DTU Library

IEA SHC Task 58 / Annex 33 Subtask 2P
On development and characterization of improved Materials

Gschwander, Stefan; Lazaro, Ana; Delgado, Monica; Rathgeber, Christoph; Brütting, Michael ; Höhlein, Stephan; Obermeyer, Melissa ; Groulx, Dominic; Haussmann, Thomas; Lager, Daniel

Total number of authors:
17

Task 58 / Annex 33 Subtask 2P Summary of Work

Subtask 2 PCM: On development and characterization of improved Materials

Task 58 / Annex 33 Subtask 2P Summary of Work

Subtask 2 PCM: On development and characterization of improved Materials

D2P1: List of novel developed PCMs as well as blends and mixtures

D2P2: Extended list of material properties for the characterization of novel PCM

D2P3: Measured material data for the maintenance and expansion of the PCM Database

Contents

1 Contributors

Stefan Gschwander, Fraunhofer ISE

Ana Lazaro, University of Zaragoza

Monica Delgado, University of Zaragoza,

Christoph Rathgeber, (ZAE-Bayern)

Michael Brütting, (ZAE Bayern)

Stephan Höhlein, University of Bayreuth

Melissa Obermeyer, Hochschule Luzern

Dominic Groulx, Dalhousie University

Thomas Haussmann, Fraunhofer ISE

Daniel Lager, Austrian Institute of Technolgy

Saman Nimali Gunasekara, KTH Royal Institute of Technology

Mohammed Farid, University of Auckland

Rocio Bayón, CIEMAT-PSA

Gonzal Diarce, University of the Basque Country (UPV/EHU)

Juan de Dios Cruz Elvira, Instituto Tecnologico de Oaxaca

Gerald Englmair, Technical University of Denmark

Thomas Aigenbauer, FH OÖ Forschungs & Entwicklungs GmbH

2 Introduction

The work of Subtask 2P is split into 4 topic:

- Material development
- Developing measurement procedures
- Filling the PCM Database
- Developing a Wiki for terms used in the context of PCMs

As the material development is done at different institution the objective of the work was to collect the materials which are under research and development to get an overview on the most relevant properties of these materials and application which are addressed.

In the frame of this subtask investigations on results obtained at different institutions using various measurement methods for different material properties have been conducted. Procedures have been developed, for thermal conductivity and the determination of viscosity.

New material properties have been feed into the database which was developed during previous tasks and annexes.

And finally a wiki for the terms uses in the context of PCMs have been developed based on a content management web based tool.

3 Material development; List of novel developed PCMs as well as blends and mixtures (D2P1)

The objective of this work was to collect the PCMs and applications focused on within the participants of the SHC Task 58 and ECES Annex 33 (TA5833). Another target is to determine the challenges of the development process. For the collection of this data a questionnaire was prepared which was send to the TA5833 mailing list. Nine different institutions reported about the development or investigation of 20 different materials and the supposed applications (Annex from page 16).

PCMs

- Organic (alkanes, fatty acids, sugar alcohols and others)
- Inorganic (salt hydrates, salt)
- Eutectic mixtures (organic, inorganic)

Applications

- Cold storages, precooling of refrigerants
- Passive cooling for buildings and industrial processes
- Heat pump applications
- Mobile heat storage
- Waste heat / process heat / steam generation
- Thermal protection of electronics / battery cooling
- Solar heating / long term storage

The main work is done on organic PCMs and on mixtures of either organic or inorganic materials. Figure 1 is indicating the materials which are under research. The majority of materials are developed for storage temperature below 100 °C. A few materials are under research in the temperature range between 100 und 150 °C. Above 150 °C many institutions doing research on d-Mannitol, which is only once rated as stable as well as a mixture of d-Mannitol and Ducitol. Another material which is under research on this temperature level is hydroquinone, which is also rated as not stable.

Supercooling of almost all materials is below 10 K except d-Mannitol, pinacone hexahydrate, and sodium acetate trihydrate. The last one was optimized to be used as supercooled storage material.

Almost all contributors tested the stability of materials by thermal cycling except one who kept the temperature above the melting point. The number of cycle has been very different and reaches from 5 up to 5000 cycles. There is no clear indication for stability e.g. based on a limit for the decrease of enthalpy or change in phase transition temperature.
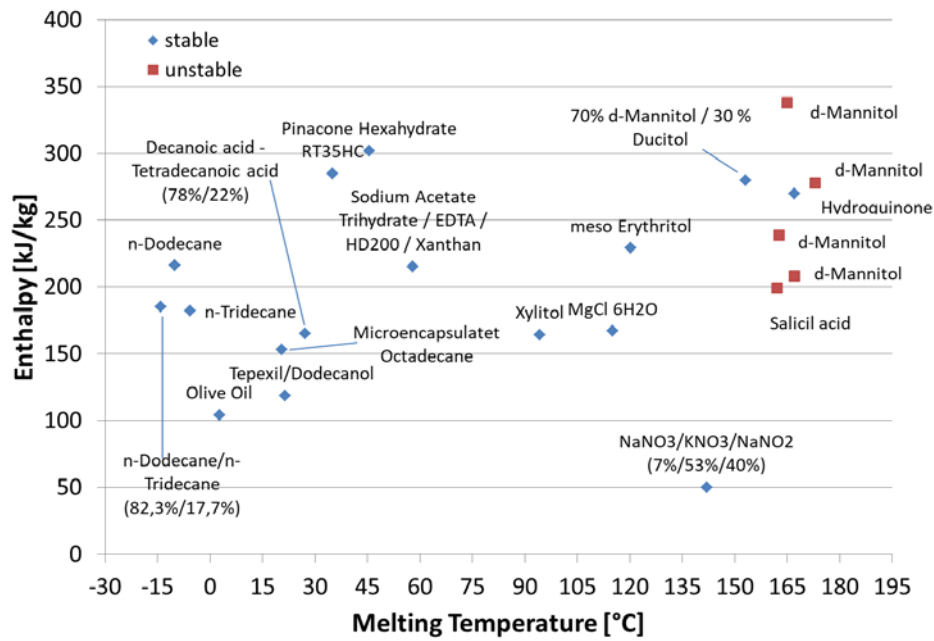
Figure 1: Overview on materials collected with their phase change enthalpy and melting temperature, red indicates the unstable materials
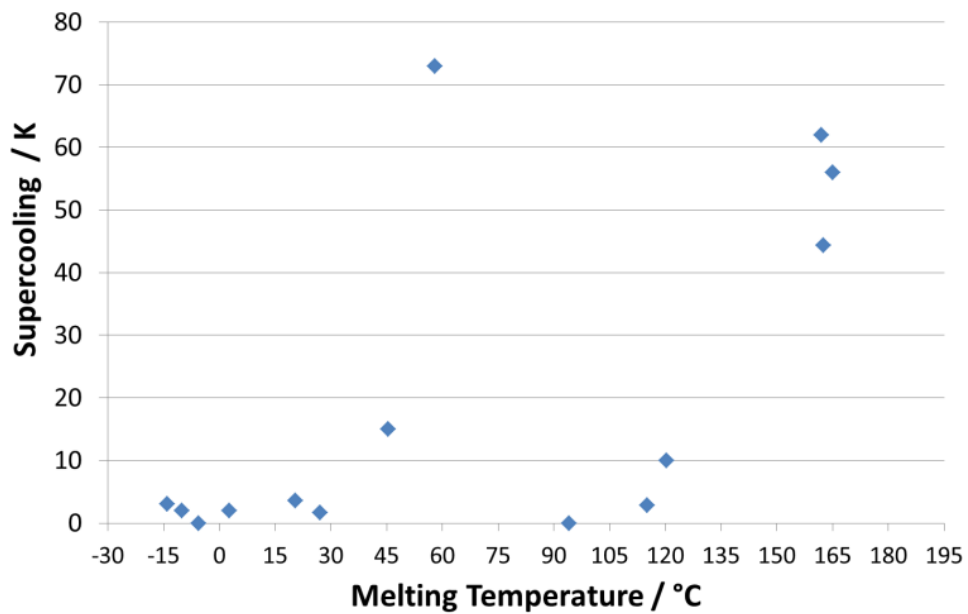

Figure 2: Supercooling behaviour of the materials
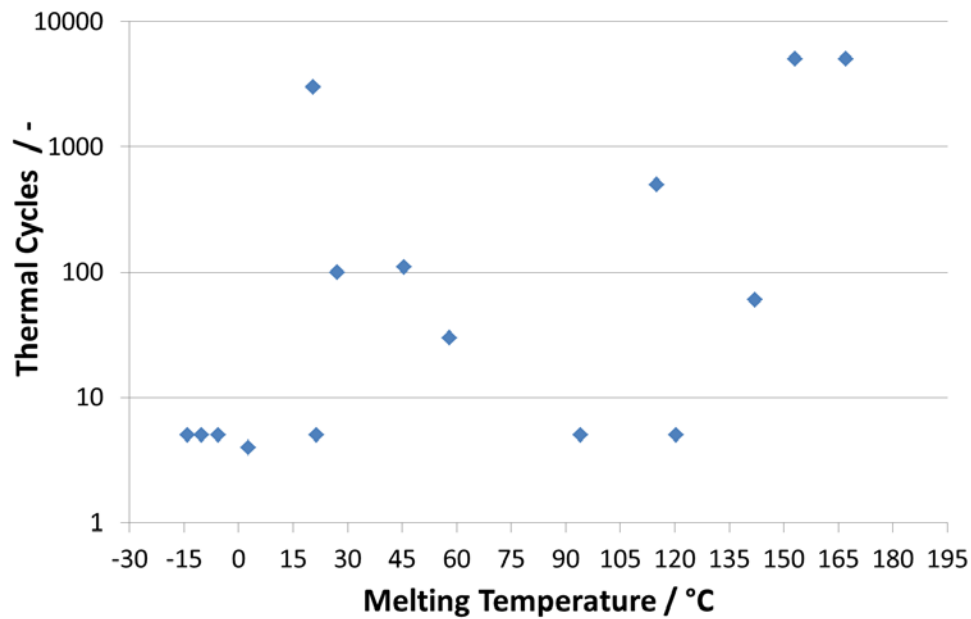
Figure 3: Stability of materials, number of cycles tested

4 Characterization of PCMs; Extended list of material properties for the characterization of novel PCM (D2P2)

4.1 Thermal conductivity

An intercomparative test of thermal diffusivity was carried out under the guidance of ZAE Bayern. The scope of the test was to develop a guideline for determination of thermal diffusivity and conductivity of PCM by means of flash technique in order to ensure reliable measurement data.

The investigated sample material was RT70HC provided by Rubitherm and was from the same batch used for the DSC intercomparison of IEA SHC Task 42 ECES Annex 29. The melting range of the PCM is around 70 °C.

In the first comparison in Task 42 Annex 29 different measurement methods were used and no procedures for the measurement and specimen preparation were defined. The results showed high deviations in the measurement results between the different laboratories (standard deviation of about $\pm\,30$ %). Therefore, it was decided to continue the work in Task 58 Annex 33.

To reduce the number of different measurement methods, it was decided to focus on the flash method, since it is the method available in most laboratories.

In order to avoid deviations caused by the sample preparation, the measured specimens were all prepared by the pilot laboratory. Since the cooling rate of the material has an influence on the crystal structure of the material and again the thermal conductivity, the specimens were prepared with two significantly different cooling rates (slow: 2 K/h; fast: LN_2).

In the first measurement round the thermal diffusivity of the solid PCM RT70HC was measured at 40 °C and 50 °C by five different laboratories with the flash technique. The results showed a lower standard deviation in the measurement results for the different cooling rates and a significant difference in the thermal diffusivity values between the different cooling rates of about 23 %.

Task 58 / Annex 33 Subtask 2P: Summary of Work

In the second measurement round the pulse energy was varied systematically. With rising pulse energy a trend towards lower thermal diffusivity can be observed for all measurements. The values at 0% pulse energy are calculated by linear extrapolation of the measured values. By this procedure the standard deviation of the results are reduced again.

In a third measurement round the specimens were prepared by the participating laboratories in order to test the influence of sample preparation. Compared to the previous measurement rounds, the standard deviation of the results was increased. In Table 1 the statistical results of the different measurement rounds are compared and in Figure 4 the results of the third measurement round are compared to the results of the previous Task/Annex.

