Sveučilište u Zagrebu / University of Zagreb
Tekstilno-tehnološki fakultet
Faculty of Textile Technology

DOCTORAL THESIS DEFENCE

ELECTROSPUN NANOFIBROUS MATERIALS AND FILMS FOR HEAT MANAGING APPLICATIONS

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Overview

1. Heat energy storage and phase change materials
2. State of the art in electrospun phase change nanofibers
3. Objectives and research questions
4. Materials and methodology
5. Results

5.1. PVA/plant oils electrospun fibrous materials

- Fibers morphology, heat managing performance, crystallinity, thermal stability and mechanical behaviour.

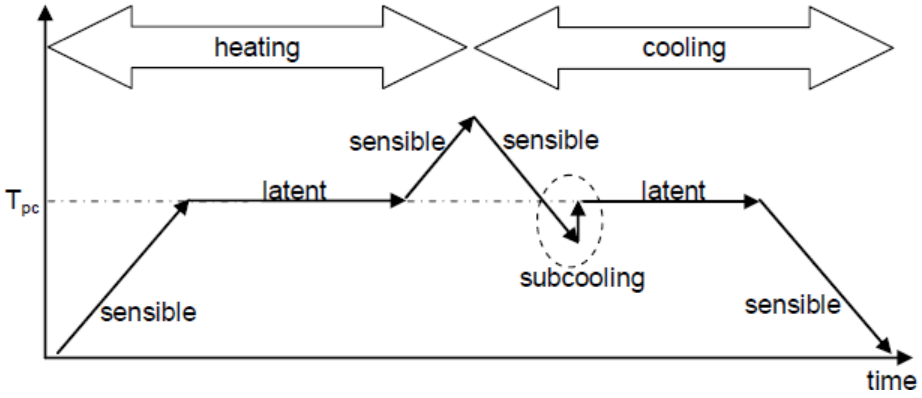
5.2. PVDF/PCL electrospun fibrous materials

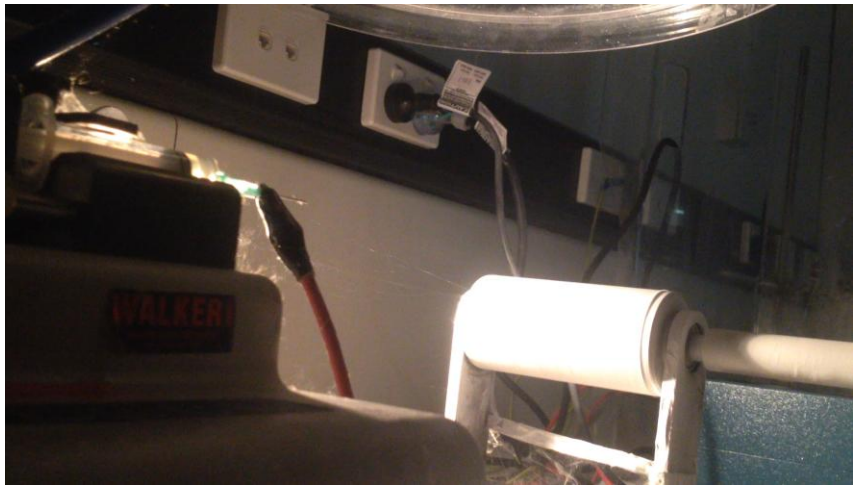
- Heat enthalpies and fibers diameter relation, comparisons with films counterparts, influence of thin layer coating and thermally conductive compound on the heat managing performance, thermal stability and mechanical behaviour.

6. Conclusions

Heat energy storage and phase change materials

1) Latent heat storage - high density of energy stored, isothermally.
2) Phase change material - solid to liquid transfer
Requirements of PCM: melting temperature in compliance with practical application, large heat enthalpy, low supercooling effect, thermal conductivity and cycling reliability.
3) PCM encapsulation





State of the art in electrospun phase change nanofibers

Electrospinning encapsulation of PCMs:

1) co-axial electrospinning (PMMA/tetradecanol; PVP/hydrocarbons – multi channel)
2) single-phase electrospinning (polymers or small molecules PCMs: e.g. PEG in PLA, CA or PA 6; paraffins, fatty acids or their esters in PA 6, PET, PU etc.
3) addition of nanoparticles (SiO_2 and CNTs into PET/LA and PVDF/PEG)

General conclusions reported:

- Fibers as form-stable media with an advantage of the core-sheath configurations;
- Nanoparticles improved thermal conductivity and thermal stability, reduced flammability and both improvement and reduction in heat storage performance.

- Little attention on fibrous form stability and mechanical behaviour at temperatures above the PCM melting point?

State of the art in electrospun phase change nanofibers

Materials	PCM wt% or mass ratio	PCM H_m/H_c (J/g)	Composite - H_m/H_c (J/g)	Ref.
LA/PET	100/100	171.13/174.91	70.76/62.14	Chen et al 2008
SA/PET	70/100	222.8/226.7	67.88/61.62	Chen et al 2008
PA-SA/PET	70/100 120/100	204.7/204.2	72.11/67.20 112.7/101.6	Ke et al 2012, Cai et al 2012
CA-SA/PET	200/100	156.8/147.1	95.24/93.67	Ke et al 2013
LA/PA6 LA/PA6+CNFs(1wt%)	100/100	173.25/172.19	70.44/57.14 75.99/61.34	Cai et al 2012 & 2014
LA/PET/SiO_2(4 wt%)	70/100	173.25/172.19	55.8/29.9	Cai et al 2011
Soy wax/PU	50	69.97/-	36.47/-	Hu et al, 2012 & 2014
SS/PET	50/100	172.7/175.0	53.77/-	Chen et al 2009
ADOEs/PET	75/100	164.3/162.4	66.5/65.4	Chen et al 2011
GMS/PET	150/100	115.1/114.8	66.99/66.02	Ke et al 2013
Octadecane/TiO2-PVP	45	-	114/-	Mc Cann et al 2006
Dodecane/zein	14	196.88/196.88	27.552	Perez-Masia et al 2012
RT5/PCL	44.35	144.7/-	-	Chalco-Sandoval et al 2014
PEG1000Da/PVDF	20	171.9/168.0	33.70/30.40	Ngyen et al 2011
PEG4000Da/PVDF	42.5	161.0/155.0	68.0/62.0	Van Do et al 2012
PEG8000Da/PLA	66.67	167.8/161.8	74.70/83.3	Chen e tal 2013
PEG/CA	50(70)	177.38/167.75	85.91(120.18) /65.15(104.40)	Chen et al 2007 & 2011
PEG/PA 6	130/100	160.93/188.98	85.95/85.42	Seipfoor et al 2011

Objectives and research questions

Developing a heat storing electrospun fibrous materials based on mixture of plant oils or polycaprolactone as phase change materials;

1. How the phase change material influences fibers morphology, thermal performance, mechanical properties and stability upon heat/cool treatment?
2. How will the polycaprolactone perform as a phase change material?
3. How will the change in the fiber diameter affect the heat enthalpies?
4. What is the effect of a polymer coating on the electrospun phase change fibers heat managing performance and mechanical behaviour after high heat exposure?
5. How will the polymer coating combined with reduced-graphene oxide affect the materials heat managing performance and thermal conductivity?

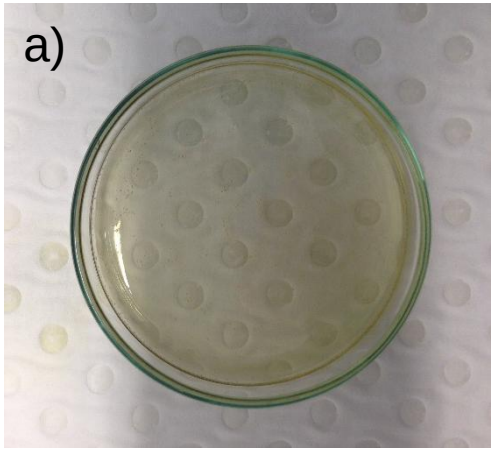
Materials and methodology

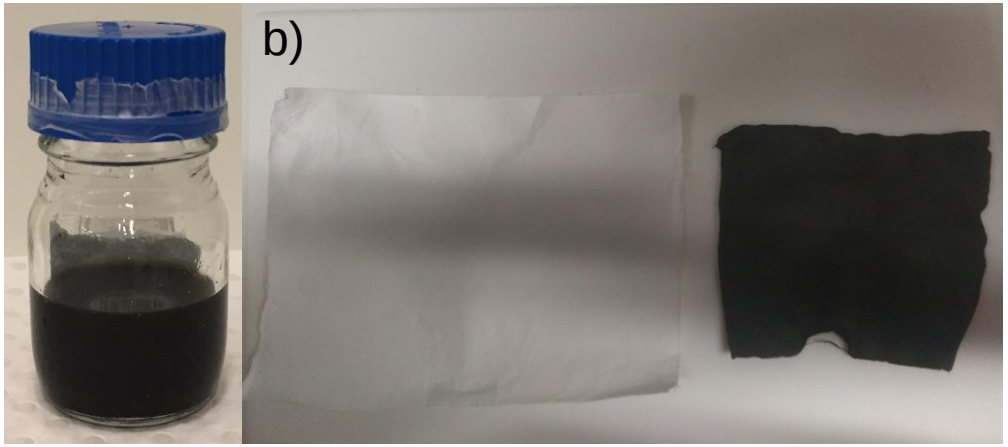
- Preparation of the electrospun heat managing fibrous materials from:

1) mixture of plant oils and PVA matrix by emulsion electrospinning;

2) PCL (with low and high Mw) and PVDF matrix by polymer solution blend electrospinning.

- Preparation of PVDF/PCL films counterparts by casting for comparison (a).
- Post-processing treatments: 1) vapour polymerization of PPy and PPy/r-GO (b).





Materials and methodology

1. Emulsion electrospinning:

- O/W emulsions (15 wt% PCM + SDS (8.4 mmol/L)) 70-80 ºC + pre-prepared PVA aqueous solutions. Shear mixing + sonification + Triton X-100 (0.07- 0.2%).
- PVA concentrations: 9 w/w% and 7 w/w%.
- PVA/PCM mass ratios: 100/10, 100/30, 100/50, 100/70, 100/90.

2. Blend electrospinning:

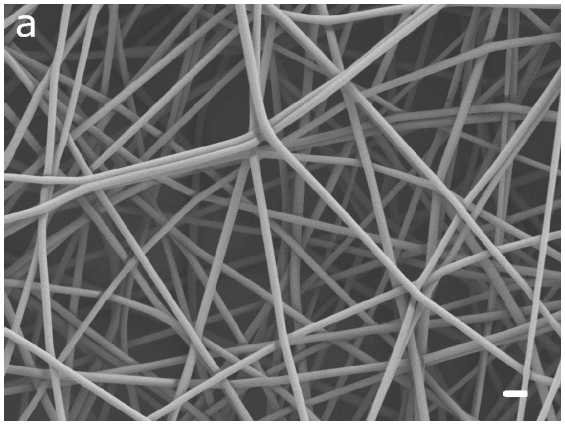
- 14% PVDF, 14% PCL (low and high Mw) in DMF:THF=4:6 and 1:1, respectively.
- PVDF/PCL (low Mw) = 14% 100/(25-100), DMF:THF=4:6
- PVDF/PCL (high Mw) = 8% 100/75, DMF:THF=3:7

3. Vapour polymerization of pyrrole:

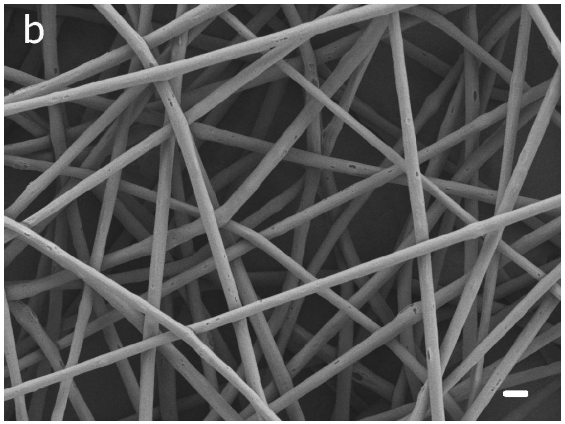
- Vacuum desiccator: exposure of the electrospun PVDF/PCL to pyrrole vapour with prior immersion in 1.5% $FeCl_3$ x $6H_2O$/ethanol solution or r-GO/$FeCl_3$ x $6H_2O$/ethanol solution.

GO reduction in L-AA/water for 48 h. Re-dispersion in ethanol with NaCl.
Concentrations of: 0.25 and 1 mg/ml.

