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Preparation & characterization of sodium sulfide hydrates for application in thermochemical storage systems

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Abstract

Bottlenecks for realizing a commercial system for thermochemical heat storage (TCS) with hygroscopic salts are the chemical, physical and mechanical stability of the salt under operation conditions. Hence, improved knowledge of thermochemical materials (TCMs) is critical to spur progress in TCS system development. Sodium sulfide hydrates (Na₂S.nH₂O, n=0-9) are highly interesting as TCMs because they exhibit a high energy density under operation conditions and are potentially readily available and affordable. Preparation methods for well-defined nonahydrate and pentahydrate crystals of Na₂S were developed and the resulting samples were subjected to cycling experiments under conditions representative for TCS. Before and after cycling, crystal samples were taken and characterized using techniques like SEM/TEM, XRD. Mechanical strength was evaluated using a salt bed stability test. Based on the extensive characterization of sodium sulfide hydrate salts, a method has been proposed to improve the stability of the salt by blending it with cellulose. First trials on these composites yielded promising results with respect to improved material stability.

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1. Introduction

As Europe and the US move more and more towards low and zero-carbon buildings, challenges arise in the management of building-integrated renewable energy technologies. Electricity generating renewable energy sources have the benefit of electrical connection and financial mechanisms allowing feed-in to the network. Thermal technologies do not have such general interconnectivity and therefore rely on sizing typically meeting 90% of thermal demands in the summer in moderate climates. This approach provides little benefit in winter, e.g. for space heating purposes. Wouldn't it be nice to be able to store the summer heat in a loss-free fashion for use in winter time in some sort of "thermal battery"?

Sodium sulfide hydrate salts ($Na_2S.nH_2O$) exhibit a large energy density (2.66 GJ/m³ going from the pentahydrate to the hemihydrate [1]) in the right temperature and pressure regime. Absorption of water vapor can take place at sufficiently high temperatures ($40-60^{\circ}C$) to deliver heat while desorption can occur at relatively low temperatures (<83 °C). This material attracted noticeable interest in the past [2,3], but application suffered from lack of demonstration results due to chemical, physical and mechanical stability issues. For thermal energy storage systems, the forms of instability and related concerns are:

- 1. Chemical instability involves chemical reactions, (a) reaction with oxygen from air (oxidation) forming sodium sulfite salt causing a loss of energy density of the material, (b) reaction with carbon dioxide from air (carbonation) forming sodium carbonate salt and release of hydrogen sulfide gas that is toxic already at low concentration. Reaction of sodium sulfide with metals forming metal sulfides weakens the system construction by corrosion while simultaneously hydrogen gas is released. Accumulation in the vapour space of the thermochemical system of inerts like hydrogen or hydrogen sulfide gas reduces the heat generating capacity of the system.
- 2. Physical instability involves phase transitions of the sodium sulfide hydrate salt that may lead to destabilization of the salt embedded in the system. In the phase diagram of sodium sulfide hydrate salts [3] it can be observed that (1) at a temperature of 49°C a phase transition of the nonahydrate salt to the pentahydrate salt occurs while simultaneously a solution of sodium sulfide forms, (2) at a temperature of 83°C the pentahydrate structure partially dissolves while it forms a solution of sodium sulfide.
- 3. Mechanical instability concerns stability of the bed of the salt hydrate crystals in the thermochemical system. The stability of the bed may decrease during absorption and desorption steps while cycling because of phase transitions of the salt hydrates, for example because the different hydrates have different densities (volume expands and shrinks).

In the p,T diagram of sodium sulfide hydrate salt [2, 3] the different phases of the sodium sulfide hydrate salt during cycling are described. Typically absorption of water vapour (hydration) takes place at a temperature of 40-60°C (depends whether the heat is delivered for heating or for warm water) while the vapour pressure is approximately 12 mbar (corresponding to a winter water temperature in the evaporator of 10°C). Desorption of water vapour (dehydration) typically occurs at a temperature of 80°C to prevent partial dissolution of the sodium sulfide hydrate salt at a vapour pressure of 24 mbar (corresponding to a summer water temperature in the condenser of 20°C).

Preferably, during operation of the thermochemical system only solid phases of the sodium sulfide salt hydrates are present. When the hydration step takes place at 40°C the dry hemihydrate form of the sodium sulfide salt will absorb water vapour while it transforms to the pentahydrate salt and if sufficient water vapour is present is will transform further to the nonahydrate. When the hydration takes place at 60°C the pentahydrate salt will form together with a solution of sodium sulfide. When this material is subsequently cooled below 49°C in the presence of more water vapour also the nonahydrate phase can form. Practically, to deal with these phase changes in previous research a stabilizing material like cellulose fibers was mixed through the sodium sulfide hydrate salt [2].