Table 1: Relative standard deviations of the measurement results in the different measurement rounds for specimens with different cooling rates.
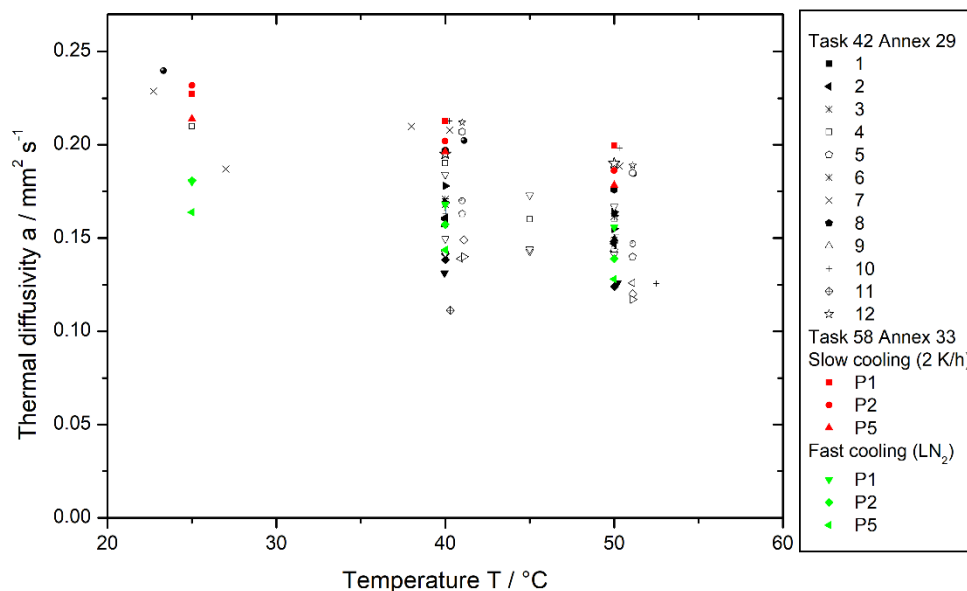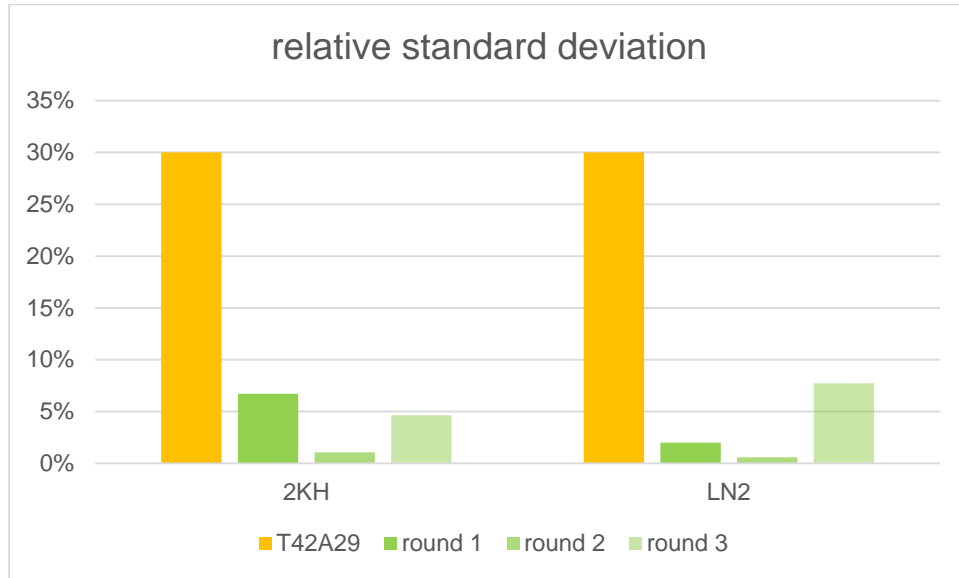
Figure 4: Comparison of thermal diffusivity measurement results from Task 42 Annex 29 and Task 58 Annex 33.

4.2 Viscosity

This work was led by the University of Zaragoza. A comparison of viscosity measurement devices was conducted for which Uni. Zaragoza, Uni. Bayreuth, and Fraunhofer ISE contributed. A publication on this was done in 2018[1]. Figure 5 depicts two results from this publication, which show the comparison of a measurement using a standard oil and of a paraffin.
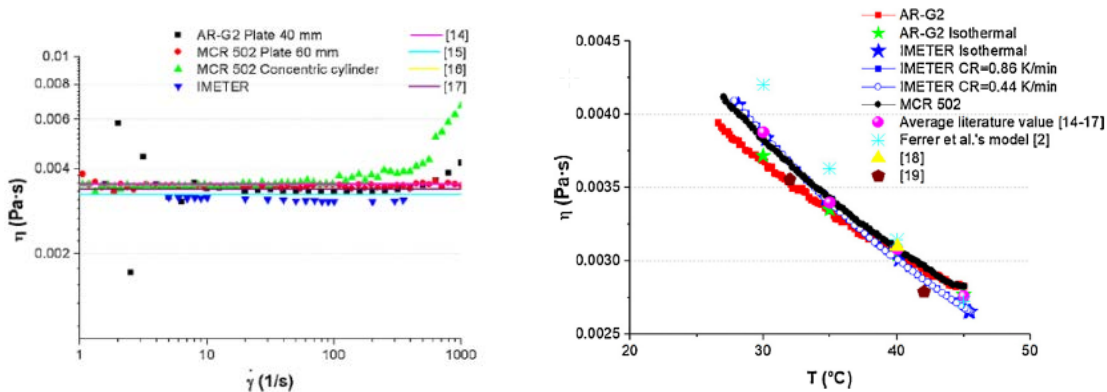
Figure 5: Comparison of deferent measurement devices for viscosity, left: comparison of a standard oil, right: comparative measurement of a paraffin

At Marche 7th and 8th 2018 a workshop on viscosity measurement of paraffin and salt nitrates was organised by the University of Zaragoza and hosted by Fraunhofer ISE in Freiburg. For the workshop Rheometers from TA-Instruments, Anton Paar and Thermo-Scientific were available in the lab. Previous to the measurement in the lab presentation were given by:

- Monica Delgado (Uni. Zaragoza) on the measurement procedure for PCMs
- Helena Navarro (Uni. Birmingham) on the measurement of high temperature PCMs
- Stephan Höhlein (Uni. Bayreuth) on the IMETER measurement principle
- Mr. Schwab, TA-Instruments, introduction into DHR 2 Rheometer

Figure 6 shows some impressions from the workshop and Figure 7 shows the viscosity of RT70HC in dependency of temperature.

---

[1] Delgado et al., Intercomparative tests on viscosity measurements of phase change materials, Thermochimica Acta 668 (2018) 159–168

Task 58 / Annex 33 Subtask 2P: Summary of Work

Figure 6: Workshop on measurement of viscosity via rotational rheometers, from above clockwise: Presentations, programming a rheometer, the high temperature measurement geometry filled with molten nitrate salt, filling the gap for the measurement of RT70HC
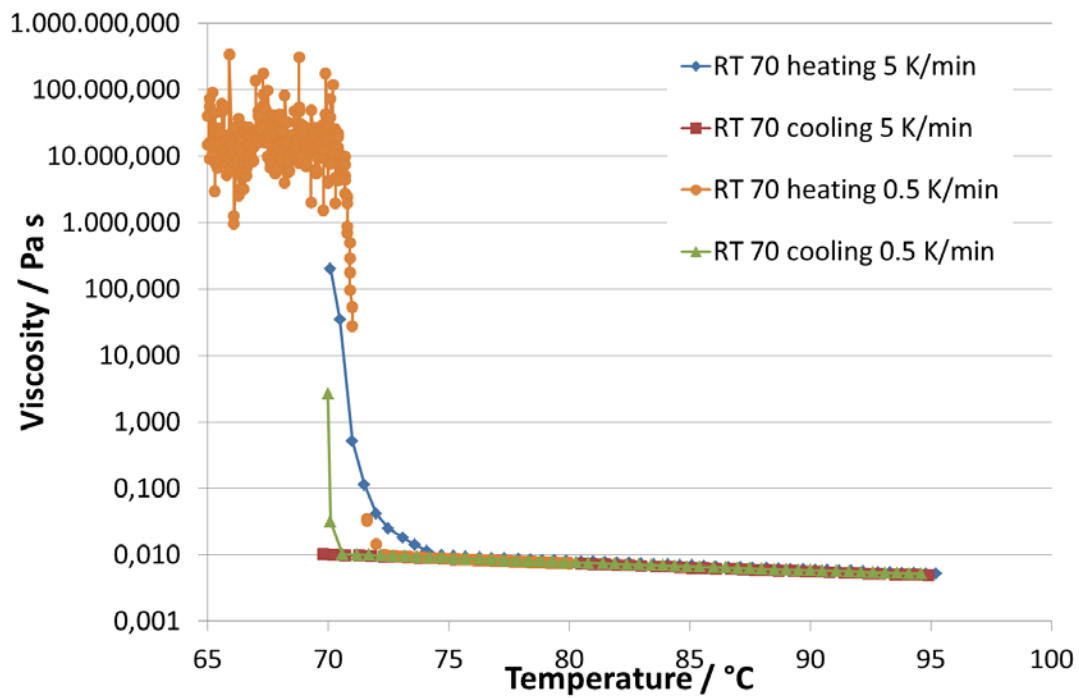
Figure 7: Example: result on the measurement of RT70HC, measuring from liquid to the phase transition, oscillatory method

4.3 cp-Measurement via DSC

4.3.1 Comparison of measurements

A comparison of specific heat capacity measurement via DSC was conducted. Five institutions contributed to this comparison. The measurements were done using different heating rates to determine the influence of low heating rates on the results. The target was to determine the resolution errors that are made using different DSCs and to check whether slow measurement is possible as the procedure to measure the enthalpy is based on slow heating rates. The results show, that for some devices slow heating rates lead to larger deviations (Figure 8), which is also observed in the comparison of the different results (Figure 9). The result shows that for a common measurement procedure to determine cp of PCMs it is not possible to uses slow heating rates. Therefore the measurement according to ASTM 1269 is suggested.
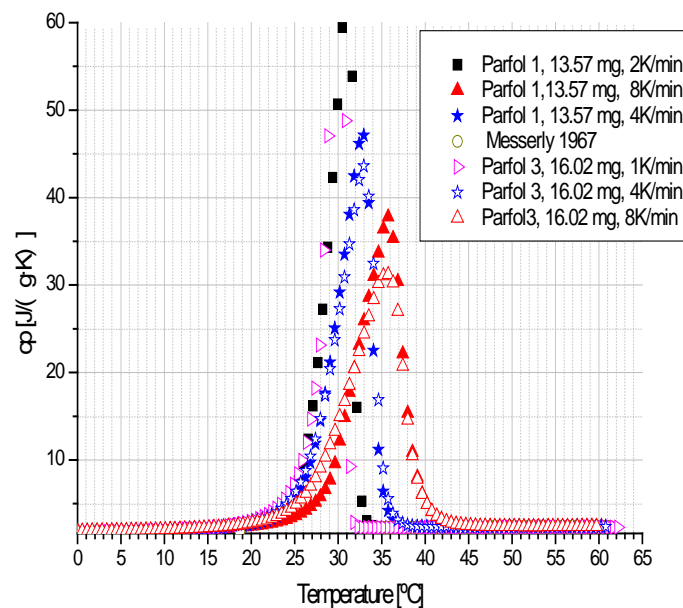
Figure 8 Influence of heating rate on the shape of the melting peak (measured by University of Zaragoza)

