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Fabrication and Thermal Performance of 3D Copper-Mesh-Sintered Foam/Paraffin Phase Change Materials for Solar Thermal Energy Storage

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Abstract: Due to its large latent heat and high energy storage capacity, paraffin as one of the phase change materials (PCMs) has been widely applied in many energy-related applications in recent years. The current applications of paraffin, however, are limited by the low thermal conductivity and the leakage problem. To address these issues, we designed and fabricated form-stable composite PCMs by impregnating organic paraffin within graphite-coated copper foams. The graphite-coated copper foam was prepared by sintering multilayer copper meshes, and graphite nanoparticles were deposited on the surface of the porous copper foam. Graphite nanoparticles could directly absorb and convert solar energy into thermal energy, and the converted thermal energy was stored in the paraffin PCMs through phase change heat transfer. The graphite-coated copper foam not only effectively enhanced the thermal conductivity of paraffin PCMs, but also its porous structure and superhydrophobic surface prevented the paraffin leakage during the charging process. The experimental results showed that the composite PCMs had a thermal conductivity of 2.97 W/(m·K), and no leakage occurred during the charging and discharging process. Finally, we demonstrated the composite PCMs can be readily integrated with solar thermoelectric systems to serve as the energy sources for generating electricity by using abundant clean solar-thermal energy.

Keywords: solar-thermal energy storage; phase change material; copper foam; paraffin



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1. Introduction

To date, energy crisis has become one of the most serious issues for sustainable development of mankind, and solar energy due to the advantages such as cleanness, large capacity, wide availability and versatility has been attracted tremendous attention in the past decades [1,2]. Solar-thermal conversion technology, which directly converts solar irradiation into thermal energy, is considered as a promising candidate to harness abundant solar energy for industrial manufactures and domestic applications [3]. Solar irradiation, however, is inherently intermittent and highly dependent on weather conditions, which results in a mismatch between the thermal energy demand and supply. Hence, the solar-thermal energy storage system is proposed to address these issues. Currently, latent heat storage based on liquid-solid phase change materials (PCMs) which have many advantages of high heat storage density, large phase change enthalpy, and the capability to maintain a near-constant phase change temperature is widely applied [4–6]. The latent heat storage systems not only provide a stable thermal energy output, but also improve the thermal performance of solar energy utilization. As a result, extensive research interest has been concentrated on the PCMs such as organic paraffin [7,8], stearic acid [9], molten salt [10], and the applications involve seawater desalination [11], water heating [12,13], and building heating [14]. The PCMs, however, have a low thermal conductivity which leads to a low phase change heat transfer rate and restricts the thermal storage efficiency. In addition,

the PCMs have a serious leakage problem when it melts from solid phase to liquid phase, leading to decaying in the thermal performance of the systems.

In order to overcome the problem of low thermal conductivity, a large amount of high thermal conductivity fillers have been blended with the PCMs such as carbon materials [15–17], metal foam [18–20], and nanoparticles [21,22]. For example, Lu et al. [23] reported a new composite PCM which was composed of expanded graphite and paraffin. The thermal conductivity of new composite PCMs containing 5wt.% of expanded graphite was about 4.2 times than that of pure paraffin. Kumar et al. [24] used silicon nitride (Si_3N_4) nanoparticles to enhance the thermal conductivity of pure paraffin. The experimental results showed that the thermal conductivity of paraffin could be increased by 33.9% as 2 wt.% of Si_3N_4 nanoparticles blended into the PCMs. Shama et al. [25] studied the impact of CuO nanoparticles on the thermal performance of paraffin. It was found that the thermal conductivity of composite PCMs was improved by 22.22% and melting time was reduced by 42.85% when 1 wt.% of CuO nanoparticles were used as enhancers. Xie et al. [26] adopted a facile blending method to prepare the new composite PCMs which consisted of polyethylene, expanded graphite, and paraffin. The thermal conductivity of the new composite PCMs was 2.7 times than that of pure paraffin, and the speed of thermal response was increased by 25.9%. Masher et al. [27] developed a high thermal conductivity composite PCM which contained silicon carbide and silver nanomaterials. The experimental results showed that when 15 wt.% of SiC and 15 wt.% of Ag nanomaterials were blended into the PCMs, the thermal conductivities of the prepared composite PCMs were improved by 58.2% and 31.2%, respectively. Although many efforts have been devoted to improving the thermal performance of the PCMs, high concentrates of high- k fillers will reduce the thermal capacity of the PCMs.

