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1	Thermal performance analysis of a solar energy storage unit
2	encapsulated with HITEC salt/copper foam/nanoparticles composite
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10	Abstract
11	HITEC salt (40 wt. % NaNO <sub>2</sub> , 7 wt. % NaNO <sub>3</sub> , 53 wt. % KNO <sub>3</sub> ) with a melting
12	temperature of about 142 °C is a typical phase change material (PCM) for solar energy
13	storage. Both aluminum oxide (Al <sub>2</sub> O <sub>3</sub> ) nanopowder and metal foam were used to
14	enhance pure HITEC salt, so as to retrieve the limitation of composite PCMs with
15	single enhancement. The morphologies and thermo-physical properties of the
16	composites were firstly characterized with Scanning Electron Microscope,
17	Fourier-transform Infrared spectroscopy and Differential Scanning Calorimeter,
18	respectively. A pilot test rig with a heater of 380 W located in the inner pipe was built,
19	which was encapsulated with HITEC salt, nano-salt (HITEC salt seeded with 2 wt. $\%$
20	$Al_2O_3$ nanopowder) and salt/copper foam composite seeded with 2 wt. % $Al_2O_3$
21	nanopowder as storage media. Then heat storage and retrieval tests of the energy

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1 storage system were conducted both for pure HITEC salt and composite PCMs at 2 various heating temperatures. The temperature evolutions and distributions of the 3 PCMs at different locations were measured, including radial, angular, and axial 4 locations, and the energy and volumetric mean powers during heat storage/retrieval 5 processes were calculated subsequently. The results show that metal foam is generally 6 compatible with the nano-salt. The maximum deviation of the melting/freezing phase 7 change temperatures of the nano-salt/copper foam composite is 3.54 °C, whereas that of the nano-salt/nickel foam composite is 3.80 °C. The specific heats of the nano-salt are 8 9 apparently enhanced with the addition of Al<sub>2</sub>O<sub>3</sub> nanopowder both in solid and liquid 10 states. The system encapsulated with the nano-salt/copper foam composite can be 11 considerably enhanced, e.g. the time-duration of heat storage process at the heating 12 temperature of 160 °C can be reduced by about 58.5%, compared to that of pure salt. 13 The volumetric mean power of heat storage for the nano-salt/copper foam composite at 14 the heating temperature of 180 °C increases to 109.32 kW/m<sup>3</sup>, compared with 53.01 15 kW/m<sup>3</sup> of pure HITEC salt. The information will be helpful for solar system design, 16 construction and application using molten salt for solar energy storage. 17 Keywords: HITEC salt; aluminum oxide nanopowder; metal foam; thermal

18 characterization; heat storage/retrieval

19

#### Nomenclature

#### $c_p$ specific heat, kJ/(kg K)

d	diameter, mm
Ε	energy, kJ/kg
JF	comprehensive performance coefficient
т	mass, kg
$\overline{P}$	mean power, kW
$\bar{P_V}$	volumetric mean power, kW/m <sup>3</sup>
Т	temperature, °C
t	time, s
V	volume, m <sup>3</sup>
Z.	position of thermocouple, mm

# Subscripts

с	charging
d	discharging
i	inner
0	outer
PCM	phase change material

1

## 2 **1. Introduction**

The huge consumption of fossil energy has brought about energy crisis and environmental issues. The applications of renewable energy are keys to developing energy-saving and clean energy technologies [1-4]. The direct usage of solar energy as 1 one of the renewable energy is attracting attention recently, and a large amount of 2 research has been carried out regarding the high-efficiency usage of solar thermal 3 energy in heating and cooling [5-6]. Phase change materials (PCMs) are proposed as a 4 principal way to store the thermal energy regarding the intermittent of solar energy [7], 5 which is named latent thermal energy storage (LTES) and can stabilize the system.

6 Nitrate salts as PCMs have been widely used in the medium temperature range 7 (100~300 °C) of solar energy application [8-9]. However, the main drawbacks of pure salts are the low thermal conductivity and specific heat. Many techniques have 8 9 addressed the issues of pure salts, including extended fins, macroand 10 micro-encapsulation of the PCMs, addition of high-conductivity materials, and 11 impregnating the PCMs into highly conductive porous structures [8-11]. Metal foam 12 with good mechanical and thermal properties can be used as thermal spreader in pure 13 PCMs, which shows attractive heat transfer performance. A number of studies extensively 14 investigated the thermo-physical properties and system performances relating to metal 15 foam composites [12-16]. Zhao et al. [13] experimentally and numerically studied the heat 16 transfer performance enhancement by embedding metal foam in paraffin. It can be seen 17 that the overall heat transfer rates were enhanced by 5~20 times in the solid region and 3~10 times in the liquid region during melting process, respectively. Zhang et al. [14] 18 19 experimentally and numerically studied the heat transfer performance of a LTES unit 20 encapsulated with the salt/metal foam composite. The results indicated that the 21 time-durations of heat retrieval process for the salt/copper foam composite and

