

Supplementary Information: Influence of the coagulation bath on the nanostructure of cellulose films regenerated from an ionic liquid solution

^a Faculty of Applied Sciences, Delft University of Technology, 2629 JB, Delft, Netherlands.

^b LEPABE - Laboratory for Process Engineering, Environment, Biotechnology and Energy, Faculty of Engineering, University of Porto, Rua Dr. Roberto Frias, 4200-465 Porto, Portugal.

^c ALICE - Associate Laboratory in Chemical Engineering, Faculty of Engineering, University of Porto, Rua Dr. Roberto Frias, 4200-465 Porto, Portugal.

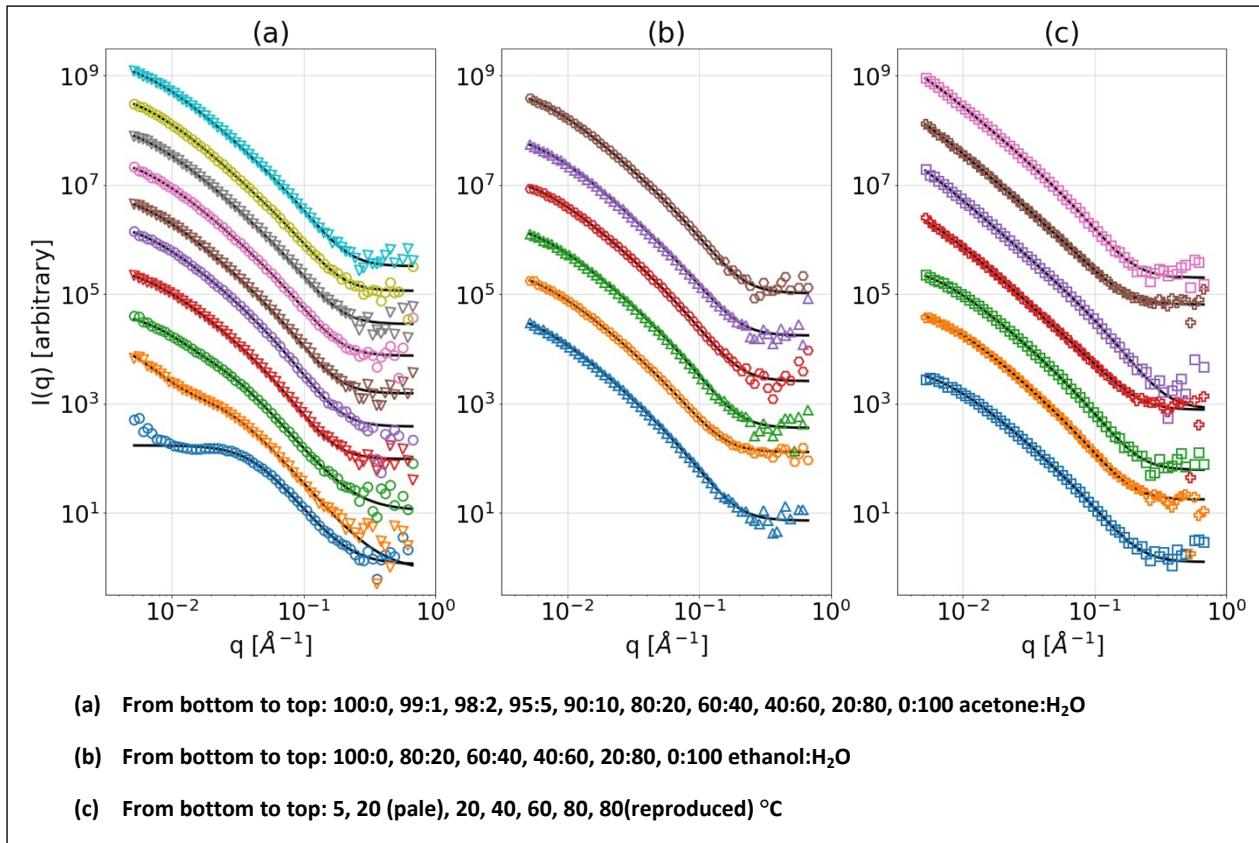
^d ISIS pulsed Neutron and Muon Source, Rutherford Appleton Laboratory, Chilton, Oxfordshire, OX11 0QX, United Kingdom