To solve the problem of leakage, a large amount of stable-form composite PCMs have been successfully prepared by impregnating PCMs within porous materials such as carbon foams [28], nickel foams [29], and copper foams [30]. Among them, copper foams were widely used as the supporters to improve the thermal performance of the paraffin due to its high thermal conductivity, high porosity, large specific surface area, and favorable mechanical properties [31–35]. For example, Zhang et al. [33] reported a new form-stable composite PCM which was composite of copper foam, graphene oxide (GO), reduced graphene oxide (rGO), and organic paraffin. The surface of copper foam was modified by GO and rGO to efficiently solve the problem of PCMs leakage. Ye et al. [34] designed a PCM-based heat pack by combining the carbon-coated copper foam with organic PCMs. After coating with carbon materials, the pores of copper foam provided a large amount of active sites to adsorb paraffin PCMs, thus solving the leakage issue. Liang et al. [35] reported a superhydrophobic oxidized copper foam which was prepared by oxidizing copper foam by using $(\text{NH}_4)_2\text{S}_2\text{O}_8$ to construct a rough surface and then filled into organic PCMs. The superhydrophobic surface effectively prevented the paraffin leakage from the modified copper foam. Zheng et al. [36] designed a form-stable phase change material with high thermal conductivity and capability of solar-thermal conversion. The graphene aerogel and copper foam not only enhanced the thermal conductivity of the PCMs, but also solved the problem of leakage. Meanwhile, the composite PCMs could directly absorb solar energy and converted it into thermal energy. In the majority of current research, however, commercial copper foams were mainly used as enhancers for PCMs. It is still a challenge to design and fabricate the three-dimensional copper foams to improve the thermal performance of the PCMs.

In this work, we proposed a facile method to fabricate copper foam-based composite PCMs which had high thermal conductivity, high thermal stability and could directly convert solar irradiation into thermal energy. Such form-stable composite PCMs were prepared by impregnating paraffin in porous graphite-coated copper foam. The graphite-coated copper foam was prepared by sintering multilayer copper meshes, and its surface was etched by strong base solution, followed by coating with a mixture of graphite nanoparticles and polydimethylsiloxane (PDMS). High thermal conductivity graphite-coated copper foam

would provide an efficient heat transfer path during the process of phase change. Meanwhile, the superhydrophobic surface and small pores of the modified three-dimensional copper foam effectively inhibited the leakage of melted PCMs. Graphite nanoparticles were used as solar absorbers to convert solar energy into thermal energy, and the converted thermal energy was directly stored in the paraffin PCMs through phase change heat transfer. The experimental results showed that the graphite-coated copper foam effectively enhanced the thermal conductivity of paraffin PCMs and prevented the paraffin leakage during the solar charging process. Finally, we demonstrated that this copper foam-based composite PCM could be integrated with a thermoelectric module to generate electricity.

2. Materials and Methods

2.1. Materials

The copper mesh (300-mesh) was brought from Hebei Kang Mei Long Wire and Mesh Co., Ltd., Shijiazhuang, China. Paraffin, potassium hydroxide, and potassium peroxodisulfate were ordered from Aladdin Reagent Co., Ltd., Shanghai, China. Polydimethylsiloxane (PDMS, Sylgard 184) was obtained from Dow Corning Corporation, Shanghai, China. The mixed gas (nitrogen, 90%; hydrogen, 5%) was brought from Dalian Date Gas Co., Ltd., Dalian, China.

2.2. Preparation of Sintered Copper Foam

Copper meshes were first cut into different sizes and then were placed into 4 mol/L hydrochloric acid solution to remove the copper oxide on the surface. After drying, the cleaned copper meshes were formed into a cube with a size of 27 mm × 18 mm × 13 mm. Then, the copper cube was sintered in the furnace at a temperature of 900 °C. The temperature was raised from room temperature to 900 °C with a rate of 10 °C/min. During the sintering process, a hydrogen-nitrogen mixture was used as protective gas with a flow rate of 80 mL/min. After the sintering temperature reached 900 °C, it was held for 1 h. After cooling to room temperature, the sintered copper foam was cleaned and dried for further experiments.

