Encapsulation of Docosane into Polyurethane Microcapsules as Latent Phase-Change Materials for Thermal Energy Storage

Dr. Paula Felix De Castro and Prof. Dmitry Shchukin

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Thermal Energy Storage (TES)

Latent Heat Storage (LHS)

- Latent heat by using **Phase Change Materials** (**PCMs**). Thermal energy is stored when the PCM undergoes a phase change (S-S, S-L, L-G, S-G transitions).
- Heat characteristics:
  - Heat can be stored/release at almost constant temperature.
  - Higher energy density storage per mass/volume.

- **Still technology in development:**
  - **Inorganic PCMs** suffer supercooling and improper resolidification process, degradation and are also corrosive to the heat transfer matrix.
  - **Organic PCMs** show low thermal conductivity and flammability.

PCM encapsulation need

1. Confinement of the liquid phase during the S-L transition and vice-versa.

2. Prevent degradation of the PCMs in contact with the outside environment.

3. Heat transfer improvement via increasing the surface/area ratio (organic PCMs).

4. Supercooling problems in inorganic PCMs are neglected after encapsulation.

5. Flexibility of incorporation of mPCMs in the application devices.

Research lines

1. Encapsulation of **n-alkanes** (organic PCMs).

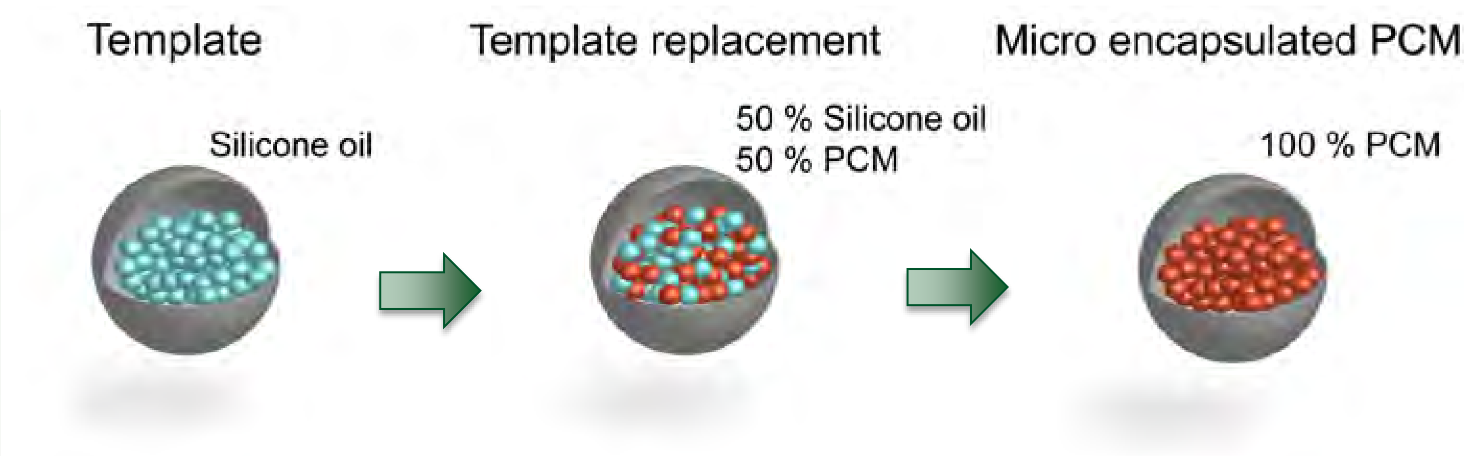
2. Encapsulation of **Salt Hydrates** (inorganic PCMs).

3. Development of hybrid systems based on:
   PCM-GO-CNTs microcapsules/GO for 2D flexible heating devices.

1. Organic n-alkanes as mPCMs

Synthesis Methodology

A template (Silicone oil) was used to optimize synthesis conditions for the encapsulation of organic PCMs (n-alkanes)



Selected n-docosane (C22) as PCM

T_m = 44 °C

ΔH_m = 249 kJ/kg

- Thermo-regulating paints, coatings
- Building components, solar cell components
- Thermo-responsive textiles

Polyurethane (PU) was chosen as a shell

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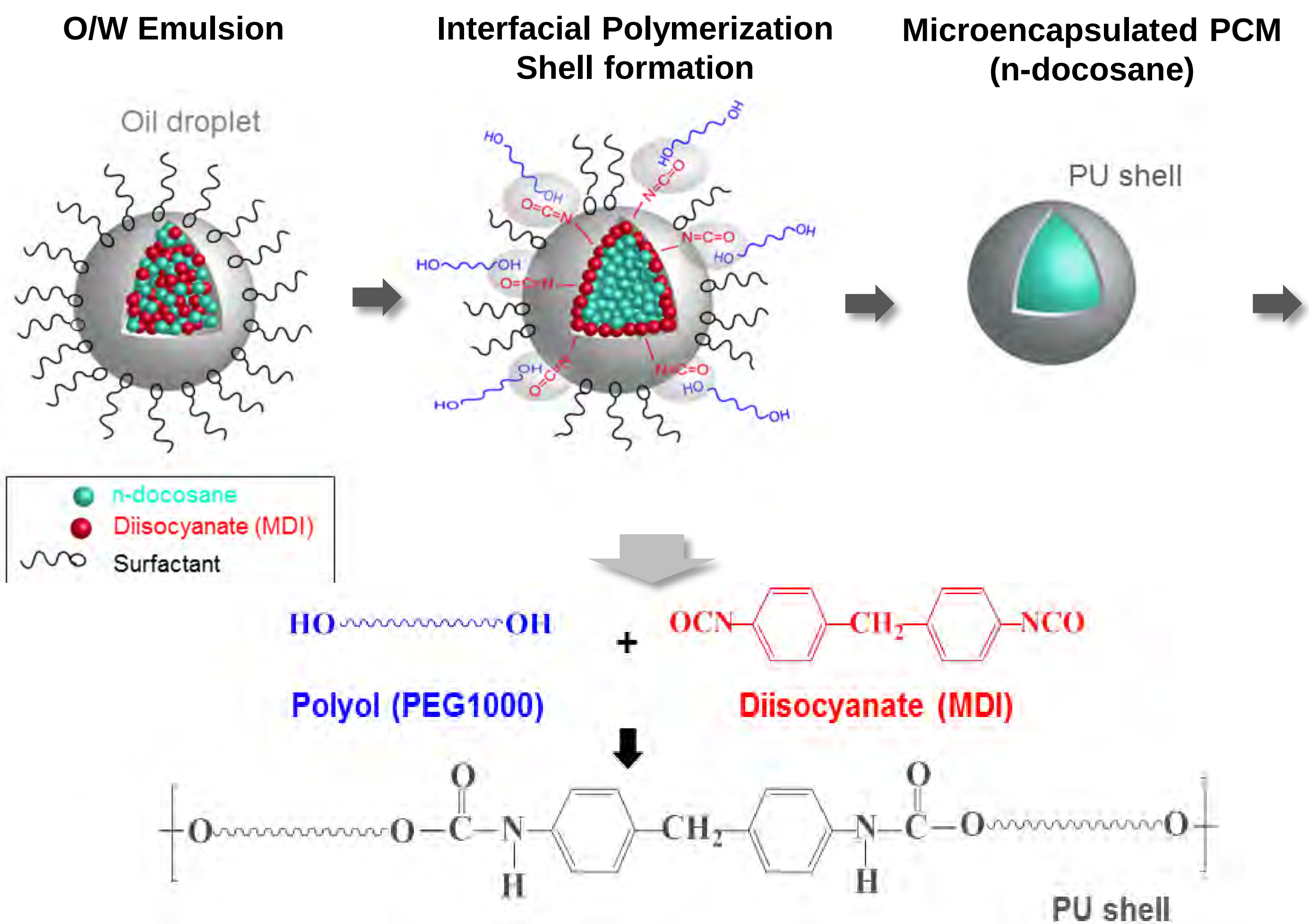
1. Organic n-alkanes as mPCMs