1	salt/nickel foam composite showed 28.8% and 19.3% reduction, respectively, when the
2	cooling temperature was 30 °C. Zhang et al. [15] experimentally and theoretically studied
3	the performance of an AISI 321 tube encapsulated with NaNO <sub>3</sub> /KNO <sub>3</sub> , whereas metallic
4	foam and metallic sponge were used to enhance the effective thermal conductivity of the
5	PCM. Yang et al. [16] numerically studied the melting characteristics of a
6	shell-and-tube LTES unit enhanced with metal foam, and the influences of metal foam
7	location and porosity on the thermal performance of the LTES unit were analyzed
8	extensively. It was found that the porosity of metal foam could affect the temperature
9	field of PCMs, and the melting time can be maximally reduced by 88.55% together with
10	<i>j</i> -f factor increased by 5186.91% in comparison with a smooth tube [16].
11	Nanoparticle has a high particle surface energy due to its large specific surface area,
12	and it can make slight improvement in thermal conductivity and lead to keep and increase
13	the specific heat of pure PCM [17-25]. The nanocomposites synthesized with PCMs and
14	nanoparticles have been studied extensively, both for thermo-physical properties and
15	system performances. Navarrete et al. [18] characterized solar salt nano-encapsulated
16	with Al-Cu alloy layer and pointed out that the total energy stored of the system can be
17	increased owing to contribution of the latent heat storage of the nano-encapsulated
18	PCM, e.g., the thermal storage capacity can be increased by 17.8% when the PCM was
19	nanoencapsulated with an aluminium oxide layer. Mayilvelnathan and Arasu [19]
20	prepared and studied the erythritol/graphene composite PCM. It was found that the
21	thermal conductivity could be increased to 1.122 W/(m K) with the addition of 1 wt. %

1	graphene nanoparticles. Zhang et al. [20] synthesized the micro-capsuled PCM
2	(mePCM) with binary carbonate salt, and the mePCM showed an enhanced effective
3	heat capacity of 1.34-fold compared to the best commercialized product in a CSP
4	system. Chieruzzi et al. [21] prepared and characterized the nanofluids with binary salt
5	(NaNO <sub>3</sub> -KNO <sub>3</sub> with 60:40 ratio) and 1.0 wt. % SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub> (silicon dioxide/aluminium
6	oxide) nanoparticles. The results showed that the nanoparticles induced a maximum
7	increase of 52.1% in solid phase and 18.6% in liquid phase of specific heat. Li et al.
8	[22] prepared the binary molten salt nanofluids with nitrates and SiO <sub>2</sub> nanoparticles,
9	where the nanoparticles with different diameters and mass fractions were investigated.
10	It was found that the specific heat was about 17.8 % higher, while the average thermal
11	conductivity was 20.2% higher, in comparison with those of pure molten salt. Liu and
12	Yang [23] investigated the specific heat and latent heat of eutectic hydrate salt doped
13	with TiO <sub>2</sub> nanoparticles. It showed that the specific heats increased by 83.5% in solid
14	state and 15.1% in liquid state with the addition of 0.3% TiO <sub>2</sub> , while the latent heat was
15	up to 6.4%. Song et al. [24] experimentally investigated the effects of the mixing time
16	and stirring rate of preparation of molten-salt nanofluids on the specific heats of
17	quaternary nitrate dispersed with SiO <sub>2</sub> nanoparticles. It showed that the specific heats of
18	the molten-salt nanofluids could be enhanced due to the formation of nanostructures.
19	Ho and Pan [25] investigated the influences of Al <sub>2</sub> O <sub>3</sub> nanoparticle concentrations on the
20	heat transfer characteristics of nano-HITEC salt flowed in a circular tube. Both the
21	mean and local Nusselt numbers exhibited significant enhancement with the addition of

Al<sub>2</sub>O<sub>3</sub> nanopowder, e.g. a maximum enhancement of the mean Nusselt number could be
up to 11.6% when the concentration of Al<sub>2</sub>O<sub>3</sub> nanoparticles was 0.25 wt. %. However,
the thermal conductivities of most nanocomposites are below 1.0 W/(m K).

4 Meanwhile, it has been proven that the combination of metal foam and nanoparticles is a promising solution to enhance the thermo-physical properties of pure salt or other 5 [26-28]. Thus it is of great importance to incorporate high-conductivity nanoparticles 6 7 into metal foam, so as to make the attractive enhancement of pure PCMs. However, it 8 can be seen from the aforementioned research that the thermo-physical properties of 9 nano-salt/copper foam composite was seldom investigated. Besides the thermo-physical 10 properties of the nano-salt at a small scale, it is indispensable to investigate the thermal 11 response of the nano-salt in an energy storage unit. None of the previous studies reported heat transfer investigations of nano-salt with and without porous medium in a 12 13 large scale rig setup, while the heat transfer performance of a storage medium (nano-salt 14 with porous medium) is interesting and needs to be revealed. In the present study, in 15 order to store the thermal energy in the temperature range of 100~200 °C for electricity 16 generation, common HITEC salt (40 wt. % NaNO<sub>2</sub>, 7 wt. % NaNO<sub>3</sub>, 53 wt. % KNO<sub>3</sub>) 17 as a ternary nitrate/nitrite mixture was used as the base material, and Al<sub>2</sub>O<sub>3</sub> nanopowder and metal foam were applied to enhance thermo-physical properties of pure HITEC salt. 18 19 The morphologies and thermal characteristics of the composites were firstly 20 characterized with Scanning Electron Microscope (SEM), Fourier-transform Infrared 21 (FT-IR) spectroscopy and Differential Scanning Calorimeter (DSC), respectively. A

1	pilot experimental rig with a heater located in the inner pipe was built, which
2	encapsulated HITEC salt, nano-salt (HITEC salt seeded with 2 wt. % $Al_2O_3$ ) and
3	nano-salt/copper foam composite as storage media. Then heat storage and retrieval tests
4	of the LTES unit were conducted both for pure salt and composite PCMs, while the
5	temperature distributions of the PCMs at different locations were measured, including
6	radial, angular, and axial locations. The heat transfer characteristics together with the
7	volumetric mean powers of the LTES unit were extensively revealed. The findings
8	from the present study can broaden the application of nano-salt with and without porous
9	medium. The accurate data of the thermo-physical properties of the nano-salt/metal
10	foam composite PCMs play key roles for designing and modeling the LTES system.
11	The output power and heat storage/retrieval rates of the LTES system can be improved
12	greatly with the composite PCMs, and therefore the energy efficiency of the LTES
13	system increases.