It is desired to evaluate experimentally the potential chemical, physical and mechanical instability of sodium sulfide hydrate salt for the application as a thermochemical material. To do so preparation, cycling and characterization of the sodium sulfide hydrate salt is required. Hence the aim of this materials study is to evaluate the chemical, physical and mechanical stability of sodium sulfide hydrate salts. This requires preparation of well-defined crystals of sodium sulfide hydrates and determination of the main characteristics and cycling stability. Additionally a composite material of sodium sulfide hydrate salt and cellulose fibers will be prepared and experimentally evaluated.

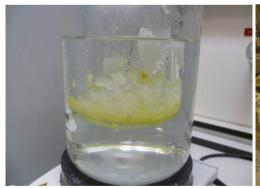
2. Materials and methods

Ideally, all relevant information regarding TCMs is known and can be used "as is", like pT-diagrams and phase diagrams (together defining operational conditions for TCS applications), cycling behavior and stability (defining possible life-time and eventual degradation properties) and cost (defining applicability). However, this kind of information is largely unavailable or insufficiently known for nearly all salt hydrates at this point. Sodium sulfide was selected as base material, mainly because of its high energy density at favorable operating conditions, potential availability and acceptable cost. Measurements include chemical analysis (titration), powder X-ray diffraction (pXRD), scanning electron microscopy (SEM), analysis of melting behavior, and measurements on crystal packed-bed height during cycling.

2.1 Preparation of the crystals

Sodium sulfide nonahydrate had the form of lumps with size of several centimeters that were not suitable for cycling or characterization experiments. By using a standard suspension cooling crystallization method in a glass double walled vessel equipped with a stirrer well defined crystals of sodium sulfide hydrate salts were obtained (fig. 1). The required cooling profile was determined from the solubility lines for the nonahydrate and pentahydrate forms of sodium sulphide as depicted in the phase diagram [3]. By cooling a warm solution of sodium sulfide nonahydrate to room temperature a suspension of nonahydrate crystals was obtained. The suspension was filtered and the filter cake was collected.

To prepare the pentahydrate crystals nonahydrate lumps were first melted/dissolved at elevated temperature and next the hot solution was cooled (fig. 1). Hence a suspension of crystals was obtained and subsequently filtered over a warm filter and next the crystal cake was collected from the filter. It was observed that at elevated temperature the solution turned from clear to yellowish. Furthermore the warm solution remained hazy. Therefore the warm solution was filtered and the particles that caused the haziness were collected for analysis.



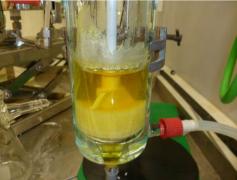


Fig. 1. Recrystallization of Na₂S.5H₂O. Left: melting/dissolving of feed material; right: yellow mother liquor with white crystals

Stabilized sodium sulfide nonahydrate salt samples were originally prepared by using the suspension cooling methods as described above but before cooling was started, cellulose powder was dispersed in the hot solution. A

second (improved) method consisted of first melting/dissolving sodium sulphide nonahydrate lumps at elevated temperature. Next cellulose powder was dispersed in the warm solution by vigorous mixing. This dispersion was cooled to room temperature in a tray without mixing. After solidification the cake made of the composite of cellulose and sodium sulfide nonahydrate salt was collected from the tray.

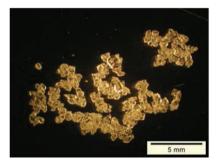
2.2. Cycling test

A test setup was constructed in order to investigate changes occurring on the sodium sulfide hydrate salt samples upon multiple cycling of the sample temperature between dehydration conditions at 80°C and hydration conditions at 40°C. The water vapour pressure was held at 10 mbar using a water bath at 7°C. The sodium sulfide salt hydrate samples were typically cycled 42 times to investigate the chemical, physical and mechanical changes of the material during a simulated life time of 20 years in a thermochemical system for summer-winter operation.