Encapsulation via Mini-Emulsion Interfacial Polymerization



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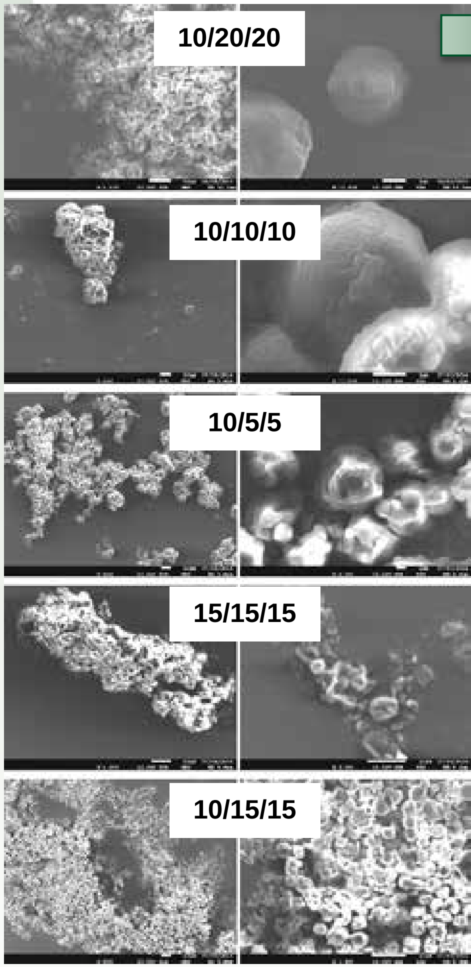
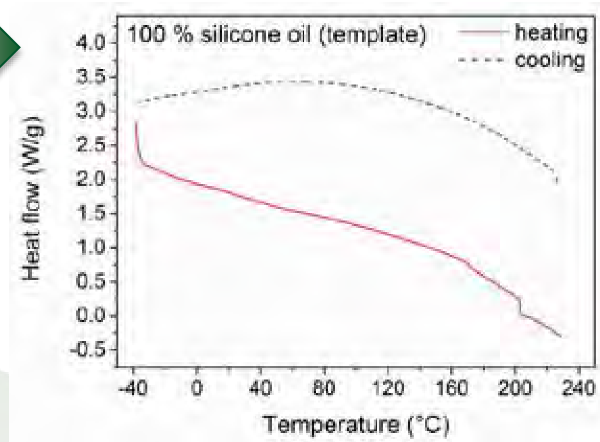
1. Organic n-alkanes as mPCMs

100 % Silicone oil as template

SEM images



DSC

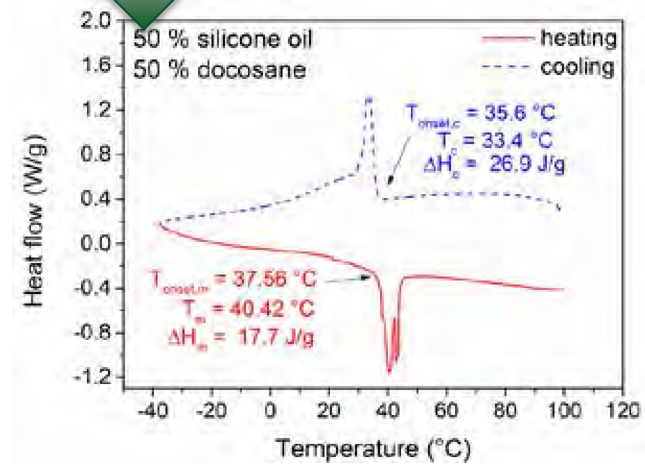


No latent heat when using silicone oil as a core template

50 % Silicone oil- 50 % PCM

SEM images

DSC



Latent heat obtained when introducing 50 % of PCM as a core

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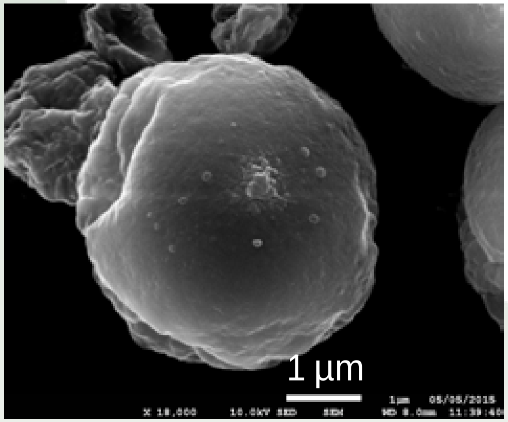
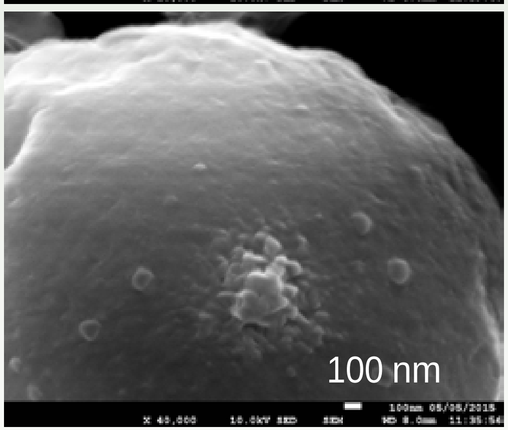
1. Organic n-alkanes as mPCMs

100 % n-docosane as PCM

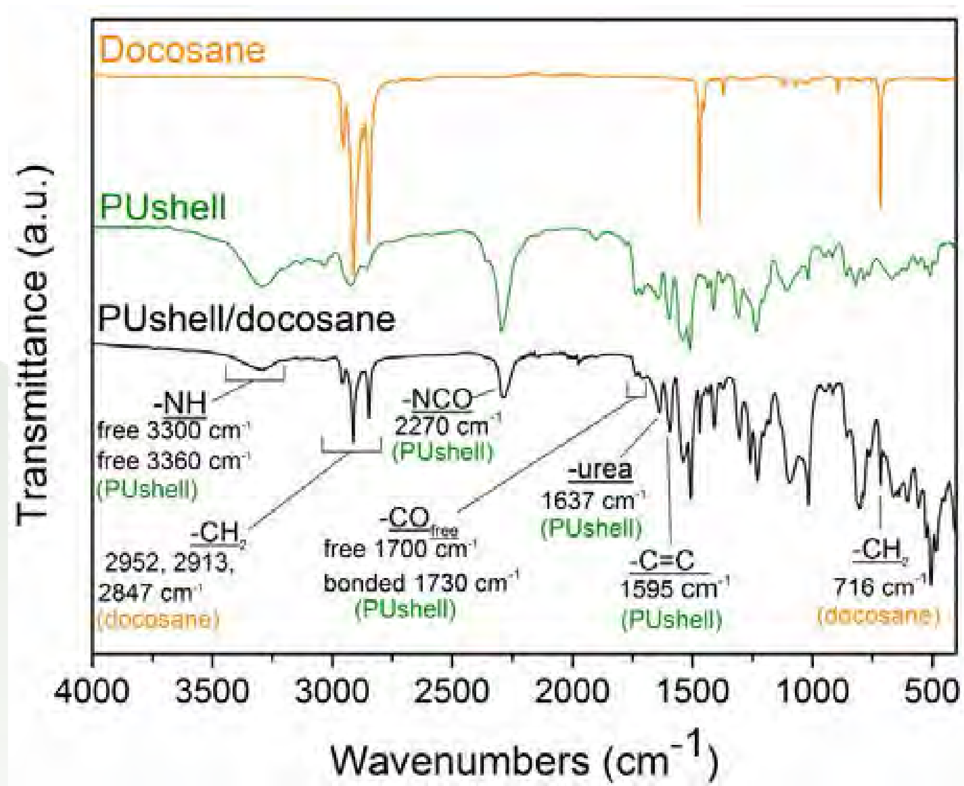
PUshell/n-docosane

(4 ± 1 µm)





