

Article



# **Experimental Research of the Heat Storage Performance of a Magnesium Nitrate Hexahydrate-Based Phase Change Material for Building Heating**

Yang Li<sup>1,2</sup>, Caixia Wang<sup>2</sup>, Jun Zong<sup>2</sup>, Jien Ma<sup>1,\*</sup> and Youtong Fang<sup>1</sup>

- <sup>1</sup> Department of Electrical Engineering, Zhejiang University, Hangzhou 310027, China; liyang83901998@163.com (Y.L.); youtong@zju.edu.cn (Y.F.)
- <sup>2</sup> New Energy Technology Research Institute, State Power Investment Corporation Research Institute, Beijing 102209, China; wangcaixia@spic.com.cn (C.W.); zongjun@spic.com.cn (J.Z.)
- \* Correspondence: majien@zju.edu.cn; Tel.: +86-137-5716-1686

**Abstract:** Phase change heat storage material is a preferred material in solar building heating or off-peak electric-heat storage heating technology and is the research focus. A compact phase change thermal storage device has been designed and experimentally studied for improving heating system load in this work. A new type, magnesium nitrate hexahydrate-based phase change material has been studied to improve the cooling degree and crystallization difficulty. The focus of this study is on the heat charging and discharging characteristics of this new phase change material. The heat storage device has two groups of coils, the inner side which carries water and the outer side which is the phase change material. A testing system was built up to value the thermal cycling performance of the heat storage device. The measurement data include phase change material temperature field, water inlet and water outlet mean temperature, heat charging and heat discharging depth, and flow rates over the operating period. The results show the phase change material has a quick response with the operating temperature range of 20–99 °C. Its latent heat is 151.3 J/g at 91.8 °C. The heat storage density of this phase change material is about 420 MJ/m<sup>3</sup>. The thermal performance degradation is about 1.8% after 800 operation cycles. The phase change thermal storage device shows flexibility and a great potential to improve the capacity and economy of heating systems.

**Keywords:** heat storage performance; phase change material; heat storage; building heating; magnesium nitrate hexahydrate

# 1. Introduction

Ecological environment and energy utilization are closely related to social health development [1,2], and more than ninety percent of the world energy budget is connected to heat conversion and storage [3]. Due to its wide applicability, heat storage has attracted more and more attention in renewable energy power generation, solar building heating, and power grid peak shaving [4,5]. Different from other existing energy storage, heat storage has significantly high stability and low cost [6,7]. Especially in recent years, responding to the nationwide policy of "coal to clean energy", solar heating and electric heating is increasingly used in northern China to reduce coal-fired heating and its resulting pollution emissions [8,9]. This brings significant market prospects for heat storage technology to fill the energy demand gap economically. Phase change heat storage materials are different from common sensible heat storage materials [10]. Because of their latent heat, phase change materials (PCMs) can store additional energy at a certain temperature [11]. This allows them to play important roles in improving energy storage cost and balancing the mismatch between heating supply and demand [12]. The PCMs used in special fields should have certain thermophysical properties. For instant requirement such as building heating, the PCMs should have better thermal conductivities to get quick response, and



