

# Insights into the levulinate-based ionic liquid class: synthesis, cellulose dissolution evaluation and ecotoxicity assessment

Andrea Mezzetta,<sup>[a]</sup> Stefano Becherini,<sup>[a]</sup> Carlo Pretti,<sup>[b,c]</sup> Gianfranca Monni,<sup>[c]</sup> Valentina Casu,<sup>[c]</sup> Cinzia Chiappe<sup>[a]</sup> and Lorenzo Guazzelli \*<sup>[a]</sup>

## *Supporting Information*

### *Table of contents*

<sup>1</sup> H- and <sup>13</sup> C-NMR spectra of Lev ILs	pages S2-S11
IR spectra of Lev ILs	pages S12-S15
Thermal gravimetric analysis (TGA) of Lev ILs	pages S16-S19
Images of dissolved MCC in Lev ILs at maximum wt%	pages S20-S25
Optical microscopy of dissolved MCC in Lev ILs at maximum wt%	pages S26-S33
IR spectra of pristine MCC and regenerated cellulose after dissolution in Lev ILs	pages S34-S38
XRD measurements of MCC and regenerated cellulose from [EMIM][Lev]	page S39

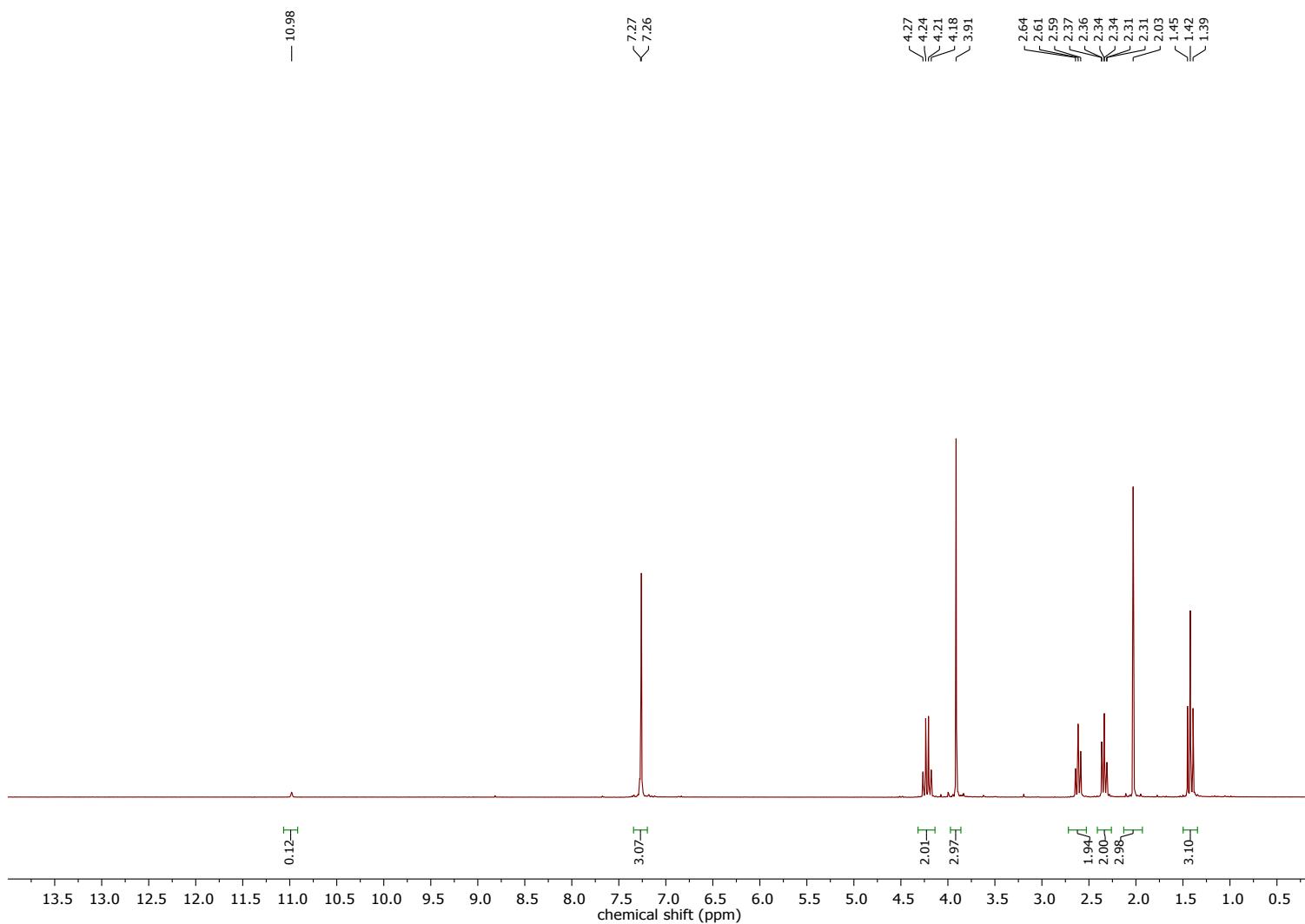
---

[a] A. Mezzetta, S. Becherini, C. Chiappe, L. Guazzelli  
Department of Pharmacy  
University of Pisa  
Via Bonanno 6, Pisa (Italy)  
\*E-mail: [lorenzo.guazzelli@unipi.it](mailto:lorenzo.guazzelli@unipi.it)

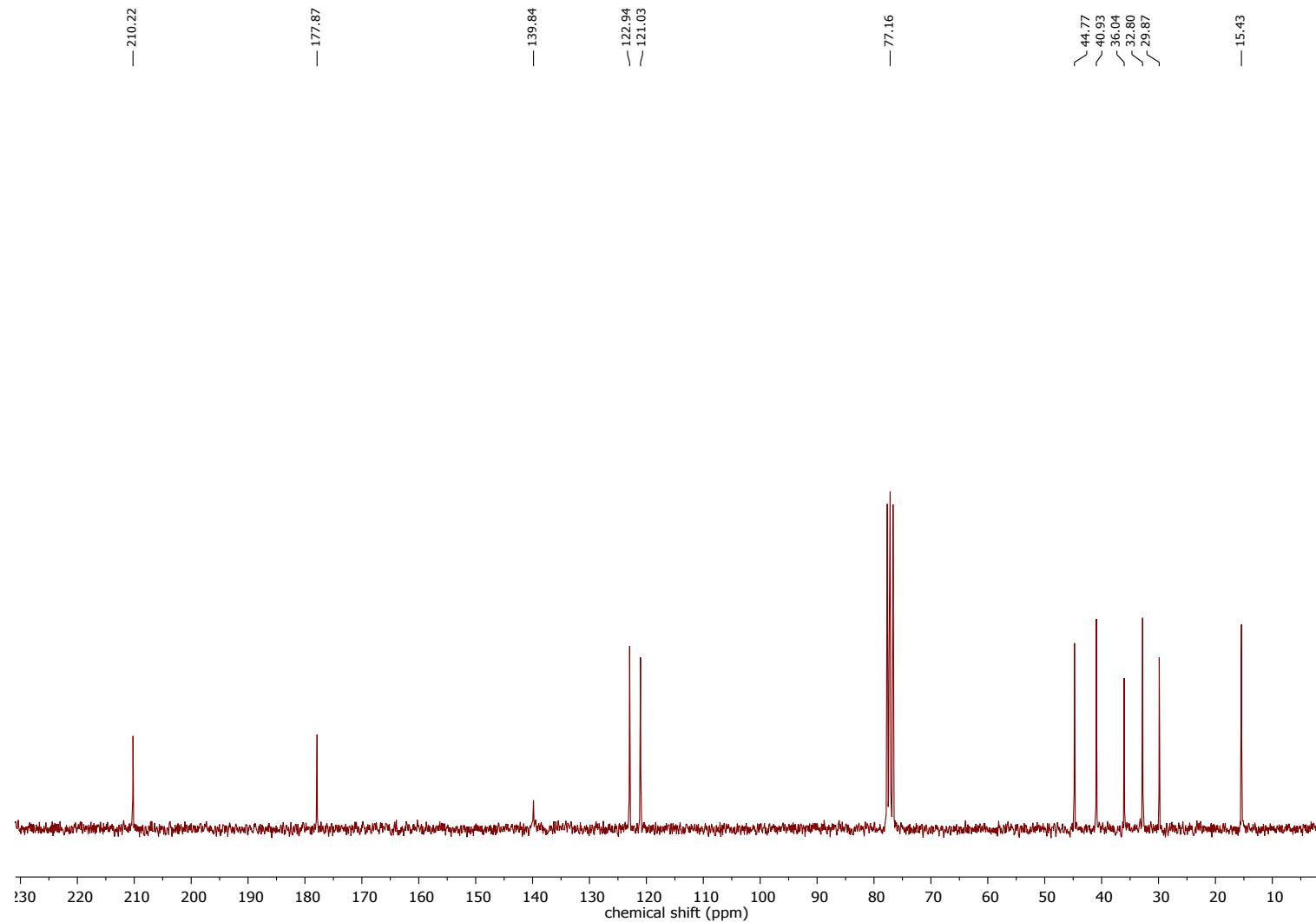
[b] C. Pretti  
Department of Veterinary Sciences,  
University of Pisa,  
Via Livornese lato monte, San Piero a Grado (PI) 56122, Pisa,  
Italy

[c] C. Pretti, G. Monni, V. Casu  
Interuniversity Consortium of Marine Biology of Leghorn “G.  
Bacci”, Livorno, 57128, Italy

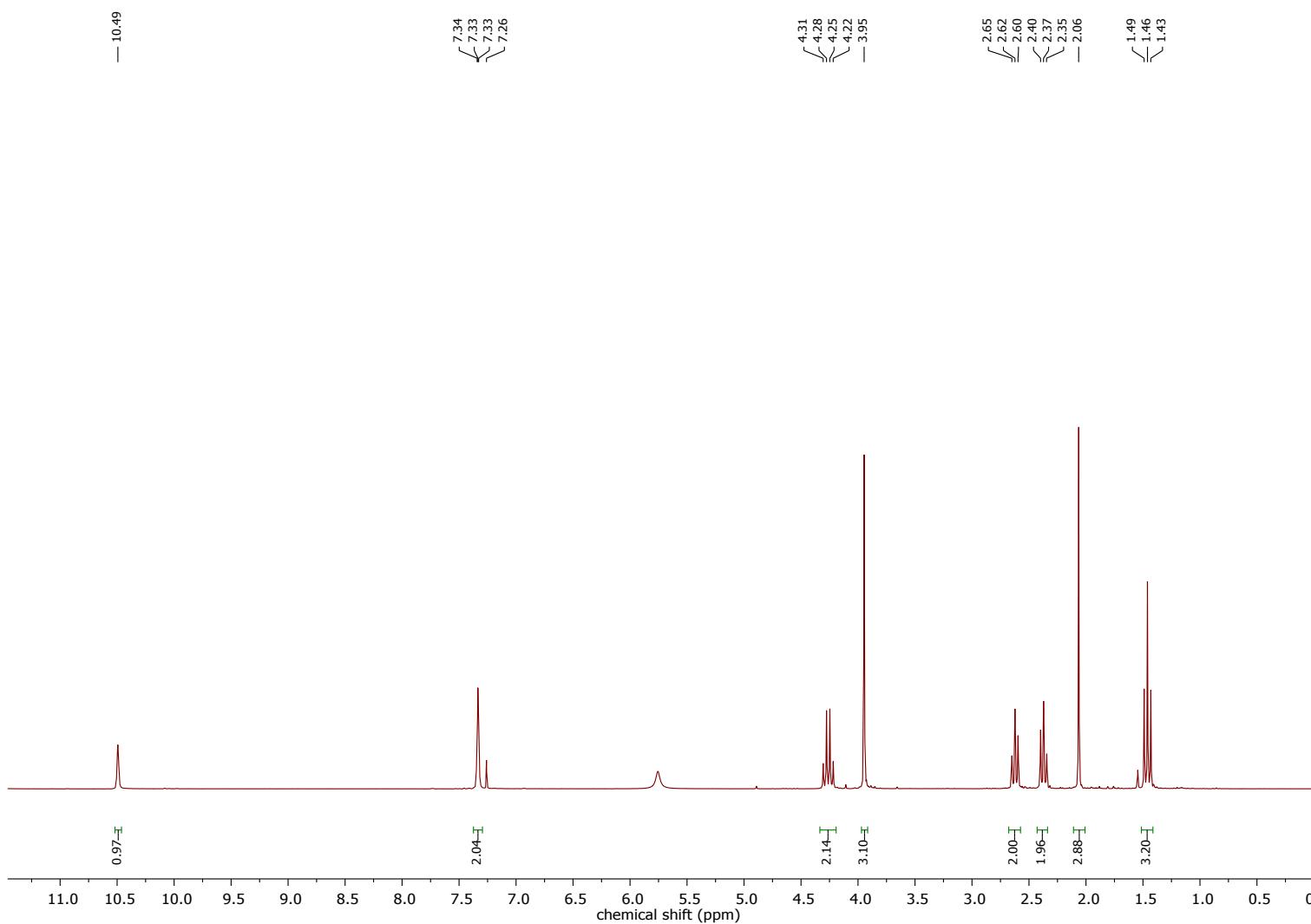
**Fig S1.**  $^1\text{H}$  NMR of EMIMLev at 25 °C



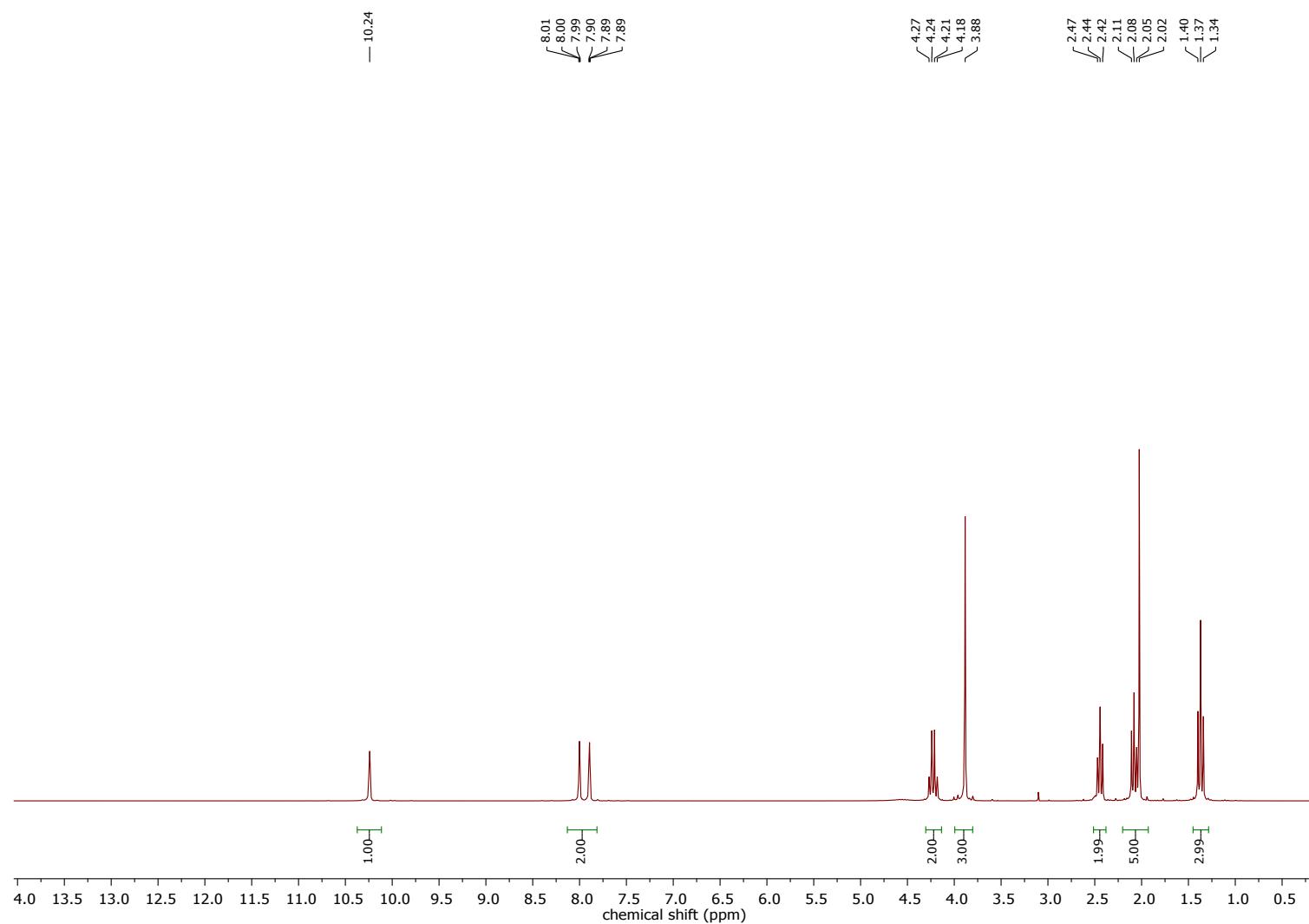
**Fig S2.**  $^{13}\text{C}$  NMR of EMIMLev at 25 °C



