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## Laboratory test of a prototype heat storage module based on stable supercooling of sodium acetate trihydrate

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### Abstract

Laboratory test of a long term heat storage module utilizing the principle of stable supercooling of 199.5 kg of sodium acetate water mixture has been carried out. Avoiding phase separation of the incongruently melting salt hydrate by using the extra water principle increased the heat storage capacity. An external expansion vessel minimized the pressure built up in the module while heating and reduced the risk of instable supercooling. The module was stable supercooled at indoor ambient temperature for up to two months after which it was discharged. The energy discharged after activating the supercooled sodium acetate water mixture was 194 kJ/kg of sodium acetate water mixture in the first test cycles dropping to 179 kJ/kg in the later test cycles. Instability of the supercooling occurred when the charging periods were short and in the last test cycles where the tube connecting the module to the expansion vessel had been blocked by the salt hydrate.

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*Keywords:* Seasonal heat storage, sodium acetate trihydrate, supercooling, prototype testing, thermal energy storage

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### 1. Introduction

Heating buildings and domestic hot water usage accounts for a large part of our energy use. Solar energy is more abundant in summer than in the winter periods when heating demands are much larger. Thermal energy storage technologies are therefore needed to match the intermittent supply of solar energy with varying heating demands. Currently available heat storage systems that use water as the storage medium work well but mainly for short term

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storage, as their continuous heat losses limit the storage period. For long storage periods alternative technologies are needed. Storages utilising the latent heat of fusion of a material has been suggested for improving the performance compared to the storage of sensible heat [1]. Subcooling/supercooling of Phase Change Material (PCM) in the latent heat storage has been seen as an undesired effect that had to be avoided by using various nucleation agents as it prevented the heat of fusion from being released as desired when the melting point of the storage material was reached during the discharge process [2]. The idea of utilizing a supercooled salt hydrate for long term storage has however been known since the late 1920s [3] and pocket-sized heat packs storing heat in supercooled sodium acetate trihydrate were patented in 1976 [4]. Well-functioning large scale applications utilizing supercooled PCM for long term storage have not previously been reported.

Sodium acetate trihydrate with a melting point of 58°C and a relatively high heat of fusion of 264 kJ/kg [5], have been shown to supercool consistently down to temperatures well below 0°C [6, 7]. As sodium acetate trihydrate is an incongruently melting salt hydrate it will tend to suffer from phase separation especially over repeated heating and cooling cycles, which will reduce the storage capacity [3, 8]. Adding extra water to the salt hydrate so that the salt water mixture composition is always at a point where all salt is dissolved in the water when it is in the supercooled liquid phase has been proposed as a solution [9, 14]. This requires soft mixing of the salt water mixture to avoid phase separation and it reduces the energy density of the storage.

When the ever-present impurities in the salt hydrate work as nucleation agents, large PCM volumes may show reduced stability of the supercooling, as the chance of a spontaneous nucleation caused by an impurity increases with the volume [7]. Heating the salt water mixture to a high temperature will cause some evaporation of the crystal water, so a closed container with no loss of water through the tank material over the lifetime of the storage unit is essential to avoid changing the composition of the salt water mixture. As the metastable state of the supercooled salt hydrate could easily be interrupted by external influences, a closed container is inherently more stable. Diffusion of the heat transfer fluid from the heat exchanger into the PCM should be avoided. When considering the choice of tank material, corrosion and chemical reactions between the salt and the tank material must be considered. Steel and stainless steel in combination with sodium acetate trihydrate has been shown to be stable over long periods [10]. When using rigid constructions such as steel the density change between the cold solid and the warm liquid salt hydrate must be considered, as the volume change of the PCM in a closed container can cause pressure changes and deformations of the tank. Just as bending a metal disk with cracks works as a triggering mechanism for the pocket sized heat packs [11], small cracks on the inside of the tank can in combination with pressure changes and deformations work as an uncontrolled activation mechanism e.g. at joints or weldings. The volume reduction that takes place in sodium acetate trihydrate when changing from the liquid to the solid state could form cavities inside the tank that reduce the heat transfer rate between the PCM and the heat transfer fluid [5].

