

Kinetic analysis of microwave-enhanced cellulose dissolution in ionic solvents

Pablo B. Sánchez^a, Shuntaro Tsubaki^b, Agílio A. H. Pádua^a, Yuji Wada^b

^aLaboratoire de Chimie, École Normale Supérieure de Lyon & CNRS, 46 Allée d'Italie, 69007 Lyon, France

^bDepartment of Chemical Science and Engineering, School of Materials and Chemical Technology, Tokyo Institute of Technology, 2-12 Ookayama, Meguro, Tokyo 152-8552, Japan

1. Experimental information

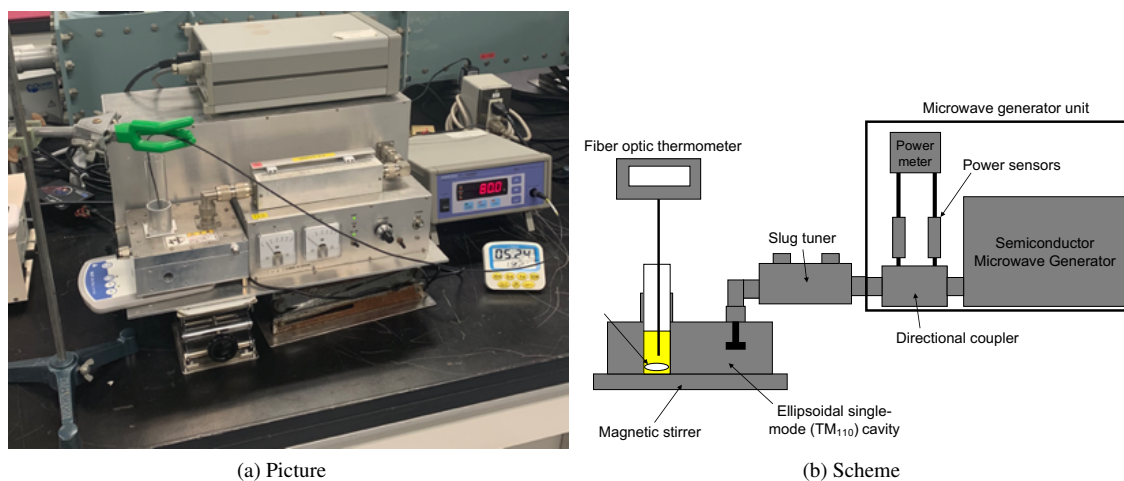


Figure S1: MW device described in the experimental section

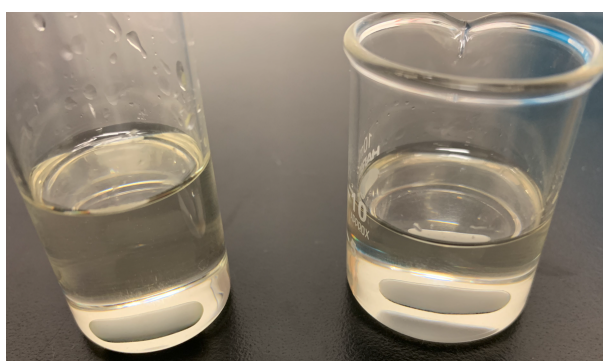


Figure S2: Vessels employed for MW (left) (diameter = 24 mm) and conventional (right) heating (diameter = 27 mm)

Email address: pablo.sanchez@ens-lyon.fr (Pablo B. Sánchez)

