

RSC Materials Advances

Supporting Information (SI)

A novel characterization technique for hydrogels – *in situ* Rheology- Raman spectroscopy for gelation and polymerization tracking

Sina Lambrecht,^a Marek Biermann,^b Selin Kara,^{c,d} Stefan Jopp,^a Johanna Meyer^{*,d}

^a Institute of Chemistry, University of Rostock, Albert-Einstein-Str. 3a, 18059, Rostock, Germany.

^b Anton Paar OptoTec GmbH, Lise-Meitner-Str. 6, 30926 Seelze, Germany.

^c Department of Biological and Chemical Engineering, Aarhus University, Gustav Wieds Vej 10, 8000 Aarhus, Denmark.

^d Institute of Technical Chemistry, Leibniz University Hannover, Callinstraße 5, 30167 Hannover, Germany.

E-mails: johanna.meyer@iftc.uni-hannover.de

Table of Contents

1	Synthetic procedures and analytical data	S3
1.1	NMR spectra	S4
1.2	IR spectra	S6
2	Rheological data	S7

1. Synthetic procedures and analytical data

Synthetic procedure for the synthesis of Methyl-6-iodo- α -D-glucopyranoside (1). Methyl- α -D-glucopyranoside (10 mmol), triphenylphosphine (15.5 mmol), iodine (14.5 mmol), and imidazole (20 mmol) were refluxed in THF (60 mL) for 4 h. The resulting solid was filtered off, the solvent was removed and the product (91 % yield) was obtained after column chromatography (CHCl₃/MeOH 12:1). ¹H-NMR (300 MHz, DMSO-d₆): δ = 2.87–2.95 (m, 1H); 3.16–3.27 (m, 3H); 3.31 (s, 3H, CH₃); 3.34–3.42 (m, 1H); 3.50–3.57 (m, 1H); 4.54 (d, 1H, ³J = 3.65 Hz, H-1); 4.78 (d, 1H, ³J = 6.43 Hz, OH); 4.86 (d, 1H, ³J = 4.99 Hz, OH); 5.17 (d, 1H, ³J = 5.83 Hz, OH). ¹³C-NMR (75 MHz, DMSO-d₆): δ = 9.5 (C-6); 54.6 (CH₃); 70.9, 71.9, 72.7, 74.1 (C-2, C-3, C-4, C-5); 99.8 (C-1). ATR-FTIR: 3430, 3285, 2911, 2880, 2840, 1455, 1032 cm⁻¹.

Synthetic procedure for the quaternization with imidazoles to 1-(Methyl- α -D-glucopyranosid-6-yl)-3-vinylimidazolium iodide (GVIM-I) (2). Methyl-6-iodo- α -D-glucopyranoside (3 mmol) and the *N*-vinylimidazole (5 mmol) were dissolved in DMF (5 mL) and stirred at 95 °C for 24 hours. After cooling down, ethyl acetate (40 mL) was added and the flask was stored in a fridge overnight. The solvent was decanted and the precipitated solid was repeatedly washed with ethyl acetate and dried under a high vacuum to achieve the product, yielding in a light-brown solid (73 % yield). ¹H-NMR (250 MHz, D₂O): δ = 3.25 (s, 3 H, OCH₃); 3.24–3.28 (m, 1H, H-4); 3.58 (dd, 1H, ³J = 9.77 Hz, ³J = 3.77 Hz, H-2); 3.66–3.75 (m, 1H, H-3); 3.95 (ddd, 1H, ³J = 9.96 Hz, ³J = 7.47 Hz, ³J = 2.46 Hz, H-5); 4.50 (dd, 1H, ²J = 14.55 Hz, ³J = 7.38 Hz, H-6a); 4.70 (dd, 1H, ²J = 14.55 Hz, ³J = 2.55, H-6b); 4.85 (d, 1H, ³J = 3.77 Hz, H-1); 5.49 (dd, 1H, ³J = 8.68 Hz, ²J = 2.84 Hz, Vinyl-CH₂); 5.86 (dd, 1H, ³J = 15.58 Hz, ²J = , Vinyl-CH₂); 7.2 (dd, 1H, ³J = 15.58 Hz, ³J = 8.70 Hz, Vinyl-CH); 7.70 (d, 1H, ³J = 2.09 Hz, H_{Ar}); 7.86 (d, 1H, ³J = 2.11 Hz, H_{Ar}); 9.16 (s, 1H, H_{Ar}). ¹³C-NMR (125 MHz, D₂O): δ = 50.2 (C-6); 55.1 (OCH₃); 69.2 (C-5); 70.5 (C-4); 71.0 (C-2); 72.8 (C-3); 99.3 (C-1); 109.8 (Vinyl-CH₂); 119.4, 123.8 (CH_{Ar}); 123.8 (Vinyl-CH); 135.0 (CH_{Ar}). ATR-FTIR 3396, 3088, 2998, 2910, 1657, 1566, 1549, 1048, 1015 cm⁻¹.