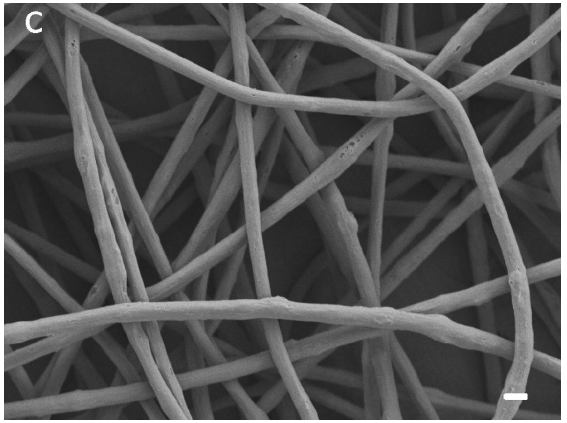
PVA/PCM fibers morphology



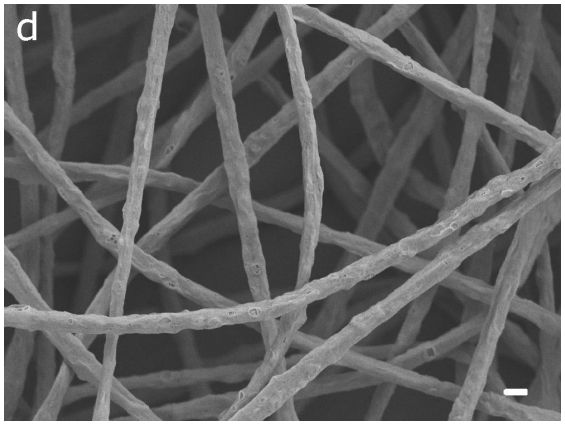
9w/w% PVA



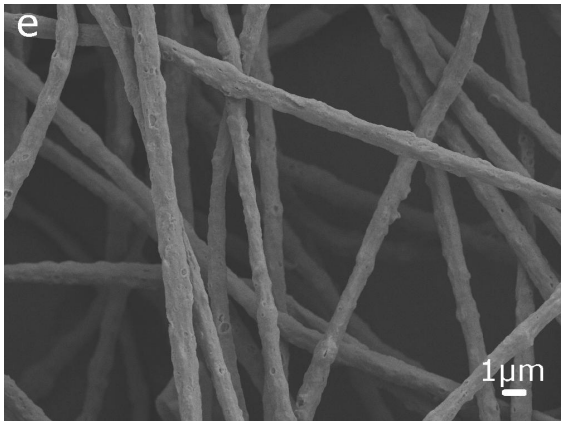
PVA/PCM-10



PVA/PCM-30

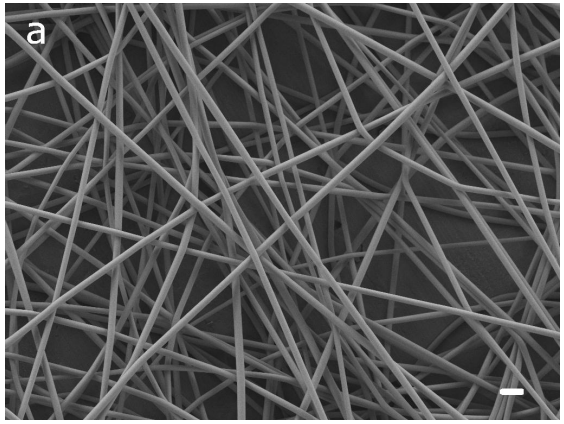


PVA/PCM-50

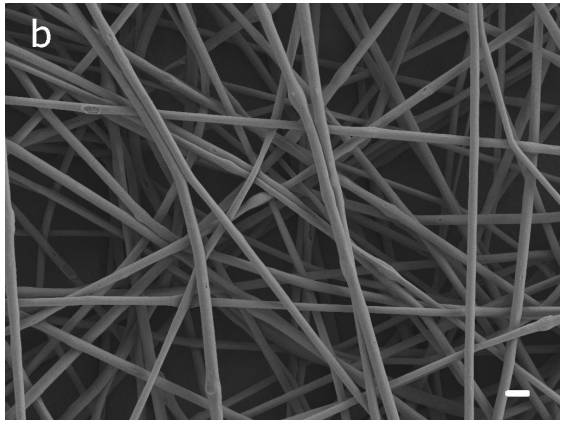


PVA/PCM-70

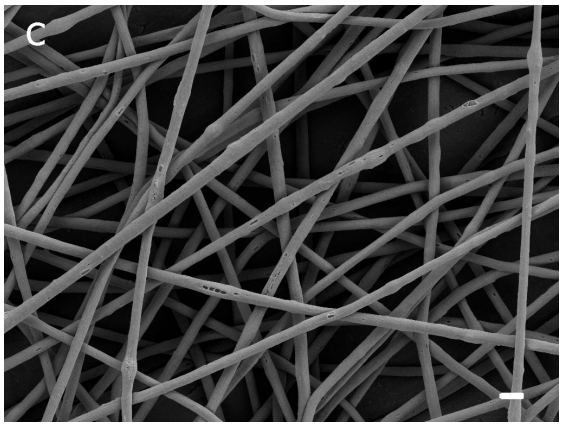
PVA/PCM fibers morphology



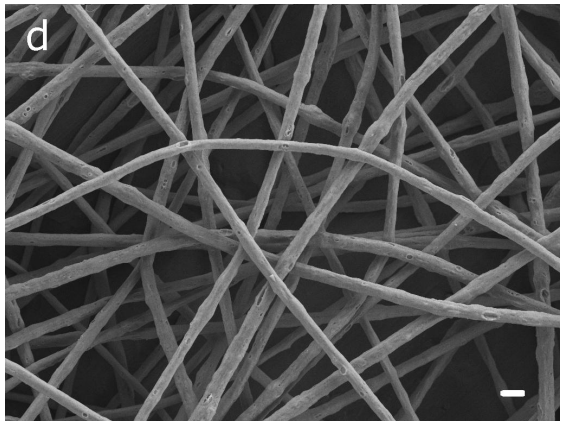
7w/w% PVA



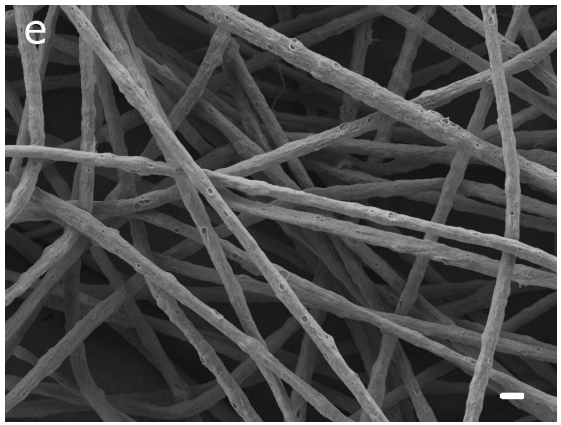
PVA/PCM-10



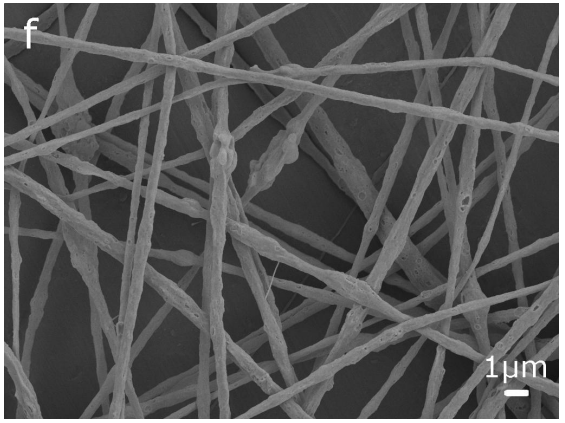
PVA/PCM-30



PVA/PCM-50

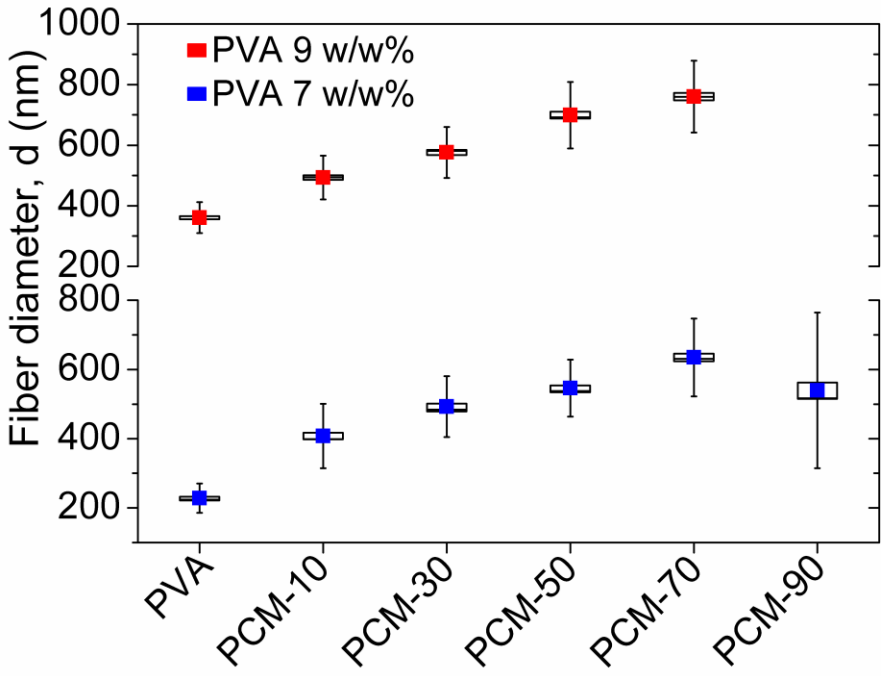


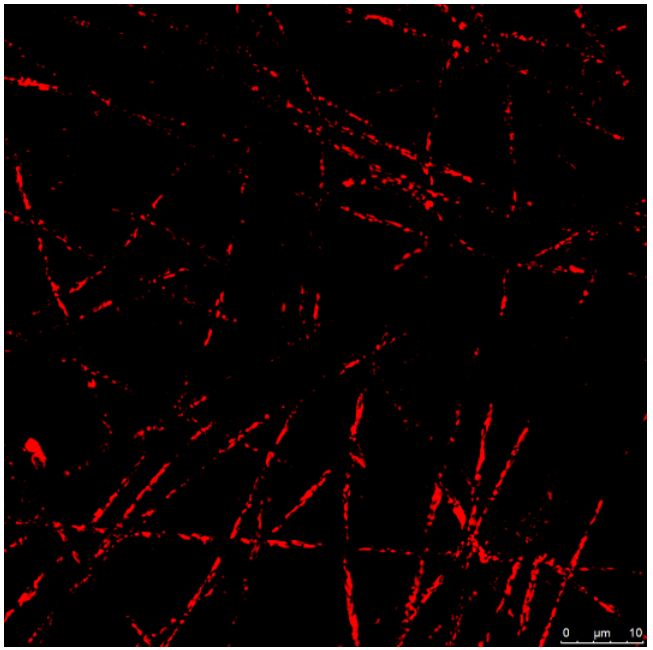
PVA/PCM-70



PVA/PCM-90

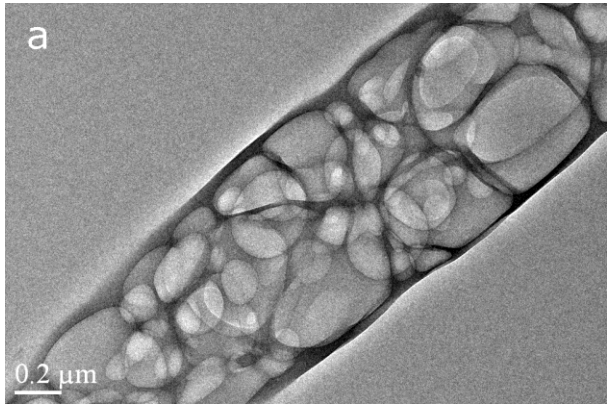
Fibers diameter and PCM distribution



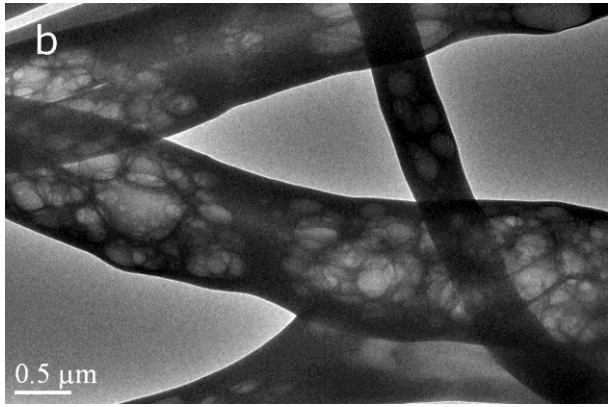


9w/w% PVA/PCM-50

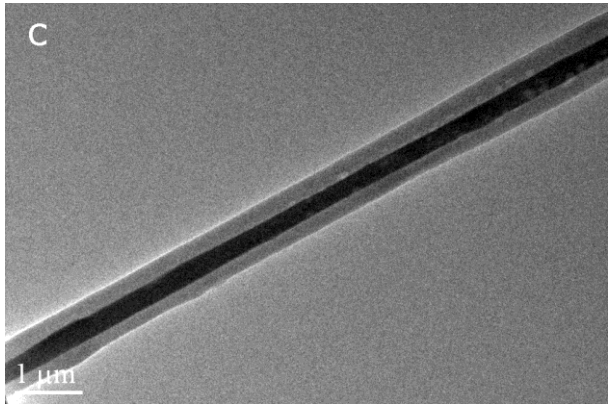
Fibers inner morphology and cross-sections



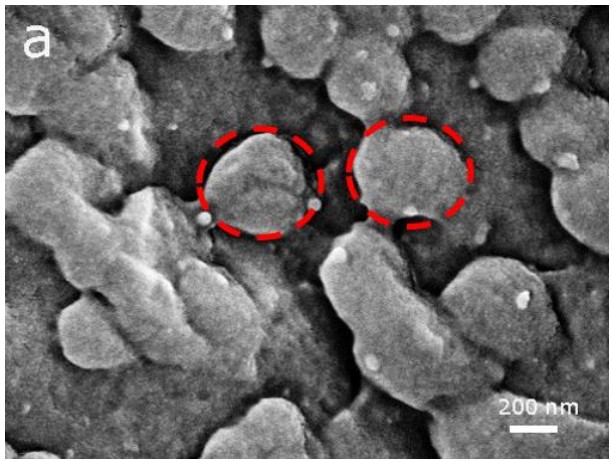
9w/w% PVA/PCM-10



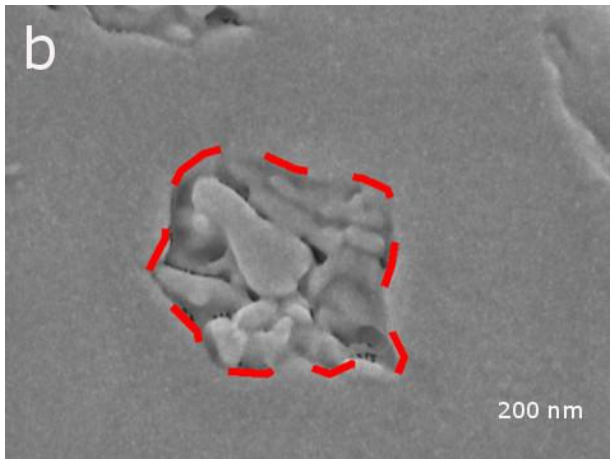
9w/w% PVA/PCM-50



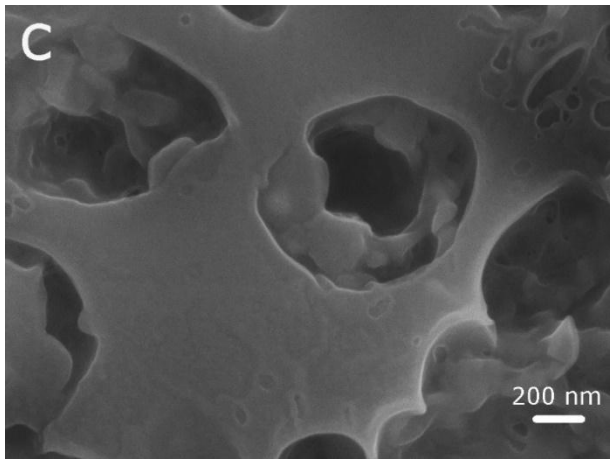
9w/w% PVA/PCM-50



9w/w% PVA

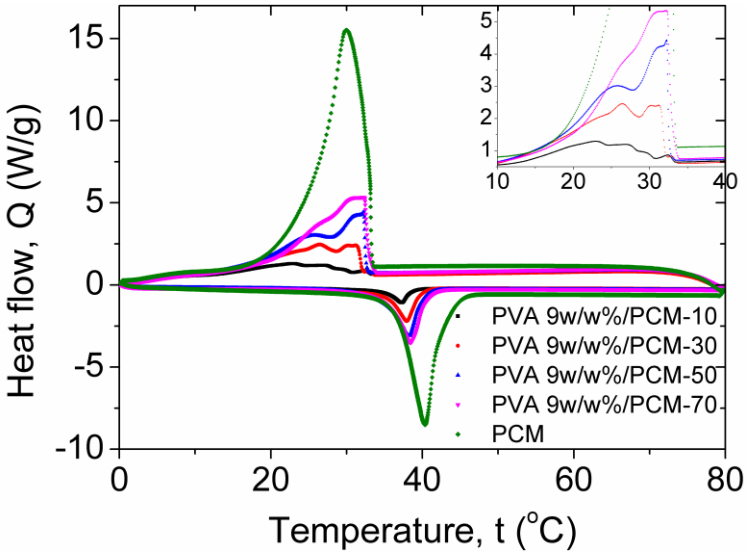


9w/w% PVA/PCM-70



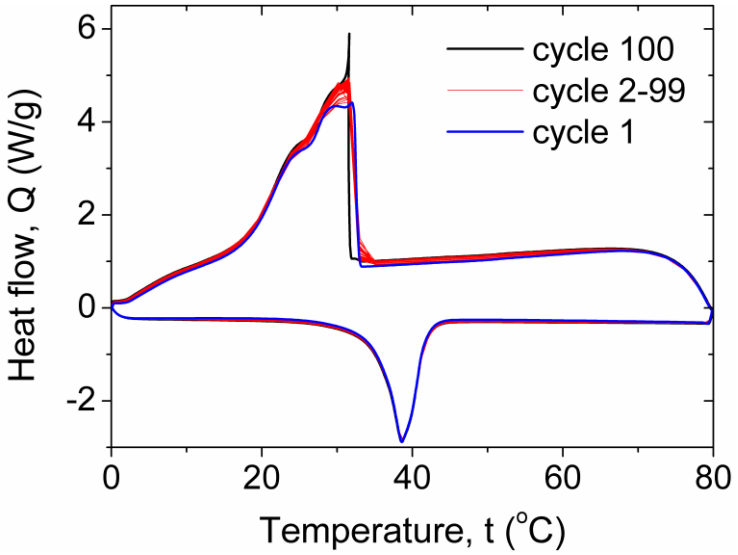
9w/w% PVA/PCM-70 heat

PVA/PCM fibrous mats heat managing performance

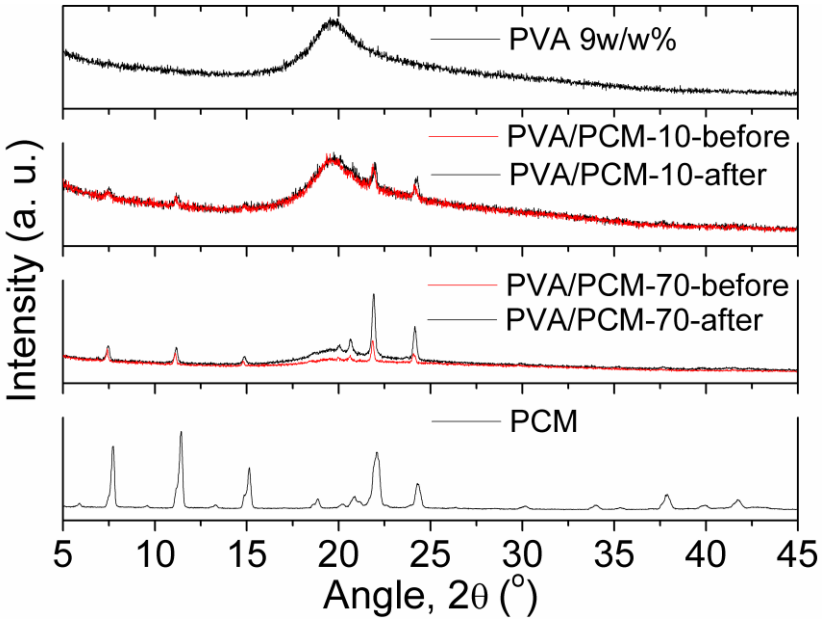


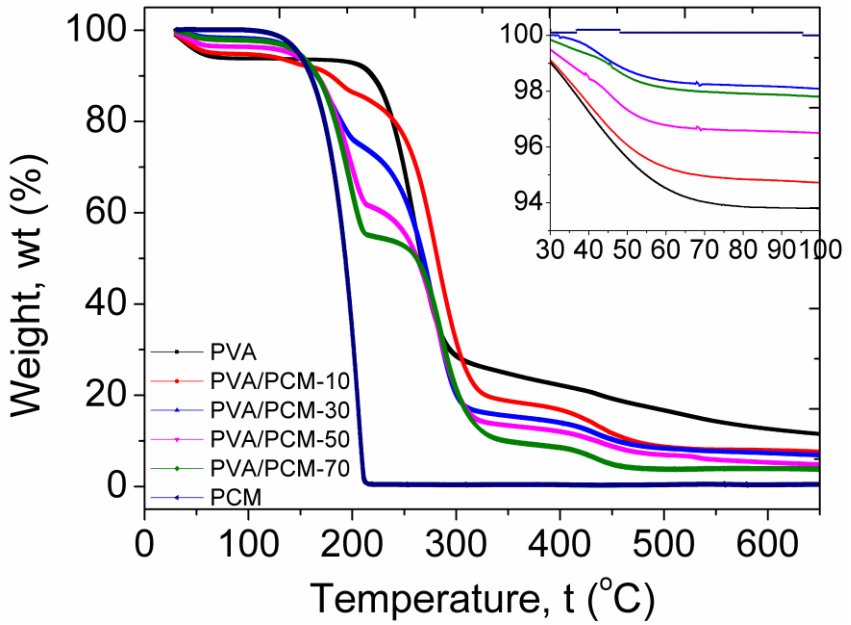
Electrospun mats	t_{om} (°C)	t_m (°C)	t_{oc} (°C)	t_{c1} (°C)	t_{c2} (°C)	t_{c3} (°C)	t_{c4} (°C)
Raw PCM	36.65	40.34	33.08	30.05	-	-	-
PCM-10 (9 w/w% PVA)	34.81	37.29	33.30	32.50	29.68	26.89	22.81
PCM-30 (9 w/w% PVA)	35.02	37.95	32.01	31.26	26.48	-	-
PCM-50 (9 w/w% PVA)	35.09	38.25	32.39	32.00	25.72	-	-
PCM-70 (9 w/w% PVA)	35.29	38.46	32.83	32.21	26.51	-	-

	Electrospun mats	H_{m1} (J/g)	H_{m10} (J/g)	H_{c1} (J/g)	H_{c10} (J/g)
	Raw PCM	221.2	221.5	221.8	221.5
9w/w % PVA	PCM-10	17.96	17.81	18.18	18.77
	PCM-30	48.70	48.06	48.78	49.27
	PCM-50	73.41	72.37	71.89	72.34
	PCM-70	84.72	84.41	83.83	84.58
7w/w% PVA	PCM-10	15.88	15.88	15.58	15.68
	PCM-30	44.05	43.35	43.89	43.47
	PCM-50	65.08	63.75	64.80	63.62
	PCM-70	80.43	79.92	79.29	78.77
	PCM-90	96.58	96.01	96.19	96.88

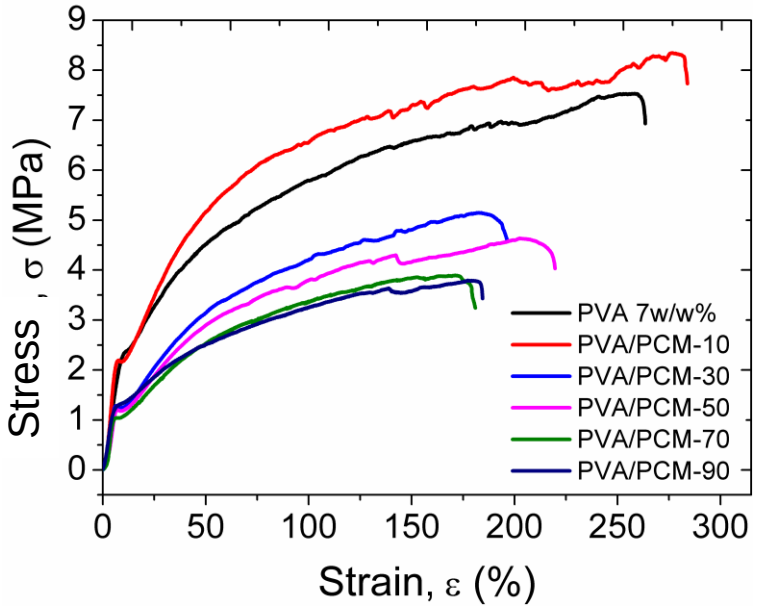


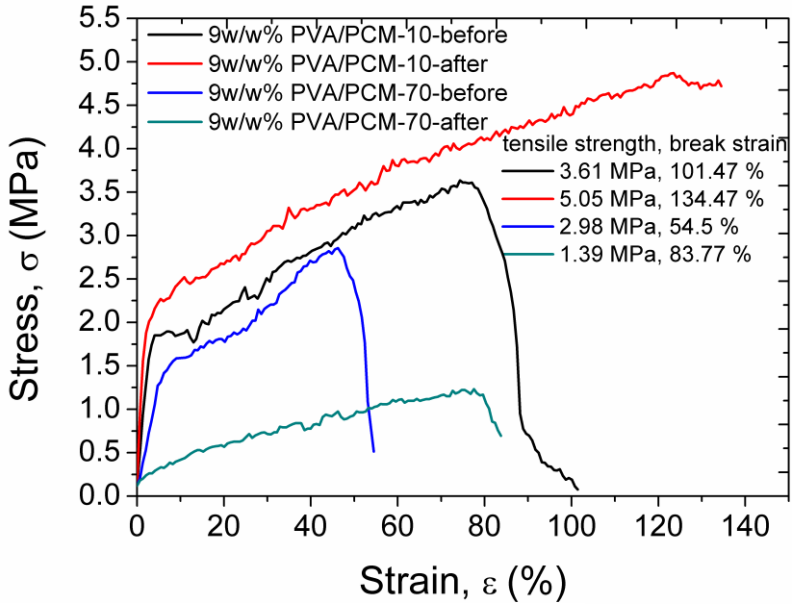
PVA/PCM fiberous mats crystallinity and temperature weight loss