2. Complementary results

2.1. SEM images

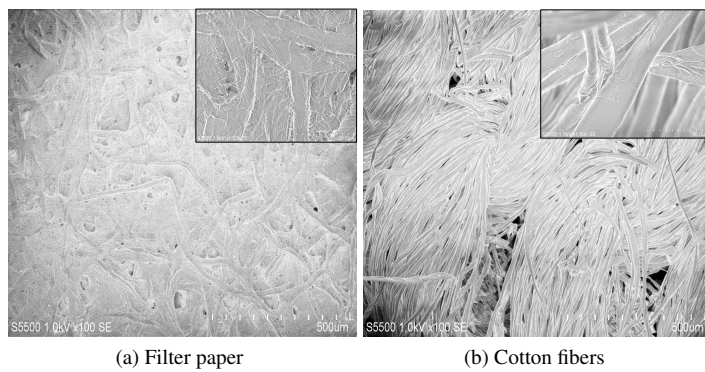


Figure S3: SEM images of the cellulose sources

2.2. XRD scans

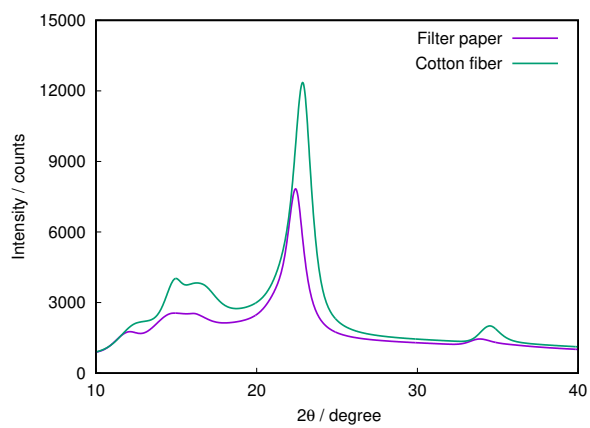


Figure S4: XRD scans of cellulose sources

2.3. Cellulose dissolution

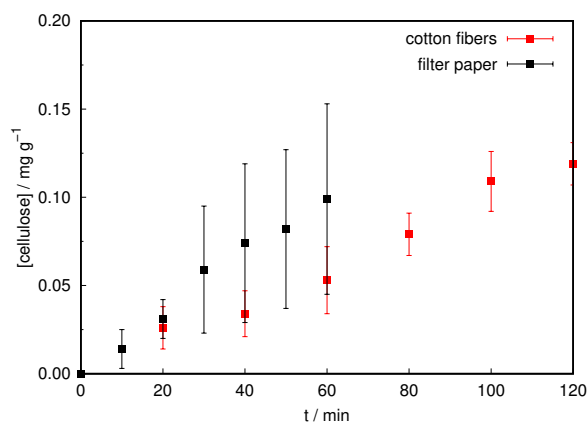


Figure S5: Rate of cellulose dissolution depending of the cellulose source

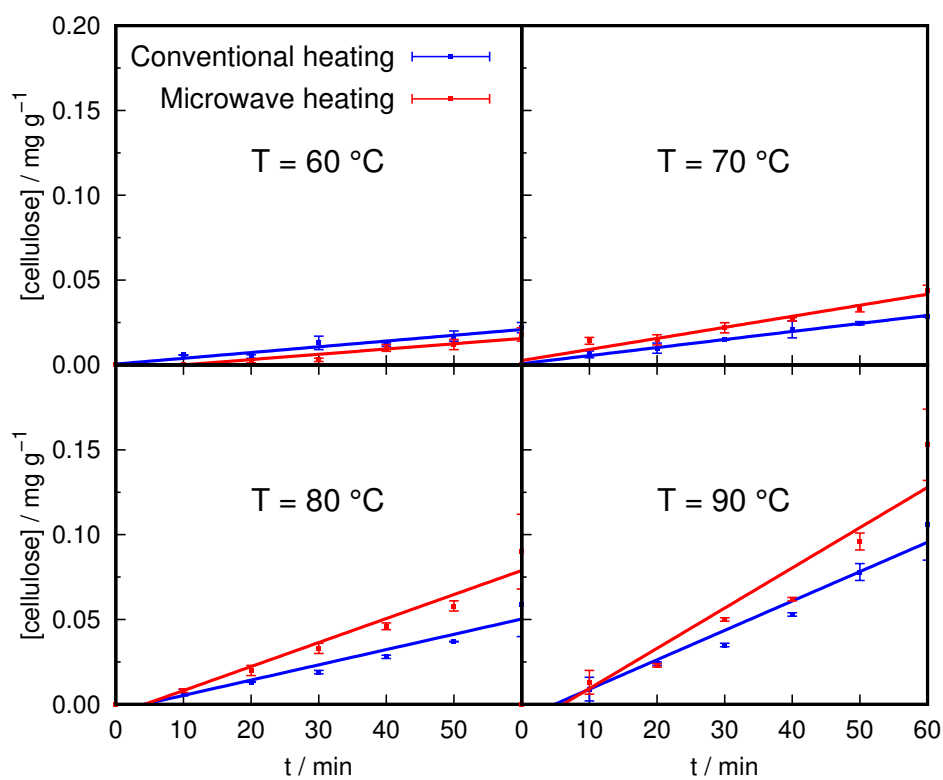


Figure S6: Linear fitting of dissolved cellulose fibers vs time for conventional and microwave heating modes at different temperatures: 60, 70, 80, 90 °C. Solvent is a mixture of $[\text{C}_2\text{C}_1\text{Im}][\text{OAc}]$ and DMSO at 50% (w/w)

T / °C	Dissolution rate / $\mu\text{g g}^{-1} \text{min}^{-1}$		Regression coefficient	
