SOLAR HEATING & COOLING PROGRAMME
INTERNATIONAL ENERGY AGENCY

Compact TES material characterization techniques

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IEA SHC Solar Academy Webinar, 1 April 2025

Introduction

TES technologies and materials

- **Sensible Thermal Energy Storage** (STES): Enthalpy change ΔH due to heat capacity C and temperature gradient ΔT of a Sensible Heat Storage Material (SHSM)

- **Latent Thermal Energy Storage** (LTES): Enthalpy change ΔH_t in solid-solid or solid-liquid phase transitions of a Phase Change Material - PCM

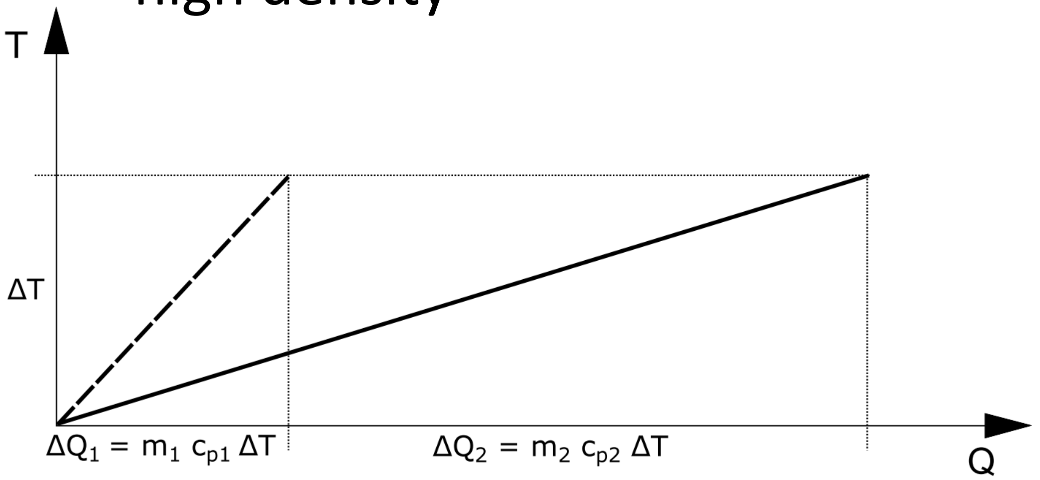
- **Thermochemical Energy Storage** (TCES): Enthalpy change due to physical sorption or a chemical reaction, respectively, of a ThermoChemical Material – TCM

For all three technologies it is essential to have a detailed knowledge of the thermophysical properties of the storage materials to predict possible storage capacities, energy densities and thermal power on material scale.

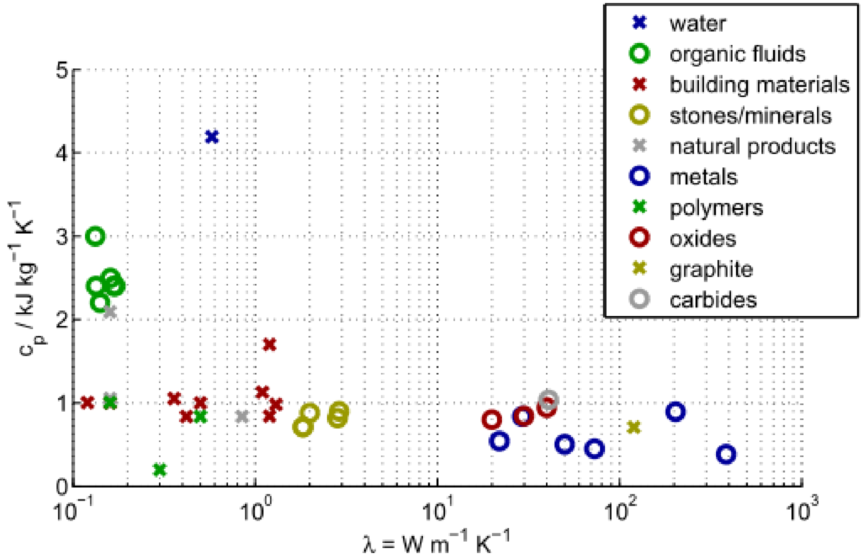
Source: Lager, D. (2017). *Evaluation of thermophysical properties for thermal energy storage materials - determining factors, prospects and limitations* [Dissertation, Technische Universität Wien]. reposiTUm. https://doi.org/10.34726/hss.2017.47756

Sensible Heat Storage

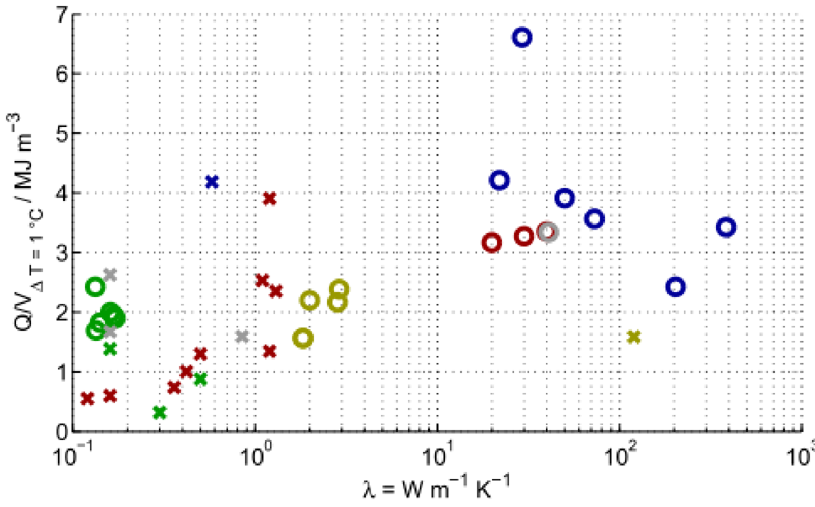
- Heat capacity and temperature difference determine the stored heat
- Material requirements:
  - high spec. Heat capacity c_p
  - high thermal conductivity λ
  - high density