† Corresponding e-mails: gbernardo@fe.up.pt; S.R.Parnell@tudelft.nl

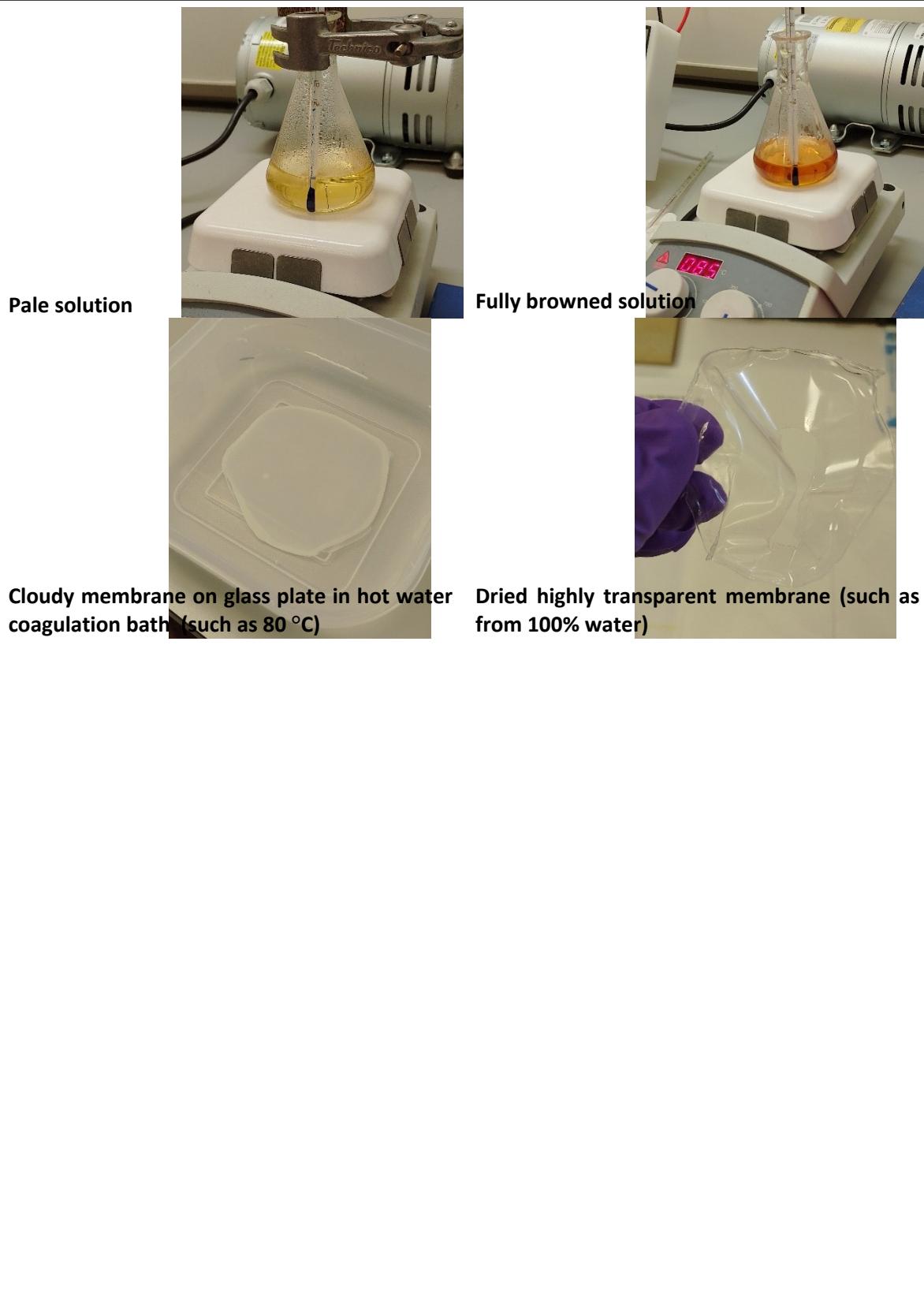
Electronic Supplementary Information (ESI) available: [details of any supplementary information available should be included here]. See DOI: 10.1039/x0xx00000x

