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# Wet Impregnation Method for Synthesis of Phase Change Composite of N-Octadecane in Porous Silica Gel

**Tien Giang Nguyen*, Minh Tam Le**
*Ho Chi Minh City University of Technology and Education, Vietnam*

*Corresponding author. *Email:* ntgiang@hcmute.edu.vn

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## ABSTRACT

Thermal energy storage is gaining more interest as people aim to utilization of solar energy. The thermal energy can be stored in the form of latent heat and used for building applications that require increasing energy. This report investigated the wet impregnation method for the synthesis of thermal storage material, namely phase change composite (PCC) for two purposes: to create a simple method for the synthesis of PCC and study behaviors of phase change material (PCM) when confined inside the pore. Particularly, the PCC was synthesized using n-octadecane and silica as PCM and porous matrix, respectively. The characterization of the product was conducted by various analytical methods: $N_2$ adsorption/desorption isotherm, differential scanning calorimetry (DSC), thermogravimetric analysis (TGA). Results showed that the n-octadecane was successfully impregnated into a silica porous network through simple steps of the wet impregnation method. The porosity analysis confirmed the confining of PCM inside matrix pores. The n-octadecane was easily kept inside pores by the capillary and surface tension forces.  without any chemical interaction. At maximum impregnation, n-octadecane accounted for 70% of the composite. This sample presented a shape-stability at up to 90 ºC, provided a latent heat of 145 J $g^{-1,}$ and was selected as optimal PCC.

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## 1. Introduction

These days, it is well-known that fossil fuel is gradually exhausted and the price of oil is in a predictable uptrend. People are eager to find substitutable energy resources with reference to non-$CO_2$ release. Solar energy is an extremely potential resource as it is abundant, renewable, and environmentally friendly. The heat from solar energy can be used for building applications such as space heating/cooling as well as hot water and electricity production [1]. However, a major drawback of solar energy is the mismatch in supply and demand periods because of its intermittence. The most intensity of the sunlight occurs during the daytime when the demand for heating is low. In contrast, the highest demand usually occurs at night or early in the morning, when the solar irradiation is low [2]. Therefore, thermal energy storage (TES) has been becoming an attractive topic.

Thermal energy storage is mainly classified into three methods: sensible, latent, and thermochemical. Their mechanisms, advantages, and disadvantages are summarized in some reviews [3]. Among the three methods, latent heat storage shows high potential for building applications because of its simple mechanism, high heat density, low cost, non-corrosion, and good thermal and chemical stability. In this method, heat is released or stored when a material changes its phase e.g., solid-solid, solid-liquid, or liquid-gas, and vice-versa. The material employed in the latent heat storage is therefore called phase change material (PCM). While the solid-solid or the liquid-gas phase change gained less interest in the past decades because of their low heat density or large volume change during the transition, most research was focused on the development of the solid-liquid phase change.  This physical phenomenon provided a relatively high latent heat in a narrow range of temperatures without large volume change. The solid-liquid phase change materials consist of organics, inorganics, and eutectics. The comparison of these materials as well as many potential and commercial PCMs were reviewed in the literature [4]. Depending on the application, one can choose an appropriate candidate.

The major drawbacks of solid-liquid PCMs come from low thermal conductivity and leakage in the liquid phase, especially in the case of organics. Many authors solved the problems by encapsulation of PCM inside polymers like high density polyethylene. This technology showed high thermal control and was believed to erupt strongly in the future despite of the current expensive cost [5]. Another solution to tackle the disadvantages of PCM was the impregnation of PCM into a porous matrix, resulting in a phase change composite. The support matrices possess higher thermal conductivity and keep PCM inside their porous network through physical interactions like capillary and surface tension forces. Several typical porous matrices have been investigated including silica, graphite, vermiculite, and perlite as they are cheap, non-toxic, and well-available. It was proved that after PCMs were impregnated into vermiculite and silica fume, the thermal conductivity of composites increased by 85% and 156%, respectively, without any leakage [6], [7]. On the other hand, it is well-known that the thermal properties of a substance inside a pore will significantly change. Accordingly, the characteristics of PCC need to be studied carefully after the impregnation process [8].