Task 58 / Annex 33 Subtask 2P: Summary of Work
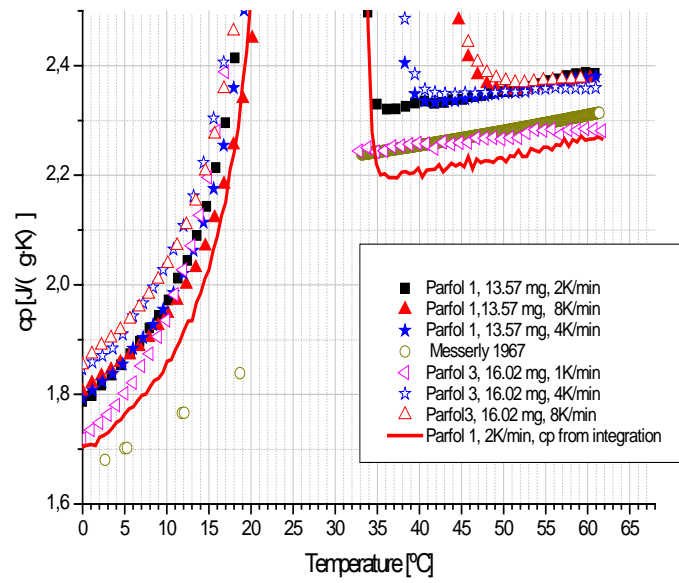
Figure 9: Influence of heating rate on result, measured by University of Zaragoza
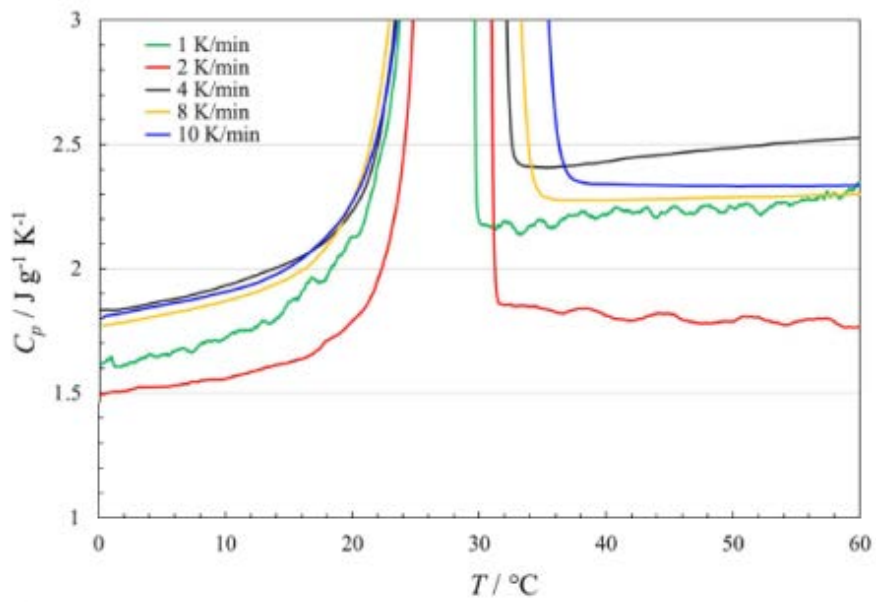
Figure 10: Influence of heating rate on result, measured by Dalhousie University
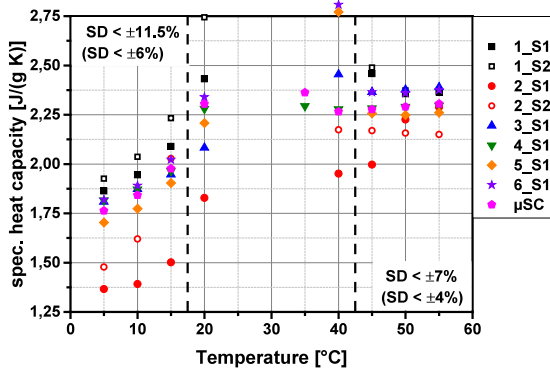
8 K/min
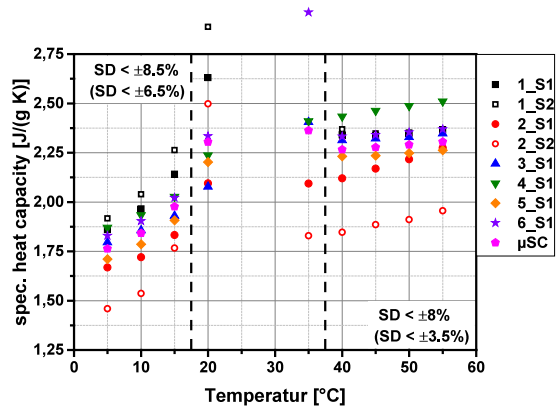
4 K/min
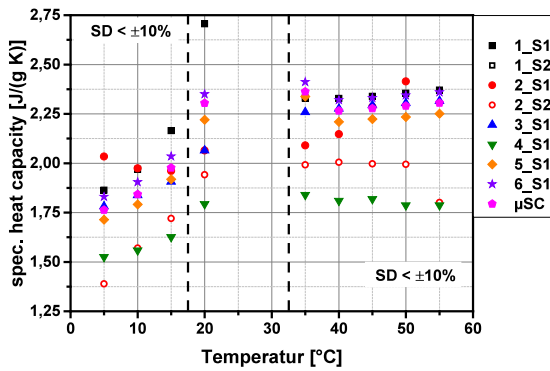
2 K/min
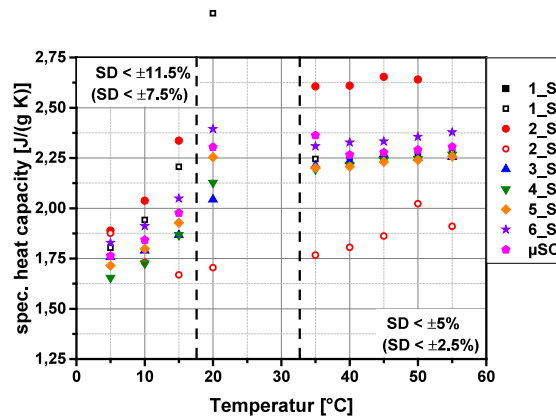
1 K/min

Figure 11: comparison of cp-measurement conducted at different institutions. Standard deviation (SD) in brackets without measurement of contributor 2, dashed lines mark the temperature range in which the material Parafol 18-97 is melting

Task 58 / Annex 33 Subtask 2P: Summary of Work

4.3.2 Method suggestion based on ASTM 1269

Short abstract from the ASTM1269 (not complete):

Reference Material—Synthetic sapphire.

1. Purge the DSC apparatus with dry nitrogen (or other inert gas) at a flow rate of 10 to 50 mL per min throughout the experiment.

2. Weigh a clean, empty specimen holder plus lid to a precision of 60.01 mg. Record as the tare weight.

3. Position the empty specimen holder plus lid and a reference specimen holder plus lid (weight-matched, if possible) in the DSC apparatus. NOTE 7—The same reference specimen holder + lid should be used for the sapphire standard run and for the test specimen run.

4. Heat or cool the DSC test chamber to the initial temperature for the experiment at 20 °C/min.

5. Hold the DSC test chamber isothermally at the initial temperature for at least 4 min to establish equilibrium. Record this thermal curve (refer to 12.4).

6. Heat the test specimen from the initial to final temperature at a rate of 20 °C/min. Continue to record thethermal curve. NOTE 8—The precision of this test method is enhanced by this high heating rate. Other heating rates may be used but shall be reported.

7. Record a steady-state isothermal baseline at the upper temperature limit. Refer to 12.4.

7.1. Terminate the thermal curve after this period.

7.2. Cool the DSC test chamber to ambient temperature.

8. Place the sapphire standard into the same specimen holder plus lid used in 13.1.2.

9. Weigh sapphire standard and specimen holder plus lid to a precision of 0.01 mg and record the weight.

Following the additional definitions (12., 13., 14., and 15.) for conditioning , procedure, calculation and report.

4.4 Density measurement

A comparison of density measurement was undertaken. The contributors used different technologies for the measurement (oscillating U-tube, Archimedes principle, helium pycnometer). Results are shown in Figure 12. The results reveal large deviations between measurement principals and different institutions. During the TA5833 it was decided not to proceed with this task.
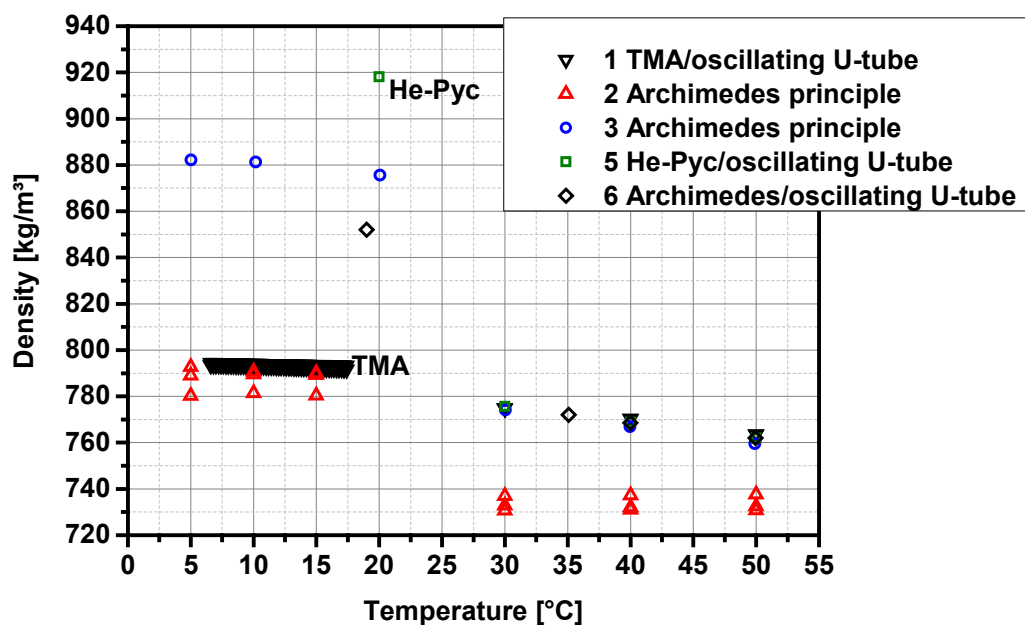
Figure 13: Comparison of density measurement done at different institutions

Task 58 / Annex 33 Subtask 2P: Summary of Work

5 Database;
Measured material data for the maintenance and expansion of the PCM Database (D2P3)

5.1.1 Database

The database has a private and a public section. Data of 56 measurements are available in the private section from which 16 datasets are publicly available. The last material uploaded was in October 2019. Figure 12 shows on the overview table of public available materials. Figure 13 illustrates one example for details available for a PCM.

| Home | User | News | Workshops | Measurement-Standards & Tools | PCM | Sorption-Materials | Wiki PCM | |

You are here: Home / PCM

Database PCM

Show 25 ▼ entries

Search:

Name	Institution	Last Change	Melting Temperature	Heat of Fusion	Density (liquid)	Thermal Conductivity (liquid)	Viscosity (liquid)
			[°C]	[kJ/kg]	[kg/m3]	[W/mK]	[mPas]
CaBr2·6H2O	ZAE-Bayern	Apr 19, 2017	33.29	135.5	1956.0		
gypsum board	Fraunhofer ISE	Oct 18, 2019	18.48	19.4			
HDPE natur NT D960/6	Fraunhofer ISE	Oct 13, 2015	128.0	219.0			
Lauric acid (dodecanoic acid)	ZAE-Bayern	Apr 19, 2017	43.5	178.2			
Lauric acid (Dodecanoic acid)	Fraunhofer ISE	Sep 07, 2017	43.65	180.0			
Methyl Stearate (methyl octadecanoate)	Fraunhofer ISE	Sep 07, 2017	36.7	208.0			
Micronal DS 5038 X	Fraunhofer ISE	Apr 09, 2018	21.5	96.0			
Micronal DS 5040 X	Fraunhofer ISE	Apr 09, 2018	19.07	93.6			
n-Octadecane, 99%	Fraunhofer ISE	Sep 07, 2017	27.5	233.0			
n-Octadecane, 99.5+%	Fraunhofer ISE	Sep 07, 2017	27.66	237.0			
NaNO3	Fraunhofer ISE	Oct 02, 2017	307.0	175.0			
Octadecan Parafol 18-97	Fraunhofer ISE	Jun 13, 2017	27.35	231.3			
PEG1000	Fraunhofer ISE	Sep 07, 2017	31.0	150.0			
PEG600	Fraunhofer ISE	Sep 07, 2017	13.0	137.0			
Potassium nitrate (KNO3)	Fraunhofer ISE	Sep 13, 2016	329.6	92.5			
RT 70 HC	Fraunhofer ISE	Oct 13, 2015	70.1	256.4			

Figure 14: Screenshot of the public available datasets (www.thermalmaterials.org)

| Contents | View | Edit | Sharing | | Actions ▼ Display ▼ Add new... ▼ State: Published ▼ |

gypsum board

by Georg Hagelstein — last modified Oct 18, 2019 11:25 AM — History

Material details

Description	gypsum board with microencapsulated paraffin
Type	composite
Manufacturer	not available
Added by	Fraunhofer ISE

- Enthalpy (T) (enthalpy-sample-1)
- Enthalpy (T) (enthalpy-sample-2)
- Enthalpy (T) (enthalpy-sample-3)
- Safety sheets
- Additional information

Figure 15: Details example for compound of gypsum and PCM

6 Wiki for PCM

A Wiki was developed to document the nomenclature which is uses in the field of PCMs. The Wiki is open, so that everybody can add new terms and definitions or to change existing ones. Figure 16 shows a screenshot.

Figure 16: Screen shot of the Wiki for PCM (www.thermalmaterials.org/wiki-pcm, 30/01/2020)

Task 58 / Annex 33 Subtask 2P: Summary of Work

7 Annex

7.1 Material Data Sheets

First Name Saman Nimali		Family Name Gunasekara
Institution KTH Royal Institute of Technology		
Address		
Street Brinellvägen 68		
Zip-Code 100 44	City Stockholm	
Country Sweden		
email saman.gunasekara@energy.kth.se		Telephone +46 73652 3339
Material Data/Information		Date: 05.12.2017
Material Designation Meso-Erythritol (99% purity), thermo-physical characterizations using the T-History, TPS and XRD methods		
PCM (single component (U)/ composite (Cm) or, a blend: binary (B)/ ternary (T).., eutectic (E)/ solid solution (Ss)/ compound (C)) 1. meso-erythritol (U) 2. 3.		Composite material(s) 1. --- 2. 3.
Data for the component/ composite/ blend		

Melting Temperature [°C] 112.6-128.0	Minimum Temperature [°C] 0 (min. of the measurement range)
Storage Capacity [kJ/kg] 229±64	Maximum Temperature [°C] 138 (max. of the measurement range)
Density [kg/l] Unevaluated	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h) ~5 cycles, and the given results exclude the very first melting. The melting enthalpy found here seem at least 19-25 % lower than the literature values, possibly due to the cycled behavior analyzed here.
Supercooling [K] ~10 K	Material compatibility Not yet tested systematically. Nevertheless, it appears to be compatible with metals, but cracks glass (especially at higher temperatures or during solidification respectively).
Technology readiness levels (TRL) Unevaluated	Additional Parameter Thermal conductivity: 0.32 W/m K (liquid at 125 °C), and 0.59 W/m K (solid at 20 °C)
If possible, please insert DSC-curve or other characteristic graph	Please insert an image/photo Erythritol X-Ray Diffraction (XRD) characteristics at room temperature

Erythritol Enthalpy vs. Temperature
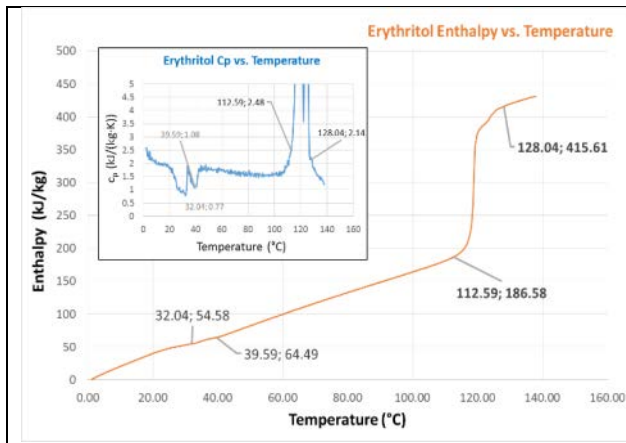
Thermally activated change in Erythritol [Gunasekara et al., 2016]:
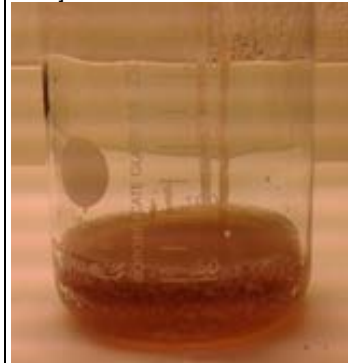
Target Applications (up to 4 most relevant)
1. district heating
2. mobile heat storage
3.
4.

