

Task 58 / Annex 33

Subtask 3P

Deliverable 1

**Properties of Phase Change Materials
(PCM) in the Lab Environment and under
Application Conditions**

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10 March 2020

DOI: [10.18777/ieashc-task58-2021-0002](https://doi.org/10.18777/ieashc-task58-2021-0002)

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1 Contributors

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2 Introduction

Deliverable 1 of Subtask 3P is an inventory of properties of Phase Change Materials (PCM) that change comparing experiments in the lab environment with tests under application conditions. Examples where no change is observed are also included.

The inventory is reported in the following sections containing a short description of the property and a table summarizing the relevant information. The considered properties are: degree of supercooling (section 3.1), phase separation (3.2), storage capacity (3.3), long-term stability (3.4), crystal structure (3.5), stability of phase change emulsions (3.6), and thermal conductivity (3.7).

The structure of the tables is as follows: The first column indicates the observation from the comparison between experiments in the lab environment with tests under application conditions. Columns 2 and 3 specify for which experimental conditions and which PCM (class) the changes of properties have been observed. Column 4 contains corresponding references and column 5 comments.

In column 4, either publications or the institutions providing input are indicated. The following abbreviations, listed in alphabetical order, were used for these institutions.

DLR:	German Aerospace Center, Germany
DTU:	Technical University of Denmark, Denmark
HSLU:	Institute of Mechanical Engineering and Energy Technology IME / CC Thermal Energy Storage, Lucerne University of Applied Sciences and Arts, Switzerland
ISE:	Fraunhofer Institute for Solar Energy Systems ISE, Germany
LAMTE:	Laboratory of Applied Multiphase Thermal Engineering, Dalhousie University, Canada
LTTT:	Lehrstuhl für Technische Thermodynamik und Transportprozesse, University of Bayreuth, Germany
TUE:	Technical University of Eindhoven, The Netherlands
UPV/EHU:	University of the Basque Country, Spain
ZAE:	Bavarian Center for Applied Energy Research (ZAE Bayern), Germany

3 Inventory tables

3.1 Degree of supercooling

As stated in the PCM Wiki [1], “Supercooling during the liquid-solid phase change is the phenomenon when a material’s crystallization initiation occurs at a temperature below its freezing temperature. That is, the nucleation starts at a temperature below the real freezing point of the material. Thus, a material that tends to supercool should be cooled considerably below its expected freezing point to initiate freezing. Once its nucleation initiates, the material temperature rises to its real freezing point, and then continues freezing at that temperature.”

The degree of supercooling depends on the applied measurement conditions: Sample size, purity, cooling conditions, level of heating above the melting point, design of PCM container and heat exchanger, and additives. Besides the experimental conditions, the degree of supercooling also depends on the material class under investigation. Usually, salt hydrates, water, and sugar alcohols exhibit a higher degree of supercooling compared with organic PCM like paraffins, fatty acids, and fatty alcohols. [2][3]

Table 1 contains the observations related to the degree of supercooling when comparing experiments in the lab environment with tests under application conditions.

Table 1: Degree of supercooling

#	OBSERVATION	EXPERIMENTAL CONDITIONS	MATERIAL (CLASS)	REF.	COMMENTS
1.1	Nucleation temperature: DSC: -11 °C 10 L: 4-8 °C 500 L: 5.1-7.8 °C	DSC device 10 L container 500 L storage	TBAB40 + water	HSLU	500 L storage: sometimes different degree of supercooling in different sensors
1.2	Nucleation temperature: DSC: --10 °C, T-History and storage: 25 – 27 °C	DSC, T-History, 1 and 1.5 m ³ storage	CaCl ₂ ·6H ₂ O	ZAE [4]	Varied test conditions: purity, sample size, cooling rate
1.3	Degree of supercooling: DSC: 32 K 12 L: 6 – 18 K	DSC device 12 L container	Erythritol	ISE	Supercooling depends on previous temperature level during melting process
1.4	Freezing point: 20 L setup: 49 °C “Easymax”: -10 °C	20 L container; “Easymax 102” cycling device and synthesis station	35% Alum amon hydrate slurries	HSLU	
1.5	Heat sodium acetate trihydrate (SAT) to temperatures +20 K above its melting point resulted in supercooling of 40 K	closed steel tanks, prototype heat storage units with SAT mixtures	SAT with additives, extra water, thickening agents	DTU [5] [6] [7] [8]	In a closed PCM tank, the expansion of SAT has to be considered, local pressure changes and tank