Normally, vacuum impregnation was the most used method for the synthesis of PCC [9], [10]. This method includes adding an excessive amount of PCM into a porous matrix under a vacuum atmosphere at a temperature higher than the melting point of PCM. When the impregnation process was completed, the matrix pores were filled with PCM, and the resulting composite was submerged in excessive PCM. The surplus PCM was then removed from the product by vacuum filter, followed by possible high-temperature treatment [11], [12]. The vacuum impregnation method has the advantage of thorough impregnation. However, the usage of the low pressure in the synthesis process and difficulty in removing surplus PCM were considered disadvantages which can cause difficulty from in-lab to scale-up production. In this report, we expected to evaluate a simple method for the synthesis of PCM composite, which was recently used by other scholars, namely wet impregnation or evaporative impregnation [13]-[15]. For this purpose, porous silica and n-octadecane were employed as support matrix and PCM, respectively. The effectiveness of the impregnation was discussed thoroughly. Besides, characteristics of composite such as chemical compatibility, thermal behavior, and shape-stability were also investigated.

## 2. Materials and Methods

### 2.1. Materials

Porous silica (SS350) was bought from S-Chemtech (South Korea) with physical properties from the manufacturer shown in Table 1. n-octadecane was purchased from Alfa Aesar with a purity of 99%, melting point of 28 $^{o}$C, boiling point of 317 $^{o}$C, density of 0.782 g cm$^3$. Petroleum ether (boiling point of 30-60 $^{o}$C, AR ), was bought from Xilong Chemical (China).

**Table 1.** *Physical properties of porous silica*

| Properties | Values |
|---|---|
| Surface area (m$^2$/g) | 310 |
| Pore volume (mL/g) | 1.6 |
| Average pore size (µm) | 4 |
| Oil Absorption (mL/100g) | 300 |

### 2.2. Preparation of maximum impregnated sample and determination of PCM quantity

To a solution of an excessive quantity of n-octadecane in petroleum ether, a calculated amount of silica was added. This mixture was subsequently stirred at 400 rpm for 1 h by magnetic stirring at room temperature. Then, the temperature was raised to 60 $^{o}$C while keeping the mixture stirred until all the solvent evaporated. After, the excessive PCM was removed from the resulted product by vacuum filtering with 1 µm filtered paper at 150 $^{o}$C using a heat gun (BOSCH GHG 18-60) until a granular sample formed. Finally, the composite was dried in a vacuum oven at 80 ºC for 12 h. The quantity of PCM in this sample was determined by TGA and the result showed that the PCM fraction as of 70%.

### *2.3. Preparation of PCCs by wet impregnation method*

After the possible maximum fraction of n-tetradecane in composite was determined as 70%, the PCCs were synthesized by using expected amounts of PCM (≤70%), as the procedure described below:

To a solution of a known quantity of n-octadecane in petroleum ether, a calculated amount of silica was added. This mixture was subsequently stirred at 400 rpm for 1 h by magnetic stirring at room temperature. Then, the temperature was raised to 60 ºC while keeping the mixture stirred until all the solvent evaporated. Finally, the material was dried in a vacuum oven at 80 ºC for 12 h. By this procedure, 5 PCCs with PCM amounts ranging from 23-70% were resulted in. These silica-octadecane composites were abbreviated as SOCX, with X standing for a fraction of octadecane (%).