1.1 NMR Spectra

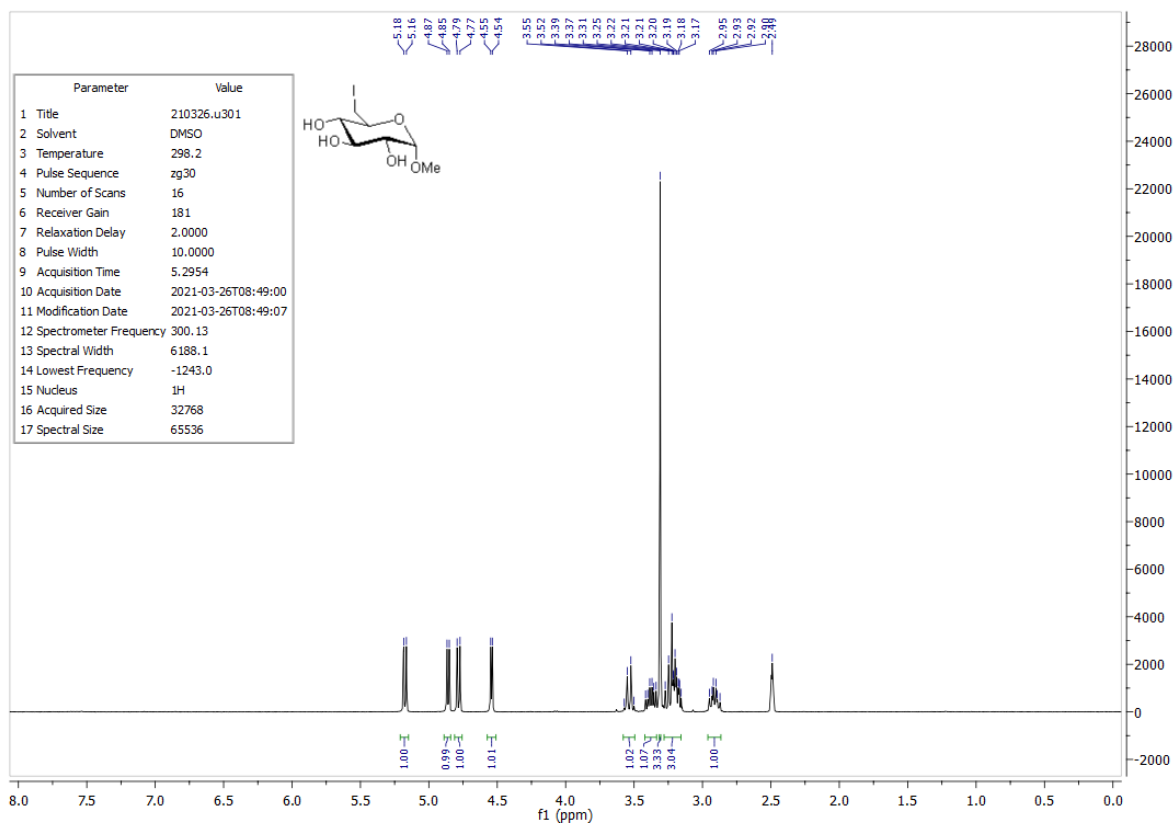


Figure S1. ¹H-NMR (DMSO) spectrum of compound 1.