FTIR



Characteristic peaks of n-docosane and PU shell present in microcapsules spectra

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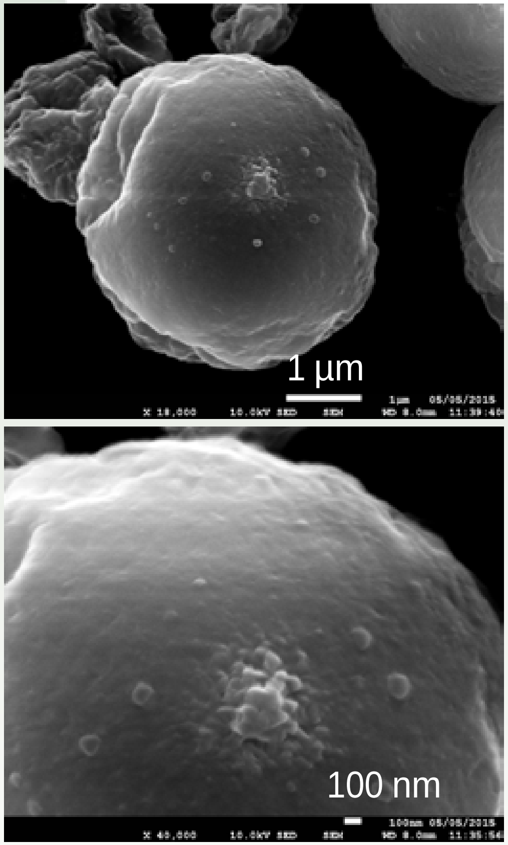
1. Organic n-alkanes as mPCMs

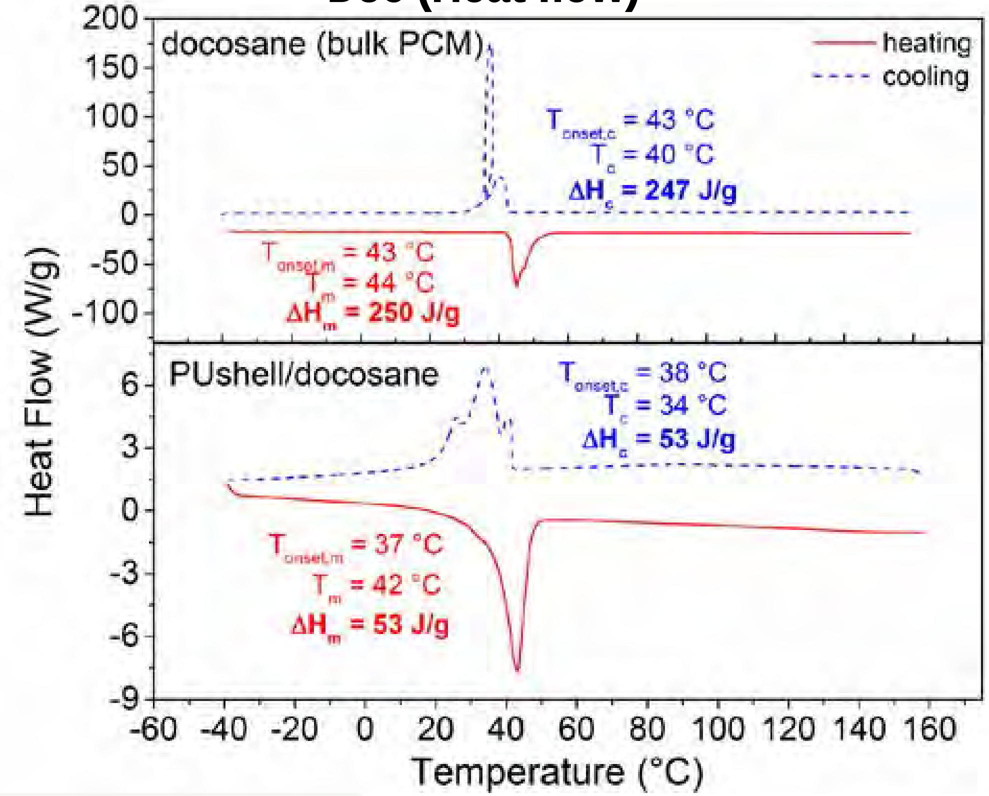
100 % n-docosane as PCM

PUshell/n-docosane

(4 ± 1 µm)



DSC (Heat flow)



Shift of T_m and T_c and latent heat decreased of when encapsulating docosane

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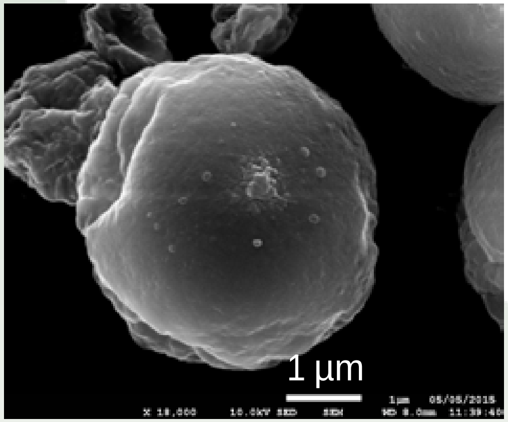
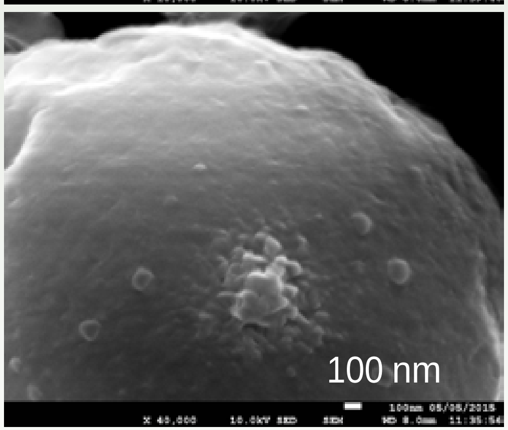
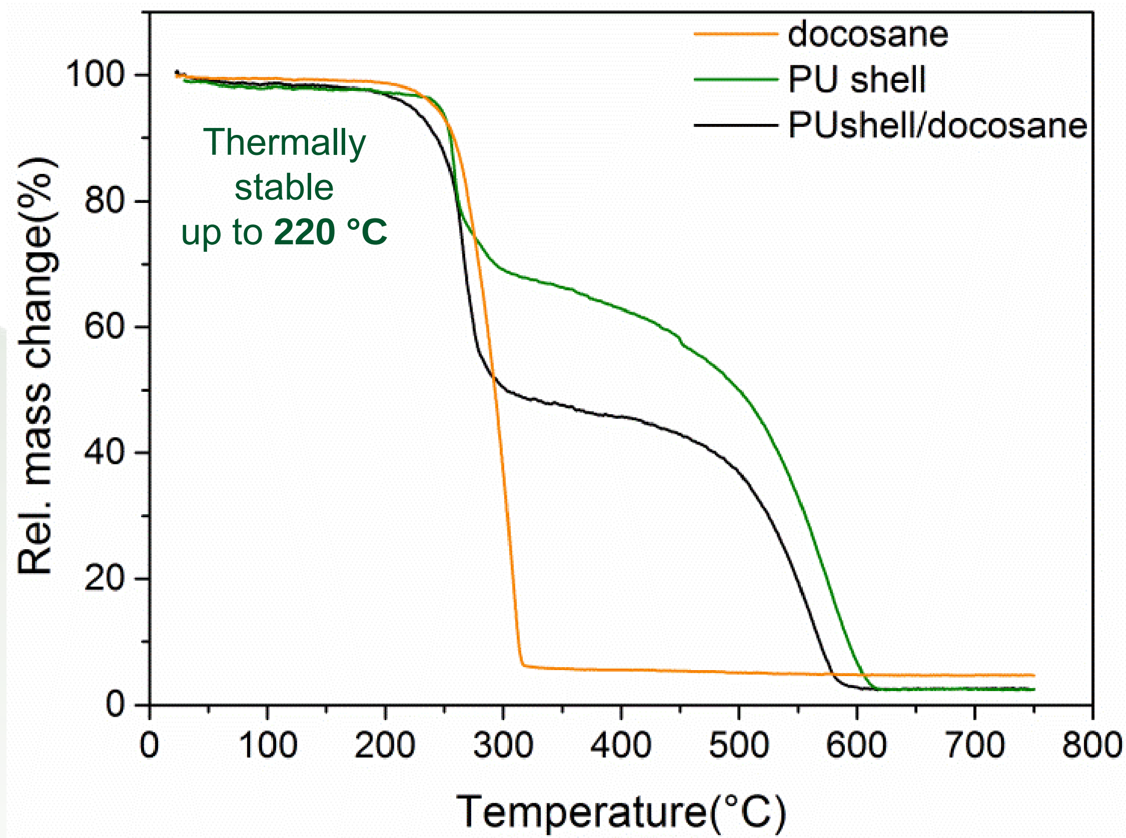




