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Experimental and application study of $Na_2SO_4 \cdot 10H_2O$ with additives for cold storage

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Abstract
Based on the current market demand for effective cold storage for food, we propose an optimal phase change material composed of $Na_2SO_4 \cdot 10H_2O$ and other agents. By means of different scanning calorimetry, transient plane source, temperature time curve, and step cooling curve analysis methods, the effects of various additives to a sodium sulfate hydrate system as a cold storage material were studied. The experimental results show that the optimal percentages of the components of the system are 75.5% $Na_2SO_4 \cdot 10H_2O$, 3% borax, 1.25% PAAS, 16%/4% NH_4Cl/KCl, and 0.25% deionized water, which effectively prevented the supercooling phenomenon. The system's phase change temperature, latent heat, and thermal conductivity were determined to be 6.4 °C, 141 J g^{-1}, and 0.547 W $m^{-1}K^{-1}$, respectively. After 20, 50, and 100 cycles of thermal cycling, the thermal properties of the system were basically unchanged, and the system exhibited good thermal stability. Apples were used as the object of preservation to analyze the sodium sulfate hydrated salt system and cold storage material loaded into an insulation box. Compared to an unloaded insulation box, the material kept the apples fresh for about 9.63 h based on the cold holding temperature of the material. The results suggest that the proposed material with optimal percentages of nucleating, thickening, and cooling agents is feasible and has good preservation characteristics for potential application in cold storage methods for food and medicinal products.

Keywords $Na_2SO_4 \cdot 10H_2O$ · Phase change material · Thermodynamic properties · Insulation box · Cold storage

Introduction

Cold chain logistics require that food must be within specific temperature ranges for production, processing, storage, transportation, sales, and consumption to ensure its quality [1–4]. A large number of food products are transported and sold without cold chain guarantee in China every year, which results in food spoilage and decay, and the loss rate is as high as 20% or more. According to required cold chain temperatures for different transportation means and the principle of phase change storage, various materials with different phase change temperatures can be configured to meet the requirements of food cold chain logistics [5]. Cold storage technology has extensive applications and energy-saving potential in the cold chain of food, such as low-temperature processing, low-temperature storage, low-temperature transportation and distribution, and low-temperature sale, among others [6]. In recent years, sodium sulfate decahydrate ($Na_2SO_4 \cdot 10H_2O$) has gained increasing attention as one of the most typical materials for phase change energy storage due to its high latent calorific value, high energy storage density, good thermal conductivity, and low price.

However, undercooling and phase separation are the two main problems encountered in inorganic phase change materials, particularly in crystallized hydrated salts [7]. Xu et al. [8] found that the composite system of $Na_2SO_4 \cdot 10H_2O$ and $Na_2HPO_4 \cdot 12H_2O$ exhibited good stability without undercooling and phase separation in their study on cold storage technology. Xie et al. [9] employed a microcalorimetric method and determined the activation energy of crystallization of the $Na_2SO_4 \cdot 10H_2O$ system to be 157.8 kJ mol^{-1} and the time index of Avrami equation to be 2.39–2.43. The latter researchers also proposed that the crystallization of $Na_2SO_4 \cdot 10H_2O$ system should follow

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the mechanism of three-dimensional nucleation. Liu et al. [10] investigated the phenomenon of undercooling and phase separation during the phase change process of $Na_2SO_4 \cdot 10H_2O$ and compared the experimental effects of three different thickening agents. Li et al. [11, 12] found that the addition of moderate amounts of thickening agent and nucleating agent could effectively enhance the stability of $Na_2SO_4 \cdot 10H_2O$ as the phase change thermal storage system. Using the physical and chemical dispersion method, Liu et al. [13] prepared nano-composite phase change energy storage materials (PCM), including C–$Na_2SO_4 \cdot 10H_2O$, Cu–$Na_2SO_4 \cdot 10H_2O$, and Al–$Na_2SO_4 \cdot 10H_2O$, and analyzed the effects of nano-C powder, nano-Cu power, and nano-Al powder on $Na_2SO_4 \cdot 10H_2O$, respectively. The results from the latter study suggest that the addition of moderate amounts of nano-materials could effectively improve the undercooling of $Na_2SO_4 \cdot 10H_2O$. In addition, while nano-Cu powder and nano-Al powder settled severely in $Na_2SO_4 \cdot 10H_2O$ system, no obvious stratification was observed after adding nano-C powder to the system. However, with the increase of nano-C, the coefficient of thermal conductivity of PCM was increased.