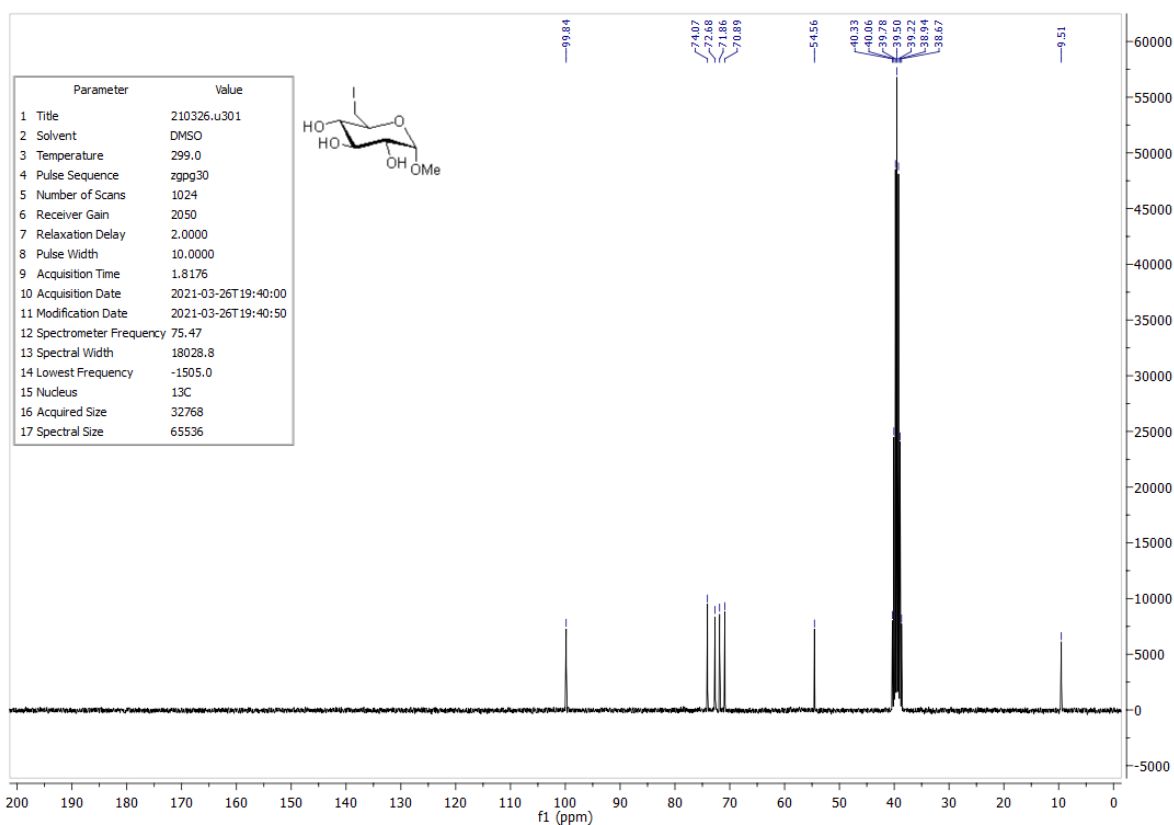


Figure S2. ¹³C-NMR (DMSO) spectrum of compound 1.

