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Thermal Performance Evaluation of Fatty Acid Ester and Paraffin Based Mixed SSPCMs Using Exfoliated Graphite Nanoplatelets (xGnP)

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Academic Editor: Mohammed Mehdi Farid

Received: 22 February 2016; Accepted: 4 April 2016; Published: 12 April 2016

Abstract: Phase change materials (PCMs) have been used in various fields including the materials of buildings. In this research, mixed shape-stabilized PCMs (Mixed SSPCMs) were prepared by impregnating coconut oil and *n*-hexadecane into exfoliated graphite nanoplatelets (xGnP) through a vacuum impregnate method. Coconut oil is fatty acid ester PCM which is relatively economical in comparison to other PCMs, and *n*-hexadecane is paraffin PCM that has high latent heat capacity. Drawbacks include leakage in a liquid state and low thermal conductivity resolved by xGnP. When preparing Mixed SSPCMs, coconut oil and *n*-hexadecane were impregnated at different proportions, namely 70:30, 50:50, 30:70 wt %. Mixed SSPCMs were analyzed through SEM, FT-IR, DSC, TGA and TCi. As a result, we confirmed the microstructure, chemical stability, thermal properties, thermal stability and thermal conductivity of Mixed SSPCMs. Latent heat capacity of Mixed SSPCMs were 89.06, 104.30 and 124.50 J/g while those of SSPCMs containing single PCM were 82.34 and 96.40 J/g. Thermal conductivity of Mixed SSPCMs was more than 284% higher than that of pure coconut oil and *n*-hexadecane. Finally, we confirmed that coconut oil and *n*-hexadecane were impregnated into xGnP, and the Mixed SSPCMs have high thermal durability.

Keywords: mixed SSPCM; fatty acid ester PCM; paraffin PCM; thermal performance; thermal storage; xGnP; impregnation

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1. Introduction

Thermal energy storage (TES) for space heating and cooling of buildings is becoming more and more important due to the rising cost of fossil fuels and environmental concerns. It saves premium fuels and leads to the system being more cost effective by reducing the waste of energy and capital costs [1,2]. Sensible heat storage (SHS) using thermal mass of the structural elements is the most common way to storage thermal energy in buildings. It can be used for the storage and release of thermal energy in a passive way. However, in comparison to latent heat storage, a much larger volume of material is required to store the same amount of energy by changing the phase of a storage material. Therefore, an effective way to reduce the building's energy consumption for heating and cooling is by incorporating phase change materials (PCMs) in latent heat thermal energy storage (LHTES) systems of various building elements such as walls, windows, ceilings and floors [3,4].

PCMs are generally divided into organic PCM, inorganic PCM, and eutectic PCM. Typical inorganic PCMs include water, aqueous solution, salt hydrate, and molten salt, while organic PCMs include paraffin, fatty acid, ester and sugar alcohol. Besides, eutectic mixtures of two or more miscible pure

PCM constituents that solidify simultaneously out of the liquid at a minimum freezing point have been widely developed to enrich the diversity of PCMs [5].

Commonly, inorganic PCMs suffer from decomposition and supercooling, which can further affect their phase change properties [6]. In contrast, organic PCMs are more stable and incorrodible than inorganic PCMs in the surrounding container [7]. In particular, paraffin PCMs are more widely used for application in building because they possess large latent heat capacity, low vapor pressure, and good thermal stability [8,9]. Also, many fatty acid ester PCMs have lower latent heat capacity than paraffin PCMs, but most of them are more commercial and economical than paraffin PCMs [10]. However, fatty acid ester and paraffin PCMs also have low thermal conductivity and leakage of liquid problems that disturb their application to energy storage [11–13]. Manufacturing shape-stabilized PCM (SSPCM) by impregnating the PCMs into high thermal conductivity materials such as carbon materials with porous structure resolves these problems. Exfoliated graphite nanoplatelets (xGnP) which are porous and nano-sized carbon materials are suitable [14–18]. Additionally, they have a layered structure and are very cost effective. The low coefficient of thermal expansion of xGnP, is a desirable property for composite structural applications [19–22].