The insulation box (also simply known as a cooler), developed in the early 1980s [14–16], is an eco-friendly, airtight, and heat-insulated sealed container used to keep food products or materials cool (or hot). Coolers are flexible in size, have good thermal insulation properties, and have become a desirable transport device for vaccine delivery as well as an effective tool for short-distance food transport and the preservation of fruits and vegetables. Margeirsson et al. [17] compare the thermal properties of corrugated cellulose propionate (CP) and foamed polystyrene (EPS) by means of experiments and numerical simulation. Experiments show that ice bags are effective for heat preservation during transportation. Singh et al. [18] and others have combined 12 kinds of heat preservation boxes and carried out thermal resistance tests. At the same time, the heat preservation performance of different heat preservation boxes, bags, and refrigerant in single package transportation was compared. Ice bags or ice boxes are commonly used as cooling materials to maintain a low temperature inside the container so that no refrigeration is needed to maintain cold insulation for cold chain distribution of fruits and vegetables over a long time period [19, 20]. Song et al. [21] investigated the qualities of different cooling materials to use inside the cooler and found that the cooling properties of the insulation box meet the required temperature interval of 2–8 °C for the transport of fruits and vegetables. Insulation boxes are traditionally made with polyurethane, which proves undesirable in thermal insulation property and causes contamination. An inorganic eutectic salt was developed for cold chain logistics, and a heat transfer model was established by Li et al. [22]. Liu et al. [23] used a vacuum-insulated panel and polyurethane foaming to produce a composite thermal insulation material, which decreased the cooling load of insulation box by 15.7% under the effective volume. Kan et al. [24–26] developed tapping polyurethane as the core material of a vacuum-insulated panel to effectively decrease the heat conductivity coefficient to < 10 mW m^{-1} K^{-1}.

The storage and transportation temperature of different food varies in which the optimum storage temperature is 2–8 °C for production, such as vegetables and fruits [27]. In this study, $Na_2SO_4 \cdot 10H_2O$ was chosen as the main heat storage material, which was analyzed with differential scanning calorimetry (DSC). Different nucleating agents, surfactants, and melting point coolants were studied by means of the step cooling curve method, and the effects of different additives on the undercooling degree, phase transition temperature, and latent heat of phase transformation were investigated. After preparing the sodium sulfate hydrated salt system as the phase change material, the phase change temperature, latent heat, thermal conductivity, density, charging and discharging cycle stability, and other parameters were tested. The construction of cold chain logistics insulation tank is by using vacuum insulation panel with $Na_2SO_4 \cdot 10H_2O$ system and cargo insulation test.

Experimental

Materials and equipment

All chemicals, including $Na_2SO_4 \cdot 10H_2O$, borax, sodium polyacrylate (PAAS), ammonium chloride (NH_4C), and potassium (KCl) chloride, used in the experiment were analytically pure and obtained from the National Drug Group Chemical Reagent Co., Ltd. The latent heat value of phase change was measured by DSC (200F3-type differential scanning calorimeter), with a temperature precision < 0.1 °C and enthalpy precision < 0.1. The thermal conductivity was measured by Hot Disk thermal conductivity (TPS2500 type, accuracy < 2).