Lassi V. Tiihonen,^a Gabriel Bernardo^{*b,c}, Robert Dalgiesh^d, Adélio Mendes^{b,c}, and Steven R. Parnell^{*a}

S1 – FULL DATA AND FIT PLOTS

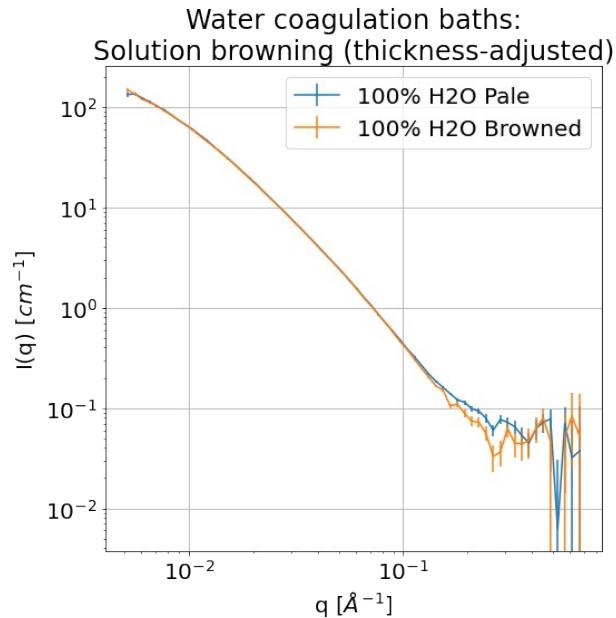


S2 – IMAGES



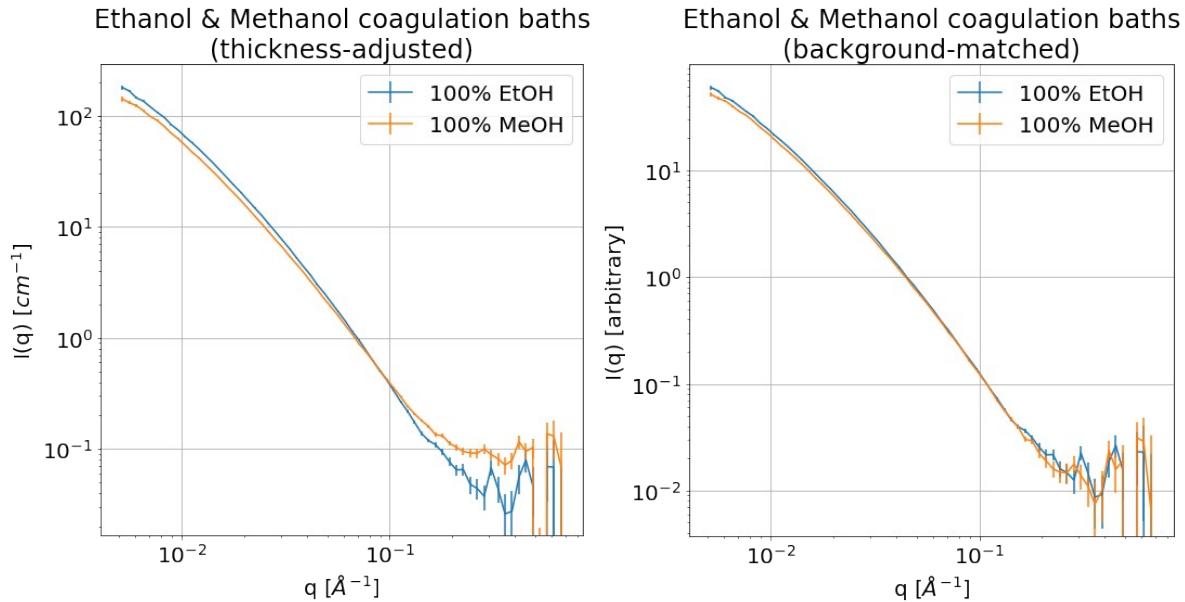
S3 – PALE-BROWNED SOLUTION COMPARISON

A “pale” cellulose solution was used to prepare a water-coagulated sample before the fully browned solution. Adjusting for calliper-measured sample thickness resulted in identical SANS data within uncertainties.



