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

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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


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

- $T_m = 44 \, ^\circ C$
- $\Delta H_m = 249 \, kJ/kg$

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

Polyurethane (PU) was chosen as a shell

1. Organic n-alkanes as mPCMs

Encapsulation via Mini-Emulsion Interfacial Polymerization

O/W Emulsion

Shell formation

Microencapsulated PCM (n-docosane)

1. Organic n-alkanes as mPCMs

100 % Silicone oil as template

SEM images

50 % Silicone oil- 50 % PCM

SEM images

DSC

No latent heat when using silicone oil as a core template

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

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

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

1. Organic n-alkanes as mPCMs

100% n-docosane as PCM

PUshell/n-docosane (4 ± 1 µm)

TGA

Thermally stable up to 220 °C

1. Organic n-alkanes as mPCMs

Obtention different microcapsule size by using different rpm

- **6000 rpm**: 10 ± 2 µm
- **14000 rpm**: 4 ± 1 µm
- **20000 rpm**: 2 ± 0.2 µm

from SEM images

1. Organic n-alkanes as mPCMs

Confinement effect on heat properties and crystallinity

DSC (Heat properties)

XRD

1. Organic n-alkanes as mPCMs

Shell thickness

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

Cycling stability

Samples exposed to 100 heating/cooling cycles

Reproducible phase change transition over 100 cycles

1. Organic n-alkanes as mPCMs

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

SEM images
1. Organic n-alkanes as mPCMs
Incorporation of mPCMs into textiles by Nanofibrillated Cellulose (NFC) coating

Capsules coated on the surface of textile with NFC

Collaboration Project with University of Georgia (USA), Prof. Sergiy Minko
2. Encapsulation salt hydrates

\[ \text{AB}_n(\text{H}_2\text{O}) \xrightarrow{T} \text{AB} + \text{H}_2\text{O} \text{ (anhydrous salt)} \]

\[ \xleftarrow{\text{melts}} \text{AB}_m\text{H}_2\text{O} + n-m\text{H}_2\text{O} \text{ (lower hydrated salt)} \]

Degradation occurs after a few cycles.

No degradation of the salt.

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
- Ultrasound-induced Emulsification
  - melted PCM
- 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)

3. Hybrid systems: PCM-GO-CNTs

Stability improvement by encapsulation

Room temp. after 30 min at 50 °C

PCM(docosane)

PCM(docosane)-GO

PCM(docosane)-GO-CNT

TGA-Thermal stability

DSC (Heat properties)

3. Hybrid systems: PCM-GO-CNTs

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

Tailored shape thickness

Vacuum filtration of PCM-GO-CNTs + GO

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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