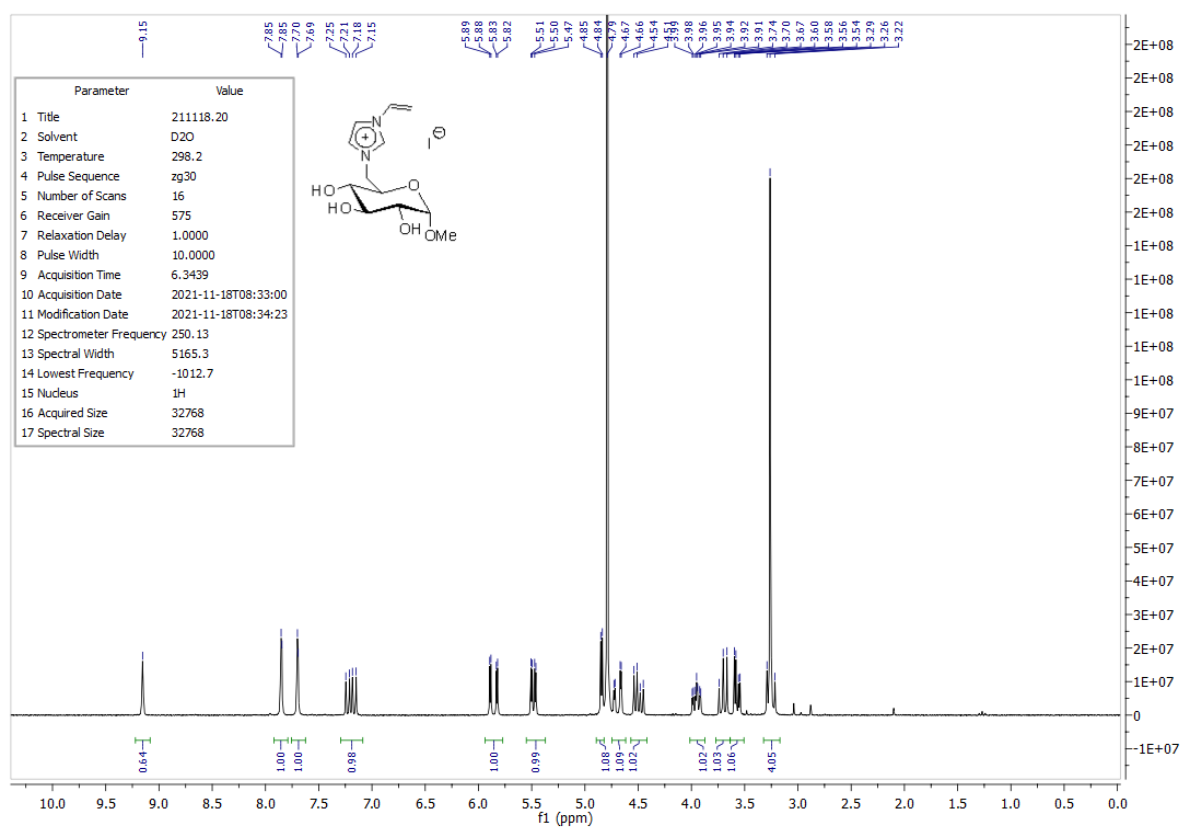


Figure S3. ¹H-NMR (D₂O) spectrum of compound 2.