Step cooling curve experimental system

The experimental system of the step cooling curve is shown in Fig. 1. The temperature of phase change storage material was placed in a glass beaker and measured by a T-type thermocouple. The data were transmitted to the computer through the Agilent data acquisition module 34970A and the data transmission module. The temperature and time data of the phase change storage material during melting and solidification were recorded by Agilent

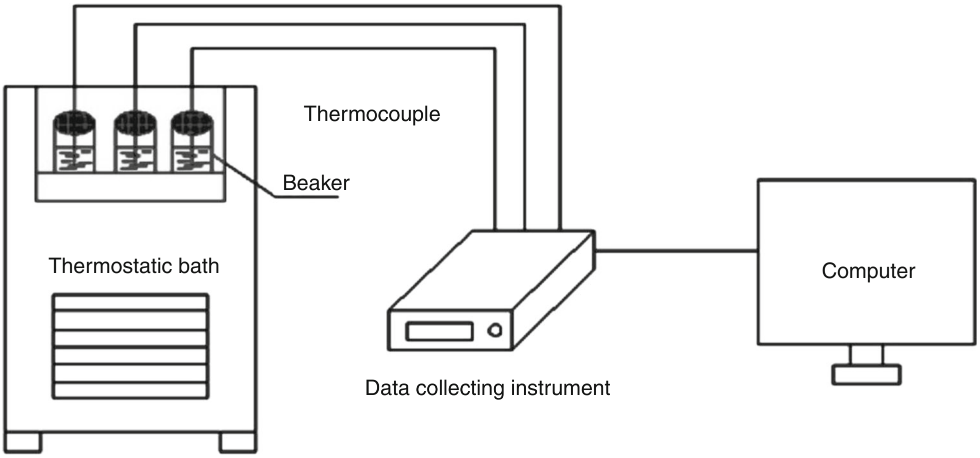

Fig. 1 Cooling curve experiment system

software. The time–temperature curve is drawn by origin software.

Cyclic stability experiment

The high- and low-temperature alternating box was used to circulate the modified phase change materials at 20–50 °C and 100 °C. The temperature range of the high- and low-temperature alternating box was −50 to 50 °C, and the cycle period was 50 min. The melting temperature and latent heat value were measured by DSC after the low-temperature phase change material cycle.

Insulation box experiment

Apples (1500 g) were selected as the test object for the loading application, and 2000 g sodium sulfate cold storage material was used. To prevent contamination by condensation during the test and inhibition of ethylene production caused by ripening of the apples, the apples were wrapped with a preservative film before the test. Because the RFID card cannot monitor the temperature of the apples' epidermis and interior of temperature and humidity, the thermocouple was used to test the temperature. The thermocouple was fixed in the apples' interior and skin, as shown in Fig. 2, then the apples were placed inside the insulation box. The temperature was measured five times at different points inside the cooler to obtain an average value.

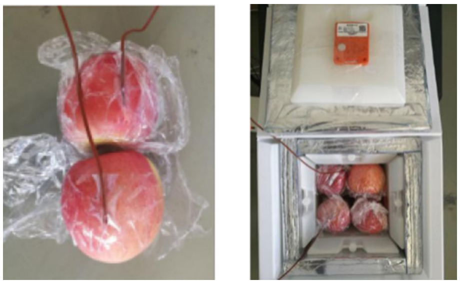

Fig. 2 Apples wrapped in preservation film

Results and analysis

Effect of nucleating agent concentration on $Na_2SO_4{\cdot}10H_2O$ undercooling

The supercooling of pure $Na_2SO_4{\cdot}10H_2O$ is serious, and the degree of undercooling is about 11 °C. Because borax can effectively restrain the undercooling of $Na_2SO4{\cdot}10H_2O$, it was chosen as the nucleating agent in this experiment [28]. Different contents of borax, ranging 1–4%, were analyzed in the 60 g sodium sulfate decahydrate system to understand its effect on the system's temperature and degree of undercooling. Melting and cooling experiments were then carried out on the four groups of samples under different contents of borax. The samples were dissolved in a constant temperature water bath at 45 °C with continuous stirring. After a period of stabilization, the samples were taken out and cooled in a constant temperature tank at 5 °C. The experimental data and step cooling curves are shown in Table 1 and Fig. 3.