### *2.4. Characterization techniques*

$N_2$ adsorption/desorption isotherm was employed to investigate the porosity of pristine support matrix and PCM composites, using a Microactive Tristar instrument. DSC was employed to investigate the thermal properties of materials such as heat, melting, and solidifying temperature, using a DSC 214 Polyma instrument. The measurement was conducted at a ramp rate of 5 ºC $min^{-1}$ with a $N_2$ purge gas at 20 mL $min^{-1}$. Before use, the DSC instrument was calibrated with indium as standard reference material. The latent heat was achieved by numerical integration of phase change peaks. The melting points were considered the extrapolated onset temperature of the melting peak. TGA was used to study the n-octadecane quantities of the composites, using Labsys Evo TG-DSC instrument. TGA was measured at a ramp rate of 5 ºC $min^{-1}$ with $N_2$ purge gas at a flow rate of 20 mL $min^{-1}$.

## 3. Results and discussion

### *3.1. Porosity characters*

The characteristics of impregnation effectiveness can be studied by $N_2$ adsorption/desorption isotherm measurement which gives information on the change in surface area, pore size, and pore volume of porous matrix. Fig.1 shows the pore size distribution and Table 2 shows the porosity parameters of pristine silica and the composites. In detail, Brunauer-Emmett-Teller (BET) method was used to achieve surface area while the Dollimore and Heal (DH) method, which accounts for the pore size distribution of mesopore, was employed for calculating pore size distribution. As seen in Fig.1, the pore size of pristine silica distributes from 1 to 46 nm in radius with an average size of 12.24 nm, accompanied by a surface area of 278 $m^2$ $g^{-1}$. The impregnation of n-octadecane at less than 50% made a deep decrease in surface area e.g., from 278 to 23 $m^2$ $g^{-1}$ for SOC50. The decline in the surface area of the composite was attributed to the occupation of pore space by n-octadecane as confined in matrix pores. It is noted that the surface area and pore volume in Table 2 were calculated relative to composite mass. This also reduced the surface area of silica, as a fraction of silica in composite decreased compared with sole silica. However, considering the fractions of PCM and the decline degree in surface area, we concluded that the successful impregnation of octadecane into matrix pores was the main reason causing a decline in surface area. The area decrease was followed by an increase in average pore radius from 12.24 to 16.29 nm at 23 and 33% loading. The saturated PCM amount ($\phi_S$) impregnated into a porous material could be calculated by the equation (1) [16] below:

$$\phi_S = \frac{V_p.\rho.\mathrm{W}_{\mathrm{PM}}.100}{\mathrm{W}_{PM} + Vp.\rho.\mathrm{W}_{\mathrm{PM}}} \tag{1}$$

Where $V_p$ is the specific pore volume of the porous silica material, ρ is the density of n-octadecane (0.77 g/$cm^3$), $W_{PM}$ is the weight of the porous silica material. Assuming that using 1 g of the porous silica material ($W_{PM}$ = 1 g), Vp is 1.69 $cm^3$/g, and ρ = 0.77 g/$cm^3$, the $\phi_S$ is calculated to be approximately 56%. Therefore, at 23 and 33%, the increase in pore size was due to a filled order of matrix pores by PCM, in which smaller pores were filled first while larger pores were partially filled.

This was confirmed by the pore size distribution curves of SOC23 and SOC33 with a strong decrease in dVp/drp at a pore radius of less than 10 nm compared with pristine silica, while at pore size higher than 10 nm, the dVp/drp value remained significantly.