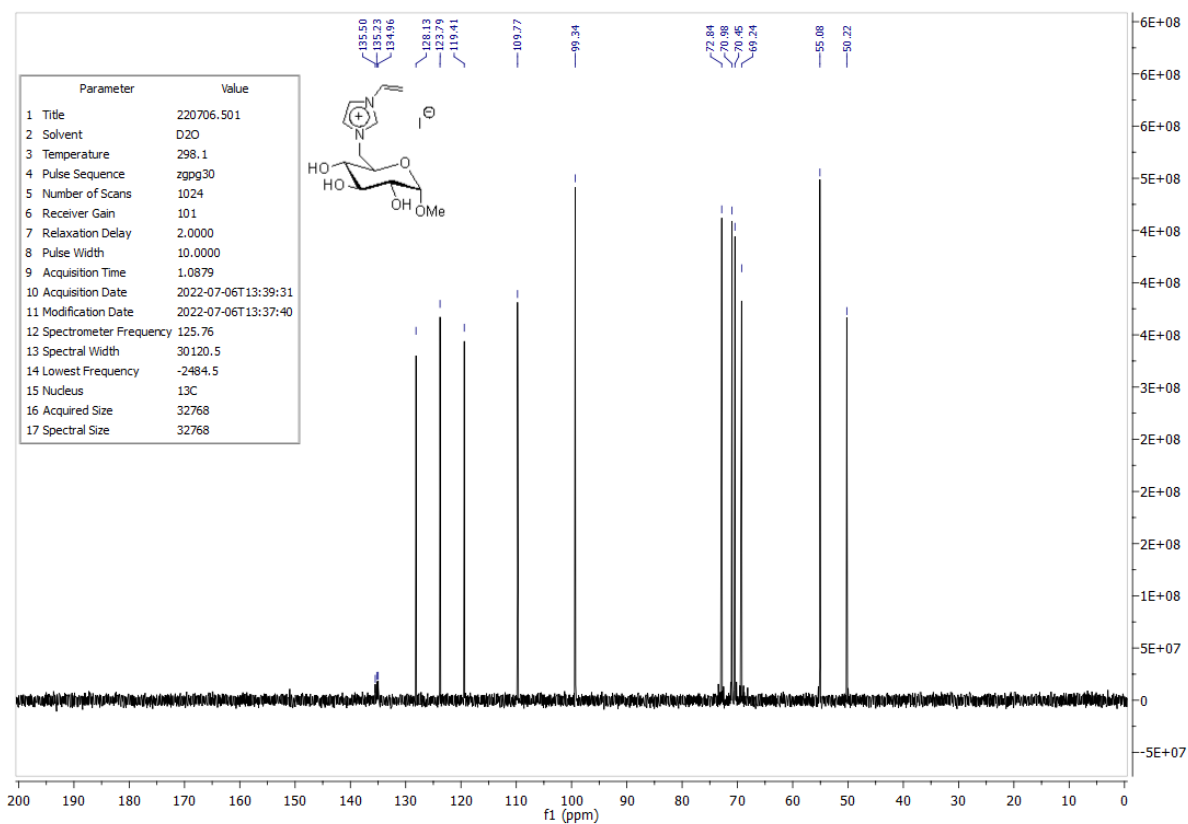


Figure S4. ¹³C-NMR (D₂O) spectrum of compound 2.