	k_{hp}	k_{mw}	r_{hp}	r_{mw}
60	0.34	0.31	0.97	0.95
70	0.47	0.65	0.99	0.98
80	0.90	1.41	0.97	0.98
90	1.73	2.37	0.98	0.96

Table S1: Dissolution rate and regression coefficients of the linear fitting presented in Figure S6

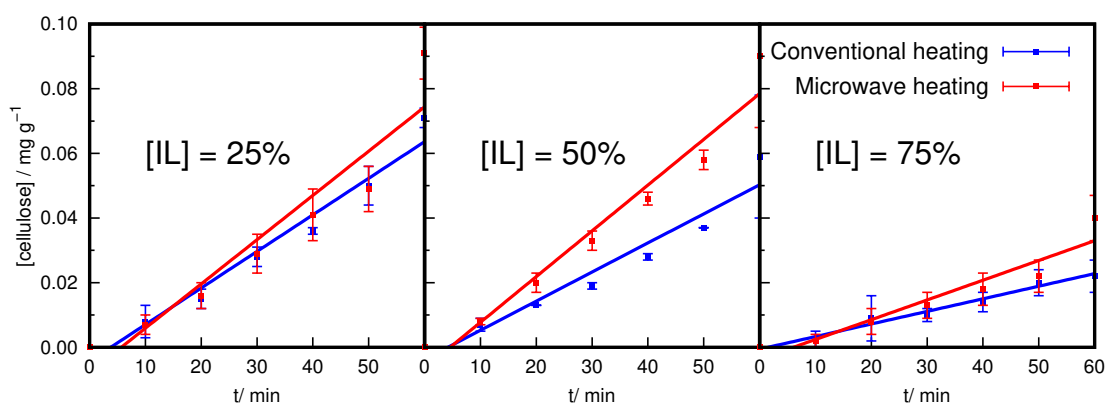


Figure S7: Linear fitting of dissolved cellulose fibers vs time for conventional and microwave heating modes at different concentrations of IL: 25, 50 and 75%. Temperature was set to 80°C in all cases.

[IL] (w/w)	Dissolution rate / $\mu\text{g g}^{-1} \text{min}^{-1}$		Regression coefficient	
	k_{hp}	k_{mw}	r_{hp}	r_{mw}
25	1.13	1.37	0.98	0.95
50	0.90	1.41	0.97	0.98
75	0.39	0.61	0.99	0.95

Table S2: Dissolution rate and regression coefficients of the linear fitting presented in Figure S7