### **2.** Materials preparation and experimental set-up

# **2.1 Synthesis of composite PCMs**

NaNO<sub>2</sub> (Alfa Aesar, UK), NaNO<sub>3</sub> (Honeywell Fluka, UK) and KNO<sub>3</sub> (Acros
Organics, UK), all with 99.0 % purity were used as the base materials in the present
study. They were uniformly mixed with the mass ratios of 40:7:53 to form HITEC salt.
Al<sub>2</sub>O<sub>3</sub> nanopowder (40-80 nm APS, Nanostructured & Amorphous Materials Inc., US)
and metal foam (Kunshan Jiayisheng Electronics Co. Ltd., CN) with the porosity of

about 95.0% and pore size of 10 PPI (pores per inch) were used to enhance the
 thermo-physical properties of pure salt. The synthesized process of HITEC salt/metal
 foam composites seeded with Al<sub>2</sub>O<sub>3</sub> nanopowder was shown in Fig. 1.

4 Pure HITEC salt was first dissolved into deionized water, and Al<sub>2</sub>O<sub>3</sub> nanopowder 5 with mass fraction of 1%, 2% or 3% was suspended in the solution, respectively, stirred 6 and sonicated for 60 mins with an ultrasonicator (FB15057) under the power of 600 W 7 and frequency of 37 kHz for good dispersion. Then nickel foam or copper foam was 8 physically immersed in the solution filled in a stainless steel disc. Subsequently the 9 solution after 10 mins sonication was heated in an eurotherm oven (Carbolite Sheffield, UK) at 200 °C to evaporate the water and ensure the impregnation. Finally the disc 10 11 filled with salt/metal foam composites seeded with Al<sub>2</sub>O<sub>3</sub> nanopowder was taken out 12 and naturally cooled, which was done at an oven with 30 °C for 2 mins. The composite 13 PCMs separated were removed and slightly polished with the abrasive paper to make 14 the surface smooth.

15

#### 16 2.2 Characterization of composite PCMs

17 Series characterizations of the properties of the composites were addressed 18 subsequently, and three specimens of each type were tested to ensure the repetition and 19 accuracy of the results. The morphologies of the composites were characterized by 20 tabletop Microscope TM3030Plus (Hitachi High-Technology, Japan), while BSE 21 (Backscattered Electrons) and EDX (Energy Dispersive X-ray Spectrometer) modes were used. FT-IR Perkin Elmer device (Thermo Scientific, NICOLET iS10) was used as the suppliment of the component analysis, contributing to analyze the chemical bonding, molecular structure and degradation effect of the specimens. During the measurement of FT-IR, the specimens were subjected to infrared light and the absorption and emission of wave length were monitored, where the covered wavelength ranged from 400 cm<sup>-1</sup> to 4000 cm<sup>-1</sup> in the present study.

7 A Mettler-Toledo DSC (Mettler Toledo Ltd., Leicester, UK) was used to characterize the phase change behaviours of the composites. The accuracy of 8 9 temperature measurement is within ±0.02 °C, and the resolution of the furnace 10 temperature is within ±0.00006 °C. All the specimens were slightly heated until they 11 reached 50 °C. Then they were subjected to melting-freezing cycles under the same test 12 conditions with the heating and cooling rates of 5 °C/min within a temperature range 13 from 50 °C to 200 °C. Two melting-freezing cycles were conducted and the average 14 values were used to characterize the phase change temperatures and latent heats of 15 HITEC salt, salt/Al<sub>2</sub>O<sub>3</sub> nanopowder nanocomposite (nano-salt), salt/metal foam 16 composite and salt/metal foam composite seeded with Al<sub>2</sub>O<sub>3</sub> nanopowder. In addition, 17 the specific heats were measured and calculated with the multiple curving methods [21, 29], including empty crucible curve, sapphire curve, and specimen curve. The crucibles 18 19 were subjected to the same heating procedure, which was isothermal at 100 °C for 10 20 min, heating from 100 °C to 300 °C with the rate of 25.0 °C/min and kept constant at 21 300 °C for 10 min. Then the specific heats of the specimen were calculated in 1 comparison with those of sapphire already known.