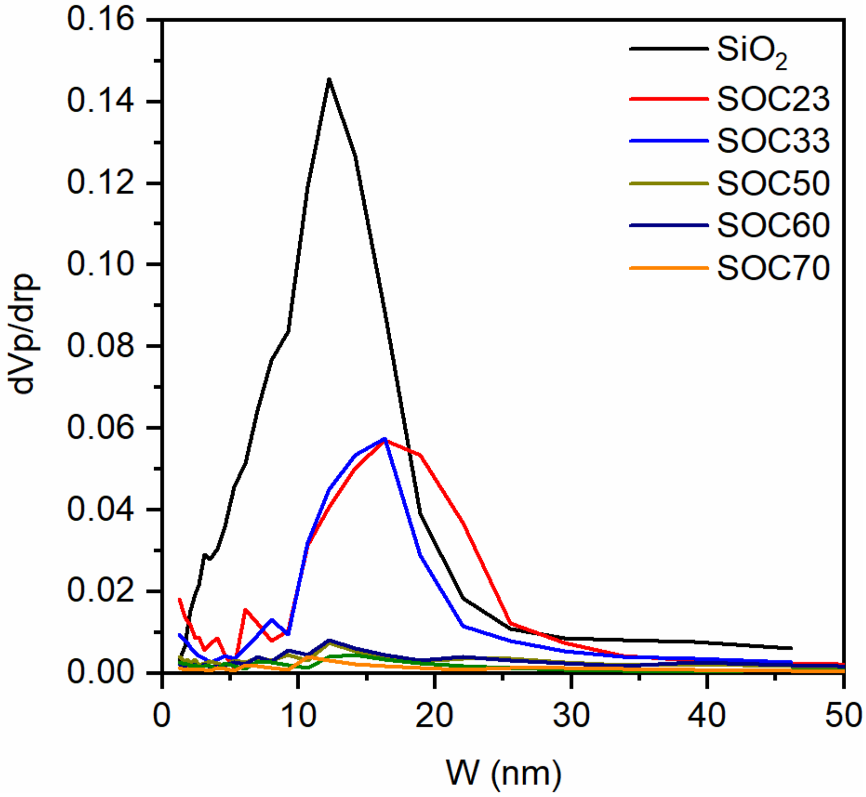

**Figure 1.** *Pore size distribution of silica and PCCs.*

**Table 2.** *Porosity information of silica and PCCs*

| Samples | Surface area (m$^2$ g$^{-1}$) | Pore volume (cm$^3$ g$^{-1}$) | Average pore radius (nm) |
|---|---|---|---|
| Silica | 278 | 1.69 | 12.24 |
| SOC23 | 135 | 0.87 | 16.29 |
| SOC33 | 91 | 0.66 | 16.29 |
| SOC50 | 23 | 0.16 | 12.24 |
| SOC60 | 20 | 0.16 | 12.24 |
| SOC70 | 9 | 0.071 | 10.65 |

At 50% loading, the nearly flat curve of pore size distribution at low dVp/drp values suggested that almost the pores of silica were filled with PCM although we cannot rule out the presence of PCM outside the pores. This was in good agreement with the theoretical fraction of n-octadecane to fill all the pores as of 56%. The decline of pore volume from 1.69 m$^3$ g$^{-1}$ (pristine silica) to 0.16 m$^3$ g$^{-1}$ (SOC50) confirmed the pore filling by PCM at 50% loadings. At higher PCM fractions e.g., 60, 70%, the matrix pores were filled more thoroughly and the excessive PCM dispersed on the matrix surface, as confirmed by further pore volume and surface area reduction, e.g. the pore volume decreased to 0.096 m$^3$ g$^{-1}$ and surface area decrease to 9 m$^2$ g$^{-1}$, in case of SOC 70.

### *3.2. Thermal stability of PCCs*

By the wet impregnation method, we can simply synthesize PCCs. One more important advantage of this method is that the PCM quantities in the composites were easily controlled with no effort to remove excessive PCM. With the usage of solvent to dissolve the PCM, the viscosity of the PCM was reduced and the PCM easily absorbed regularly into the silica matrix. The thermogravimetric analysis confirmed the expected PCM fractions in the PCCs (Fig.2). Indeed, it was calculated from the TGA that the practical PCM quantities of PCCs with 23-70% were 22.5, 34.1, 49.2, 60.5, and 71.0%, respectively, which were closed to the prepared values. Regarding thermal stability, pure n-octadecane showed mass loss within a temperature of 137-206 °C, which is consistent with previous studies [17], [18]. Interestingly, the PCCs exhibited mass loss at greater temperature range of 158-228 °C, indicating capillary and surface tension force could lower the thermal degradation of Oct. It could be stated that the thermal stability of Oct was enhanced as combined with the silica material. Similar phenomenon was also observed when Oct was impregnated in other supports such as kaolinite [17] and $MnO_2$ [18].