Comments
The main reason for the low melting enthalpy is possibly the thermally activated change (browning and thickening of material at the end of the T-history cycles, conducted within air). However, the literature data for the melting enthalpy of erythritol are also very disparate, within a very wide range: 281–370 kJ/kg, most often presented without specifying the number of cycles these values represent, and if specified, mostly representing only the first melting.
The reuse of this given photograph of the browned erythritol may require permission from the publisher of Gunasekara et al., 2016 (Elsevier).

Task 58 / Annex 33 Subtask 2P: Summary of Work

First Name		Family Name
Saman Nimali		Gunasekara

Institution
KTH Royal Institute of Technology

Address

Street
Brinellvägen 68

Zip-Code	City
100 44	Stockholm

Country
Sweden

email	Telephone
saman.gunasekara@energy.kth.se	+46 73652 3339

Material Data/Information	Date: 21.12.2017

Material Designation
Xylitol (99% purity), thermo-physical characterizations using the T-History, TPS and XRD methods

PCM (single component (U)/ composite (Cm) or, a blend: binary (B)/ ternary (T).., eutectic (E)/ solid solution (Ss)/ compound (C))	Composite material(s)
1. xylitol (U)	1. ---
2.	2.
3.	3.

Data for the component/ composite/ blend	
Melting Temperature [°C] 90.6–97.7	Minimum Temperature [°C] 0 (min. of the measurement range)
Storage Capacity [kJ/kg] 164±46	Maximum Temperature [°C] 138 (max. of the measurement range)
Density [kg/l] Unevaluated	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h) ~5 cycles, and the given results exclude the very first melting. This given melting enthalpy is for the 2nd melting, because, afterwards the melting enthalpy was extremely subtle, due to heavy influence of glass transition. This enthalpy seems at least 19-25 % lower than the literature values, possibly due to the cycled behavior analyzed here.
Supercooling [K] Very large (as it becomes glassy)	Material compatibility Not yet tested systematically. Nevertheless, it appears to be compatible with metals, but cracks glass (especially at higher temperatures or during solidification respectively).
Technology readiness levels (TRL) Unevaluated	Additional Parameter Thermal conductivity: 0.41-0.43 W/m K (liquid at 110 °C), and 0.37 W/m K (solid at 20 °C)

If possible, please insert DSC-curve or other characteristic graph	Please insert an image/photo Xylitol X-Ray Diffraction (XRD) characteristics at room temperature
	Thermally activated change in xylitol (browned thickened material) [Gunasekara et al., 2016]:

Glassy nature of xylitol (the spatula is stuck within the extremely thick glassy liquid) [Gunasekara et al., 2016]:
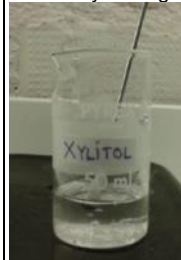
Target Applications (up to 4 most relevant)
1. district heating
2. mobile heat storage
3.
4.

Comments

The main reasons for the low melting enthalpy are possibly the heavy influence of glass transition as well as thermally activated change (browning and thickening of material at the end of the T-history cycles, conducted within air). Another reason is that xylitol, before its stable melting, indicated a minor change at a lower temperature (see figures at left-side). The literature data for the melting enthalpy of xylitol are also disparate, within a wide range: 219-280 kJ/kg, most often presented without specifying the number of cycles these values represent, and if specified, mostly representing only the first melting.

The reuse of this given photographs of the browned xylitol and glassy xylitol may require permission from the publisher of Gunasekara et al., 2016 (Elsevier).

First Name Saman Nimali	Family Name Gunasekara
Institution KTH Royal Institute of Technology	
Address	
Street Brinellvägen 68	
Zip-Code 100 44	City Stockholm
Country Sweden	
email saman.gunasekara@energy.kth.se	Telephone +46 73652 3339
Material Data/Information	Date: 05.12.2017

Material Designation
n-tridecane (99%+ pure), thermal characterizations using T-History method

PCM <mark>(single component (U)/ composite (Cm) or, a blend: binary (B)/ ternary (T).., eutectic (E)/ solid solution (Ss)/ compound (C))</mark>	<mark>Composite</mark> material(s)
1. *n*-tridecane $CH_3(CH_2)_{11}CH_3$ (U) 2. 3.	1. --- 2. 3.

Data for the <mark>component/ composite/ blend</mark>	
Melting Temperature [°C] -6.7 to -4.5 (average over 2nd to 4th melting cycles)	Minimum Temperature [°C] -28 (min. of the measurement range)
Storage Capacity [kJ/kg] 182±18 (average over 2nd to 4th melting cycles)	Maximum Temperature [°C] 8 (max. of the measurement range)
Density [kg/l] Unevaluated	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h) 5 cycles, while the given results exclude the very first melting. The results agree well with the literature values.
Supercooling [K] Negligible	Material compatibility Not yet tested systematically. Nevertheless, it appears to be compatible with metals and glass but incompatible with plastics.
Technology readiness levels (TRL) Unevaluated	Additio<mark>nal</mark> Parameter Also undergoes a polymorphic phase change, between −19.4 °C and −18.2 °C (in heating) and −19.5 °C and −20.3 °C (in cooling) with the respective enthalpy changes 66 ± 7 kJ/kg and 46 ± 5 kJ/kg.
If possible, please insert DSC-curve or other characteristic graph	Please insert an image/photo

n-tridecane Melting Enthalpy vs. Temperature

n-tridecane Cp vs. Temp.

Data point labels on melting enthalpy curve: -4.57; 335.82; -6.67; 152.74; -18.14; 101.63; -19.40; 34.96

n-tridecane Freezing Temperature Profile (negligible supercooling)

Data point labels on freezing profile: -6.19; -6.39
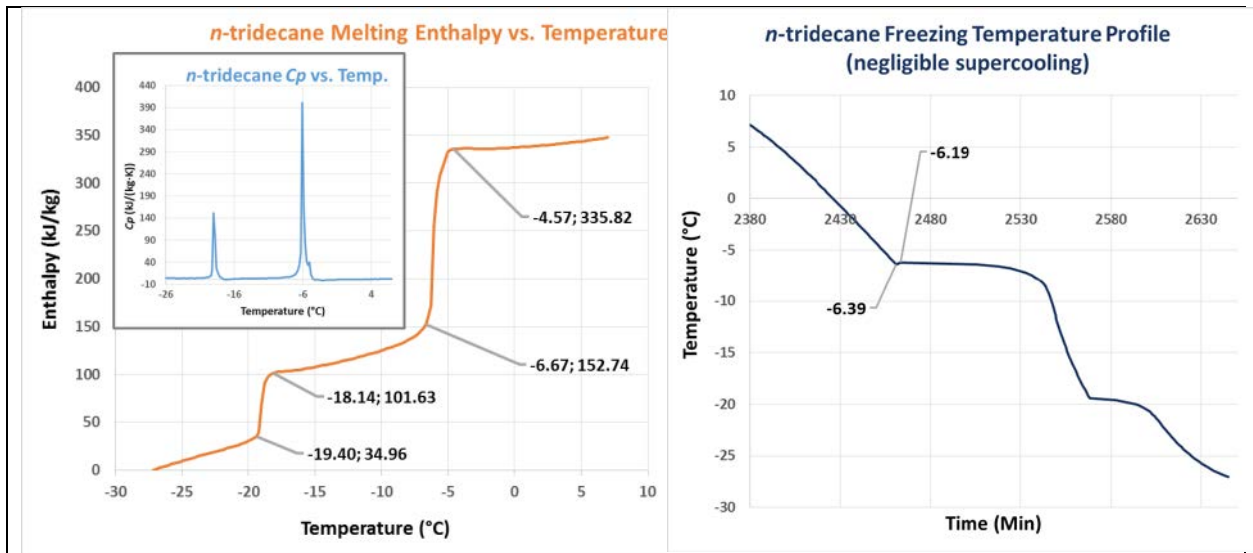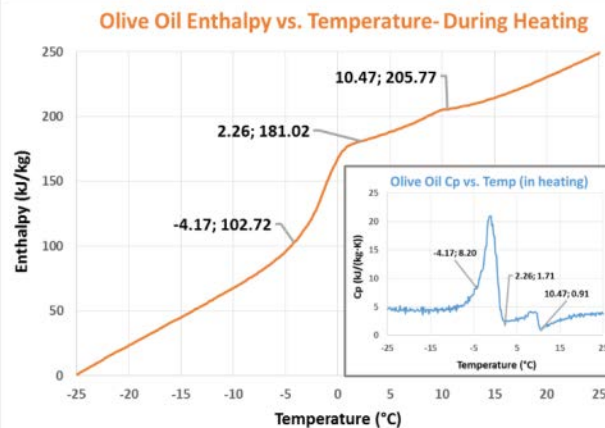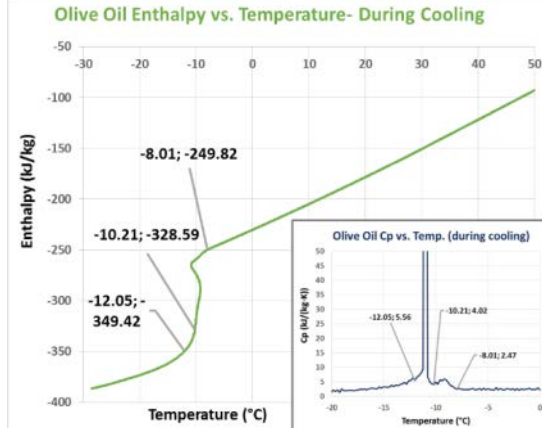
Target Applications (up to 4 most relevant)

1. pre-cooling of refrigerants
2. cold storage below 0 °C
3. solid-solid PCM applications for cooling (considering the polymorphic phase)
4.

Comments

The results shown are for the 3rd melting (and freezing) cycle (in the T-history evaluation). Nonetheless, for all the evaluated cycles the sample displayed very consistent behaviors during both melting and freezing. This material (n-tridecane) also undergoes a polymorphic phase change (detailed under 'Additional Parameters).

Task 58 / Annex 33 Subtask 2P: Summary of Work

First Name		Family Name
Saman Nimali		Gunasekara
Institution		
KTH Royal Institute of Technology		
Address		
Street		
Brinellvägen 68		
Zip-Code		City
100 44		Stockholm
Country		
Sweden		
email		Telephone
saman.gunasekara@energy.kth.se		+46 73652 3339
Material Data/Information		Date: 04.01.2018

Material Designation
Olive Oil (virgin oil, commercial grade, unknown purity), thermal characterizations using T-History method

PCM (single component (U)/ composite (Cm) or, a blend: binary (B)/ ternary (T).., eutectic (E)/ solid solution (Ss)/ compound (C))	Composite material(s)
1. Olive oil (multicomponent blend)	1. ---
2.	2.
3.	3.

Data for the component/ composite/ blend

Melting Temperature [°C]	Minimum Temperature [°C]
-4.5 to 10.3 (average over 2nd to 4th melting cycles, of 2 identical samples)	-30 (min. of the measurement range)
Storage Capacity [kJ/kg]	Maximum Temperature [°C]
104±10 (average over 2nd to 4th melting cycles, of 2 identical samples)	80 (max. of the measurement range)
Density [kg/l]	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h)
Unevaluated	4 cycles, while the given results exclude the very first melting. The results agree well with the available literature values.
Supercooling [K]	Material compatibility
Minor (~2 K)	Not yet tested systematically. Nevertheless, it appears to be compatible generally with metals, glass and plastics.
Technology readiness levels (TRL)	Additional Parameter
Unevaluated	Hysteresis is rather considerable (3.5 -22 °C), primarily due to its wide melting temperature range.

If possible, please insert DSC-curve or other characteristic graph	Please insert an image/photo

Olive Oil Enthalpy vs. Temperature- During Heating

Data points labeled: 10.47; 205.77 ; 2.26; 181.02 ; -4.17; 102.72

Inset: Olive Oil Cp vs. Temp (in heating) — -4.17; 8.20 ; 2.26; 1.71 ; 10.47; 0.91

Olive Oil Enthalpy vs. Temperature- During Cooling

Data points labeled: -8.01; -249.82 ; -10.21; -328.59 ; -12.05; -349.42

Inset: Olive Oil Cp vs. Temp. (during cooling) — -12.05; 5.56 ; -10.21; 4.02 ; -8.01; 2.47

Frozen Olive oil samples (from a simple freezing pre-test):

Target Applications (up to 4 most relevant)
1. pre-cooling (within -4.5 to 10.3 °C) in chilling applications
2.
3.
4.

Comments
The results shown are for the 3rd melting (and freezing) cycle (in the T-history evaluation), for one of the 2 tested identical samples. For all the evaluated cycles the samples anyways displayed very consistent behaviors respectively during melting and freezing.