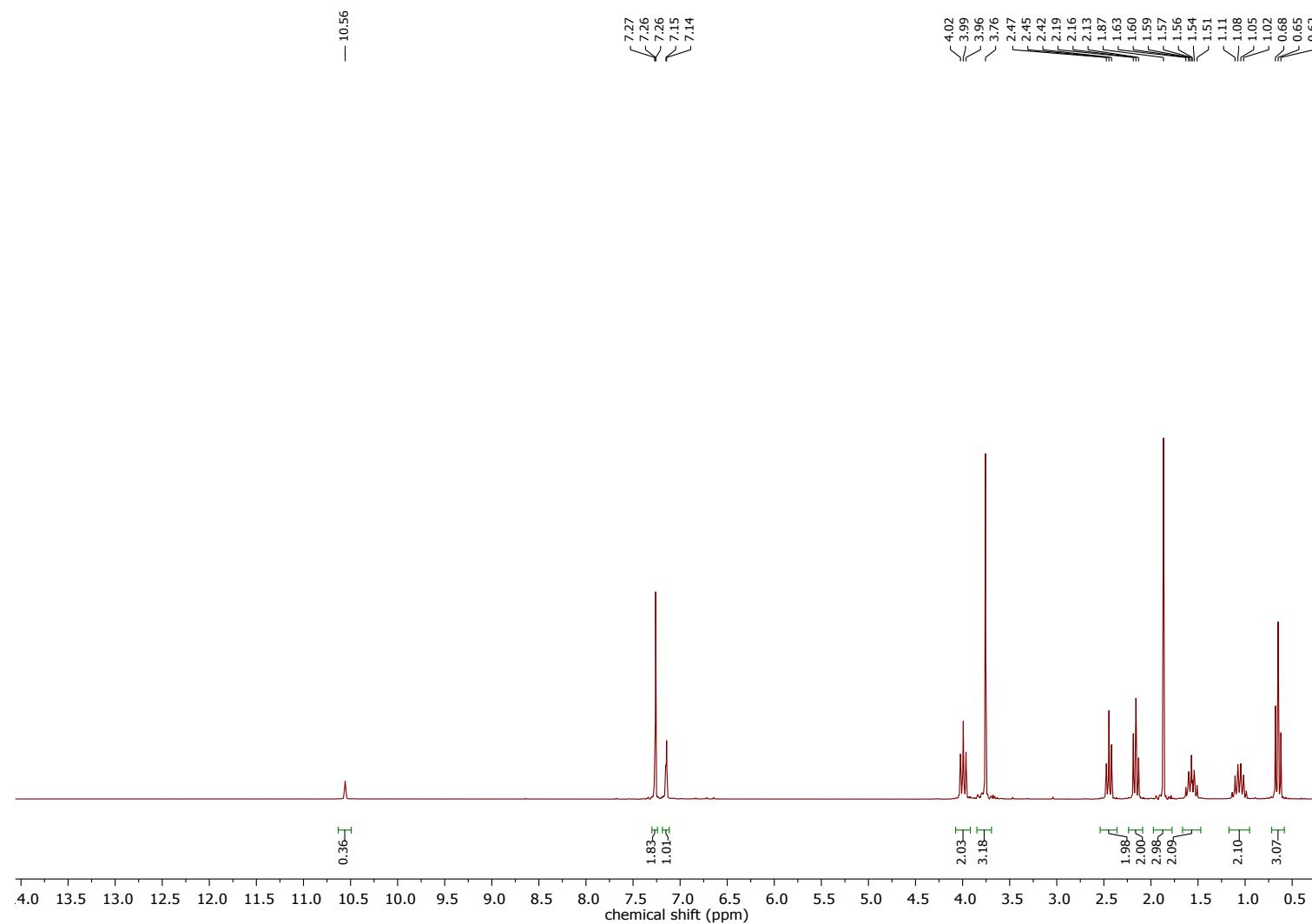
**Fig S3.**  $^1\text{H}$  NMR of **EMIMLev** (25 °C) recycled after the second cycle of dissolution of MCC at 100 °C



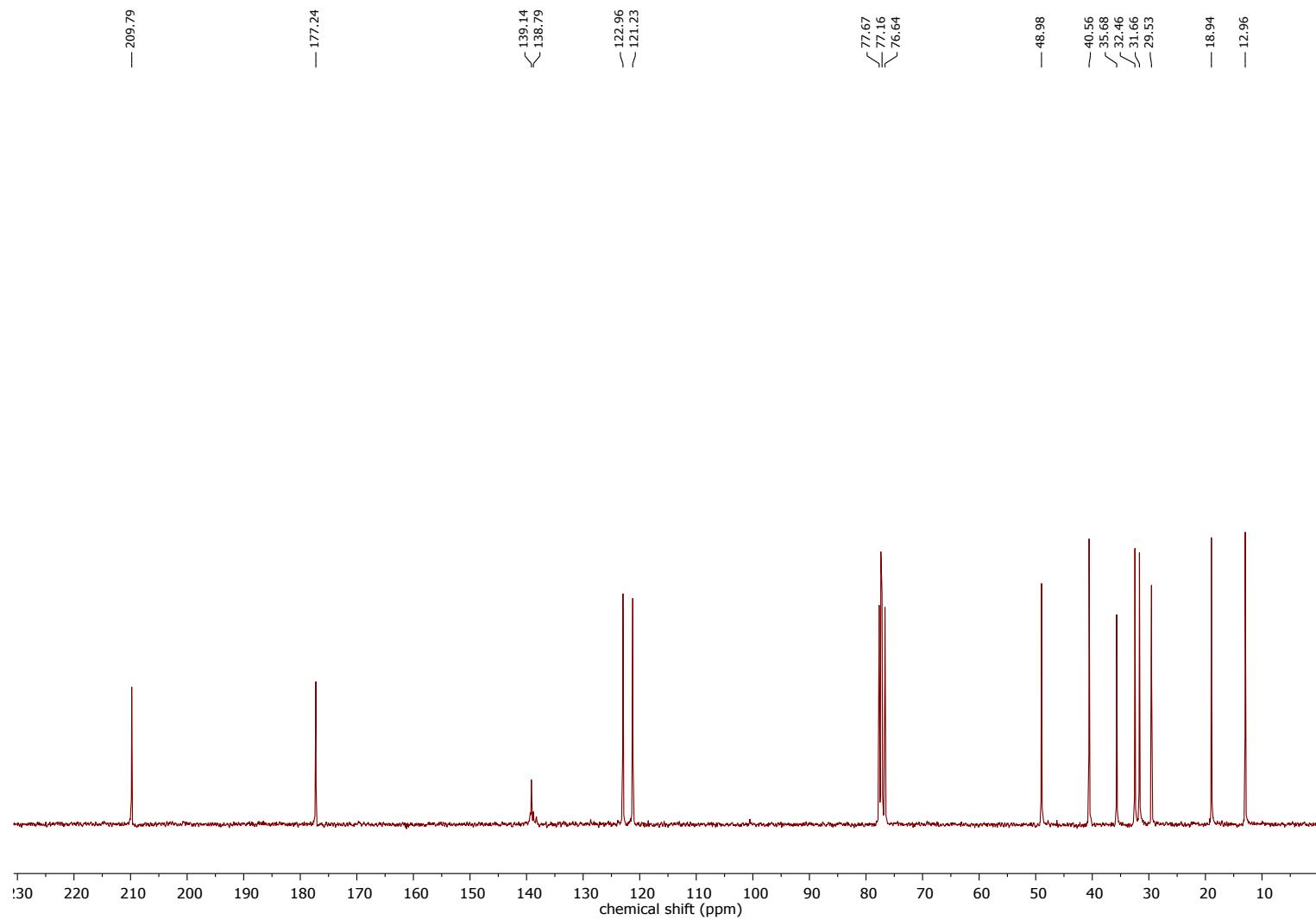
**Fig S4.**  $^1\text{H}$  NMR of **EMIMLev** (coaxial, 25 °C) after 12h at 120 °C (thermal stability test)



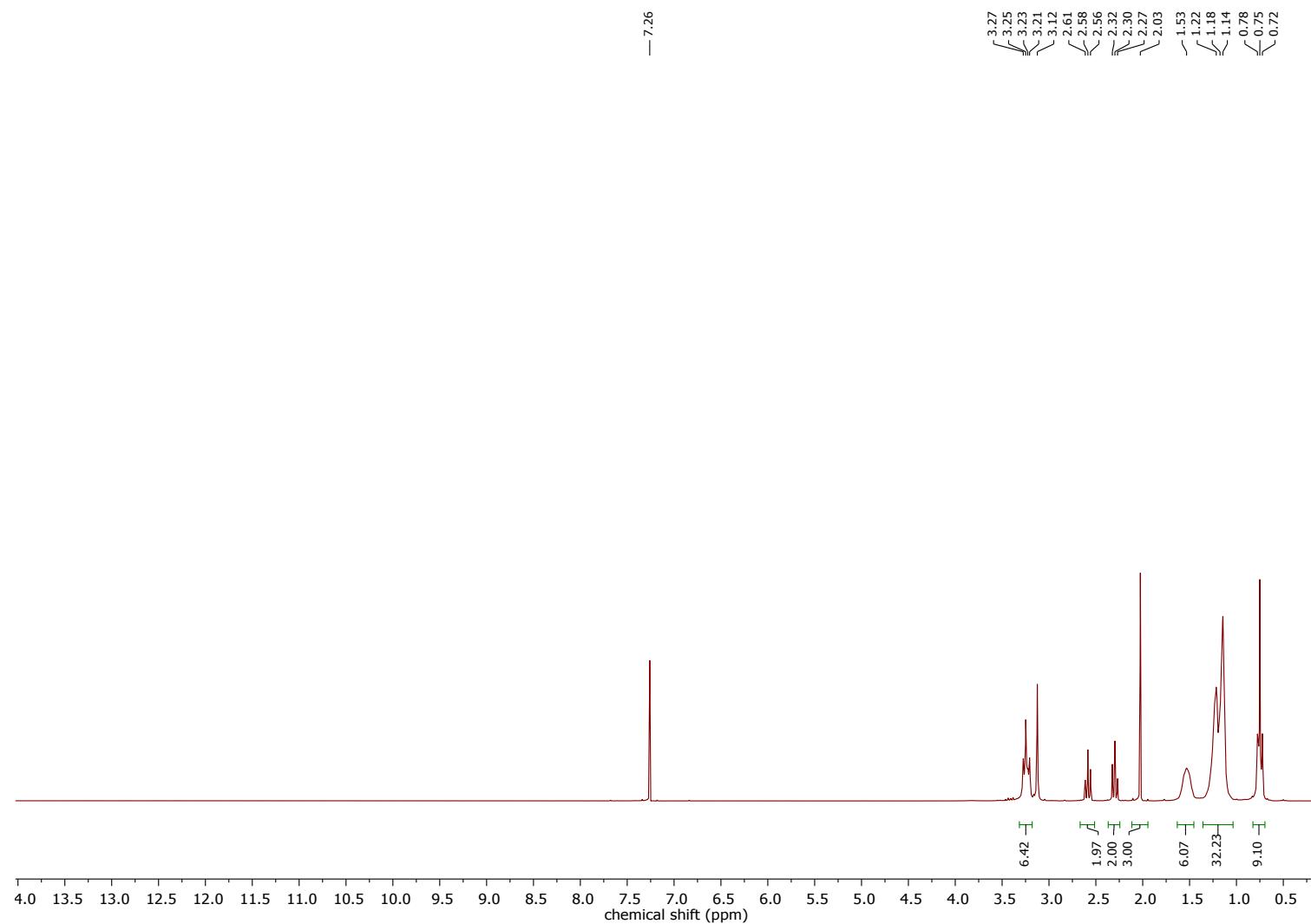
**Fig S5.**  $^1\text{H}$  NMR of **BMIMLev** at 25 °C



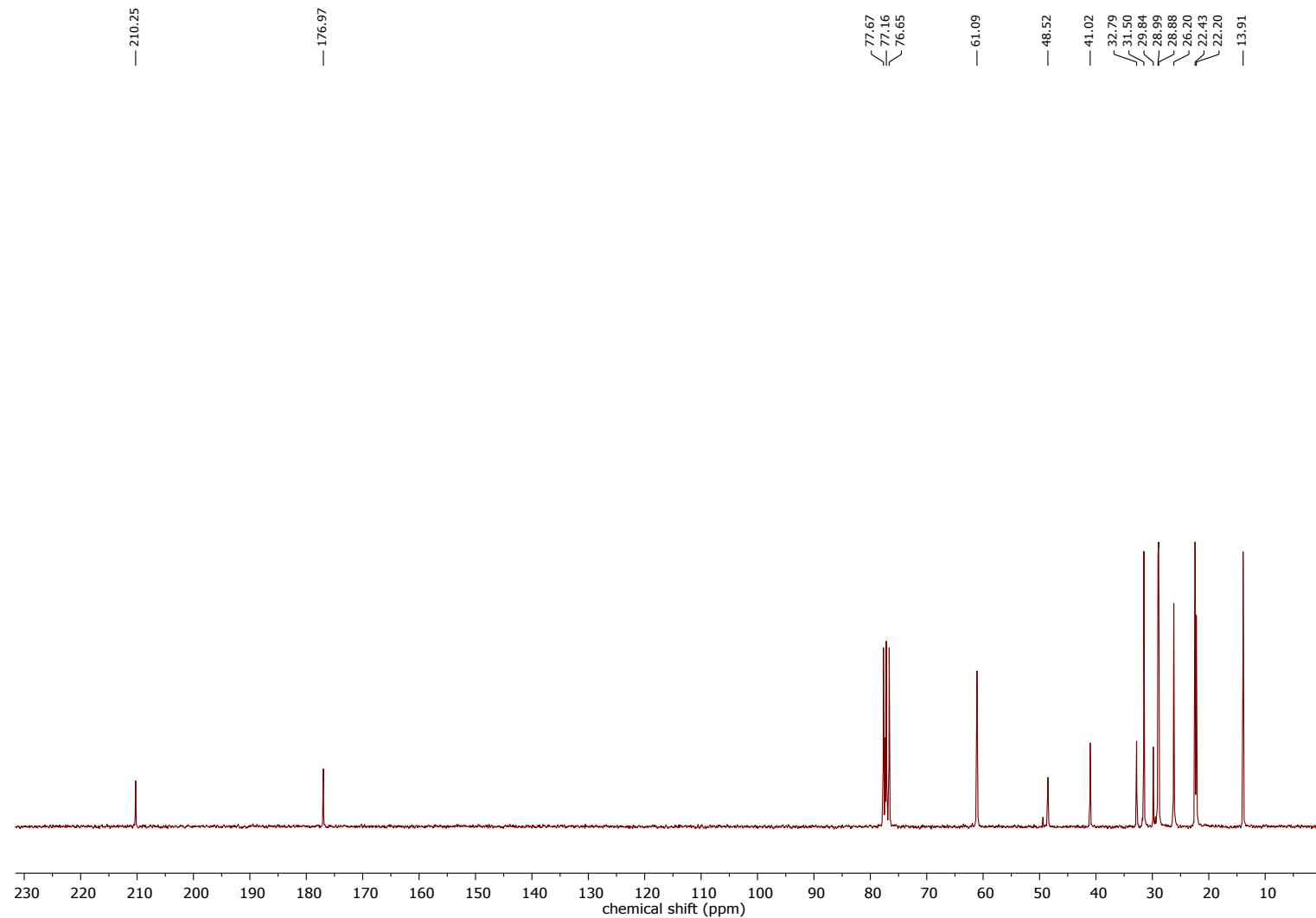
**Fig S6.**  $^{13}\text{C}$  NMR of BMIMLev at 25 °C