2

#### 3 2.3 Heat storage/retrieval tests of pure salt and composite PCMs

4 **2.3.1 Experimental rig and procedure** 

5 Fig. 2 shows the schematic of the LTES unit, which is with a diameter of 95.0 mm 6 and a height of 300.0 mm. It can be seen from Fig. 2(a) that a cartridge heater, which 7 worked as heat source, was located in the inner pipe made of the stainless steel with a 8 diameter of 25.0 mm, while the surrounding was encapsulated with pure salt or 9 composite PCMs. The power of the heater was provided via a control system using 10 LabVIEW software, with a maximum power of 380 W. Various thermocouples of type 11 K with a diameter of 3.0 mm were inserted at different locations inside the PCMs 12 region, so as to measure the temperature distributions at radial, angular, and axial 13 locations, as shown in Fig. 2(b). The thermocouples labelled with TD2, TD3, TD4 and 14 TD5 were located in the middle axial positions at z=150 mm. Those thermocouples 15 were different at radial direction, and the distances from the centre of the heater are 16 22.5 mm, 27.5 mm, 32.5 mm, 37.5 mm, respectively. Other thermocouples labelled 17 with T3, T4 and T5 were located at z=125 mm, and T6 in the farthest radial direction were at the deepest point. During the tests, T4, T4', T4'' and T4''' located in four 18 19 angular directions were used to check the homogenous heat transfer within the PCMs. 20 In addition, a power meter was used to measure the power consumption as a reference 21 in each experiment. The test rig was covered with multilayer thermal insulation and

radiant shielding (aluminium fibre) materials, so as to reduce the heat loss of the LTES
unit. Because of the safety consideration, a safety pressure relief valve was added to
ensure no pressure accumulated inside the LTES unit.

4 The LTES unit was encapsulated with three types of PCMs, including HITEC salt, 5 salt/2 wt. % Al<sub>2</sub>O<sub>3</sub> nanopowder nanocomposite (nano-salt), salt/copper foam composite 6 seeded with Al<sub>2</sub>O<sub>3</sub> nanopowder, respectively. Because salt and nano-salt were filled in 7 powder state, only 90.0% of the volume of the LTES unit was filled and the left space 8 was used for adapting the thermal expansion of about 7% for the PCM [30]. The 9 HITEC salt powder filled in three cases were 2300 g, 2250g and 2100 g, respectively, 10 with the addition of Al<sub>2</sub>O<sub>3</sub> nanopowder and copper foam in the two latter cases. Thus 11 the mass of the nano-salt and nano-salt/copper foam composite were 2295 g and 2780 g, 12 respectively.

13 The whole LTES unit was at a surrounding temperature of about 10~20 °C initially, 14 then the heat storage tests were conducted. The heating temperatures were set as 160 °C, 15 180 °C and 200 °C for HITEC salt and its composites, respectively, so as to investigate 16 the performances of the LTES unit at various conditions. The heat retrieval tests were 17 started subsequently after the main unit approximately reached the heating temperatures. 18 During heat retrieval process, the whole unit was cooled down naturally at the 19 surrounding temperature of about 13~18 °C. Although the test rig was located in the 20 ventilating cabine of the lab with air conditioning, the temperature of the ventilating 21 cabine still fluctuated, which caused the variation of the surrounding temperature. The

temperature evolutions of all thermocouples were instantly recorded with the time interval of 1.4 s once the tests started, where the interval were determined by a program in Labview software. A first thermal cycle was made to remove the air in salt powder, and the temperature evolutions during heat storage/retrieval processes presented in the present study were obtained under the second thermal cycle.

6

#### 7 2.3.2 Energy and volumetric mean power of the LTES unit

8 The specific heats of pure salt and composite PCMs can be considered 9 approximately as a function of the temperature in the entire temperature range [31]. The 10 latent heat accounting for the phase change process can be incorporated into the 11 specific heat of the PCM, which is named apparent specific heat. In the present study, 12 the energy absorbed by the LTES unit can be obtained with the summation of the latent 13 and sensible energy stored by the PCMs. As a result, the apparent specific heat was 14 used to calculate the energy based on the mass of the PCMs filled in the LTES unit. 15 Then the energy and volumetric mean powers of the LTES unit during heat storage 16 process were calculated as follows, respectively:

$$E_c = m_{PCM} \int_{30}^{T_{heating}} c_{p, PCM} dT$$
<sup>(1)</sup>

$$(2)$$

17 Where  $t_c$  is the time-duration of heat storage considered from 30 °C to the heating 18 temperatures. The energy and volumetric mean powers of the LTES unit during heat

#### 1 retrieval process were defined as follows, respectively:



where 
$$t_d$$
 is the time-duration of heat retrieval from the heating temperatures to the end  
temperatures. As the initial temperatures during heat retrieval were a little larger than  
the heating temperatures,  $t_d$  was adjusted to start from  $T=T_{heating}$ . In the present study,  
the end temperatures during heat retrieval were set as 50 °C, 60 °C and 70 °C,  
respectively, so as to get the insight of the optimal temperature difference between the  
final temperature and surrounding for the application.

8

#### 9 **2.3.3 Experimental uncertainty analysis**

10 The thermocouples were pre-calibrated with the uncertainty of 0.5 °C, and the 11 uncertainty of the position of the thermocouple was about 3.0 mm. The expanded 12 uncertainty of the temperature evolutions can be obtained by the uncertainty 13 propagation analysis, shown as below:

$$\frac{\delta T}{T} = \sqrt{\left(\frac{\delta T_{thermocoulpe}}}{T_{thermocoulpe}}\right)^2 + \left(\frac{\delta z}{z}\right)^2} \tag{5}$$

Thus the maximum uncertainty for the temperature evolutions was determined to be 2.02%. Both the uncertainties of the weight and specific heat of the PCM were 0.5%, and the uncertainty of the time-duration was 1.0%. The overall uncertainty of the volumetric mean power, mainly attributed to the uncertainties in weight and specific
 heat of PCM, temperature evolution and time-duration, was determined to be 2.36%
 with the following equation.