3. Results and discussion

3.1 Characterization of the crystals

Samples of sodium sulfide nonahydrate and pentahydrate salt and of the composite material consisting of cellulose and the nonahydrate form that were prepared as described above were characterized by (1) light microscopy, (2) Scanning Electron Microscopy (SEM),(3) powder-X-Ray Diffraction (pXRD), and (4) visual melting point test. The crystals of both nonahydrate and pentahydrate consisted of free flowing crystals of approximately 1 mm. On light microscopy images it was observed that pentahydrate and nonahydrate samples had well-defined and distinctly different crystal shapes (fig. 2). The structure of the crystals was confirmed by pXRD; the nonahydrate sample consisted for 100% of the nonahydrate form while the pentahydrate sample consisted of 95% pentahydrate and 5% nonahydrate.



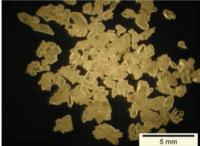


Fig. 2. Microscope images of recrystallized Na₂S.9H₂O (left) and of recrystallized Na₂S.5H₂O (right)

SEM images showed that the surface of the nonahydrate was covered with pores, while the surface of the pentahydrate was covered by a layer of a different material (fig. 3). Based on SEM observations and on the pXRD analysis it was concluded that nonahydrate crystals on the surface of the pentahydrate crystals had formed during cooling down of the wet crystal cake by crystallization of the adhering mother liquor at a temperature below the transition point of 49°C.

Samples of the filter cake obtained by filtering the hazy warm solution of sodium sulfide were subjected to pXRD analysis and turned out to consist of sodium carbonate crystals, probably formed by reaction with carbon dioxide from air during exposure of the sodium sulfide raw material. This is illustrative for the chemical stability of the sodium sulfide hydrate salt that should not be exposed to air but blanketed with nitrogen gas during storage.

Samples of hydrate salt crystals prepared with cellulose by the suspension crystallization method consisted of free flowing white crystals with an average size in the order of ~1mm. However, SEM pictures revealed clearly

recognizable pentahydrate and nonahydrate crystals that were attached to cellulose fibers. This mixed material was considered to be insufficiently homogeneously dispersed at crystal/fiber level.

Solidification of well pre-mixed dispersion of cellulose in a warm solution of dissolved sodium sulfide hydrate salt resulted in lumps of solidified material that could be easily broken and ground to particles of a few mm size. SEM images of this material showed that the cellulose was evenly distributed in between the crystals (fig. 3). This is desirable for stabilization of the salt hydrate, and hence is the latter method is recommended for preparation of cellulose-stabilized sodium sulfide hydrate salt.



Fig. 3. SEM images of (a) recrystallized Na₂S.9H₂O (left) (b) of recrystallized Na₂S.5H₂O (middle) and (c) composite of Na₂S.9H₂O with cellulose, prepared by melt solidification (right) (scale bar = 300 micron)

In previous work with sodium sulfide hydrate salts [1] it was observed that stabilization of the hydrate salt with cellulose resulted in higher melting points compared to pure salt hydrate crystals. To get an indication of the effect of cellulose stabilization on the melting point of the materials, a visual melting point test was performed. This was done by using an instrument consisting of a metal plate that was electrically heated while creating a temperature profile over the length of the plate By sliding two samples next to each other over the plate until melting was visually observed the difference in approximate melting temperature could be noted. This test gave an indication that cellulose-stabilized samples have a higher melting point than the pure salt hydrate samples.

3.2 Results chemical stability (H_2S test and pXRD)

Hydrogen sulfide formation during cycling was investigated by taking water samples from the evaporator in the test setup. The samples were analysed with a HACH LANGE Kuvettentest. The detection limit of this test is 0.1 mg $\rm H_2S/L$. With up to 9 g $\rm Na_2S.nH_2O$ and down to 0.3 L water present in the set-up, this limit would correspond to 0.3% of the sodium sulfide hydrate salt present being converted to hydrogen sulfide gas. However, in water samples taken from the evaporator section of the set-up during the cycling experiments, the presence of hydrogen sulfide dissolved in the water could not be observed above the detection limit. A second analysis of a water sample from the evaporator with gas chromatography, with a comparable detection limit of 0.1 mg $\rm H_2S/L$, confirmed this result. Only in the extreme case of copper (Cu) powder being added to the $\rm Na_2S$ sample before cycling was started, dissolved $\rm H_2S$ with a concentration of 1.4 mg /L was found to be present above the detection limit. Also a black deposit of copper sulfide on top of the sodium sulfide hydrate salt sample could be observed in the cycling set-up.