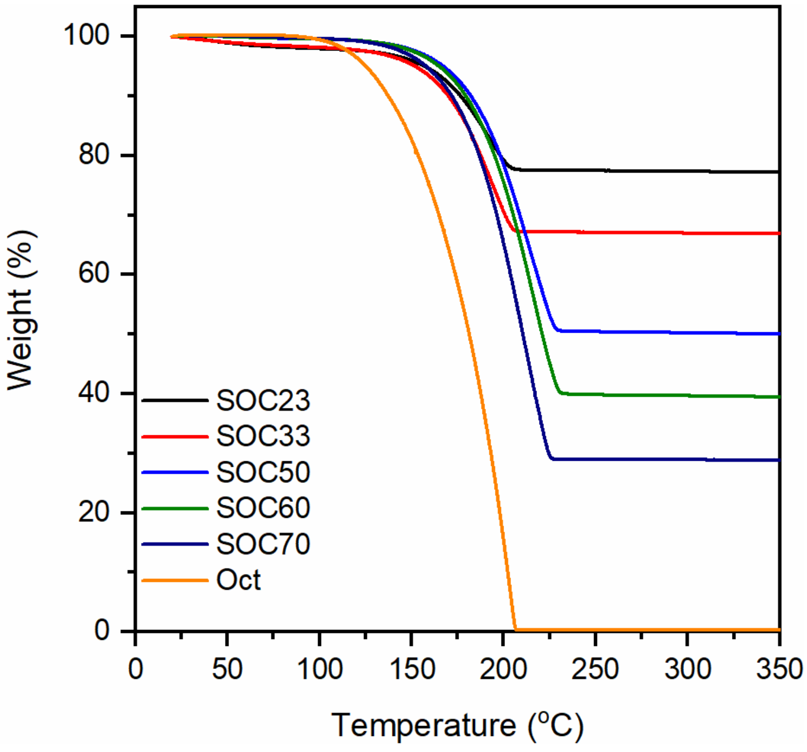

**Figure2.** *TGA curves of PCCs and n-octadecane*

### *3.3. Phase change properties*

DSC analysis gives important information about the thermal properties of PCM composite such as heat value and phase change temperature. DSC curves of n-octadecane and composites are shown in Fig.3, followed by their latent heat and melting points shown in Table 3. The melting temperature of pure n-octadecane was at 27.47 °C. When impregnated in silica pores at less than 50% however, the n-octadecane melted at a lower temperature, e.g., 13.83°C at 23 %. The shift to lower phase change temperature of PCM when being inserted into pores was due to strain in the molecular structure of PCM inside pores [8]. At the lowest PCM loadings, the smaller pores of silica were filled first. At composites with PCM fractions from 60 to 70%, DSC curves appeared second melting peaks which melting temperatures approached that of pure n-octadecane. These PCM quantities were higher than the needed PCM quantity for saturating silica pores (56%). Therefore, the appearance of the second peak from 60% was attributed to the n-octadecane outside the host pores. Since the excessive PCM dispersed on the silica surface without strain in structure or interaction with the pore surface, it melted at a temperature similar to that of the bulk n-octadecane.