$$\frac{\delta \bar{P}}{\bar{P}} = \sqrt{\left(\frac{\delta m}{m}\right)^2 + \left(\frac{\delta c_p}{c_p}\right)^2 + \left(\frac{\delta T}{T}\right)^2 + \left(\frac{\delta t}{t}\right)^2} \tag{6}$$

4

#### 5 **3. Results and discussion**

#### 6 **3.1 Morphologies of composite PCMs**

7 Fig. 3 shows the SEM images of the salt/Al<sub>2</sub>O<sub>3</sub> nanocomposite with and without 8 metal foam. It can be seen from Fig. 3(a) that Al<sub>2</sub>O<sub>3</sub> nanopowder can mix well with salt, 9 with a granular appearance. The structural interaction with salt molecules can be 10 generated as it is difficult to distinguish nanoparticle from salt. Fig. 3(b)-(c) show that 11 the salt/Al<sub>2</sub>O<sub>3</sub> nanocomposite is generally compatible with metal foam, where the 12 impregnation ratio of the present sample can reach 90.0% [32]. The rugged surface in 13 Fig. 3(c) is caused by the shrinkage of the salt during freezing, as the density of the salt 14 in solid state is larger than that in liquid state [30]. The phenomenon is similar to the 15 previous study of the paraffin/metal foam composites [32].

# Fig. 4 shows the FT-IR analyses of HITEC salt and composite PCMs, where the Y-axis indicates the transmittance ratio of infrared light. It can be seen that the FT-IR absorption spectra are nearly the same for all the specimens, indicating that the existence of the physical bonding of $Al_2O_3$ nanopowder with nitrate molecule does not disturb the

chemical structure interaction for chemical stability. The peaks between 1200 cm<sup>-1</sup>~1400
 cm<sup>-1</sup> are slightly obvious with the addition of Al<sub>2</sub>O<sub>3</sub> nanopowder and metal foam,
 indicating that the good bonding between Al<sub>2</sub>O<sub>3</sub> nanopowder and nitrate molecule exists.

#### 5 **3.2 Phase change behavior of composite PCMs**

6 The variations of the phase change temperatures of pure salt and composite PCMs 7 were characterized with DSC, as shown in Fig. 5. The extrapolated onset melting 8 temperatures of the salt/nickel foam and salt/copper foam composites shifts to 139.67 9 °C and 139.56 °C, respectively, from 138.67 °C for pure HITEC salt. The deviated 10 tendencies of the melting temperatures can be explained by the Clapeyron-Clausius 11 equation, and the same phenomenon was reported in a previous study [32]. It seems that 12 the slight increase of the phase change temperature can be attributed to the restriction of 13 the metal skeleton on the volume expansion. On the contrary, the addition of Al<sub>2</sub>O<sub>3</sub> 14 nanopowder can slightly decrease the extrapolated onset melting temperature and 15 increase the extrapolated onset freezing temperature of pure HITEC salt, e.g., the extrapolated onset melting temperature shifts from 138.67 °C to 137.74 °C, while the 16 extrapolated onset freezing temperature shifts from 141.79 °C to 144.98 °C, compared 17 with those of HITEC salt. The phenomenon of the early occurrence of phase change 18 19 can be attributed to the good combination and dispersion performance of the PCM and 20 nano-promoter [29, 33], as depicted in Fig. 3(a). The combined effects of metal foam 21 and Al<sub>2</sub>O<sub>3</sub> nanopowder contribute to the variation of phase change temperature of the 1 salt/metal foam composite seeded with 2 wt. % Al<sub>2</sub>O<sub>3</sub> nanopowder to some extent, and
2 the maximum deviation of the melting/freezing phase change temperatures of the
3 nano-salt/copper foam composite is 3.54 °C, whereas that of the nano-salt/nickel foam
4 composite is 3.80 °C.

5 Fig. 6 shows the examples of the apparent specific heats of HITEC salt and 6 composite PCMs, which incorporated with latent heat. Table 1 also lists the mean 7 specific heats of HITEC salt and composite PCMs. The specific heats in solid state 8 were calculated within the temperature range of 105~120 °C, while those in liquid state 9 were calculated in the temperature range of 180~280 °C. It can be seen that the specific 10 heats of the nano-salts are apparently enhanced with the addition of Al<sub>2</sub>O<sub>3</sub> nanopowder 11 both in solid and liquid states, while those of the salt/metal foam composite PCMs 12 decrease, compared with that of pure HITEC salt. The maximum enhancement is about 13 12.1% in solid state, and 5.8% in liquid state with a nanopowder concentration of 2 14 wt. %. Therefore, in order to keep the heat storage capacity of the LTES unit, HITEC 15 salt/copper foam composite seeded with 2 wt. % Al<sub>2</sub>O<sub>3</sub> nanopowder was used as the 16 storage medium in the following system tests.