1.2 ATR-FTIR Spectra

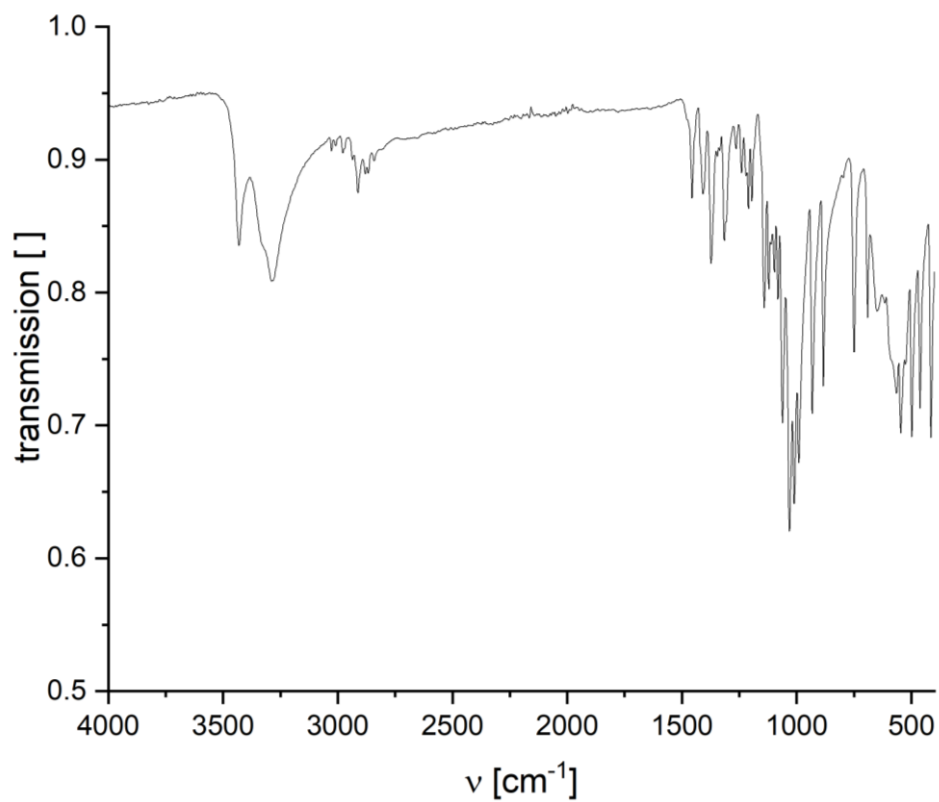


Figure S5. ATR-FTIR spectrum of compound 1.

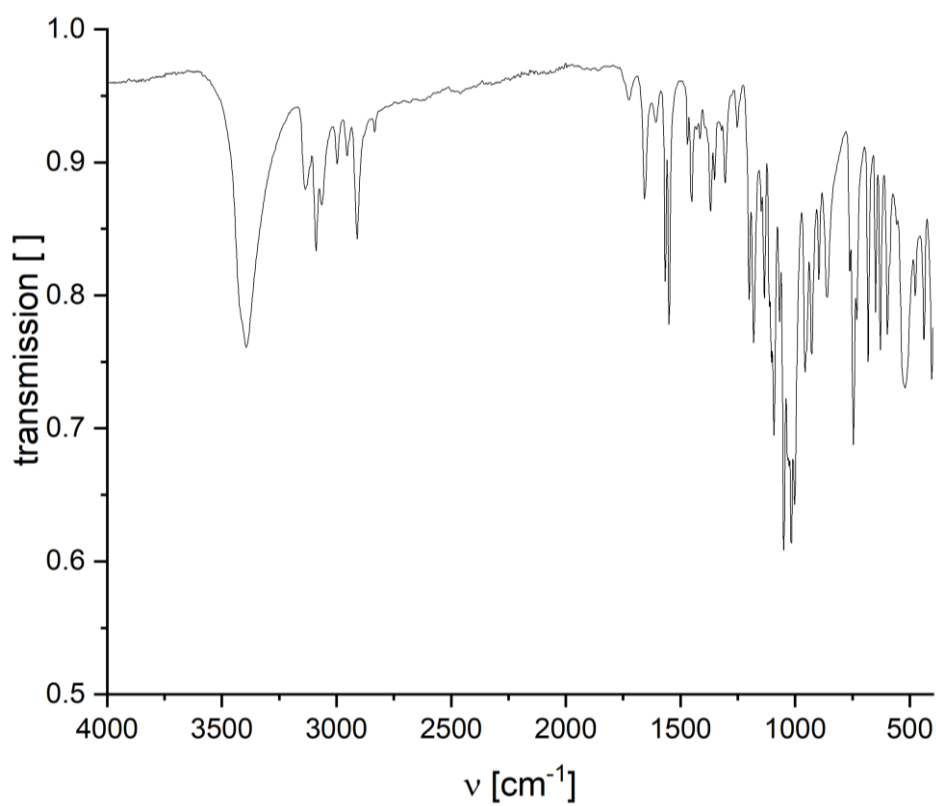


Figure S6. ATR-FTIR spectrum of compound 2.