In some samples, after cycling, a small amount of sodium sulphite (Na₂SO₃) was found in varying quantities using powder X-ray diffraction. This was especially the case for samples that were taken at high temperature from the cycling set-up. During sampling the set-up is opened and air enters the set-up. Oxygen from the air may react with the warm salt sample resulting in the formation of sodium sulphite crystals A preliminary recommendation is to handle samples in an oxygen free environment (nitrogen gas blanketing).

3.3 Results physical stability (visual, SEM and pXRD)

During cycling of the material, some physical processes were observed. The appearance of the sodium sulfide hydrate salts changes during the cycling experiment: initially white crystals become yellowish when the temperature increases. When the temperature decreases the crystals become white again (fig. 4). Moreover, the crystals appearance becomes increasingly opaque at higher temperature and with increasing number of cycles. Finally, when a power failure occurred, it was observed that the sodium sulfide hydrate salt sample kept absorbing water vapour until it was completely molten/dissolved.







Fig. 4. Cycling test vessel (left) and physical appearance of the sodium sulfide hydrate salt at T=40°C white crystals (middle) and at T=80°C yellowish crystals (right)

After cycling of the sodium sulfide hydrate salt, physical changes were observed. SEM images showed that the morphology of the salt hydrate crystals changed from well-defined angular crystals to a highly porous, sponge like shape after multiple cycles (fig. 5). The hydrate salt crystals became more and more spongy with increasing number of cycles. Similar changes were observed for composite samples of cellulose-stabilized salt hydrates. Initially it was assumed that the spongy structure originated from partial melting/dissolution of nonahydrate crystals in the temperature range between 40 and 49°C. However, during cycling between 60 and 80°C instead of between 40 and 80°C the spongy structure was observed as well. In the temperature range between 60 and 80°C the nonahydrate can not exist so it is now assumed that the spongy structure is created by partial melting/dissolution of the pentahydrate salt. It is noted that the occurrence of the porous, spongy structure may be beneficial for water vapour mass transfer during cycling because the surface area of the crystals is significantly increased.

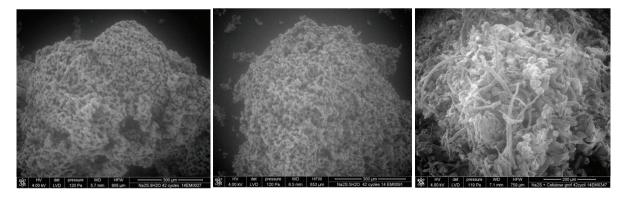


Fig. 5. SEM images of samples after cycling 42 times between T=40 an 80° C: (a) recrystallized $Na_2S.9H_2O$ (left) (b) recrystallized $Na_2S.5H_2O$ (middle) (a and b - scale bar = 300 micron) and (c) composite of $Na_2S.9H_2O$ and cellulose melt solidified (right) (c - scale bar = 200 micron)

To determine the crystal structure and chemical composition of the salt hydrates, powder X-ray diffraction was performed. The effect of cycling on the crystal composition is primarily observed in the hydration state of the

sodium sulfide salt hydrate. Samples of pentahydrate and nonahydrate were taken from the set-up at 40°C after multiple cycling and turned out to consist of similar mixtures of pentahydrate and nonahydrate salt forms. The samples taken from the cycling set-up at 80°C consisted partially of sodium sulfide anhydrate and dihydrate while also a considerable fraction of pentahydrate (36-50%) was observed. This is an indication that conversion of the salt hydrates was not complete during cycling. The results of the composition analysis by pXRD are summarized in table 1.

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Sample	Na ₂ S.9H ₂ O	Na ₂ S.5H ₂ O	Na ₂ S.2H ₂ O	Na ₂ S.0H ₂ O
Na ₂ S.9H ₂ O before cycling	100			
$Na_2S.9H_2O 42$ cycles; $T_{max} = 40C$	25	71		
Na ₂ S.9H ₂ O 42 cycles T 80 °C	3	36	~10	42
Na ₂ S.5H ₂ O before cycling	5	100		
Na ₂ S.5H ₂ O 42cycles; T ₁₁ 40 °C,	11	89		-
Na ₂ S.5H ₂ O 42 cycles T 80 °C	4	50	~10	22
Na ₂ S.9H ₂ O + cellulose before cycling	100			
Na ₂ S.9H ₂ O + cellulose 42 cycles	6	94		
Na ₂ S.5H ₂ O +cellulose before cycling	8	92		
Na ₂ S.5H ₂ O + cellulose 42 cycles	38	62		

3.4 Results mechanical stability (volumetric expansion test, bed strength test)

During cycling of the thermochemical storage system, the pentahydrate salt converts to the hemihydrate salt (actually a mixture of anhydrate and dihydrate forms) and this phenomenon may be accompanied by a change in volume of the material. Due to the confinement of the sodium sulfide hydrate salt in the heat exchanger a significant volumetric increase or decrease may have a negative effect on contact between the salt hydrate crystals and heat exchanger fins. Also the resulting stress on the crystals resulting in brittleness and powder forming.