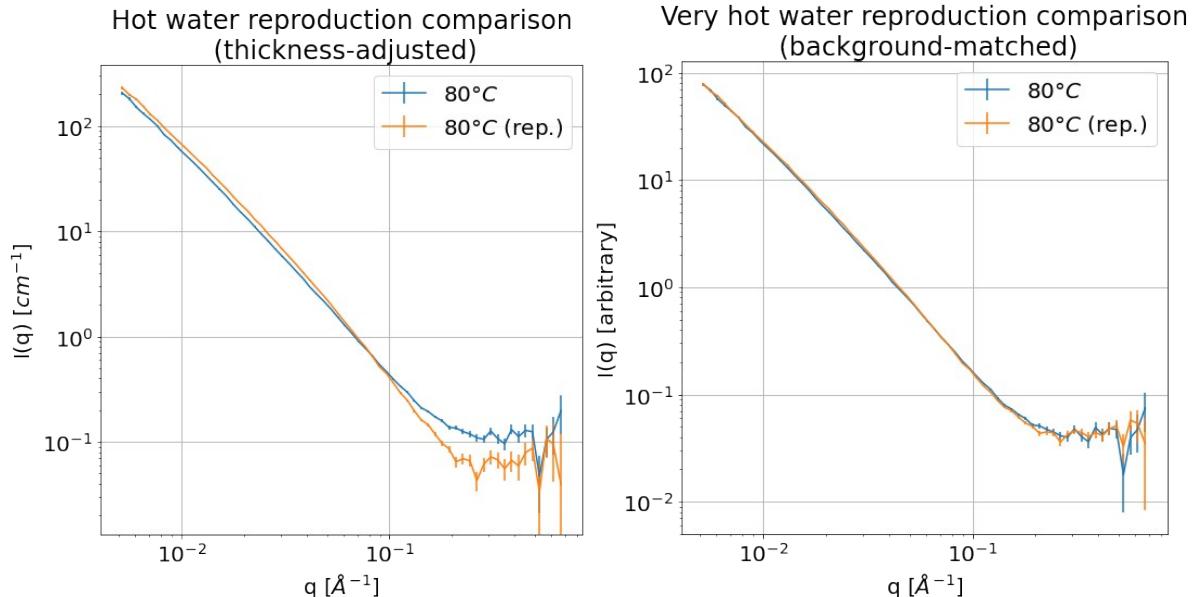
S4 – METHANOL-ETHANOL COMPARISON

Pure methanol and ethanol resulted in highly similar SANS results. Subtraction of the estimated excess high-q background from the methanol signal shows the data are nearly identical.



S5 – REPRODUCTION: HIGH-TEMPERATURE WATER

Reproduction of a high-temperature water sample shows the high reproducibility of SANS results for water coagulation baths.



S6 – MICROBALANCE RESULTS: ACETONE-WATER

Mass fractions as measured by microbalance method for acetone-water samples. The standard deviations were corrected based on the control chart constant c_4 for unbiased uncertainty estimates.
*: for the single-sample cases, the highest adjacent standard deviation was assumed.

vol% H ₂ O	$mean(\phi_{m,cel})$	$N_{samples}$	$STD(\phi_{m,cel})$	$c_4(N)$	$STD_{corr}(\phi)$
0	18.56	9	1.56	0.97	1.61
1	22.07	2	0.81	0.80	1.02
2	20.49	3	0.46	0.89	0.52
5	18.04	4	1.36	0.92	1.48
10	13.37	3	0.68	0.89	0.76
20	13.01	3	0.20	0.89	0.23
30	12.46	1	0.28*	-	0.31*
40	12.25	1	0.28*	-	0.31*
50	12.32	3	0.28	0.89	0.31
100	12.02	2	0.12	0.80	0.15

S7 – MICROBALANCE RESULTS: WATER TEMPERATURE

Mass fractions as measured by microbalance method for water temperature samples. The standard deviations were corrected based on the control chart constant c_4 for unbiased uncertainty estimates.
*: for the single-sample cases, the highest adjacent standard deviation was assumed.