TGA



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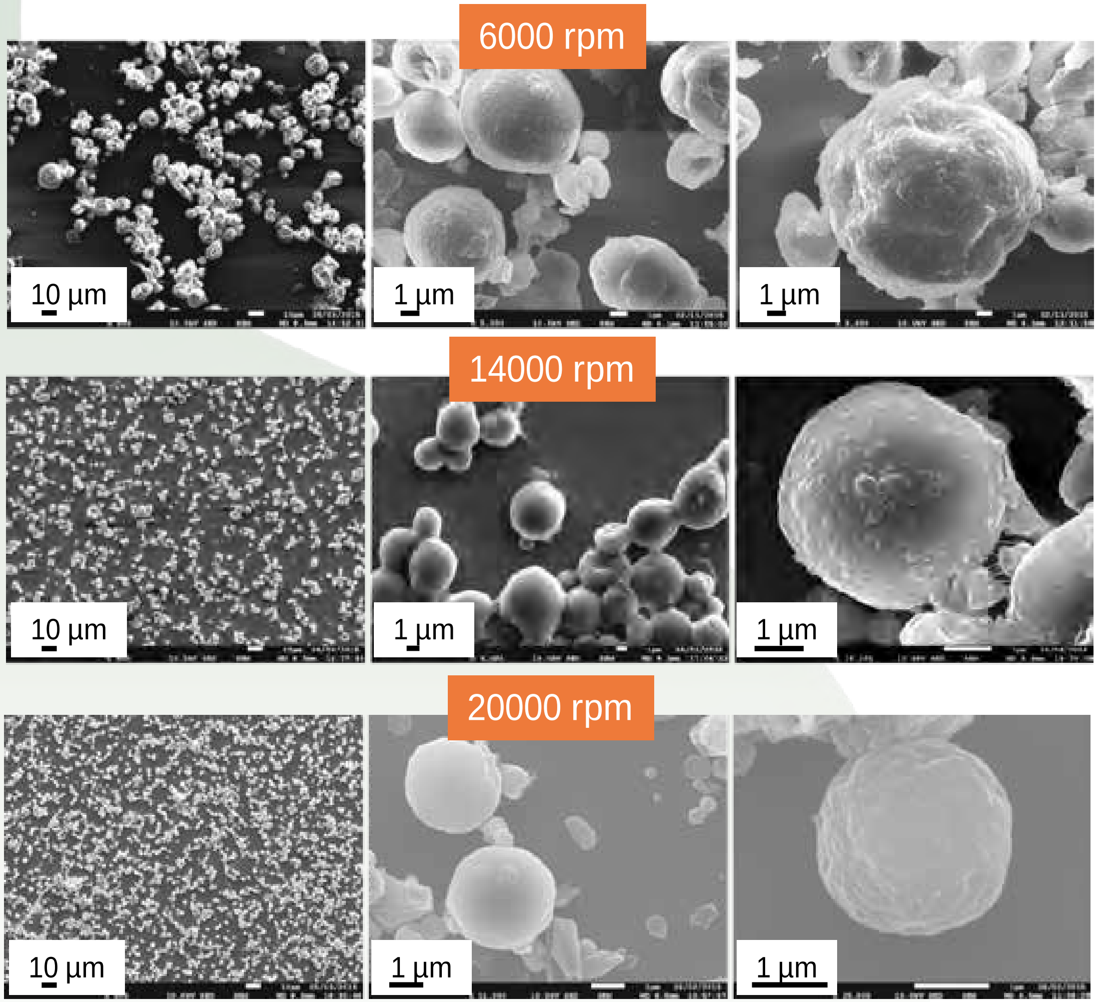
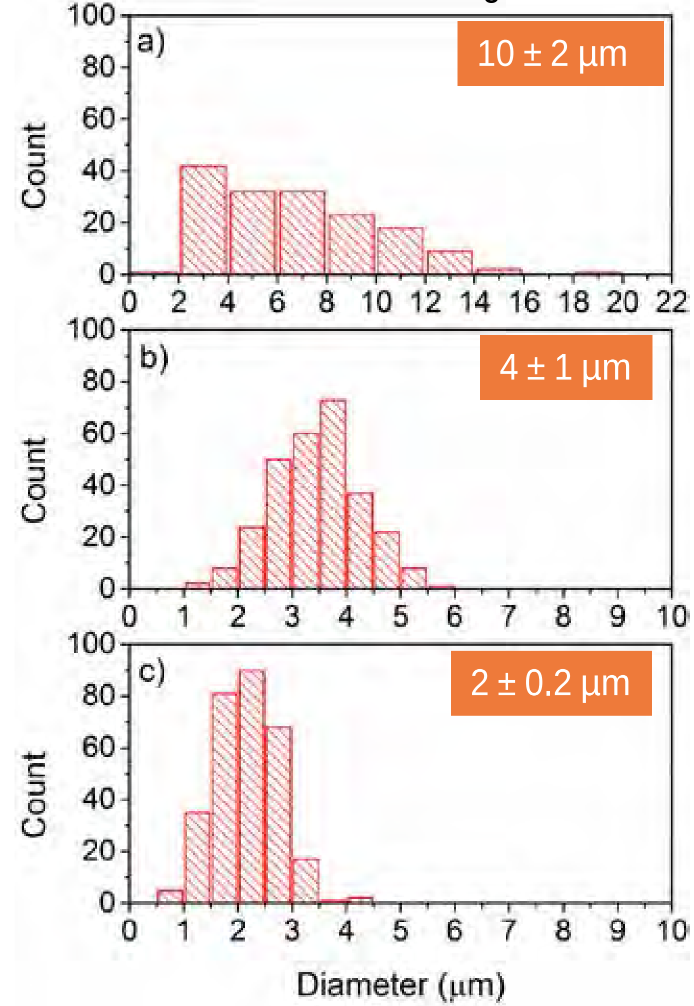
1. Organic n-alkanes as mPCMs