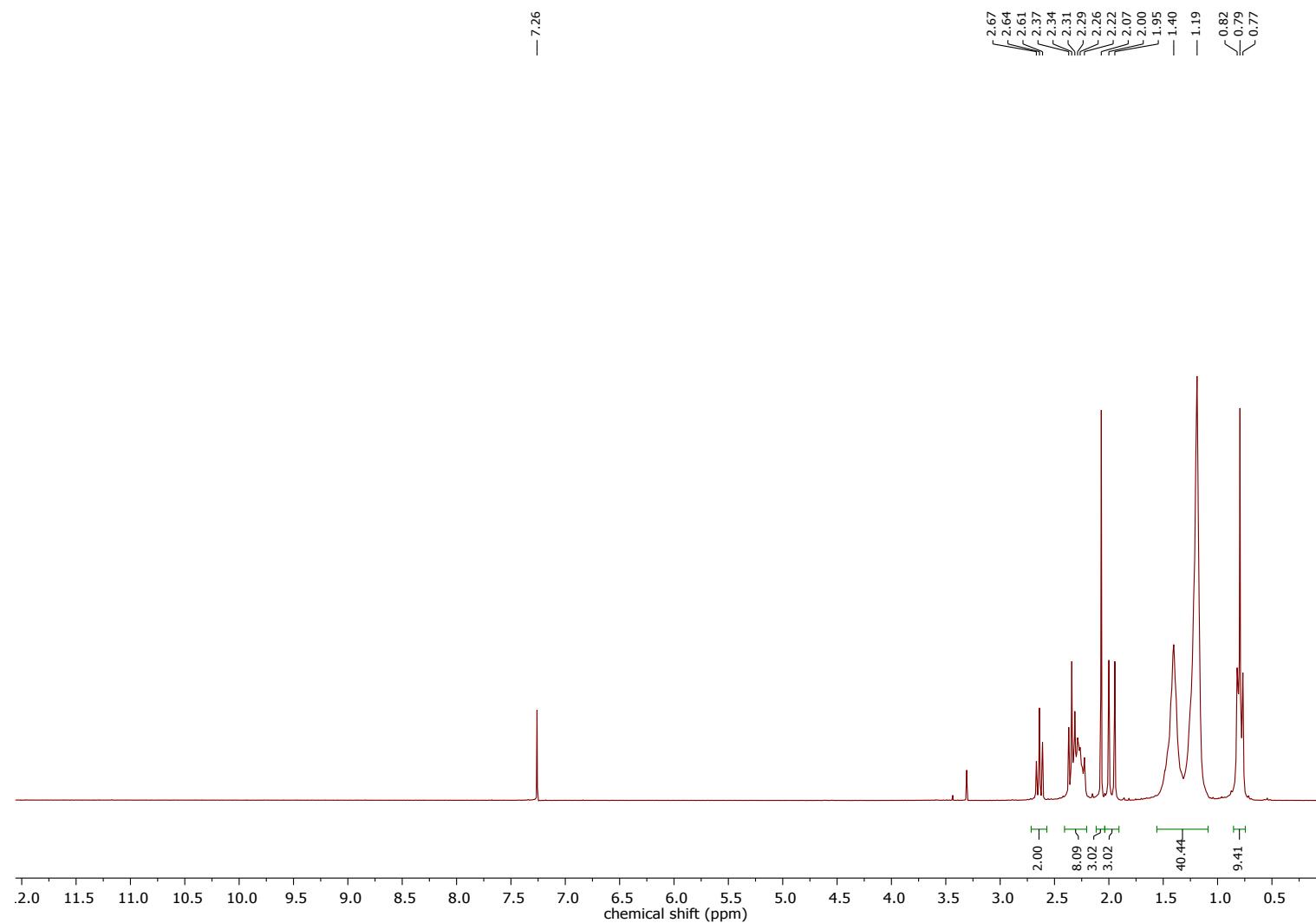
**Fig S7.**  $^1\text{H}$  NMR of **N<sub>8881</sub>Lev** at 25 °C



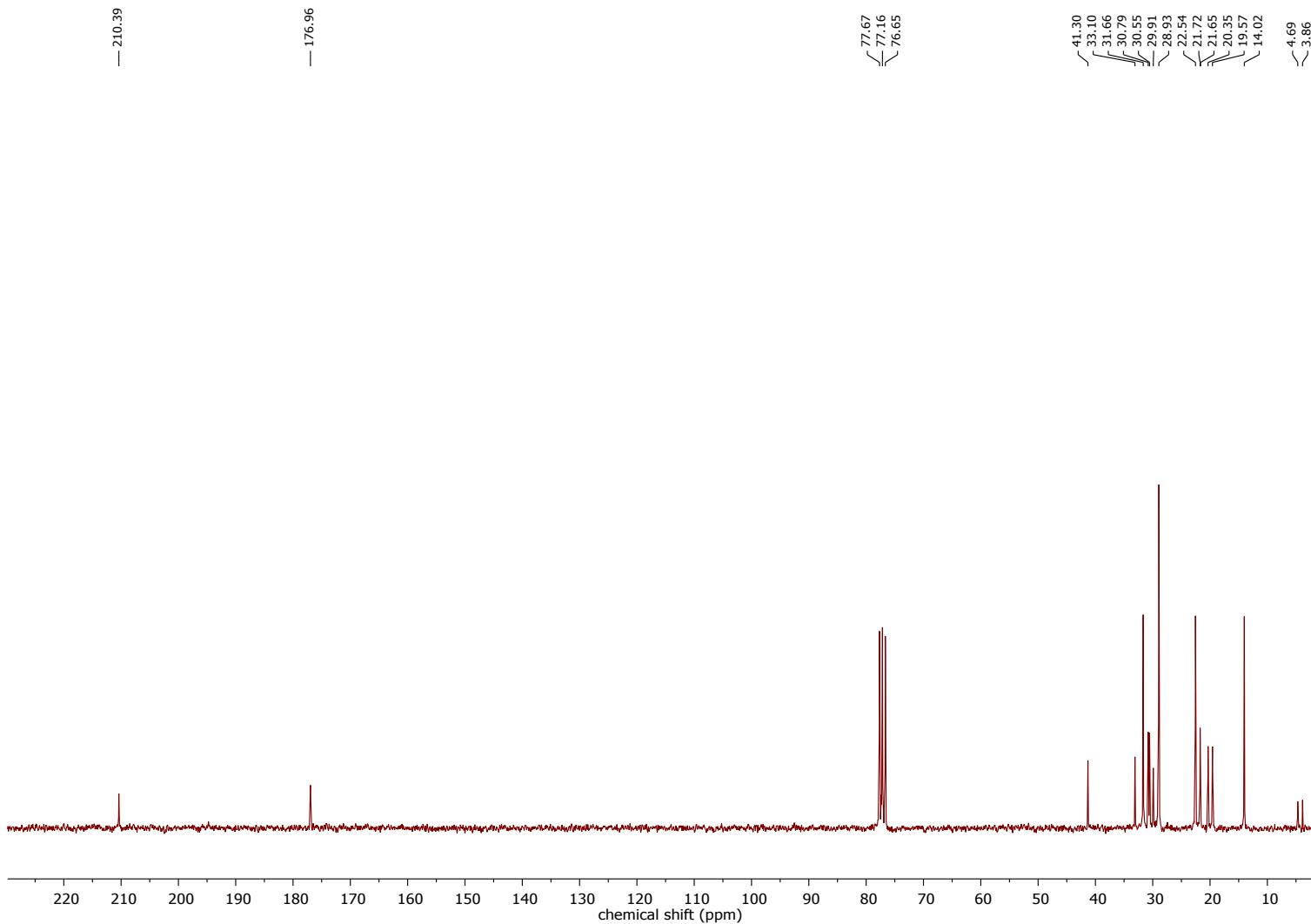
**Fig S8.**  $^{13}\text{C}$  NMR of **N<sub>8881</sub>Lev** at 25 °C



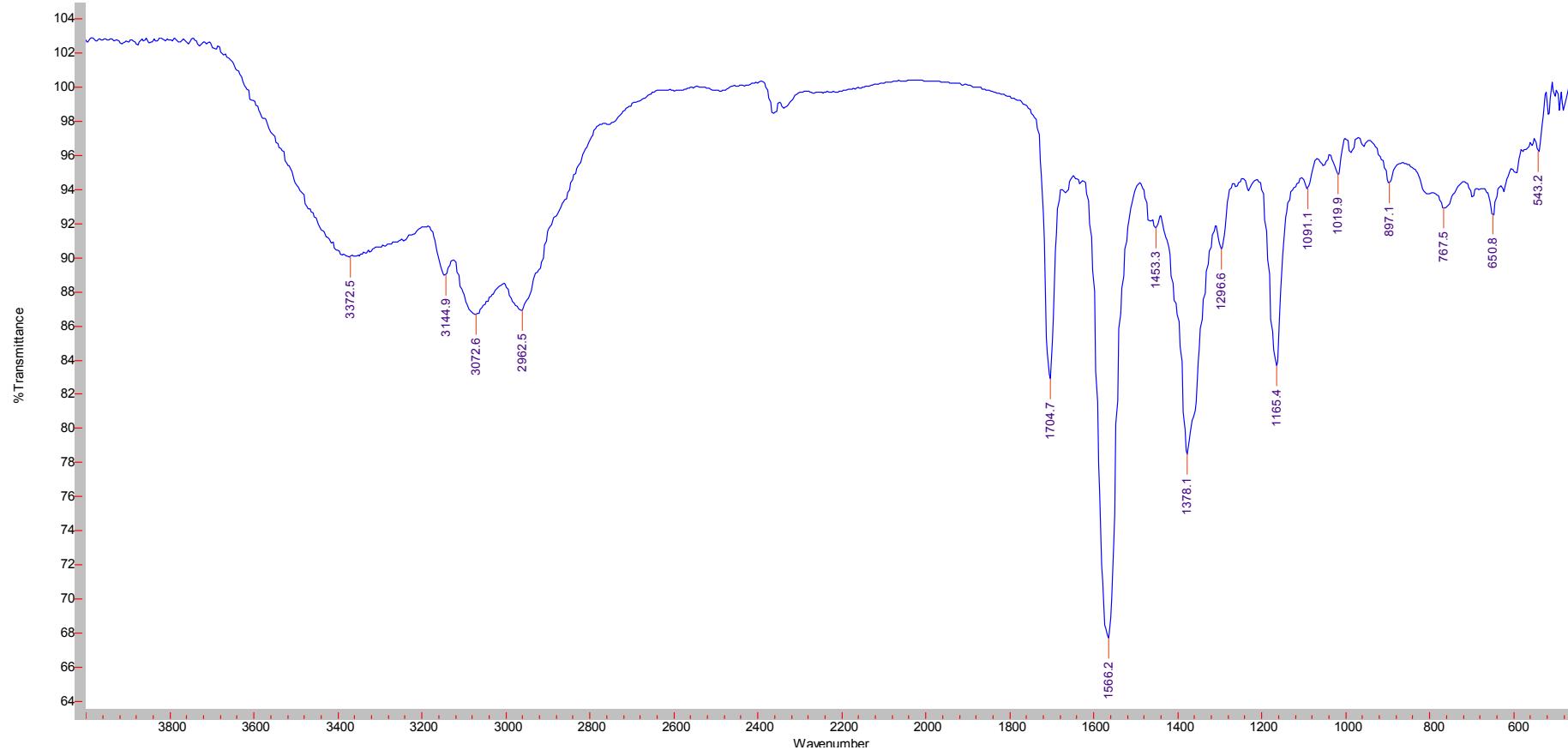
**Fig S9.**  $^1\text{H}$  NMR of **P<sub>8881</sub>Lev** at 25 °C



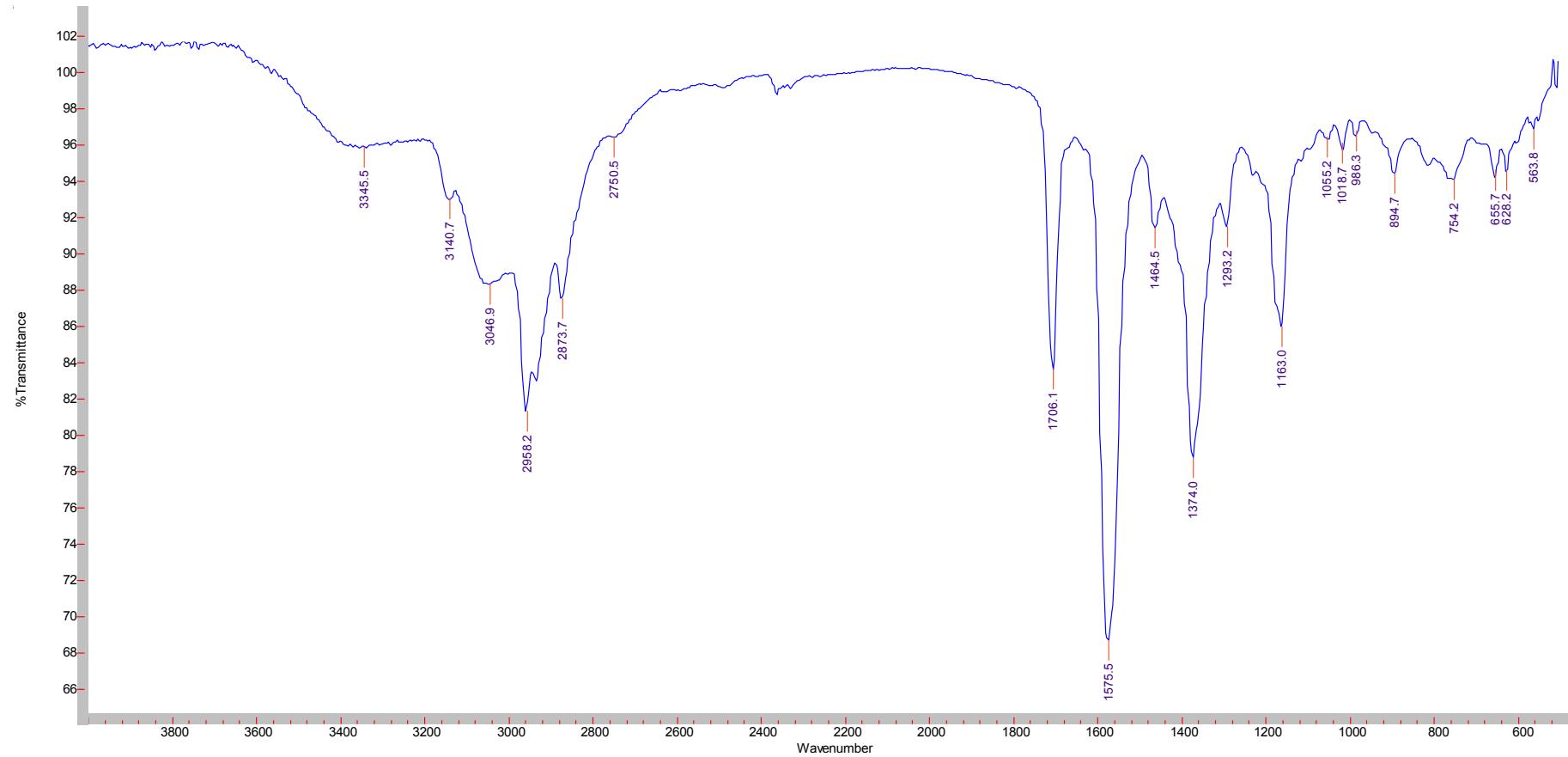
**Fig S10.**  $^{13}\text{C}$  NMR of **P<sub>8881</sub>Lev** at 25 °C