In this research, we prepared Mixed SSPCMs by impregnating two types of PCMs, coconut oil and *n*-hexadecane into the xGnP at different proportions to improve thermal performance and investigate the difference between their performances. We used a vacuum impregnation process that guarantees high heat storage of fatty acid ester PCM and paraffinic PCM due to capillary forces and surface tension forces during the incorporation process [23]. Further, we analyzed the microstructure, chemical stability, thermal storage performance, thermal stability, and thermal conductivity of the Mixed SSPCMs by using SEM, FT-IR, DSC, TGA, and TCi.

2. Experimental

2.1. Materials

In this research, we used fatty acid ester PCM and paraffin PCM, which possess different melting points. Fatty acid ester PCM is coconut oil and paraffin PCM is *n*-hexadecane in this research. The melting point and latent heat capacity at solid-liquid melting of pure coconut oil are 26.78 °C and 110.4 J/g, and those of pure *n*-hexadecane are 20.84 °C and 254.7 J/g [23,24]. The coconut oil is obtained from the Korea Similac Corporation in Gyeonggi, South Korea, and the *n*-hexadecane is obtained from the Celsius Korea Corporation in Seoul, South Korea. The exfoliated graphite nanoplatelets (xGnP) were arranged from sulfuric acid-intercalated expandable graphite (3772) obtained from Asbury Graphite Mills, Inc., in Asbury, NJ, USA by applying a cost- and time-effective exfoliation process initially proposed by Drzal's group [19]. Table 1 shows the physical properties of coconut oil and *n*-hexadecane, and Table 2 shows those of xGnP.

Table 1. Physical properties of coconut oil and *n*-hexadecane [23,24].

Property	Value	
	Coconut Oil	***n*-Hexadecane**
Melting point (°C)	26.78	20.84
Latent heat of melting (J/g)	110.4	254.7
Thermal conductivity (W/mK)	0.321	0.154

Table 2. Physical properties of xGnP [14].

Property	Value
Surface area (m^2/g)	20.41
Bulk density (g/cm^3)	0.0053–0.010
Pore volume (cm^3/g)	0.081
Thermal conductivity (W/mK)	2–300
Specific heat capacity (J/kgK)	710

2.2. Preparation

We prepared the Mixed SSPCMs by impregnating the coconut oil and *n*-hexadecane into xGnP at different proportions: 70:30 wt %, 50:50 wt %, and 30:70 wt %. To prepare the Mixed SSPCMs, we mixed the coconut oil and *n*-hexadecane in each proportion at room temperature and strongly stirred. The xGnP was then dried at 105 °C for 24 h in a vacuum oven. Next, we used the vacuum impregnation process according to following procedure. The xGnP was put inside a filtering flask, which was connected to a water tromp apparatus to evacuate air from the porous structure. Then, a valve between the flask and a container with a total of 100 g of liquid coconut oil and *n*-hexadecane was opened, to allow the liquid PCMs to flow into the flask and cover the nanoparticles of xGnP. After the vacuum process was continued for 90 min, the air was allowed to enter the flask again, to force the liquid PCMs to penetrate the porous structure of the xGnP. In this process, excess PCMs which were not impregnated remained in the flask. Those were removed through a filtering process. The·coconut oil + *n*-hexadecane Mixed SSPCMs in the colloidal state were filtered through 1 μm filter paper until a granule type of sample appeared on the filter paper, and then those were dried in a vacuum drier at 80 °C for 48 h.