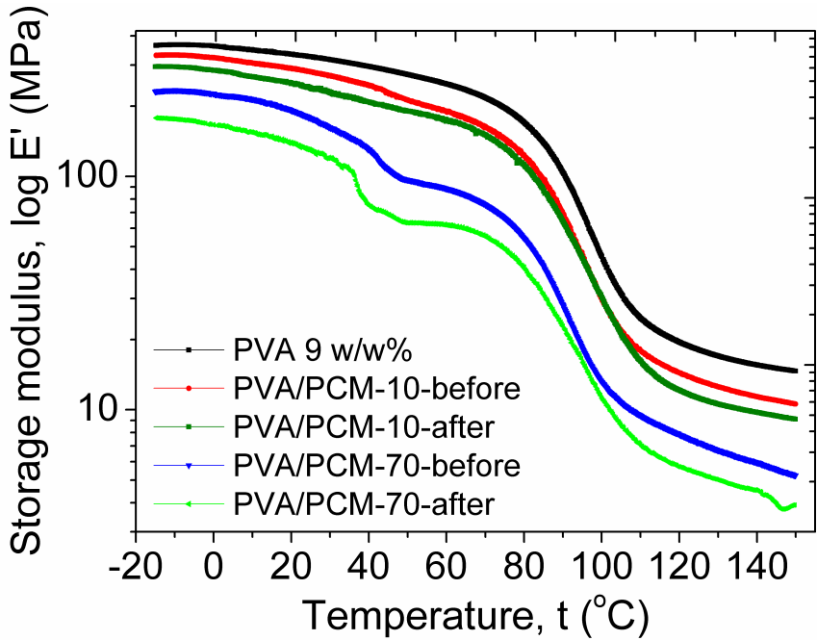
PVA/PCM fibrous mats tensile behaviour

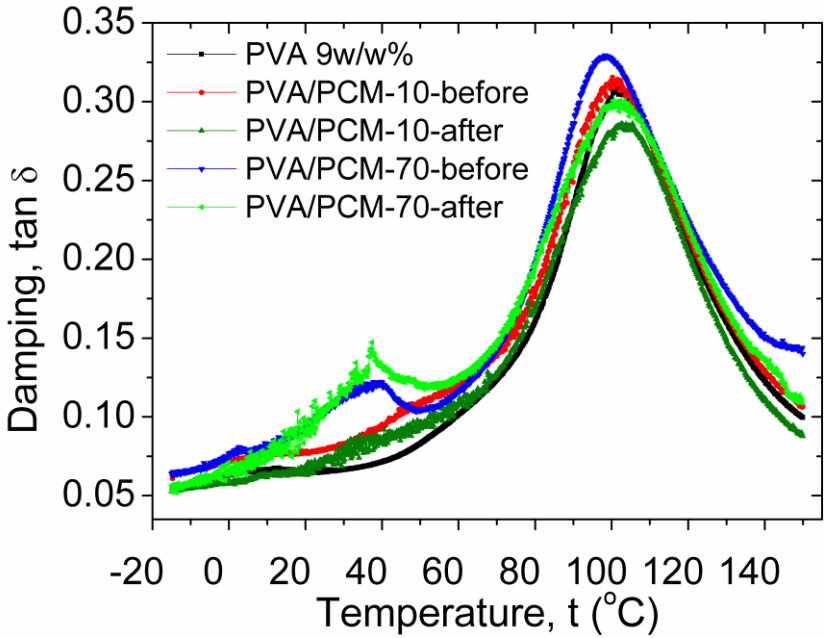




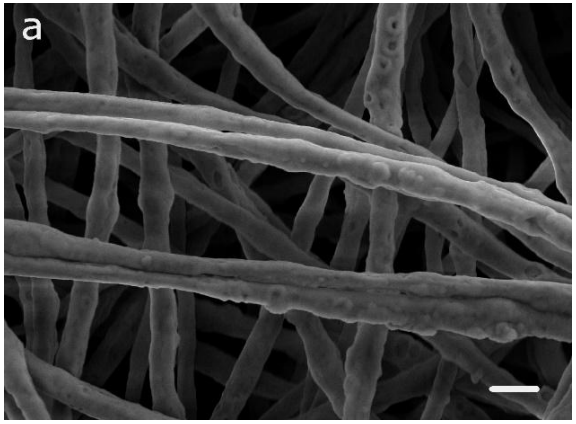
Electrospun mats	Modulus (MPa)	Tensile strength (MPa)	Tensile strain at break (%)
PVA 7w/w%	7.67±1.24	6.59±0.88	257.35±10.33
PVA/PCM-10	8.89±2.04	6.82±1.36	273.50±17.58
PVA/PCM-30	5.36±0.39	4.07±0.48	195.12±5.21
PVA/PCM-50	4.48±0.80	3.51±0.45	217.31±9.28
PVA/PCM-70	4.26±0.51	3.33±0.34	190.50±16.97
PVA/PCM-90	4.43±1.07	2.92±0.55	182.16±7.83

PVA/PCM fibrous mats dynamic mechanical behaviour - before and after heat treatment

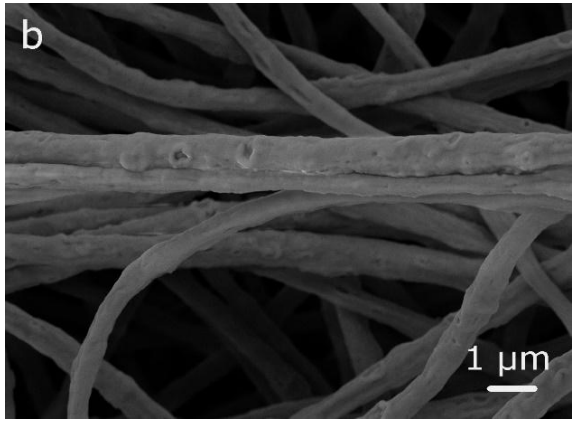




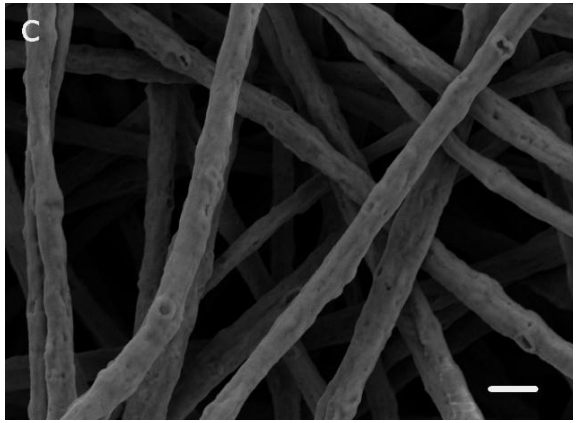
7w/w% PVA/PCM-70 fibers morphology after heat exposure



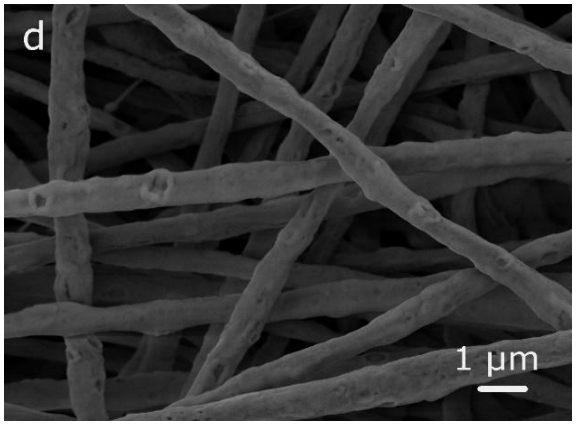
50 °C, 30 min



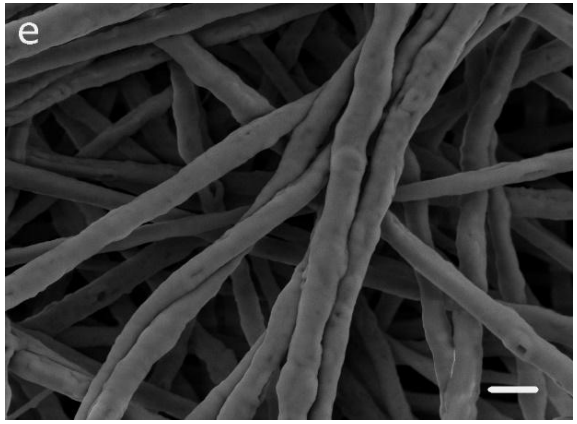
50 °C, 2 h



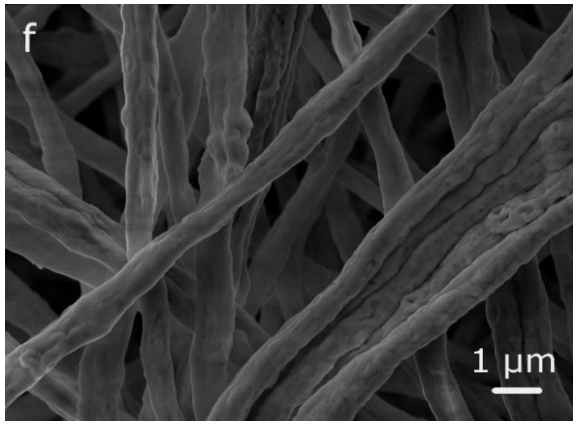
60 °C, 30 min



60 °C, 2 h

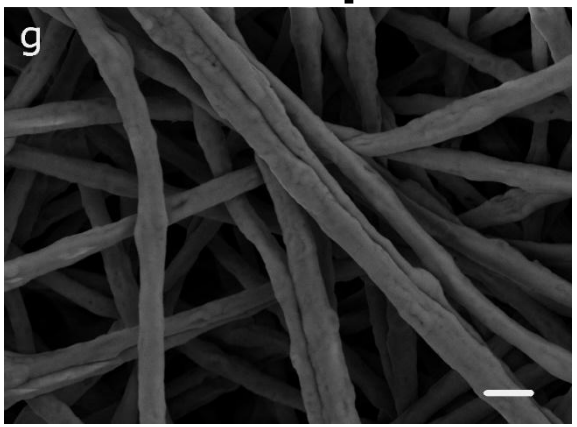


70 °C, 30 min

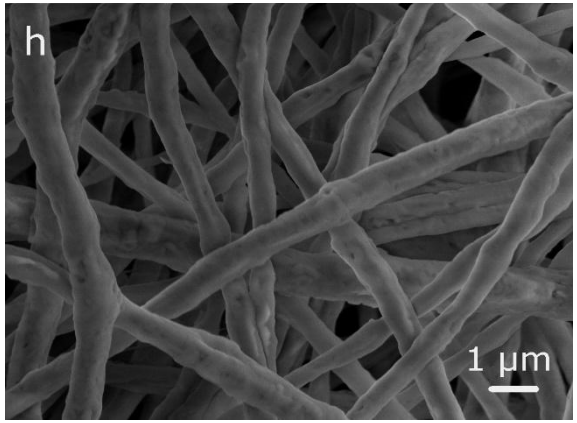


70 °C, 1 h

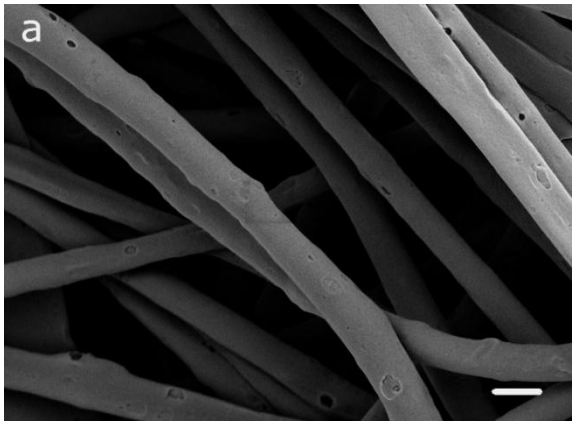
7w/w% (g, h) & 9 w/w% (a-d) PVA/PCM fibers morphology after heat exposure



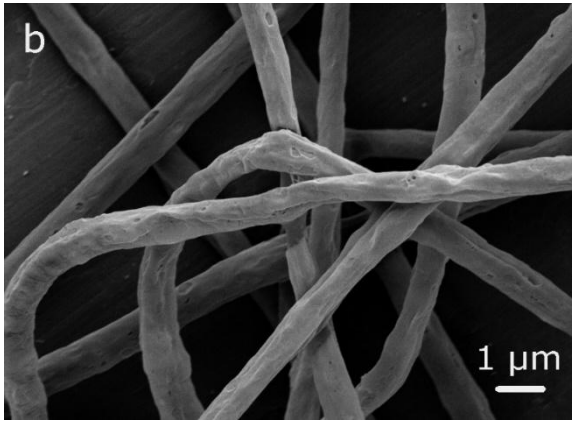
80 °C, 5 min



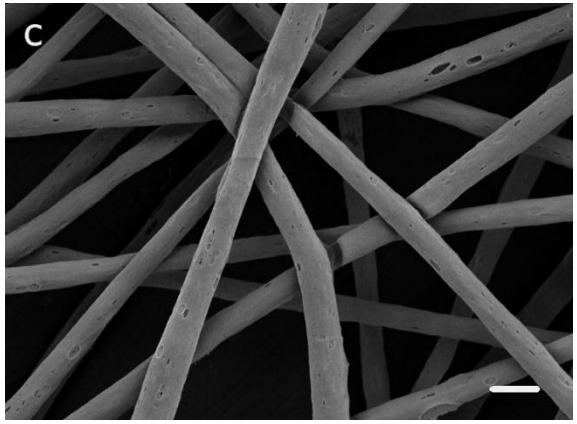
80 °C, 2 h



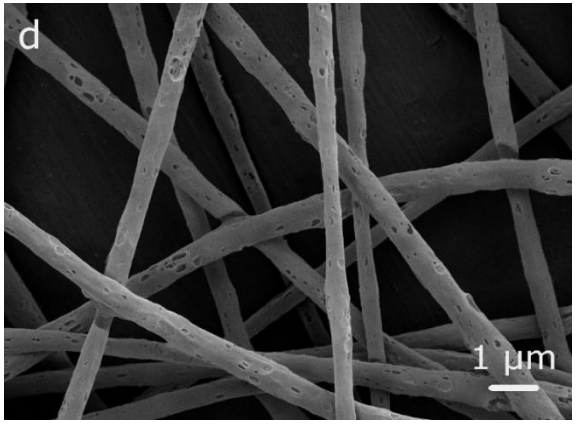
65 °C, 10 cycles



65 °C, 10 cycles

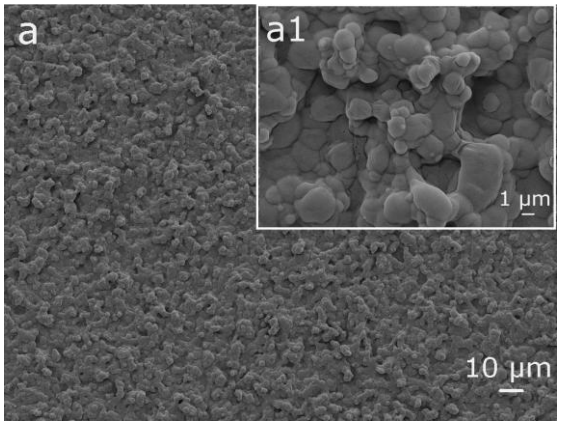


65 °C, 10 cycles

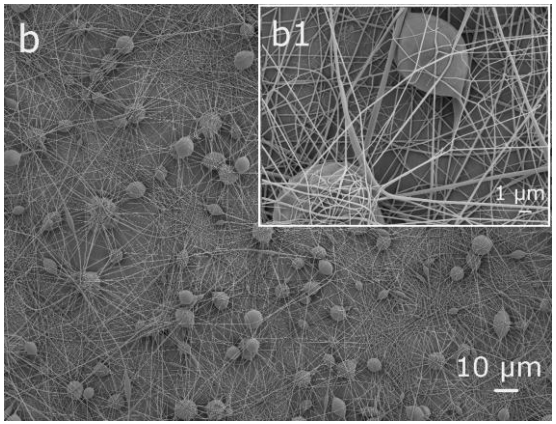


65 °C, 10 cycles

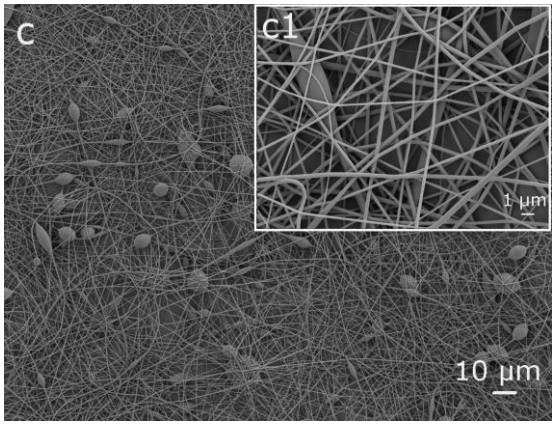
PVDF/PCL (low Mw) fibers morphology



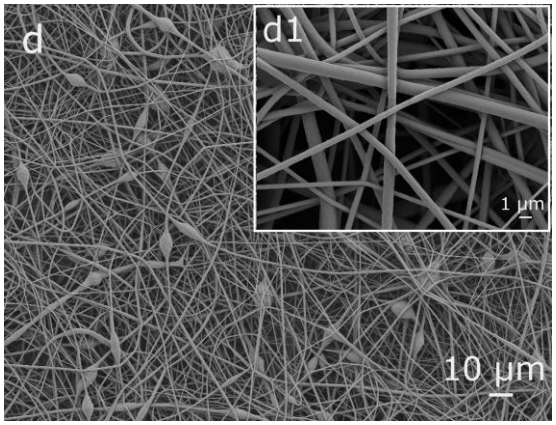
14% PCL



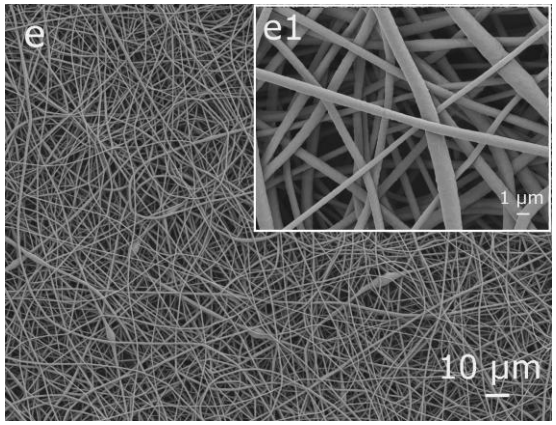
14% PVDF



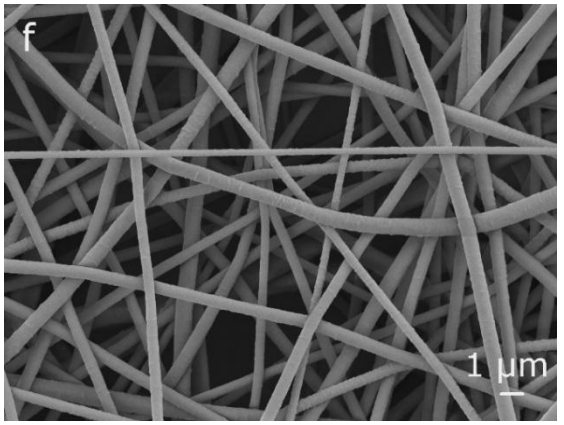
14%PVDF/PCL:100/25



14%PVDF/PCL:100/50

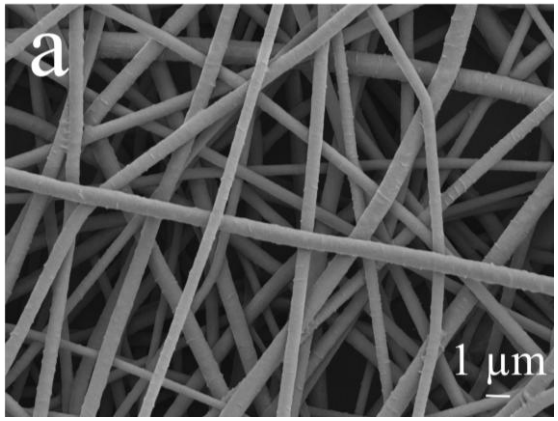


14%PVDF/PCL:100/75

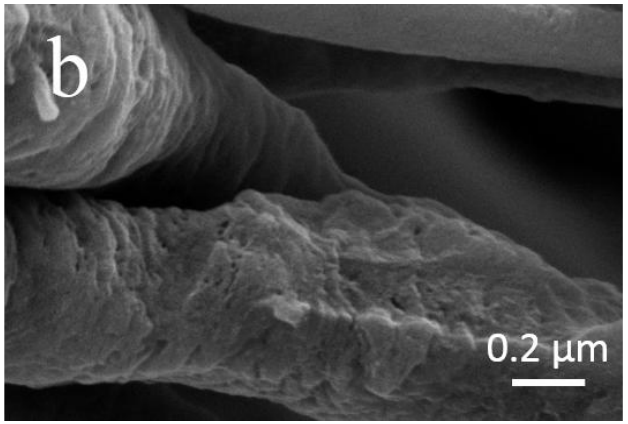


14%PVDF/PCL:100/100

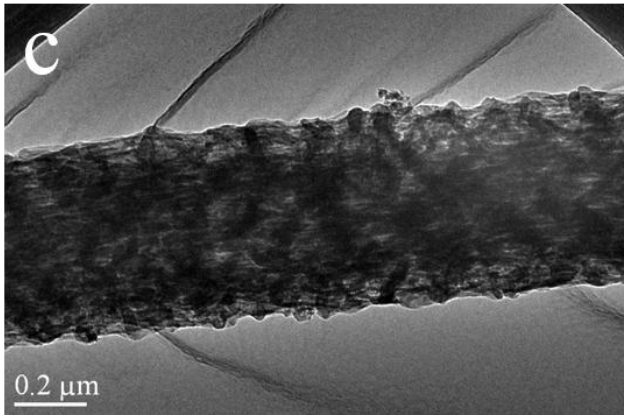
14% PVDF/PCL (low Mw) 100/100 fibers morphology