In Table 1 and Fig. 3, the undercooling of sodium sulfate hydrated salt system with borax improved obviously as the borax mass percentage increased. The more nucleating agent content there is in the composite phase change material, the more adhesion points are formed to promote the nucleation of the solution, causing a decrease in the undercooling degree of the system. When the content of borax was 3%, the supercooling of the system was basically eliminated. However, as the borax content increased past 3%, the supercooling degree of the system did not change significantly and the phase transition platform became shorter. This also caused volume reduction and

Table 1 Thermophysical properties of borax systems with different contents

Borax content/%	Phase transition initiation temperature/°C	Phase transition peak temperature/°C	Degree of supercooling/°C
1	24.5	30.0	5.5
2	27.7	29.8	2.1
3	28.6	28.6	0
4	28.3	28.3	0

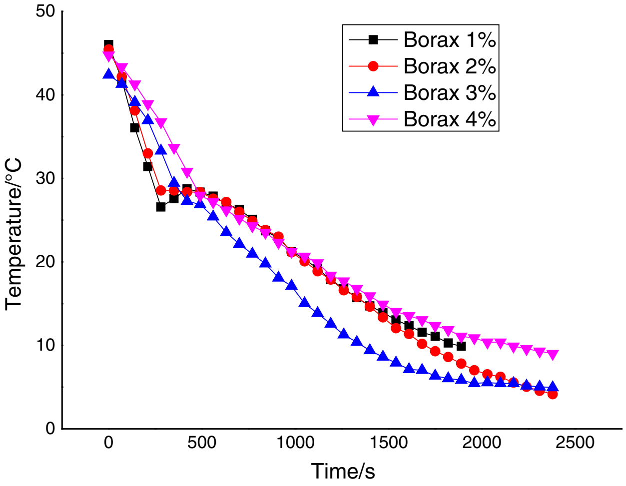

Fig. 3 Cooling curve of different borax contents

increased thermal conductivity of the solution, whereby latent heat affects cold storage of the material; therefore, the optimum borax content was 3%. In addition, the addition of borax also had a certain effect on the phase transition temperature of the sodium sulfate hydrated salt system. As the amount of borax was increased, the nucleation of the solution was promoted, and the phase transition temperature of the system decreased. Thus, the addition of borax in the sodium sulfate hydrated salt system improved the undercooling problem of the system, which is consistent with the purpose of preparing a low-temperature storage agent in this work to reduce the phase change temperature of the system.

Effect of different thickeners on $Na_2SO_4 \cdot 10H_2O$ phase separation

Due to the serious phase separation phenomenon of pure $Na_2SO_4 \cdot 10H_2O$ after melting, it is necessary to add a suitable thickener to enhance the viscosity of the solution. The solid particles in the liquid can be distributed more evenly around the solution rather than deposited on the bottom, which basically eliminates the separation of crystal and liquid [27]. Polyacrylamide (PAM) and sodium polyacrylate (PAASA) were selected as the solution thickening agents. A matrix was prepared with 3% borax and 60 g sodium sulfate, to which 1–3% of each thickener was added separately. The experimental results are shown in Fig. 4 and Table 2.

Thickeners contain a large number of carboxyl polar groups so they easily form hydrogen bonds with water molecules, which increases the viscosity of the system and forms a gel-like substance. In the experiment, the carboxyl polar groups of the thickener increased the viscosity of the sodium sulfate solution in which sodium sulfate inorganic salts were more uniformly distributed. Although no phase separation occurred, the liquid became more viscous with poor fluidity. This further resulted in low thermal conductivity and enhanced the cold volume transfer in the hydrated salt systems containing 3/3% PAAS sulfate and 2%/3% PAAS sodium sulfate.