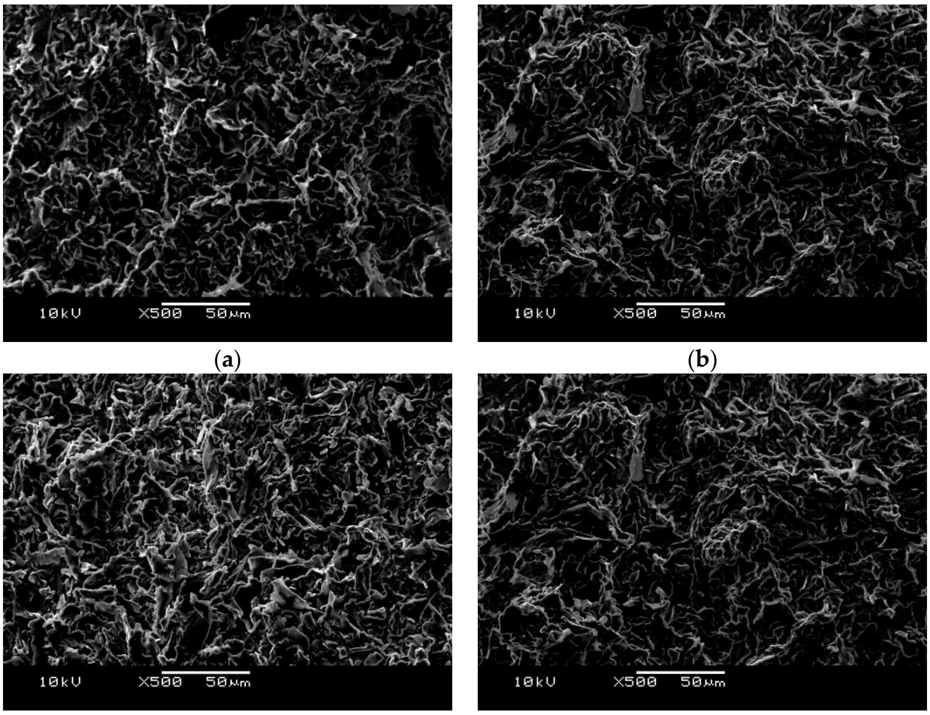
2.3. Characterization Techniques

The microstructure of the Mixed SSPCMs were analyzed through scanning electron microscopy (SEM, JSM-6360A, JEOL, Tokyo, Japan) at room temperature. The SEM images of the samples were obtained with an accelerating voltage of 12 kV and a working distance of 12 mm, and the samples were gold coated at a few nanometers in thickness to increase their electrical conductivity [19]. Fourier transform infrared spectroscopy (FT-IR, Nicolet 6700, Thermo Scientific, Waltham, MA, USA) was used to confirm the change of chemical groups of the Mixed SSPCMs at room temperature. Clear potassium bromide (KBr) disks were made from powder and used as backgrounds. The Mixed SSPCMs were analyzed over the range of 650–4000 cm^{-1}, with a spectrum resolution of 8 cm^{-1}. All spectra were averaged over 32 scans. This analysis of the Mixed SSPCMs was performed by point-to-point contact with a pressure device [25]. Thermal properties of the Mixed SSPCMs, such as melting and freezing point and latent heat capacities, were analyzed by the differential scanning calorimetry (DSC, DSC-Q1000, TA instrument, New Castle, PA, USA). The melting and freezing point were measured by drawing a line at the point of maximum slope of the leading edge of the peak and extrapolating to the base line, and the latent heats of the Mixed SSPCMs were analyzed as total by numerical integration of the area under the peaks that represents the solid-solid and solid-liquid phase transition [17]. The DSC measurements were performed at a 3 °C/min heating and cooling rate and a temperature range of −20 to 80 °C. Thermal stability of the Mixed SSPCMs was analyzed by thermogravimetric analysis (TGA, Q-5000 IR, TA Instruments, New Castle, PA, USA) on approximately 11–14 mg of the samples within the range from 20 to 600 °C at a heating rate of 10 °C/min under a nitrogen flow of 20 mL/min. The TGA was measured with the composites placed in a high quality nitrogen (99.5% nitrogen, 0.5% oxygen content) atmosphere to prevent unwanted oxidation [26]. The thermal conductivity of the Mixed SSPCMs were measured by TCi thermal conductivity analyzer (C-Therm Technologies Ltd., Fredericton, NB, Canada) at the temperature of 10 °C. The TCi is a device for conveniently measuring the thermal conductivity of a small sample by using the Modified Transient Plane Source (MTPS) method. It is the advantage of the TCi that can measure the thermal conductivity of materials in the states of solid, liquid, powder, and mixed [27].