2.3. Preparation of the PCM Composite

First, the sintered copper foam was cleaned by diluted hydrochloric acid and deionized water, respectively. Then, the cleaned copper foam was immersed in a hot base solution of 0.065 M $K_2S_2O_8$ and 2.5 M KOH at 60 °C for 60 min. The etched copper foam was rinsed by deionized water for 5 times and dried in an oven. The surface of the sintered copper foam was further modified by 20 g n-hexane solution which dissolved with graphite nanoparticles, PDMS, and curing agent (10:10:1). The modified copper foam was placed into a drying oven at 60 °C for 24 h to cure the PDMS. To obtain the PCM composite, the graphite-coated copper foam was immersed into the molten paraffin for 10 min and then taken out followed by cooling to room temperature.

2.4. Measurement and Characterization

The morphology was observed by using a Field Emission Scanning Electron Microscope (SEM, Sirion 2000, FEI, Shanghai, China). IR images were obtained by using an infrared camera (HM-TPH36-10VF/W HIKVISION, Hangzhou, China). A solar simulator (CEL-PE300L-3A, Beijing China Education Au-light Technology Co., Ltd., Beijing, China) was used as the solar light source to generate the solar illumination, and the solar flux was measured by a solar power meter (CEL-NP2000-2A, Beijing China Education Au-light Technology Co., Ltd.). To evaluate the thermal conductivity of the sample, the temperature was real-time monitored by K-type thermocouples (Omega SMPWTT-K, Shanghai, China) which were connected to a multichannel data acquisition system (Agilent 34970a, Agilent Technologies Inc., Beijing, China). A cooling bath (Julabo Bilon Equipment, Beijing, China) was used to provide circulating running water with a consistent temperature of 10 °C.

3. Results and Discussion

In the composite PCMs, the graphite-coated copper foam played a significant role in the process of solar-thermal energy storage. The copper foam with high thermal conductivity provided a fast heat transfer path for storing thermal energy into the paraffin PCMs during the charging process. The graphite nanoparticles were excellent solar-thermal conversion materials, and directly converted solar energy into thermal energy. The porous structure of copper foam efficiently trapped the incident solar light and reduces scattered reflection. In addition, both the superhydrophobic surface and small pores provided a sufficient capillary force to inhibit the leakage of paraffin PCMs. Figure 1 exhibits the synthesis process of the copper foam-based composite PCM. First, copper meshes (60-mesh) were cut into different sizes to construct a cube with a length of 27 mm, a width of 18 mm, and a height of 13 mm. In the cube, 5 copper meshes were used as internal supporters. The whole cube was sintered at a temperature of 900 °C, and then cooled to room temperature to obtain copper-mesh-sintered foam. In order to obtain superhydrophobic and solar-thermal conversion properties, the copper-mesh-sintered copper foam was oxidized by strong base solution, and further immersed within a mixture of graphite nanoparticles and PDMS in n-hexane solution. After drying, the modified copper foam was impregnated with paraffin PCMs.

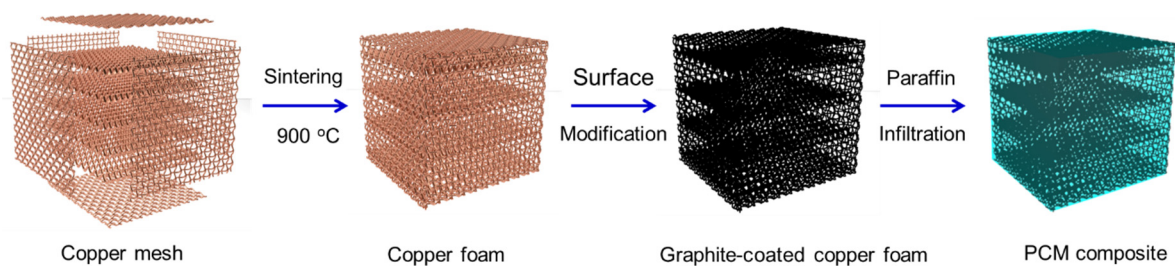


Figure 1. Fabrication process of the copper foam-based composite PCM.