17

#### 18 **3.3 Performance enhancement with Al<sub>2</sub>O<sub>3</sub> nanopowders and metal foam**

#### 19 **3.3.1** Comparisons between pure salt and composite PCM

Fig. 7 shows the temperature evolutions and distributions of HITEC salt and composite PCMs both for heat storage and retrieval processes. Because the

1	thermocouple of $T6$ locates near the wall of the LTES unit, the temperature is lower
2	than other thermocouples due to the heat loss from the lateral surface of the LTES unit,
3	as shown in the pentagonal region of Fig. 7. It can be seen from Fig. 7 (a-I) that the
4	temperature of $T3$ increases more quickly than that of $TD3$ , which is due to natural
5	convection during melting. Based on the calculation with empirical correlation [31], it
6	is found that the equivalent thermal conductivity is five times of pure salt considering
7	natural convection. As shown in the rectangular region in Fig. 7 (a-I), there are slightly
8	difference among T4, T4', T4'' and T4''', which could be attributed to the following
9	reasons. On one hand, there might be several cavities caused by the shrinkage of salt
10	during freezing, inducing the temperature difference during heat storage process while
11	the salt is still in solid state. However, the temperatures of T4, T4', T4'' and T4''' are
12	almost the same while the salt is in liquid state. On the other hand, the positions of the
13	four thermocouples might not be at the radial and axial positions exactly which also
14	slightly affect the comparisons among those test points. In general, the heat transfer
15	seems homogenous at the same radial and axial positions. The heat retrieval process
16	shown in Fig. 7(b) seems very slow, which is due to natural air cooling domination
17	inducing large thermal resistance.

Fig. 8 shows the comparisons of temperature evolutions and distributions of HITEC salt and composite PCMs, whereas points T3, T4 and T5 are selected as the representative thermocouples. The temperature of point T5 of HITEC salt increases more quickly than that of the nano-salt, as shown in **Fig. 8(a)**. The viscosity of the

1	nano-salt increased slightly with the addition of nanoparticles [34], which restricts
2	natural convection to some extent accordingly. It can be seen from Fig. 8(a) that the
3	time-durations of the nano-salt and nano-salt/copper foam composite for heat storage
4	are considerably reduced, e.g., the charging times are 9422 s and 4606 s for the nano-salt
5	and nano-salt/copper foam composite, indicating a reduction of the time-duration by
6	15.1% and 58.5%, respectively, compared with 11095 s for HITEC salt. The apparent
7	enhancement is similar to the previous research [14-15]. However, the discharging times
8	( $T_{end}$ =50 °C) are 35527 s, 42686 s and 41286 s for HITEC salt, nano-salt, nano-salt/copper
9	foam composite, respectively. The phenomenon of slight improvement can be attributed
10	to natural air cooling with large thermal resistance outside the LTES unit dominated the
11	whole process, despite the enhancement of the thermo-physical properties of the
12	nano-salt and nano-salt/copper foam composite. Due to the enhanced heat transfer, the
13	LTES unit with the nano-salt and nano-salt/copper foam composite can reach the higher
14	temperature after heat storage, inducing the larger time-duration of heat retrieval. In
15	addition, due to the variation of the inner temperature of the ventilating cabine in the
16	lab, the experiments of pure HITEC salt were conducted at the surrounding
17	temperatures of about 13-15 °C, and those of nano-salt and nano-salt/copper foam
18	composite were conducted at the surrounding temperatures of about 16-18 °C.

# **3.3.2 Different heating temperatures**

21 Fig. 9 shows the temperature evolutions and distributions of HITEC salt/copper

1 foam composite seeded with 2 wt. % Al<sub>2</sub>O<sub>3</sub> nanopowder at different heating 2 temperatures. It can be seen from Fig. 9(a) that the higher heating temperature 3 controlled can accelerate the melting process, e.g., the charging times are 4711 s and 4 3577 s at the heating temperatures of 180 °C and 200 °C, respectively. This is due to large temperature difference between the heater and PCMs leads to the enhanced heat 5 6 transfer. Fig. 9(b) shows the temperature evolutions during heat retrieval process. The 7 discharging times are about 44258 s and 46298 s when the starting temperatures are 180 8 °C and 200 °C, respectively. More energy can be stored with the higher heating 9 temperature, which induces larger time-duration of heat retrieval subsequently as air 10 cooling dominated the heat transfer process.

11

#### 12 **3.4 Energy and volumetric mean power for heat storage/retrieval**

13 Latent and sensible enthalpy change were estimated according to the mass and 14 apparent specific heats of the PCMs encapsulated in the LTES unit. Then the heat 15 storage and retrieval powers were approximately calculated accordingly, based on the 16 time-duration of heat storage and retrieval processes. The energy and volumetric mean 17 powers of the LTES unit are listed in **Tables 2** and **3**, respectively. Because of the slight 18 decrease of specific heat and large mass of the nano-salt/copper foam composite 19 encapsulated in the LTES unit, the energy stored by the composite PCMs is slightly 20 higher, as shown in **Table 2**.



The volumetric mean powers of heat storage in the LTES unit range from 36.54 to

1 157.91 kW/m<sup>3</sup>, based on different heating temperatures and PCMs. While the volumetric mean powers of heat retrieval from the LTES unit are in the range of 2 8.66~11.93 kW/m<sup>3</sup>, 9.31~13.84 kW/m<sup>3</sup> and 11.99~16.92 kW/m<sup>3</sup> when the final 3 4 temperatures are 50 °C, 60 °C and 70 °C, respectively. It can be seen from Table 3 and 5 Fig. 10 that the mean powers of the LTES unit during heat storage process were 6 significantly improved with the application of the composite PCM, e.g., the mean 7 power for the nano-salt/copper foam composite at the heating temperature of 180 °C increases to 109.32 kW/m<sup>3</sup>, compared with 53.01 kW/m<sup>3</sup> of pure HITEC salt. However, 8 9 the mean powers during heat retrieval process are quite different. There are slight 10 difference among various PCMs, which is due to natural air cooling dominated the 11 whole process, inducing the large time-duration of heat retrieval process.