Specific Heat Capacity vs. Thermal Conductivity



Vol. energy density vs. Thermal Conductivity



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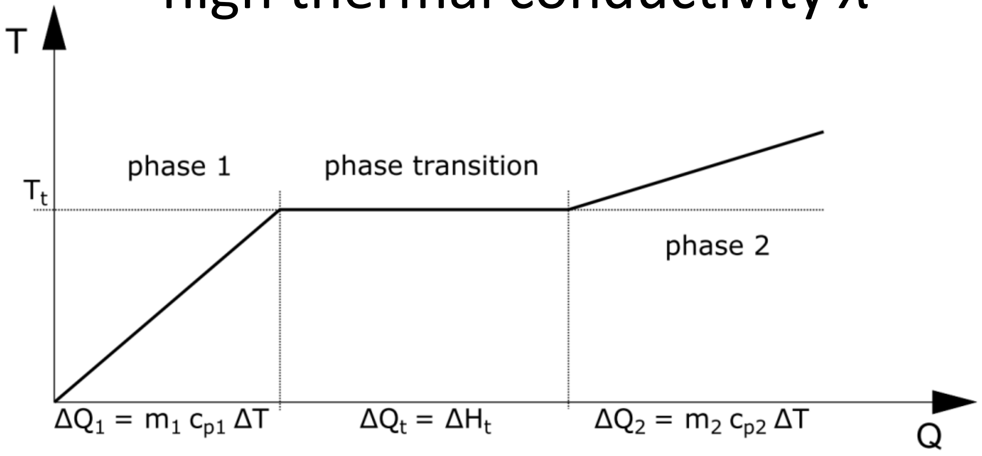
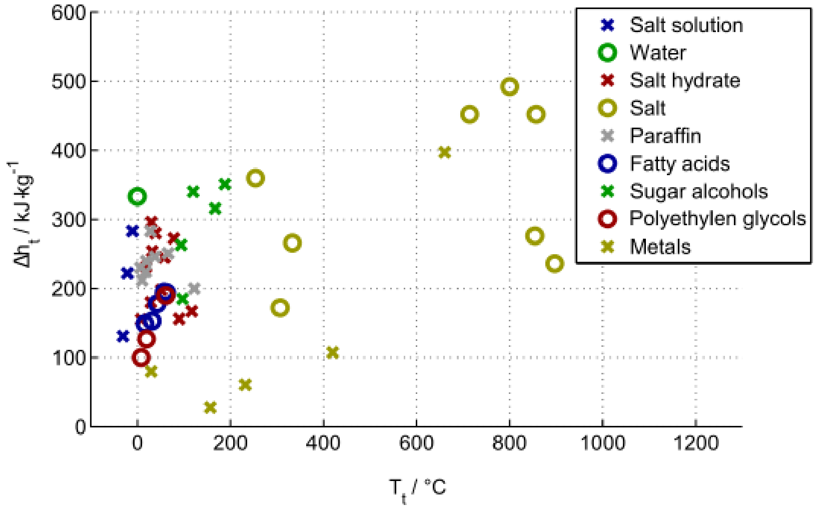
Latent Heat Storage

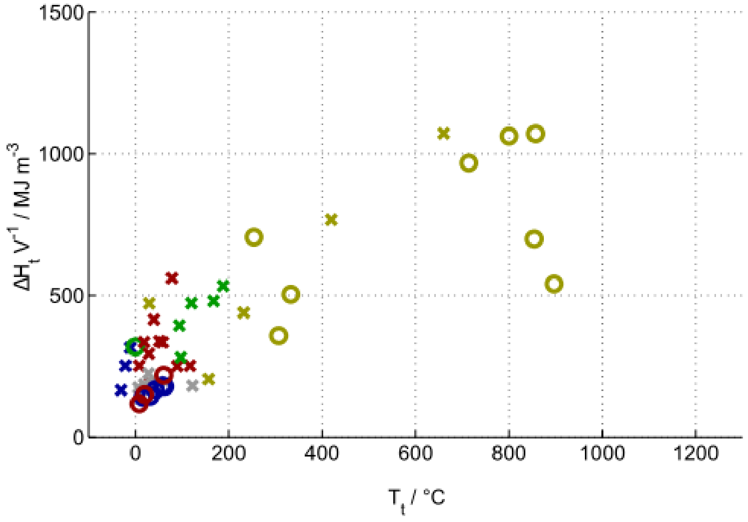
- Use of transition enthalpy in phase changes
- Material requirements:
  - phase change temperature *Tt*
  - high phase change enthalpy *ΔHt*
  - low subcooling
  - high thermal conductivity *λ*



spec. phase change enthalpy vs. Transition temperature



Vol. enthalpy change vs. Transition temperature



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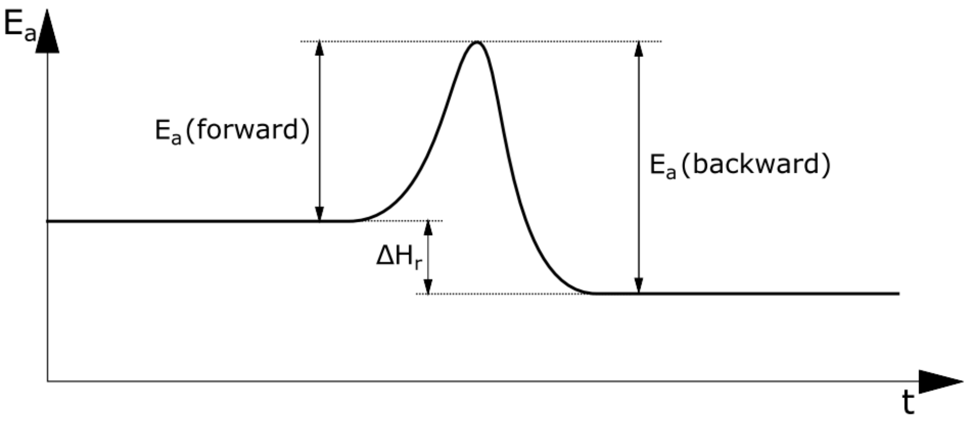
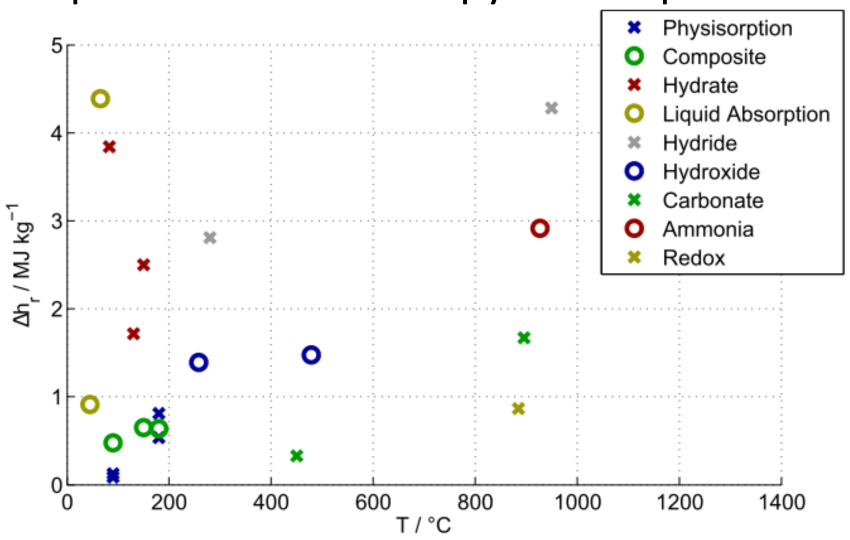
Thermochemical Heat Storage

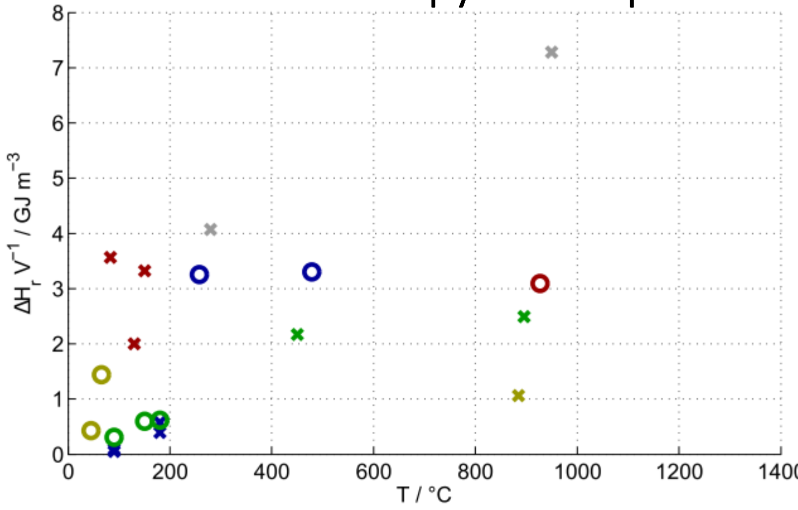
- Use of reaction or/and sorption enthalpy
- Material requirements:
  - high enthalpy of reaction *ΔHr*
  - fast reaction rates *r*
  - high thermal conductivity *λ*,
  - reversible and stable in cycles