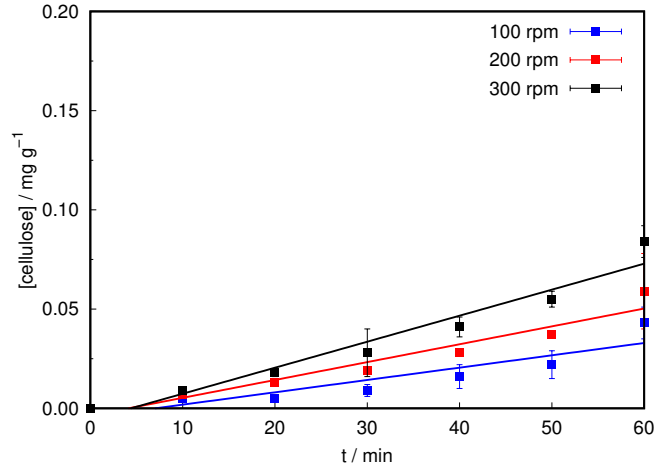


Figure S8: Linear fitting of dissolved cellulose fibers vs time for conventional at different stirring speeds: 100, 200 and 300 rpm. Concentration of IL is 50% and temperature was set to 80°C

stirring speed /rpm	Dissolution rate / $\mu\text{g g}^{-1} \text{min}^{-1}$	Regression coefficient
	k_{hp}	r_{hp}
100	0.62	0.92
200	0.90	0.97
300	1.31	0.97

Table S3: Dissolution rate and regression coefficients of the linear fitting presented in Figure S8

2.4. Evolution of viscosity and water content during dissolution

[IL] (w/w)	before cellulose dissolution			after cellulose dissolution		
	A	B	T_0	A	B	T_0
25	0.102	550.4	152.5	0.109	568.0	152.9
50	0.122	604.2	165.0	0.123	634.9	162.9
75	0.052	961.0	150.0	0.090	821.1	162.4

Table S4: Parameters of Vogel-Fulcher-Tamman fitting shown in figure 6

2.5. Dielectric spectra

The contribution of conductivity in dielectric spectra in the Figure 7 of the manuscript was done according to the equation 1 where ϵ''_{diel} accounts for dielectric loss, σ is the conductivity, ω is frequency and ϵ_0 the absolute permittivity.

$$\text{Material loss} = \epsilon''_{diel} + \frac{\sigma}{\omega\epsilon_0} \quad (1)$$

Given than the second term of the addition accounts for the contribution of conductivity, we have proceeded as follows:

1. Material loss in the lower frequency range was fitted to $1/\omega$. Dielectric spectra given by the impedance analyser was used with this purpose.
2. According to equation 1, slope obtained in the previous step equals to σ / ϵ_0 .
3. Once σ is obtained, equation 1 can be used to disentangle dielectric and conductive contributions to material loss.

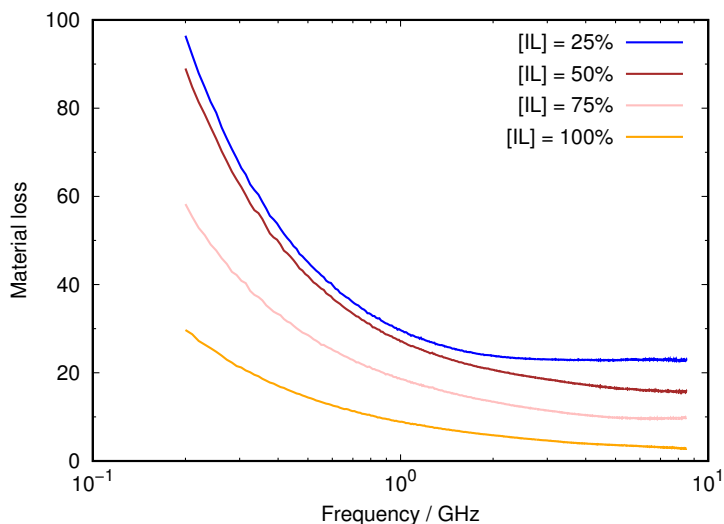


Figure S9: Dielectric spectra of $[C_2C_1Im][OAc]$ + DMSO mixtures from 200 MHz and 8.5 GHz. Concentration range of $[C_2C_1Im][OAc]$ between 25 to 100%