					deformations may cause nucleation starting at crack in a similar way as the metal disc in the hand warmers.
1.6	Minimum crystallization temperatures of supercooled SAT composites: * SAT and SAT +9% water: - 24 °C; * SAT +5% CMC and SAT +3% liquid polymer -18 °C; * All SAT composites in contact with rusty steel: -15 °C	Supercooled SAT composite samples (60 g) were cooled down in a freezer	SAT with additives, extra water, thickening agents, liquid polymers	DTU [9]	Varied test conditions: purity, supercooling duration.
1.7	DSC: - Erythritol: 60 K - Xylitol: > 90 K - MgCl ₂ ·6H ₂ O: 30 K 3-Layer-Calorimeter: - Erythritol: 47 K - Xylitol: > 90 K - MgCl ₂ ·6H ₂ O: < 3 K Macro-capsule: - MgCl ₂ ·6H ₂ O: < 3 K	3 different setups: DSC (sample size 10 mg), 3-Layer-Calorimeter (100 g), macro-capsules (281 g)	Technical grade sugar alcohols (Erythritol, Xylitol) and salt hydrate (MgCl ₂ ·6H ₂ O)	LTTT [10] [11]	Degree of supercooling depends on applied sample volume. The sample mass of 100 g in the 3-Layer-Calorimeter is in the magnitude of PCM mass of the later on applied macro-capsules.
1.8	Highly increased supercooling in small volumes	Micro/nano scale	All material classes, also paraffins	ISE	
1.9	Sample size, maximum holding temperature and holding time affect significantly the nucleation process of Glauber´s salt nucleated with borax. Glauber´s salt nucleated with borax: DSC measurements will render a value of the supercooling reduction well below the actual values for large samples [20].	DSC (10 mg) vs 25 ml flasks Several cooling rates and maximum holding temperatures	Glauber´s salt (Na ₂ SO ₄ ·10H ₂ O) with additives (borax)	UPV/EHU [12]	Within the ranges of the work referred, the cooling rate seems not to play an important role.
1.10	No supercooling at large scale, slight	DSC to multi liter scale	Dodecanoic acid	LAMTE [13]	At large scale, no supercooling

supercooling (1-2 K) in
DSC

[14]

[15]

(maximum
temperature of the
PCM during testing
never exceeds 80
°C, less than 40 K
above melting
temperature)
In [13] and [14]:
solidification plateau
at the expected
melting temperature.

3.2 Phase separation

In the case of PCM consisting of more than one component, a separation of different phases might occur during repeated melting and crystallization cycles. If phase separation occurs, a part of the material does not contribute to the melting and crystallization enthalpy any longer, thereby usually reducing the storage capacity of a latent heat storage system. [16]

Phase separation depends on the applied test measurement conditions, such as sample size, purity, thermal cycling conditions, heat exchanger design, additives, and container dimensions.

Table 2 contains observations related to phase separation when comparing experiments in the lab environment with tests under application conditions.

Table 2: Phase separation

#	OBSERVATION	EXPERIMENTAL CONDITIONS	MATERIAL (CLASS)	REF.	COMMENTS
2.1	100 ml no separation, 10 L separation	Varying sample container volumes	Salt hydrates or Mixtures of materials with significantly different densities	TUE, ZAE	More related to height of storage rather than volume
2.4	Containments with a large height are worse	Encapsulated PCM	Different material classes	ISE	Example: FlateICE manufacturer recommends to lay the containers
2.2	No difference in separation observed for different sample sizes (during the phase change trough the melting range between solid and liquid state)	Sample sizes from 20 mg to 156 kg	Non eutectic salt mixture of 30 wt.% KNO ₃ and 70 wt.% NaNO ₃	DLR [17]	
2.3	In PCM storage with SAT and extra water phase separation occurs over time/repeated cycles when the PCM is not actively mixed. Thickening agents can effectively reduce phase separation also in tall storages. There is also high potential for using various polymers for reduction of phase separation.	200 g to 200 kg	SAT with additives; extra water, thickening agents (CMC or Xanthan rubber), EDTA, liquid polymers	DTU [6] [18] [19] [20] [21]	Results from prototype storage unit testing have been compared to experiments with smaller sample sizes. Also EDTA and similar additives with chelating effect can be used for avoiding phase separation.