The optical image of the fabricated composite PCM was shown in Figure 2a. As it shown, the size of the PCMs composite was 27 mm × 18 mm × 13 mm. The wettability of the graphite-coated copper foam was evaluated by the water contact angle. The graphite/PDMS layer of the copper foam remained superhydrophobic with a water contact angle of 133.4° as shown in Figure 2b, while the water contact angle of untreated copper foam was only 65.7° as shown in Figure S1. The low- and high-magnification SEM images in Figure 2c,d show the morphologies of the graphite-coated copper foam. After being modified with graphite nanoparticles, the surface of the copper foam became rough, and a layer of graphite nanoparticles was observed on the surface of it. In contrast, the untreated copper foam exhibits a relatively smooth surface as shown in Figure S2. The superhydrophobicity together with the roughness surface will help trap the paraffin PCMs in the copper foam. In addition, through comparing the weight and volume, the porosity of the copper foam was calculated to be 92.97%. Such high porosity would enable the copper foam loading more paraffin PCMs.

In addition, form stability has great effects on the solar-thermal charging and discharging processes. To evaluate the thermal stability, the composite PCM sample and pure paraffin sample were placed on a heating plate at 80 °C for 10 min as shown in Figure 3a,b. It should be noted that the composite PCM sample still kept the original shape and no leakage was observed after heating. On the contrary, the pure paraffin sample was completely melted within 1 min under the same condition. We further measured the weight change of the composite PCM before and after heating as shown in Figure S3. The experimental results showed that there was no obvious weight loss even after 50 thermal cycles and the leakage was less than 6%.

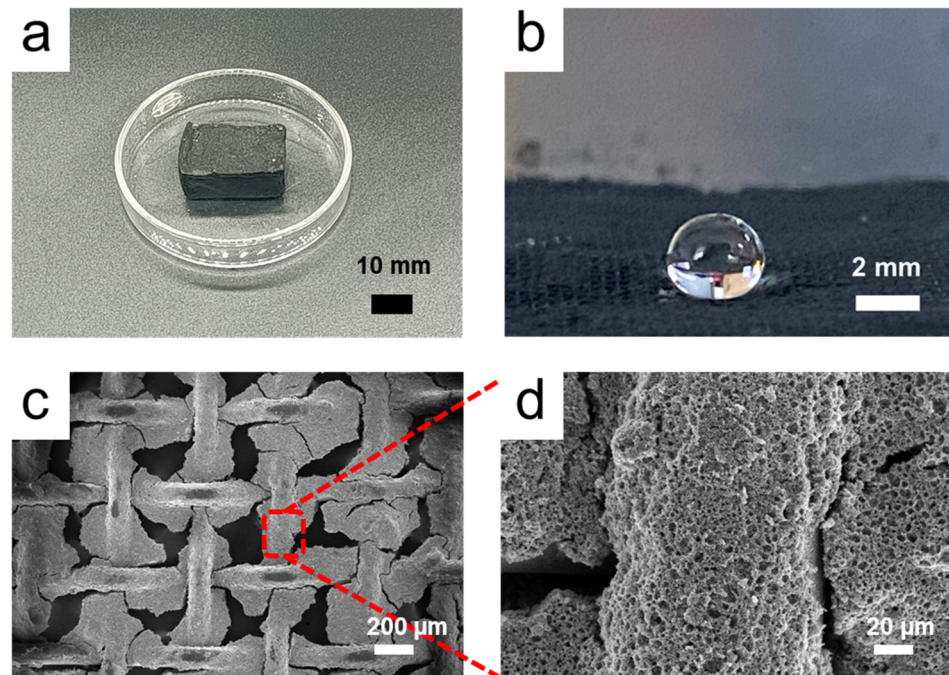


Figure 2. Characterization of morphology and wettability of the graphite-coated copper foam. (a) Photograph of the prepared composite PCM. (b) Photograph of a water droplet on the graphite-coated copper foam. (c,d) SEM images at different magnifications of the graphite-coated copper foam.