12 It can also be seen from Table 3 that the volumetric mean powers of heat retrieval 13 for the nano-salt/copper foam composite at the heating temperature of 160 °C are 10.05 kW/m<sup>3</sup>, 10.92 kW/m<sup>3</sup> and 12.90 kW/m<sup>3</sup> when the end temperatures are 50 °C, 60 °C and 14 15 70 °C, respectively. Because the time-duration decreases more apparently in 16 comparison with the reduction of released energy, the volumetric mean power of heat 17 retrieval increases when the end temperature changes from 50 °C to 70 °C. It can be attributed to the reason that the heat transfer process becomes slow when the temperature 18 19 difference between the LTES unit and surrounding decreases. It should be noted that if the 20 high end temperature is considered, the usable energy will be reduced although the 21 volumetric power increases. As a result, the suitable end temperature should be considered in real application. A LTES system can be constructed by aligning many
such LTES units in a heat storage tank with or without heat transfer fluid. The heat
transfer performance of such LTES unit presented is the fundamental information for
performance evaluation of a LTES system, which can be very useful in the real
application of nano-salt with and without porous medium.

6

#### 7 **5** Conclusions

8 HITEC salt/2 wt. % Al<sub>2</sub>O<sub>3</sub> nanopowder (nano-salt) and salt/metal foam composite 9 seeded with 2 wt. % Al<sub>2</sub>O<sub>3</sub> nanopowder were synthesized, and the morphologies and 10 thermal characteristics of the composites were extensively characterized. The heat 11 storage and retrieval characteristics of a pilot test rig were experimentally investigated, 12 which was encapsulated with pure HITEC salt, nano-salt and nano-salt/copper foam 13 composite as storage media. The main conclusions can be drawn as follows:

(1) Both Al<sub>2</sub>O<sub>3</sub> nanopowder and metal foam can mix well with pure HITEC salt, the
addition of Al<sub>2</sub>O<sub>3</sub> nanopowder can slightly decrease the extrapolated onset melting
temperature and increase the extrapolated onset freezing temperature of pure salt.
The maximum deviation of the melting/freezing phase change temperatures of the
nano-salt/copper foam composite is 3.54 °C, whereas that of the nano-salt/nickel
foam composite is 3.80 °C. The specific heats of the nano-salts are apparently
enhanced with the addition of Al<sub>2</sub>O<sub>3</sub> nanopowder both in solid and liquid states.

21 (2) The tests of the LTES unit indicate that both  $Al_2O_3$  nanopowder and copper foam

1	can significantly improve the heat transfer of pure HITEC salt, e.g., the
2	time-duration of heat storage can be reduced by 15.1% and 58.5% for the nano-salt
3	and nano-salt/copper foam composite, respectively. But the time-durations of heat
4	retrieval process are seldom improved as the process is dominated by natural air
5	cooling. Furthermore, the higher heating temperature can accelerate the melting
6	process, while the heat retrieval process will be much slower as more energy stored
7	at higher heating temperature accordingly.

8 (3) The volumetric mean powers of the LTES unit during heat storage process were
9 significantly improved with the application of the composite PCM, e.g., the
10 volumetric mean power for HITEC salt/copper foam composite seeded with 2
11 wt. % Al<sub>2</sub>O<sub>3</sub> nanopowder at the heating temperature of 180 °C increases to 109.32
12 kW/m<sup>3</sup>, compared with 53.01 kW/m<sup>3</sup> of pure HITEC salt. However, the volumetric
13 mean powers during heat retrieval process are quite different, and slight difference
14 among various PCMs exists.

15

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20

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Specific	HITEC	+Al <sub>2</sub> O <sub>3</sub> [26]			HITEC	HITEC	HITEC salt	HITEC salt
heat	salt	1 wt.	2 wt.	3 wt.	salt/nickel	salt/copper	/2 wt. %	/2 wt. %
(kJ/(kg·°C))	[26]	%	%	%	foam	foam [26]	Al <sub>2</sub> O <sub>3</sub> /nickel	Al <sub>2</sub> O <sub>3</sub> /copper
							foam	foam
Solid state	1.40	1.57	1.57	1.54	1.18	1.32	1.39	1.42
Liquid state	1.56	1.64	1.65	1.63	1.23	1.50	1.41	1.55
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**Table 1** Specific heats of HITEC salt and composite PCMs