spec. reaction enthalpy vs. temperature



Vol. reaction enthalpy vs. temperature



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Measurement Challenges

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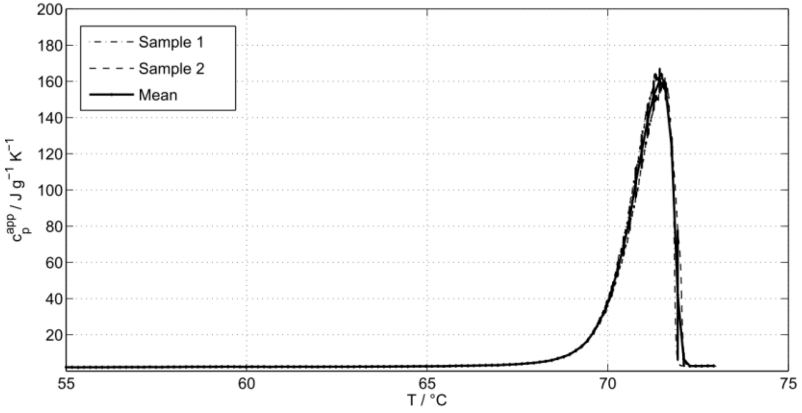
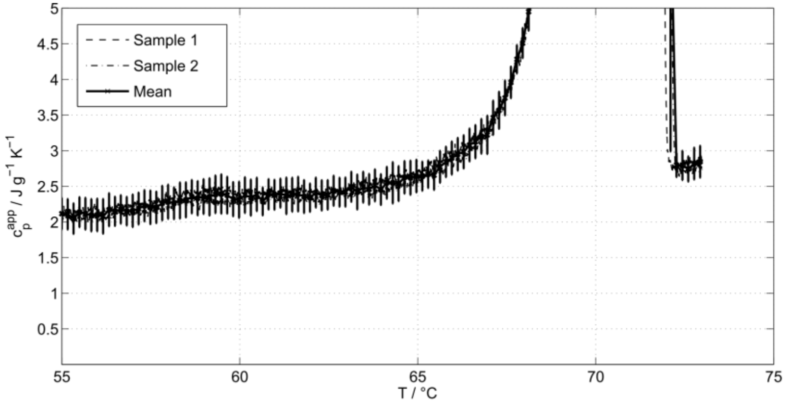
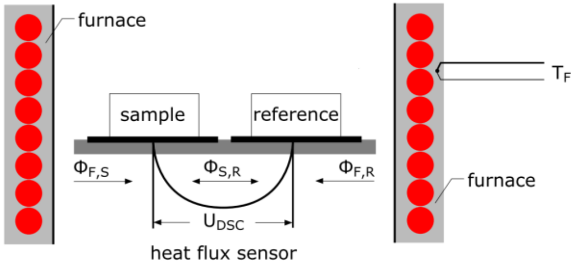
Measurement methods for PCM
Paraffin Wax / Calorimetry