Obtention different microcapsule size by using different rpm



from SEM images



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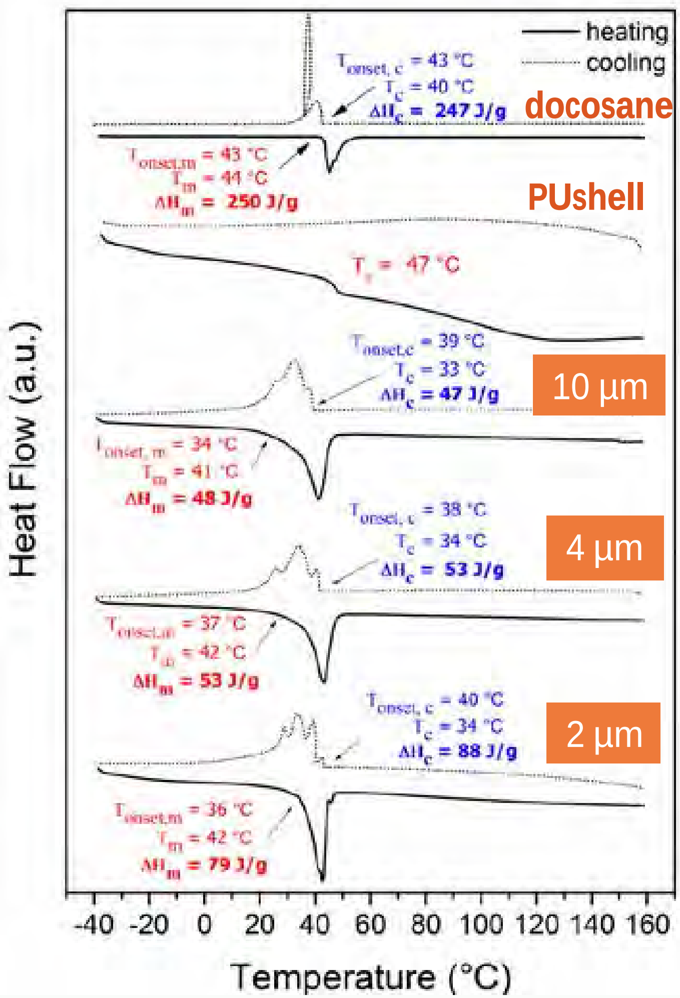
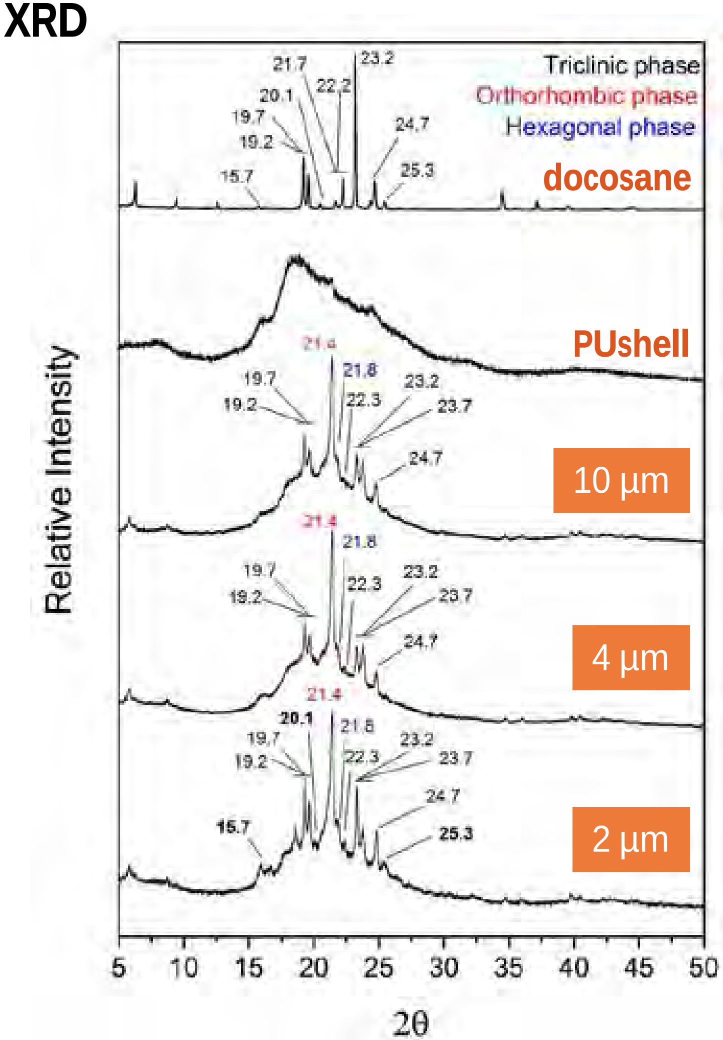
1. Organic n-alkanes as mPCMs

Confinement effect on heat properties and cristallinity

DSC (Heat properties)



XRD



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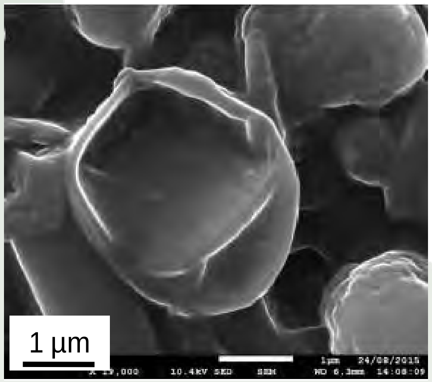
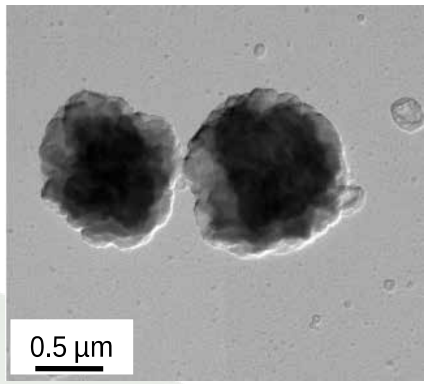
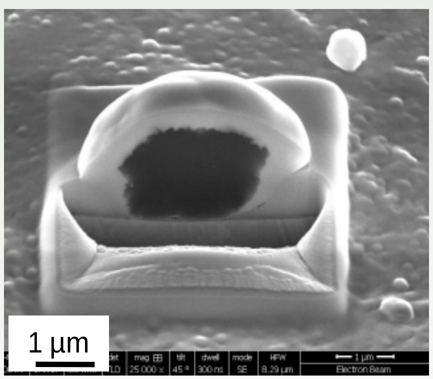
1. Organic n-alkanes as mPCMs

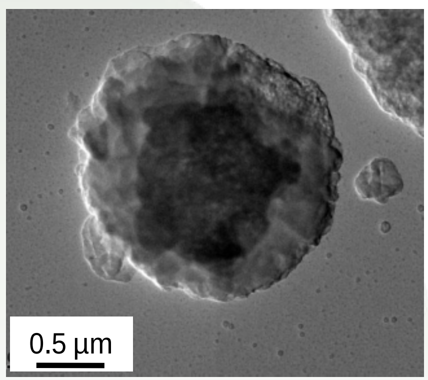
Shell thickness

SEM, FIB-SEM images of artificially broken microcapsules and TEM images





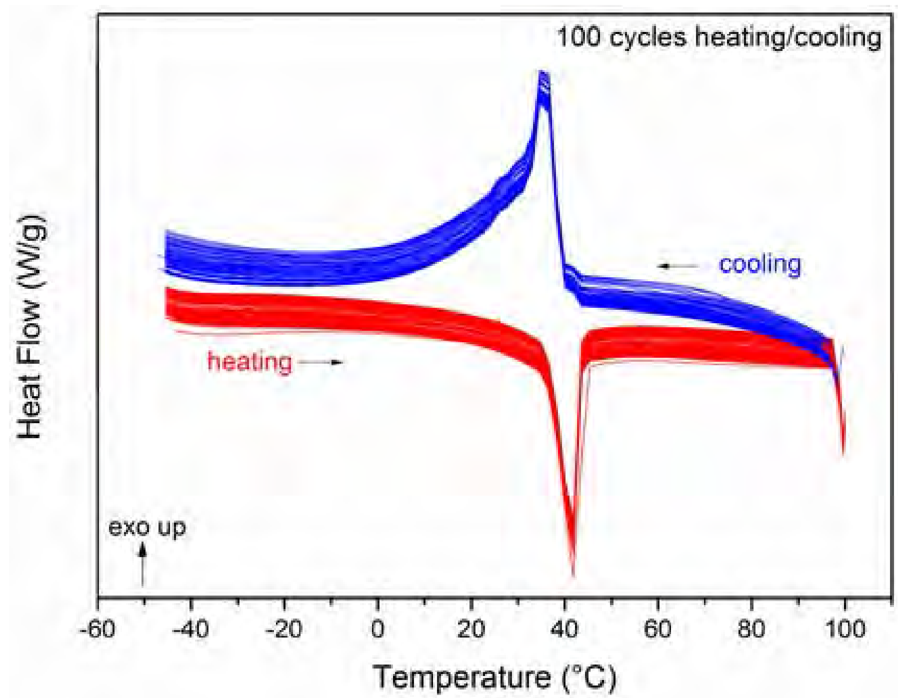




(150-200 nm thickness shell)