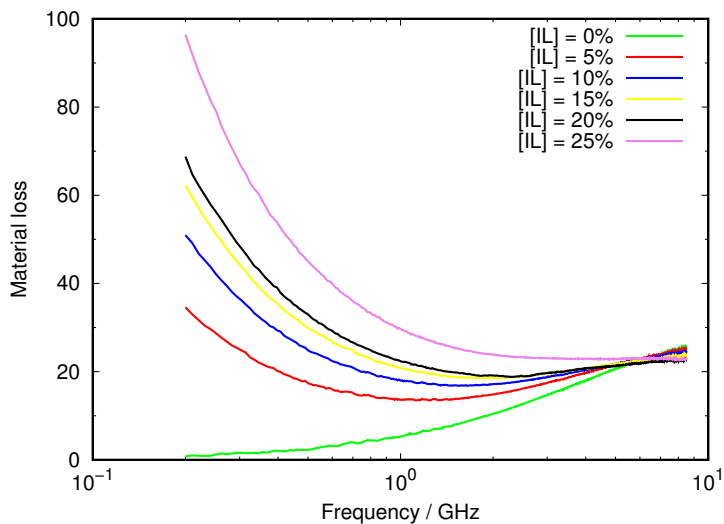


Figure S10: Dielectric spectra of $[C_2C_1Im][OAc]$ + DMSO mixtures from 200 MHz and 8.5 GHz. Concentration range of $[C_2C_1Im][OAc]$ between 0 to 25%

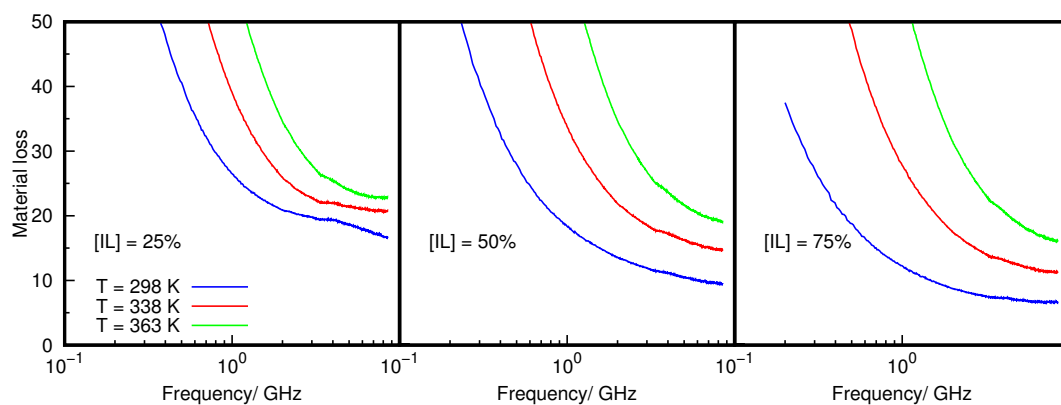


Figure S11: Dielectric spectra of $[\text{C}_2\text{C}_1\text{Im}][\text{OAc}] + \text{DMSO}$ at three concentrations - 25, 50, 75% - and temperatures - 298, 338, 363 K.

2.6. Heating rate

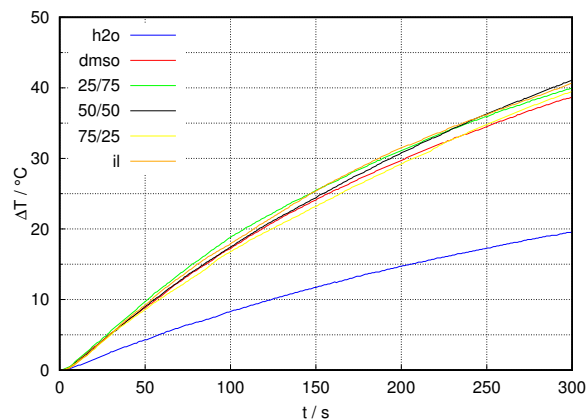


Figure S12: Heating rate of $[\text{C}_2\text{C}_1\text{Im}][\text{OAc}] + \text{DMSO}$ mixtures at different concentrations, $[\text{DMSO}] = 25\%$ (black), 50% (green) and 75% (red, respectively).