2. Rheological data

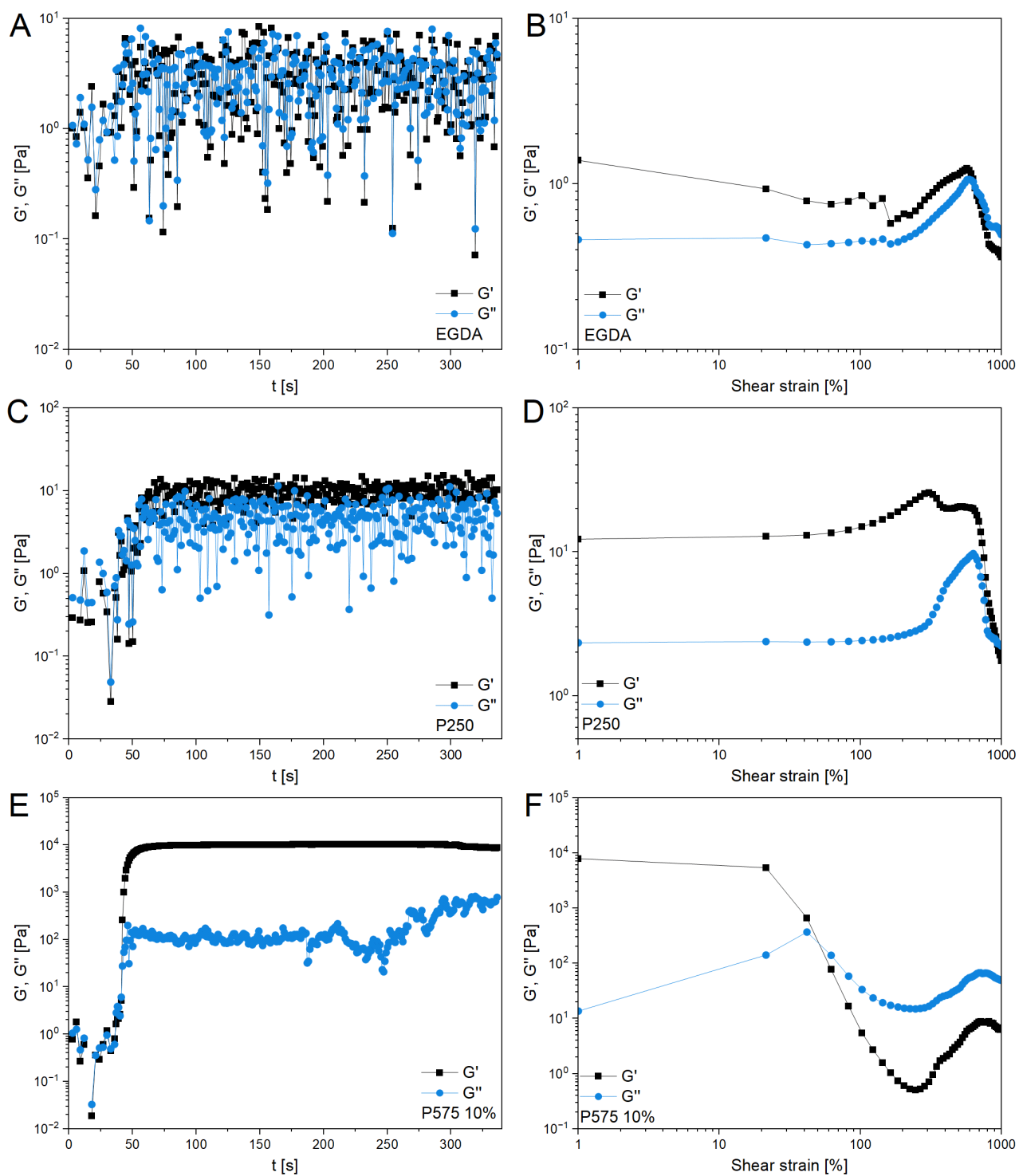


Figure S7. Gelation tracking and amplitude sweep measurements of the samples EGDA (A and B), PEGDA 250 (C and D) and PEGDA 575 10% (E and F) [$C_{\text{GVIM-1}} = 1.25$ mol/L; LAP as photoinitiator, gelation was tracked with a constant strain sweep of 0.1% and at a constant frequency of 1 Hz at 37 °C, the amplitude sweep was measured with varying the oscillation from 0.1 to 1000% at 37 °C].