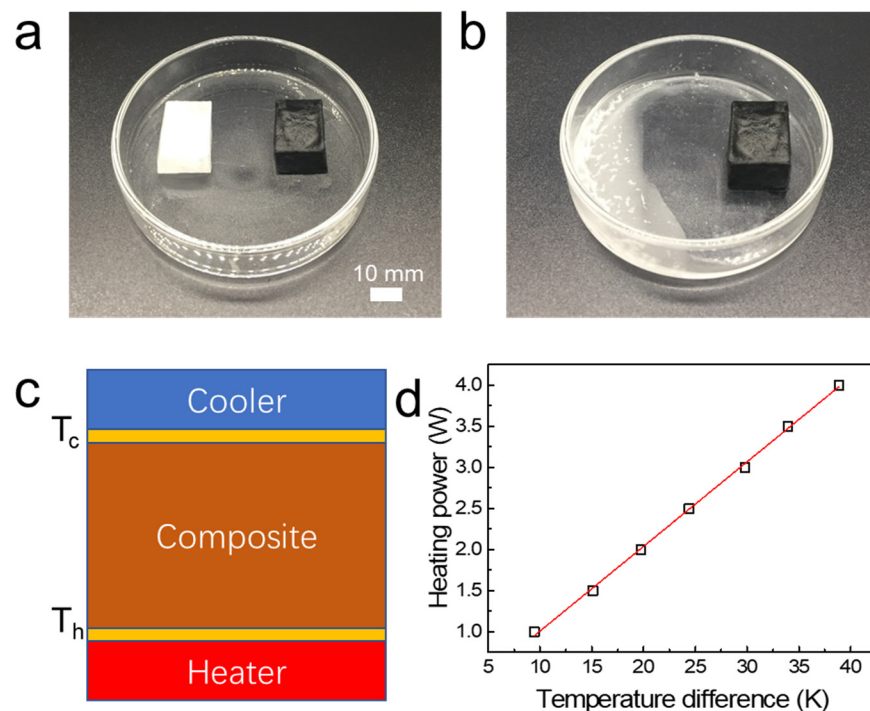


Figure 3. Form stability and thermal conductivity of the PCM composite. (a) Photograph of the PCM composite sample and paraffin sample before heating. (b) Photograph of the two samples after heating. (c) A schematic of the setup for differential steady state method. (d) Measurement result of the PCM composite.

Thermal conductivity was another important factor of the composite PCM. Figure 3c presented the measurement mechanism of thermal conductivity based on the Fourier's law. The experimental setup consisted of a water cooling bath, a multichannel data acquisition

system, a ceramic heating plate, and thermocouples as shown in Figure S4. The composite PCM sample was placed between the heater and the cooler, and the temperature difference was monitored by thermocouples under increasing heating inputs. During the test, the sample was wrapped by a layer of insulating foam with a thickness of 10 mm to reduce heat loss. Based on the experimental results, the thermal conductivity of the composite PCM sample can be obtained by the following formula:

$$Q = Ak \frac{T_h - T_c}{h} + Q_{ex} \quad (1)$$

where A is the cross-sectional area of the composite PCM sample, h is the thickness of the sample, k is the thermal conductivity, and Q_{ex} is the heat loss between the heater and environment. According to Equation (1), the heating input Q is linearly proportional to the temperature difference $T_h - T_c$, when Q_{ex} is assumed to be constant. Figure 3d shows the measurement result of the thermal conductivity of the composite PCM sample. By using a linear fitting of the temperature differences under varied heating inputs, the thermal conductivity of the composite PCM (k) is calculated to be 2.97 W/(m·K). Table 1 shows comparison of the prepared PCM composite with other reported works. As it shown, the high-porosity metal foam significantly enhances the thermal conductivity of phase change materials. Compared to other previous research, the prepared PCM composite in this work presents a high thermal conductivity.

Table 1. Comparison of the prepared PCM composite with other reported works.

Composite	Porosity	Thermal Conductivity (W/(mK))
Paraffin-Copper foam [32]	95%	1.13
Paraffin-Copper foam [18]	97.3%	2.879
Paraffin-Copper foam [37]	97.59%	1.238
	95.92%	1.439
Paraffin-Nickel foam [38]	95.2%	1.44
Paraffin-Nickel foam [39]	90.61%	2.33
Myrtle alcohol-Nickel Foam [40]	97%	0.48
This work	92.97%	2.97

To evaluate the solar-thermal charging performance, the composite PCM sample and paraffin sample were placed on a silicone pad under a solar flux of 400 mW/cm². The temperature change of the composite PCM sample was real-time monitored and recorded by an IR camera. Figure 4a shows the IR images of the composite PCM and pure paraffin samples during the charging process. Before charging, both the temperatures of composite PCM and pure paraffin samples were equal to the ambient temperature. After charging for 5 min, the composite PCM sample was obviously heated, while the pure paraffin sample was still at a relatively low temperature. We further investigated the temperature evolutions of the composite PCM and pure paraffin samples. Figure 4b shows that the pure paraffin sample was not melted even heated for 15 min. In addition, its temperature difference between the top surface and bottom was about 8 °C, meaning that the pure paraffin had a low thermal conductivity and low solar absorption.