pure

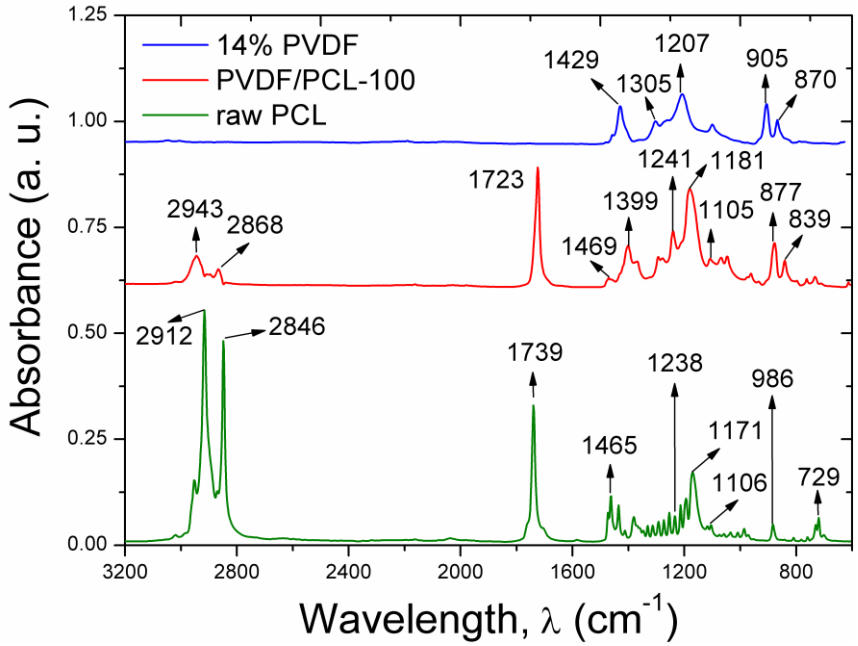


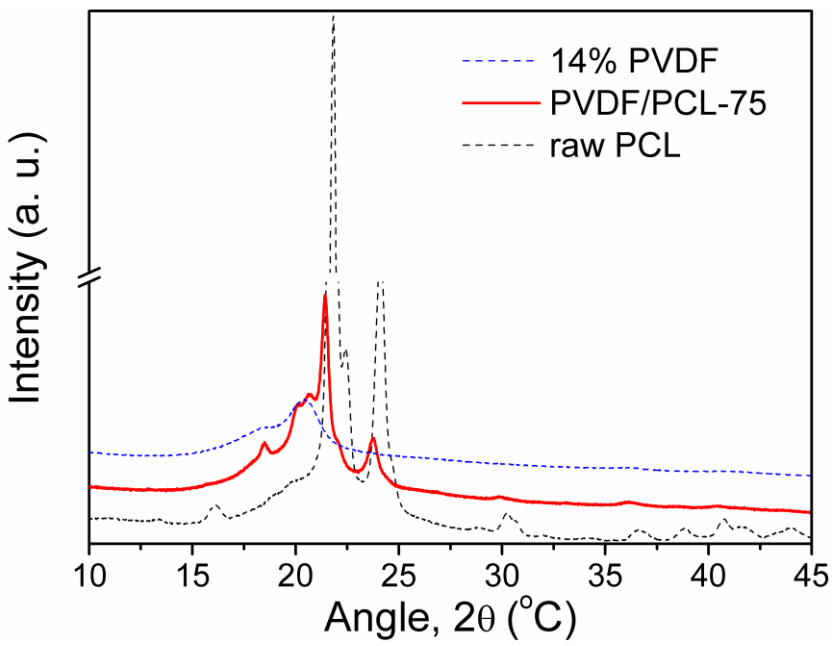
after PCL extraction



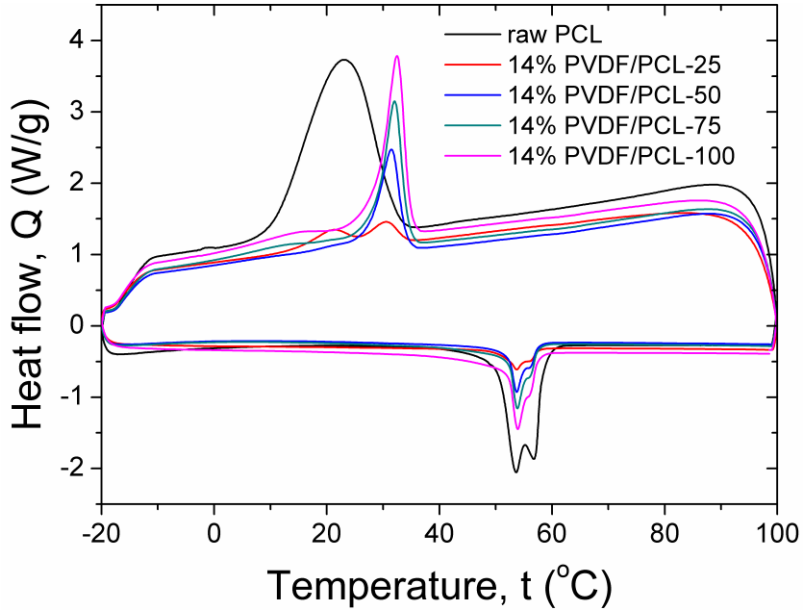
after PCL extraction

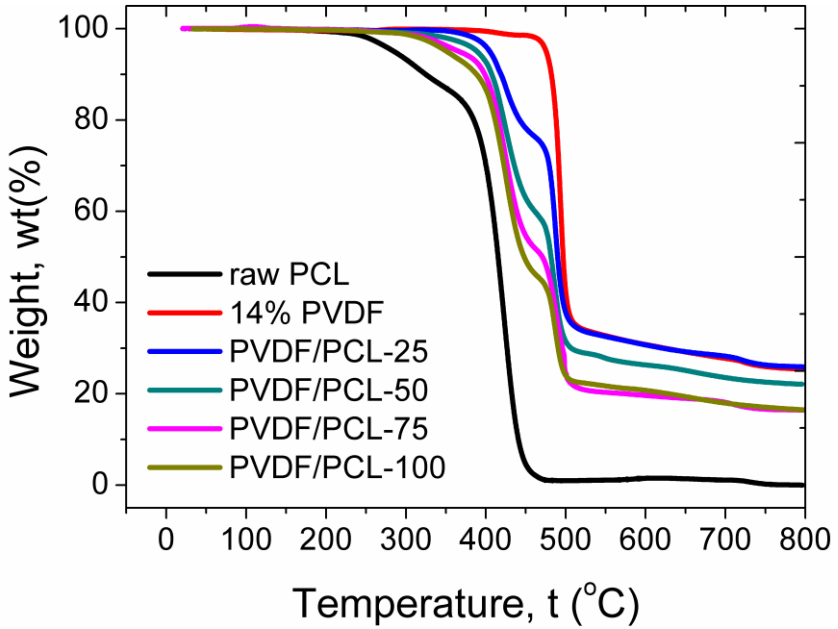
PVDF/PCL (low Mw) fibrous mats chemical composition and crystallinity





PVDF/PCL (low Mw) fibrous mats heat managing performance and thermal stability

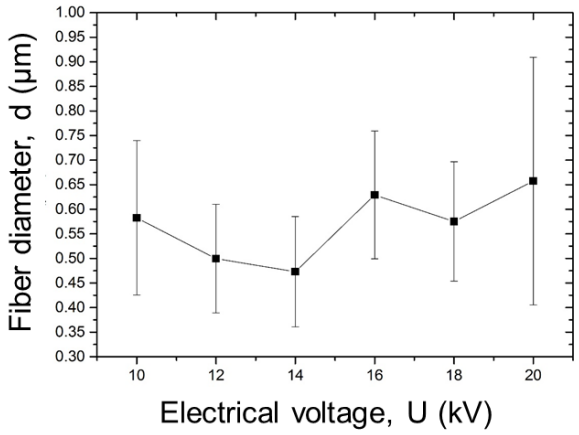


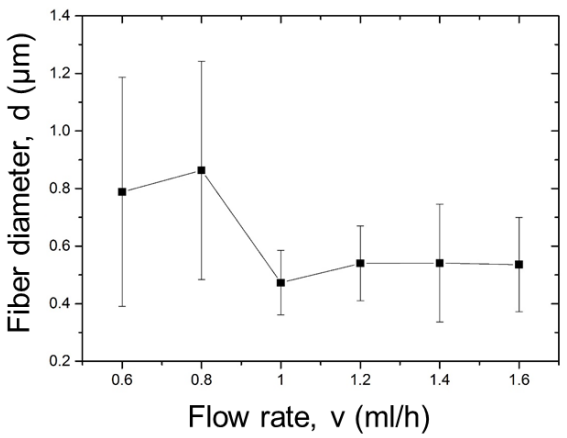


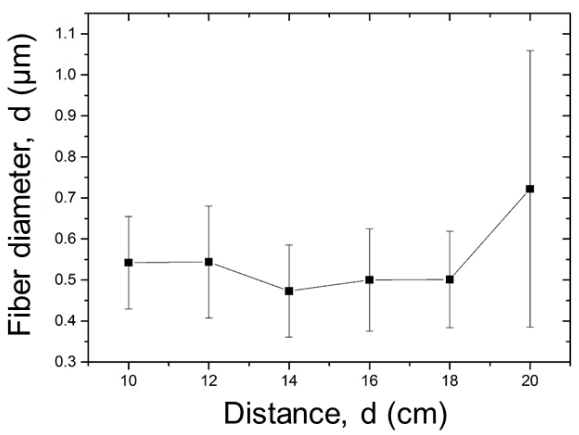
Samples	H_{m1} (J/g)	H_{m10} (J/g)	H_{c1} (J/g)	H_{c10} (J/g)
Raw PCL	73.02	70.94	71.32	70.43
14% PVDF/PCL-25	9.31	9.33	8.68	7.96
14% PVDF/PCL-50	18.02	17.56	18.10	17.60
14% PVDF/PCL-75	24.91	24.90	25.56	24.56
14% PVDF/PCL-100	33.90	34.10	33.08	32.16

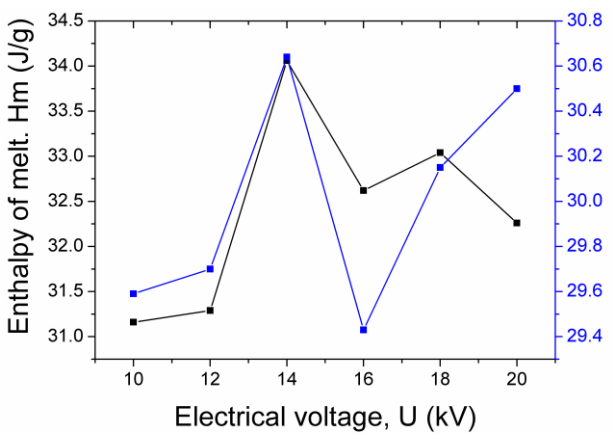
Samples	t_{om} (°C)	t_{m1} (°C)	t_{m2} (°C)	t_{oc} (°C)	t_{c1} (°C)	t_{c2} (°C)
Raw PCL	50.48	53.63	56.88	32.27	22.91	-
14% PVDF/PCL-25	51.21	53.72	-	34.23	30.51	21.22
14% PVDF/PCL-50	52.11	53.73	-	34.10	31.48	-
14% PVDF/PCL-75	52.17	53.87	-	34.46	32.07	-
14% PVDF/PCL-100	52.09	53.92	-	34.90	32.52	-

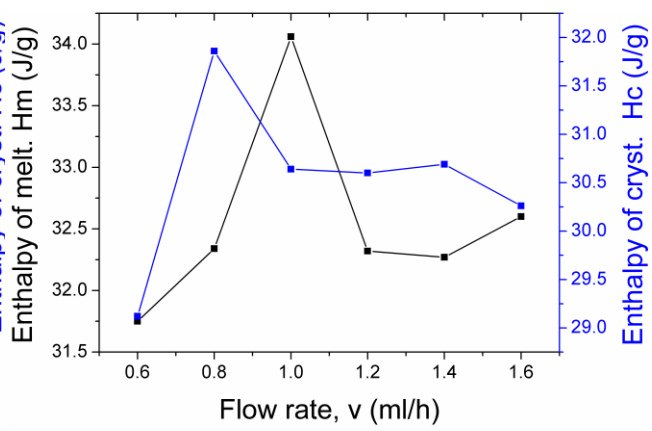
Fibers diameter and heat enthalpies relation

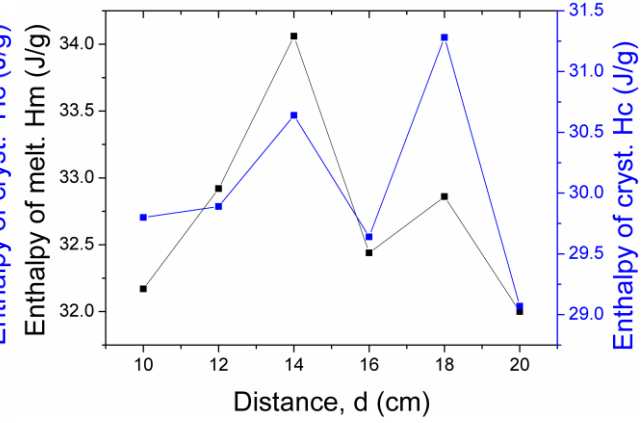




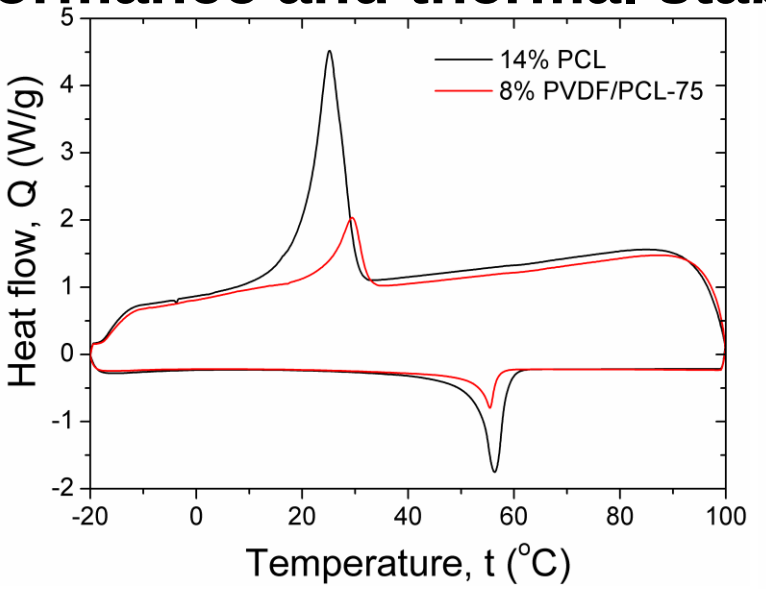


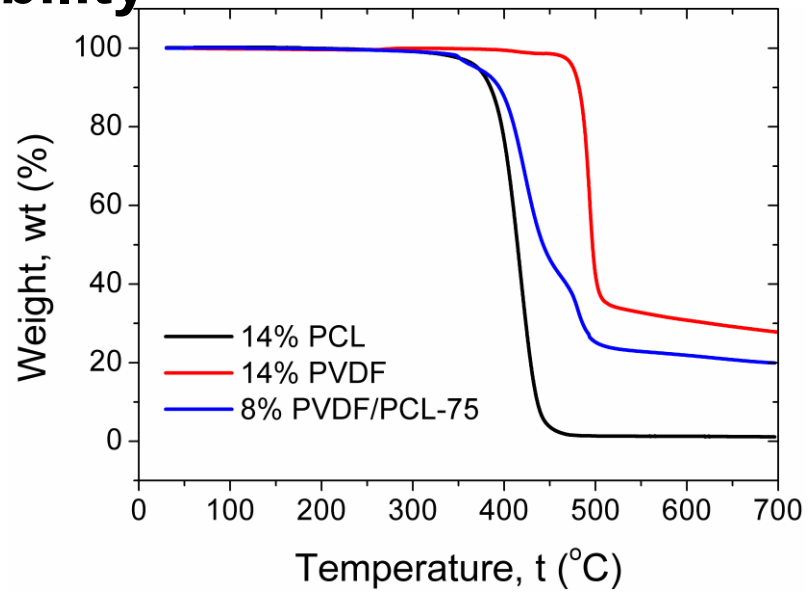






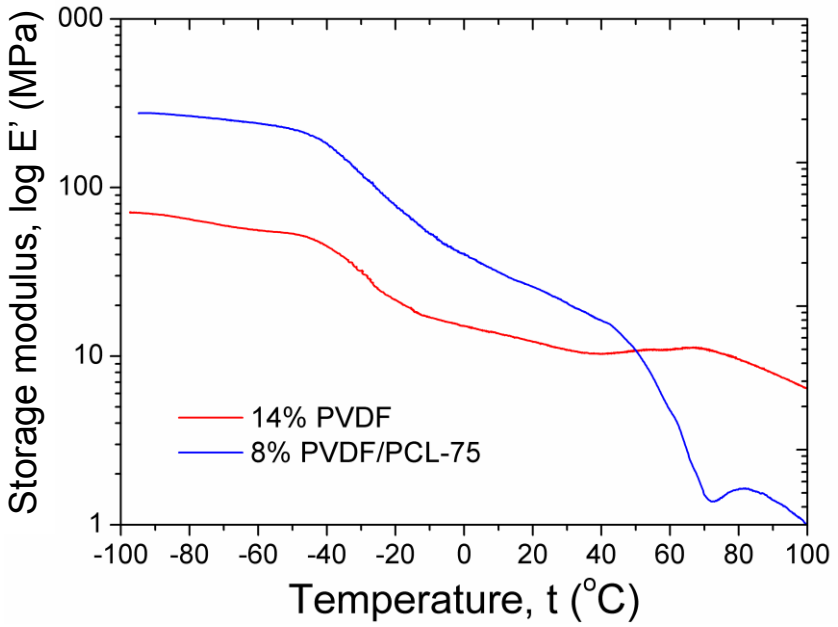
PVDF/PCL (high Mw) fibrous mats heat managing performance and thermal stability