According to the above experimental results, 1% PAAS and 2% PAM had the best effect on eliminating phase separation. In order to further determine which thickener can more effectively improve the performance of the sodium sulfate hydrated salt system, a DSC test was performed on the systems with 1% PAAS and 2% PAM. The test results are shown in Figs. 5 and 6.

The results of DSC tests show that the phase transition temperature of the 1% PAAS system was 0.425 °C lower than that of the 2% PAM system. The latent heat of phase change is higher than that of 11.1 J g^{-1}. Because we aimed to obtain a phase change material with a low phase transition temperature and high latent heat in this work, 1% PAAS was selected as the thickener additive in the sodium sulfate hydrated salt system.

Determination and analysis of cooling agent

Salt ions weaken the attraction of hydrogen bonds between water molecules and decrease the freezing point of a solution. Therefore, inorganic salts, such as NaCl, NH_4Cl, KCl, and $NaNO_3$, can reduce the eutectic temperature after forming a salt solution with water [17]. In addition, Na ions in NaCl or $NaNO_3$ weaken the strength or decrease hydrogen ions and have a large negative effect on lowering the solidification temperature of eutectic salt solutions. Therefore, we combined NH_4Cl and KCl, instead of NaCl or $NaNO_3$, as the melting point coolants for the $Na_2SO_4 \cdot 10H_2O$ system and studied its effect on the phase transition temperature. Due to the limited eutectic concentration of the inorganic salt solution and to ensure that the temperature of the system could reach 2–8 °C, the content of the total melting point coolant was set to 20%. Mass ratios of 8%/12%, 12%/8%, and 16%/4% of NH_4Cl/KCl were

PAAS PAM

Fig. 4 Effects of different thickeners on phase separation of solution

Table 2 Different amounts of thickener system thermal properties

Name	Content/ %	System phase separation
Polyacrylamide (PAM)	1	Slight phase separation
	2	No phase separation, good effect
	3	Thicker, poor fluidity
Sodium polyacrylate (PAAS)	1	No phase separation, good effect
	2	Thicker, poor fluidity
	3	Thicker, poor fluidity