On the contrary, the temperature curves of different parts of the composite PCM sample were almost overlapped during the whole charging process, as shown in Figure 4c. According to the temperate curves, it should be noted that the composite PCM sample began to undergo the phase transition from solid phase to liquid phase at 5 min. After charging for 8 min, the PCM composite was completely melted, and then the solar-thermal energy storage would be transformed from latent heat storage to sensible heat storage. Due to its high solar-thermal absorption of the graphite nanoparticles on the surface, the composite PCM sample was heated to more than 70 °C, which was much higher than the pure paraffin temperature. In addition, the high thermal conductivity of the composite

PCM would facilitate the heat transfer during the whole charging process, meaning that the sample could be uniformly heated.

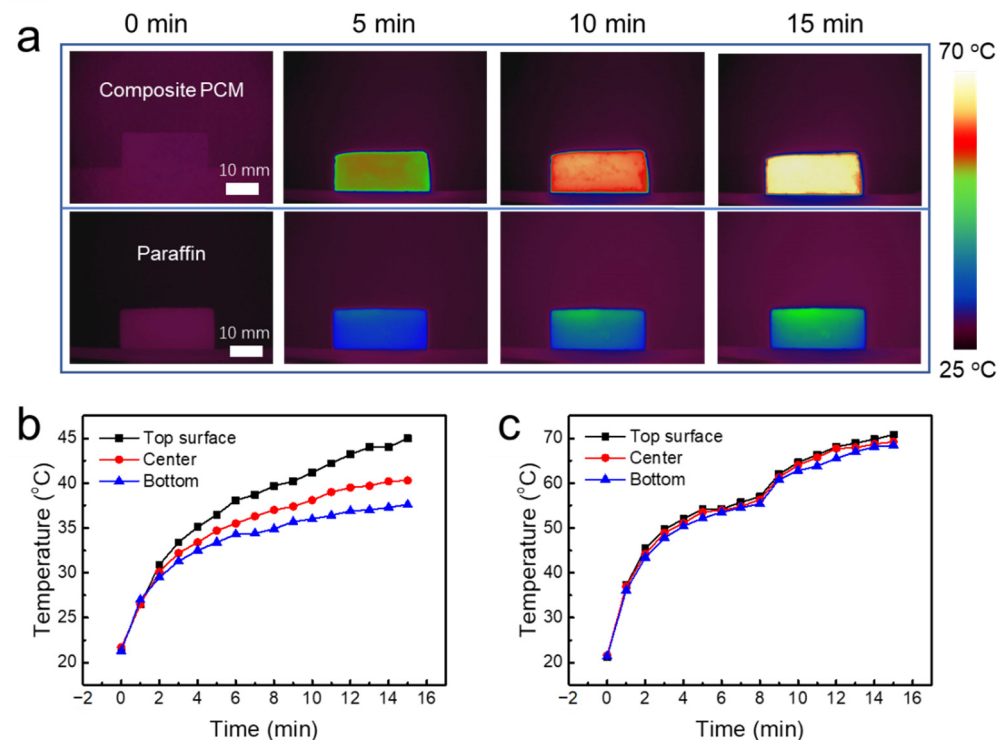


Figure 4. Thermal performance of the PCMs in the charging process. (a) IR images of the pure paraffin sample and the composite PCM sample at different charging time. (b,c) Temperature evolutions of the pure paraffin sample (b) and the composite PCM sample (c).

We also explored the thermal performance of the composite PCM during the discharging process. The discharging behavior of charged composite PCM was monitored by four thermocouples as shown in Figure S5. The composite PCM sample was heated to 70 °C, and then cooled to room temperature. As it shown, all the measured temperature curves are almost overlapped in the discharging process, which is attributed to the high thermal conductivity of the composite PCM sample.