To achieve supercooling of the sodium acetate water mixture it is necessary that all crystals of the bulk are melted so that the sodium acetate water mixture will not crystallise as it cools down. There appears to be a link between the level of heating above the melting point and the stability of supercooling [12], and a temperature of around 20 K above melting point is needed in the entire volume to achieve stable supercooling.

Crystallization and release of the heat of fusion from the supercooled sodium acetate trihydrate is initialised when the first seed crystal of a certain size is present in the solution, after which the crystallization will spread to the entire volume and the temperature will rise to close to the melting point of 58°C [7]. Supplying the seed crystal to trigger the crystallization in a reliable and controllable way is essential for such a system. Ultra-sonic sound has been tried and found reliable as an activation mechanism [13]. The metallic disc of the pocket sized heat packs also works reliably but needs moving parts submerged in the solution and therefore also poses a risk of uncontrollable activation. Mechanically dropping a crystal into the supercooled solution might also be a viable solution. Lastly, it has been shown that cooling a supercooled sodium acetate water mixture to a low temperature will eventually cause crystallization of the supercooled solution [6].

As the entire volume will crystallize once it is activated, it is desirable to divide the storage unit into a number of separate modules that can be operated individually. The size and number of modules would depend on the application into which it is to be integrated.

**Nomenclature**

$Q$	power [W]
$V$	volume flow rate [m <sup>3</sup> /s]
$c_p$	specific heat capacity [J/kgK]
$C$	heat capacity [j/K]
$\rho$	density [kg/m <sup>3</sup> ]
$T$	temperature [°C]
$H_{loss}$	heat loss coefficient [W/K]
$E$	energy [J]
$t$	time [s]
$\eta$	efficiency [-]
$m$	mass [kg]
$L$	latent heat of fusion [J/kg]
Subscript:	
$in$	inlet
$out$	outlet
$amb$	ambient
$s$	storage
$supercool$	supercooled state
$melt$	melting temperature
$start$	starting temperature
$max$	maximum temperature
$module$	storage module
$end$	ending temperature
$cont$	energy content
$s$	solid
$l$	liquid
$dis$	discharging
$charge$	charging
$theo$	theoretical

**2. Method***2.1. Experimental investigation*

A prototype module was developed incorporating solutions to the above issues that affect the stability of the supercooling and the avoidance of phase separation. The module was designed as a flat rectangular chamber of steel with an internal height of 5 cm to reduce the risk of phase separation by allowing mixing of the salt water mixture by convection during heating. In one end of the module a 300mm wide 100 mm high extension in the width of the module was incorporated for allowing expansion of the sodium acetate water mixture as it is heated. Heat exchangers with manifolds on two sides, 16 parallel channels covering the entire bottom surface and 14 channels covering the top surface of the PCM chamber were incorporated onto the outer surface of the PCM chamber to transfer heat through the PCM chamber walls, as shown in Fig. 1 and 2. In this way, the PCM could be heated to a relatively uniform temperature to obtain complete melting of all crystals. The internal height of the heat exchanger channels were 4 mm and the width of the channels were 130 mm separated by 20 mm spacers. The width and length of the module was 1200 x 2400 mm (Fig. 2). The internal volume of the chamber was approximately 144 litres for the PCM and an extra 30 litres for the expansion. The material thickness of the steel plates used was 2 mm. The total mass of the empty module was 235.6 kg. The total volume of heat transfer fluid in the heat exchangers including manifolds was estimated to be 32 litres. An inflatable plastic bag or an expansion vessel without prepressure was

connected to the expansion volume via a tube to allow for expansion of the sodium acetate water mixture without pressure build up inside the module.

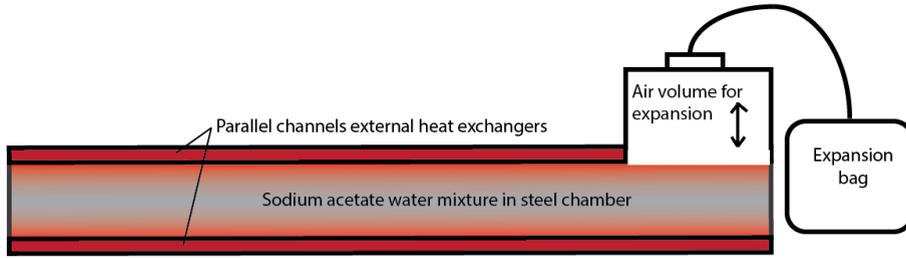


Fig. 1. Diagram of module with PCM chamber, external heat exchangers on top and bottom surface, integrated and external expansion.