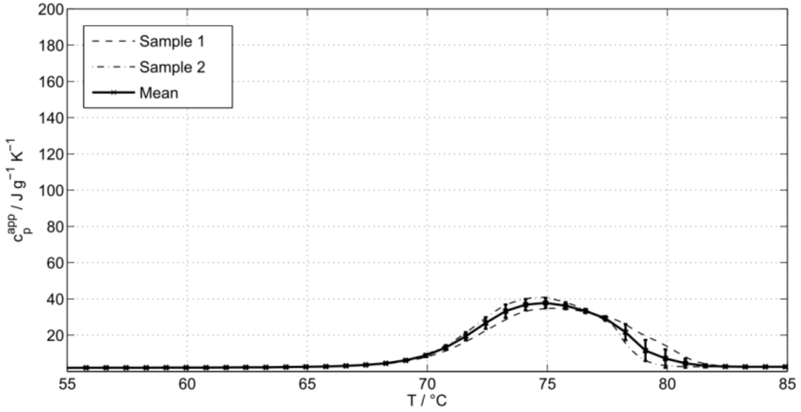


(A) Evaluated $c_p{}^{app}(T)$ with $\beta = 0.25\,\mathrm{K\,min^{-1}}$

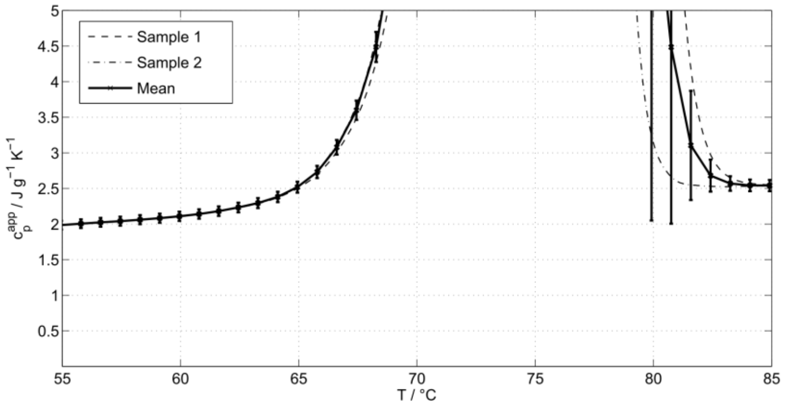


(A) Detailed view of $c_p{}^{app}(T)$ with $\beta = 0.25\,\mathrm{K\,min^{-1}}$

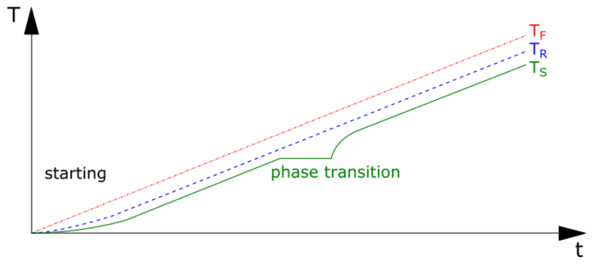


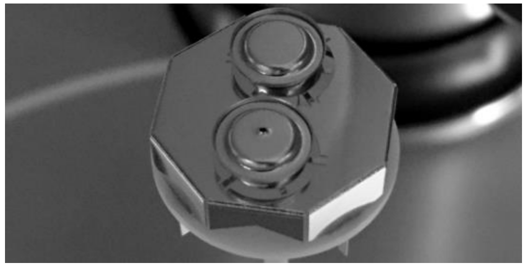


(B) Evaluated $c_p{}^{app}(T)$ with $\beta = 10\,\mathrm{K\,min^{-1}}$



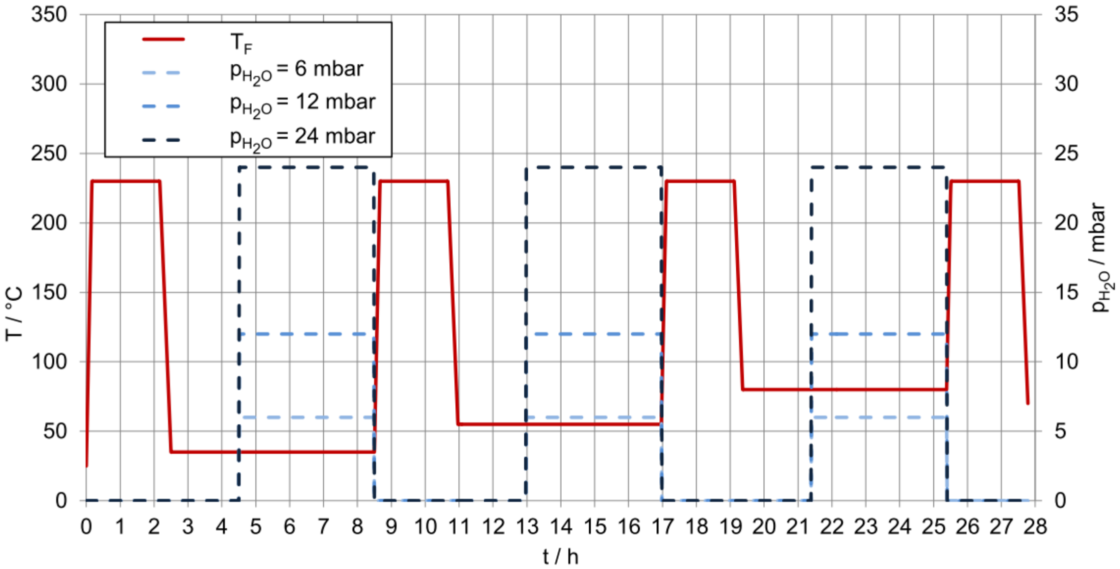
(B) Detailed view of $c_p{}^{app}(T)$ with $\beta = 10\,\mathrm{K\,min^{-1}}$





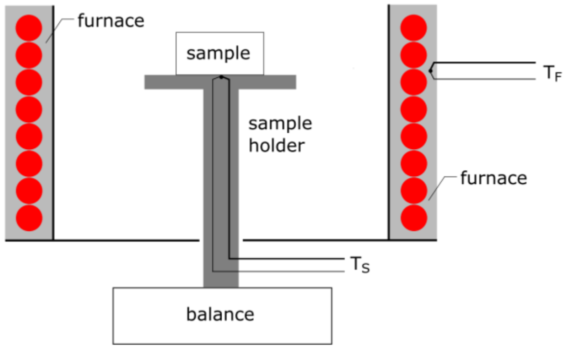
Measurement methods for TCM
Gravimetric Sorption Measurements

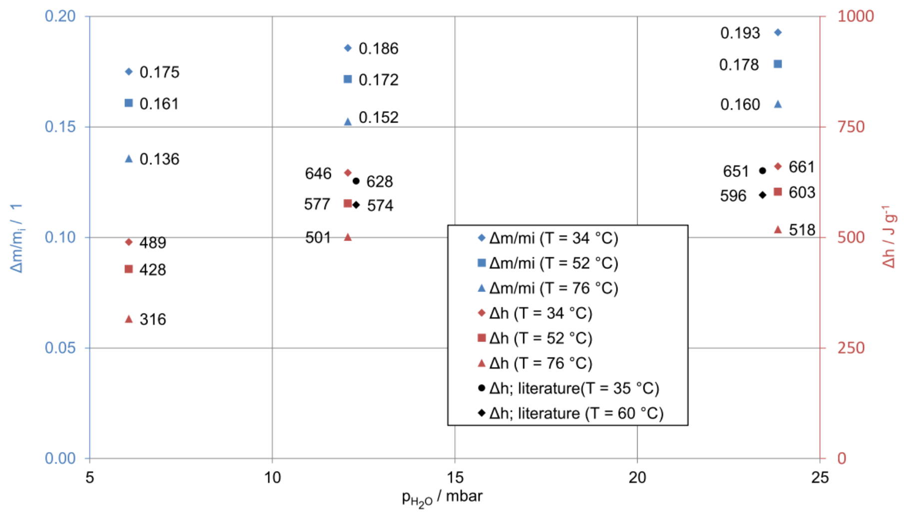
- Characterization of sorption isotherms under defined gas (water vapor) conditions
- Comparison of the measured data to existing literature using the example of a zeolites