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**Copyright:** © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). the phase change temperature of the PCMs should be between 40 and 90 °C. PCMs include organic and inorganic materials [13]. The organic PCMs include fatty acids and paraffin wax, most of them have relatively stable chemical properties. However, these organic PCMs are always expensive and burn easily, and present poor thermal conductivity. The inorganic PCMs usually include salts, salt hydrates, and eutectic. Unlike organic materials, inorganic compounds are generally cheap and nonflammable, and have better thermal conductivity performance and higher density, all of these are helpful to obtain high heat storage performance. However, so far, most of the PCM used in buildings are organic materials. Inorganic PCMs lack application due to the problems such as supercooling and unstable circulation caused by phase separation [14]. Supercooling brings extra cooling capacity to release latent heat, and phase separation may lead to the failure of inorganic hydrate to maintain its phase change characteristics in long-term thermal cycle. Because the temperature of building heating is usually below 100 °C, it is valuable to study hydrated salt PCMs. Through comparison and selection, magnesium nitrate hexahydrate (MNH) with phase change temperature close to 89  $^{\circ}$ C and latent heat about 162 J/g is more suitable for this temperature range, and it has relatively higher chemical stability, lower corrosivity, and better economy. There are also cycle stability and supercooling problems in pure MNH. In order to solve the supercooling problem of pure MNH, Lane et al. [15] selected a variety of isomorphic additives as candidate nucleating agents, and freezing experiments show that the addition of 0.5 wt% sulfate hydrate can greatly inhibit the supercooling of pure MNH. Wang H et al. [16] proposed to prepare composite PCMs by combining photo-thermal materials. Solar-driven composite PCMs can absorb photons and convert them into thermal energy, most of which are stored in PCMs in the form of latent heat. The driving energy of PCMs comes from solar energy or other heat sources. First, a new solar-driven composite PCM containing MNH, carboxymethyl cellulose, and graphene was prepared, and its absorption capacity and photo to heat storage properties were measured by full spectrum system. Compared with pure MNH, the adsorption capacity of graphene composites increased from 46.54% to 79.12%. The photo to heat conversion and storage efficiency of composite PCMs can reach 69.73%, while pure MNH is not enough to absorb a large amount of latent heat because of its low absorption capacity. The thermal conductivity of the composite PCM with 5% graphene increased by 191.18%.

Zhang MH et al. [17] systematically studied the kinetic characteristics and its behavior of multi-step thermal decomposition of MNH. The results show that the pyrolysis of MNH is a complex multi-step reaction. It includes three reaction processes. The first two reaction processes are characterized by the precipitation of  $H_2O$ , while the third reaction process is characterized by the precipitation of  $NO_2$ . The order of the three processes is diffusion, orderly nucleation, and growth mechanism. Neeraj et al. [18] experimentally studied the effects of carbon materials of different sizes and shapes on the thermophysical properties of MNH. The experimental results show that the thermal conductivity of MNH can be increased by 100% by adding 0.5 wt.% carbon spheres. The results of differential scanning calorimetry show that the addition of carbon material improves the phase change and reduces the supercooling effect without affecting the latent heat capacity of the material. Honcova et al. [19] tried to reduce the supercooling of pure MNH by adding some additives, the mass fractions is from 0.5 to 2%. DSC results show that Mg (OH)<sub>2</sub> is very effective which can reduce the supercooling of pure MNH by about 90%. Honcova P. et al. [20] carried out a study on the thermal stability of MNH by adding carbon nanomaterials. The results show that graphite and graphene provide the highest energy, and the supercooling inhibition decreases from about 30 K to 2.2 K in 50 cycles. The addition of 3% graphite and 3% graphene can significantly increase 9% and 15%, respectively. Nagano et al. [21] also studied the corrosivity of MNH to metals such as aluminum and steel. The experimental results show that aluminum has good corrosion resistance to MNH, but carbon steel is not suitable. The experimental results show that the aluminum container can maintain the storage performance of MNH and its modified composites even after 1000 charge-discharge cycles. Li TX et al. [22–24] developed a modified MNH composite containing a certain

amount of nucleating agent for low-cost energy storage applications at medium and low temperatures. The results show that the supercooling of the composites can be maintained at about 2 K during solidification. So far, the research on the thermophysical properties modification and cycle performance evaluation of MNH including the modified composites with MNH as the main material all basically proved the applicability of MNH in medium and low temperature energy storage. However, for the large area application potential of MNH, such as industrial waste heat recovery and the use of "peak valley electricity price" for thermal regulation of electric building heating, there still are uncertainties. At present, the research on thermal design and performance evaluation of actual scale storage equipment using this material is quite few. In addition, some important results obtained in previous studies, such as the theoretical heat storage capacity and supercooling of materials, heat charging and heat discharging performance, need to be further analyzed and evaluated in practical application. In this work, the main thermal properties and supercooling stability of the modified MNH named as MNH90 were characterized. After confirming that the material can be recycled, we designed and manufactured a small latent heat storage device for the phase change material, and tested the charging and discharging performance of the energy storage device under certain working conditions with the electric boiler as the heat source and the cold water tank as the cold source. Some suggestions for thermal design and operation of the equipment are also put forward in the end of this work. The purpose of this paper is to prove the potential of MNH90 in the field of medium and low temperature heat storage and building heating.