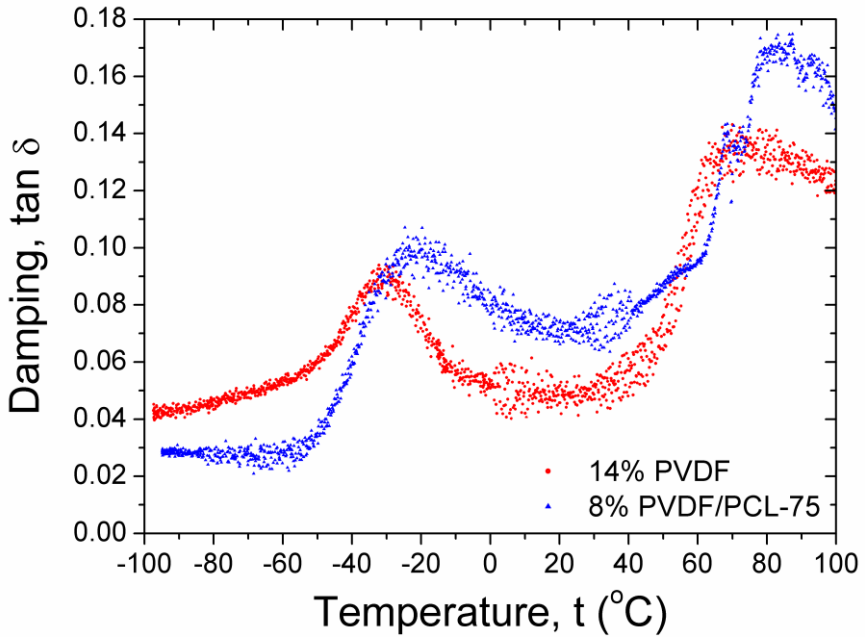




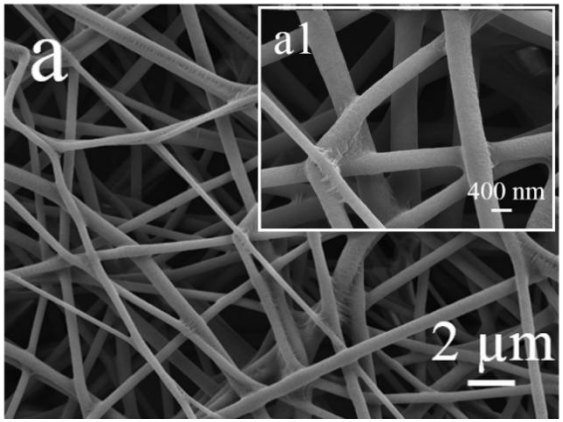
Samples	t_{om} (°C)	t_m (°C)	t_{oc} (°C)	t_c (°C)
14% PCL	53.08	56.35	30.25	25.18
8% PVDF/PCL-75	52.79	55.44	32.42	29.43
Samples	H_{m1} (J/g)	H_{m10} (J/g)	H_{c1} (J/g)	H_{c10} (J/g)
14% PCL	55.17	54.77	58.13	56.54
8% PVDF/PCL-75	19.77	19.51	19.99	19.81

PVDF/PCL (high Mw) fibrous mats dynamic mechanical behaviour

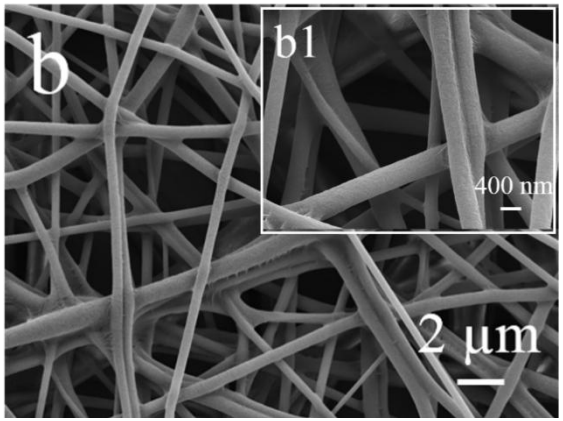




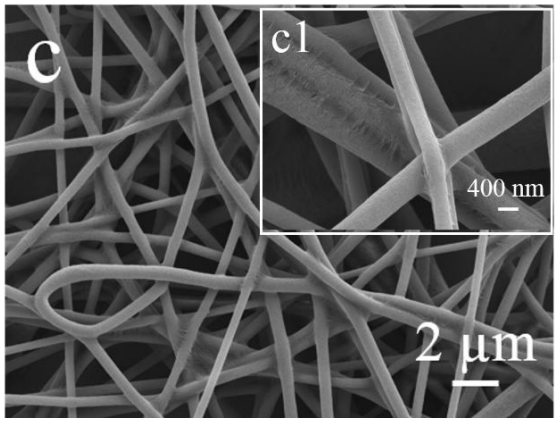
PVDF/PCL (low Mw) fibers morphology after heat exposure



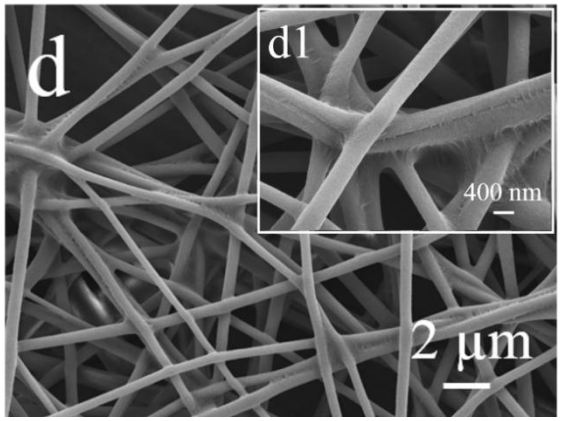
65 °C, 30 min



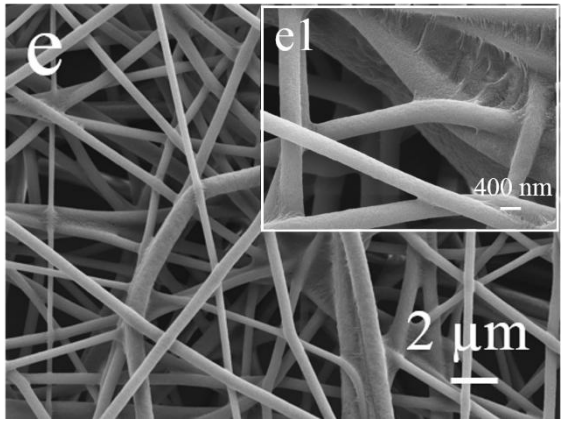
65 °C, 1 h



65 °C, 2 h



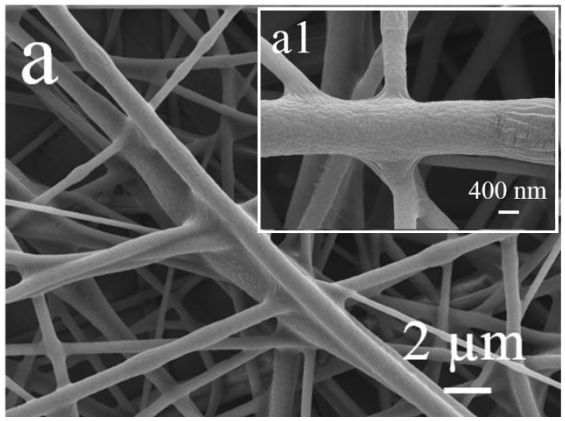
65 °C, 4 h



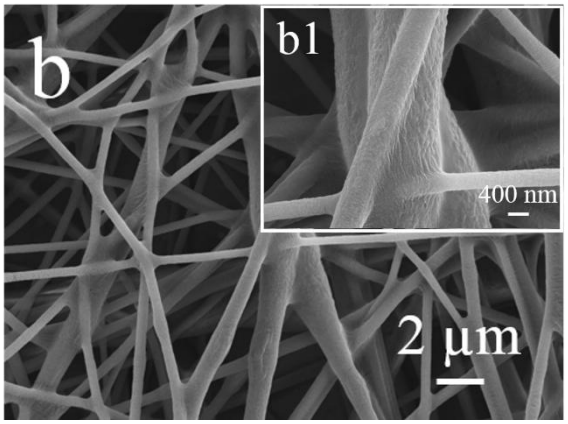
65 °C, 24 h

14% PVDF/PCL:100/100

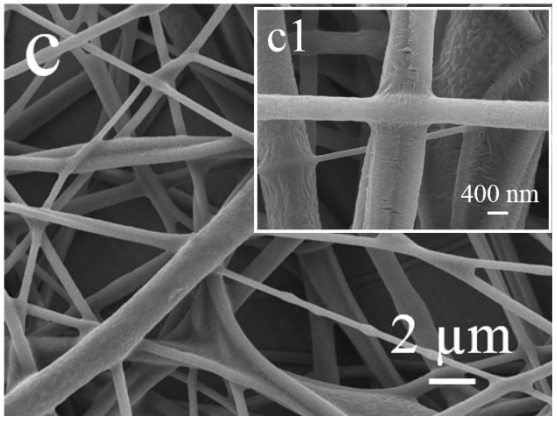
PVDF/PCL (high Mw) fibers morphology after heat exposure



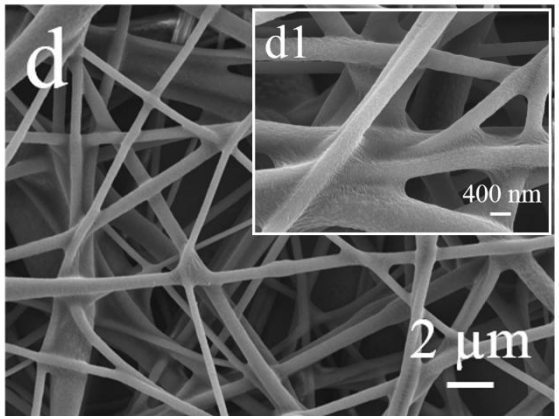
65 °C, 30 min



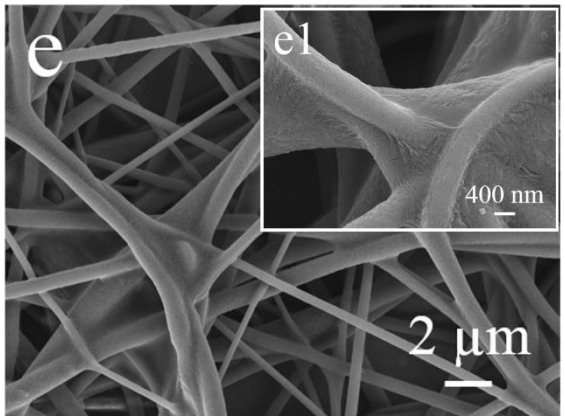
65 °C, 1 h



65 °C, 2 h



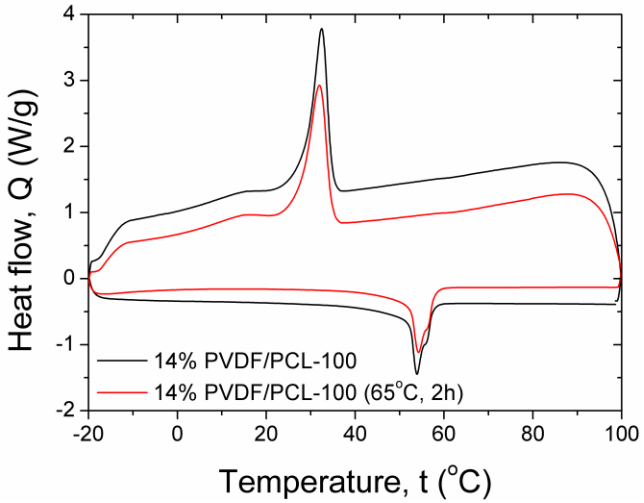
65 °C, 4 h

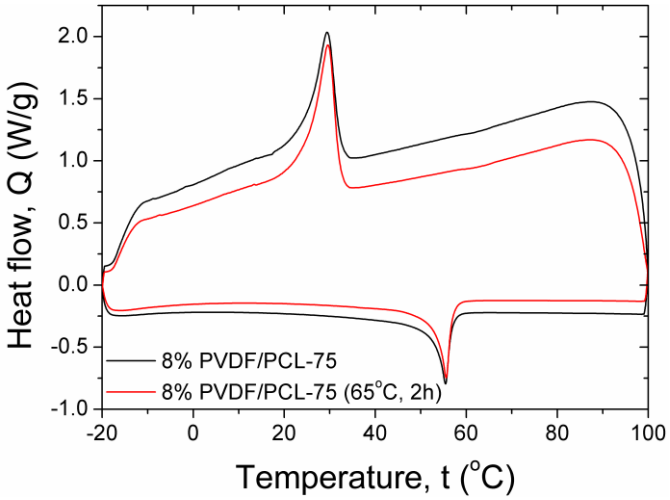


65 °C, 24 h

8% PVDF/PCL:100/75

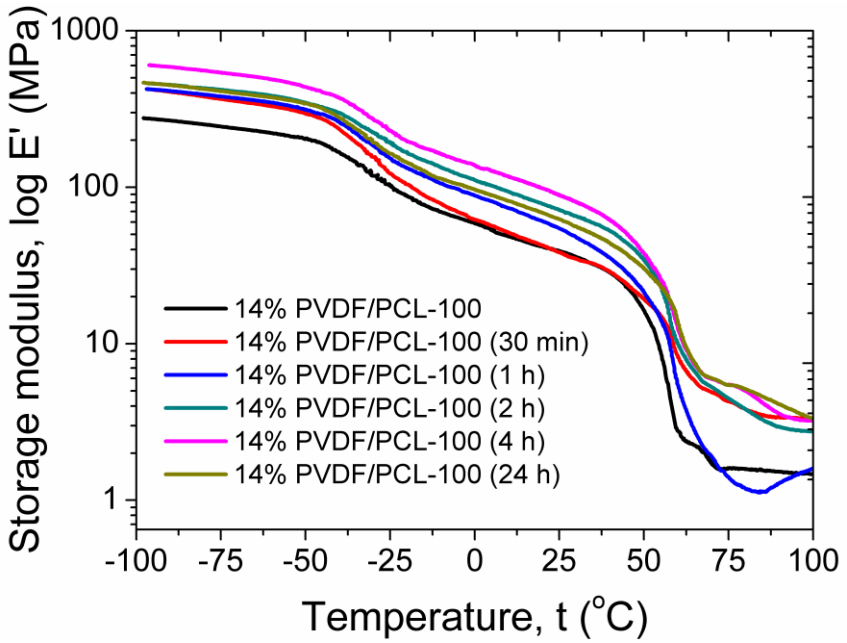
PVDF/PCL (low Mw) fibrous mats heat managing performance after heat exposure

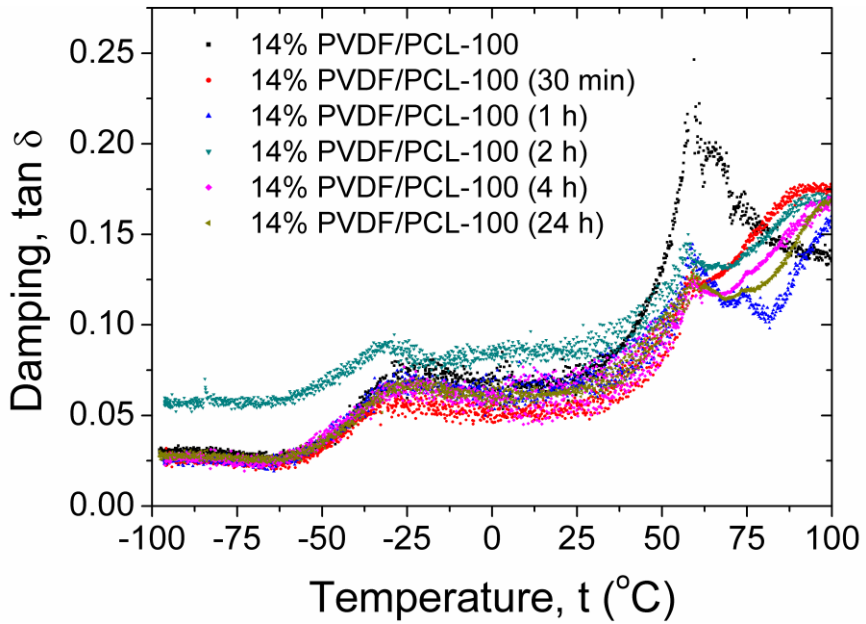




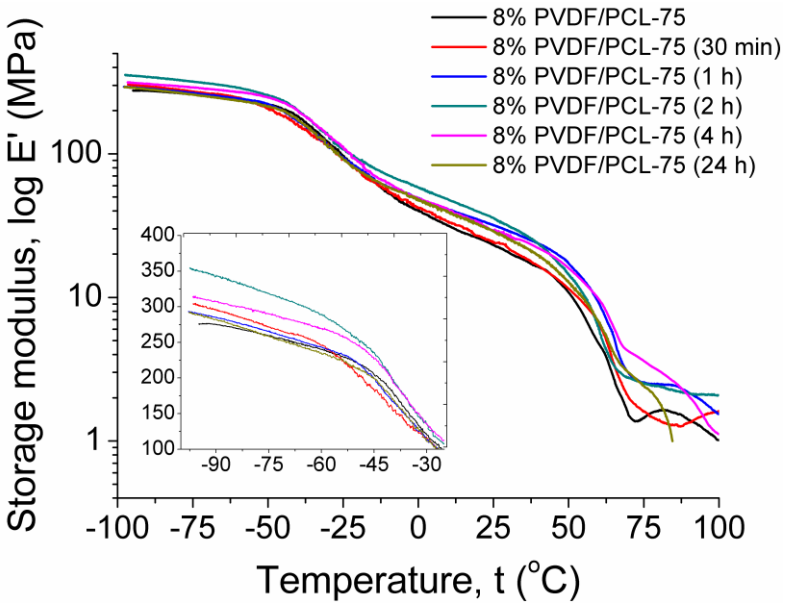
Samples	t_{om} (°C)	t_m (°C)	t_{oc} (°C)	t_c (°C)
14% PVDF/PCL-100	52.10	53.92	34.89	32.52
14% PVDF/PCL-100 (65°C, 2h)	52.17	54.28	33.46	31.91
8% PVDF/PCL-75	52.79	55.44	32.42	29.43
8% PVDF/PCL-75 (65°C, 2h)	53.08	55.59	32.32	29.58
Samples	H_{m1} (J/g)	H_{m10} (J/g)	H_{c1} (J/g)	H_{c10} (J/g)
14% PVDF/PCL-100	32.26	33.16	29.46	29.93
14% PVDF/PCL-100 (65°C, 2h)	32.93	33.25	32.77	33.46
8% PVDF/PCL-75	19.77	19.51	19.99	19.81
8% PVDF/PCL-75 (65°C, 2h)	20.16	19.97	22.10	20.52