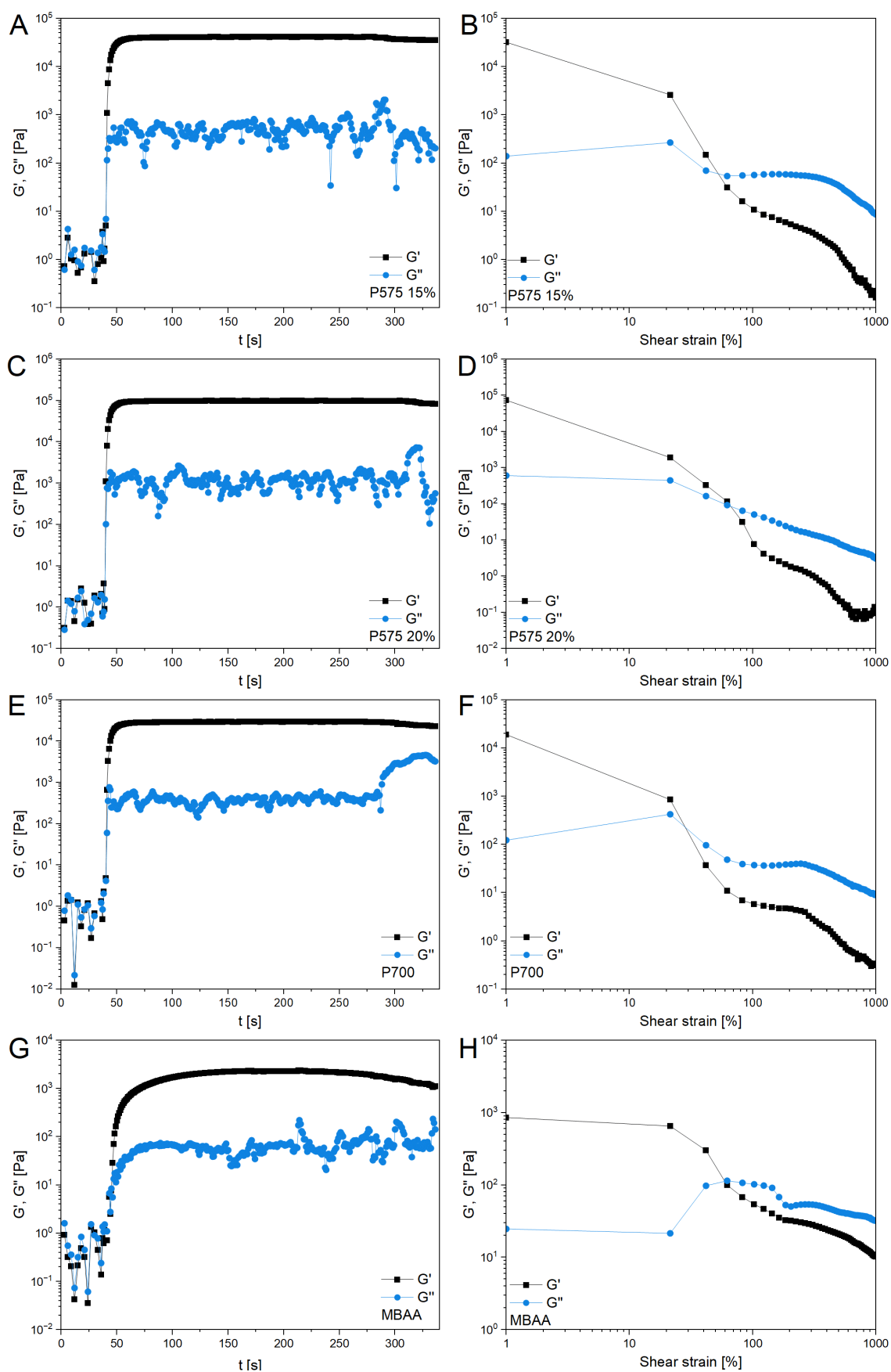


Figure S8. Gelation tracking and amplitude sweep measurements of the samples PEGDA 575 15% (A and B), PEGDA 575 20% (C and D), PEGDA 700 (E and F) and MBAA (G and H) [$C_{\text{GVIIM-I}} = 1.25 \text{ mol/L}$; LAP as photoinitiator, gelation was tracked with a constant strain sweep of 0.1% and at a constant frequency of 1 Hz at 37 °C, the amplitude sweep was measured with varying the oscillation from 0.1 to 1000% at 37 °C].