## 2. Experimental Set Up

## 2.1. Phase Change Material

The thermal–physical properties of the MNH90 include latent heat, melting point and specific heat were measured by using a differential scanning calorimeter (DSC214, NETZSCH-Gerätebau GmbH). Figure 1 shows the specific heat of phase change material MNH90. The mass of sample analyzed here is about 20.75 mg. Concavus aluminum crucibles were used to fix samples in the DSC. The operating temperature range for these experiments was from 20 to 120 °C, the heating rate of the measurement was set to 1 K/min, and the flow rate of purge nitrogen and protective nitrogen is set at 40 mL/min and 60 mL/min, respectively. Sapphire is taken as the standard sample, and its mass is 20.75 mg. It can be seen from the results that the specific heat of MNH90 is  $1.6 J/(g\cdot K)$  at 20 °C. The specific heat increases slowly with the increase of working temperature. The specific heat reaches 4.1 J/(g·K) at 120 °C.



Figure 1. Specific heat capacity of phase change material MNH90.

The mass of MNH90 sample to be analyzed for latent heat is about 16.51 mg. Heat the sample from 20  $^{\circ}$ C to 110  $^{\circ}$ C and then cool it to 20  $^{\circ}$ C, the heating rate was set to

1 K/min, and the flow rate of purge nitrogen and protective nitrogen is set at 40 mL/min and 60 mL/min, respectively. Figure 2 reveals that in the heating stage, MNH90 has two endothermic peaks corresponding to 74.4 °C and 94.3 °C. The melting point corresponding to the larger peak is around 91.8  $^{\circ}$ C, so the melting point of the material is 92  $^{\circ}$ C, the latent heat is about 151.3 J/g, and in the cooling stage, the solidification temperature of the material is about 68  $^{\circ}$ C. Compared with pure MNH, due to the addition of 0.3 wt% nanoparticles and 2 wt% additive A, the supercooling degree and the stability of charging and discharging of MNH90 have been greatly improved, and the density, thermal conductivity, and specific heat of MNH90 are also higher than pure MNH. However, these nanoparticles and additive A do not have the ability to store latent heat in this operating temperature range, resulting in the latent heat of MNH90 to decrease slightly than that of pure MNH. According to the results, the MNH90 material can be used for heat storage and building heating system, which can meet the user's demand of 60 °C heating/hot water consumption. It can be estimated that the heat storage density of MNH90 is about  $420 \text{ MJ/m}^3$ , which is about 2.5 times than that of water, assuming the heat storage working temperature range is 40 °C. The thermal properties for MNH and MNH90 are provided in Table 1.



Figure 2. DSC curve of phase change material MNH90.

<b>Fable 1.</b> Thermal p	roperties for N	MNH and 1	MNH90.
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Material	T <sub>m</sub>	ΔH <sub>m</sub>	ρ	C <sub>P</sub>	λ
	(°C)	(J/g)	(g/m)	[J/(gial)]	[W/(mial)]
Pure MNH	89.9	162.8	1.636	1.8	0.67
MNH90	91.8	151.3	1.886	2.2	0.77