The samples displayed a secondary phase change peak (which is a possible solid-solid phase change) consecutively before the melting c_p peak or, after the freezing c_p peak, respectively. This secondary peak was however smaller during cooling, as compared to that observed during heating.

This secondary peak could be an indication of a near-eutectic composition in this multicomponent blend, or could be a polymorphic phase incurred due to the major triglyceride component in olive oil: triolein. The study indicates that: olive oil is not recommendable as a PCM as it is, however, compositional refinements could yield an attractive renewable PCM out of it.

First Name	Family Name
Saman Nimali	Gunasekara
Institution KTH Royal Institute of Technology	
Address	
Street Brinellvägen 68	
Zip-Code 100 44	City Stockholm
Country Sweden	
email saman.gunasekara@energy.kth.se	Telephone +46 73652 3339
Material Data/Information	Date: 21.12.2017

Material Designation
25-30 mol% Erythritol in Xylitol, thermal characterizations using the T-History and TPS methods

PCM (single component (U)/ composite (Cm) or, a blend: binary (B)/ ternary (T).., eutectic (E)/ solid solution (Ss)/ compound (C))	Composite material(s)
1. Erythritol-Xylitol (BE), @ 25-30 mol% Erythritol	1. ---
2.	2.
3.	3.

Data for the component/ composite/ blend (shown for 25 and 30 mol% Erythritol respectively)

Melting Temperature [°C] 80.6–101.6 and 80.0–91.1	Minimum Temperature [°C] 0 (min. of the measurement range)
Storage Capacity [kJ/kg] 59.5±17 and 45±13 (Total including melting as well as glassy and intermediate changes: 172±48 and 200±56)	Maximum Temperature [°C] 138 (max. of the measurement range)
Density [kg/l]	Cycle Stability (how many thermal cycles tested, if possible reduction in

Unevaluated	kJ/kg·h)
	~5 cycles, and the given results exclude the very first melting. This given melting enthalpy is for the 2nd melting, because, afterwards the melting enthalpy was extremely subtle, due to heavy influence of glass transition. This enthalpy seems at least 19-25 % lower than the literature values, possibly due to the cycled behavior analyzed here.
Supercooling [K]	Material compatibility
Very large (as it becomes glassy)	Not yet tested systematically. Nevertheless, it appears to be compatible with metals, but cracks glass (especially at higher temperatures or during solidification respectively).
Technology readiness levels (TRL)	Additional Parameter: **for 25 mol% erythritol**
Unevaluated	Thermal conductivity: 0.40 W/m K (liquid at 110 °C), and 0.39 W/m K (solid at 20 °C)
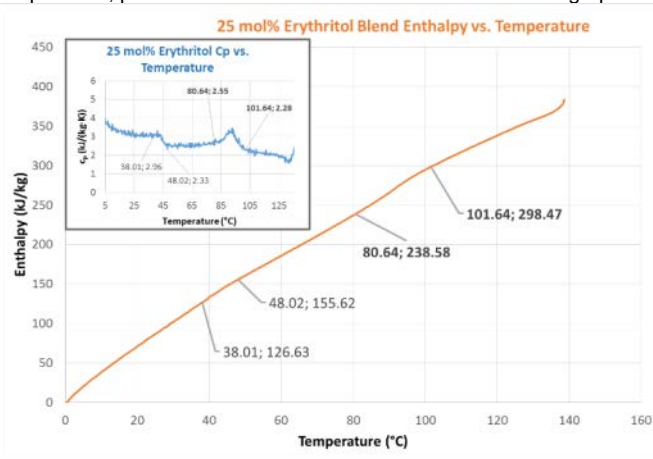
If possible, please insert DSC-curve or other characteristic graph	Please insert an image/photo
	Erythritol-Xylitol Binary Phase Diagram [Gunasekara et al., 2018]
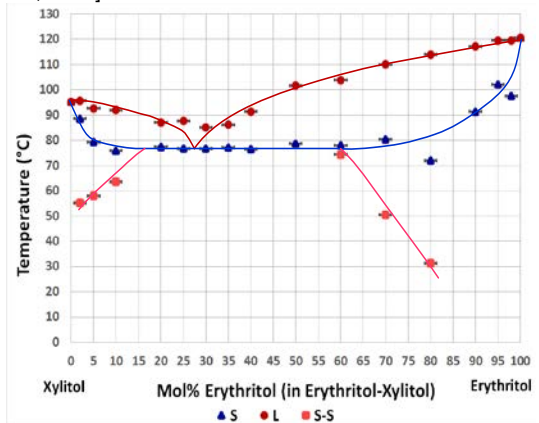
Target Applications (up to 4 most relevant)
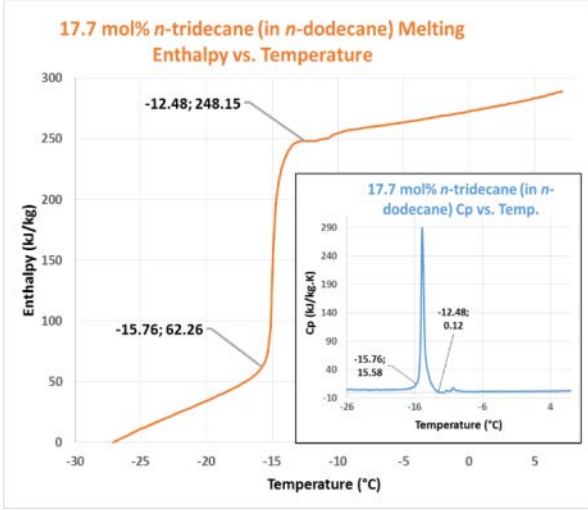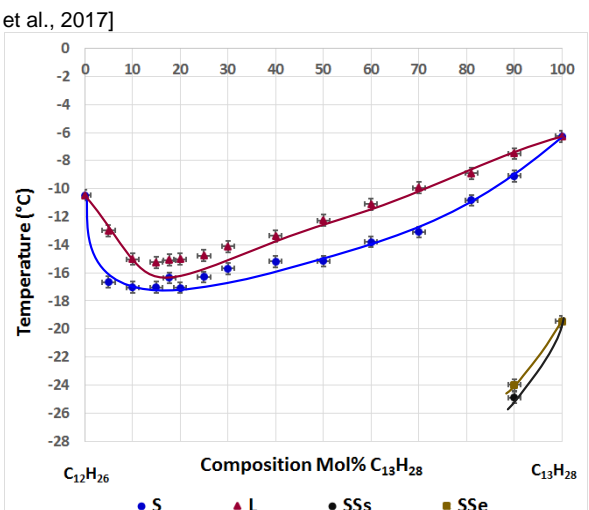
1. district heating
2. mobile heat storage
3.
4.

Comments

The eutectic was also heavily affected by glass transition, thus resulting in a very low melting enthalpy. Furthermore, after the 2nd melting (which is what's shown here) the phase change was almost completely overcome by glass transition. This composition also underwent thermally activated change (browning and thickening of material at the end of the T-history cycles, conducted within air), which is another possible reason for lowered melting enthalpy. The literature data for the melting enthalpy of this eutectic blend is larger, however cannot be compared directly as those studies do not specify what cycle it represented.

The reuse of this given phase diagram may require permission from the publisher (Elsevier).

First Name		Family Name
Saman Nimali		Gunasekara
Institution		
KTH Royal Institute of Technology		
Address		
Street		
Brinellvägen 68		
Zip-Code	City	
100 44	Stockholm	
Country		
Sweden		
email	Telephone	

saman.gunasekara@energy.kth.se	+46 73652 3339
Material Data/Information	Date: 05.12.2017

Material Designation

n-dodecane-*n*-tridecane binary system (made from *n*-dodecane and *n*-tridecane (i.e., $CH_3(CH_2)_{11}CH_3$ and $CH_3(CH_2)_{11}CH_3$) of 99% and 99%+ purity), thermal characterizations using T-History method

PCM (single component (U)/ composite (Cm) or, a blend: binary (B)/ ternary (T).., eutectic (E)/ solid solution (Ss)/ compound (C))	Composite material(s)
1. *n*-dodecane-*n*-tridecane (B, possibly congruent melting Ss), @ ~17.7 *n*-tridecane	1. ---
2.	2.
3.	3.

Data for the component/ composite/ blend

| Melting Temperature [°C] −15.7 °C to −12.4 °C (average over 2nd to 4th melting cycles)	Minimum Temperature [°C] -28 (min. of the measurement range)
Storage Capacity [kJ/kg] 185±19 (average over 2nd to 4th melting cycles)	Maximum Temperature [°C] 8 (max. of the measurement range)
Density [kg/l] Unevaluated	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h) 5 cycles, while the given results exclude the very first melting. The results agree well with the literature values.
Supercooling [K] Negligible	Material compatibility Not yet tested systematically. Nevertheless, it appears to be compatible with metals and glass but incompatible with plastics.
Technology readiness levels (TRL) Unevaluated	Additional Parameter Hysteresis of this blend is also minor, of around 1-3 °C.

If possible, please insert DSC-curve or other characteristic graph	Please insert an image/photo *n*-dodecane-*n*-tridecane Binary Phase Diagram [Gunasekara et al., 2017]

Target Applications (up to 4 most relevant)

1. pre-cooling of refrigerants
2. cold storage below 0 °C
3. solid-solid PCM applications for cooling (considering the polymorphic phase)
4.

Task 58 / Annex 33 Subtask 2P: Summary of Work

Comments

The results shown are for the 3rd melting (and freezing) cycle (in the T-history evaluation). Nonetheless, for all the evaluated cycles the blend displayed very consistent behaviors during both melting and freezing. This system appears to form a congruent minimum melting solid solution at around 17.7 mol% *n*-tridecane composition. That therefore appears to be ideal as a PCM for freezing applications. However, as the phase diagram here was presented only based on thermal characterizations, physicochemical characterizations and cycling stability tests are necessary future steps to confirm its PCM-suitability.

The reuse of the phase diagram given may require permission from the publisher (Elsevier).

First Name		Family Name	
Saman Nimali		Gunasekara	

Institution	
KTH Royal Institute of Technology	

Address	

Street	
Brinellvägen 68	

Zip-Code		City	
100 44		Stockholm	

Country	
Sweden	

email	Telephone
saman.gunasekara@energy.kth.se	+46 73652 3339

Material Data/Information	Date: 05.12.2017

Material Designation	
n-dodecane (99% pure), thermal characterizations using T-History method	

PCM (single component (U)/ composite (Cm) or, a blend: binary (B)/ ternary (T).., eutectic (E)/ solid solution (Ss)/ compound (C))	Composite material(s)
1. *n*-dodecane $CH_3(CH_2)_{10}CH_3$ (U)	1. ---
2.	2.
3.	3.

Data for the component/ composite/ blend	

Melting Temperature [°C]	Minimum Temperature [°C]
-11.4 to -8.8 (average over 2nd to 4th melting cycles)	-28 (min. of the measurement range)

Storage Capacity [kJ/kg]	Maximum Temperature [°C]
216±22 (average over 2nd to 4th melting cycles)	8 (max. of the measurement range)

Density [kg/l]	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h)
Unevaluated	5 cycles, while the given results exclude the very first melting. The results agree well with the literature values.

Supercooling [K]	Material compatibility
Minor (~2 K)	Not yet tested systematically. Nevertheless, it appears to be compatible with metals and glass but incompatible with plastics.

Technology readiness levels (TRL)	Additional Parameter
Unevaluated	--

If possible, please insert DSC-curve or other characteristic graph	Please insert an image/photo

Task 58 / Annex 33 Subtask 2P: Summary of Work

Target Applications (up to 4 most relevant)

1. pre-cooling of refrigerants
2. cold storage below 0 °C
3.
4.

Comments

The results shown are for the 3rd melting (and freezing) cycle (in the T-history evaluation). Nonetheless, for all the evaluated cycles the sample displayed very consistent behaviors during both melting and freezing.

First Name		Family Name
Christoph		Rathgeber
Institution		
ZAE Bayern		
Address		
Street		
Walther-Meissner-Str. 6		
Zip-Code	City	
85748	Garching	
Country		
Germany		
email		Telephone
christoph.rathgeber@zae-bayern.de		+49 89 329 442 88
Material Data/Information		Date: 20.03.2018
Material Designation		
Pinacone hexahydrate		
PCM		Compound material(s)
1. Pinacone hexahydrate		1.

Data for the compound or PCM if no compound

Melting Temperature [°C]	Minimum Temperature [°C]
45.5 °C (onset) [1]	not available
Heat of fusion [kJ/kg]	Maximum Temperature [°C]
302 ± 15 [1]	Not tested yet
Density [kg/l]	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h)
0.97 (in liquid state) [1]	Stable after 110 thermal cycles between 20 and 60 °C (according to PCM RAL stability criteria) [2]
Supercooling [K]	Material compatibility
~40 K (DSC), ~15 K (T-History), ~10-15 K (thermal cycling device, 60 ml sample) [1, 2]	Not tested yet
Technology readiness levels (TRL)	Additional Parameter(s)
?	