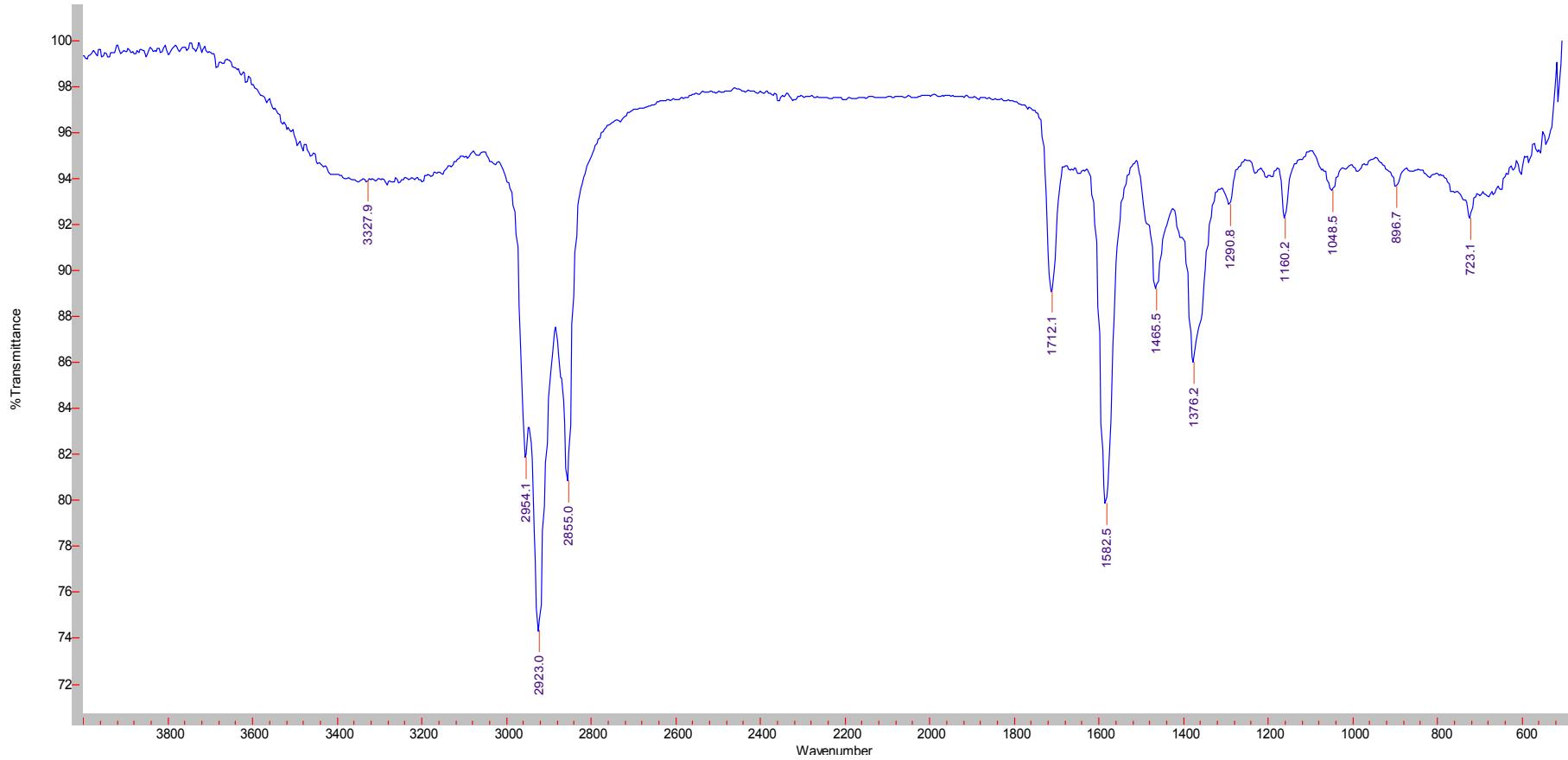
**Fig S11.** IR of **EMIMLev** at 25 °C



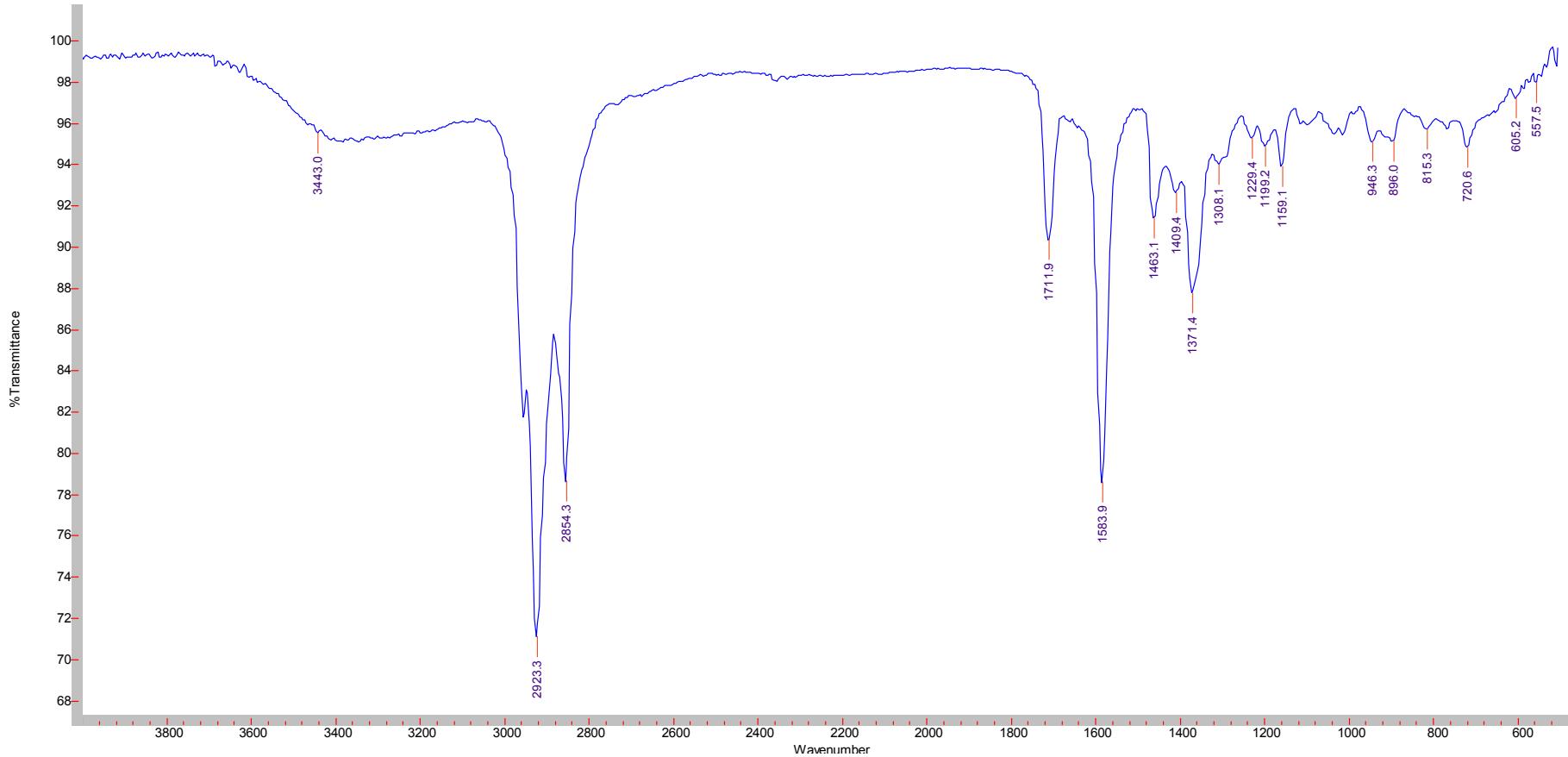
**Fig S12.** IR of **BMIMLev** at 25 °C



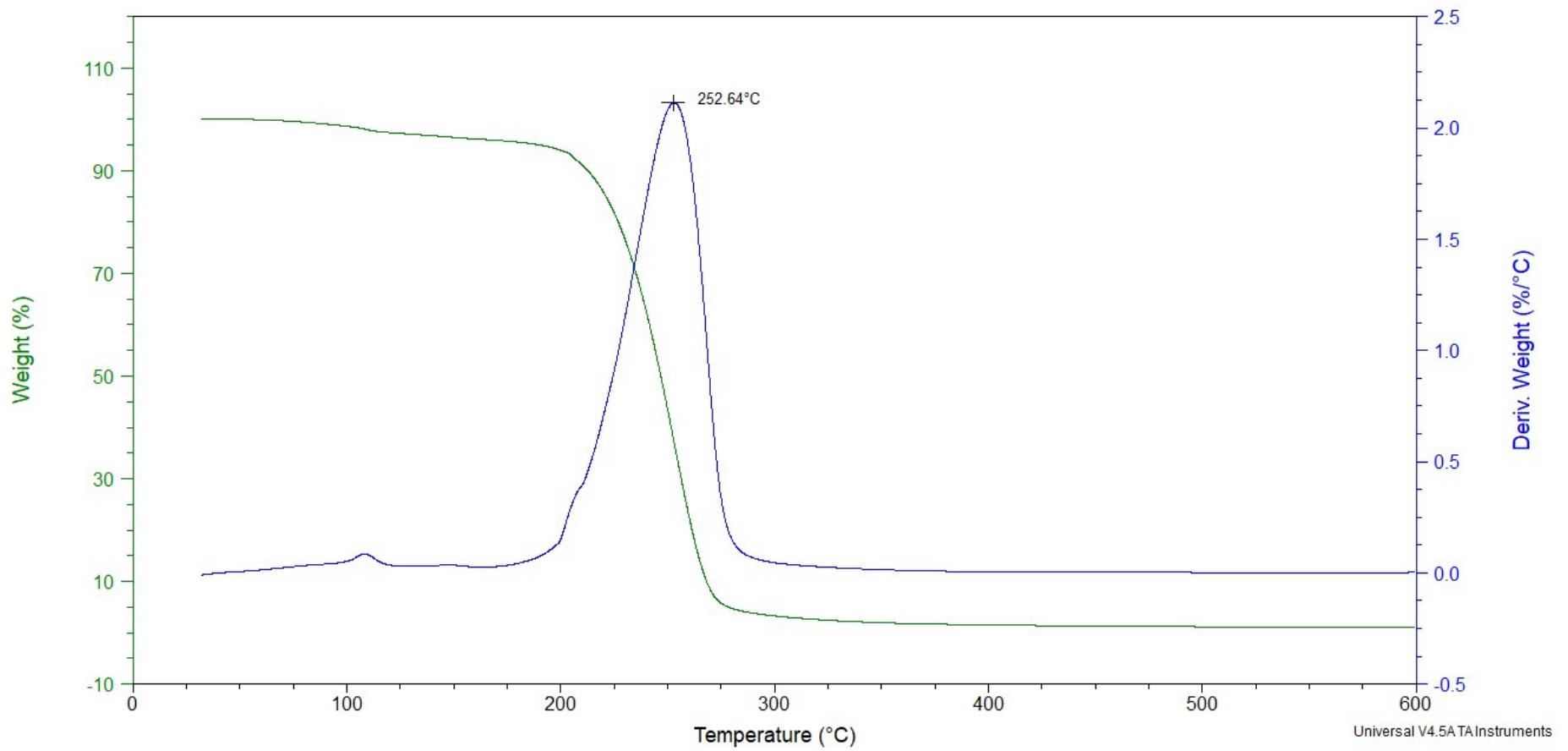
**Fig S13.** IR of **N<sub>8881</sub>Lev** at 25 °C



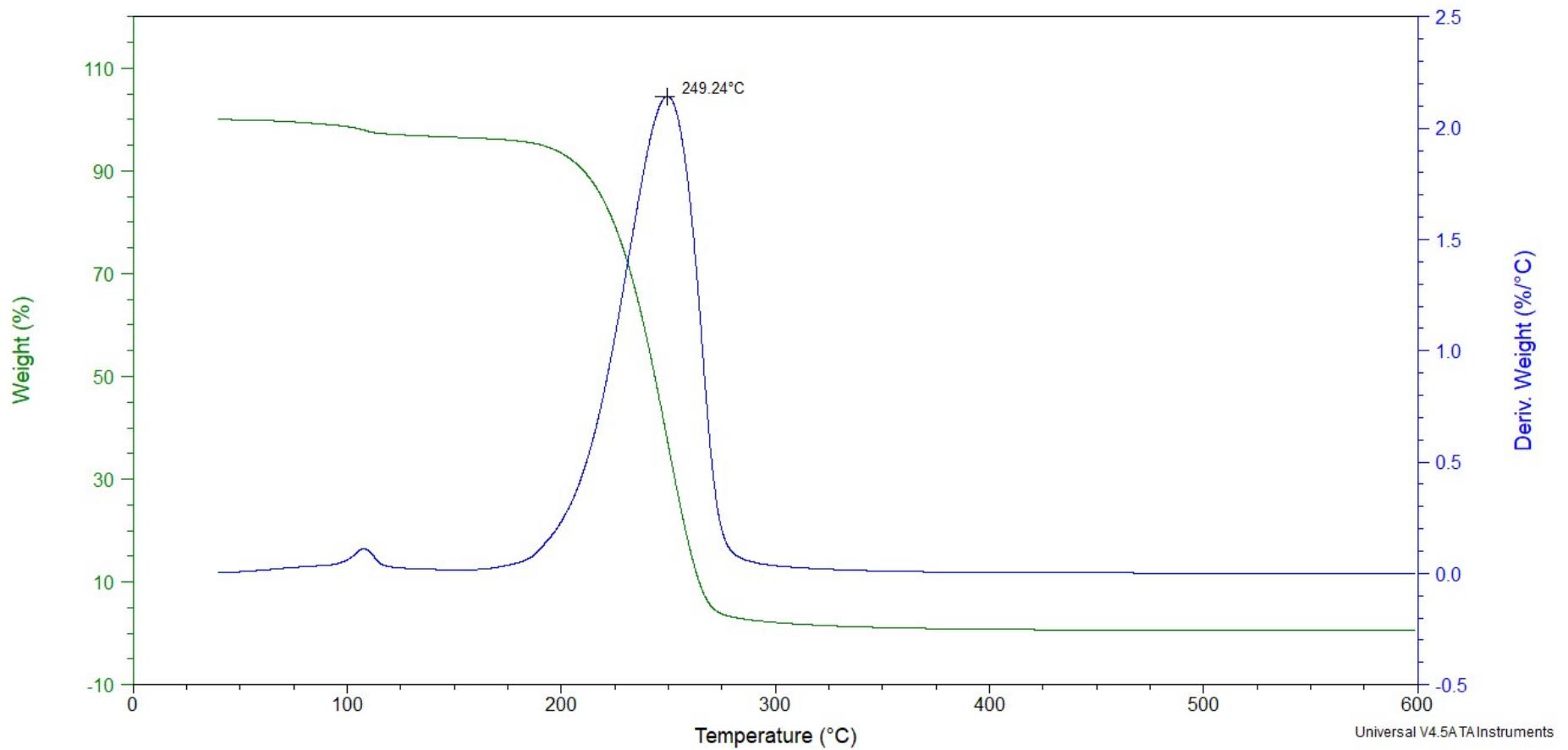
**Fig S14.** IR of P<sub>8881</sub>Lev at 25 °C



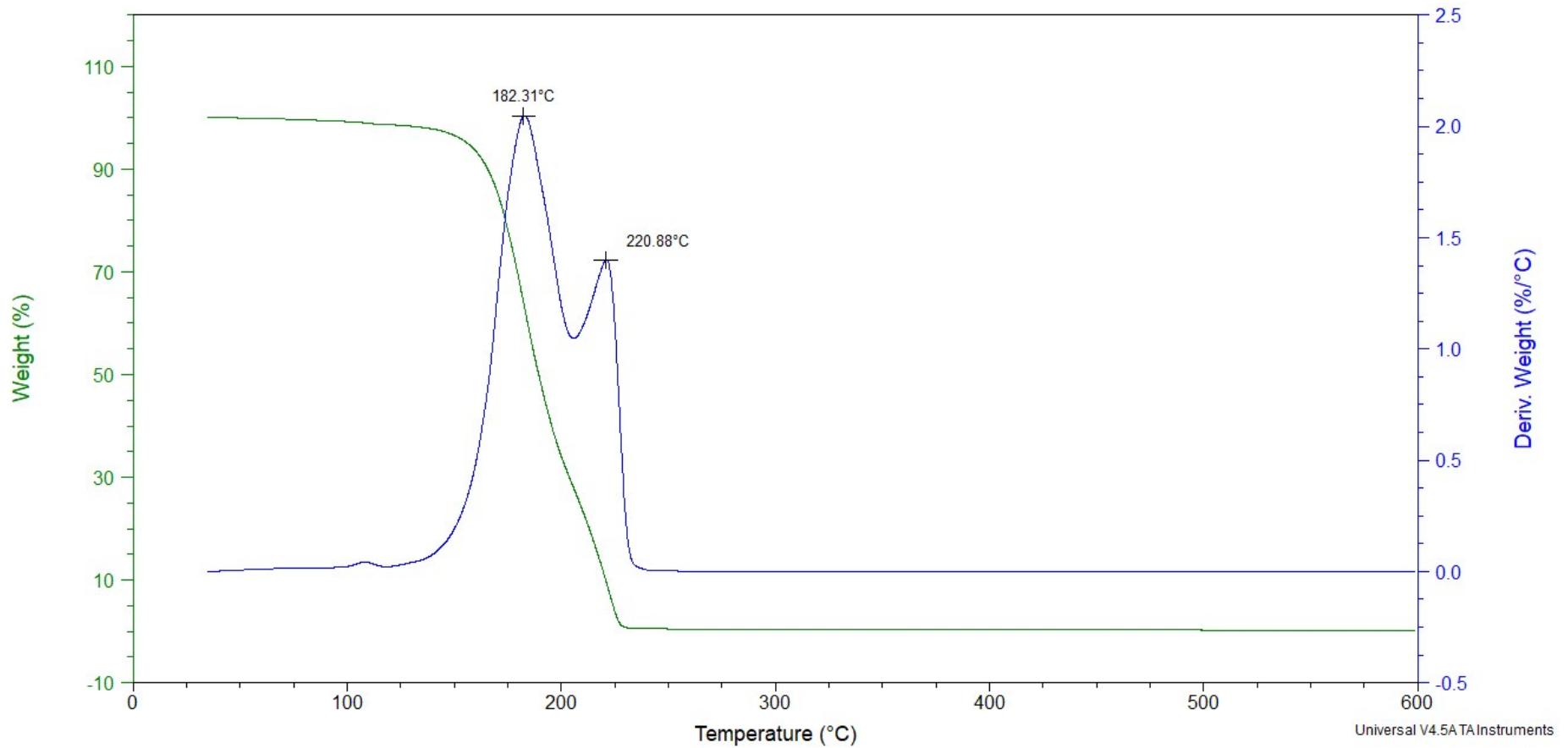
**Fig S15.** Thermal gravimetric analysis (TGA) of **EMIMLev**