The latent heat of n-octadecane was measured and gave a value of 231 J $g^{-1}$. The experimental heat of the composites increased as PCM content increased, with the largest value of 145 J $g^{-1}$ for SOC70. The second column of Table 3 presents the ideal heats of PCM composites which were calculated by the mass ratio of n-octadecane in the composites. We can see that all of the experimental heats are lower than those of ideal heats. Nomura et al. [8] found that the formation of a “non-freezing layer” which acts physically as part of the pore wall resulted in the falling of phase change heat. Furthermore, porous materials with smaller pore sizes lead to an increase in the fraction of the non-freezing layer and consequently cause stronger reduction in latent heat. In this study, at low PCM loadings where almost PCM stayed inside matrix pores, the difference between experimental and ideal heats value becomes largest. For example, the experimental and ideal heats of SOC23 were 27 and 53 J g$^{-1}$, corresponding to a difference of 49.2%. It is reminded that at low PCM loadings, the smaller pores of silica were filled first. This could explain the largest decrease in melting heat. As the PCM quantities increase, the difference became narrower. Especially from 60% where excessive PCM was distributed on the matrix surface, the experimental melting heat approached much nearer to that of the ideal value e.g., at 70% loading, the difference between experimental and ideal heat was of 10.3%.

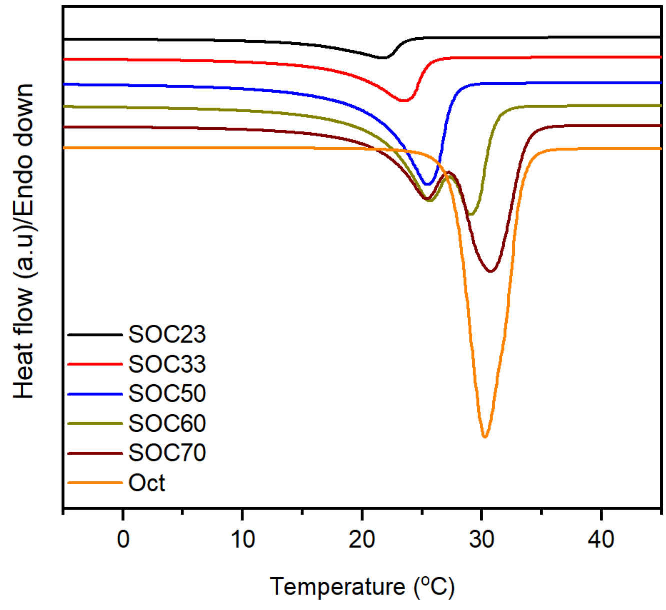

***Fig.3.*** *DSC curves of PCCs and n-octadecane*

***Table 3.*** *Thermal characters of PCCs and n-octadecane*

| Samples | Ideal heat (J g$^{-1}$) | Experimental heat (J g$^{-1}$) | Melting temperature (°C) | |
|---|---|---|---|---|
| SOC23 | 53 | 27 | 13.83 | - |
| SOC33 | 76 | 46 | 17.82 | - |
| SOC50 | 115 | 82 | 20.76 | - |
| SOC60 | 138 | 114 | 20.84 | 26.27 |
| SOC70 | 161 | 145 | 20.94 | 27.02 |
| Oct | 231 | 231 | - | 27.47 |

### *3.4. Shape-stability test of optimal composite*

For a good PCM composite, we considered latent heat and shape-stability main criteria. As a composite with the highest latent heat, SOC70 was chosen for the shape-stability test. This experiment was conducted by placing the sample at desired temperatures for 1 h and their digital photos are shown in Fig.4. The SOC70 sample existed in dried powder form over all tested temperatures even though these values were higher than the melting point of n-octadecane. As an alkane with simple C-H bonds, n-octadecane showed no chemical interactions with silica materials [19], [20]. This result indicated that the capillary and surface tension force firmly retained Oct inside $SiO_2$ pores, preventing the leakage of melted Oct. Consequently, SOC70 was selected as the optimal PCM composite. This material possessed latent heat of 145 J g$^{-1}$. Furthermore, this material can be used for applications at high temperatures (up to 90 °C) without damaging its properties.