Fig. 2. 1200 x 2400 x 60 mm module with integrated heat exchangers on the top and bottom surface and expansion volume.

The inner surfaces of the PCM chamber were designed to be simple and smooth without cracks or gaps, in order to avoid spontaneous nucleation caused by pressure changes in any cracks. 30 pipe segments of 50 mm length were welded inside in the PCM chamber to the top and bottom surface to work as supports and to provide a rigid construction of the module (Fig. 3a). Three openings to the PCM chamber were provided for filling the PCM into the chamber and for installing the tube for the expansion bag and a probe to measure the temperature of the PCM. These filling necks were closed with lids and 3 mm thick rubber gaskets (Fig. 3b). The filling necks were placed in the upper area of the expansion volume to avoid contact between the PCM and the lid or gasket during operation.

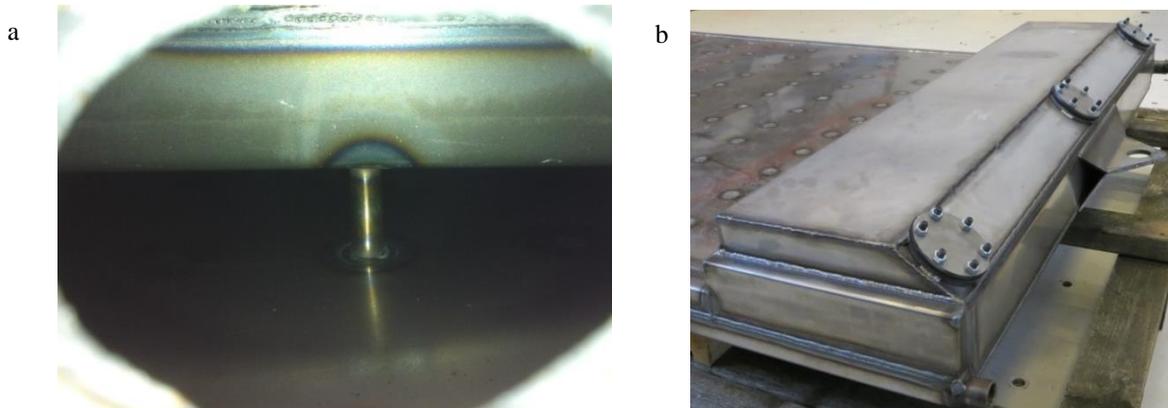


Fig. 3. (a) View of round stabilizer through filling neck; (b) Expansion volume with filling necks with rubber gaskets and lids.

A 100 ml chamber was welded to the outside of the module, in good thermal contact with the PCM chamber in the opposite end of the expansion volume. In this chamber liquid CO<sub>2</sub> with a pressure of 5-6 bars could be flushed through to cool a small part of the PCM through the chamber wall to a low temperature as the CO<sub>2</sub> evaporated, in order to initialise nucleation [6], as shown in Fig. 4.



Fig. 4. CO<sub>2</sub> container for activating supercooled sodium acetate water mixture by cooling.

A sodium acetate water mixture was prepared by melting the solid sodium acetate trihydrate in a barrel with a lid in a large oven. Water was added to increase water content from 39.7 % by weight of the sodium acetate trihydrate composition to approximately 45 % water. The module was filled with the liquid sodium acetate water mixture at a temperature of approximately 80 °C in a slightly tilted position to achieve full filling of the PCM chamber. In the end of the module with the expansion volume the liquid sodium acetate water mixture was filled to a level of approximately 4 cm higher than the internal height elsewhere in the module of 5 cm. With the density of the liquid sodium acetate water mixture at 80 °C of 1275 kg/m<sup>3</sup> this would correspond to a uniform height of the PCM of 5 cm in solid state at 25 °C in the entire module if the crystallization would occur homogeneously without air pockets in the PCM chamber. During filling, samples of the sodium acetate water mixture were taken to determine the actual water content of the sodium acetate water mixture that was filled into the module.