H ₂ O temp	$mean(\phi_{m,cel})$	$N_{samples}$	$STD(\phi_{m,cel})$	$c_4(N)$	$STD_{corr}(\phi)$
5	11.4	1	0.17*	-	0.21*
20	11.3	2	0.17	0.80	0.21
40	9.97	4	0.34	0.92	0.37
60	8.62	3	0.33	0.89	0.37
80	8.55	3	0.19	0.89	0.21

S8 – SASVIEW FITTING SAMPLE LEGEND & THICKNESSES:

Label	Description	Thickness [mm]
Wet01	Pale solution - Room temp water (lab room temp 19.7 C)	0.403
Wet02	Browned solution - Room temp water (lab room temp 19.7 C)	0.310
Wet03	" Ice water (~5 C)	0.267
Wet04	" Hot water (~60 C)	0.350
Wet05	" Very hot water (~80 C)	0.377
Wet06	" Warm water (~40 C)	0.340
Wet07	" Very hot water (~80 C) (reproduced with shorter delay)	0.337
Wet08	100% acetone	0.184
Wet09	80:20% acetone:H ₂ O	0.283
Wet10	60:40% acetone:H ₂ O	0.317
Wet11	40:60% acetone:H ₂ O	0.283
Wet12	20:80% acetone:H ₂ O	0.277

Wet13	100% H ₂ O	0.283
Wet14	90:10% acetone:H ₂ O	0.330
Wet15	95:5% acetone:H ₂ O	0.227
Wet16	98:2% acetone:H ₂ O	0.187
Wet17	100% ethanol	0.333
Wet18	100% methanol	0.363
Wet19	80:20% ethanol:H ₂ O	0.333
Wet20	60:40% ethanol:H ₂ O	0.313
Wet21	40:60% ethanol:H ₂ O	0.300
Wet22	20:80% ethanol:H ₂ O	0.283
Wet23	99:1% acetone:H ₂ O	0.117
Wet24	99.5:0.5% acetone:H ₂ O	0.117

S9 – SASVIEW FITTING OUTPUTS: ACETONE-WATER

PMF fitting outputs (gray = fixed values):

	scale	scale_err	background_d	background_err	xi1	xi1_err	xi2	xi2_err	d1	d2	d2_err	sld_polymer	sld_nonsolvent
Wet02	0.040147	0.001394	0.012366	0.00082	8.8269	0.24773	170.8	4.0327	2	2.0722	0.010147	1.75	6.4
Wet08	0.048441	8.66	0.018946	0.001873	-16.23	1485.7	16.224	1091.4	2	2.3578	5.8942	1.75	6.4
Wet09	0.040347	0.001795	0.014846	0.000719	10.956	0.29993	170.32	4.5817	2	2.0657	0.012695	1.75	6.4
Wet10	0.04496	0.001665	0.018231	0.000773	10.005	0.25376	176.3	4.356	2	2.0645	0.010688	1.75	6.4
Wet11	0.041577	0.001508	0.017327	0.00078	9.6572	0.25366	174.87	4.2367	2	2.0758	0.010485	1.75	6.4
Wet12	0.035553	0.00131	0.017487	0.000987	8.5683	0.2829	175.38	4.4343	2	2.0962	0.010689	1.75	6.4
Wet14	0.060817	0.002934	0.01477	0.000826	12.337	0.31262	171.49	4.7554	2	2.0249	0.013643	1.75	6.4
Wet15	0.059418	0.004135	0.014954	0.000647	14.01	0.42469	169.27	6.3575	2	1.9431	0.019604	1.75	6.4
Wet16	0.26403	0.044644	0.01253	0.000618	20.896	0.98862	544.5	178.01	2	1.4434	0.048539	1.75	6.4
Wet23	0.71495	0.076519	0.001058	0.000876	23.293	0.84381	2.08E+08	1.00E+08	2	1.05	0.027291	1.75	6.4