Source: [1]

Source: [2]

Target Applications (up to 4 most relevant)

1. Intermediate storage for heat pumps in space heating systems
2. Thermal protection of electronics / battery systems

Comments

The main obstacle for an application of pinacone hexahydrate are its costs: 300 €/kg for 99% purity. The costs of technical grade pinacone are unknown. [2]

Task 58 / Annex 33 Subtask 2P: Summary of Work

References
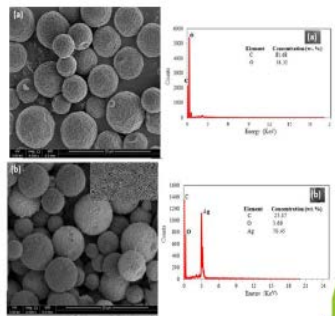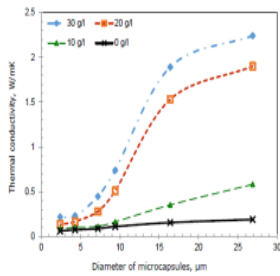
[1] Rathgeber, C., Schmit, H., Hennemann, P., & Hiebler, S. (2014). Investigation of pinacone hexahydrate as phase change material for thermal energy storage around 45 C. *Applied Energy*, *136*, 7-13.

[2] Grisval, A. (2017). Investigation on organic hydrates as phase change materials (PCM). Master's Thesis, Technical University Munich.

First Name		Family Name
Stephan		Höhlein
Institution		
Chair of Engineering Thermodynamics and Transport Processes (LTTT), University of Bayreuth		
Address		
Street		
Universitätsstraße 30		
Zip-Code	City	
95447	Bayreuth	
Country		
Germany		
email		Telephone
Stephan.Hoehlein@uni-bayreuth.de		+49 921 55 7520
Material Data/Information		Date: 12.01.2018
Material Designation		
Magnesiumchloride Hexahydrate, MgCl$_2$ 6H$_2$O		
PCM		Compound material(s)
1. Magnesiumchloride Hexahydrate		1.
2.		2.
3.		3.

Data for the compound or PCM if no compound	
Melting Temperature [°C] 115,1 (onset) [1]	Minimum Temperature [°C]
Storage Capacity [kJ/kg] 166,9 [1]	Maximum Temperature [°C]
Density [kg/l] 1,5955 (20 °C) [1] 1,4557 (120 °C) [1]	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h) 500 cycles at DSC-scale, ~ 1 % reduction in melting enthalpy [1]
Supercooling [K] 30 (sample size ~10 mg) [1] 2,8 (sample size ~100 g) [1]	Material compatibility Anodized aluminium [2]
Technology readiness levels (TRL)	Addition Parameter

Enthalpy and heat capacity of MgCl$_2$ 6H$_2$O [1]

Crystallization of MgCl$_2$ 6H$_2$O within an aluminium capsule

Target Applications (up to 4 most relevant)
1. Waste heat
2. Process heat
3. Mobile storage systems
4.

Task 58 / Annex 33 Subtask 2P: Summary of Work

Comments
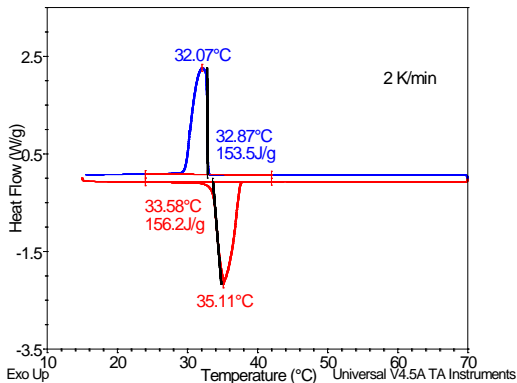
[1] S. Höhlein, A. König-Haagen, and D. Brüggemann, "Thermophysical Characterization of MgCl2·6H2O, Xylitol and Erythritol as Phase Change Materials (PCM) for Latent Heat Thermal Energy Storage (LHTES)," Materials (Basel). vol. 10, no. 4, p. 444, Apr. 2017.

[2] D. Brüggemann, A. König-Haagen, R. R. Kasibhatla, S. Höhlein, U. Glatzel, R. Völkl, and N. Agarkov, "Entwicklung makroverkapselter Latentwärmespeicher für den straßengebundenen Transport von Abwärme (MALATrans): Laufzeit: 01.07.2013 bis 31.12.2016 (Abschlussbericht)," Bayreuth, 2017.

First Name		Family Name
Mohammed		Farid
Institution		
University of Auckland		
Address		
Street		
University of Auckland, Department of Chemical and Materials Engineering, Grafton		
4-6 Park Ave, Auckland, New Zealand		
Zip-Code	City	
	Auckland	
Country		
New Zealand		
email		Telephone
m.farid@auckland.ac.nz		+6421812678

Material Data/Information		Date: 19/05/2018
Material Designation		
PCM (		Composite material(s)
1. Any PCM		1. metal coated microencapsulated PCM
2.		2.
3.		3.

Data for PCM or composite

Melting Temperature [°C]	Minimum Temperature [°C]
Storage Capacity [kJ/kg]	Maximum Temperature [°C]
Density [kg/l]	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h)
Supercooling [K]	Material compatibility
Technology readiness levels (TRL)	Additional Parameter

Metal Coated PCM Microcapsules
(surface activation by dopamine followed by silver coating

SEM photos and EDX patterns of (a) RT21 microcapsules and (b) RT21-PDA/Ag (Al Shannaq and Farid (2016))

If possible, please insert DSC-curve or other characteristic graph that shows the temperature dependency

Please insert an image/p

Thermal Conductivity Enhancement

Measured apparent thermal conductivity versus mean diameter of the PCM microcapsules at different silver nitrate concentration used in the coating process (Al Shannaq and Farid (2016))

hoto

Target Applications (up to 4 most relevant)
1. Cooling of electronic devises using slurry of microencapsulated PCM
2.
3.
4.
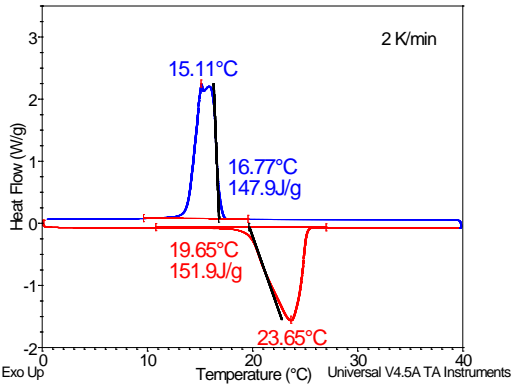
Task 58 / Annex 33 Subtask 2P: Summary of Work

Comments
References

First Name		Family Name	
Samer		Kahwaji	

Institution			
Dalhousie University – Department of Chemistry- Group of Prof. Mary Anne White			

Address

Street			
6274 Coburg road			

Zip-Code	City		
B3H 4R2	Halifax		

Country			
Canada			

email	Telephone	
sam@dal.ca	1-902-494-6538	

Material Data/Information	Date: May 22, 2018

Material Designation

Eutectic mixture of fatty acids, new melting point (see Ref. [1])

PCM	Composite material(s)
1. Dodecanoic acid (98% pure), 68 mass %	1.
2. Tetradecanoic acid (98% pure), 32 mass %	2.
3.	3.

Data for PCM or composite

Melting Temperature [°C]	Minimum Temperature [°C]
33.6 ±1.5 °C (onset)	0

Storage Capacity [kJ/kg]	Maximum Temperature [°C]
160 ± 16 kJ/kg	70

Density [kg/l]	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h)
0.865 (liquid phase)	Mixture not cycled. Individual fatty acids tested for 3000 cycles with no significant loss in enthalpy (see Ref. [2])

Supercooling [K]	Material compatibility
0.7 (onset points)	Compatible with stainless steel and aluminum (see Ref. [2])

Technology readiness levels (TRL)	Additional Parameter
	Heat capacity (C_p) of solid = 1.95 J/g K (at 10 °C), C_p of liquid = 2.21 J/g K (at 50 °C).

If possible, please insert DSC-curve or other characteristic graph that shows the temperature dependency	Please insert an image/photo

DSC curve showing Heat Flow (W/g) vs Temperature (°C) at 2 K/min. Peaks labeled: 32.07°C, 32.87°C 153.5J/g, 33.58°C 156.2J/g, 35.11°C. Exo Up. Universal V4.5A TA Instruments.

Target Applications (up to 4 most relevant)

1. Passive cooling of electronics and batteries.
2. Thermal energy storage in solar thermal collectors.

Comments

Individual fatty acid PCMs with melting temperature around 34 °C are not available, so this mixture makes this temperature accessible. The specified melting temperature and storage capacity are determined from averages of multiple samples and measurements at 2 K/min and 10 K/min, respectively.

References

[1] S. Kahwaji, M.A. White, Thermochim. Acta. 660 (2018) 94–100.

[2] S. Kahwaji, M.B. Johnson, A.C. Kheirabadi, D. Groulx, M.A. White, Sol. Energy Mater. Sol. Cells. 167 (2017) 109–120.
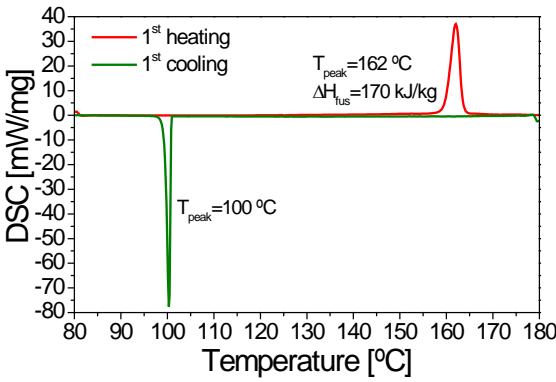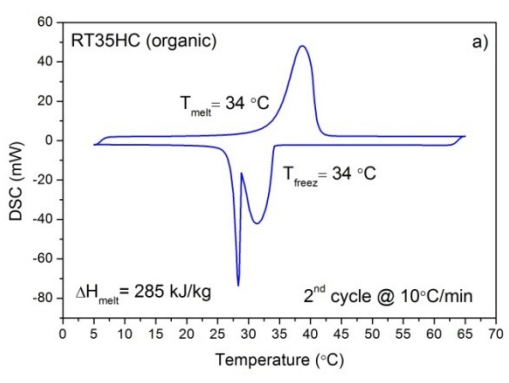
First Name		Family Name	
Samer		Kahwaji	

Institution	
Dalhousie University – Department of Chemistry- Group of Prof. Mary Anne White	

Address	

Street		
6274 Coburg road		

Zip-Code	City	
B3H 4R2	Halifax	

Country	
Canada	

email	Telephone
sam@dal.ca	1-902-494-6538

Material Data/Information	Date: May 22, 2018

Material Designation	
Eutectic mixture of fatty acids, new melting point (see Ref. [1])	

PCM	Composite material(s)
1. Decanoic acid (99% pure), 78 mass %	1.
2. Tetradecanoic acid (98% pure), 22 mass %	2.
3.	3.

Data for PCM or composite	

Melting Temperature [°C]	Minimum Temperature [°C]
20.5±1.5 °C (onset)	0

Storage Capacity [kJ/kg]	Maximum Temperature [°C]
153 ± 15 kJ/kg	70

Density [kg/l]	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h)
0.874 (liquid phase)	3000 cycles, no significant loss in enthalpy

Supercooling [K]	Material compatibility
3.6 (onset points)	Compatible with stainless steel and aluminum (see Ref. [2])

Technology readiness levels (TRL)	Additional Parameter
	See Ref. [1] for heat capacity, thermal conductivity and thermal diffusivity.

If possible, please insert DSC-curve or other characteristic graph that shows the temperature dependency	Please insert an image/photo

If possible, please insert DSC-curve or other characteristic graph that shows the temperature dependency

2 K/min

15.11°C

16.77°C
147.9J/g

19.65°C
151.9J/g

23.65°C

Heat Flow (W/g)
Temperature (°C)
Exo Up
Universal V4.5A TA Instruments

Target Applications (up to 4 most relevant)

1. Passive cooling of buildings / integration in building materials.
2. Thermal energy storage in solar thermal collectors.

Comments

Individual fatty acid PCMs with melting temperature around 20 °C are not available, so this mixture makes this temperature accessible. Purity of mixed fatty acids may affect the exact composition and melting point of the eutectic. The specified melting temperature and storage capacity are determined from averages of multiple samples and measurements.

Task 58 / Annex 33 Subtask 2P: Summary of Work

References
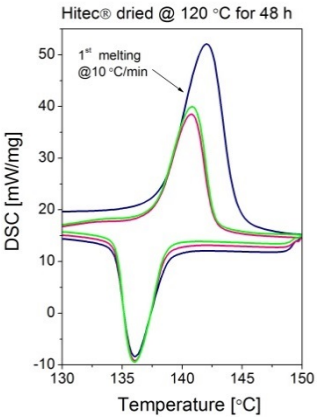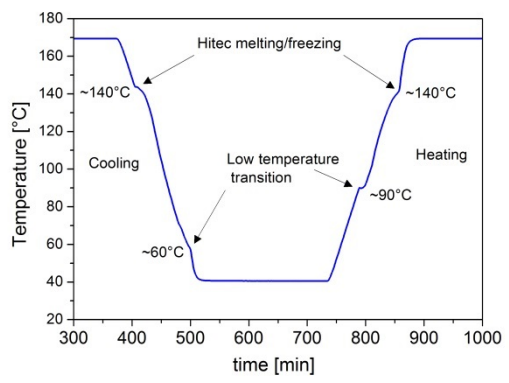
[1] S. Kahwaji, M.B. Johnson, A.C. Kheirabadi, D. Groulx, M.A. White, Appl. Energy. 168 (2016) 457–464.