The module contained 199.5 kg of sodium acetate water mixture consisting of 44.8 % water and 55.2 % sodium acetate by weight. The module was supported on a bed of 100 mm of insulating material and a layer of 100 mm foam insulation was placed on the top and around the sides of the module.

The module was connected to a heat storage test facility so that it could be heated and cooled under controlled conditions. 20 test cycles with different flow rates, charge and discharge powers, temperatures settings and durations were carried out over an 8 month period.

## 2.2. Test procedure

The following describes the conditions for the 14<sup>th</sup> test cycle. For this test the power of the heating element in the test facility was 6 kW. The module was charged for a period of 21 hours with an inlet temperature of 91-92°C with a flow rate of 10.5 – 11 l/min in each heat exchanger. Inlet, outlet and storage temperatures were stable at 89-92°C after approximately 8 hours. After the stable period the module was discharged to supercooled state with a flow rate of 2 l/min in each heat exchanger to stable temperatures of approximately 26 °C.

After the module had remained in a supercooled state for 3 days the crystallization was started by flushing CO<sub>2</sub> through the CO<sub>2</sub> container and the energy released after the activation was discharged with a flow rate of 2 l/min in each heat exchanger. Water was used as heat transfer fluid.

The charge and discharge powers of the storage module were determined by:

$$Q_{charge} = V \cdot c_p \cdot \rho \cdot (T_{in} - T_{out}) \quad (1)$$

where,  $T_{in}$  is the inlet temperature,  $T_{out}$  is the outlet temperature,  $V$  is the volume flow rate of the heat transfer fluid measured at the inlet,  $c_p$  is the specific heat capacity of the heat transfer fluid at mean temperature between  $T_{in}$  and  $T_{out}$ ,  $\rho$  is the density of the heat transfer fluid at  $T_{in}$ .

The heat loss coefficient of the storage module was measured by heating the module to a stable temperature over a long period in which no additional energy was added to the module. The energy balance of the system was used to determine the heat loss experimentally e.g. the energy added to the system was equal to the heat loss when the storage temperature remained stable over a period. In this way a heat loss coefficient with a constant value could be determined for the specific temperatures.

$$H_{loss} = Q_{charge} / (T_s - T_{amb}) \quad (2)$$

where  $T_s$  is the heat storage temperature of the module at a stable hot period,  $T_{amb}$  is the ambient temperature. The heat loss coefficient for the storage module was used when calculating energy content of the storage based on the measured data.

The change of heat content in the storage module over a specific time period during a charge is determined by:

$$E_{chargemeasured} = \int_0^{t_c} [Q_{charge} - H_{loss} \cdot (T_s - T_{amb})] dt \quad (3)$$

where  $T_s$  is the heat storage temperature in the relevant time step,  $t_c$  is the duration of the charge period.

The efficiency of the module in long term storage was evaluated as the energy discharged from the module after activation of the supercooled sodium acetate water mixture  $E_{supercool}$  related to the energy charged in the storage module excluding the heat losses  $E_{charge}$  calculated by using Eq. 3. By this definition it is assumed that all of the sensible heat from the fully heated module to the supercooled state is not utilized.

$$\eta_{long term} = E_{supercool} / E_{charge} \quad (4)$$

Copper/constantan thermocouples were used to measure the surface temperature of the module at the outside of the heat exchangers and at the front and back of the module and near the lid. One thermocouple in a probe inserted through one of the filling necks measured the temperature of the PCM. The heat storage temperature  $T_s$  is determined with some inaccuracy as the average temperature of the thermocouples on the outer surface of the heat storage module. Thermopiles were used to measure the temperature difference across the inlets and outlets and two Brunata flow meters, type HGQ1 were used to record the flow rate in the top and bottom heat exchanger. Solartron cards were used with a PC to record data every 10 seconds.

### 2.3. Theoretical calculations

The measured thermal energy charged to the storage module was compared with the theoretical energy content. The following theory explains the thermal energy content of the sodium acetate water mixture in a simplified way, assuming that the salt water mixture behaves as an ideal compound which changes phase from solid to liquid at a specific melting temperature. When extra water is added to the salt hydrate the phase change will happen over a temperature range [14]. In these investigations where the focus is on the stable initial, fully charged, supercooled and discharged conditions the simple compound theory with melting at a specific temperature provides a sufficient basis for comparison.