#### 2.2. Heat Storage Cycle Experimental Device

In order to test the feasibility of MNH90 for heat storage, a small-scale heat storage unit was designed and manufactured. Figure 3 shows the heat storage device has two groups of stainless-steel (304 SS) coils as heat exchanger, the inner side of heat exchanger coils is water and the outer side is phase change material. The heat exchanger has 2 header pipes, 2 serpentine branch-pipe groups (20 layers) and the length of the two water channels is equal. The total mass of MNH90 is 700 kg, and it fills 92% of the space between the inside of the heat storage tank and the outside of the pipeline. The outside surface of the heat storage tank is a layer of rock wool thermal insulation with an average thickness of 100 mm and an external steel sheets shell. The overall size of the heat storage device is 1788 mm (length)  $\times$  220 mm (width)  $\times$  1776 mm high. The designed heat storage capacity of the heat storage device is 70 kWh, and its designed working temperature range is 20–100 °C, which is mainly aimed at the working temperature of building heating. The heat storage



system is manufactured, assembled, and debugged in Henan Energy Storage Technology Co., Ltd. Cc (Pingdingshan, China).

Figure 3. Heat storage device for MNH90 test: (a) plane section diagram, (b) vertical view, and (c) internal structure of heat storage.

## 2.3. Testing System and Its Heat Performance Test

A testing system was built to evaluate the charge and discharge capacity and cycling efficiency of MNH90. Figure 4a shows the system employs a hot water tank with 10 kW electric heater and a cold water tank with cooling coil to provide constant inlet temperature for the heat storage device. The heat transfer fluid for the heat storage device is water, and it was driven by a centrifugal water pump with a rated mass flowrate of 453 kg/h. The flow rate of water for heating or cooling is measured by an electromagnetic flowmeter. Total of 11 temperature measuring points, including the mean temperature of the water inlet (Tin) and water outlet (Tout), and the temperature of the MNH90 in the heat storage tank (between the two groups of coils, and the distance from the top of the tank inner surface are 498 mm, 1138 mm and 1618 mm) are arranged to present the temperature in charging and discharging. Figure 4b presents the full view of the experiment system. It is needed to mention that the current design of the heat storage device is a modular design. By means of series and parallel multiple such modules, it is easy to realize the amplification application of the phase change heat storage unit. The ambient temperature around the experimental system ranges from 23 °C to 25 °C.



**Figure 4.** Performance test system of heat storage device: (**a**) schematic the experimental system and (**b**) the full view.

(b)

energy

storage device

The detailed system operation strategy and test steps of this experiment are as follows: During the charging process, the electric heater is set to a constant thermal output with a given maximum outlet water temperature of 99.5  $\pm$  0.5 °C. When the average difference of mean water temperature between water inlet and water outlet drops below 3.0 °C, heat charging stops. During heat discharging, the electric heater is shut down and the cooling water from cold water tank with the temperature of 20.5  $\pm$  0.5 °C flows through the heat storage tank. When the temperature of outlet water drops below the given cut-off temperature of 38.5  $\pm$  0.5 °C, heat discharging is terminated. In this experiment, data acquisition instrument (34970A, Keysight) is used for automatic data acquisition, and the data acquisition interval is 10 s.

#### 2.4. Performance Evaluation

Water

pump

The heat storage capacity of MNH90 can be obtained by this equation [25]:

$$Q = m \int_{T_{min}}^{T_{max}} c_{P,PCM} dT \tag{1}$$

where *m* is the total mass of MNH90,  $T_{min}$  and  $T_{max}$  are the start and end temperature of the MNH90 during charging process, and  $c_P$  is the specific heat capacity of the MNH90.