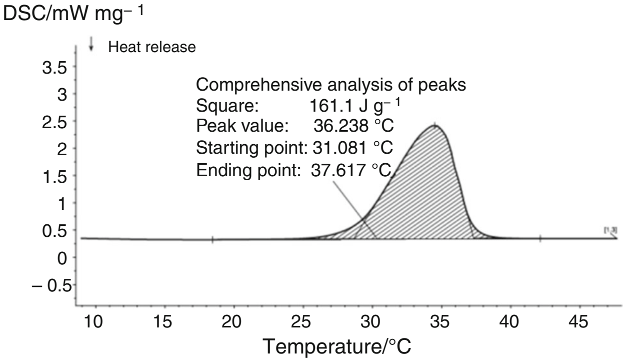

Fig. 5 1% PAAS DSC test system curve

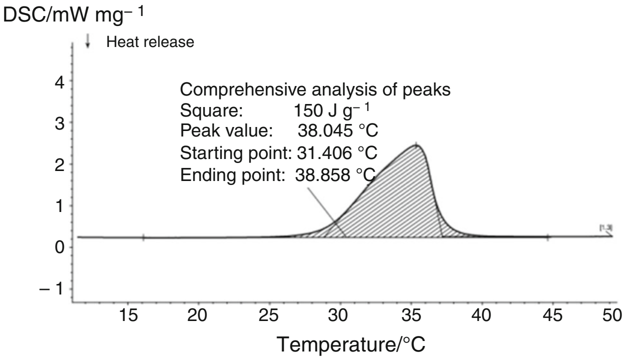

Fig. 6 2% PAM DSC test system curve

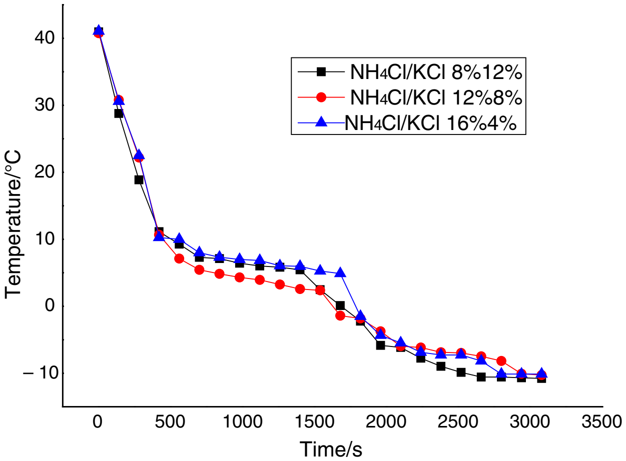

Fig. 7 Cooling curves of different contents of NH_4Cl/KCl

analyzed. To ensure that KCl and NH_4Cl were completely and uniformly dissolved in the sodium sulfate hydrated salt system, 1.25% PAAS thickener was added. The phase change materials of sodium sulfate hydrated salt system of KCl and NH_4Cl with 3% borax 1.25 and different mass ratios were placed in a − 10 °C constant temperature bath for cooling. The step cooling curve and the results of phase transition temperature and latent heat measurement are shown in Fig. 7 and Table 3.

In Fig. 7, it can be seen that the hydrated sodium sulfate system with NH_4Cl and KCl had no undercooling, and the phase transition temperature was about 4 ~ 7 °C, which fits the required temperature range for this work. Table 3 reveals that the phase transition temperature of the system had no obvious change at 2–8 °C, but the latent heat of the phase change increased as the NH_4Cl mass percentage increased. Because we need to prepare a phase change material with high latent heat, we chose the 16%/4% NH_4Cl/KCl combination to further enhance the latent heat of the phase change of the system. Based on the above results, the NH_4Cl/KCl mass percent was adjusted to 16%/4%,16.5%/3.5%, and 17%/3%, and experiments were conducted again. The experimental results show that the maximum latent calorific value was obtained when the mass percent content of NH_4Cl/KCl is adjusted to 16%/4%.

Table 3 Different content of NH4Cl/KCl system DSC

NH_4Cl/KCl mass percentage	Phase transition temperature/°C	Latent heat/ J g^{-1}
8%/12%	6.423	85.66
12%/8%	4.311	108.79
16%/4%	6.986	137.2