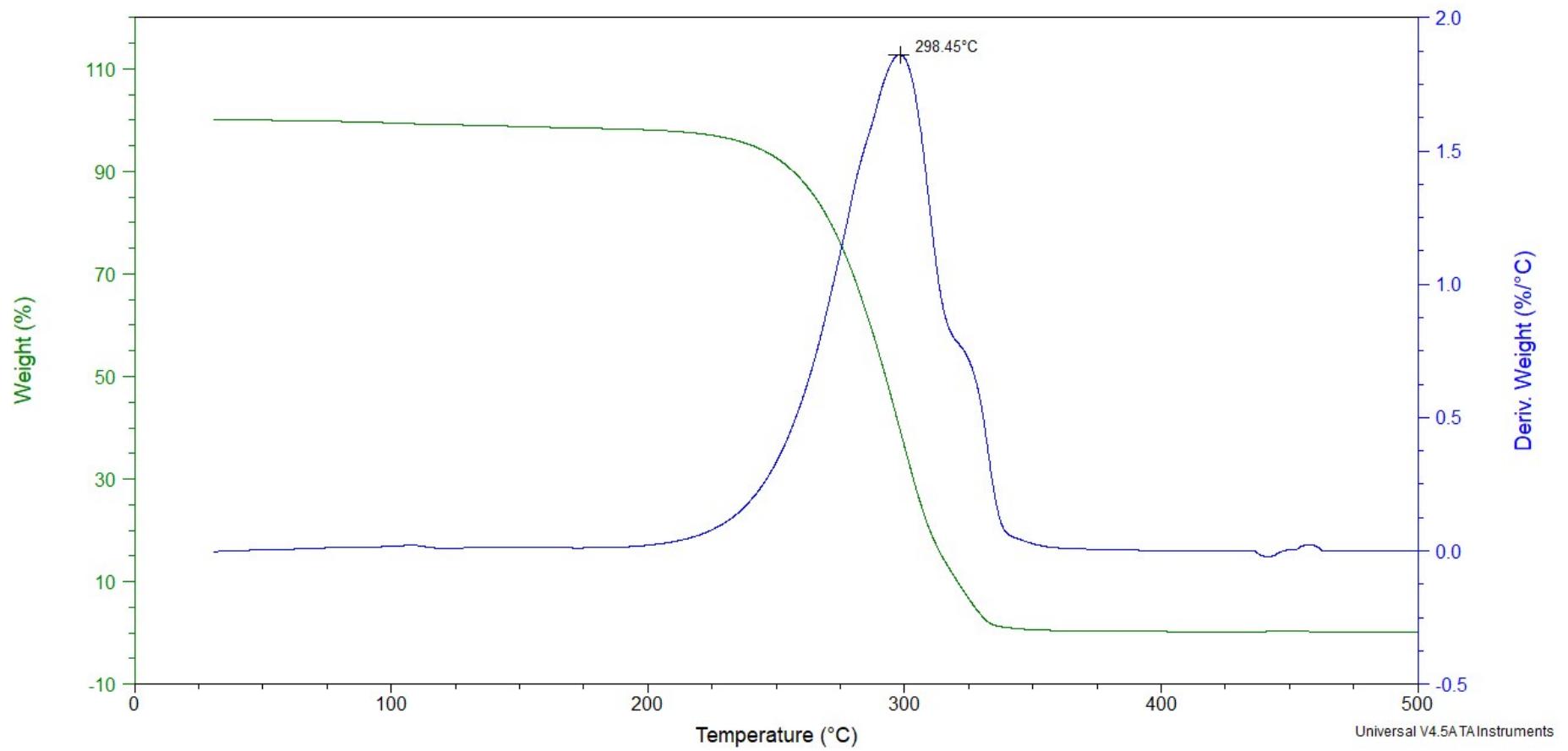
**Fig S16.** Thermal gravimetric analysis (TGA) of **BMIMLev**



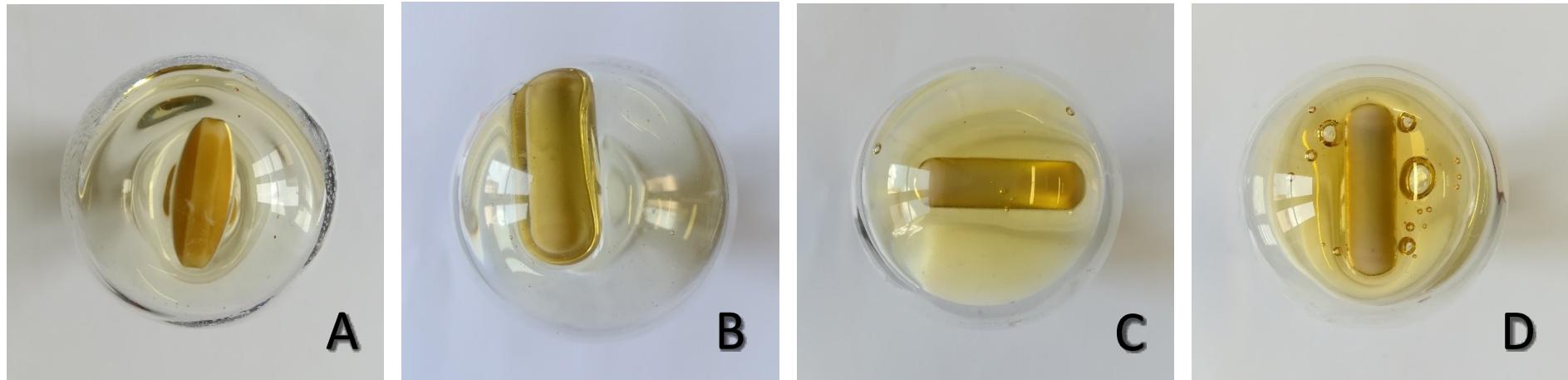
**Fig S17.** Thermal gravimetric analysis (TGA) of N<sub>8881</sub>Lev



**Fig S18.** Thermal gravimetric analysis (TGA) of **P<sub>8881</sub>Lev**



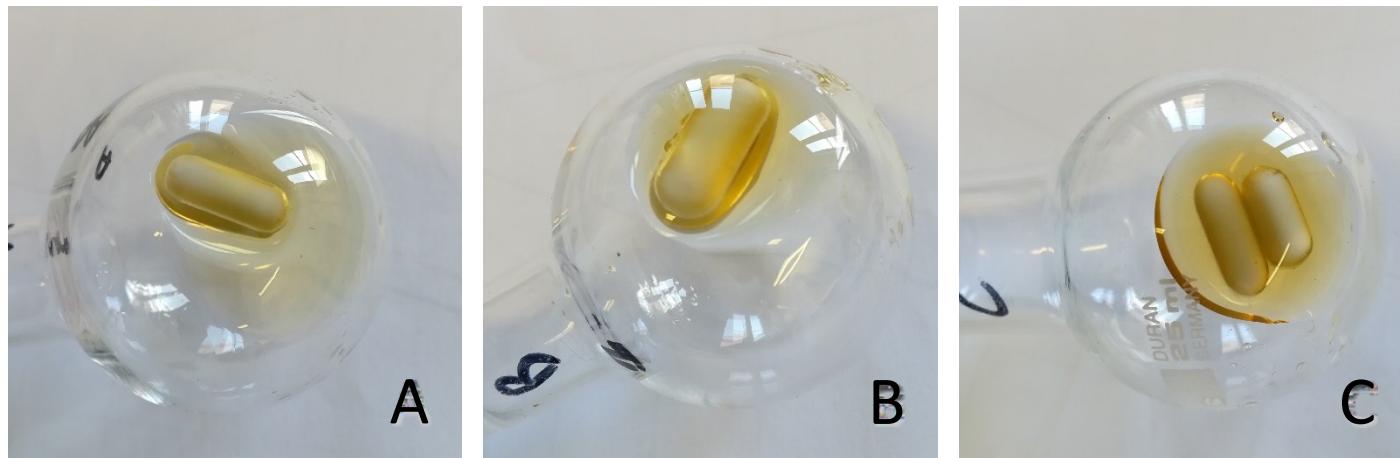
**Fig S19:** Pictures of dissolved MCC in **EMIMLev**: 25 °C, 6 wt% (**A**); 40 °C, 8 wt% (**B**); 60 °C, 18 wt%(**C**); 80 °C, 26 wt%(**D**); 100 °C, 29 wt%(**E**)



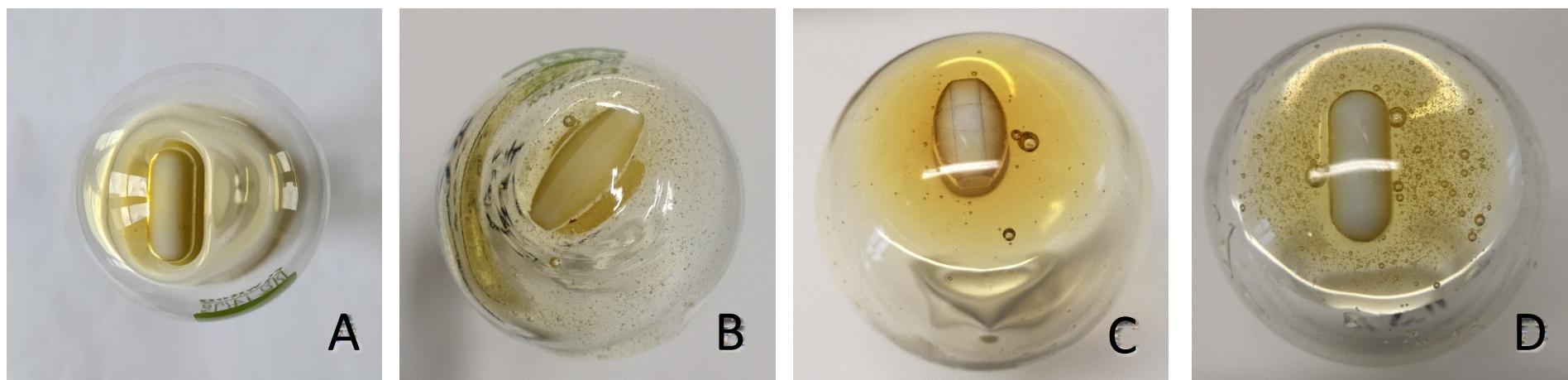
**Fig S20:** Picture of undissolved MCC in **EMIMLev** (100 °C, 30 wt%)

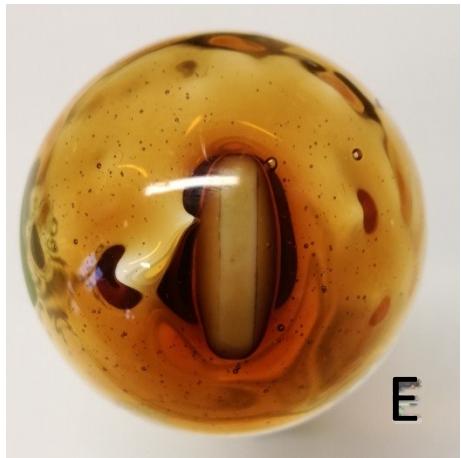


**Fig S21:** Dissolved MCC in **EMIMLev**, at 60 °C, with addition of a precise amount (10% mol) of contaminants: H<sub>2</sub>O (**A**), MeOH (**B**), EtOH (**C**)

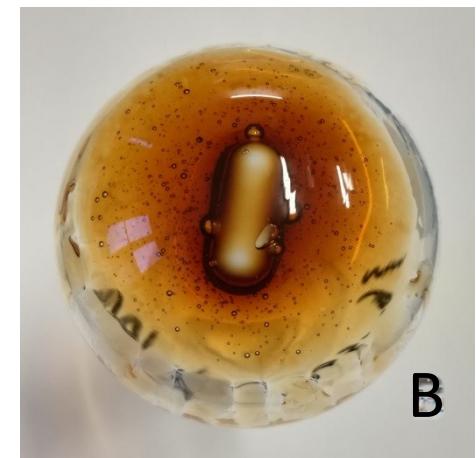
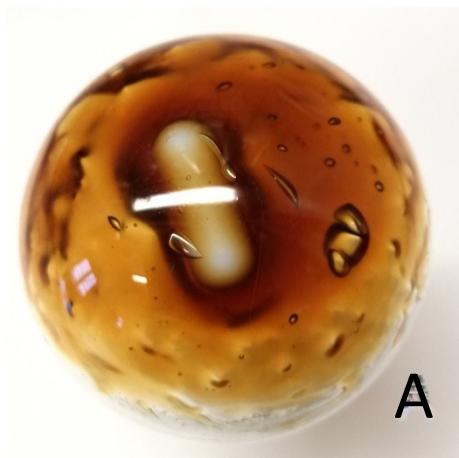


**Fig S22:** Pictures of under vacuum dissolved MCC in **EMIMLev**: 25 °C, 8 wt% (**A**); 40 °C, 12 wt% (**B**); 60 °C, 20 wt% (**C**); 80 °C, 33 wt% (**D**); 100 °C, 38 wt% (**E**)

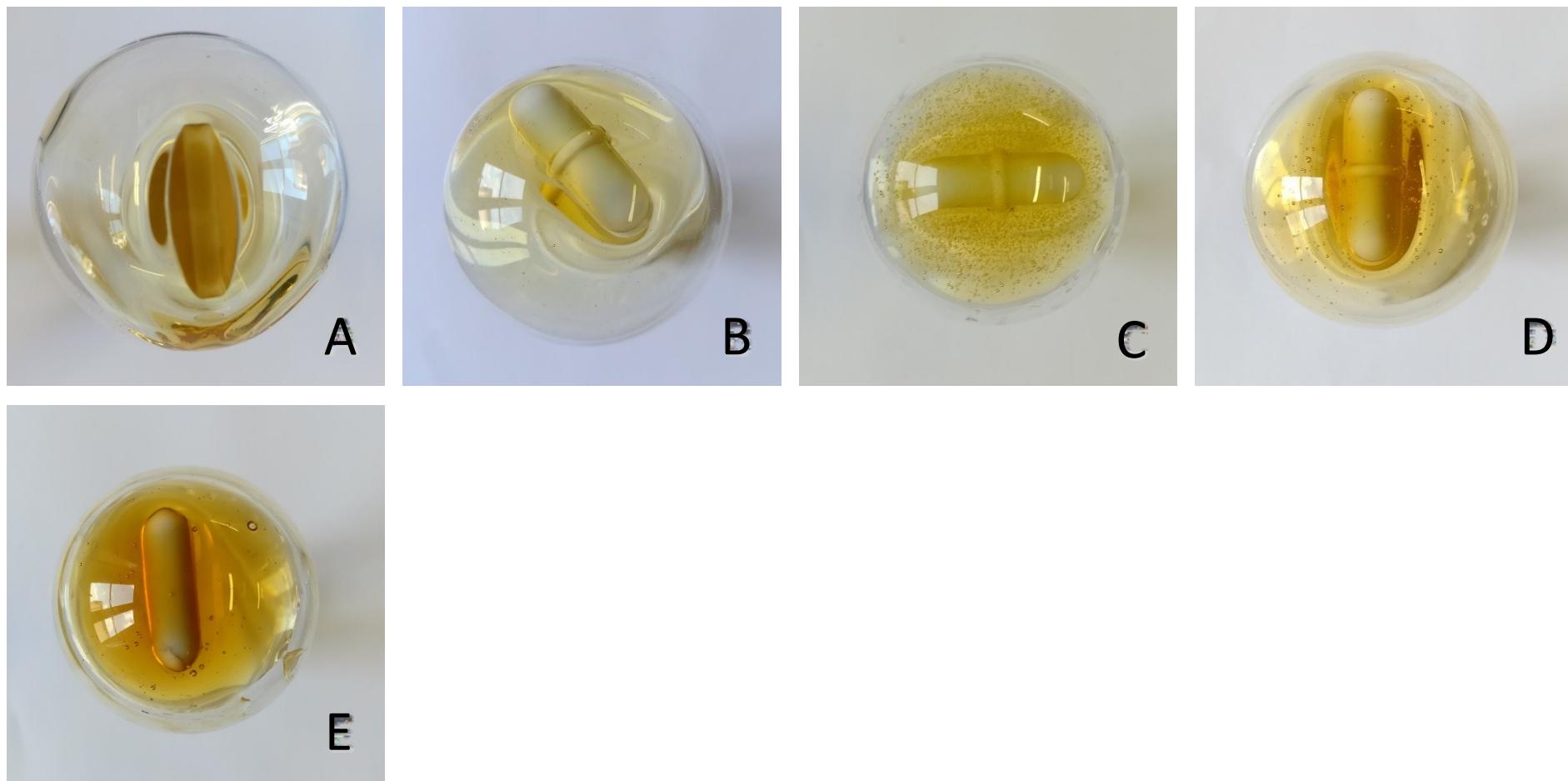




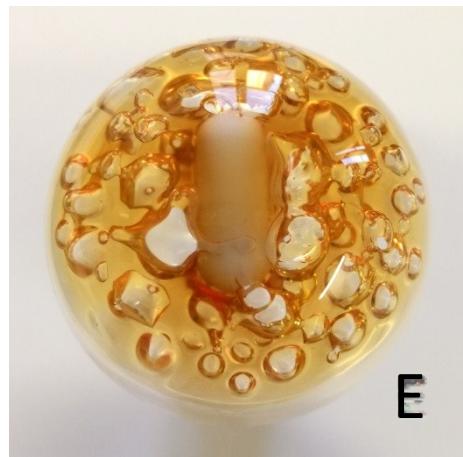
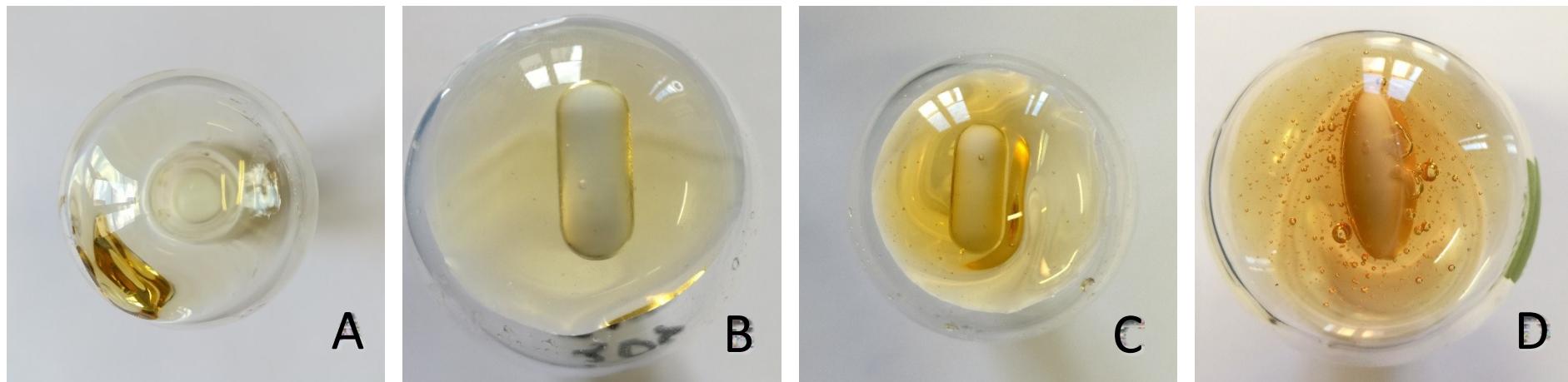
**Fig S23:** Pictures of under vacuum undissolved MCC in **EMIMLev** (A) (100 °C, 38.5 wt%) and under vacuum dissolved MCC in two-time recycled **EMIMLev** (B) (100 °C, 37% MCC)