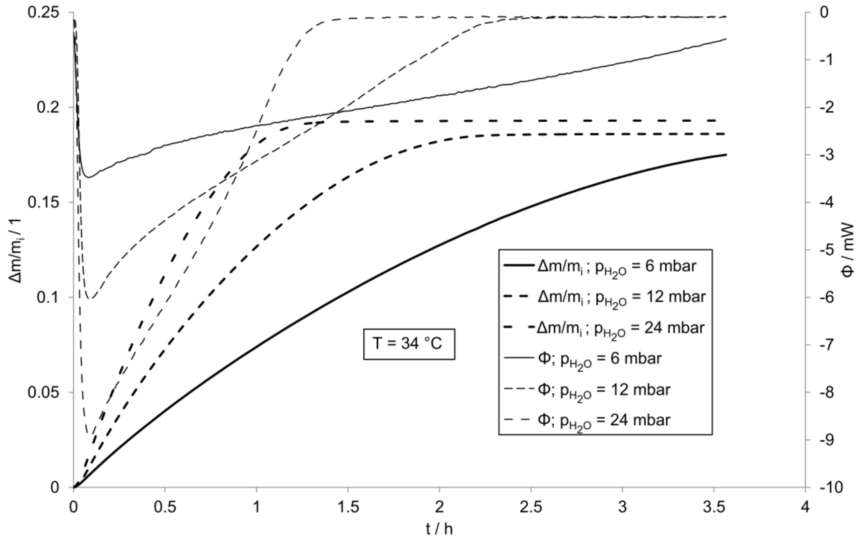


Measurement methods for TCM Gravimetric Sorption Measurements

- Results:
  - Water uptake (TGA)
  - Heat of adsorption (DSC)
  - Kinetics

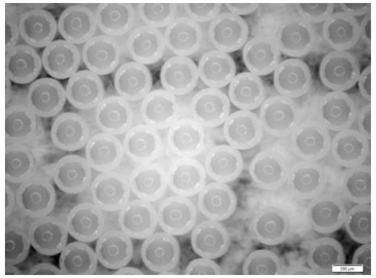


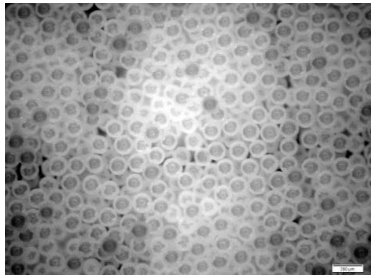


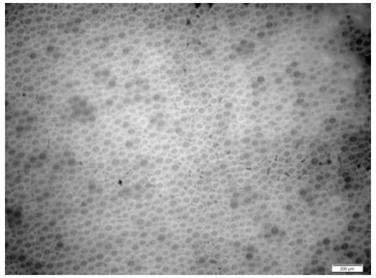


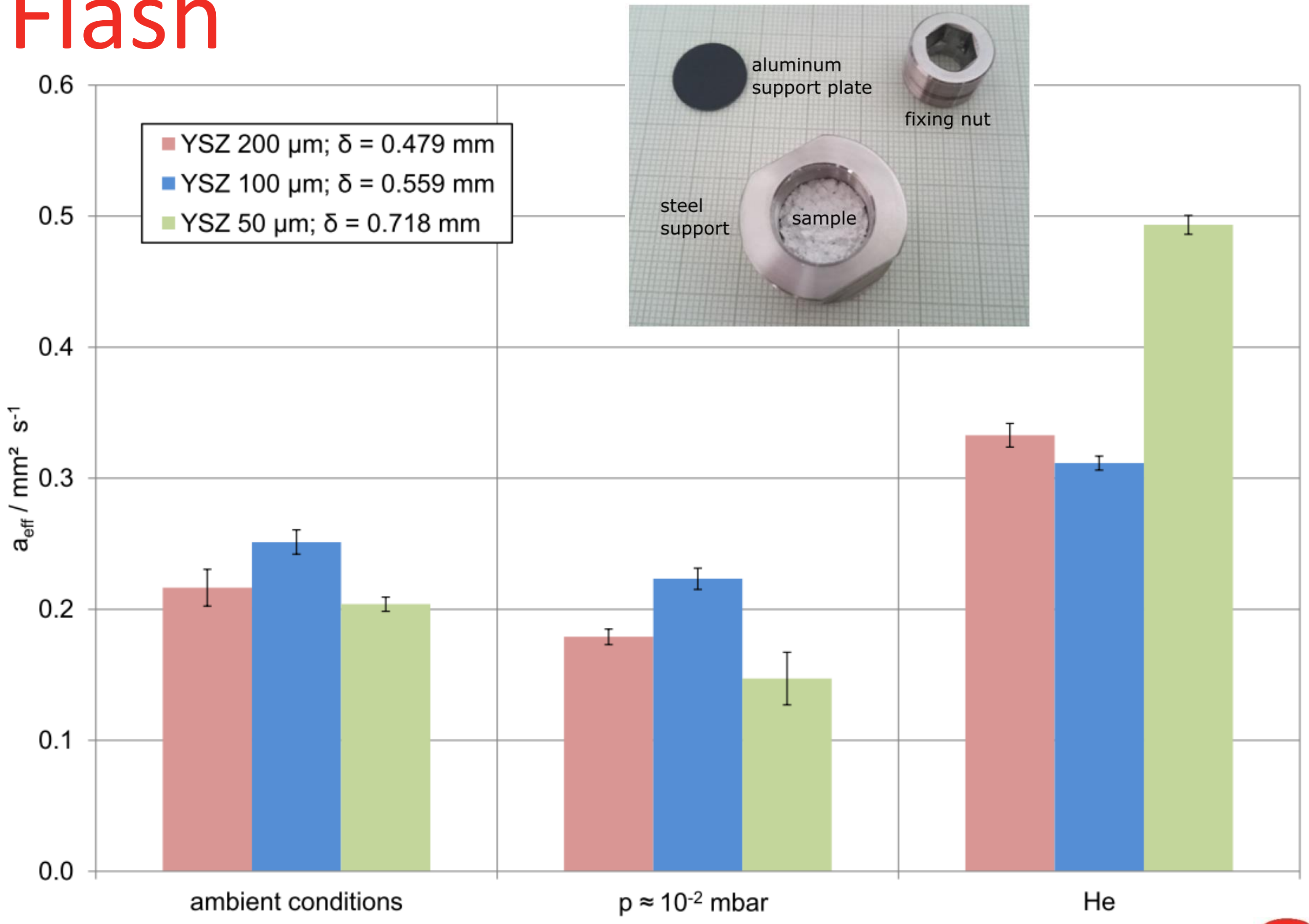
Measurement methods for TCM packed beds: Laser Flash

YSZ - Yttrium Stabilized Zirconia

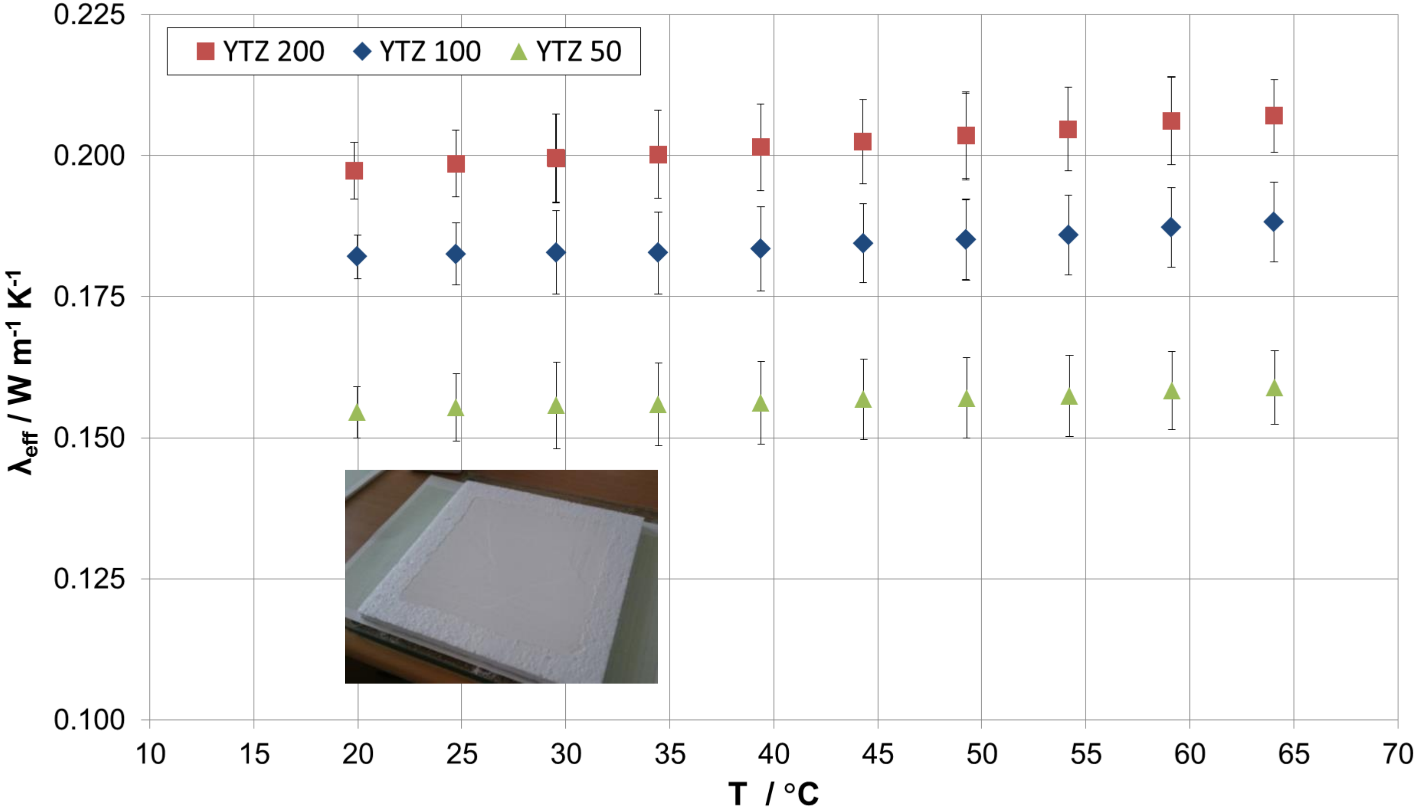


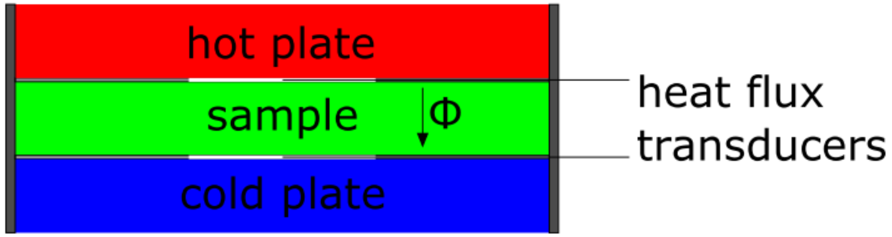






Measurement methods for TCM packed bed: Heat Flow Meter





$\rho_{B\ (YSZ\ 200)} = 4.021\ g/cm^3$

$\rho_{B\ (YSZ\ 100)} = 3.928\ g/cm^3$

$\rho_{B\ (YSZ\ 50)} = 3.819\ g/cm^3$

IEA SHC TASK 67 - Compact TES

Text and pictures from the final report of the IEA SHC TASK 67: https://task67.iea-shc.org/