[2] S. Kahwaji, M.B. Johnson, A.C. Kheirabadi, D. Groulx, M.A. White, Sol. Energy Mater. Sol. Cells. 167 (2017) 109–120.
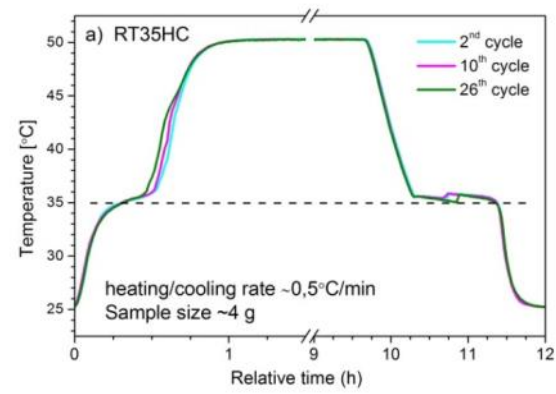
[3] S. Kahwaji, M.A. White, Thermochim. Acta. 660 (2018) 94–100.

First Name		Family Name
Rocío		Bayón
Institution		
CIEMAT-PSA		
Address		
Street		
Av. Complutense 40		
Zip-Code	City	
28040	Madrid	
Country		
Spain		
email		Telephone
rocio.bayon@ciemat.es		+34913466048
Material Data/Information		Date:
Material Designation		
Salicylic acid		
PCM (s)		Composite material(s)
1. Salicylic acid		1.
Data for PCM or composite		

Melting Temperature [°C] 162 ºC	Minimum Temperature [°C] -
Storage Capacity [kJ/kg] 199 kJ/kg	Maximum Temperature [°C] -
Density [kg/l] 	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h) -
Supercooling [K] In DSC: 70 K	Material compatibility -
Technology readiness levels (TRL) -	Additional Parameter

Sample appearance upon heating/cooling

Target Applications (up to 4 most relevant)

1. Not suitable as storage medium

Comments

- Strong supercooling
- White vapors evolve from sample upon melting
- White needles deposit back on sample surface after cooling

References

- R. Bayón, E. Rojas, Characterization of organic PCMs for medium temperature storage, in: A Méndez-Vilas (Ed.), Materials and Technologies for Energy Efficiency, Brown Walker Press, Boca Ratón, Florida (US), 2015, pp: 157–161.

Task 58 / Annex 33 Subtask 2P: Summary of Work

First Name		Family Name	
Rocío		Bayón	
Institution			
CIEMAT-PSA			
Address			
Street			
Av. Complutense 40			
Zip-Code	City		
28040	Madrid		
Country			
Spain			
email		Telephone	
rocio.bayon@ciemat.es		+34913466048	
Material Data/Information		Date:	
Material Designation			
RT35HC from Rubitherm®			
PCM (s)		Composite material(s)	
1. commercial organic mixture		1.	
Data for PCM or composite			
Melting Temperature [°C]	Minimum Temperature [°C]		
35 ºC	70 ºC (given by manufacturer)		
Storage Capacity [kJ/kg]	Maximum Temperature [°C]		
240 kJ/kg (manufacturer)	-		
285 kJ/kg (measured in DSC)			
Density [kg/l]	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h)		
	-		
Supercooling [K]	Material compatibility		
-	-		
Technology readiness levels (TRL)	Additional Parameter		
10 (commercial material)			

Daily heating/cooling cycles in oven under air (T-history)

Target Applications (up to 4 most relevant)
1. Refrigeration
2. Dry cooling

Comments
- No supercooling is observed
- Sample behavior remains constant after 26 daily heating/cooling cycles
- Melting/freezing plateau is observed in T-history curve even when temperature interval is $T_{melt} \pm 2 ºC$.

References

- R. Bayón, M. Biencinto, E. Rojas, N. Uranga. STUDY OF HYBRID DRY COOLING SYSTEMS FOR STE PLANTS BASED ON LATENT STORAGE. To be presented at ISEC conference in Graz, October 2018.

Task 58 / Annex 33 Subtask 2P: Summary of Work

First Name		Family Name	
Rocío		Bayón	

Institution			
CIEMAT-PSA			

Address			

Street			
Av. Complutense 40			

Zip-Code		City	
28040		Madrid	

Country			
Spain			

email		Telephone	
rocio.bayon@ciemat.es		+34913466048	

Material Data/Information		Date:	

Material Designation			
HITEC® commercial eutectic mixture			

PCM (s)		Composite material(s)	
1. $NaNO_3$:7 % w		1.	
2. KNO_3 :53 % w			
3. $NaNO_2$:40 % w			

Data for PCM or composite			
Melting Temperature [°C]		Minimum Temperature [°C]	
142 ºC			
Storage Capacity [kJ/kg]		Maximum Temperature [°C]	
83 kJ/kg (literature)		535 ºC	
50 kJ/kg (measured in DSC)			
Density [kg/l]		Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h)	
		-	
Supercooling [K]		Material compatibility	
-		-	
Technology readiness levels (TRL)		Additional Parameter	
10 (commercial material)			

DSC cycles

Heating/cooling cycle in oven under air (T-history): solid-liquid and solid-solid transition

Target Applications (up to 4 most relevant)
3.   Medium temperature storage although storage capacity is not very high
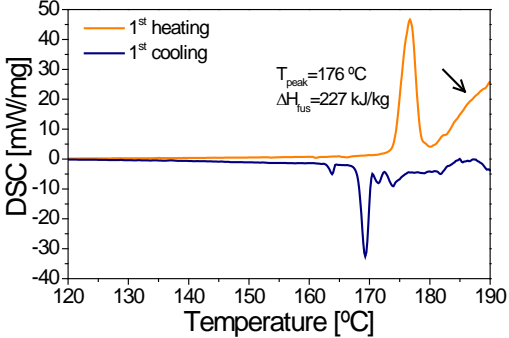
Comments
- Chemical stability after 50-60 daily cycles under air, N_2 and Ar
- No increase in nitrate percentage is observed
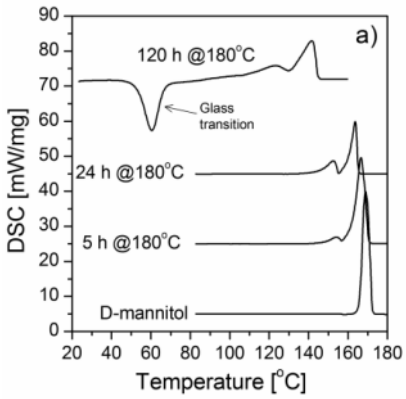
References
- M.M. Rodríguez-García, E. Rojas, R. Bayón, Test campaign and performance evaluation of a spiral latent storage module with Hitec® as PCM, Solar Heating and Cooling Conference 2017, Abu Dhabi, November 2017. Accepted for AIP Conference proceedings

Task 58 / Annex 33 Subtask 2P: Summary of Work

Questionnaire Phase Change Materials ECES Annex 33 / SHC Task 58
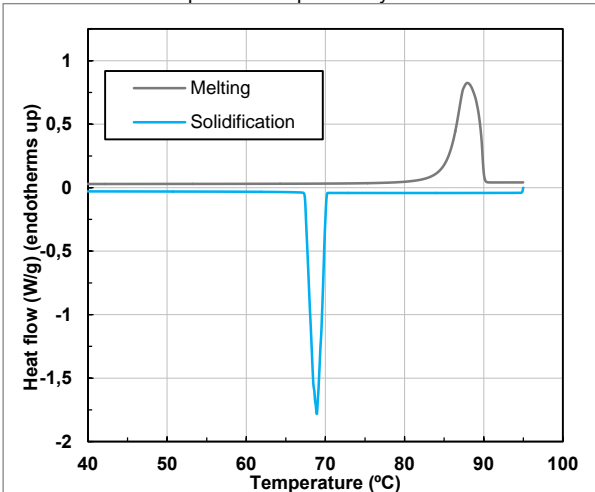
First Name	Family Name
Rocío	Bayón

Institution
CIEMAT-PSA

Address

Street
Av. Complutense 40

Zip-Code	City
28040	Madrid

Country
Spain

email	Telephone
rocio.bayon@ciemat.es	+34913466048

Material Data/Information	Date:

Material Designation
Hidroquinone

PCM (s)	Composite material(s)
1. Hidroquinone	1.

Data for PCM or composite

Melting Temperature [°C]	Minimum Temperature [°C]
173 ºC	-
Storage Capacity [kJ/kg]	Maximum Temperature [°C]
192-278 kJ/kg	-
Density [kg/l]	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h)
	-
Supercooling [K]	Material compatibility
-	-
Technology readiness levels (TRL)	Additional Parameter
-	

DSC scan showing degradation upon melting

$T_{peak}=176\ ºC$
$\Delta H_{fus}=227\ kJ/kg$

Hydroquinone sample appearance upon heating/cooling

Target Applications (up to 4 most relevant)
1. Not suitable as storage medium

Comments
- White vapors evolve from sample upon melting
- White needles deposit back on sample surface after cooling
- Bulk sample browning

References
- R. Bayón, E. Rojas, Characterization of organic PCMs for medium temperature storage, in: A Méndez-Vilas (Ed.), Materials and Technologies for Energy Efficiency, Brown Walker Press, Boca Ratón, Florida (US), 2015, pp: 157–161.

Send to stefan.gschwander@ise.fraunhofer.de

First Name		Family Name	
Rocío		Bayón	

Institution			
CIEMAT-PSA			

Address

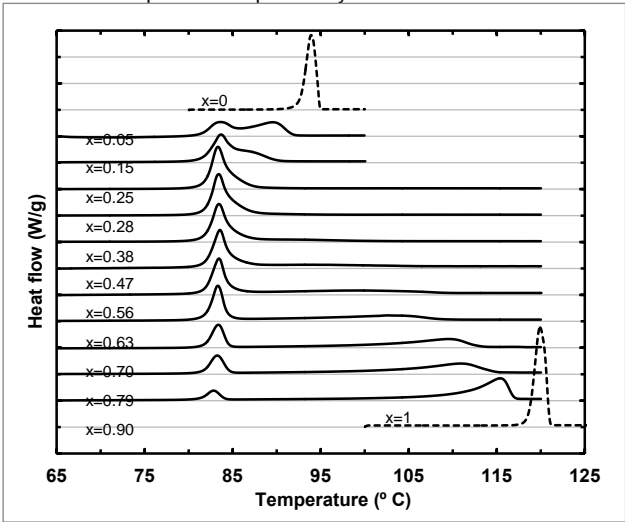| Street			
Av. Complutense 40			

Zip-Code	City		
28040	Madrid		

Country			
Spain			

email		Telephone	
rocio.bayon@ciemat.es		+34913466048	

Material Data/Information		Date:	

Material Designation			
D-mannitol			

PCM (s)		Composite material(s)	
1. D-mannitol		1.	

Data for PCM or composite

Melting Temperature [°C]		Minimum Temperature [°C]	
165 ºC		-	

Storage Capacity [kJ/kg]		Maximum Temperature [°C]	
246-338 kJ/kg		-	

Density [kg/l]		Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h)	
		-	

Supercooling [K]		Material compatibility	
In DSC: ~56 K		-	
In T-history: ~30 K			

Technology readiness levels (TRL)		Additional Parameter	
-			

DSC scan variation after stability tests

D-mannitol after 166 h melted under air @180 ºC

Target Applications (up to 4 most relevant)

1. Not suitable as storage medium

Comments
- This material degrades very quickly even under inert atmosphere (N$_2$, Ar)
- It undergoes caramelization even under O$_2$-free atmosphere.
-

References
- R. Bayón, E. Rojas, Feasibility study of D-mannitol as phase change material for thermal storage, AIMS Energy 5 (3) (2017) 404–424. https://doi.org/10.3934/energy.2017.3.404.
- M.M. Rodríguez-García, R. Bayón, E. Rojas, Stability of D-mannitol upon melting/freezing cycles under controlled inert atmosphere, Energy Procedia 91 (2016) 218–225. https://doi.org/10.1016/j.egypro.2016.06.207.

- R. Bayón, E. Rojas, Characterization of organic PCMs for medium temperature storage, in: A Méndez-Vilas (Ed.), Materials and Technologies for Energy Efficiency, Brown Walker Press, Boca Ratón, Florida (US), 2015, pp: 157–161.

First Name		Family Name	
Gonzalo		Diarce	
Institution			
University of the Basque Country (UPV/ EHU)			
Address			
Street			
Rafael Moreno Pitxitxi 2			
Zip-Code	City		
48012	*Bilbao*		
Country			
Spain			
email		Telephone	
gonzalo.diarce@ehu.es		*+34946014952*	
Material Data/Information		Date:	
Material Designation			
Eutectic mixture of Urea and Sodium Nitrate; phase diagram, thermal properties, crystallization behaviour, degradation, influence of water uptake			
PCM (		Composite material(s)	
1. Urea (71 % w/w) – NaNO$_3$ (29 % w/w) (eutectic mixture) 2. 3.		1. 2. 3.	