**Fig S24:** Picture of dissolved MCC in **BMIMLev**: 25 °C, 2 wt% (**A**); 40 °C, 7 wt% (**B**); 60 °C, 16 wt% (**C**); 80°C, 22 wt%(**D**); 100 °C, 24 wt%(**E**)



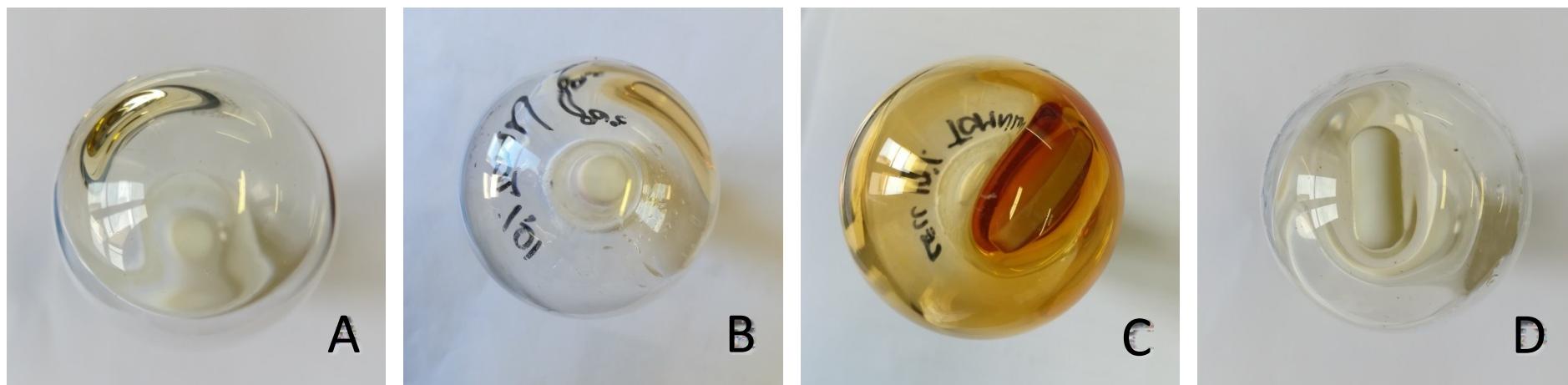
**Fig S25:** Picture of under vacuum dissolved MCC in **BMIMLev**: 25 °C, 3 wt% (A); 40 °C, 12 wt% (B); 60 °C, 25 wt% (C); 80 °C, 31 wt% (D); 100 °C, 34 wt% (E)



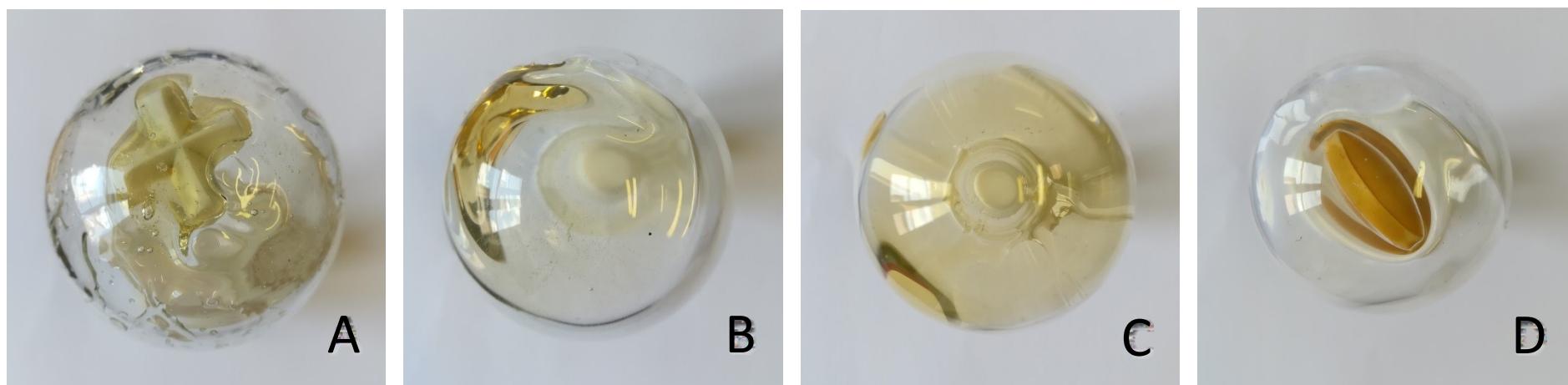
**Fig S26:** Picture of under vacuum undissolved MCC in **BMIMLev** (100 °C, 34.5 wt%)



**Fig S27:** Pictures of dissolved MCC in **N<sub>8881</sub>Lev/DMSO**: 60 °C, 9 wt% (**A**); 80 °C, 10 wt% (**B**); 100 °C, 12 wt%(**C**). MCC dissolved in **N<sub>8881</sub>Lev/DMSO** under vacuum at room temperature: 25 °C, 13 wt% (**D**)



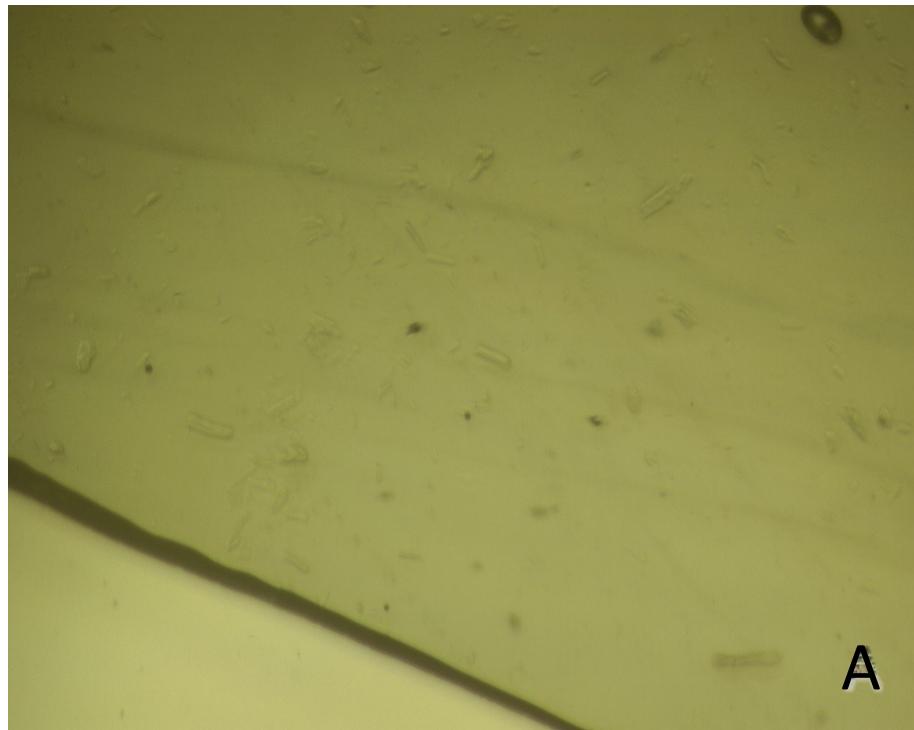
**Fig S28:** Pictures of dissolved MCC in **P<sub>8881</sub>Lev/DMSO**: 60 °C, 7 wt% (**A**); 80 °C, 8 wt% (**B**); 100 °C, 10 wt%(**C**). Cellulose dissolved in **P<sub>8881</sub>Lev/DMSO** under vacuum at room temperature: 25 °C, 11 wt% (**D**)



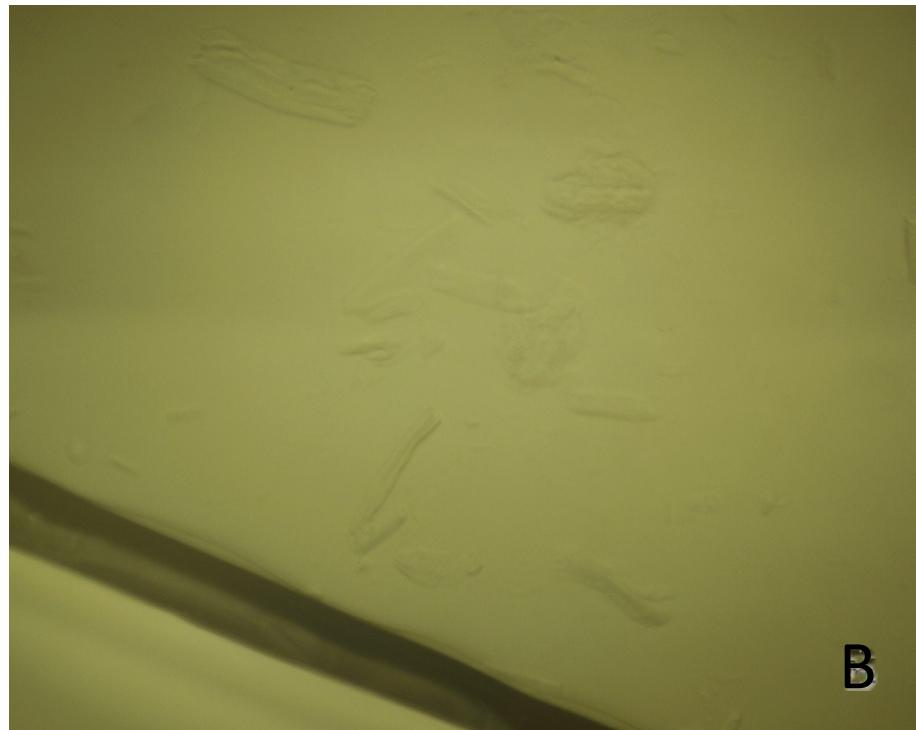
**Fig S29:** Optical microscopy of MCC dissolved in **EMIMLev** (100 °C, 29 wt%), 4x(A) and 15x (B)