3.3 Storage capacity

“In applications, the maximum storage capacity (MSC) of a PCM is a crucial value. It corresponds to the enthalpy difference between minimum and maximum operating temperature and thus, besides the latent heat in the temperature interval under consideration also contains sensible heat.” [22]

The storage capacity (SC) observed in experiments under application-oriented conditions depends on, amongst others, the sample purity and the cooling conditions. In the case of PCM mixtures, a possible deviation from the correct concentration also affects the storage capacity.

Table 3 contains observations related to storage capacity when comparing experiments in the lab environment with tests under application conditions.

Table 3: Storage capacity (SC)

#	OBSERVATION	EXPERIMENTAL CONDITIONS	MATERIAL (CLASS)	REF.	COMMENTS
3.1	Decreased SC due to a deviation from the correct concentration	DSC; 1 to 1.5 m ³ storage	Salt hydrates	ZAE [22]	“In large latent heat storages with the used PCM being a salt hydrate, it is difficult to assure the stoichiometrically correct hydrate concentration of the salt hydrate and to verify the concentration.” [22]
3.2	Change in SC due to impurities	DSC; 1 to 1.5 m ³ storage		ZAE	Impurities can act as “inert” material and thus decrease SC. Impurities can affect crystallinity.
3.3	SC (melting enthalpy) of technical grade PCM agrees with results from analytical grade samples.		Technical grade sugar alcohols (Erythritol, Xylitol) and salt hydrate (MgCl ₂ ·6H ₂ O)	LTTT [10]	Comparison of technical grade PCM with values from literature
3.4	SC of polymers is affected by cooling conditions	DSC with varying cooling rate and different minimum temperatures reached upon cooling	Polymers	ZAE	Crystallinity of polymers depends on cooling conditions → different phase change temperature ranges due to different phase fraction of amorphous and crystalline (or different crystals) solid material
3.5	Polymorphism affects SC as different melting points and enthalpies can be obtained in succeeding thermal cycles	DSC	Sugar alcohol (Erythritol)	LTTT [10]	Repeated melting and crystallisation cycles with same heating and cooling rate

3.6	Reduction of melting enthalpy due to decreased purity of fatty acids		Fatty acids: Stearic Acid (SA) and Palmitic Acid (PA)	UPV/EHU [23]	“Industrial grade SA and PA rendered melting enthalpies 24 and 20% lower than the ones for the analytical purity compounds. A noticeable decrease was also noticed for the melting temperatures, being also the melting interval for the industrial compounds significantly wider than the analytical one. “[23]
3.7	Maximum storage capacity, i.e. enthalpy difference in temperature range of 15 K (25-40 °C): Pure: 164 J/g Technical: 134 J/g	DSC, 2 K/min heating and cooling rate	CaBr ₂ ·6H ₂ O Pure: crystals grown from solution Technical: prepared from aqueous CaBr ₂ solution	ZAE [24]	The purity of the used basic raw material to prepare the salt hydrate has an influence on the maximum storage capacity.
3.8	No difference in storage capacity	DSC measurements and tests at experimental scale	Dodecanoic acid	LAMTE [14][25][26]	For the work that was done with dodecanoic acid, the authors have not seen any difference between thermal storage as determined from DSC data, and then measured through the charging or discharging at the experimental scale. Papers [14][25][26] show the theoretical prediction (from measured DSC properties) matching the experimental prediction within the error measurements

The storage capacity is also affected by changes of the phase change temperature range.