Therefore, a volumetric expansion test was performed, in which the volumetric increase and decrease was studied by measuring the height of a bed of material during cycling. The bed height was seen to decrease and increase during every cycle (fig. 6). The height deviation is around 10-15%. A volumetric decrease was expected to be seen during this dehydration at 80°C. However, the observed volumetric decrease already occurred during the 40°C stage, contrary to expectations. A possible explanation for the volumetric increase and decrease during the 40°C stage, is partial melting/dissolution of both nonahydrate and/or pentahydrate crystals.

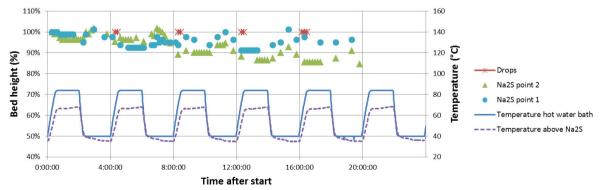


Fig. 6. Salt hydrate bed height, relative to starting height (left axis), occurrence of drops, and salt hydrate bed temperature (right axis) versus time.

Besides these volumetric effects, small droplets were observed on the glass dish with the sodium sulfide during 10-25 minutes, coinciding with the temperature increase. These droplets are expected to be water released from the sodium sulfide hydrate salt crystals during dehydration.

A mechanical bed strength test was constructed based on putting a weight on top of a salt crystal bed in a glass test tube and putting these in an oven. The change in bed height was observed while the temperature of the oven was stepwise and gradually increased. It was observed that the salt bed height decreased as a function of increasing temperature for both nonahydrate and pentahydrate salt samples with and without cellulose stabilizer added (fig. 7).





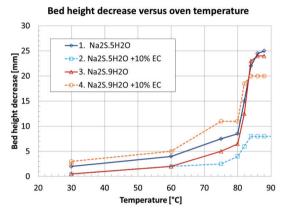


Fig. 7. Salt bed partial melting and weight partially submerged in liquid (left). Samples in oven with only sample Na₂S.5H₂O + cellulose still solid and present as a packed bed while showing marked coloration (middle). Change in bed height [mm] for the samples as a function of oven temperature [°C] (right)..

For the nonahydrate crystals no significant difference in liquefying temperature was observed between the salt samples with and without cellulose stabilizer, both melted in the range 70-82°C at a temperature above that for recrystallization/melting temperature of the nonahydrate (T=49°C). The pentahydrate salt crystals with cellulose stabilizer appeared to have a higher melting temperature than the crystals without stabilizer that started to melt at 82°C. This bed test confirmed a melting bank test with samples of pentahydrate with and without cellulose stabilizer where the pentahydrate salt with stabilizer was observed to remain solid until a higher temperature than the pentahydrate salt without stabilizer.

4. Conclusions

Both for the nonahydrate and the pentahydrate structure of sodium sulfide hydrate salts a reproducible crystallization procedure was developed. Subsequently, samples were subjected to cycling in a dedicated set-up. Characterization of the chemical, physical and mechanical stability of Na₂S hydrate crystals before and after cycling resulted in a highly valuable set of novel data on this potentially interesting TCM. Based on the stability measurements, guidelines are proposed for handling the salts during manufacturing and operation of the TCS reactor. Furthermore, a method to stabilize the sodium sulfide hydrates with cellulose as a stabilizer material has been proposed. Initial measurements on these composite materials show promising behavior for application in TCS reactors.

4.1. Preparation, cycling, characterization

- A suspension cooling crystallization method was developed to prepare well defined crystals of pentahydrate and nonahydrate forms of sodium sulfide.
- A melting / solidifying method was developed to prepare a composite material of sodium sulfide hydrate salt crystals and cellulose fibers (stabilizer).

- Samples of pure crystals and cellulose stabilized crystals were exposed to temperature cycling at reduced pressure to mimick operation in a thermochemical system.
- Samples of crystals and composite before and after cycling were characterized by microscopic imaging, SEM and pXRD.
- Cellulose stabilized hydrate salts were observed to melt at higher temperature level than the corresponding pure salt hydrate crystals.