3. Results and Discussion

3.1. Microstructure of the Fatty Acid Ester and Paraffin Based Mixed SSPCMs

Scanning electron microscopy (SEM) observations were performed for the coconut oil + *n*-hexadecane Mixed SSPCMs. Figure 1 shows the microstructure of the Mixed SSPCMs and xGnP that were investigated by SEM analysis. Figure 1a–c show the Mixed SSPCMs and Figure 1d shows the porous structure of the xGnP. We found some nano particles of xGnP in Figure 1d. Through the SEM analysis, we confirmed that the coconut oil and *n*-hexadecane were fully filled into the xGnP. As shown in Figure 1a–c, liquid PCMs were well impregnated into the each layer of the xGnP. The porous structure of the xGnP is good for impregnation of the PCMs. The microstructure of the Mixed SSPCMs are similar to one another, regardless of the ratio of the PCMs. Consequently, heat storage of the PCMs are maintained in the Mixed SSPCMs despite loading the xGnP.



(**a**) (**b**)

(**c**) (**d**)

Figure 1. SEM images of (**a**) 70 wt % coconut oil + 30 wt % *n*-hexadecane Mixed shape-stabilized phase change materials (SSPCM, (**b**) 50 wt % coconut oil + 50 wt % *n*-hexadecane Mixed SSPCM, (**c**) 30 wt % coconut oil + 70 wt % *n*-hexadecane Mixed SSPCM, and (**d**) exfoliated graphite nanoplatelets (xGnP).

3.2. FT-IR Analysis of the Fatty Acid Ester and Paraffin Based Mixed SSPCMs

Most of the edible oils, including coconut oil, are composed of triglycerides. As shown in Figure 2, a triglyceride is a fatty acid ester formed by esterifing glycerol with three fatty acids. Coconut oil consists of medium-chain triglycerides (MCTs), which are triglycerides that possess 8 to 12 carbons of fatty acids. So the coconut oil contains $–CH_3$, $–CH_2$, C=O, and C–O bonding. As shown in Figure 3, the FT-IR absorption spectrum of the Mixed SSPCMs appears with nearly the same absorption peaks. There are absorption peaks of 2919–2920 cm^{-1} and 2850–2851 cm^{-1} occurred by $–CH_3$ and $–CH_2$ bonding, and 1735–1744 cm^{-1} and 1149–1160 cm^{-1} occurred by C=O, and C–O bonding of ester groups. However, the 30 wt % coconut oil + 70 wt % *n*-hexadecane Mixed SSPCM that contains a small percentage of coconut oil gives rise to small peaks in C=O, and C–O bonding of ester groups. The *n*-hexadecane is a paraffin PCM that is consists of 16 carbons and 34 hydrogens of single chain. The condensed structure formula of that is $CH_3(CH_2)_{14}CH_3$. So the *n*-hexadecane also generate the peaks of $–CH_3$ and $–CH_2$ bonding. The FT-IR absorption peaks of the Mixed SSPCMs are shown in Table 3.

In this experiment, we mixed two types of PCMs and impregnated the mixed PCMs into xGnP to enhance thermal performance. Through the FT-IR analysis, we confirmed the chemical stability of the Mixed SSPCMs. As shown the result, the both absorption peaks of coconut oil and *n*-hexadecane are appeared in all Mixed SSPCMs. So we reason that the coconut oil and *n*-hexadecane did not join together chemically. In addition, by reviewing previous work [24,28], we confirmed the chemical stability of the PCMs in xGnP. According to [24,28], the FT-IR spectrum of coconut oil/xGnP and *n*-hexadecane/xGnP SSPCMs is the same as the each pure PCMs. This means that there are not chemical incorporations between coconut oil and xGnP, and *n*-hexadecane and xGnP. Therefore, we reasonably measured that the coconut oil + *n*-hexadecane Mixed SSPCMs are chemically stable.