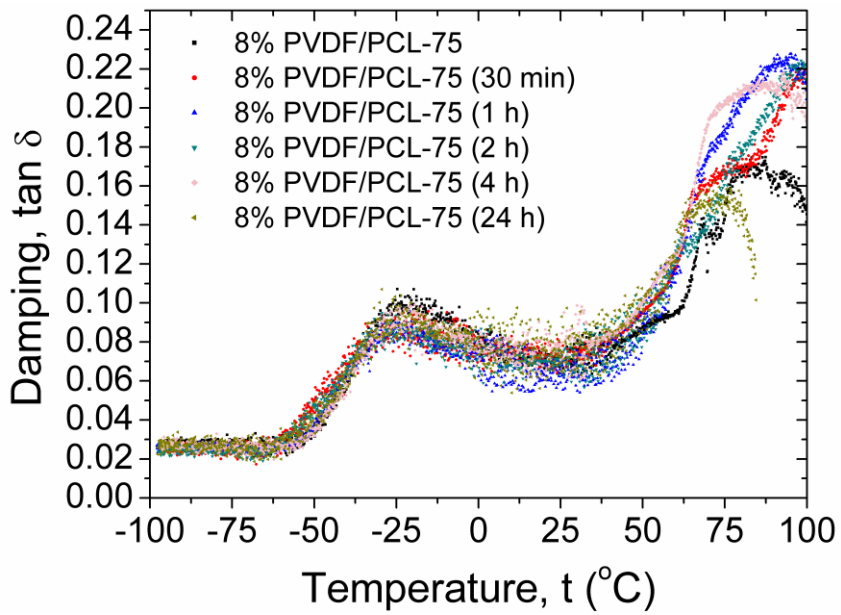
PVDF/PCL (low Mw) fibrous DM behaviour after heat exposure



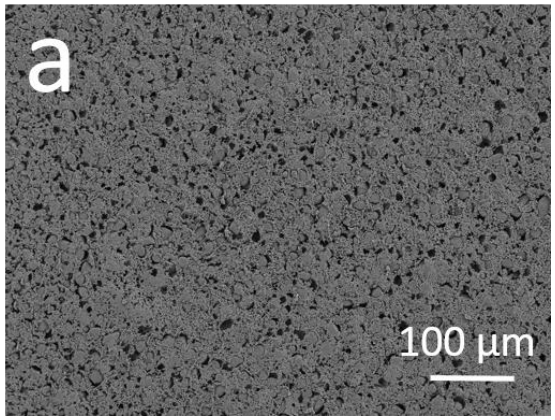


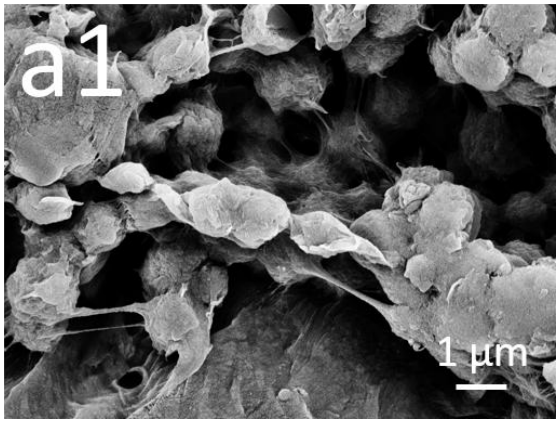
PVDF/PCL (high Mw) fibrous mats DM behaviour after heat exposure



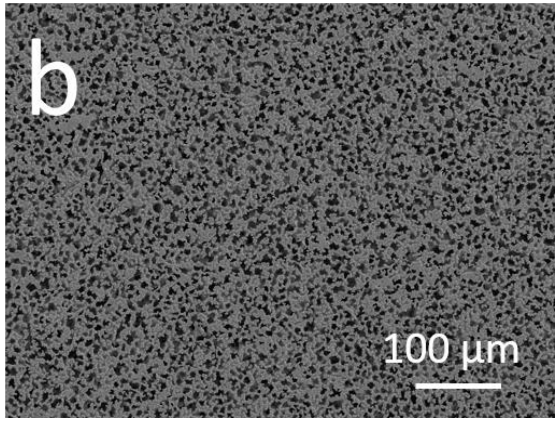


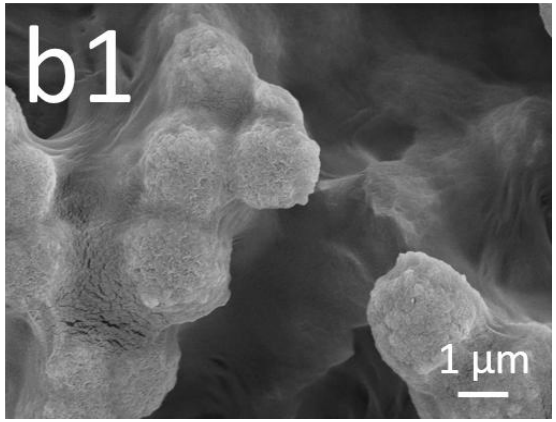
PVDF/PCL (low Mw) cast films before and after heat exposure





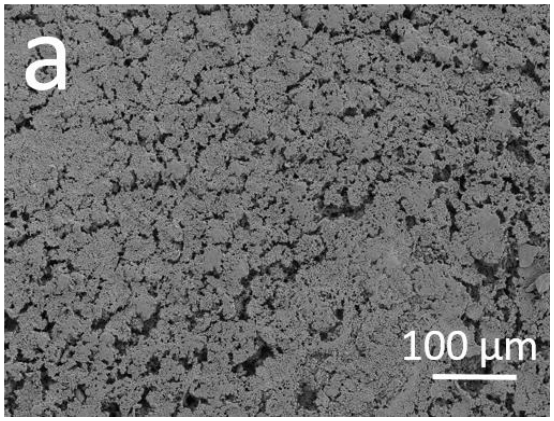
14% PVDF/PCL:100/100 - before

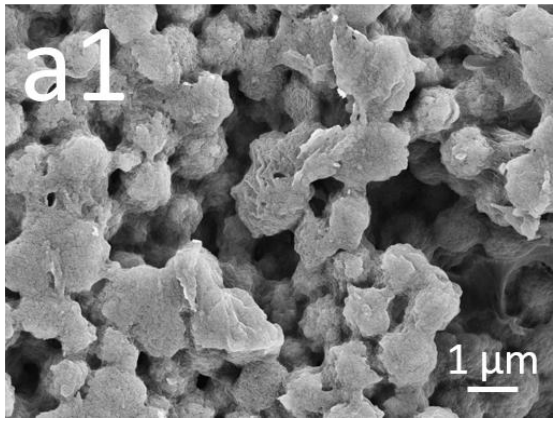




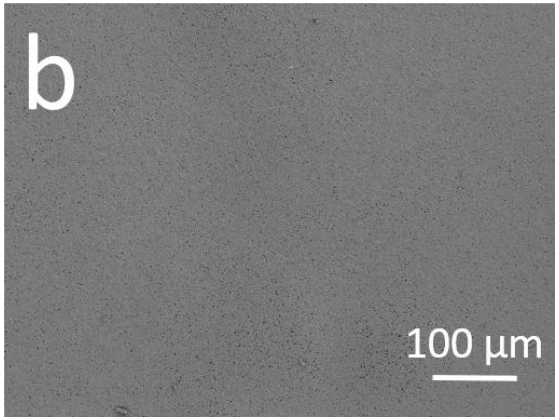
14% PVDF/PCL:100/100 - after heat at 65°C, 24 h

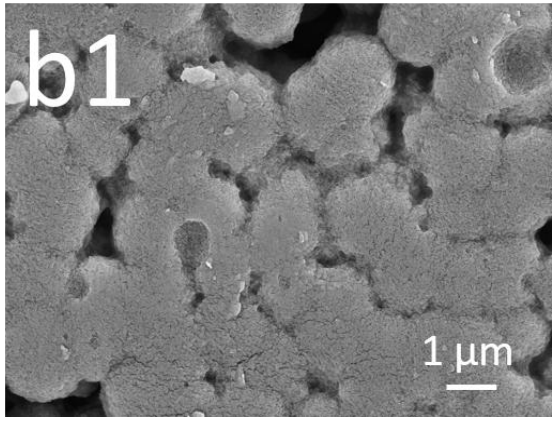
PVDF/PCL (high Mw) cast films before and after heat exposure



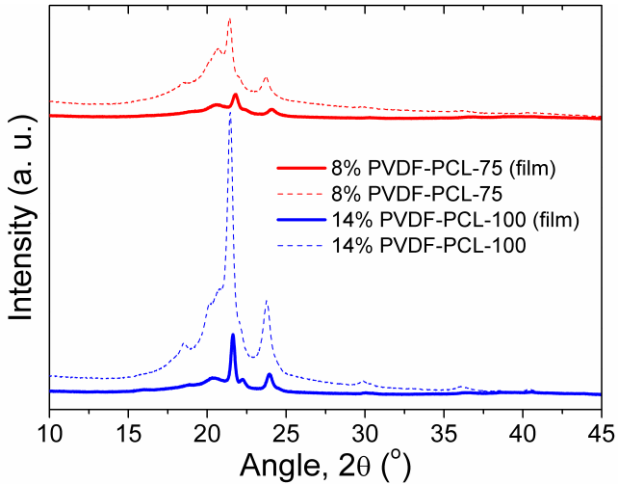


8% PVDF/PCL:100/75 - before





8% PVDF/PCL:100/75 - after heat at 65°C, 24 h



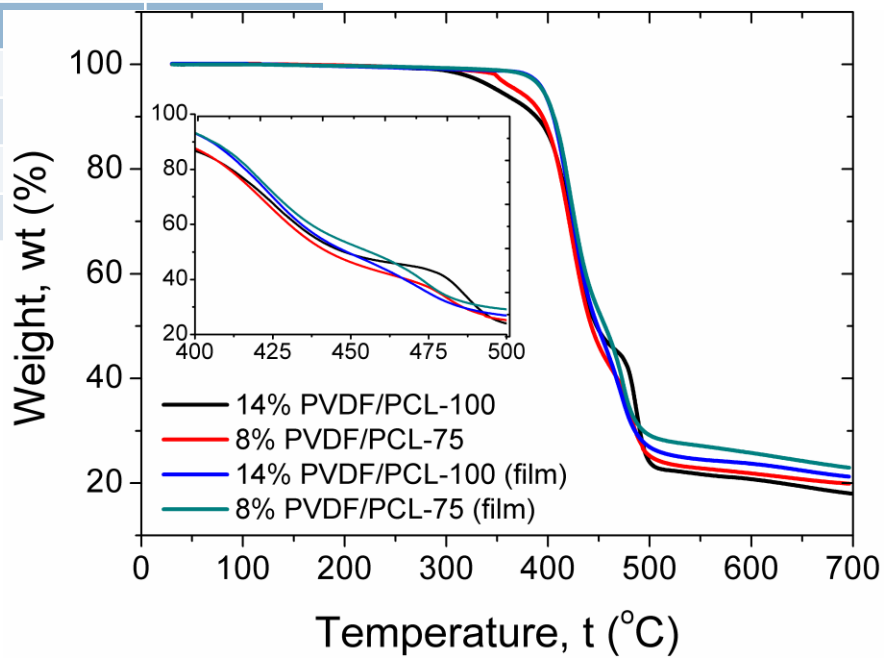
PVDF/PCL (low Mw) cast films heat managing performance and thermal stability before and after heat exposure

Samples	t_{om} (°C)	t_m (°C)	t_{oc} (°C)	t_c (°C)
14% PVDF/PCL-100	52.10	53.92	34.89	32.52
14% PVDF/PCL-100 (cast film)	53.67	55.96	30.78	27.52
8% PVDF/PCL-75	52.79	55.44	32.42	29.43
8% PVDF/PCL-75 (cast film)	53.68	56.30	32.38	28.95

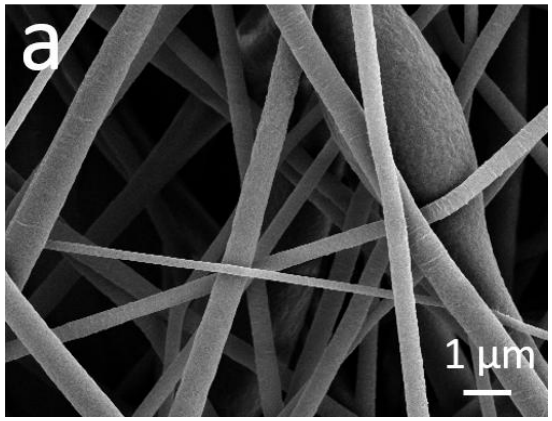
Samples	H_m (J/g)	H_c (J/g)
14% PVDF/PCL-100	32.26	29.46
14% PVDF/PCL-100 (cast film)	19.46	22.11
8% PVDF/PCL-75	19.77	19.99
8% PVDF/PCL-75 (cast film)	22.58	22.88

29 °C – supercooling
8 °C greater

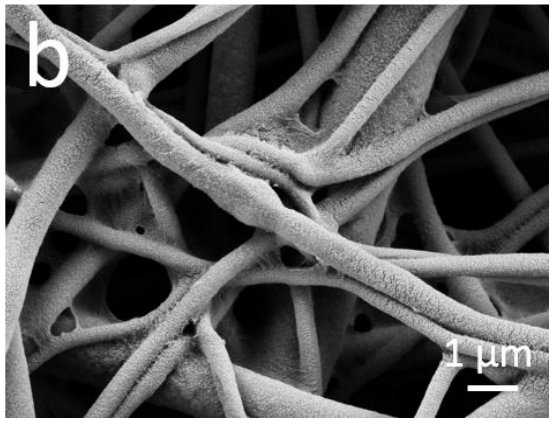
Hm and Hc / 13 J/g and 7 J/g



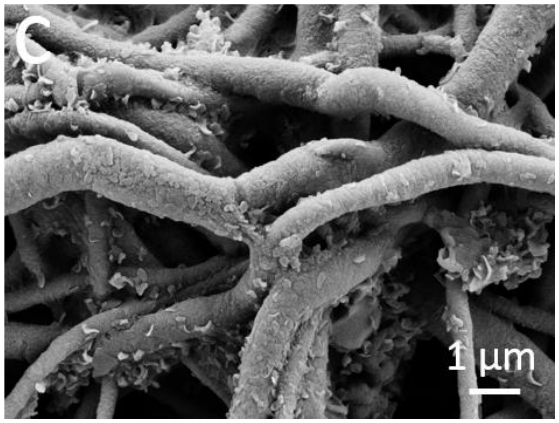
PVDF/PCL fibers morphology after coating



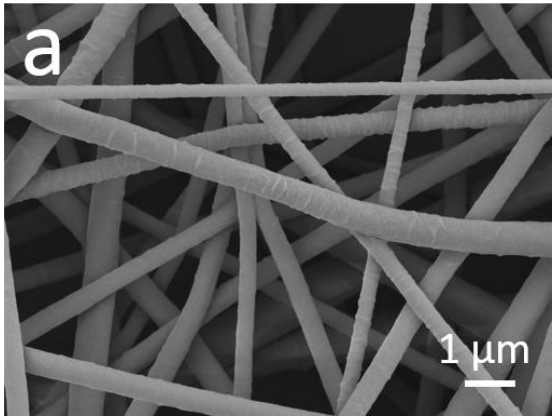
14% PVDF/PCL:100/100



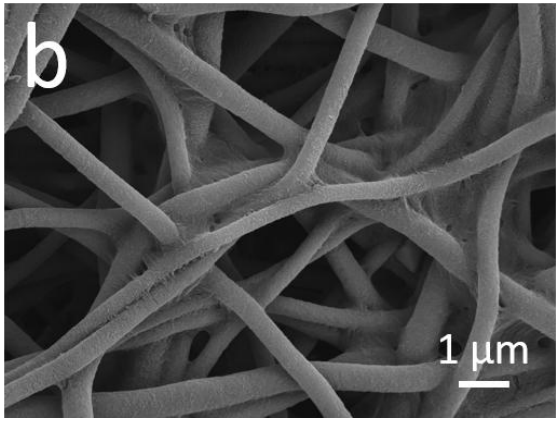
14% PVDF/PCL:100/100-PPy



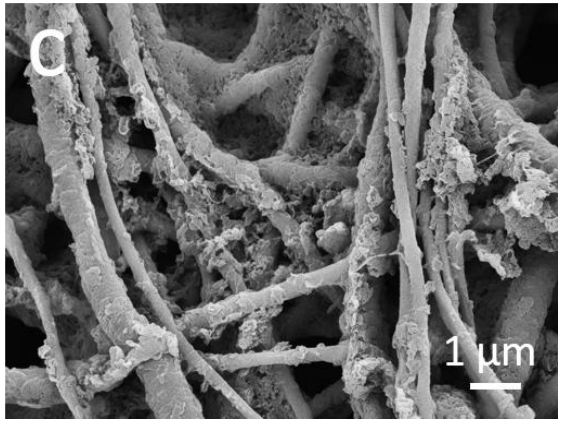
14%PVDF/PCL:100/100-PPy/r-GO



8% PVDF/PCL:100/75

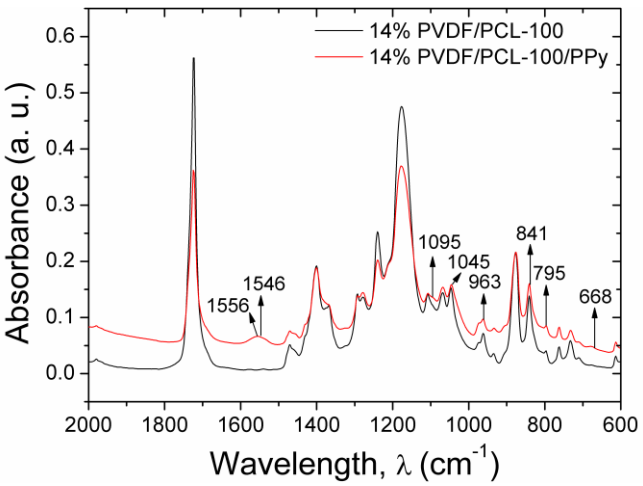


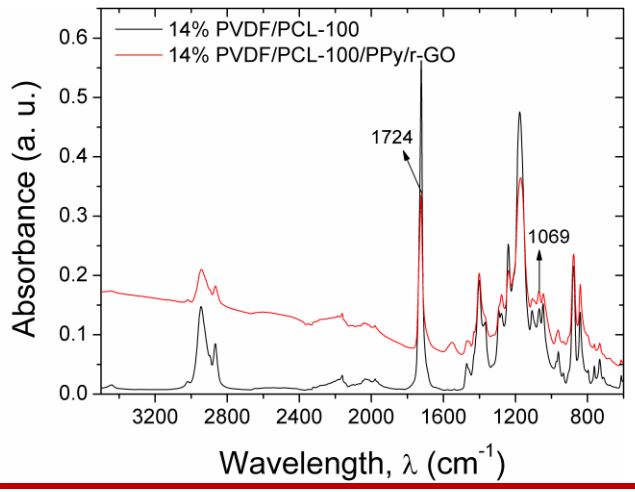
8% PVDF/PCL:100/75-PPy

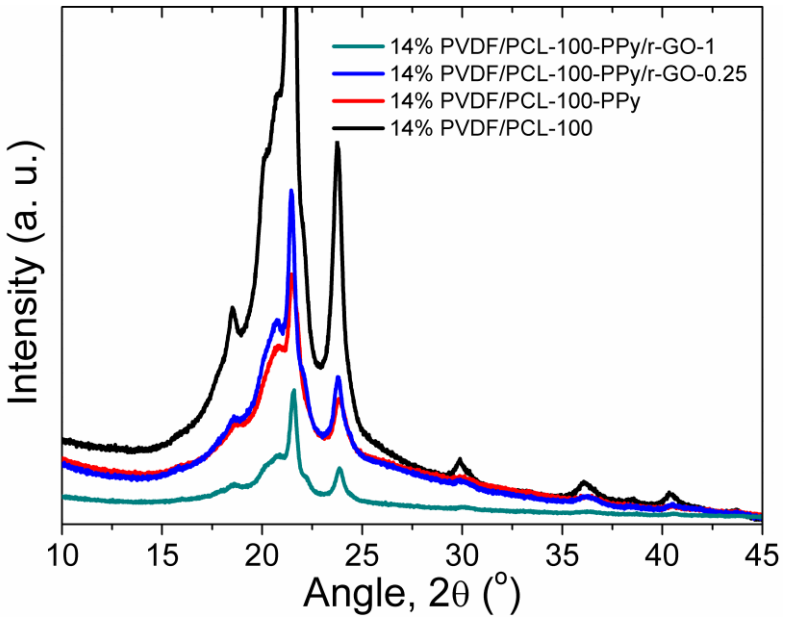


8%PVDF/PCL:100/75-PPy/r-GO

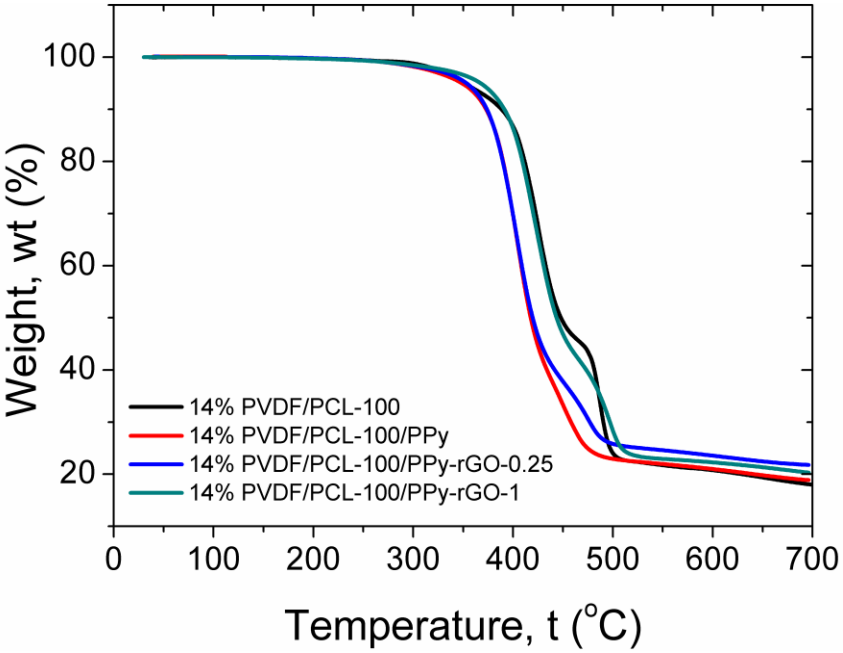
PVDF/PCL (low Mw) fibrous mats FTIR and XRD spectra after coating