The following equations show the theoretical change of thermal energy in the heat storage module for a given charge. The equation consists of the sensible heat of the salt hydrate in solid and liquid state, the latent heat of fusion of the salt hydrate and the sensible heat of the storage module material.

$$E_{chargetheo} = m \cdot ((T_{melt} - T_{start}) \cdot c_p(s) + L + (T_{max} - T_{melt}) \cdot c_p(l)) + C_{module} \cdot (T_{max} - T_{start}) \quad (5)$$

where  $m$  is the mass of the salt water mixture,  $T_{melt}$  is the melting temperature of the sodium acetate water mixture of 58°C,  $T_{start}$  is the mean storage temperature at the start of the charge,  $c_p(s)$  is the specific heat of the sodium acetate water mixture in solid phase,  $c_p(l)$  is the specific heat of the sodium acetate water mixture in its liquid phase,  $L$  is the latent heat of fusion,  $C_{module}$  is the heat capacity of the storage module material including the heat transfer fluid in the heat exchangers, and  $T_{max}$  is the mean maximum temperature the heat storage module reaches during a charge.

When the storage module is discharged from the fully charged state at  $T_{max}$  to a temperature  $T_{supercool}$  without crystallising, just the sensible heat of the module and the liquid sodium acetate water mixture is discharged. Assuming that the thermo physical properties of the supercooled sodium acetate water mixture are the same as the liquid sodium acetate water mixture the discharged energy  $E_{dis}$  can be expressed by:

$$E_{dis,theo} = (m \cdot c_p(l) + C_{module}) \cdot (T_{max} - T_{supercool}) \quad (6)$$

where  $T_{supercool}$  is the storage temperature at supercooled state.

The stored thermal energy at supercooled state can be expressed using Eq. 7. Here the discharged energy expressed in Eq. 6 is subtracted from the charged energy expressed by Eq. 5.

$$E_{supercool,theo} = E_{charge,theo} - E_{dis,theo} \quad (7)$$

If the temperature of the storage module at supercooled state  $T_{supercool}$  and end temperature  $T_{end}$  after the activation and discharge are the same.

$$T_{supercool} = T_{end} \quad (8)$$

then the thermal energy stored at supercooled state  $E_{supercool,theo}$  at a temperature  $T_{supercool}$  can be expressed by:

$$E_{supercool,theo} = m \cdot (L - (T_{melt} - T_{supercool}) \cdot (c_p(l) - c_p(s))) \quad (9)$$

If the storage is discharged to a temperature that differs from that of the supercooled state, the discharged energy is corrected for the difference in sensible heat of the module material and the sodium acetate water mixture in its solid state.

$$E_{correct,theo} = (T_{supercool} - T_{end}) \cdot (C_{module} + m \cdot c_p(s)) \quad (10)$$

The energy released per unit mass from activating a supercooled sodium acetate water mixture at a specific temperature, corrected for a difference between the final discharge temperature and the temperature of the supercooled sodium acetate water mixture before activation is:

$$E_{cont,theo}(T_{supercool}) = \frac{E_{supercool,theo} - E_{correct,theo}}{m} \quad (11)$$

#### 2.4. Material properties

The specific heat capacities for the solid and liquid phase of the salt water mixture were determined using the findings of Araki [15]. Non temperature dependent specific heat capacities for the solid and liquid phase sodium acetate water mixture were determined as the average over the relevant temperature intervals. The latent heat of fusion according to the findings of Araki was estimated to be 189.4 kJ/kg for the 44.8% water 55.2% sodium acetate mixture. Values are listed in Table 1.

Table 1. Thermal properties for sodium acetate water mixture for 44.8 % water 55.2 % sodium acetate.