The phase change temperature range is the temperature range in which the phase change occurs. In the case of an ideal PCM – being described by the model of a perfect single crystal of infinite expansion – a single melting temperature (or melting point) is obtained: Since all atoms are fixed at their lattice locations and thus have the same binding energy, all atoms undergo a phase transition at the same temperature. However, most PCM show a widened phase transition range, since the crystal has a finite expansion and slightly different binding energies can be assigned to the individual crystal components (ions, molecules, etc.) due to impurities, crystal defects, grain boundaries, and surface effects. The real crystal then has a melting temperature range, which extends to lower temperatures compared to the perfect, infinitely extended crystal. The upper limit of the melting range of the real crystal corresponds to the melting point of the ideal crystal.

During experiments, the measured phase change temperature range can be broadened depending on the experimental conditions, such as purity of the sample and applied heating / cooling rates. For example, a decreased purity of the sample lowers the melting temperature. Increased heating and cooling rates lead to an increased temperature gradient within the sample and, thus, to an apparently broadened phase change temperature range.

3.4 Long-term stability

In this report, stability refers to the stability of the PCM itself subjected to different static and/or dynamic conditions (temperature, contact to atmosphere, and contact to container/heat exchanger). Stating that a PCM is stable means that the relevant properties of the PCM remain constant within specific limits (which are to be identified) over the desired period of time.

Long-term stability depends on the applied test and measurement conditions, such as the interaction with the environment or the storage component materials, the stability of additives, and the temperature the PCM is subjected to. In the case of organic PCM, the thermal stability and stability in contact with oxygen is crucial. In the case of salt hydrates and PCM mixtures, long-term stability is affected by a decreased thermal cycling stability due to phase separation processes.

Table 4 contains observations related to long-term stability when comparing experiments in the lab environment with tests under application conditions.

Table 4: Long-term stability

#	OBSERVATION	EXPERIMENTAL CONDITIONS	MATERIAL (CLASS)	REF.	COMMENTS
4.1	NaNO ₃ thermally stable at 350 °C	Isothermal long-duration test, batch furnace, mass losses at specific times with an analytical balance $T_{\max} = 350 \text{ °C}$, $T = 2600 \text{ h} = 108 \text{ d}$	NaNO ₃ , ($T_m = 305 \text{ °C}$), from BASF, purity of min. 99%	DLR [27]	small amount of nitrite formed (equilibrium reaction)
4.2	Phase change temperature detected at the same temperature level as in laboratory experiments ($T_m = 305 \text{ °C}$)	Long-term test in a lab-scale module $T_{\max} = \text{above } 350 \text{ °C}$, typical cycle temperature: 280 – 330 °C, 200 cycles	NaNO ₃ ($T_m = 305 \text{ °C}$) Amount of PCM: 320 kg	DLR [28]	
4.3	Thickening agents such as CMC and Xanthan rubber may degrade over time at high temperatures.		SAT with thickening agents	DTU [29]	The colour of a SAT mixture with CMC or Xanthan will change when exposed to high temperatures over a period. The thickening effect may or may not remain.
4.4	No change in onset melting temperature or latent heat of fusion over 3000 to 4000 cycles	Setup built and used to run thermal cycling is presented in [30]	Paraffins and other organic PCM	LAMTE [26] [30] [31]	Study on a dozen PCM (half paraffin, half other organics): no change in melting temperature onset or latent heat of fusion over 3000 to 4000 cycles

4.5	500 cycles in DSC between 55 and 155 °C: → 1 % decrease of melting enthalpy (below the accuracy of the DSC device) → Small change of melting temperature (0,3 K) 30 cycles with macro-capsules (between 95 and 135 °C): → No decrease of the stored energy observed.	DSC with sealed crucibles; macro-capsules in lab-scale storage unit	Salt hydrate (MgCl ₂ ·6H ₂ O)	LTTT [10] [11]
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3.5 Crystal structure

“The crystal structure itself is obtained by associating with each lattice point an identical assembly of asymmetric units. The asymmetric units might be as small as an atom of neon or as large as a virus.” [32] In the context of solid-liquid PCM, a change in the crystal structure of a PCM can affect many properties, such as the melting temperature and enthalpy of the PCM.