Cycling stability

Samples exposed to
100 heating/cooling cycles



Reproducible phase change transition over 100 cycles

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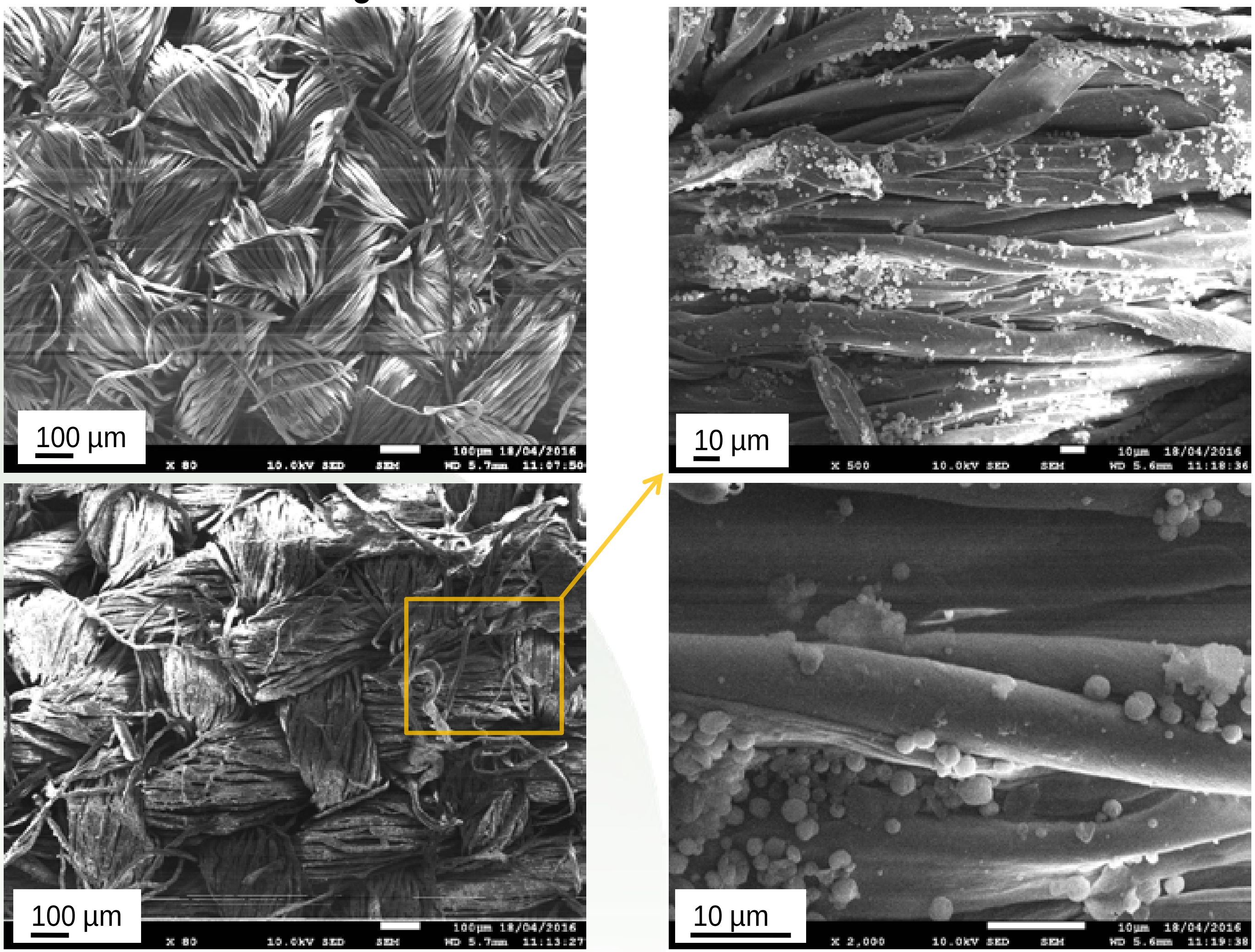
1. Organic n-alkanes as mPCMs

Incorporation of mPCMs onto textiles – (Thermo-regulating textiles)

SEM images



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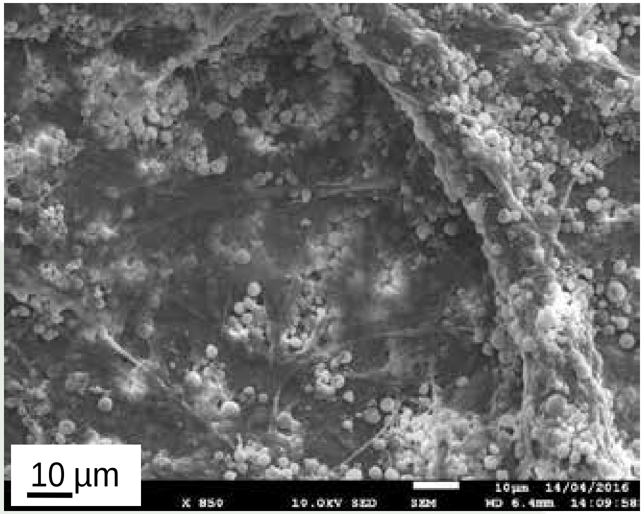
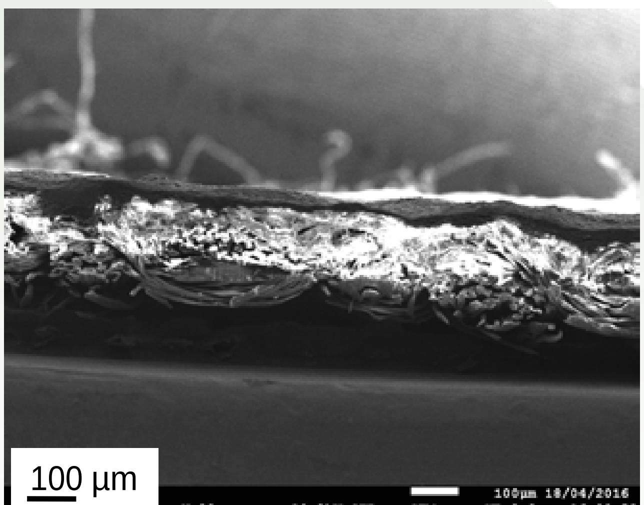
1. Organic n-alkanes as mPCMs

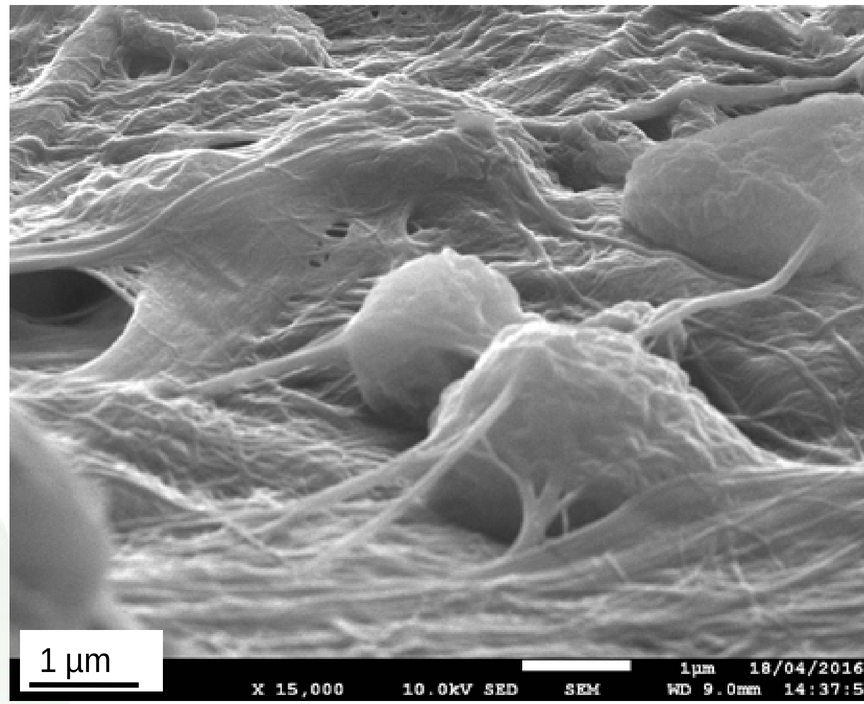
Incorporation of mPCMs into textiles by Nanofibrillated Cellulose (NFC) coating