Material property	value
Solid specific heat $c_p(s)$	2.09 kJ/kgK
Liquid specific heat $c_p(l)$	3.17 kJ/kgK
Latent heat of fusion L	189.4 kJ/kg

With a specific heat of steel of 500 J/kgK, and 32 litres of water in the heat exchangers the theoretical heat capacity of the storage module was determined to be:

$$C_{module} = 235.6 \text{ kg} \cdot 500 \frac{\text{J}}{\text{kgK}} + 32 \text{ kg} \cdot 4180 \frac{\text{J}}{\text{kgK}} = 252 \frac{\text{kJ}}{\text{K}} \quad (12)$$

### 3. Results

The following summarizes the results for the 14<sup>th</sup> test cycle. With a starting temperature of 24.6°C the module was charged with a flow rate of 21 l/min combined in both heat exchangers. Stable conditions with an average heat storage temperature of 90.1°C were reached after approximately 8 hours of charging. The heat loss coefficient was experimentally determined to be  $H_{loss} = 8 \text{ W/K}$  in the stable hot state from the 9<sup>th</sup> to the 21<sup>st</sup> hour.

The total energy stored in the module, including sodium acetate water mixture and steel, was for this temperature rise after 8 hours 91,400 kJ based on Eq. 3. The accumulated heat loss over time, the energy stored in the module and the total energy charged into the module including the heat loss are shown in Fig. 5. The accumulated heat loss after 8 hours was 10,800 kJ.

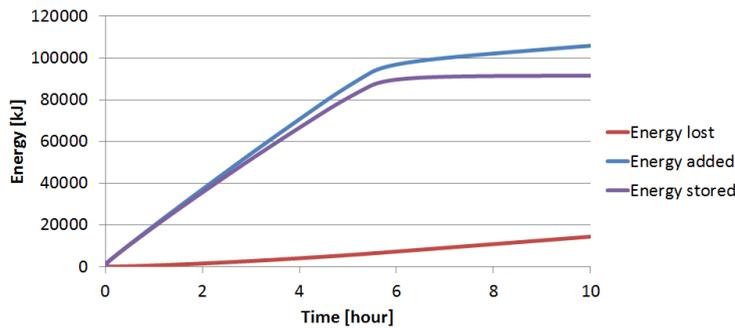


Fig. 5. Energy content of module, heat loss and total energy added

The theoretical thermal energy change in the module for this temperature set and for the material properties in Table 1 is 88,500 kJ calculated from Eq. 5. The measured energy content was 3% higher than the theoretical energy content. The measured energy discharged from the stable hot state to the supercooled state was 58,200 kJ. The theoretical discharged energy calculated using Eq. 6 was 56,300kJ, 3% lower than the measured.

After the module had remained in supercooled state for 3 days, the crystallization was started using the CO<sub>2</sub> container at a storage temperature of 26.4°C. The maximum temperature measured by the probe in the PCM after activation was 53°C. The module was discharged with a flow rate of 2 l/min in each heat exchanger to a temperature of 26.4°C. After 8 hours all surface temperature sensors had stabilized and the module was fully discharged. The total thermal energy discharged 8 hours after activation was 35,700 kJ which corresponds to 179 kJ/kg sodium acetate water mixture calculated from Eq. 11. The theoretical thermal energy discharged for this temperature set calculated from Eq. 9 and values in Table 1 was 31,000 kJ. The measured energy discharged after activation of the supercooled sodium acetate water mixture was 13% higher than the theoretical. Using the specific heat in Table 1 and the measured energy discharged after activation as input in Eq. 9 gives a latent heat of fusion of 213 kJ/kg.

During the test cycle the average ambient temperature was 26.7°C. The discharge powers, after activation for the top and bottom heat exchangers are shown in Fig. 6a. The probe, inlet and outlet temperatures are shown in Fig. 6b.