IEA TASK Compact Thermal Energy Storage – Materials within Components within Systems

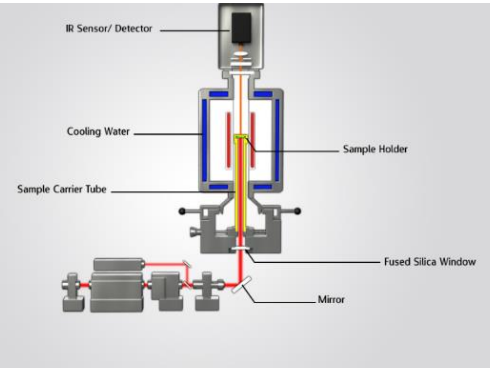
Motivation

...push forward the compact thermal energy storage (CTES) technology developments to accelerate the market introduction of these technologies through the **international collaboration of experts** from **materials research**, components development and system integration, and industry and research organizations.

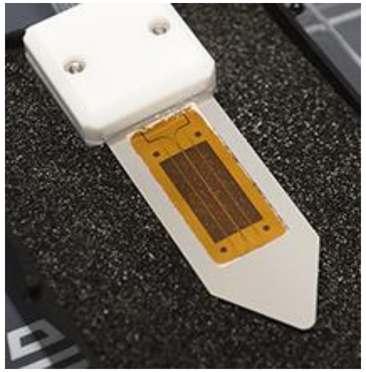
One of the main objectives:

- **characterize TES materials in a reliable and reproducible manner**

Thermal conductivity and diffusivity



- **Material:** Paraffin with a melting point between 53-58 °C
- **Methods:** 8 different transient methods and steady-state methods. Three main measuring techniques were used:
    - Laser Flash (LFA)
    - Hot Wire (HW)
    - Transient Plane Source (TPS)



Paraffin

(0)

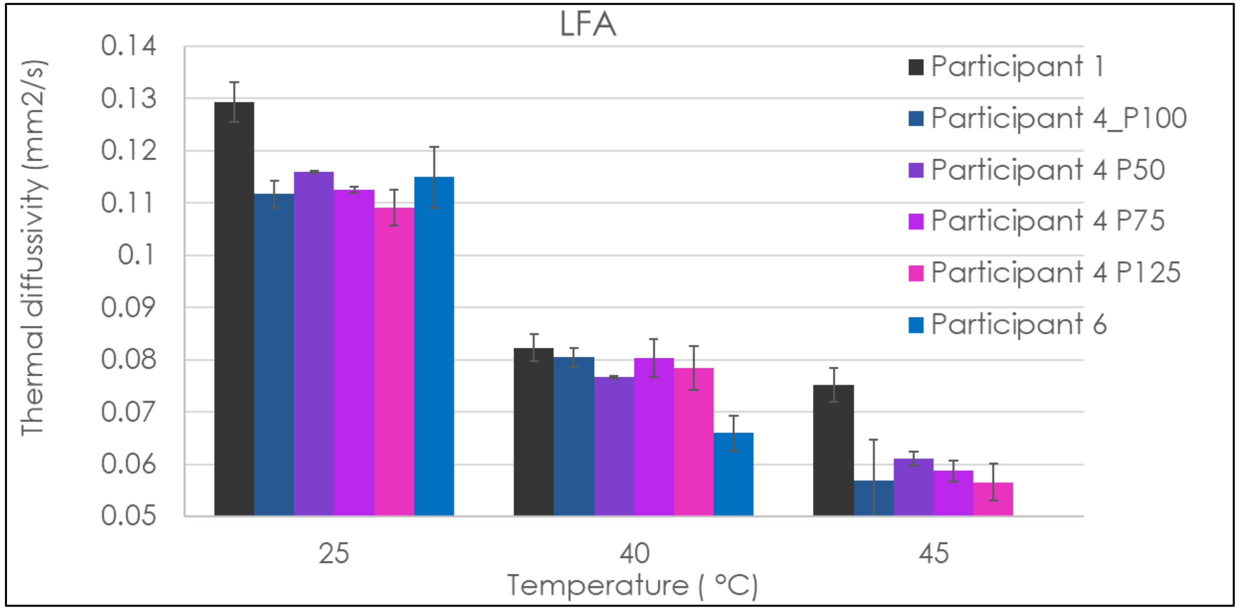
mp 53-58 °C (ASTM D 87)

Alle Fotos (1)

CAS-Nummer:	8002-74-2	EC-Nummer:	232-315-6
MDL-Nummer:	MFCD00132833	NACRES:	NA.23

Results thermal diffusivity / conductivity

Example for thermal diffusivity results (Laser Flash)

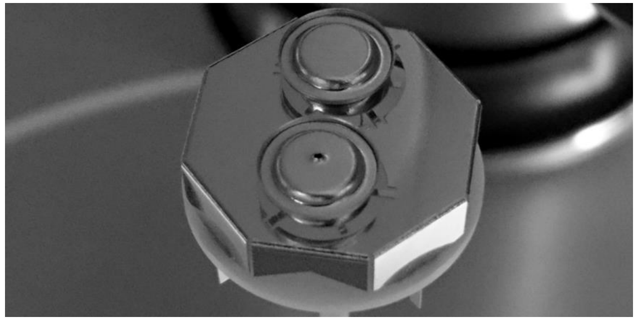


- equipment availability supposes a challenge to establish standardized measurement protocols. The procedures were split according to the available equipment into LFA, TPS, HW and other steady-state equipment.

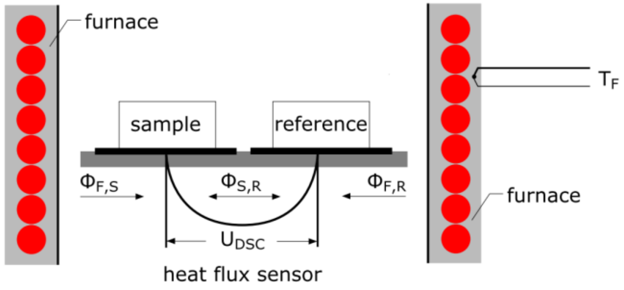
- Both experimental and systematic uncertainty have to be accounted for to compare the results.