The operating thermal power of the heat storage unit named as *P* can be calculated by Equation (2),  $T_{in}$  is the mean temperature of water inlet,  $T_{out}$  is the mean temperature of water outlet,  $\dot{m}$  is the mass flow rate of water,  $c_{P,water}$  is the temperature-dependent specific heat capacity (using the mean temperature of  $T_{in}$  and  $T_{out}$ ) of water.

$$P = \dot{m}c_{P,water}(T_{in} - T_{out}) \tag{2}$$

The charging and discharging capacity of the heat storage device can be listed by the transient operating thermal power calculated by Equation (3):

$$\begin{cases} Q_C = \int_t^{t+t_c} P dt \\ Q_{DC} = \int_t^{t+t_{DC}} P dt \end{cases}$$
(3)

where  $t_C$  and  $t_{DC}$  are the time in heat charging and heat discharging procession, respectively. The cycling efficiency of charging and discharging (also be defined as the thermal efficiency) of the heat storage unit, which can show the energy storage and reuse efficiency of the heat storage device, can be defined as the ratio between  $Q_{DC}$  and  $Q_C$ :

$$\eta = \frac{Q_{DC}}{Q_C} \times 100\% \tag{4}$$

The storage capacity factor f which reflects the utilization of the heat storage capacity of the phase change heat storage device can be defined as the ratio of  $Q_C$  and Q:

$$f = \frac{Q_C}{Q} \times 100\% \tag{5}$$

The heat loss of the phase change heat storage device can be calculated by Equation (6):

$$Q_{loss} = \frac{A_{surf}(T_{PCM} - T_{amb})}{\frac{1}{h} + \frac{\delta_{ins}}{\lambda_{ins}}}$$
(6)

where  $T_{PCM}$  is the mean temperature of the MNH90 within the heat storage tank.  $T_{amb}$  is the room temperature. *h* is the coefficient of natural convective heat transfer, assigned to 10 W/(m<sup>2</sup>·K) in this work.  $\delta_{ins}$  is the thickness of the thermal insulation layer and the thermal conductivity  $\lambda_{ins}$  is 0.05 W/(m·K).  $A_{surf}$  is the surface area of the phase change heat storage device.

#### 2.5. Uncertainty Analysis of the Research Results

The measurement ranges and measurement accuracy of the main detectors and instruments used in this research are listed in Table 2. The aluminum crucible which is used in the DSC analyzer can work above 300 °C. It is calibrated with indium at the heating rate of 10 K/min through the standard calibration procedure. The uncertainty for latent heat measurements is around 0.34%, and the uncertainty for melting temperature is around 0.14%. The Hot Disk analyzer is only suitable for experiments at room temperature. The measurement accuracy of electromagnetic flowmeter comes from the manufacturer's manual. The temperature sensors are A-grade PT-100 thermal resistance with external armors for inlet and outlet water temperature measurements, and K-type thermocouples for MNH90 temperature tests in the heat storage tank.

Instruments	Measurement Ranges	Accuracy	
DSC214	Room temperature to 750 °C Heating rate: 0.1 °C/min to 50 °C/min	$\pm 0.1$ °C for temperature sensor $\pm 2\%$ for calorimeter	
D3C214	Cooling rate: $0.1 \degree C/min$ to $2 \degree C/min$ (<30 °C) $0.1 \degree C/min$ to $10 \degree C/min$ ( $\geq$ 30 °C)		
Hot Disk TPS2500S	$0.005 \text{ W/m} \cdot \text{K}$ to $500 \text{ W/m} \cdot \text{K}$	$\pm 5\%$	
K type thermocouple	-40 °C to 375 °C 375 °C to 1000 °C	$\pm 1.5 \ ^{\circ}\text{C}$ $\pm 0.004 \   t \  $	
PT-100 Flowmeter	-100-400 °C 1 m <sup>3</sup> /h to 12 m <sup>3</sup> /h	$\pm (0.15 + 0.002   t  ) \pm 0.5\%$	

 Table 2. Information of the main detectors and instruments.

Through the linear regression calculation of the temperature difference between the water temperature measured at the typical temperature point of the high-precision constant temperature water bath (the temperature control accuracy is  $\pm 0.01$  °C), the measurement error introduced by the external armor and cable of the sensors were corrected by 50–120 °C (the temperature interval is 10 °C). The uncertainty of key parameters of instruments and equipment involved in this study can be estimated according to the basic uncertainty in the list.