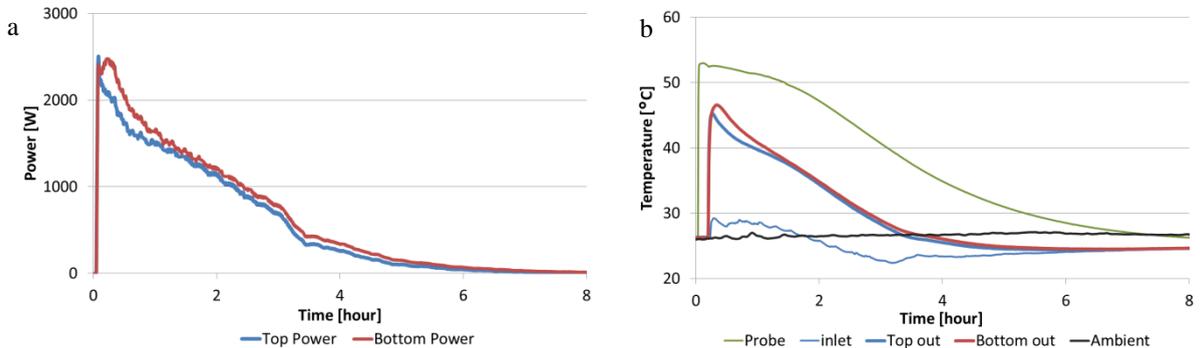


Fig. 6 (a) Discharge powers for top and bottom heat exchangers after activation; (b) probe, ambient, inlet, top and bottom outlet temperatures.

Assuming the module would supercool after the 8 hour charging period, the long term storage efficiency was determined by Eq. 4 and the measurements from the test cycle to be 39%. With the given temperature set and material properties the theoretical long term efficiency was calculated to 35.5%

In the first test cycle where a stable supercooled state was obtained the energy discharged after activation was 194 kJ/kg sodium acetate water mixture, 7.5% higher than the 14<sup>th</sup> test cycle. In one of the test cycles the module remained in supercooled state for 2 months before it was activated and discharged. In 13 of the 20 test cycles the crystallization started uncontrolled most likely due to too low temperatures in the salt during charge. In the later test cycles the module activated spontaneously in three consecutive test cycles after which the expansion bag was dismantled and it was observed that the tube between the module and the expansion vessel was blocked with salt. After cleaning the tube and remounting the expansion vessel stable supercooling was again achieved.

#### 4. Discussion

There were deviations of 3% between the measured and the theoretical energy content for the charge and the discharge to supercooled state. Further there was a deviation of 13% between the measured and calculated energy discharged after activation of the supercooled sodium acetate water mixture. This could be due to measurement uncertainties or the material properties differing from what was assumed. A larger theoretical value for the latent heat of fusion would have given better agreement between measurements and calculations. Also the simplification of the melting behaviour of the sodium acetate water mixture assumed in the theoretical calculations could have led to deviations.

The measured long term efficiency of  $\eta_{\text{long term}} = 39\%$  may be improved. The sodium acetate water mixture may have been able to supercool with a shorter charge period or at a lower temperature leading to a higher efficiency. The minimum charging required to obtain stable supercooling should be determined in future experiments. Reducing the mass of the storage module material could also lead to increased efficiency. A setup with a storage module with lower specific heat or a sodium acetate mixture with a higher latent heat of fusion will lead to a higher efficiency. Further the definition of the long term efficiency can also be debated.

A scaled up module should be tested for stability as the volume size itself could potentially reduce the stability. The optimal size of the storage modules will depend on the system where it is implemented and the heating demand.

#### 5. Conclusions

The experiments showed that it was possible to utilize the principle of stable supercooling for 199.5 kg sodium acetate water mixture to store thermal energy over a 2 month period. The later test cycles showed a decrease in energy released after activation of the supercooled sodium acetate water mixture of 7.5% compared to the first test

cycle most likely due to phase separation. An expansion volume in the PCM chamber was found to be required, to allow for the expansion and contraction of the PCM as it was heated and cooled, in order to reduce deformation of the module. An external expansion vessel without pre-pressure, connected to the air volume in the PCM chamber, decreased the pressure change and deformation of the module as it was heated and cooled and stable supercooling was achieved for some test cycles. Blockage with migrated salt of the tube connecting the expansion volume in the module to the external expansion bag showed to cause unstable supercooling. Activation with liquid CO<sub>2</sub> flushing through a container attached to the outside of the PCM chamber worked as a mechanism for triggering the crystallization by cooling a small part of the sodium acetate water mixture to its maximum degree of supercooling.

The efficiency of this storage module was 39 % when the sensible heat of cooling the module from its hot state to its supercooled state was considered a loss. The long term storage capability and the possibility of utilizing solar energy for heating purposes over long periods without sunshine mean that the potential applications for this storage concept are very promising.

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