**Fig S30:** Microcrystalline cellulose not completely dissolved in **EMIMLev** (100 °C, 30 wt%), 4x(A) and 15x (B),

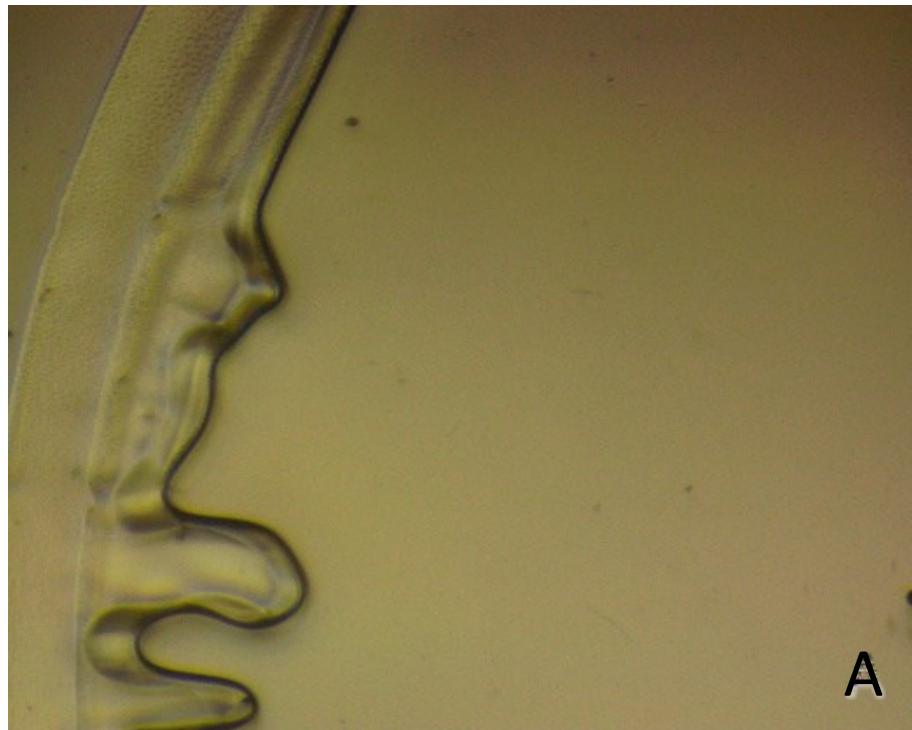


A

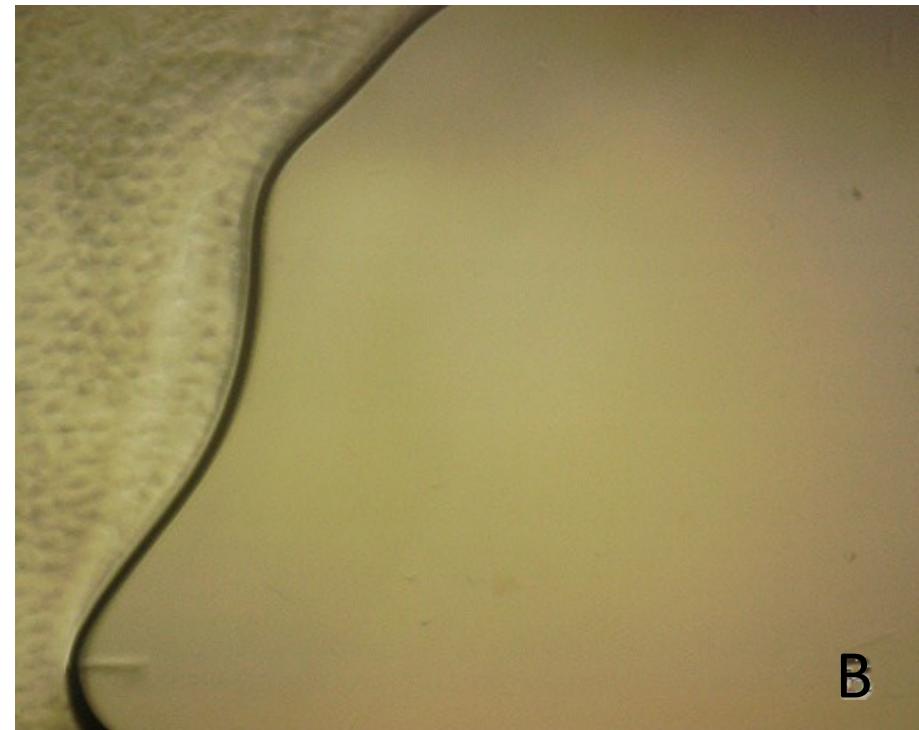


B

**Fig S31:** Optical microscopy of under vacuum dissolved MCC in **EMIMLev** (100 °C, 38 wt%), 4x(A) and 15x (B)

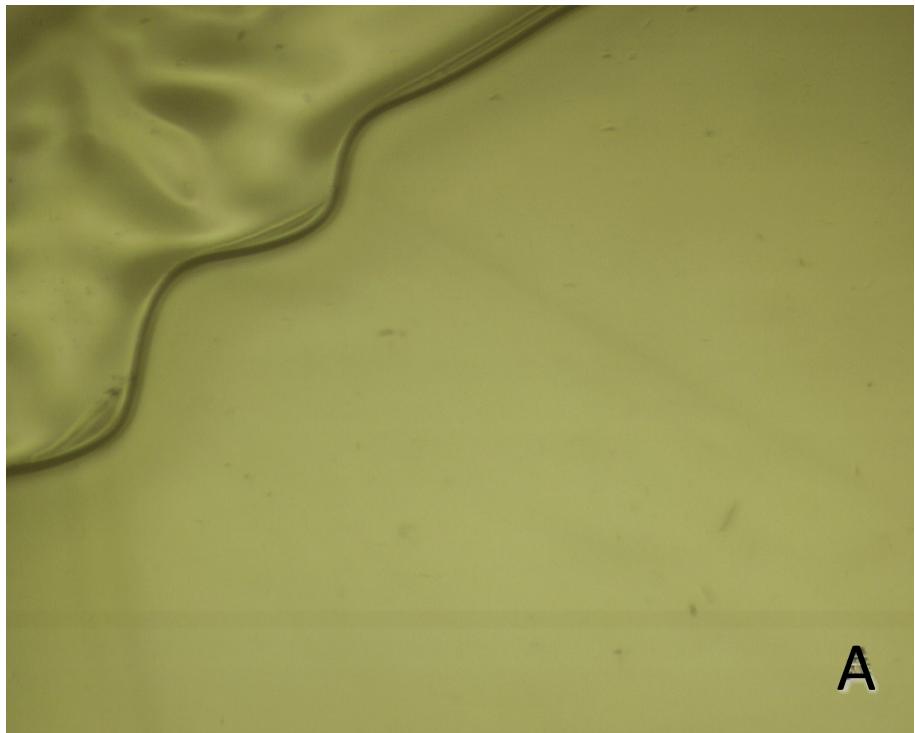


A

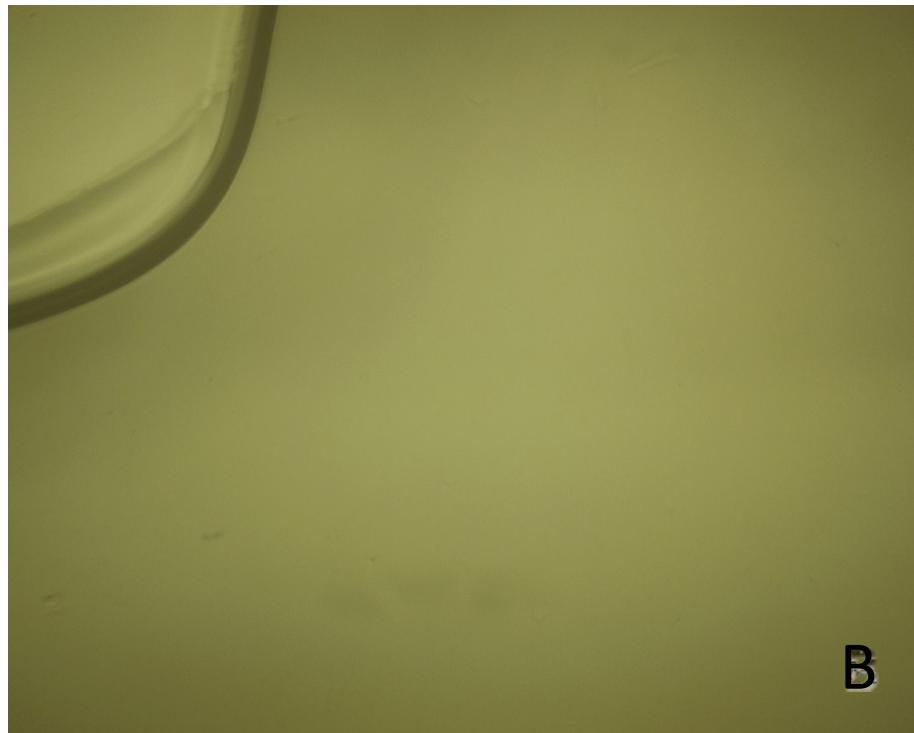


B

**Fig S32:** Optical microscopy of MCC dissolved in **BMIMLev** (100 °C, 24 wt%), 4x(A) and 15x (B)

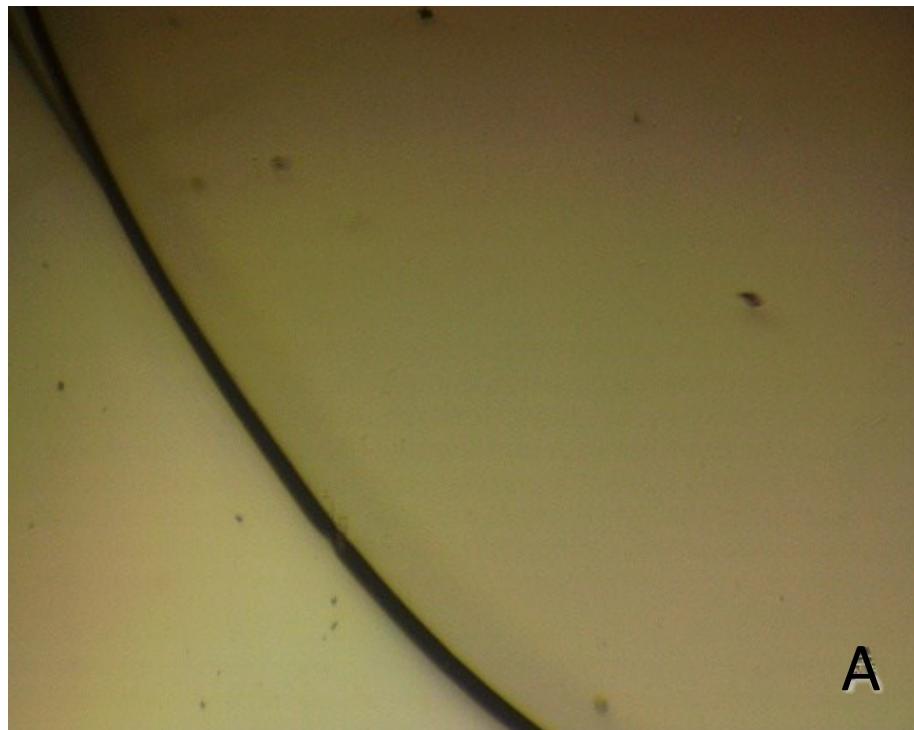


A

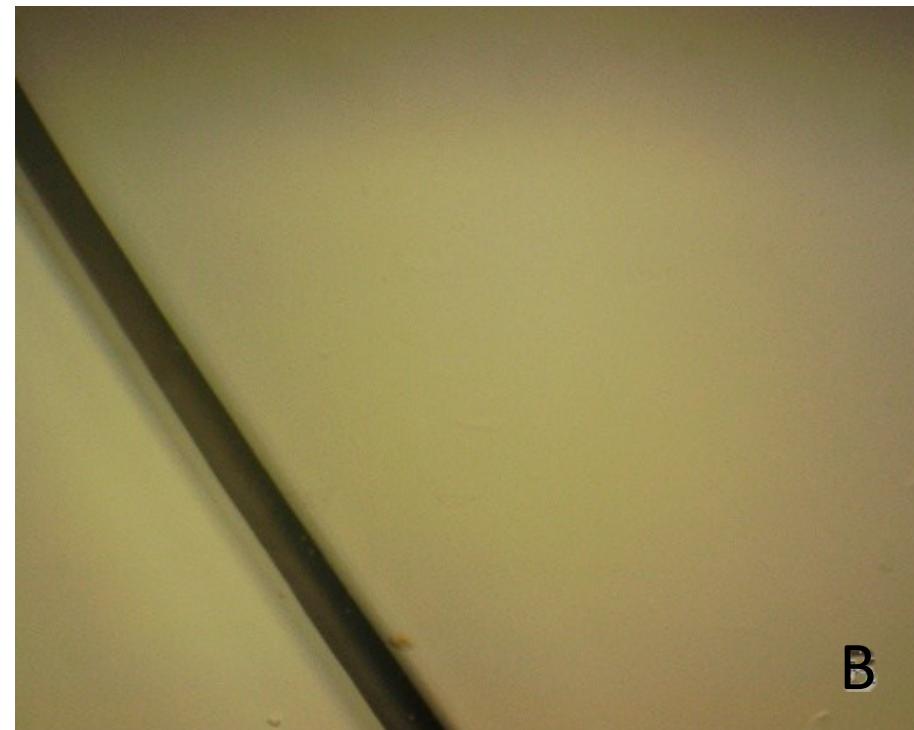


B

**Fig S33:** Optical microscopy of under vacuum dissolved MCC in **BMIMLev** (100 °C, 34 wt%), 4x(A) and 15x (B)

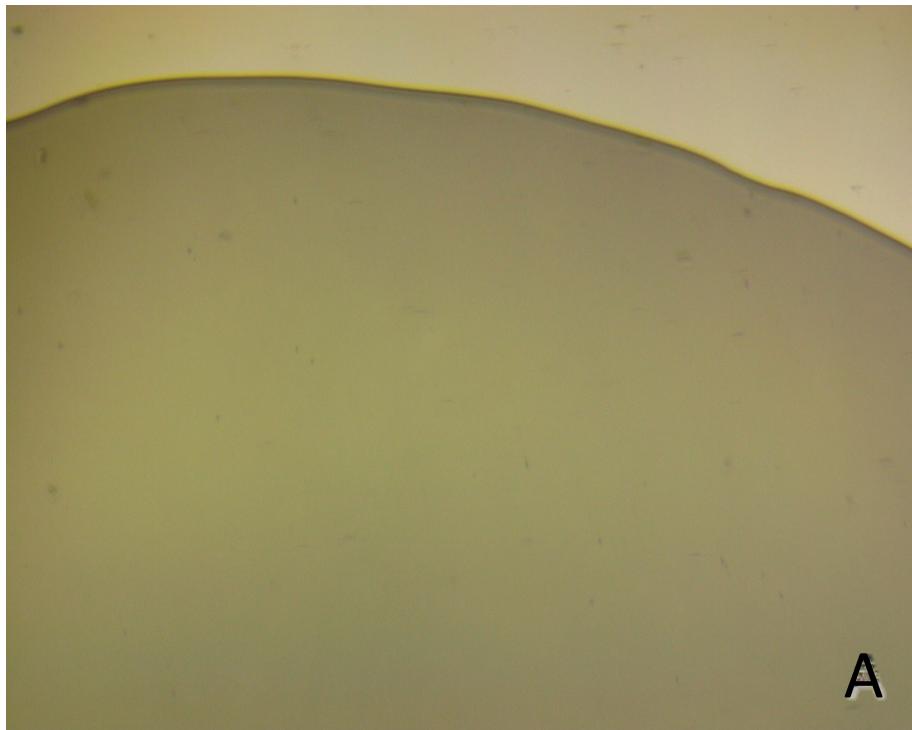


A



B

**Fig S34:** Optical microscopy of MCC dissolved in **N<sub>8881</sub>Lev/DMSO** (100 °C, 12 wt%), 4x(A) and 15x (B)

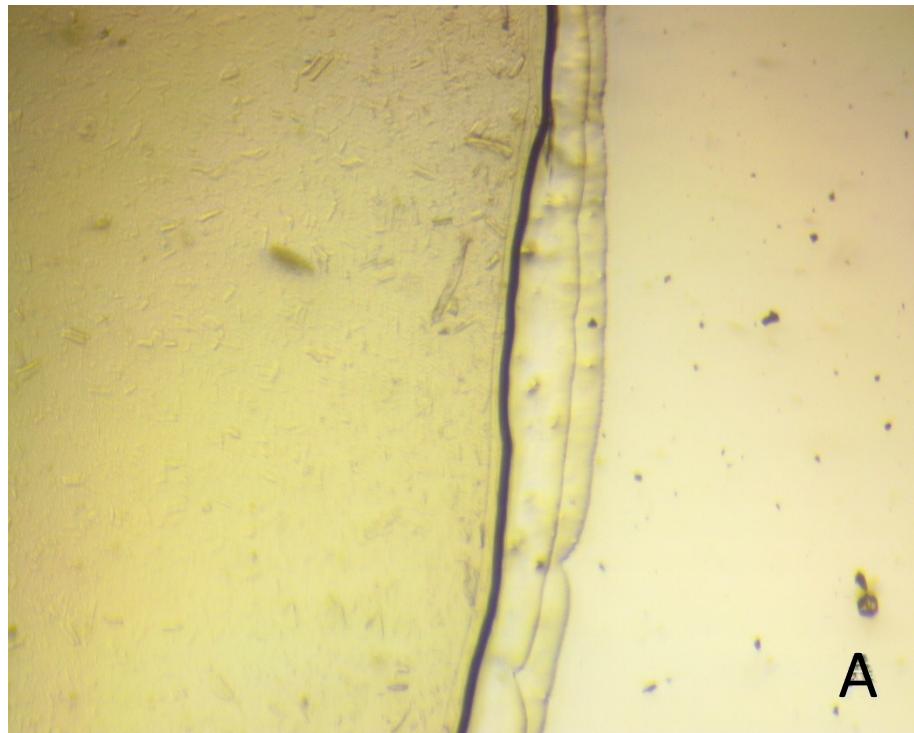


A



B

**Fig S35:** Microcrystalline cellulose not completely dissolved in **N<sub>8881</sub>Lev/DMSO** (100 °C, 13 wt%), 4x(A) and 15x (B),