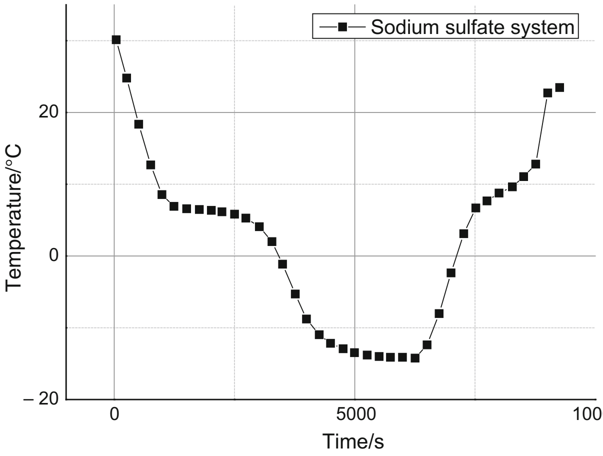

Fig. 8 Solidification melting curve of sodium sulfate hydrate system

Thermal properties and cyclic stability analysis

Based on the experiment results, the optimum proportion of the nucleating, thickening, and cooling agents for the sodium sulfate hydrated system are as follows: 3% borax, 1.25% PAAS, and 16%/4% NH_4Cl/KCl, respectively. It was found that sodium sulfate decahydrate lost some crystalline water in the melting process and 0.25% deionized water was added to the system. By doing so, both the durability of the phase change material and latent heat of the phase change were improved. The optimal contents for the sodium sulfate hydrated salt system were determined to be: 75.5% $Na_2SO_4 \cdot 10H_2O$ + 3% borax + 1.25% PAAS + 16%/4% NH_4Cl/KCl + 0.25% deionized water. The phase transition temperature and latent heat of phase change of the prepared sodium sulfate hydrated salt system were measured by DSC. The phase transition temperature was 6.4 °C, and the latent heat of phase change was 141 J g^{-1}. Hot Disk thermal conductivity analysis was performed on the prepared sodium sulfate hydrated salt system phase change material, and the thermal conductivity coefficient was 0.547 W m^{-1} K^{-1}. The melting curve of the system is shown in Fig. 8, which suggests that the phase change material with one cycle had no undercooling, the phase transition occurred, and completion time of phase transition was about 1000 s.

The DSC and thermal conductivity test of the sodium sulfate hydrated salt system were performed 50 times and 100 times, respectively, and the test results are shown in Table 4. The phase change temperature of sodium sulfate hydrated salt system was about 6 °C, which meets the requirement for the phase change temperature in this work. The latent heat of phase change was determined to be about 137 J g^{-1}; therefore, the material has good stability and can be used as a low-temperature storage agent.

Study on temperature field distribution of preservation of fruit and vegetables

The effects of the insulation box loaded with apples and not loaded were compared in terms of the heating rate, and the results are shown in Fig. 9 and Table 5. It was found that the heating rate of the insulation box loaded with apples was faster than that of the non-loaded box by 6.4 °C, because the apples were not pre-cooled at room temperature. The specific heat capacity of an apple is much larger than that of air, which causes the cold storage agent to absorb heat faster, thus the heating rate of the loaded insulation box was larger than that of the unloaded box. The average time that the loaded box maintained a cool temperature was shorter than that of the unloaded box. It can be seen from Table 5 that the average cold holding time of the loaded box was 9.63 h, which is 0.77 h shorter than that of the unload box. However, the phase change temperature of the cold storage agent was still in the range of 6–8 °C, which means the freshness of the apples was effectively preserved.

In a practical application process for cold storage, we should consider all aspects that may affect the quality of goods being stored to determine reasonable pre-cooling temperature of the cooling agent. Although a low pre-cooling temperature of the cold storage agent can prolong the cold preservation time, it can freeze and damage the quality of the goods.

Table 4 Thermal cycling system

Cycle index	Phase transition temperature/°C	Latent heat/J g^{-1}	Thermal conductivity/W $m^{-1}K^{-1}$
20	6.3	140	0.547
50	6.3	139	0.545
100	6.2	137	0.544