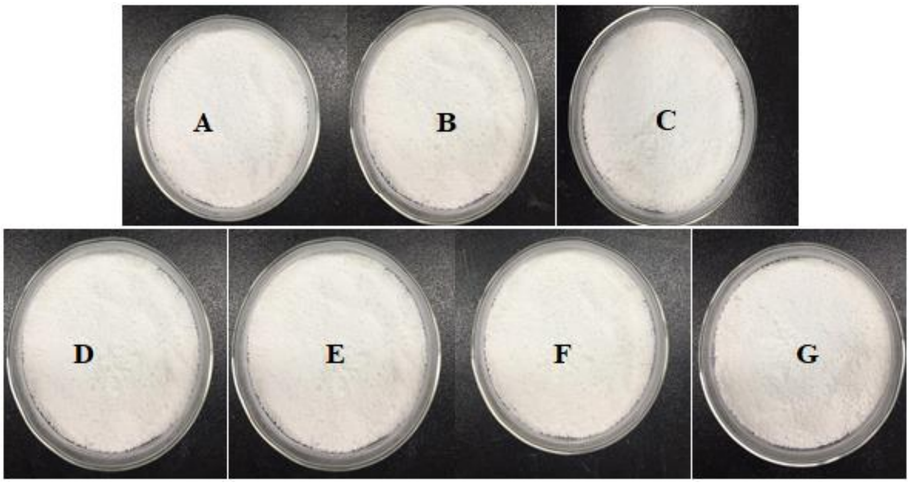

***Fig.4.*** *SOC70 sample at A (room temperature) and after 1 h at B (40 °C), C (50 °C), D (60 °C), E (70 °C), F (80 °C), G (90 °C)*

## 4. Conclusions

PCM composite consisting of n-octadecane and porous silica was successfully synthesized by the wet impregnation method. $N_2$ adsorption/desorption isotherm analysis of composites confirmed the presentation of PCM inside silica pores. The PCM was easily kept inside matrix pores by physical interactions like capillary and surface tension forces without change in the nature of the PCM and porous matrix. The confining of PCM inside the meso-pore caused a significant change in the thermal properties of PCM e.g., the reduction of melting temperature and latent heat. These changes were attributed to the interaction of PCM and pore wall, strain in the molecular structure of PCM, and non-freezing layer. At maximum impregnation, the n-octadecane quantity accounted for 70%. This sample presented good shape stability without leakage at 90 °C and a latent heat value of 145 J $g^{-1}$. Accordingly, this PCM fraction was selected as the optimal sample. By the wet impregnation method, the quantity of PCM in the composite was easily controlled. The wet impregnation method requires neither expensive apparatus nor a complicated design of the experiment. In addition, the used solvent could be collected during the evaporation process by simple condensation tools. Thus, it can be reused as well as the environmental impact is minimized. We highly recommend this method for massive production.

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## Conflict of Interest

The authors declare no conflict of interest.

## Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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**Nguyen Tien Giang** got his bachelor (2012) and master degree (2015) at the department of analytical chemistry from Ho Chi Minh City University of Science. After achieving his Ph.D degree from Korea University of Science and Technology (2021), he returned to Vietnam and working as a lecturer at Faculty of Chemical and Food Technology, HCMC University of Technology and Education. His research is concentrating on the synthesis of porous materials, surface modification, and thermal energy storage. Email: ntgiang@hcmute.edu.vn. Orcid: https://orcid.org/0000-0002-5326-7812

**Le Minh Tam** received PhD at Max Planck Institute for Dynamics of Complex Technical Systems, Magdeburg, Germany. Postdoctoral research at Vietnam Academy of Science and Technology. Editorial Board Member of JCERU journal. Expertise in the fields of advanced materials, enantiomer separation, crystallization, chromatography, process analytical technology (PAT), chemical and bio-process engineering. Email: tamlm@hcmute.edu.vn Orcid: https://orcid.org/0000-0002-5372-346X