SEM images





Capsules coated
on the surface of textile with NFC



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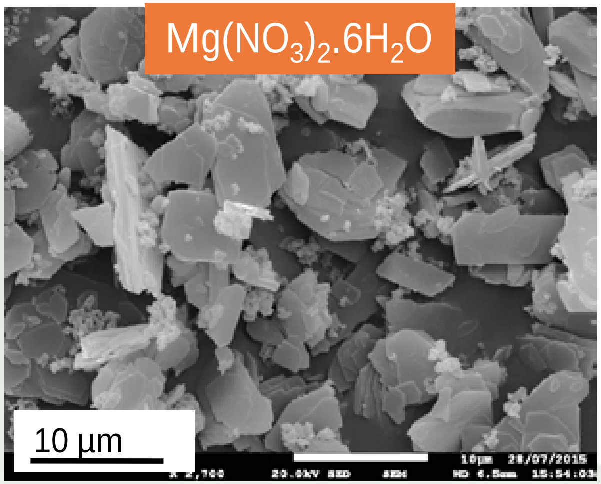
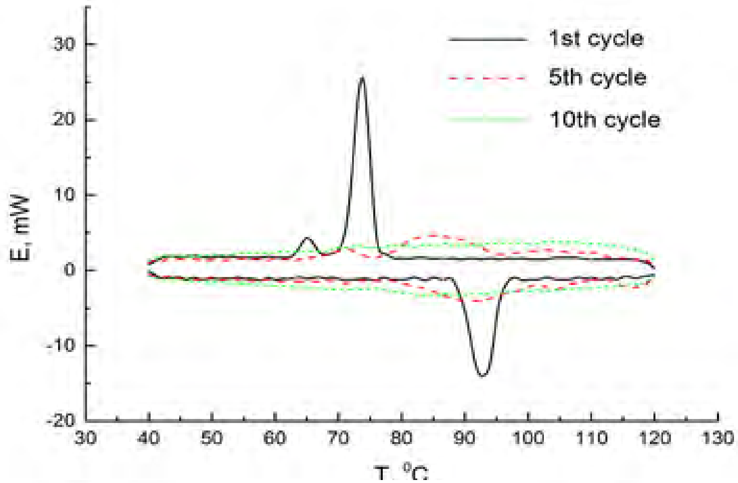
***Collaboration Project** with University of Georgia (USA), Prof. Sergiy Minko*

2. Encapsulation salt hydrates

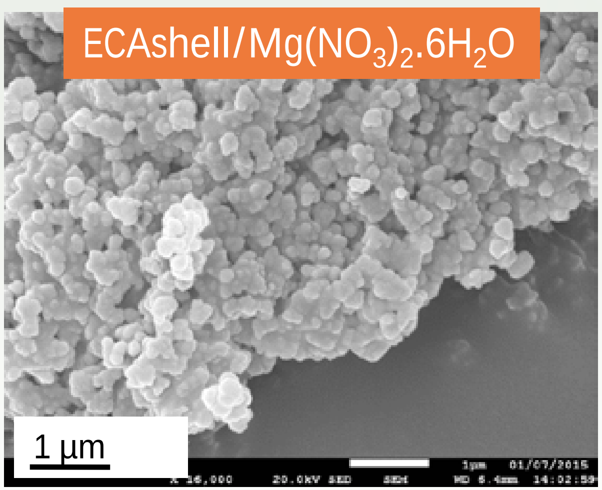
$AB._n(H_2O) \xrightarrow[\text{melts}]{\uparrow T}$ $AB + H_2O$ (anhydrous salt)

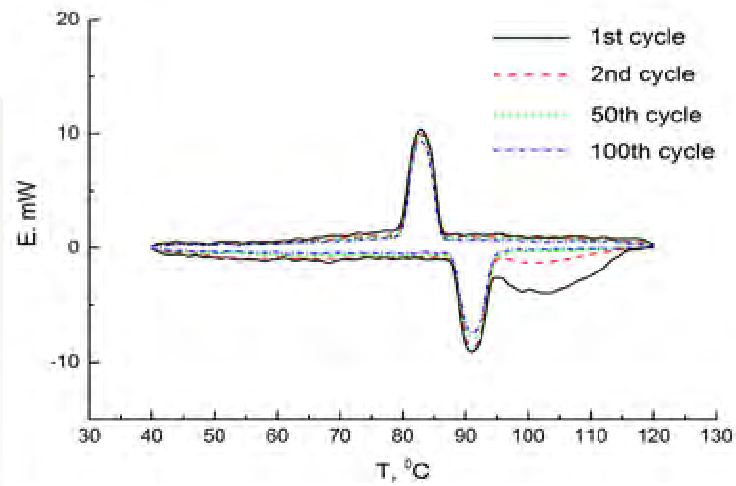
$AB._n(H_2O) \xrightarrow[\text{melts}]{\uparrow T}$ $AB._mH_2O + {}_{n-m}H_2O$ (lower hydrated salt)





Degradation occurs after a few cycles





No degradation of the salt

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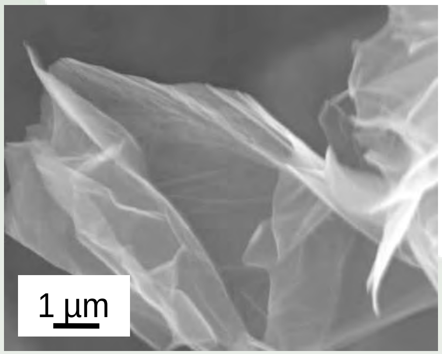
__M. Graham__ and D. Shchukin submitted to JACS.

3. Hybrid systems: PCM-GO-CNTs

PCM-GO-CNTs microcapsules for 2D Joule Heating Devices

Fabrication of PCM-GO-CNTs microcapsules

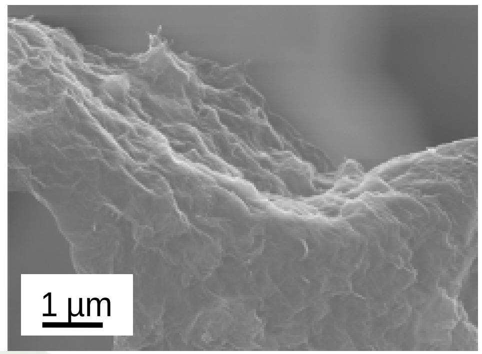
GO sheets



CNTs

Hybridization
pulsed tip sonication

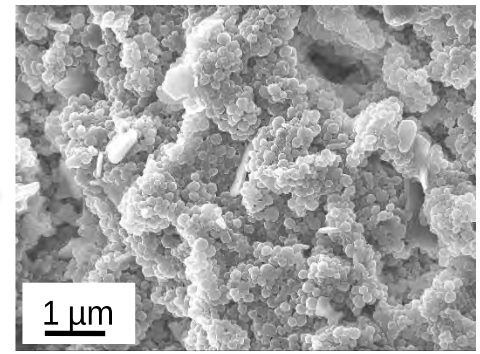
GO-CNTs hybrids

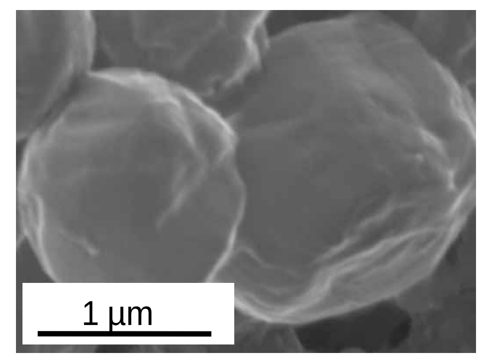


melted PCM

Ultrasound-induced
Emulsification

PCM(docosane)-GO-CNT
microcapsules (1.3 ± 0.3 µm)





It was extended to other PCM alkanes based:

n-hexadecane (C16)

n-octadecane (C18)

n-eicosane (C20)