Data for PCM or composite

Melting Temperature [°C]	Minimum Temperature [°C]
85 ºC (onset)	n.a.
Storage Capacity [kJ/kg] 250 kJ/kg (60 to 95 ºC) 172 kJ/kg (melting latent heat)	Maximum Temperature [°C] n.a.
Density [kg/l] 1.48 solid / 1.42 liquid	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h) - DSC: Crucibles closed in air. 200 cycles. Reduction of 1.2 % of the original melting enthalpy. - Thermal stability of larger samples is currently under study. The results suggest that there is a significant reduction on the enthalpy in these larger samples, caused by a phase segregation phenomenon that is coupled to the thermal degradation of the sample (formation of ammonia due to the urea decomposition).
Supercooling [K] DSC: around 20 ºC Larger samples: 3-5 ºC	Material compatibility Not checked. It may be corrosive in contact with some construction materials
Technology readiness levels (TRL) 2/3	Additional Parameter none
If possible, please insert DSC-curve or other characteristic graph that shows the temperature dependency	Please insert an image/photo

Task 58 / Annex 33 Subtask 2P: Summary of Work

DSC results (heat flow vs. temperature) for the eutectic composition (71.25 % (w/w) urea)

Target Applications (up to 4 most relevant)
1. TES systems for Heating and DHW
2. TES systems for low to medium temperature industrial residual heat
3.

Comments
We are currently focused on the thermal degradation of the material. However, it is a complex procedure, because Urea undergoes thermal decomposition above its melting point. This effect is kinetic and depends on the time that the PCM stays above the melting temperature. Thus, accelerated thermal cycling studies are not useful to study it. Besides, the mixture tends to segregate when it crystallizes. This is dependent on the sample size and shape, but the effect cannot be decoupled from the thermal decomposition. Other variables such as the gas surrounding the mixture and the moisture content can have also a significant effect. All these factors together become the degradation study really complex.

References

G. Diarce, E. Corro-Martínez, L. Quant, Á. Campos-Celador, A. García-Romero. The sodium nitrate–urea binary mixture as a phase change material for medium temperature thermal energy storage. Part I: Determination of the phase diagram and main thermal properties Solar Energy Materials and Solar Cells, 2016; 157, 1065 - 1075

G. Diarce, E. Corro-Martínez, Á. Campos-Celador, A. García-Romero, J.M. Sala. The sodium nitrate–urea eutectic binary mixture as a phase change material for medium temperature thermal energy storage. Part II: Accelerated thermal cycling test and water uptake behavior of the material Solar Energy Materials and Solar Cells, 2016; 157, 1076 - 1083
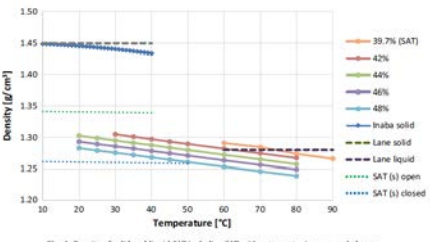
First Name		Family Name
Gonzalo		Diarce

Institution
University of the Basque Country (UPV/ EHU)

Address

Street
Rafael Moreno Pitxitxi 2

Zip-Code	City
48012	*Bilbao*

Country
Spain

email	Telephone
gonzalo.diarce@ehu.es	*+34946014952*

Material Data/Information	Date:

Material Designation
Eutectic mixtures of sugar alcohols; phase diagram, thermal properties, crystallization behaviour, degradation

PCM	Composite material(s)
1. Erythritol (21 % w/w) - Xylitol (79 % w/w) (eutectic mixture)	1.
2.	2.
3.	3.

Data for PCM or composite	
Melting Temperature [°C]	Minimum Temperature [°C]
82 ºC (onset)	n.a.
Storage Capacity [kJ/kg]	Maximum Temperature [°C]
250 kJ/kg (latent melting heat)	n.a.
Density [kg/l]	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h)
n.a.	n.a
Supercooling [K]	Material compatibility
Significant, combined with a slow crystallization rate	Compatible with common construction materials
Technology readiness levels (TRL)	Additional Parameter
2/3	none

If possible, please insert DSC-curve or other characteristic graph that shows the temperature dependency	Please insert an image/photo

DSC thermograms obtained for the erythritol–xylitol system

Target Applications (up to 4 most relevant)
1. TES systems for Heating and DHW
2. TES systems for low to medium temperature industrial residual heat
3.

Comments

So far, the studies have been focused on the thermal and crystallization behaviour. We have plans to investigate in the near future the thermal degradation of the mixture, as well as its potential application within a storage device that includes a crystallization triggering system.
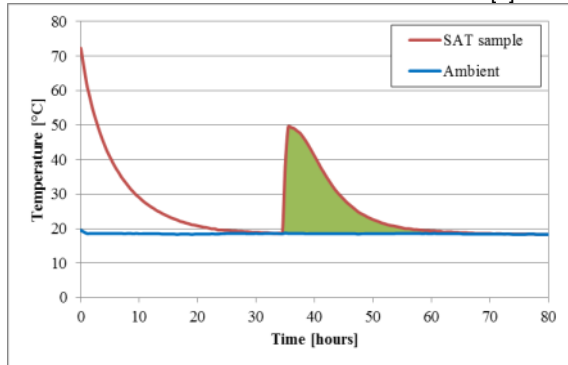
References

G. Diarce, I. Gandarias, A. Campos-Celador, A. García-Romero, J.M. Sala Eutectic mixtures of sugar alcohols for thermal energy storage in the 50-90 ºC temperature range *Solar Energy Materials and Solar Cells,* 2015; 134, 215 - 226

|		
First Name Gerald		Family Name Englmair
Institution Technical University of Denmark		
Address		
Street Nordvej Building 119		
Zip-Code 2800	City Kgs. Lyngby	
Country Denmark		
email gereng@byg.dtu.dk		Telephone
Material Data/Information		Date:
Material Designation		
PCM Sodium Acetate Trihydrate (SAT)		Composite material(s). **Examples listed. SAT with different weight contents of water, thickening agents, polymer materials can be found in** [1] 1. SAT with 1% (wt %) CMC (Carboxymethyl Cellulose) 2. SAT with 1% (wt %) water + 1% (wt %) EDTA (Disodium Ethylenediaminetetraacetic acid) 3. SAT with 2% (wt %) EDTA 4. SAT with 4% (wt %) water 5. SAT with 2% (wt %) HD 200 (Polymer material) 6. SAT with 0.5% (wt %) Xanthan Gum
Data for PCM or composite		
Melting Temperature [°C] 58		Minimum Temperature [°C] -15 °C (crystallization of liquid SAT) [3]
Storage Capacity [kJ/kg] [1] 1. 211 2. 216 3. 215 4. 194 5. 216 6. 214		Maximum Temperature [°C] ~100 °C for SAT composition except: 80 °C for compositions containing CMC (assuming atmospheric pressure conditions).
Density [kg/l] Depending on the state (liquid, solid, liquid supercooled), see reference [2] and diagram below Fig. 4. Density of solid and liquid SAT including SAT with extra water in supercooled state.		Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h) Stable with above mentioned compositions. Cyclic stability was proven in large containers (heat storage prototypes) up to ~30 cycles. Long-term investigations are ongoing.
Supercooling [K] Down to -15 °C with 73 K degree of supercooling [3]		Material compatibility
Technology readiness levels (TRL) 6-7 [4]		Additional Parameter Thermal conductivity 1. SAT with 1% CMC 0.57-0.65 W/mK 2. SAT with 0.5 % Xanthan Gum 0.5-0.65 W/mK For more composites, reference [5]

Task 58 / Annex 33 Subtask 2P: Summary of Work

If possible, please insert DSC-curve or other characteristic graph that shows the temperature dependency	Please insert an image/photo Bulk sample below: SAT with 1% (wt. %) CMC
Use heat loss method to measure the heat content [1] 	
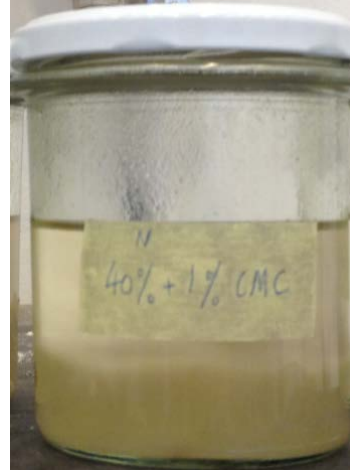
Target Applications (up to 4 most relevant)
1. Solar thermal heating system for DHW & SH
2. Domestic heating systems with heat pumps
3. Smart grid (increased demand flexibility of buildings)
4. Overall: stable supercooling for combined short – long term heat storage below 100 °C

Comments

References
[1]   W. Kong *et al.*, "Experimental investigations on heat content of supercooled sodium acetate trihydrate by a simple heat loss method," *Sol. Energy*, vol. 139, pp. 249–257, 2016.
[2]   M. Dannemand *et al.*, "Porosity and density measurements of sodium acetate trihydrate for thermal energy storage," *Appl. Therm. Eng.*, vol. 131, pp. 707–714, 2018.
[3]   G. Englmair *et al.*, "Crystallization by local cooling of supercooled sodium acetate trihydrate composites for long-term heat storage," *Energy Build.*, vol. 180, pp. 159–171, 2018.
[4]   G. Englmair, C. Moser, S. Furbo, M. Dannemand, and J. Fan, "Design and functionality of a segmented heat-storage prototype utilizing stable supercooling of sodium acetate trihydrate in a solar heating system," *Appl. Energy*, vol. 221, no. April, pp. 522–534, 2018.
[5]   M. Dannemand, J. B. Johansen, and S. Furbo, "Solidification behavior and thermal conductivity of bulk sodium acetate trihydrate composites with thickening agents and graphite," *Sol. Energy Mater. Sol. Cells*, vol. 145, pp. 287–295, Feb. 2016.
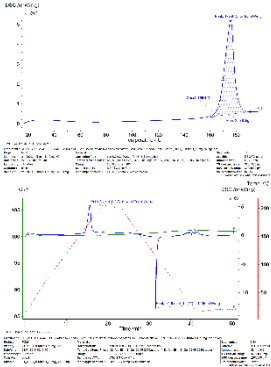
First Name		Family Name	
JUAN DE DIOS		CRUZ ELVIRA	

Institution	
INSTITUTO TECNOLOGICO DE OAXACA	

Address	

Street		
Calz. Tecnologico Esq. Ing. Victor Bravo Ahuja No. 125,Oaxaca de Juarez,Oax.		

Zip-Code	City	
68030	OAXACA	

Country	
MEXICO	

email	Telephone
juko_reto@hotmail.com	+5219512353381

Material Data/Information	Date:

Material Designation	
Composite Tepexil /Dodecanol	

PCM (	Composite material(s)
1.Dodecanol	1.Tepexil
2.	2.
3.	3.

Data for PCM or composite	

Melting Temperature [°C]	Minimum Temperature [°C]
24.5	21.4

Storage Capacity [kJ/kg]	Maximum Temperature [°C]
118.35	25.1

Density [kg/l]	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h)
1045	5

Supercooling [K]	Material compatibility

Technology readiness levels (TRL)	Additional Parameter
	TGA (125ºC stability)

If possible, please insert DSC-curve or other characteristic graph that shows the temperature dependency	Please insert an image/photo

Target Applications (up to 4 most relevant)

1. building applications
2. construction materials
3.
4.

Comments

 This work aims to evaluate the thermal properties and performance of a composite material dodecanol/tepexil for thermal comfort in housing.

First Name		Family Name
Thomas		Aigenbauer
Institution		
FH OÖ Forschungs & Entwicklungs GmbH		
Address		
Street		
Ringstraße 43a		
Zip-Code	City	
4600	Wels	
Country		
Austria		
email		Telephone
Thomas.aigenbauer@fh-wels.at		+43 50804 46919
Material Data/Information		Date: 19.09.2019

Material Designation

D-Mannitol 97+% (Alfa Aesar) and Dulcitol 97% (Alfa Aesar)

PCM (	Composite material(s)
1. D-Mannitol (70 %)	1.
2. Dulcitol (30%)	2.
3.	3.

Data for PCM or composite

Melting Temperature [°C]	Minimum Temperature [°C]
153	
Storage Capacity [kJ/kg]	Maximum Temperature [°C]
280	
Density [kg/l]	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h)
1,505	5000; no separation detected;
Supercooling [K]	Material compatibility
	Good with copper and aluminium
Technology readiness levels (TRL)	Additional Parameter
5	

If possible, please insert DSC-curve or other characteristic graph that shows the temperature dependency	Please insert an image/photo

Target Applications (up to 4 most relevant)

1. passive cooling for coating applications
2. mid-temperature industry processes

Comments
References

First Name		Family Name
Thomas		Aigenbauer

Institution
FH OÖ Forschungs & Entwicklungs GmbH

Address

Street
Ringstraße 43a

Zip-Code	City
4600	Wels

Country
Austria

email	Telephone
Thomas.aigenbauer@fh-wels.at	+43 50804 46919

Material Data/Information	Date: 19.09.2019

Material Designation
D-Mannitol 97+% (Alfa Aesar)

PCM (1. D-Mannitol 2. 3.	Composite material(s) 1. 2. 3.

Data for PCM or composite	
Melting Temperature [°C] 167	Minimum Temperature [°C]
Storage Capacity [kJ/kg] 270	Maximum Temperature [°C]
Density [kg/l] 1,52	Cycle Stability (how many thermal cycles tested, if possible reduction in kJ/kg·h) 5000; after 456h at 180°C → 208,2 kJ/kg (in copper tube)
Supercooling [K]	Material compatibility Good with copper and aluminium
Technology readiness levels (TRL) 5	Additional Parameter

If possible, please insert DSC-curve or other characteristic graph that shows the temperature dependency	Please insert an image/photo

Target Applications (up to 4 most relevant)
1. passive cooling for coating applications
2. mid-temperature industry processes
3.
4.

Comments
References