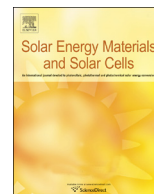




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Solidification behavior and thermal conductivity of bulk sodium acetate trihydrate composites with thickening agents and graphite



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ABSTRACT

Sodium acetate trihydrate is a promising phase change material for long term storage of solar thermal energy if supercooling is actively utilized. Well performing thermal energy storages need to be able to charge and discharge energy at a high rate. The relatively low thermal conductivity of the phase change material limits the heat exchange capacity rate to and from the storage. Another factor that limits the heat transfer is the contraction and expansion of the salt hydrate during the phase change. This density change causes formation of cavities inside the solid storage material. Investigations of the solidification behavior, the formation of cavities and thermal conductivity of composites based on sodium acetate trihydrate crystalizing with or without supercooling are presented in this paper. The thermal conductivity was measured with an ISOMET hot disc surface measurement probe. Samples that crystalized without supercooling tended to form solid crystals near the heat transfer surface and cavities away from the heat transfer surface. The measured thermal conductivity was up to 0.7 W/m K in solid sodium acetate trihydrate. Samples that crystalized from supercooled state formed fewer large cavities but had a lower thermal conductivity. A composite with sodium acetate trihydrate, thickening agent and 5% graphite flakes had a thermal conductivity of up to 1.1 W/m K.

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1. Introduction

The phase change material (PCM) sodium acetate trihydrate (SAT) is a promising material for heat storages. It has a relatively high latent heat of fusion of 264 kJ/kg at a melting point of 58 °C [1]. This melting point makes it suitable for applications for space heating and domestic hot water preparation combined with solar thermal energy [2]. SAT has the ability to supercool in a stable way down to ambient temperature and is therefore a candidate material for long term heat storage. Long term or seasonal heat storage is possible by melting SAT by solar energy and then letting it remain in supercooled state at ambient temperature in the storage period [3]. Once the crystallization of the supercooled SAT is initialized, the latent heat of fusion is released and can be discharged for heating purposes. Dannemand et al. describe a number of barriers and solutions for operating a seasonal heat storage based on stable supercooling of SAT as well as some numerical calculations elucidating the potential [4]. One of the limiting factors in using PCMs in a heat storage is the low thermal conductivity of the PCM itself, which limits the heat exchange capacity rate and discharge power of the storage [5].

The enhancement of the thermal conductivity of PCMs has been investigated by several researchers. One method is to create PCM composites with enhanced thermal properties by mixing additives in the PCM. Another method is to have fixed highly conductive structures or fins inside the PCM storage or to impregnate a highly conductive porous media with the PCM. Li et al. investigated the effect of adding powdered expanded graphite (EG) into SAT and found that the thermal conductivity could be almost doubled by adding 10% EG to the SAT mixture, which also contained Carboxymethyl cellulose (CMC) [6]. Lee et al. investigated composites of EG and erythritol and found that EG as an additive is a highly promising material for improving heat transfer in heat storages with PCMs [7]. Cabeza et al. studied the effect of adding steel pipes or copper pieces as fins in a storage with water as PCM to increase heat transfer, but found that a graphite matrix was better [8]. Sari and Karaipekli showed that the thermal conductivity of a paraffin could be increased significantly by adding a few percentage of EG and this would decrease the melting time of the PCM [9]. Mills et al. also showed that the thermal conductivity of a graphite matrix impregnated with PCM is a viable choice for enhancing thermal conductivity [10]. Also Py et al. showed that paraffin impregnated in compressed expanded natural graphite significantly increased the thermal conductivity compared to the paraffin alone [11]. Zhang and Fang carried out investigations on paraffin and EG composites and likewise found this an effective way of increasing thermal

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conductivity [12]. Fan and Khodadadi did a review on thermal conductivity enhancement of PCMs for thermal energy storage [13]. Jegadheeswaran and Pohekar did a review on several other techniques for enhancing the performance of latent heat storages [5]. Kousksou et al. also did a review listing some important characteristics of energy storage including latent heat storages with salt hydrates [14]. Considering a storage with stable supercooling of SAT, Johansen et al. had SAT samples mixed with graphite powder in stable supercooled condition for five months after it had been heated to 85 °C for 12 h [15]. They concluded that it is possible to have stable supercooled composites of SAT and graphite powder at ambient temperature.