Specific heat capacity of powdery materials - Calorimetry

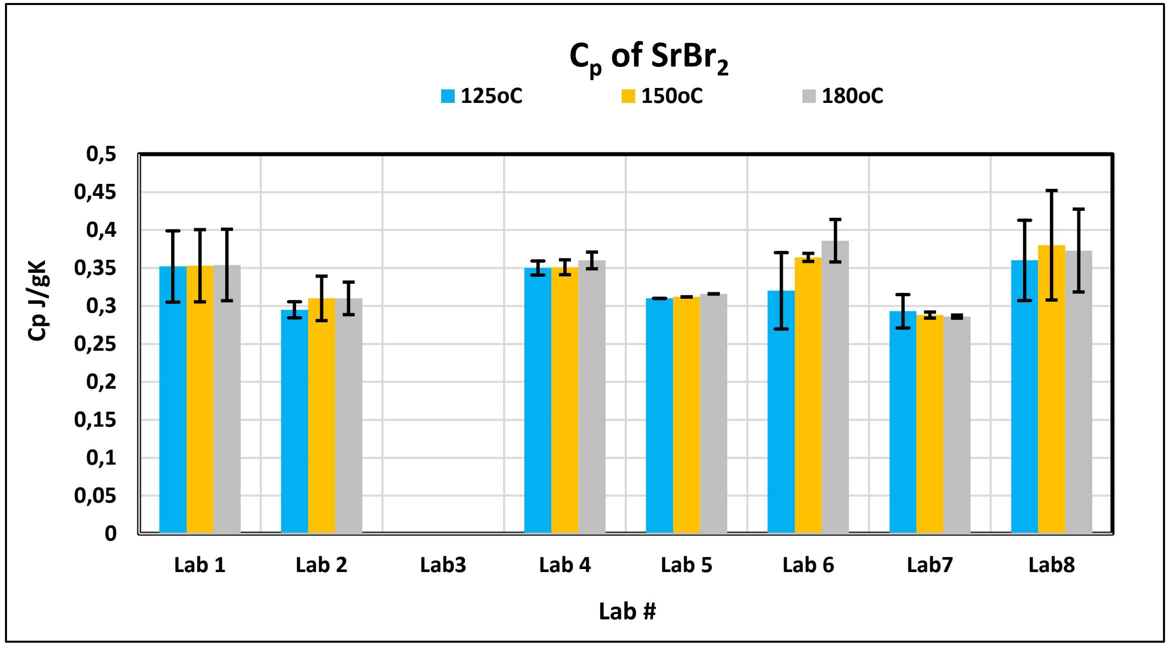
Material:
- Anhydrous strontium bromide ($SrBr_2$)
- Strontium bromide hexahydrate ($SrBr_2*6H_2O$)
- Synthetic zeolite Z-13X



Proposed Measuring Procedures:
- Instrument calibration (temperature, sensitivity)
- Baseline runs
- Reference material run
- Sample preparation
- Sample run and Cp evaluation



Results specific heat capacity of powdery materials



- Hydrate measurements showed strong deviations which may be connected to different hydration states in the measurement. Due to that the sample preparation and measurement procedure routines must be corrected.
- Especially the occurrence of water in the sample (hydrated or sorption samples) should be examined in more detail.

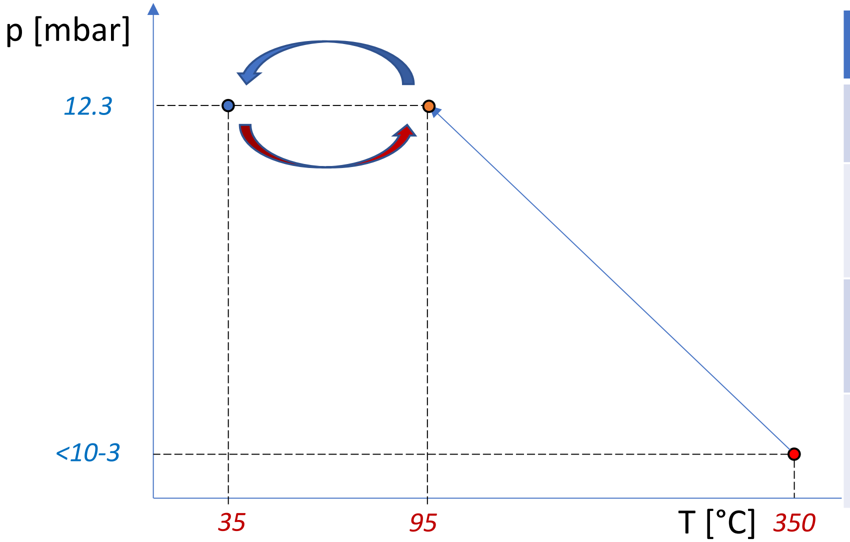
Enthalpy change due to sorption

Material: Zeolite 13X

Formula: Na86[(AlO2)86(SiO2)106] · xH2O

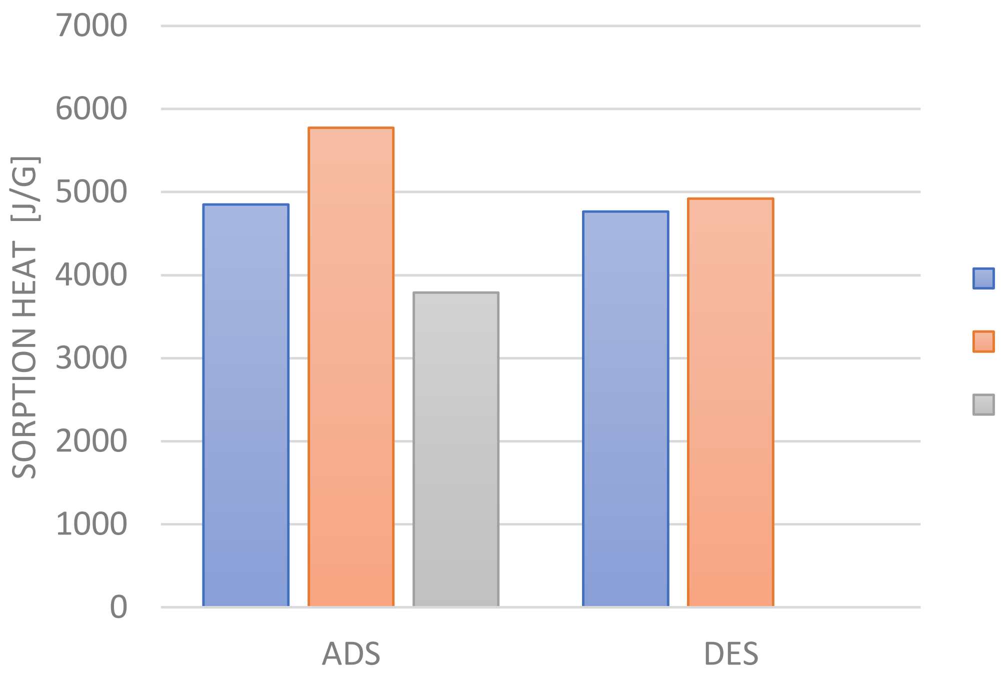
1.6 mm Pellets

Procedure:



Phase	Temperature [°C]	Pressure [mbar]	Duration	Heating/cooling rate	Crucible material
Desorption	350 °C	< 10-3	> 6 h (dry mass)		Aluminum (preferred)
Equilibrium	95 °C	12.3	Until stabilization		Aluminum (preferred)
Adsorption	95->35 °C	12.3	Until stabilization	5 K/min (same as the calibration)	Aluminum (preferred)
Desorption	35->95 °C	12.3	Until stabilization	5 K/min (same as the calibration)	Aluminum (preferred)

Results enthalpy change due to sorption



- Strong dependence on the performance of the different equipment available in the laboratories and the ability of these instruments to carefully follow the planned conditions.