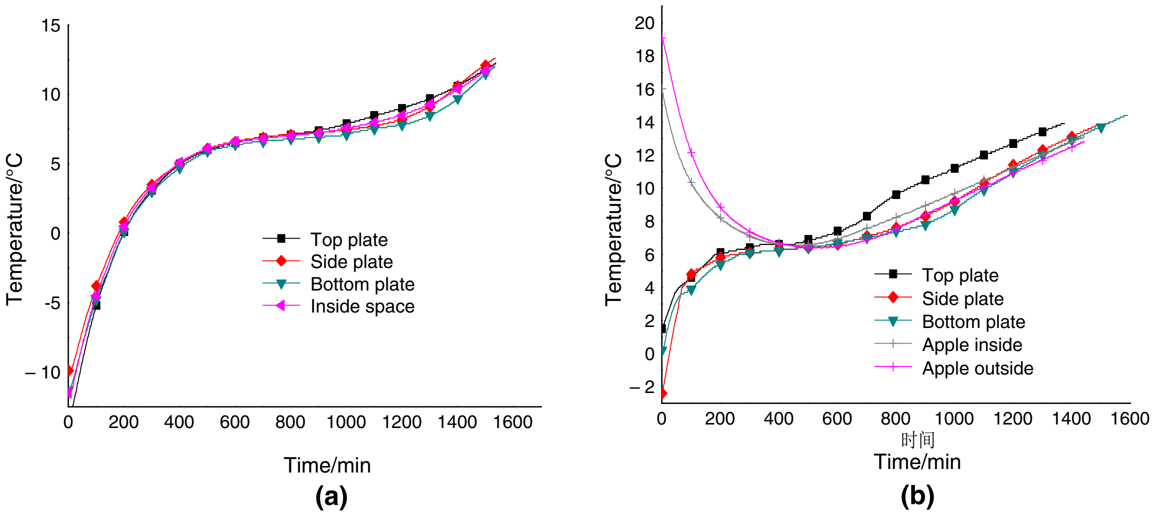

Fig. 9 Temperature variation of loaded and unloaded insulation box. **a** Unloaded, **b** loaded

Table 5 Temperature of measuring point of loaded or unloaded

Cargo situation	Ambient temperature/°C	Location of points to be measured	Cold holding temperature/°C	Cold holding time/h	Average cold holding time/h
Unloaded	21 ± 1.5	Top plate	6–8	8.9	10.77
		Side plate	6–8	11.48	
		Bottom plate	6–8	12.18	
		Inside space	6–8	10.5	
Loaded	21 ± 1.5	Top plate	6–8	8.38	10.00
		Side plate	6–8	10.5	
		Bottom plate	6–8	11.13	
		Apple inside	6–8	9.183	9.63
		Apple outside	6–8	10.067	

Conclusions

Sodium sulfate decahydrate was used as the main energy storage liquid to meet the required 2–8 °C temperature zone for efficient food preservation in cold chain transportation using an insulation box. Different additives with various concentrations were tested to determine the best thickening and cooling agents to add to the sodium sulfate hydrated salt system. The insulation box was constructed with a vacuum insulation board and optimal phase change material, composed of the sodium sulfate hydrated salt system with nucleating, thickening, and cooling agents. Tests were performed on the insulation box that was unloaded and loaded with apples, and the preservation of apples was analyzed. Based on all experimental results, the following conclusions can be made:

1. The undercooling of sodium sulfate decahydrate can be basically eliminated by adding 3% borax and 4% borax, where 3% borax has little effect on the phase transformation platform.
2. 1% PAAS and 2% PAM effectively solved the phase separation phenomenon of sodium sulfate decahydrate solution. The phase transition temperature of the solution with 1% PAAS was 0.425 °C lower than that of solution with 2% PAM, and the latent heat of phase transition of the solution with 1% PAAS was higher than that of 2% PAM.
3. The optimal composition of the sodium sulfate hydrated salt system was determined to be: 75.5% $Na_2SO_4 \cdot 10H_2O$ + 3% borax + 1.25% PAAS + 16%/4% NH_4Cl/KCl + 0.25% deionized water. The phase change temperature was 6.4 °C, which is in accordance with the required temperature range of 2–8 °C for food preservation and storage. The latent heat of phase

change was 141 J g^{-1}, and thermal conductivity was 0.547 W m^{-1} K^{-1}. After 20, 50, and 100 cycles, the phase transition temperature, latent heat, and undercooling had little change and the system had good thermal stability, which is suitable for food preservation and transportation.

4. Apples were chosen as the object of preservation to analyze the sodium sulfate hydrated salt system and vacuum insulation board cold insulation box, which was effectively kept fresh for about 9.63 h. This cold holding time was 0.77 h less than the unloaded insulation box. Overall, the insulation box composed of the proposed cold storage material was determined to have high feasibility and excellent cold preservation characteristics.

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