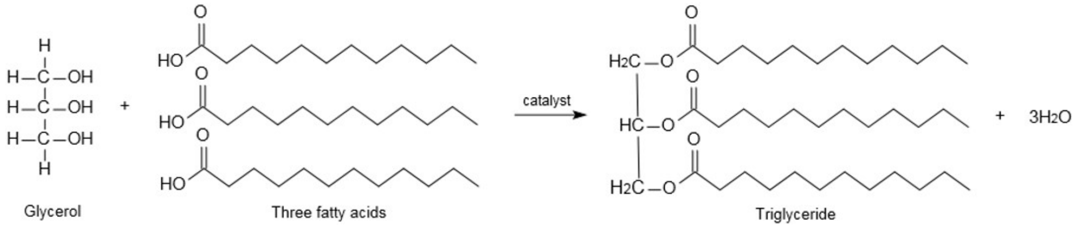


Figure 2. Chemical formation of triglyceride by esterification of glycerol with fatty acids.

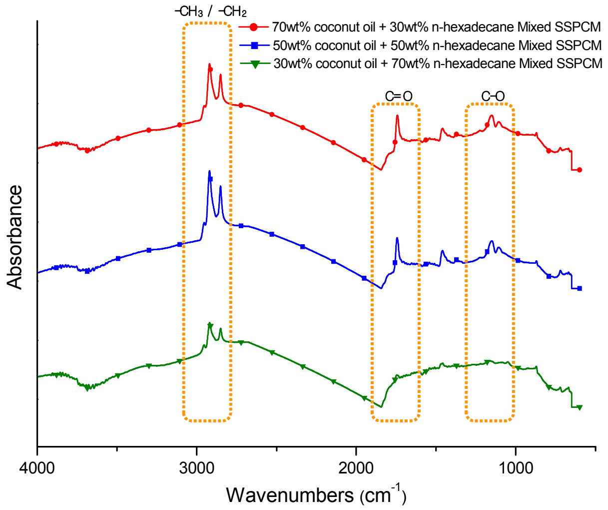


Figure 3. Fourier transform infrared spectroscopy (FT-IR) absorption spectra of Mixed SSPCMs.

Table 3. FT-IR absorption spectra peaks of Mixed SSPCMs.

Bonding	Wave Number Range (cm^{-1})
$-CH_3$	2919–2920
$-CH_2$	2850–2851
C=O	1735–1744
C–O	1149–1160

3.3. Thermal Properties Analysis

Figure 4 shows the heating and freezing curves from DSC measurements of the coconut oil + *n*-hexadecane Mixed SSPCMs. Table 4 represents the phase transition temperature and latent heat capacity of coconut oil + *n*-hexadecane Mixed SSPCMs and Table 5 represents those of coconut oil/xGnP and *n*-hexadecane/xGnP SSPCMs. As shown in Figure 4, the phase transition of the Mixed SSPCMs during heating and melting occurred at the lower temperature of the each melting and freezing point of the coconut oil and *n*-hexadecane. The Mixed SSPCMs display a tendency that the more the Mixed SSPCM contains a high rate of *n*-hexadecane, the greater the melting and freezing points. The melting temperatures at peak points of the Mixed SSPCMs were 12.53 °C, 14.51 °C, and 16.92 °C, while the freezing temperatures at peak points of them were 5.55 °C, 6.54 °C, and 10.31 °C.

The difference between melting and freezing points of the Mixed SSPCMs is because of supercooling of PCMs.