- Possible future activities could be focused on enlarging the number of laboratories involved in the testing campaign, using different apparatuses under the proposed simplified protocol. Once the approach is validated, it can be extended to a wider set of conditions and different materials to reach a common approach to be considered by the scientific community

Density

Measured Materials:

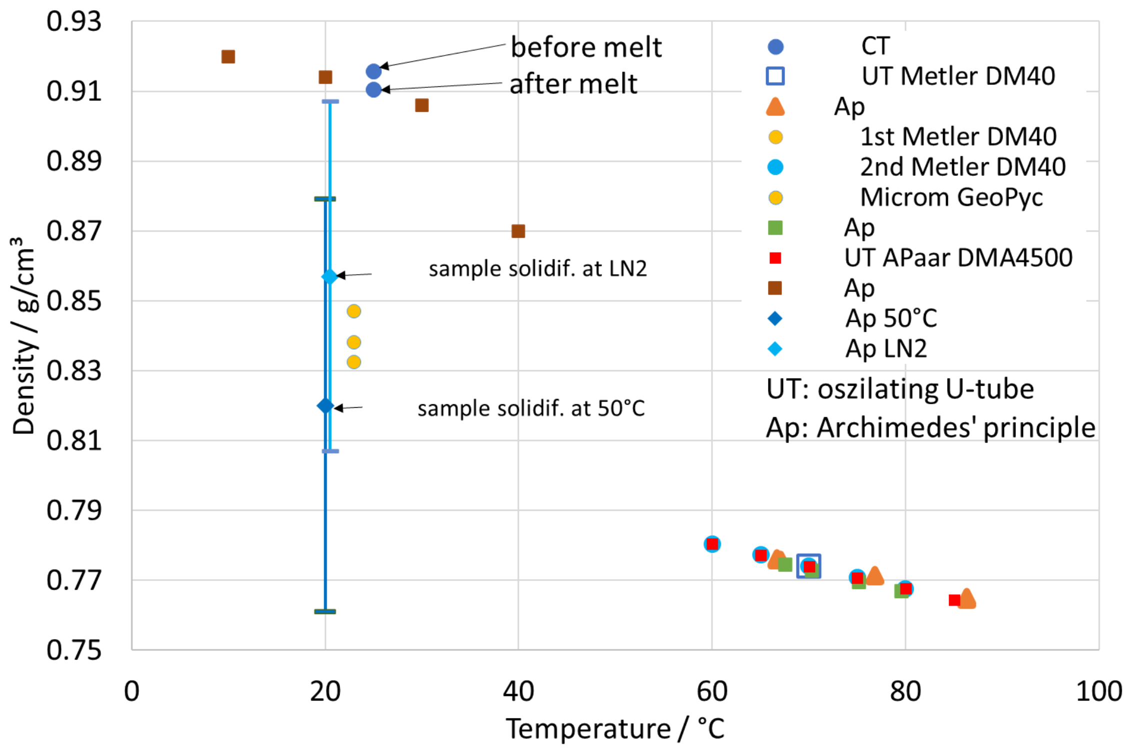
- In the round robin for density measurements in the liquid and solid phase were carried out and compared on a paraffin wax with a melting point between 53-58 °C

Procedure and methodology:

A lot of different methods were used to characterize the actual density of the solid and liquid phase:

- Computer tomography
- Oscillating U-tube
- Archimedes principle
- Pycnometer

Results density



- Results in the liquid phase are comparable while there are strong variations for the solid samples
- A new procedure based on the Archimedes principle was documented and sent to all participants for an additional round robin

Viscosity

Materials

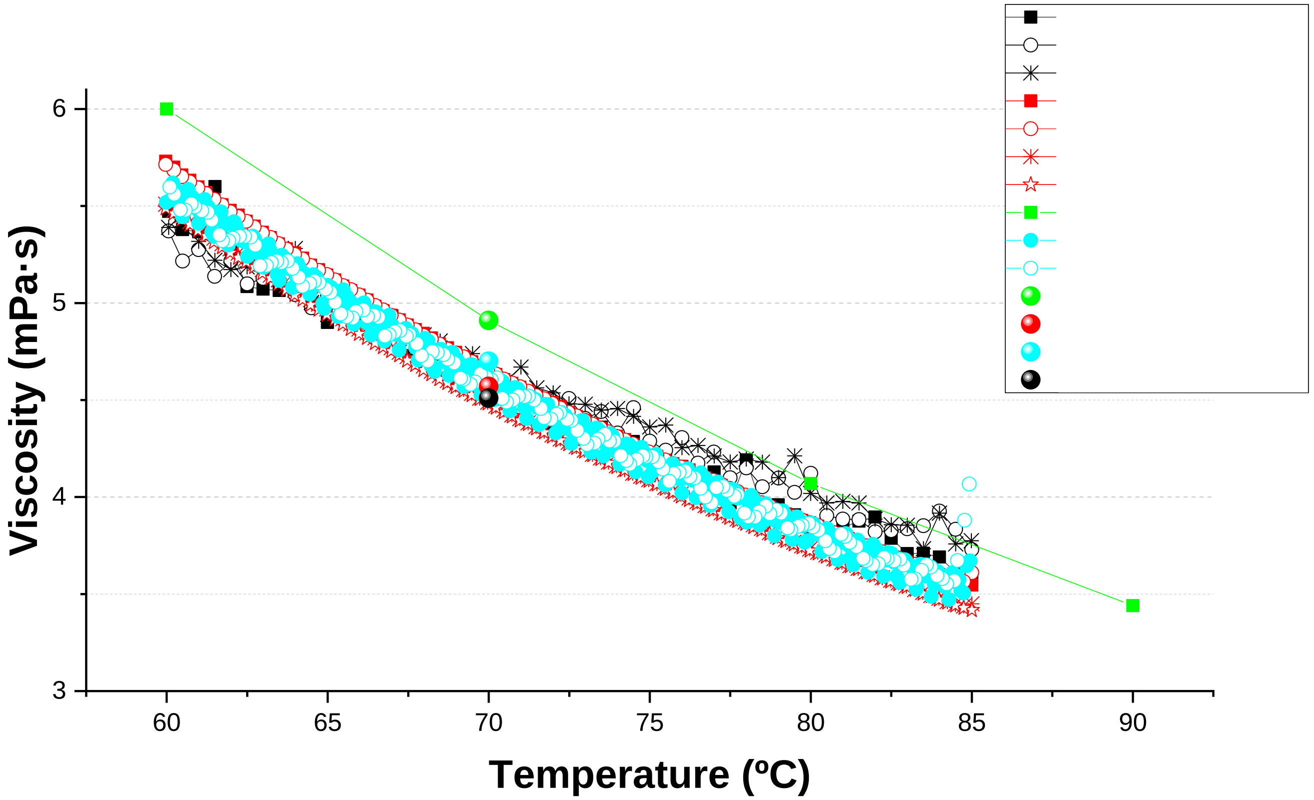
- Standard oil S3 from Paragon Scientific to check the performance of the viscometers/rheometers. Measurement at 80 °C

- A paraffin with a melting temperature range between 53-58 °C (CAS 8002-74-2)

Procedure and methodology:

Shear sweep measurement - geometry, times and temperatures defined

Results viscosity



- measurement procedure is adequate for measuring paraffins in the molten phase

- Viscosity measurements of other PCM families following the proposed measurement protocol should be considered. Furthermore, it would be interesting to characterize other storage materials that may exhibit non-Newtonian behavior (e.g. PCM slurries), to determine how the measurement protocol should be adapted

Conclusion

- the characterisation of TES materials sometimes requires new measurement procedures in order to determine the thermophysical properties in a comparable way
- The application of classical standards for some procedures can lead to incorrect evaluations of the measured quantities
- The work in the IEA TASK enables international comparisons to be made, allowing the measurement methods and procedures used to be harmonized

Thank you for your attention!

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