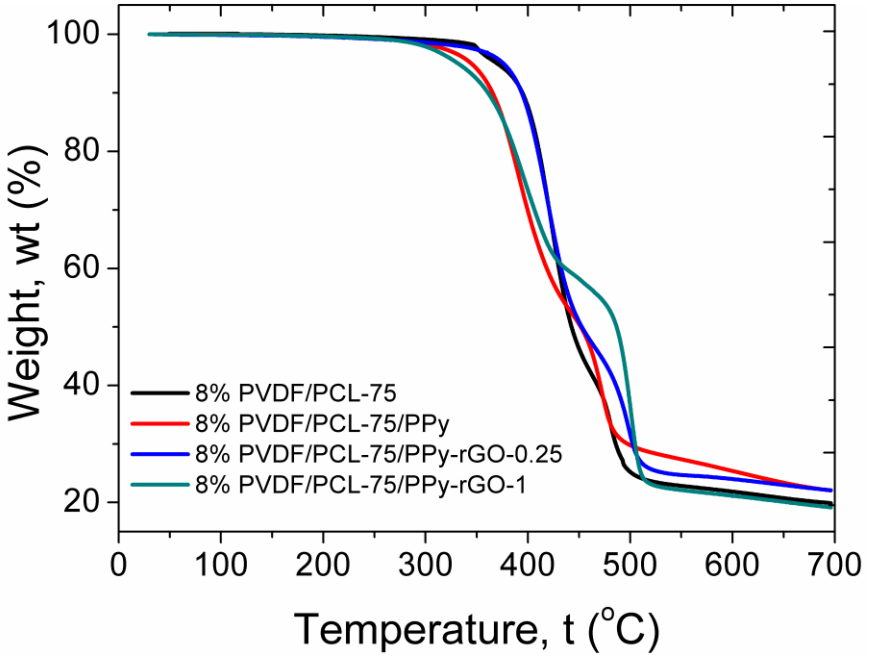




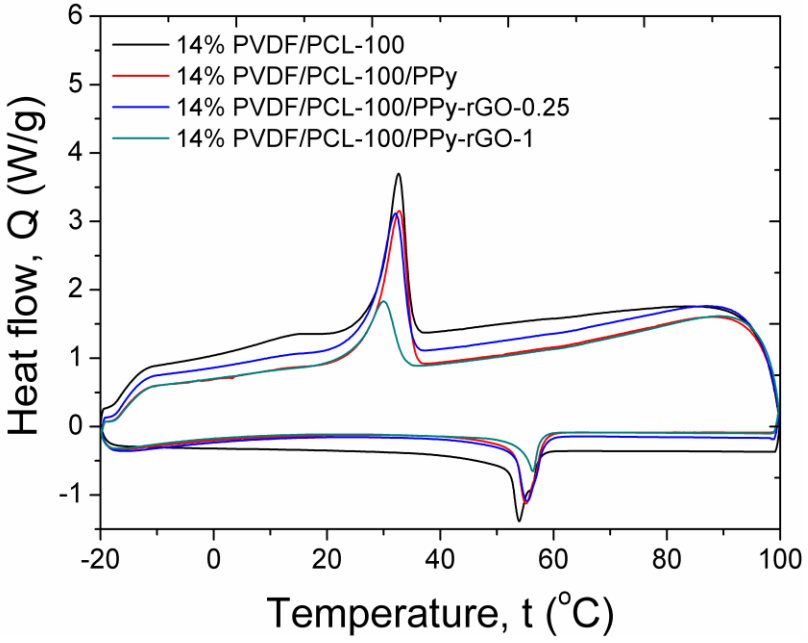


Coating influence on temperature weight loss



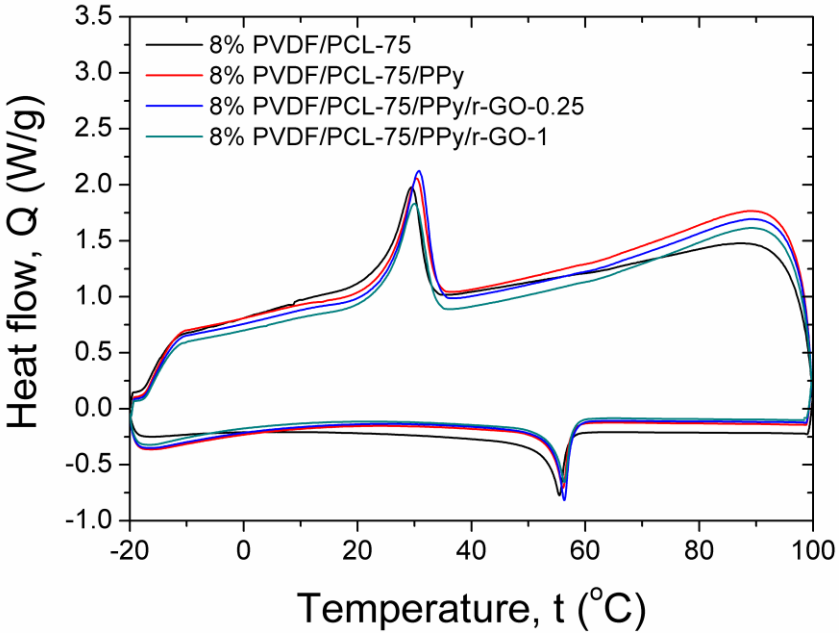


PVDF/PCL (low Mw) fibrous mats heat managing performance after coating



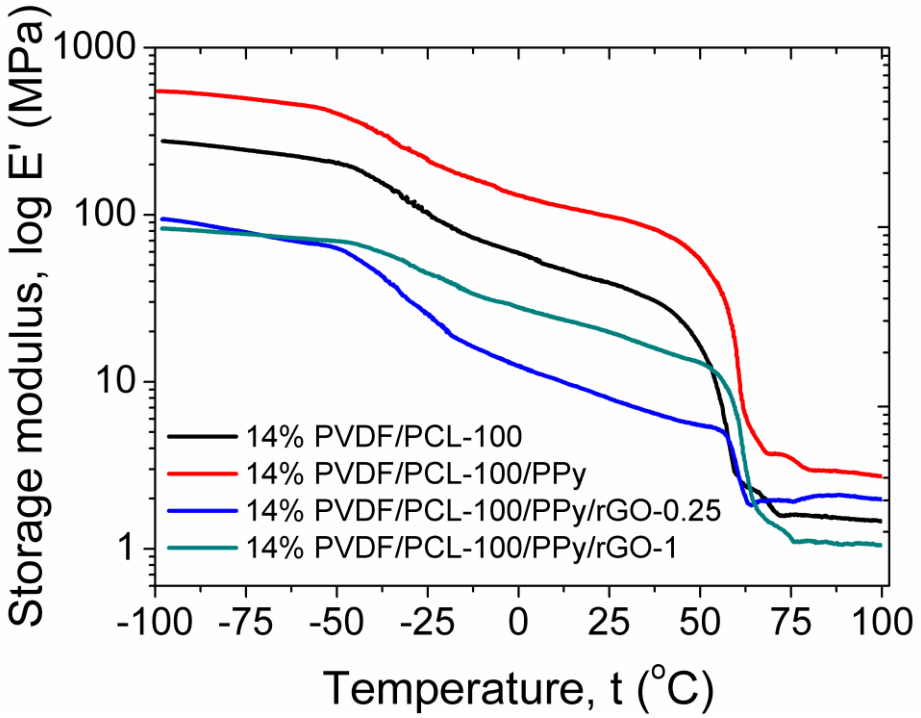
Samples	t_{om} (°C)	t_m (°C)	t_{oc} (°C)	t_c (°C)
14% PVDF/PCL-100	52.10	53.94	34.95	32.65
14% PVDF/PCL-100/PPy	52.97	55.11	35.19	32.73
14% PVDF/PCL-100/PPy/rGO-0.25	53.02	55.32	34.87	32.04
14% PVDF/PCL-100/PPy/rGO-1	52.85	55.09	35.40	32.54
Samples	H_m (J/g)	H_c (J/g)		
14% PVDF/PCL-100	33.90	33.08		
14% PVDF/PCL-100/PPy	31.56	31.49		
14% PVDF/PCL-100/PPy/rGO-0.25	30.16	29.58		
14% PVDF/PCL-100/PPy/rGO-1	31.25	31.73		

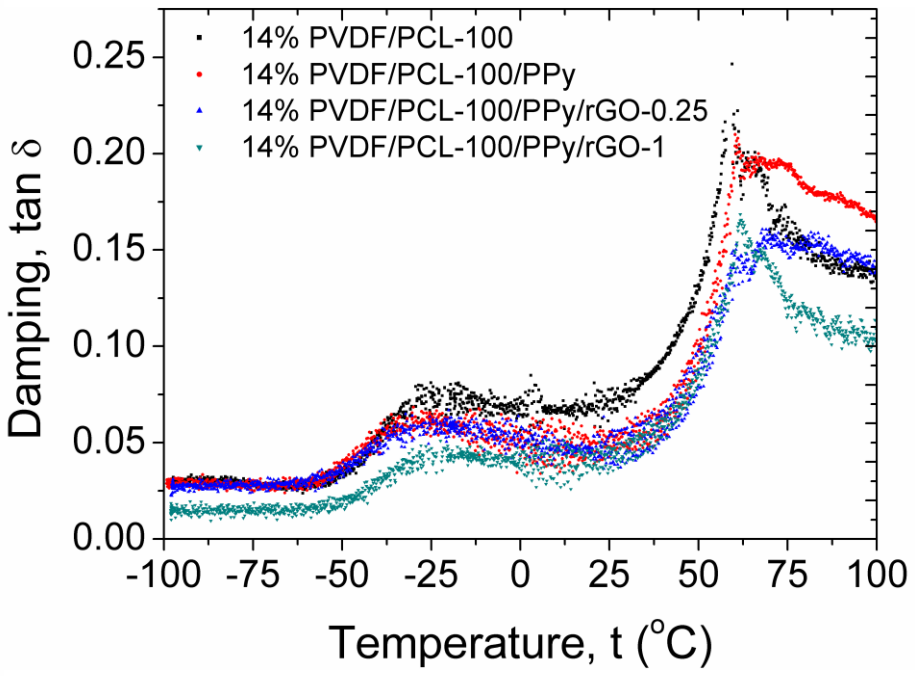
PVDF/PCL (high Mw) fibrous mats heat managing performance after coating



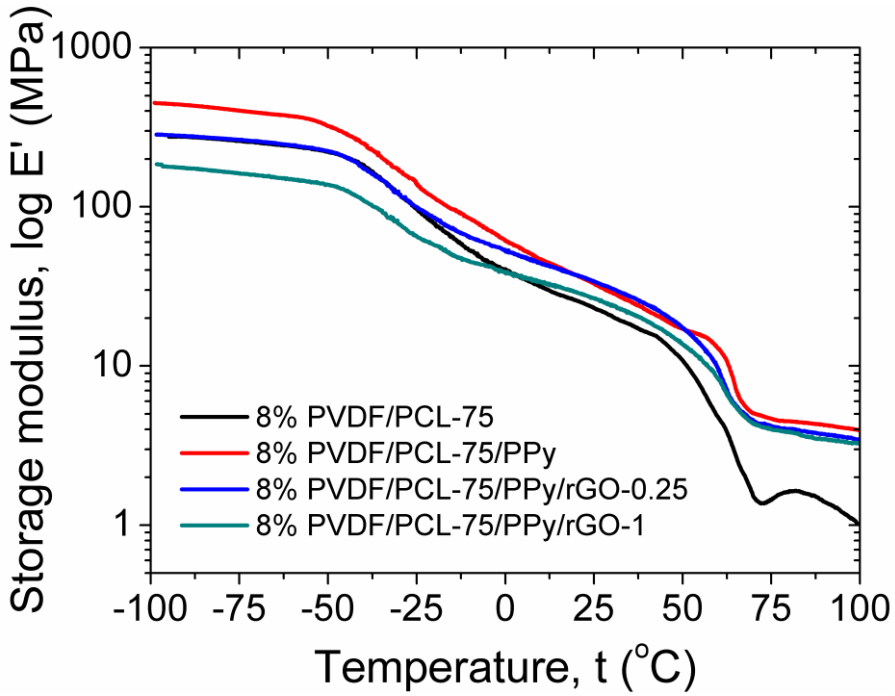
Samples	t_{om} (°C)	t_m (°C)	t_{oc} (°C)	t_c (°C)
8% PVDF/PCL-75	52.79	55.45	32.55	29.44
8% PVDF/PCL-75/PPy	53.48	56.07	33.55	30.37
8% PVDF/PCL-75/PPy/rGO-0.25	54.27	56.32	33.93	30.79
8% PVDF/PCL-75/PPy/rGO-1	53.83	56.33	33.50	30.01
Samples	H_m (J/g)	H_c (J/g)		
8% PVDF/PCL-75	20.11	19.51		
8% PVDF/PCL-75/PPy	17.42	18.10		
8% PVDF/PCL-75/PPy/rGO-0.25	19.12	19.34		
8% PVDF/PCL-75/PPy/rGO-1	18.09	18.31		

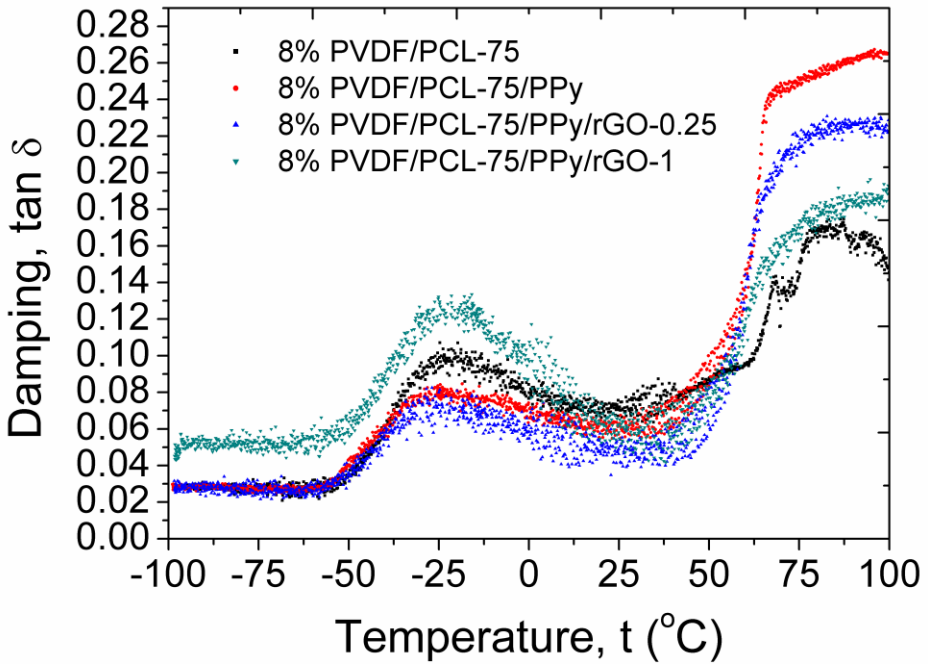
PVDF/PCL (low Mw) fibrous mats DM behaviour after coating





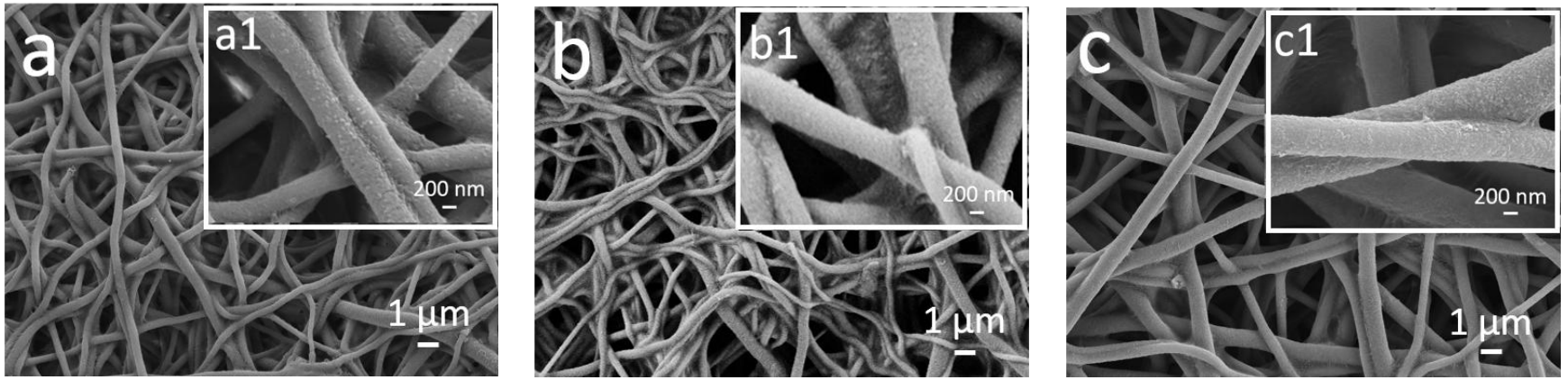
PVDF/PCL (high Mw) fibrous mats DM behaviour after coating