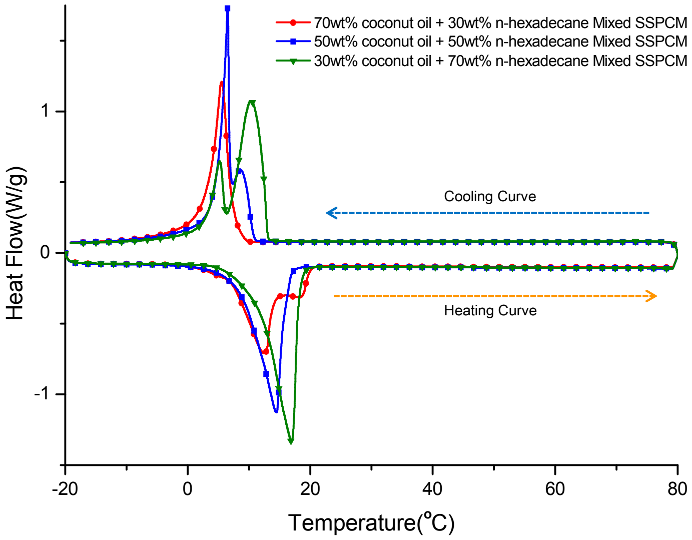


Figure 4. Differential scanning calorimetry (DSC) graphs of Mixed SSPCMs.

Table 4. DSC analysis of Mixed SSPCMs.

Mixed SSPCM (Coconut Oil: *n*-hexadecane)	**Melting Point (°C)**	**Freezing Point (°C)**	**Latent Heat (J/g)**	
			Melting	**Freezing**
70:30 (wt %)	12.53	5.55	89.06	92.82
50:50 (wt %)	14.51	6.54	104.30	106.20
30:70 (wt %)	16.92	10.31	124.50	123.40

Table 5. DSC analysis of coconut oil/xGnP and *n*-hexadecane/xGnP SSPCMs [24,28].

SSPCM	**Melting Point (°C)**	**Freezing Point (°C)**	**Latent Heat (J/g)**	
			Melting	**Freezing**
Coconut oil/xGnP	26.93	14.95	82.34	77.64
n-hexadecane/xGnP	21.80	14.60	96.40	94.80

The noticeable result in this research is the latent heat capacity of Mixed SSPCMs. The latent heat capacities of the Mixed SSPCMs were 89.06, 104.30, and 124.50 J/g during melting, and those of during freezing were 92.82, 106.20, and 123.40 J/g. Although these values are lower than pure PCMs because 3-dimentional network structure of xGnP disturbs the heat flow of PCMs, these are the high values in consideration of the latent heat capacities of coconut oil/xGnP and *n*-hexadecane/xGnP SSPCMs that contain single PCM. The latent heat capacities of the Mixed SSPCMs also appear to follow the law that the more the Mixed SSPCM contains a high rate of *n*-hexadecane, the greater the latent heat capacity.

3.4. Thermal Stability Analysis

TGA analysis was performed for thermal stability of the coconut oil + *n*-hexadecane Mixed SSPCMs at temperatures of 20 to 600 °C. Because xGnP is a carbon material that has high thermal durability, it does not decompose at temperatures under 600 °C. So only the coconut oil and *n*-hexadecane of the Mixed SSPCMs decompose in the test temperature range. Figure 5 and Table 6 show the TGA results of the Mixed SSPCMs. As shown in Figure 5, the TGA graphs of the all Mixed SSPCMs appear 2 degradation curves caused by thermal decomposition. The first degradation curves of the graphs represent the decomposition of *n*-hexadecane. The first degradation peaks of the *n*-hexadecane occurred at the temperature of 165.14 °C, 172.62 °C, and 178.38 °C. The second degradation curves of the graphs represent the decomposition of coconut oil. The second degradation peaks of the coconut oil occurred at temperatures of 363.68 °C, 357.68 °C, and 339.79 °C. In other words, the coconut oil has a greater thermal durability than the *n*-hexadecane.