4.2. Chemical, physical, mechanical stability

- Sodium sulfide hydrate salt was observed to be chemically reactive, reaction products with carbon dioxide and oxygen from air were observed.
- During cycling no detectable quantity of released hydrogen sulfide gas could be detected. Only when reactive copper metal powder was mixed with the salt a detectable quantity of hydrogen sulfide dissolved in the evaporator water was found.
- With pXRD the phase changes of the salt hydrate forms occurring during cycling could be observed. After multiple cycling samples taken at 40°C consisted of mixtures of pentahydrate and nonahydrate forms. Samples taken at 80°C consisted mostly of anhydrate and dihydrate with a considerable fraction of pentahydrate (36-50%). Partial dehydration of the salt bed will have a negative effect on the capacity of a heat storage system
- SEM images showed that during cycling the morphology of the salt hydrate crystals changed from well-defined angular crystals to a highly porous, sponge like shape after multiple cycles. This change in morphology is attributed to the crystals going through a stage where both the pentahydrate and a solution of sodium sulfide are present. Crystals stabilized with cellulose showed the same behaviour, it is assumed that the cellulose fibers present may improve mechanical stability of the salt bed during this stage.
- A change in volume up to 10-15% of the salt bed height was observed during cycling. When cooling the
 bed first an increase in bed height is observed followed by a decrease while upon heating a further decrease
 in bed height is observed.
- In a mechanical bed strength test it was observed that the salt bed height decreased with increasing temperature for both nonahydrate and pentahydrate salt samples, both with and without cellulose stabilizer. The pentahydrate salt crystals with cellulose stabilizer added appeared to resist melting up to a temperature higher than that of the crystals without stabilizer that melted between 70 and 82°C.

4.3. Recommendations

- a. To prevent chemical reactions of the sodium sulfide hydrate salt with oxygen or carbon dioxide from air exposure to air should be prevented, for example by blanketing the material during preparation and storage with nitrogen gas. Furthermore, direct contact between the sodium sulfide hydrate salt and metal surfaces should be prevented.
- b. For a high capacity of the TCS the (de)hydration of the salt hydrate crystals during cycling should be as complete as possible. In the current experiments a dehydration level in the order 50-65% was observed. It is recommended to increase this rate further.
- c. Stabilization of the sodium sulfide hydrate salt with cellulose is beneficial because it improves the mechanical strength of the composite compared to that of pure hydrate salt crystals. Stabilization with cellulose allows dealing with material phase changes like partial dissolution of salt crystals above 50°C during cycling. It is recommended to test further whether it also allows to cope with melting of pentahydrate above 83°C. The use of a more accurate test method (DSC) is recommended.
- d. The porous sponge like structure of the hydrate salt crystals that is created during cycling of the material may promote mass transfer of water vapour during the hydration and dehydration stages. It is recommended to make an estimate of this effect.

e. The phase changes of the sodium sulfide hydrate salt during cycling should be followed using more advanced measurement techniques like TGA/DSC en pXRD equipped with stages for which the water vapour pressure can be adjusted.

Acknowledgements

The research leading to these results has received funding from the European Commission Seventh Framework Programme (FP/2007-2013) under grant agreement No ENER/FP7/295983 (MERITS). MERITS is a R&D project supported by the European FP7 program with the aim to build a prototype of a compact rechargeable thermal battery. Such a product would offer a new solution for improved use of renewable sources for domestic heating, cooling and hot water appliances and thus greatly contribute to the European ambition of an energy-neutral built environment by 2050.

The project is carried out by four research institutes (TNO, VITO, Tecnalia, Fraunhofer ISE), two universities (Ulster University, University of Lleida), two SME's (De Beijer RTB, Zonne-Energie Nederland BV), and two industries (Mostostal, Glen Dimplex). The team will work with novel high energy density thermochemical materials that can supply required heating, cooling and domestic hot water for a dwelling with up to 100% renewable energy sources (e.g. the sun) throughout the year. The key development issues are:

- The delivery of heat on different dedicated temperature levels for heating, cooling and domestic hot water
- The tailoring to the requirements of individual dwellings
- The design and development of a dedicated solar collector
- The integrated design for the components and enhanced thermo-chemical materials, including the control system

Furthermore the project includes the development of business models and market strategies to foster market takeup before 2020. More information on MERITS is available on our public website "www.MERITS.eu".

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