Pristine PA shows a typical one-stage decomposition process and thiol-ene resin shows a two-stage decomposition process. The introduction of carbon aerogels exhibited no influence on the decomposition process, and the residue of 50PA/TE@CCA800, 50PA/TE@CCA1000, 50PA/TE@PCA800, and 50PA/TE@PCA1000 after decomposition process are 4.3%, 4.2%, 8.9%, and 5.6%, respectively. According to the results of TGA and DSC, the latent heat of composite PCMs with melting enthalpy is mainly determined by the mass ratio of pure PA in the composite PCMs.

In a thermal energy storage system, the shape stability is highly important. As shown in figure 9, the carbon aerogels prevented the PCMs from leaking even the temperature over the melting point of the PCM, which resulted from the porous structure of carbon aerogels, suggesting that the involvement of carbon aerogels led to outstanding improvement in shape stability of the pristine PA and 50PA/TE composite.

| Samples           | \(\Delta H_m/\text{J g}^{-1}\) | \(T_m/°C\) | Samples           | \(\Delta H_m/\text{J g}^{-1}\) | \(T_m/°C\) |
|-------------------|-------------------------------|------------|-------------------|-------------------------------|------------|
| Pure PA           | 197.78                        | 62.8       | 50PA/TE          | 104.24                        | 65.6       |
| PA@CCA800        | 196.51                        | 62.9       | 50PA/TE@CCA800   | 94.94                         | 61.2       |
| PA@CCA1000       | 194.20                        | 64.4       | 50PA/TE@CCA1000  | 103.22                        | 60.1       |
| PA@PCA800        | 189.92                        | 62.4       | 50PA/TE@PCA800   | 88.26                         | 64.7       |
| PA@PCA1000       | 190.49                        | 62.4       | 50PA/TE@PCA1000  | 88.86                         | 60.6       |

Figure 7. DSC curves of palmitic acid and composites.

Table 2. The melting enthalpy and melting point of palmitic acid and its composites.
3.3. Thermal conductivity of the form-stable PCMs composites

The fast storage/release of heat energy by the PCMs is highly significant for a thermal energy storage system. The thermal performance of composite PCMs can be characterized by measuring the thermal conductivity and temperature-time curves of the PCMs. The dominant method of heat transfer in PCMs is heat conduction and it is necessary to enhance the thermal conductivity of PA/TE composite for fast storing and releasing thermal energy. The thermal conductivities of PA and composite PCMs are shown in figures 10(a) and (b). The thermal conductivity of PA is 0.029 $\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$, which is too low to apply in a thermal energy storage system. As shown in figures 10(a) and (b), the introduction of CA improved the thermal conductivities of PA and PA/TE significantly. The influence factors of thermal conductivities of PCM composites include the degree of graphitization of CA and the mass ratio of pristine PA encapsulated in the composite PCMs. The thermal conductivities of PA@CCA1000 and PA@PCA1000 are almost 10 times higher than that of pristine PA, the corresponding thermal conductivity are 0.245 $\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ and 0.253 $\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$. Moreover, the thermal conductivity of 50PA/TE@PCA1000 (0.149 $\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$) is 60.5% higher than that of 50PA/TE (0.093 $\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$). The low thermal conductivity of 50PA/TE@CCA1000 (0.106 $\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$) mainly attributes to the high content of PA/TE in the composite PCM, and the thermal conductivities of PA/TE@CA lower than that of PA@CA owing to higher interface thermal resistance within the composites.

The thermal energy storage efficiency could be characterized by temperature-time curves during the charging process. As shown in figures 10(c) to (h), the samples with higher thermal conductivities showed higher temperature raising rates. In conclusion, a high thermal conductivity is more efficient for charging and discharging of thermal energy from the PCMs.

Figure 8. The TGA curves of PA and composites.

Figure 9. Digital photos of PCMs composites located under 100°C at (a) $t = 0\ \text{s}$ and (b) $t = 60\ \text{s}$. 

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**4. Conclusion**

Biomass-based carbon aerogels were successfully fabricated using carrot and pumpkin as raw materials. The porous carbon aerogels with high thermal conductivity were infiltrated with palmitic acid and thiol-ene resin to prepare form-stable PCMs for a thermal energy management system. The high porosity of carbon aerogels, mainly influenced by the micro-structure of raw materials, endowed the PCMs with excellent form stability, and the thermal conductivity of 50PA/TE@PCA1000 was 60.5% higher than that of 50PA/TE without incurring a significant loss in the latent heat owing to the lightweight and high thermal conductivity of carbon aerogels. An eco-friendly approach was introduced in this work to prepare carbon aerogel, which could be potentially adopted as a promising thermal conductivity enhancer in thermal management and renewable energy utilization.

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