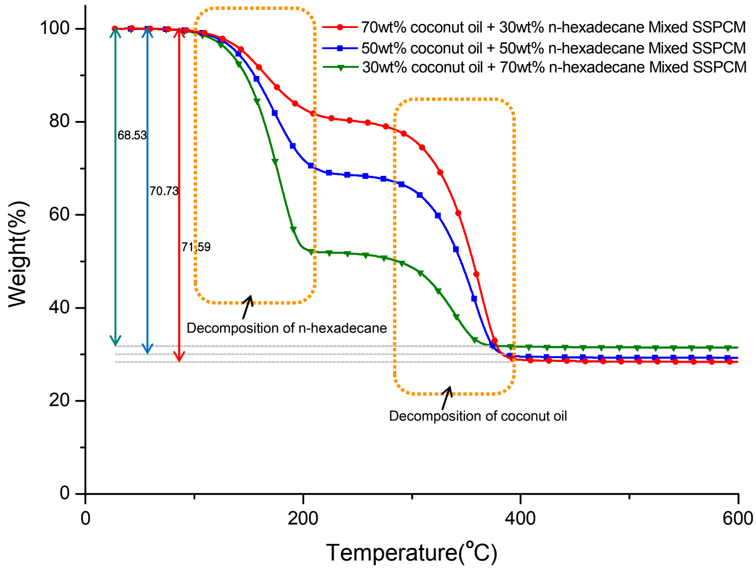


Figure 5. Thermogravimetric analysis (TGA) graphs of Mixed SSPCMs.

Table 6. TGA analysis of Mixed SSPCMs.

Mixed SSPCM (Coconut Oil: *n*-hexadecane)	First Peak of Derivative Weight (°C)	Second Peak of Derivative Weight (°C)	Total Decomposition Rate (%)
70:30 (wt %)	165.14	363.68	71.59
50:50 (wt %)	172.62	357.68	70.73
30:70 (wt %)	178.38	339.79	68.53

It is also possible to compute the percentages of impregnated coconut oil and *n*-hexadecane in xGnP by investigating the total decomposition rates. The three Mixed SSPCMs appeared similar values of total decomposition rate. The total decomposition rate were 71.59%, 70.73%, and 68.53%. In the case of the Mixed SSPCMs, xGnP remained in them although the two PCMs were fully decomposed. This means that the two PCMs were impregnated into xGnP, and the Mixed SSPCMs have high thermal durability.

3.5. Thermal Conductivity Analysis

The thermal conductivity analysis of the coconut oil + *n*-hexadecane Mixed SSPCMs using TCi at the temperature of 10 °C is shown in Figure 6. As shown in Figure 6, the thermal conductivities of the Mixed SSPCMs are 1.70, 1.61, and 1.23 W/mK. The Mixed SSPCMs appear to follow the tendency that the more coconut oil they contain, the greater the thermal conductivity because the pure coconut oil has larger thermal conductivity than pure *n*-hexadecane. The thermal conductivities of pure coconut oil and *n*-hexadecane are 0.321 and 0.154 W/mK, respectively [23,24]. The overall Mixed SSPCMs have larger thermal conductivities which are 284% higher than the pure PCMs. This implies that the xGnP led to an enhancement of the thermal conductivity of coconut oil and *n*-hexadecane. Consequently, we concluded that Mixed SSPCMs can be usefully applied to various categories such as buildings.