## 3. Results and Discussion

Figure 5 demonstrates the temperature evolution of key measuring points of the phase change heat storage device. About 800 cycles of heat charging and heat discharging were performed. Figure 5a shows the temperature variations of water inlet and water outlet, the filled phase change material MNH90 during the first heat charging and discharging operation. Figure 5b illustrates the measurement results after 800 cycles. The temperature difference between the mean temperature of MNH90 and the mean temperature of outlet water is also showed in Figure 5. It can be found that the entire charge and discharge cycle can be divided into six stages according to the temperature evolution of water and MNH90. Stages I, II, and III are charging phases and detected by whether the temperature of outlet water reaches the desired charging temperature, while stages IV, V, and VI are discharging phases and marked by whether the temperature of outlet water reaches the desired charging temperature. Stage I is sensible heat storage. MNH90 in this stage is pure solid, with the continuous heating by high-temperature inlet water, solid MNH90 and outlet water temperature increase rapidly, and the outlet water temperature is about 2 to 4 degrees higher than that of MNH90. When the MNH90 working temperature exceeds 90 °C, the trend of temperature change slows down, and stage II can be regarded as the latent heat storage stage as the change of temperature tends to be gentle. In stage III, the water outlet temperature and MNH90 have obvious temperature jump, indicating that MNH90 is in the stage of sensible heat storage of pure liquid, further heating the results in a significant temperature rise. Stage IV is a transition stage, where the heat charging process ends and the heat discharging process starts. At a specific time, the outlet temperature of water is equal to the MNH90 temperature. Stage V is the phase change process in heat discharging, and MNH90 changes from liquid phase to solid phase and releases latent heat. In stage VI, MNH90 is already in the solid, and depends on the solid heat conduction to exchange heat with the water in the coil.



**Figure 5.** Temperature evolution of key measuring points of the phase change heat storage device (**a**) within the first typical charge–discharge operation (**b**) within the 800th typical charge–discharge operation.

Discharging performance and heat storage efficiency have always been the most concerned problems in the designing of a heat storage device. Figure 6 shows the discharging performance and heat storage efficiency of the small scale latent heat storage unit during the 800 cycles. The high efficiency of heat storage benefits from the thick insulation layer and rapid charging and discharging. However, it can still be seen that after 800 cycles of charging and discharging, the heat storage capacity of the heat storage device still has a slight decline, and it drops from 70.4 kWh to 69.1 kWh, the heat storage performance decreases about 1.8%. When the output temperature is customized according to different application scenarios, this phase change heat storage device with the maximum heat storage temperature of 110 °C is still much higher than that of atmospheric water storage device.





# 4. Conclusions

Using the salt hydrates as PCM is a promising method to replace the traditional atmospheric hot water heat storage. In this paper, the feasibility of a new developed phase change material named MNH90 is analyzed by the characterization of main thermophysical parameters and the experimental cycling performance study of heat charging and discharging. The main research conclusions received through the experiment can be listed as:

- (1) The specific heat capacity of MNH90 at 20 °C is 1.6 J/g·K, and it increases slowly with the increase of temperature. When the temperature rises to 120 °C, the specific heat capacity reaches 4.1 J/g·K.
- (2) The melting point of the material is 91.8 °C, the latent heat of phase change is 151.3 J/g, and in the cooling stage, the solidification temperature of the material is about 68 °C.
- (3) The heat storage density of MNH90 is about 420 MJ/m<sup>3</sup>, which is about 2.5 times of that of water, assuming the temperature difference is 40 °C.
- (4) With thick insulation layer, quick heat charging and heat discharging control method, the heat storage system can get a higher performance. The cycling test shows that the heat storage capacity decreases with the increase of cycle times, and the heat storage performance decreases about 1.8% after 800 operation cycles.

In conclusion, this paper proves the main thermophysical properties of MNH90 and its feasibility as a low temperature heat storage medium by experiments, and reveals the operation characteristics of MNH90 phase change heat storage equipment, which can provide some valuable information for the industrialization of this technology.

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