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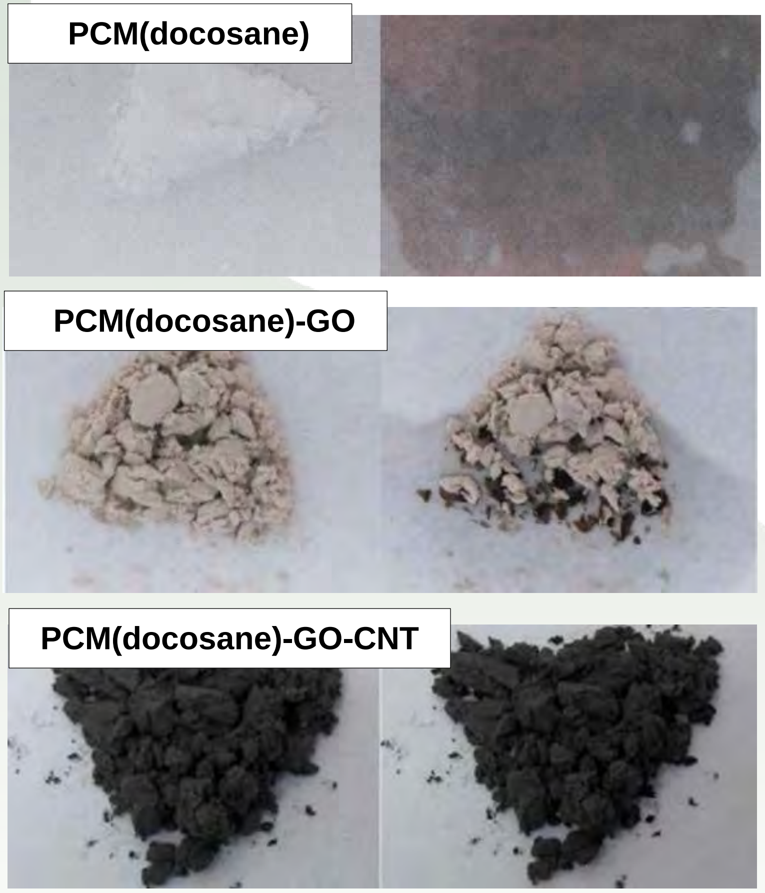
Z. Zheng, J. Jin, J. Zou, U. Wais, A. Beckett, T. Heil, S. Higgins, L. Guan, Y. Wang and D. Shchukin. *ACS Nano*. **2016**. *10* , 4695-4703.

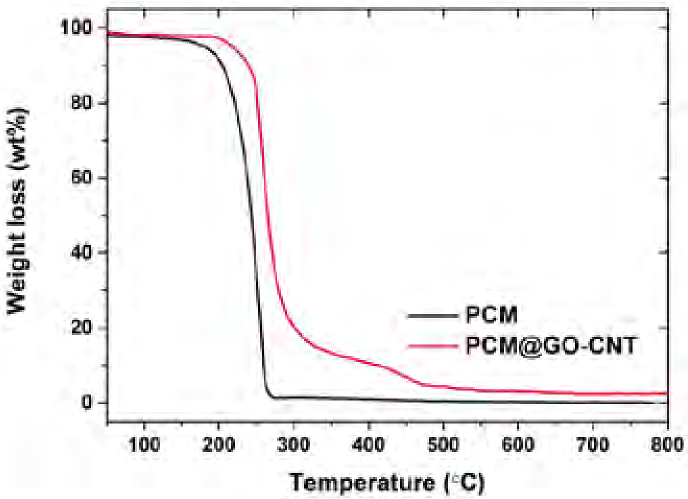
3. Hybrid systems: PCM-GO-CNTs

Stability improvement by encapsulation

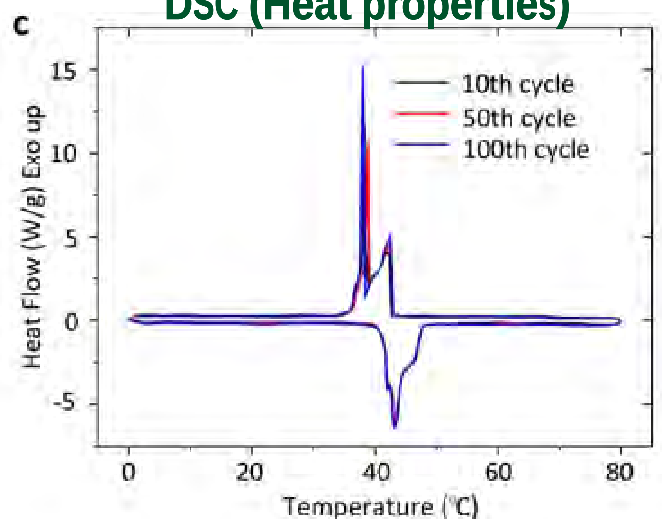
Room temp. **after 30 min at 50 °C**



TGA-Thermal stability



DSC (Heat properties)



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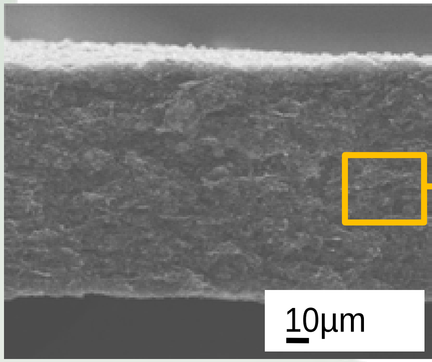
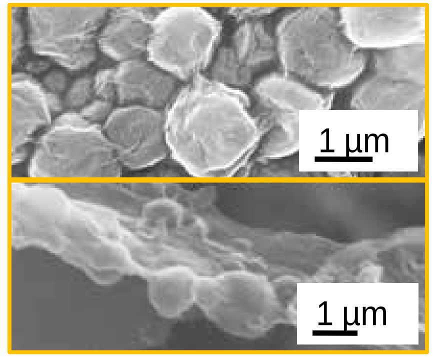
3. Hybrid systems: PCM-GO-CNTs

Fabrication of 2D Joule Heating devices à PCM-GO-CNT/GO



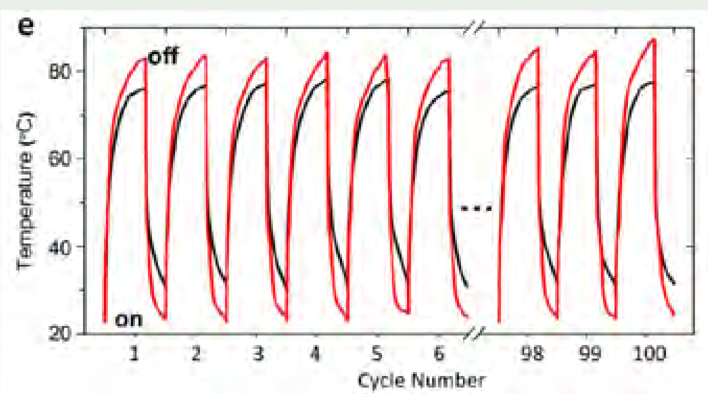
vaccum filtration of
PCM-GO-CNTs + GO

Tailored shape thickness



2D Joule heating device shows electrical and flexibility properties

Thermal enhancement properties



1 cycle consists on:

Power input applied to device: **on** → device heats up (T recorded)

Power input switch **off** → device cools down (T recorded)

Measured over 100 cycles for:

- 2D Joule Heating devices made of **PCM-GO-CNTs/GO**
- Reduced GO (standard device)

10 % thermal enhancement

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Acknowledgments

University of Liverpool

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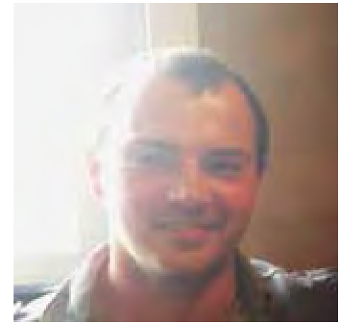
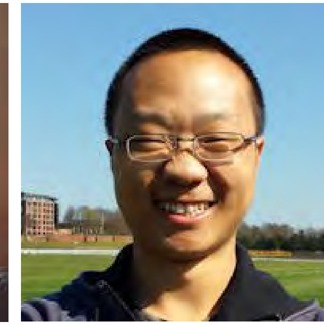


Michael Graham
PhD Student
Salt hydrates encapsulation



Dr. Zhaoliang Zheng
Hybrid systems based on PCM-GO-CNTs
Thermal enhancement

University of Georgia collaboration

Prof. Segiy Minko
Dr. Yunsang Kim

Funding

- NanoBarrier
- Byefouling
- Sono Engineering

- ENERCAPSULE ERC
- InnovateUK

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Thank you very much for your attention

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