<i>E</i> (kJ)		Pure HITEC	Salt/2 wt. % Al <sub>2</sub> O <sub>3</sub>	Salt/copper foam composite seeded
		salt	nanopowder	with 2 wt. % Al <sub>2</sub> O <sub>3</sub> nanopowder
Heat storage	<i>T<sub>initial</sub></i> =30 °C, <i>T<sub>end</sub></i> =160 °C	722.06	736.55	828.14
	T <sub>initial</sub> =30 °C, T <sub>end</sub> =180 °C	803.17	814.22	917.41
	<i>T<sub>initial</sub></i> =30 °C, <i>T<sub>end</sub></i> =200 °C	875.58	890.19	1006.13
Heat retrieval	$T_{initial}$ =160 °C, $T_{end}$ =50 °C	652.04	658.35	739.45
	$T_{initial}$ =180 °C, $T_{end}$ =50 °C	733.14	736.02	828.72
	$T_{initial}$ =200 °C, $T_{end}$ =50 °C	805.56	811.99	917.44
	$T_{initial}$ =160 °C, $T_{end}$ =60 °C	617.04	621.03	696.84
	$T_{initial}$ =180 °C, $T_{end}$ =60 °C	698.27	697.02	784.04
	$T_{initial}$ =200 °C, $T_{end}$ =60 °C	770.55	774.39	874.34
	$T_{initial}$ =160 °C, $T_{end}$ =70 °C	582.01	581.93	652.49
	$T_{initial}$ =180 °C, $T_{end}$ =70 °C	663.26	657.92	739.70
	$T_{initial}$ =200 °C, $T_{end}$ =70 °C	735.54	735.29	829.99

**Table 2** Heat storage and retrieval energies for pure salt and composite PCMs

$\overline{P}_{\rm e}$ (k)	$W/m^3$ )	Pure HITEC	EC Salt/2 wt.% Al <sub>2</sub> O <sub>3</sub> Salt/copper foam composite	
T <sub>V</sub> (R	(())))))))))))))))))))))))))))))))))))	salt	nanopowder	with 2 wt.% Al <sub>2</sub> O <sub>3</sub> nanopowder
Volumetric mean power of	<i>T<sub>initial</sub></i> =30 °C, <i>T<sub>end</sub></i> =160 °C	36.54	43.89	100.94
neat storage	<i>T<sub>initial</sub></i> =30 °C, <i>T<sub>end</sub></i> =180 °C	53.01	66.63	109.32
	T <sub>initial</sub> =30 °C, T <sub>end</sub> =200 °C	81.84	96.09	157.91
Volumetric mean power of	T <sub>initial</sub> =160 °C, T <sub>end</sub> =50 °C	10.30	8.66	10.05
heat retrieval	<i>T<sub>initial</sub></i> =180 °С, <i>T<sub>end</sub>=50</i> °С	11.07	9.01	10.51
	<i>T<sub>initial</sub></i> =200 °C, <i>T<sub>end</sub></i> =50 °C	11.93	9.47	11.12
	T <sub>initial</sub> =160 °C, T <sub>end</sub> =60 °C	10.94	9.31	10.92
	T <sub>initial</sub> =180 °C, T <sub>end</sub> =60 °C	11.97	9.51	11.33
	<i>T<sub>initial</sub></i> =200 °C, <i>T<sub>end</sub>=</i> 60 °C	13.84	10.00	11.93
	T <sub>initial</sub> =160 °C, T <sub>end</sub> =70 °C	13.62	11.99	12.90
	T <sub>initial</sub> =180 °C, T <sub>end</sub> =70 °C	14.74	12.27	13.99
	<i>T<sub>initial</sub></i> =200 °C, <i>T<sub>end</sub></i> =70 °C	16.92	12.12	14.85

1 Table 3 Volumetric mean powers of heat storage and retrieval for pure salt and

2 composite PCMs



Fig. 1. Schematic of synthesis of salt/metal foam composites seeded with  $Al_2O_3$ 

nanopowder. [26]





Fig. 2. Schematic of experimental test rig (a) section of test rig (b) layout of thermocouples. (Unit: mm)







(c)

**Fig. 3.** SEM images of HITEC salt/Al<sub>2</sub>O<sub>3</sub> nanopowder with and without metal foam (a) salt/2 wt. % Al<sub>2</sub>O<sub>3</sub> nanopowder [26] (b) salt/nickel foam composite seeded with 2 wt. % Al<sub>2</sub>O<sub>3</sub> nanopowder (c) salt/copper foam composite seeded with 2 wt.% Al<sub>2</sub>O<sub>3</sub>

nanopowder.



Fig. 4. FT-IR analyses of HITEC salt and composite PCMs.



Fig. 5. DSC curves of HITEC salt and composite PCMs



Fig. 6. Apparent specific heats of HITEC salt and composite PCMs.





**Fig. 7.** Temperature evolutions and distributions of HITEC salt and composite PCMs ( $T_{heating}$ =160 °C) (a) heat storage (b) heat retrieval. I, II, III marks represent HITEC salt, HITEC salt/2 wt. % Al<sub>2</sub>O<sub>3</sub> nanopowder

and HITEC salt/copper foam composite seeded with 2 wt. % Al<sub>2</sub>O<sub>3</sub> nanopowder, respectively.

1







(b)

**Fig. 8.** Comparison of temperature evolutions and distributions of HITEC salt and composite PCMs ( $T_{heating}$ =160 °C) (a) heat storage (b) heat retrieval. The closed,

half-open and open marks represent HITEC salt, HITEC salt/2 wt. % Al2O3

nanopowder and HITEC salt/copper foam composite seeded with 2 wt. %  $Al_2O_3$ 

nanopowder, respectively.



**Fig. 9.** Temperature evolutions and distributions of HITEC salt/copper foam composite seeded with 2 wt. % Al<sub>2</sub>O<sub>3</sub> nanopowder at different heating temperatures (I: heat storage; II: heat retrieval)

(a) 180 °C (b) 200 °C.



(a)



(b)

Fig. 10. Volumetric mean powers for HITEC salt and composite PCMs (a) heat

storage (b) heat retrieval ( $T_{end}$ =50 °C).