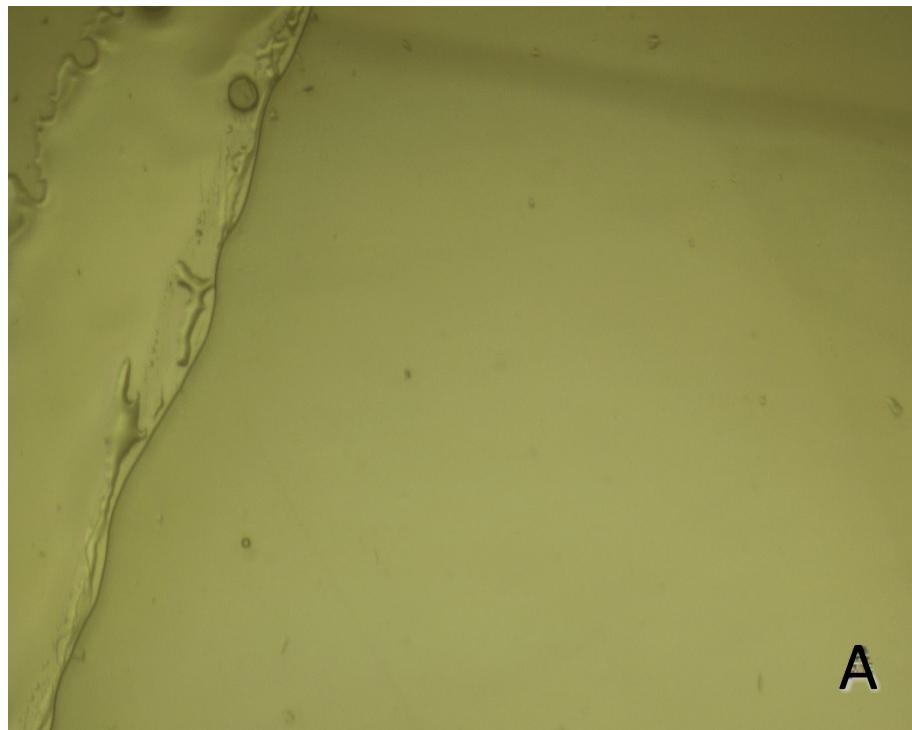


A



B

**Fig S36:** Optical microscopy of MCC dissolved in **P<sub>888</sub>1Lev/DMSO** (100 °C, 10 wt%), 4x(A) and 15x (B)

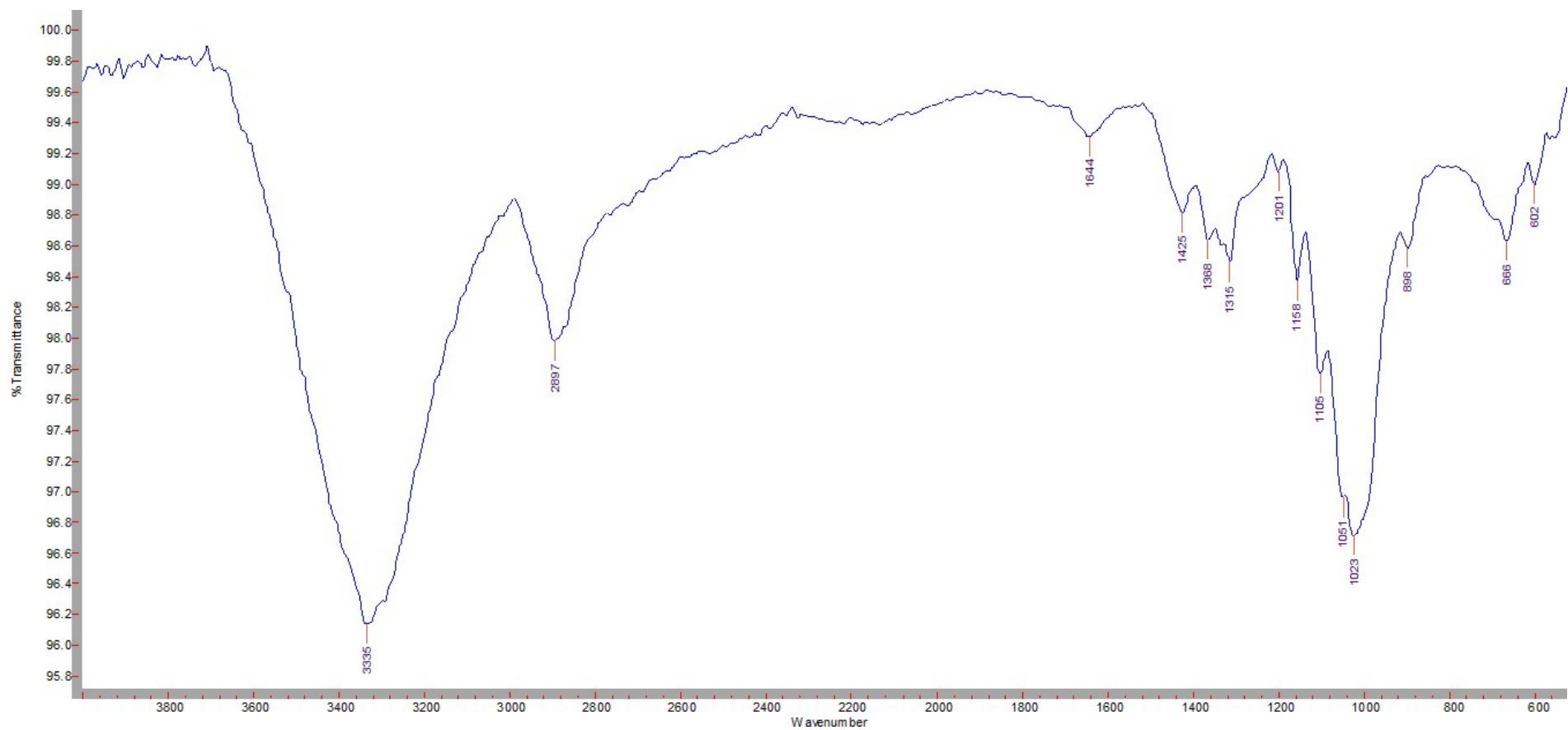


A

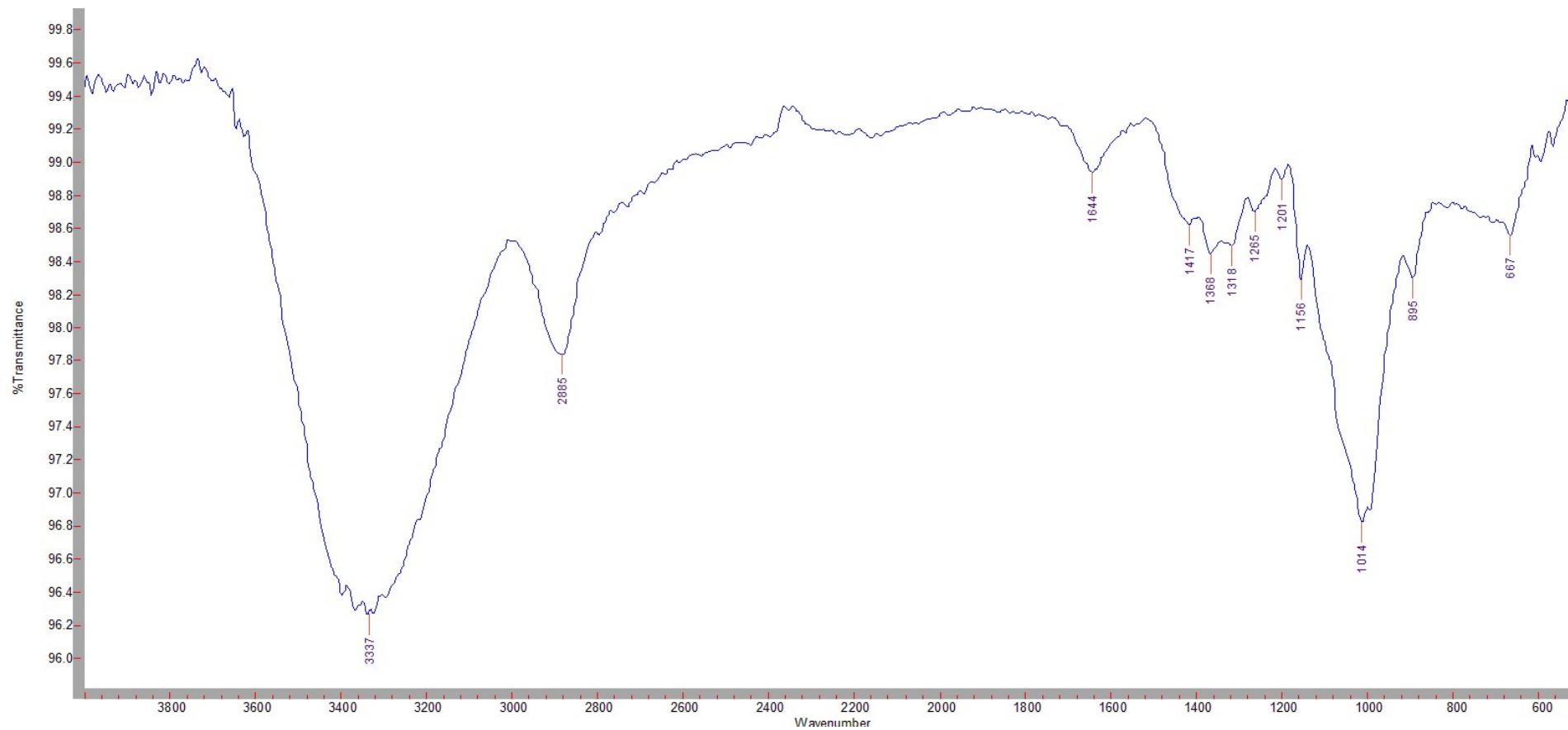


B

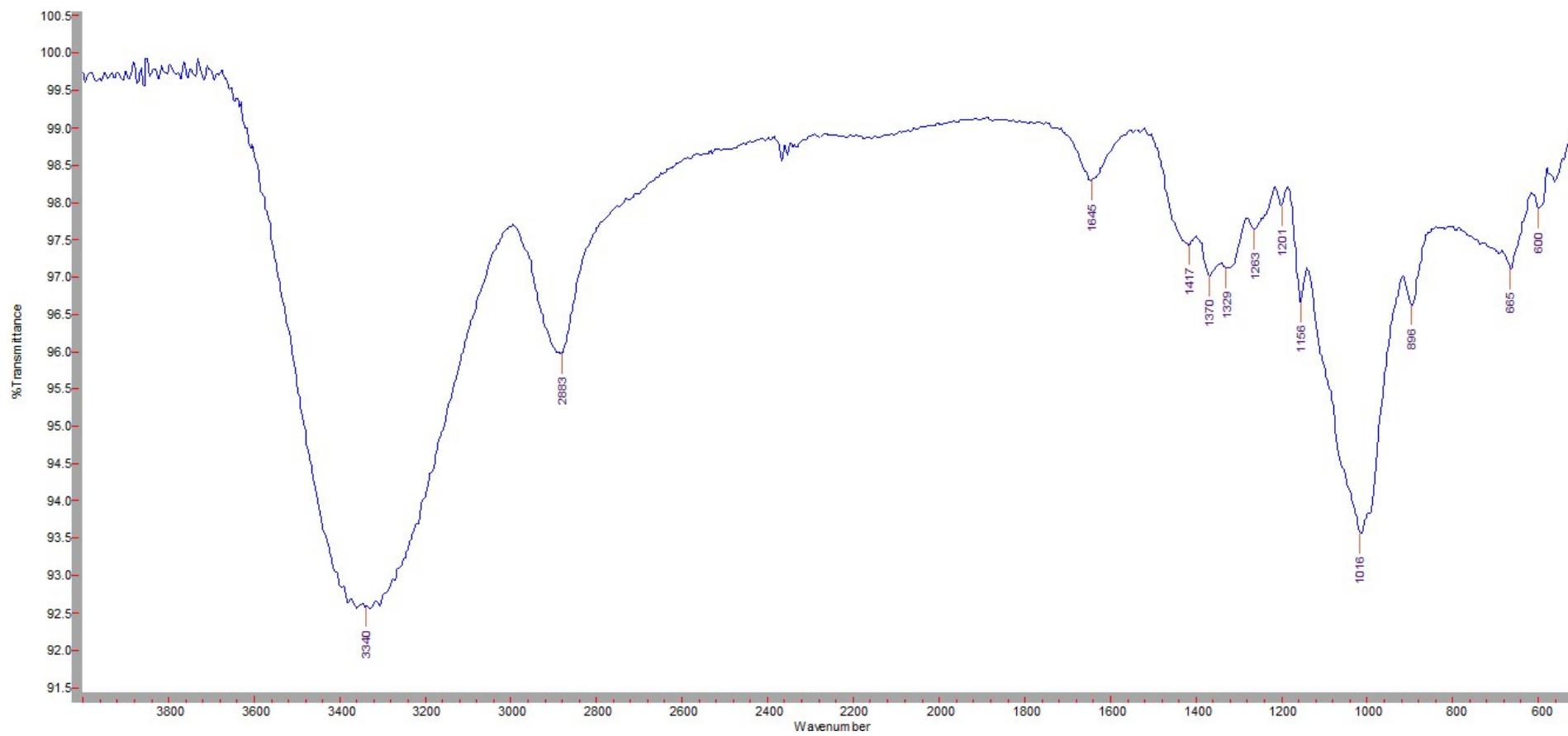
**Fig S37: IR of MCC**



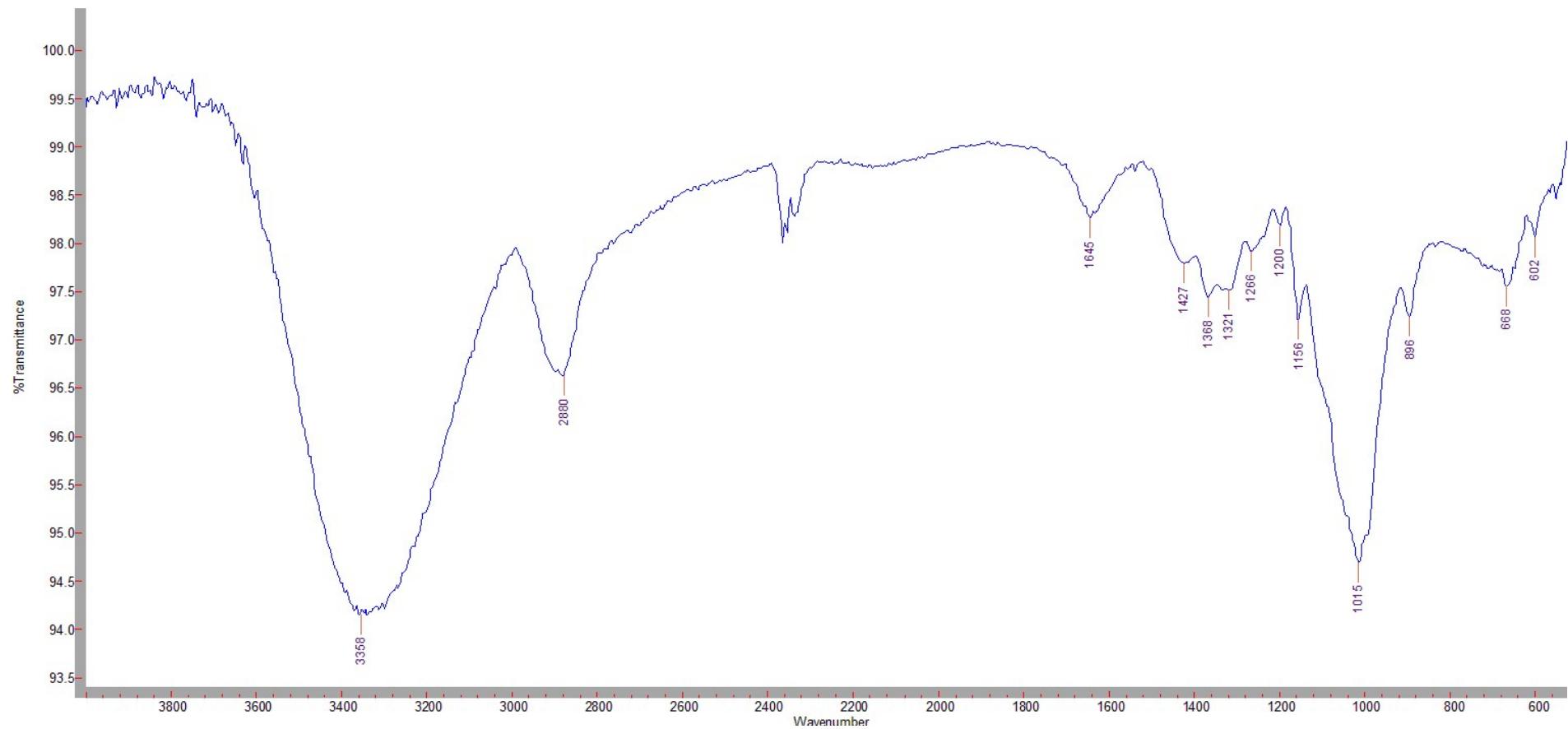
**Fig S38:** IR of regenerated cellulose after dissolution in **EMIMLev** at 100 °C



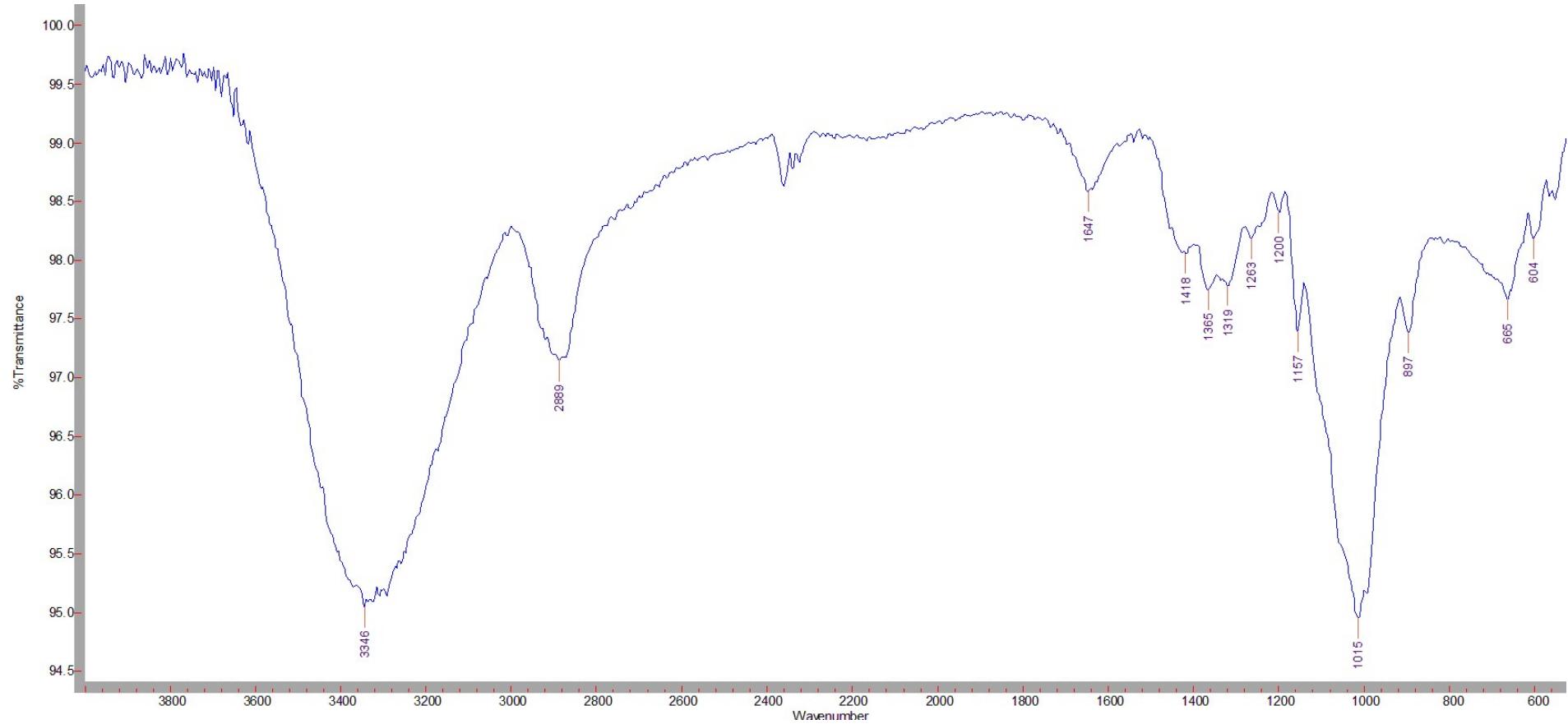
**Fig S39:** IR of regenerated cellulose after dissolution in **BMIMLev** at 100 °C



**Fig S40:** IR of regenerated cellulose after dissolution in **N<sub>8881</sub>Lev/DMSO** at 100 °C



**Fig S41:** IR of regenerated cellulose after dissolution in **P<sub>8881</sub>Lev/DMSO** at 100 °C



**Fig S42:** Interferograms of MCC and regenerated cellulose from [EMIM][Lev] at various temperatures