Especially in the case of organic materials, the crystal structure that is obtained during experiments depends on the applied test conditions. The cooling program and the sample size under investigation are two test conditions influencing the crystal structure.

Table 5 contains observations related to crystal structure when comparing experiments in the lab environment with tests under application conditions.

Table 5: Crystal structure

#	OBSERVATION	EXPERIMENTAL CONDITIONS	MATERIAL (CLASS)	REF.	COMMENTS
5.1	Formation of different polymorphic crystal structures depending on the applied cooling conditions	DSC and storage tank	Sugar alcohols, e.g. D-mannitol	[33]	“The experiments performed by DSC have shown that the D-mannitol presents polymorphic structural changes and, therefore, its thermal properties are not always the same. Depending on the polymorphic phase obtained, D-mannitol has different melting temperature. This behaviour was corroborated in a storage tank, where it may be seen that the cooling rate of the D-mannitol is a key parameter in the formation of the different polymorphic phases.” [33]
5.2	Formation of different crystals		TBAB	HSLU	It is not sure if this is affected by the storage volume

5.3	Formation of different polymorphic crystal structures depending on the applied cooling conditions		Sugar alcohols, e.g. erythritol, xylitol, sorbitol and their eutectic mixtures	EHU [34]	“Under the cooling conditions employed for the preparation of the samples (quench cooling down to room temperature), erythritol crystallized forming the metastable polymorph: form II. When the samples were subsequently heated from room temperature, form II transformed to form I. Xylitol revealed no formation signs of the metastable structure” [35]
5.4	“During a DSC thermal cycling of the eutectic mixture, the metastable Form II of erythritol crystallized 6 times out of 85 cycles. On those cycles, the mixture crystallized at 71 °C instead of 82 °C. Besides, the enthalpy of the mixture displayed a value of 190–195 J/g instead of the shown value on the remaining cycles: 240 J/g.” [36]	DSC	Organic eutectic mixtures (erythritol and urea)	EHU [36]	Note that these two observations also influence the storage capacity and phase change temperature range.

3.6 Stability of phase change emulsions

Phase change emulsions are fluids containing PCM and a carrier fluid. A phase change emulsion is stable if the emulsified PCM droplets do not agglomerate. Among others, the stability of a phase change emulsion depends on the investigated sample and particle size.

Table 6 contains observations related to stability of phase change emulsions when comparing experiments in the lab environment with tests under application conditions.

Table 6: Stability of phase change emulsions

#	OBSERVATION	MATERIAL (CLASS)	REF.
6.1	Solidification of some PC dispersions has been observed in small scale (50-100 ml), but not in large scale (10 kg)	Paraffin-in-water emulsions	HSLU
6.2	Separation and shear stability depend on particle sizes: <1 µm: stable, >1 µm unstable	Paraffin-in-water emulsions	ISE

3.7 Thermal conductivity

Thermal conductivity is a material property that determines the heat flow through a material on the basis of thermal conduction. In the PCM context, the thermal conductivity of the PCM affects the thermal power during charging and discharging of the latent heat storage. The

measured thermal conductivity depends on the applied test conditions, such as purity of the sample, heating and cooling rates, and the existence of voids in the solid sample.

Table 7 contains observations related to thermal conductivity when comparing experiments in the lab environment with tests under application conditions.

Table 7: Thermal conductivity

#	OBSERVATION	MATERIAL (CLASS)	REF.	COMMENTS
7.1	Voids within the sample can reduce the measured thermal conductivity	All material classes	ZAE	Internal voids in the solid phase or gas bubbles in the liquid phase
7.2	Different cooling rates can lead to different crystal structures with different thermal conductivities.	All material classes	ZAE	High cooling rates in small lab scale samples, but low cooling rates in application size.

4 Acknowledgements

This deliverable was prepared within the framework of IEA SHC Task 58 / ECES Annex 33.

The work of ZAE Bayern was part of the project properPCM and was supported by the German Federal Ministry of Economic Affairs and Energy under the project code 03ET1342A.

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