Finally, we explored the application of the form-stable composite PCM by integrating it with a thermoelectric module to generate electrical energy. Figure 5a shows the schematic of the experimental setup. The hot side of the thermoelectric module was attached to the composite PCM sample, and the cold side was attached to a cold block. Once the solar irradiation was shining on the top surface of the composite PCM sample, the graphite coating layer would absorb solar energy and converted it into thermal energy, which was used to drive the thermoelectric module to generate power. The temperature of the cold block was maintained at 10 °C to transport the generated heat. In the controlled device, the only difference was that the composite PCM sample was replaced by the pure paraffin. Figure 5b shows the measured open circuit voltage evolution with time under the solar flux of 400 mW/cm². As it shown, when the composite PCM sample was used as the solar-thermal conversion and storage materials, the system generated a voltage of 0.14 V. In the controlled device, the generated open circuit voltage was only 0.04 V. In addition, when the solar light was turned off, the output voltage of both the composite PCM-based device and the pure paraffin-based device quickly dropped. In the composite PCM-based device, the device could continuously generate output for 500 s after the sunlight was turned off, which was longer than the lasting duration achieved when the pure paraffin was used in the system (125 s). Therefore, the experimental results indicated that the new PCM composite had a promising potential to be served for the solar-thermal applications.

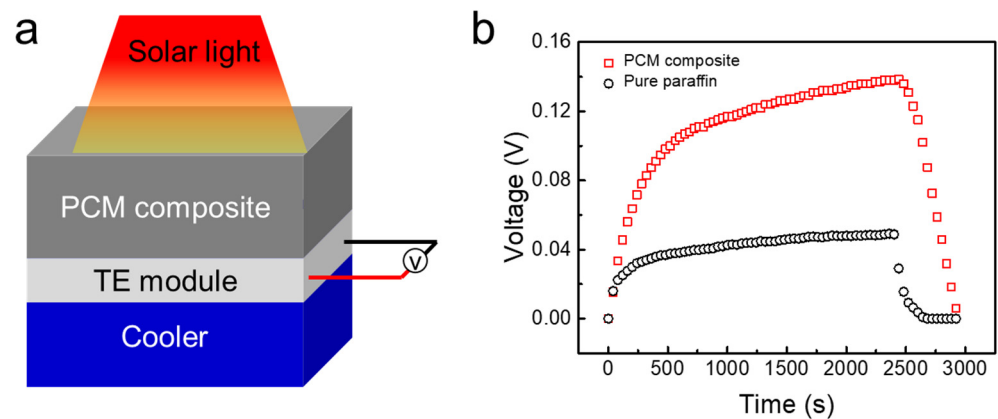


Figure 5. Solar-thermal-electric conversion based on the PCM. (a) Schematic illustration of the solar-thermal-electric device based on the composite PCM. (b) Open circuit voltage of the thermoelectric module with time under the simulated solar irradiation.

4. Conclusions

In summary, we demonstrated a new form-stable composite PCM by impregnating paraffin PCMs into a graphite-coated copper foam, which was prepared by sintering multi-layer of copper meshes. The prepared composite PCM had a high thermal conductivity of 2.97 W/m K, which attributed to the porous sintered copper foam. In addition, the graphite-coated copper foam had achieved a direct and rapid solar-thermal charging of the composite PCM through the graphite nanoparticles absorbing solar irradiation, efficiently prevented the leakage problem through trapping the liquid paraffin PCMs into the composite, and significantly improved the heat transfer capability of the composite PCMs through fast conducting the stored thermal energy along with the high thermal heat transfer path. Owing to the advantages of high thermal conductivity, form stability, large thermal energy storage capacity, and near-constant phase change temperatures, we demonstrated such prepared composite PCM could be used to provide the solar-thermal-electric system with much higher energy utilization efficiency and longer power generation time than the pure paraffin PCMs. Taking into account the general applicability of our method to develop composite PCM with different phase change temperatures by this direct and facile preparation process, we expected that the composites PCM would offer a promising strategy in the solar-thermal conversion applications and other energy-related fields.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/pr10050897/s1>. Figure S1: Photograph of a water droplet on the untreated copper foam. Figure S2: SEM images at different magnifications of the untreated copper foam. Figure S3: Leakage measurements of the composite PCM. Figure S4: Schematic of the experimental setup for evaluating thermal conductivity of the composite PCM sample. Figure S5: Temperature profiles of the composite PCM sample during the discharging process.

Author Contributions: C.C. designed the experiment. G.C. and F.W. carried out the experiment. Z.H. and L.P. analyzed the experimental data. C.C. wrote the manuscript. All authors have read and agreed to the published version of the manuscript.

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

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