1.1. Phase separation and additives

SAT is an incongruently melting salt hydrate and suffers from phase separation especially over repeated melting and solidification cycles [16]. The problem has been sought to be solved by adding extra water [17] or thickening agents to the SAT. Ramirez et al. studied the use of CMC and silica gel to avoid phase separation of SAT and reported thermal cycling stability in cycles from 30 °C to 72 °C by use of differential scanning calorimetry (DSC) [18]. Hu et al. investigated the reduction of supercooling by the use of Aluminum Nitride nanoparticles in the SAT composite with 4% CMC and found the thickening agent suitable to avoid phase separation and suspend the nucleation agent in the mixture [19]. Ryu et al. investigated several salt hydrates and found that for some salt hydrates a super absorbent polymer (SAP) was an effective thickening agent however CMC was more effective for SAT [20]. Shin et al. investigated the combination of CMC and expanded graphite as additives for SAT and found that composites with 2.5 wt% EG and 5 wt% CMC had a thermal conductivity of 1.85 W/m K [21]. Meisingset and Grønvold investigated thermodynamic properties of salt hydrates and suggested that 0.5–1% xanthan rubber can solve the problem of phase separation in SAT [22]. A suitable thickening agent could both solve phase separation and keep small sized conduction enhancers evenly distributed in the PCM composite at the temperature the storage is meant to operate at and over its lifetime.

1.2. Solidification

The density difference between the solid and liquid SAT is approximately 10% [23]. The resulting volume change of the PCM in a rigid storage tank will cause formation of cavities [24]. These cavities act as thermal resistances, reducing the effective thermal conductivity of the bulk PCM. This was also reported by Choi et al., who investigated heat transfer in storages with SAT [25]. Once a supercooled PCM is nucleated, it crystallizes almost instantly [26]. A PCM that solidifies without supercooling crystallizes at a slower rate as the crystallization front moves with the heat being released and the temperature drops below the melting point. Whether the PCM solidifies without supercooling or with a high degree of supercooling may affect the formation of the solid PCM and the location of the cavities, which affect the thermal conductivity of the bulk PCM. Additives such as extra water and thickening agents added to avoid phase separation may also affect the thermal conductivity of the PCM.

1.3. Applications

In designing and planning thermal systems including heat storages, numerical simulation models are an important tool. To do numerical calculations of PCM storages, an accurate value for the thermal conductivity of the PCM in bulk sizes that resemble the usage in full scale applications is desired. Lele et al. found the

importance of investigating the effective thermal conductivity of the storage material using a measuring setup representative of the considered application [27]. They found that a self-made guarded hot cartridge method gave results more representative for the real storage compared to DSC measurements.

Literature values for the thermal conductivity of solid SAT range from 0.17 W/m K [21] to 0.7 W/m K [28,29]. In previous literature there was no distinction between the thermal conductivity of SAT composites crystallizing from supercooled state or without supercooling.

This article reports the investigations by a simple suspension test on how well different concentrations of the thickening agents CMC and xanthan rubber suspend graphite particles in SAT composites. The second part of this article reports on the solidification behavior and the formation of cavities. Additionally, the thermal conductivity in bulk size SAT composites, which are meant to resemble the conditions in an actual heat storage with or without utilizing supercooling, were investigated. Measurements showed how extra water, thickening agents and graphite powder or graphite flakes affected the thermal conductivity of bulk size PCM composites.

2. Method

Sodium acetate trihydrate was purchased from the company IG Chemicals GmbH. Carboxymethyl cellulose and xanthan rubber was received as samples from the company CP Kelco under the product names Cekol 30000 and Keltrol Advanced performance-F. Fine graphite powder with a particle size less than 50 µm and graphite flakes mesh size 10 (see Fig. 1) were purchased from the company VWR International.

2.1. Suspension of graphite in PCM mixture

To ensure a PCM composite with graphite uniformly distributed in the sample simple suspension tests were carried out. The aim was to investigate the ability of the thickening agents to suspend the graphite in the PCM composite at a temperature of 90 °C which could be the maximum operating temperature of a PCM storage with SAT in a solar combi system. Graphite powder and flakes have a higher density than SAT. The graphite will separate from the PCM and settle to the bottom of the container if no precautions are taken. Mixtures of 200 g SAT with 1%, 2.5% and 5% CMC and SAT with 0.25%, 0.5% and 1% xanthan rubber (all wt%) were prepared without graphite. Then a layer of 40 g graphite powder or graphite flakes mixed with SAT in the ratio 1:10 was placed on top of the thickened PCM mixture.

The samples were heated in an oven to 90 °C for 14 days. During this period the samples inspected to see if the graphite settled to the bottom of the sample or remained suspended on the top of the sample. It was assumed that if the graphite stayed suspended on the top of the sample, the viscosity of the thickened PCM mixture would also be high enough to keep a uniform composite. On the other hand, if the graphite would fall through the thickened PCM mixture during the test period, it would also eventually settle to the bottom of a container in an application intended to function for years. In this case the graphite would not give the desired effect of increasing the thermal conductivity in the entire bulk.

Shaking the samples would also affect the mixing, therefore the sample were carefully handled when being inspected.

2.2. Solidification and thermal conductivity sample preparation

To evaluate the thermal conductivity and the formation of cavities in PCM composites based on SAT in bulk size, a series of 1.3 kg samples were prepared in glass jars with airtight lids (see

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