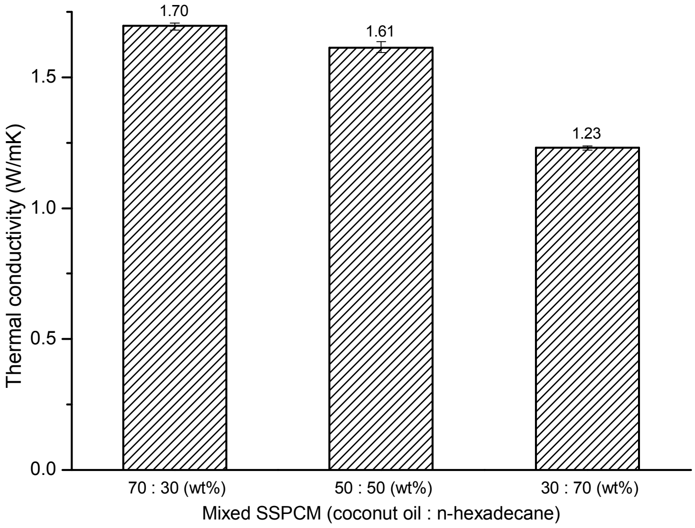


Figure 6. Thermal conductivity of Mixed SSPCMs.

4. Conclusions

With the rising cost of fossil fuels and additional environmental concerns, the importance of latent heat thermal energy storage (LTES) for space heating and cooling is increasing. Accordingly, phase change materials (PCMs) have been used in various fields. Among the PCMs, fatty acid ester PCMs are relatively economical and paraffin PCMs have higher latent heat capacities than other PCMs. So we prepared coconut oil + *n*-hexadecane mixed shape-stabilized PCMs (Mixed SSPCMs) using exfoliated graphite nanoplatelets (xGnP) through a vacuum impregnation method to enhance their thermal performance with supplementation of drawbacks of PCMs such as the leakage in liquid state and low thermal conductivity. When preparing the Mixed SSPCMs, we impregnated the coconut oil and *n*-hexadecane at different proportions, namely 70:30, 50:50, 30:70 wt %. We analyzed the microstructure, chemical stability, thermal properties, thermal stability, and thermal conductivity of the samples using SEM, FT-IR, DSC, TGA, and TCi. Through the SEM analysis, we confirmed that both of the PCMs were well impregnated into the porous structure of the xGnP. The FT-IR graphs show all peaks of the bonding of both of the PCMs. So, we confirmed that there was no chemical interaction in the all Mixed SSPCMs. From the DSC analysis, we confirmed that the latent heat capacity of the coconut oil + *n*-hexadecane Mixed SSPCMs was higher than the coconut oil/xGnP and *n*-hexadecane/xGnP SSPCMs containing single PCM. The values of latent heat during melting were 89.06, 104.30, and 124.50 J/g while those of SSPCMs containing single PCM were 82.34 and 96.40 J/g. The greater the amount of *n*-hexadecane, the greater the latent heat capacity becomes. Also, we analyzed the decomposition temperatures and the total decomposition rates of Mixed SSPCMs through TGA analysis. As a result, we confirmed that the coconut oil and *n*-hexadecane were impregnated into xGnP, and the Mixed SSPCMs have high thermal durability. The thermal conductivities of the Mixed SSPCMs analyzed by TCi were over 284% higher than those of pure coconut oil *n*-hexadecane. Consequently, we expect the Mixed SSPCMs to be useful in applications in various fields due to their high thermal performances. For example, considering their phase change temperatures, they can be applied to improve energy loss in building design and operation in countries with a contained, microthermal climate.

Acknowledgments: This work was supported by the National Research Foundation of Korea(NRF) grant funded by the Korea government(MSIP) (No.NRF-2014R1A2A1A11053829).

Author Contributions: Hwayoung Lee, Su-Gwang Jeong, Seong Jin Chang, Yujin Kang and Seunghwan Wi designed and conducted the experiment. Sumin Kim designed the project. All authors contributed to the analysis and conclusion.

Conflicts of Interest: The authors declare no conflict of interest.

Abbreviations

The following abbreviations are used in this manuscript:

PCM	Phase change material
SSPCM	Shape-stabilized phase change material
xGnP	Exfoliated graphite nano platelets
TES	Thermal energy storage
SHS	Sensible heat storage
LHTES	Latent heat thermal energy storage
SEM	Scanning electron microscopy
FT-IR	Fourier transform infrared spectroscopy
DSC	Differential scanning calorimetry
TGA	Thermogravimetric analysis

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