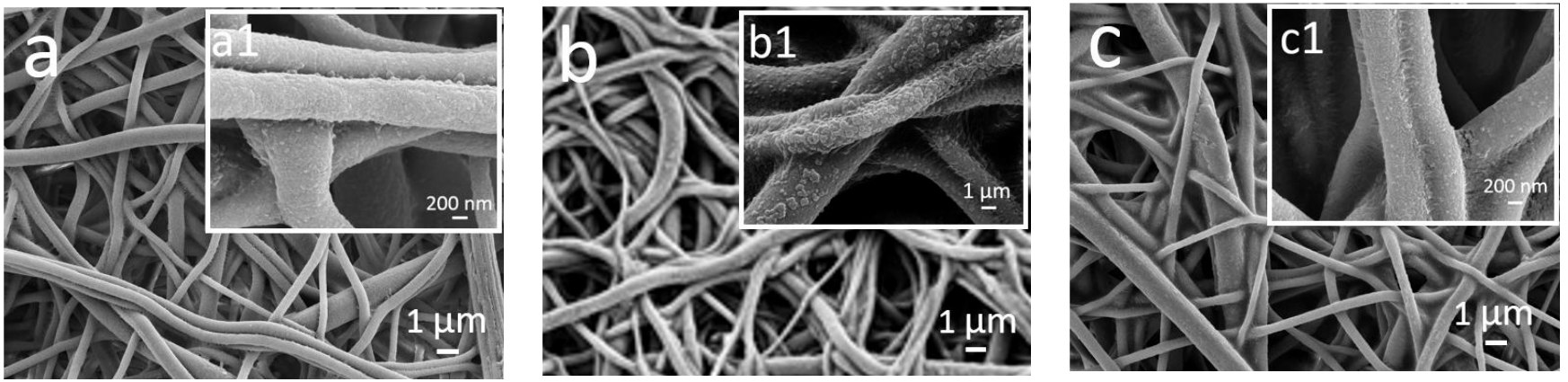


PVDF/PCL/PPy fibers morphology after heat exposure

before | after: 65 °C, 24 h | after: 80 °C, 24 h

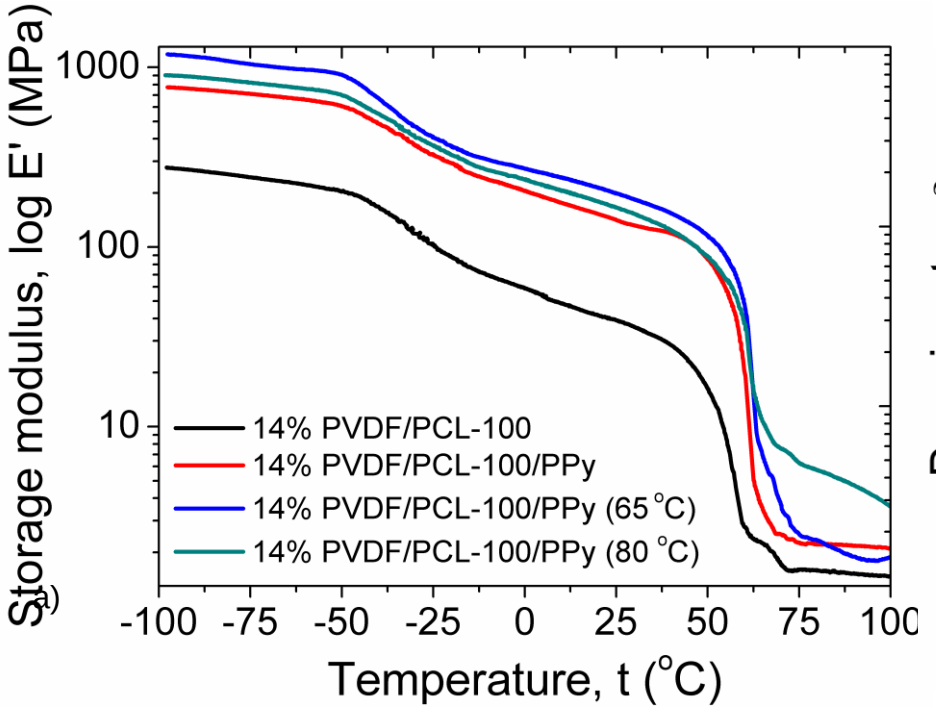


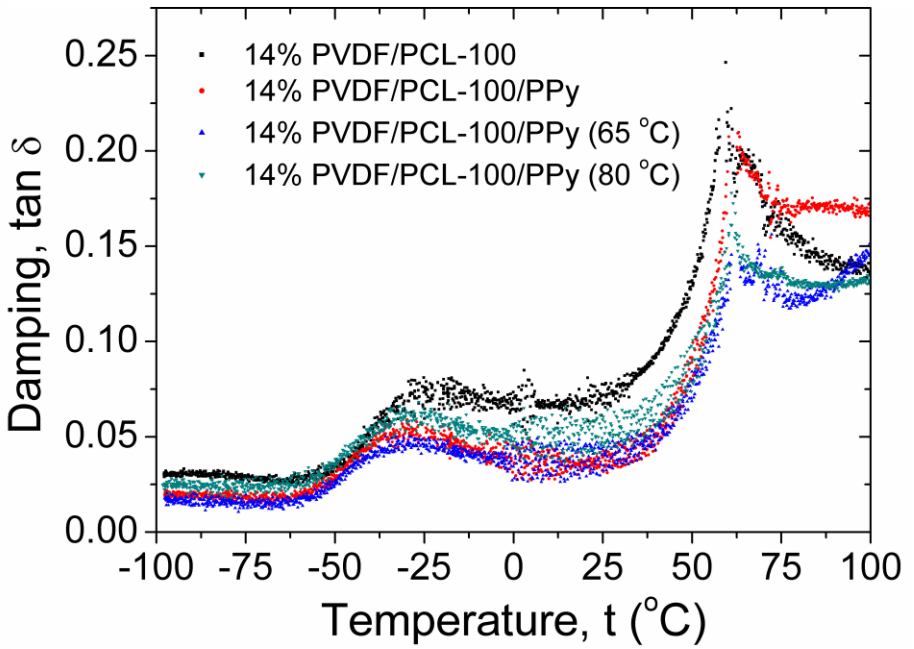
1) 14% PVDF/PCL:100/100



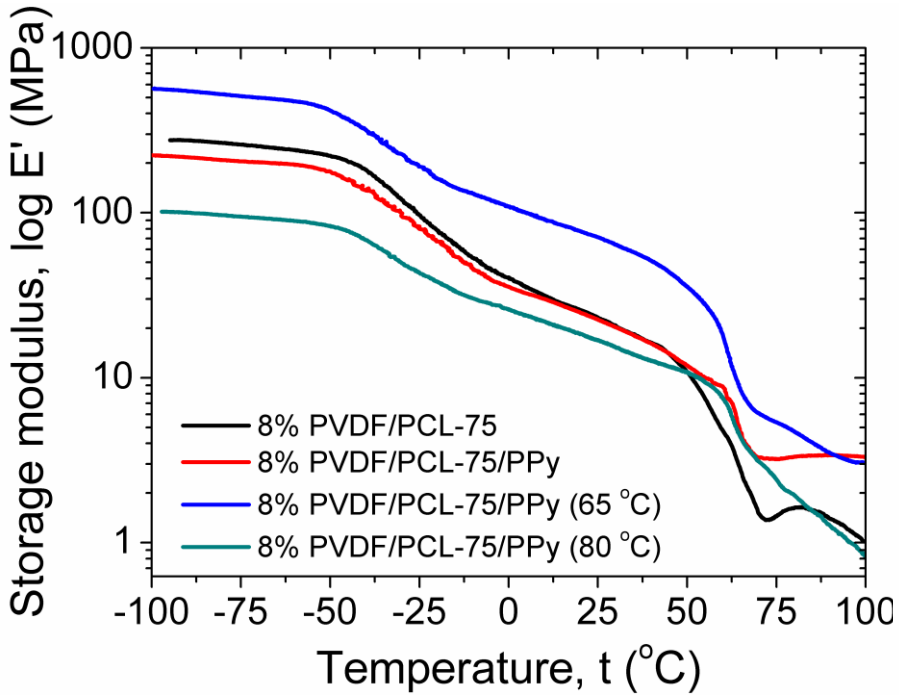
2) 8% PVDF/PCL:100/75

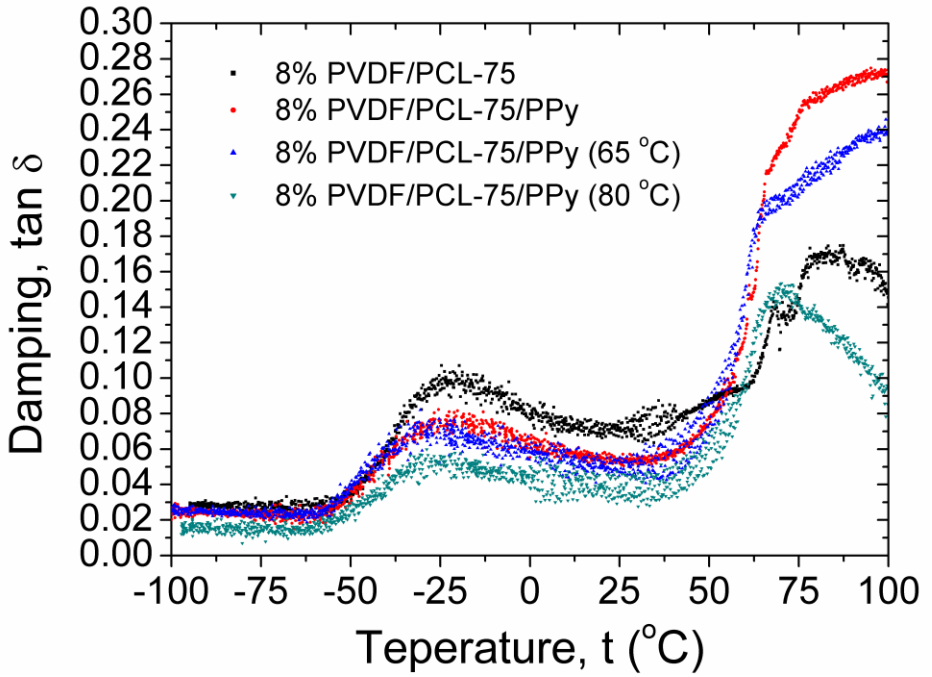
PVDF/PCL/PPy (low Mw) fibrous mats DM behaviour after heat exposure



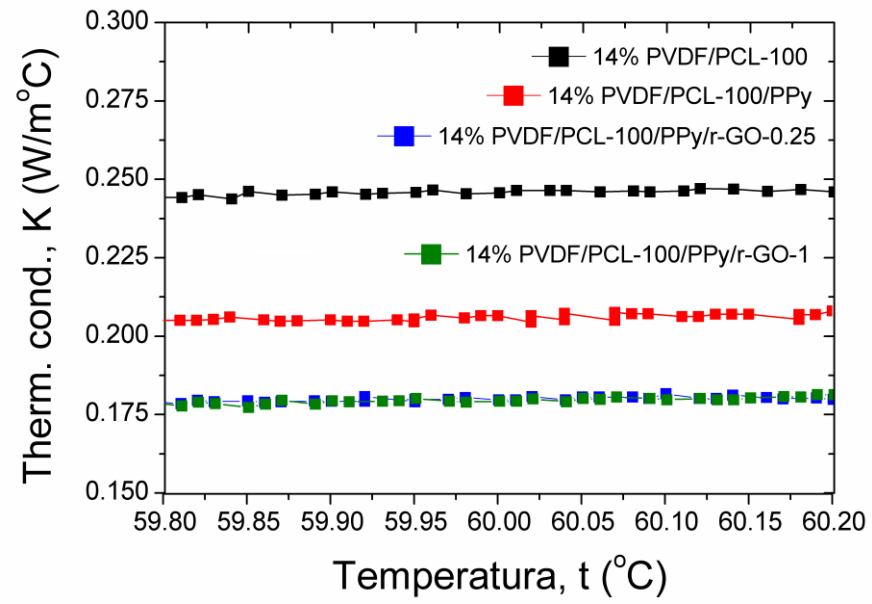


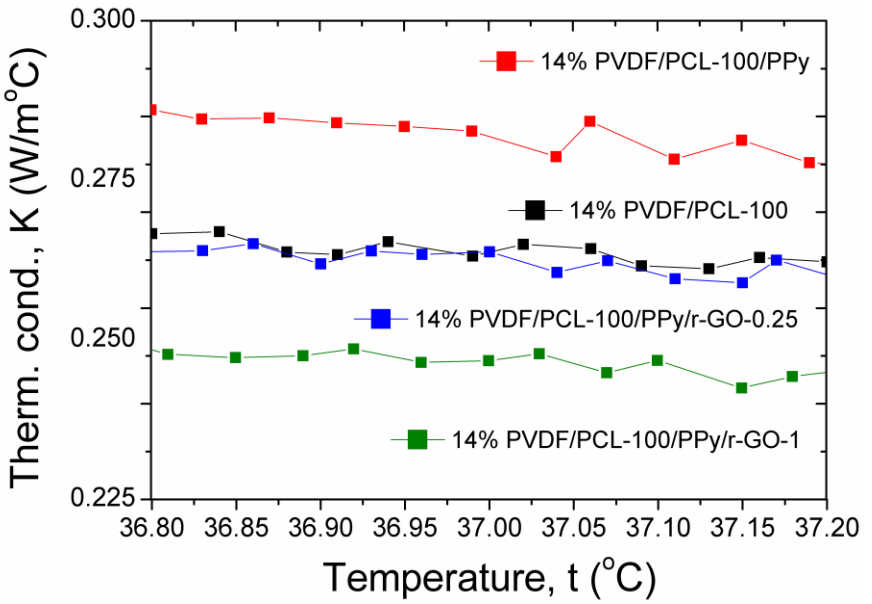
PVDF/PCL/PPy (high Mw) DM behaviour after heat exposure



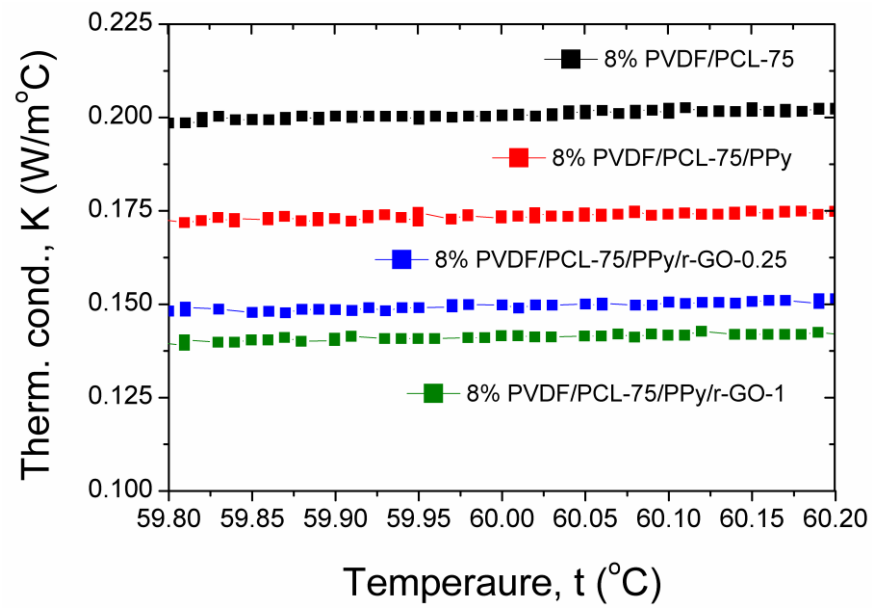


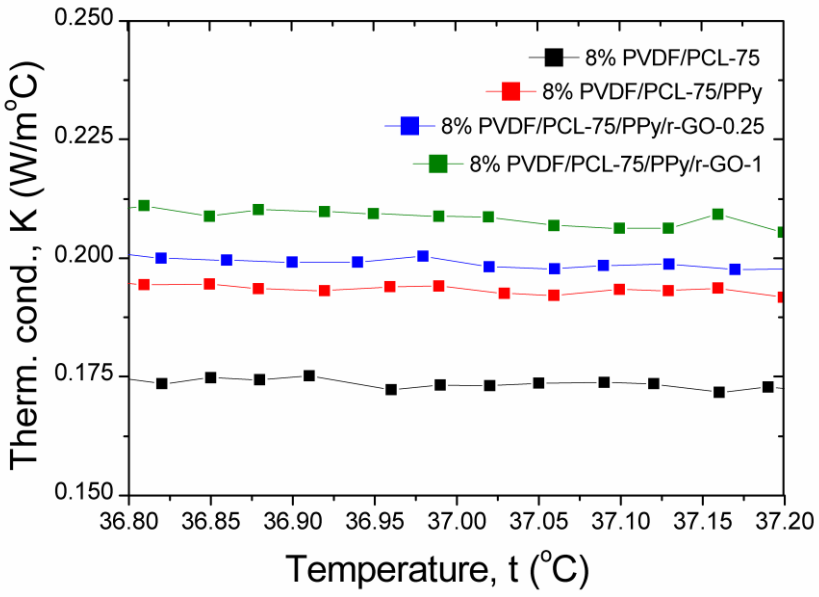
PVDF/PCL (low Mw) fibrous mats thermal conductivity





PVDF/PCL (high Mw) fibrous mats thermal conductivity





17%, 13%, 9.8%

Conclusions 1

PVA/mixture of plant oils electrospun nanofibrous materials:

- rough surface with irregular cross-sections for contents above the mass ratio of 100/50 for the PVA/PCM, as well as increased number or pores and fiber diameter.
- discontinuous distribution of the PCM domains with a few fibers having the core-sheath structure.
- heat storage/release reliability after 100 cycles of heating and cooling with negligible change in the melting/crystallization temperatures and enthalpies (~80 J/g).
- better thermo-mechanical behaviour for the lowest PCM containing fibers and increased strength and break strain, after heat treatment, due to higher uniformity of the PCM distribution.
- stability of the structure at temperatures firmly above the PCM's melting temperature.

Conclusions 2

PVDF/PCL electrospun nanofibrous materials

- 14% PVDF/PCL-100 and 8% PVDF/PCL-75 fibers showed almost 50 % and 34% of encapsulation efficiency, respectively, with reduction in the supercooling effect when blended in the PVDF matrix.
- The change in the heat enthalpies (highest of 3 J/g) was in accordance with the fiber diameter change as a function of electrical voltage and the flow rate, with the main dependence on the PCM content.

PVDF/PCL cast films counter parts

- The films were unfavourable in regard to heat storing ability due to greater supercooling effect and lower crystallinity. Both low and high Mw PCL based cast films revealed polymer separation during drying and after heat exposure.

Coated PPy and PPy/r-GO electrospun fibrous materials

- The total weight loss of the PVDF/PCM materials was reduced by 10% for the high Mw PCL based fibers coated with PPy/r-GO-1 indicating improved thermal stability.

Conclusions 3

Coated PPy and PPy/r-GO electrospun fibrous materials

-The PPy and the PPy/r-GO coatings revealed non significant changes of the melting and crystallization temperatures (increase of ~1 °C). The coating provided fiber protective layer and presumably due to r-GO agglomerations reduction in the enthalpies.

-PPy coating revealed firmly improved fibers form stability even when the fibers were exposed to temperatures of 80 °C for 24 h.

-Materials stiffness improved after heat treatment of the PPy coated low Mw PCL based fibers, thus suggesting improved crystallinity. This effect was only observed for the lower temperature (65 °C) heat treated high Mw PCL fibers.

Thermal conductivity

-The high Mw PCL based fibers in PCL solid phase showed gradual increase of the thermal conductivity by 17%, 13% and 9.8% for the PPy coated, PPy/r-GO-0.25 and PPy/r-GO-1 fibers, respectively.

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