DL fitting outputs (gray = fixed values):

	scale	background_d	background_d_err	A_scale	A_scale_err	A_cor_length	A_cor_length_err	B_lorentz_scale_1	B_lorentz_scale_1_err	B_lorentz_length_1	B_lorentz_length_1_err	B_lorentz_length_h_1_err	B_lorentz_exp_1	B_lorentz_exp_1_err
Wet 02	1	0.003598	0.001006	6.77	1.80E-05	93.839	1.3212	3.7711	0.19925	36.635	0.9566		2.6224	0.021607
Wet 08	1	0.011362	0.00085	0	0	0	0	1.6933	0.01004	24.279	0.11087		3.0266	0.018871
Wet 09	1	0.006024	0.000911	5.99	1.85E-05	98.588	1.6218	4.2451	0.23575	39.421	1.0038		2.7085	0.020326
Wet 10	1	0.008508	0.000968	6.94	1.85E-05	97.866	1.4143	4.5036	0.22621	38.304	0.91428		2.6703	0.019334
Wet 11	1	0.007414	0.000964	6.51	1.88E-05	9.96E+01	1.5413	4.6404	0.24819	39.65	1.0084		2.6376	0.019064
Wet 12	1	0.00677	0.001167	5.67	2.16E-05	103.88	2.054	5.0557	0.35613	43.306	1.4804		2.5353	0.021177
Wet 14	1	0.005642	0.001072	8.31	2.06E-05	93	1.23	4.562	0.2097	36.007	0.75165		2.8194	0.021037
Wet 15	1	0.007865	0.000877	5.53	1.86E-05	90.631	1.5432	3.4869	0.18565	36.424	0.8566		2.8325	0.022904
Wet 16	1	0.006896	0.00087	2.88	1.30E-05	100.79	2.5148	3.5273	0.16427	37.968	0.83739		2.7698	0.022017
Wet 23	1	0.002269	0.00124	9.15	4.55E-07	174.57	11.959	3.1749	0.11528	37.291	0.80422		2.7387	0.031133

S10 – SASVIEW FITTING OUTPUTS: ETHANOL-WATER

PMF fitting outputs (gray = fixed values):

	scale	scale_err	background	background_err	xi1	xi1_err	xi2	xi2_err	d1	d2	d2_err	sld_polymer	sld_nonsolvent
Wet13	0.041577	0.001508	0.017327	0.00078	9.6572	0.25366	174.87	4.2367	2	2.0758	0.010485	1.75	6.4
Wet17	0.042117	0.001522	0.014374	0.000732	10.549	0.25385	188.36	4.7451	2	2.0923	0.010308	1.75	6.4
Wet19	0.054	0.00228	0.036835	0.000753	11.388	0.27628	182.4	5.0578	2	2.027	0.01207	1.75	6.4
Wet20	0.051413	0.00201	0.014505	0.000737	10.884	0.25861	179.68	4.6359	2	2.0431	0.011219	1.75	6.4
Wet21	0.047649	0.001665	0.014888	0.000768	10.002	0.23925	178.26	4.2204	2	2.0656	0.010089	1.75	6.4
Wet22	0.032548	0.001115	0.014568	0.000823	8.5376	0.2596	169.87	3.8966	2	2.118	0.00995	1.75	6.4

DL fitting outputs:

	background	background_err	A_scale	A_scale_err	A_cor_length	A_cor_length_err	B_lorentz_scale_1	B_lorentz_scale_1_err	B_lorentz_length_1	B_lorentz_length_1_err	B_lorentz_exp_1	B_lorentz_exp_1_err
Wet 02	0.0035979	0.001006	6.77E-05	1.80E-06	93.839	1.3212	3.7711	0.19925	36.635	0.9566	2.6224	0.021607
Wet 17	0.0028949	0.000902	6.25E-05	1.92E-06	111.41	1.9468	6.3686	0.34132	44.622	1.085	2.6518	0.015686
Wet 19	0.02709	0.000971	7.19E-05	1.85E-06	97.252	1.3681	4.5934	0.21478	37.593	0.81823	2.7364	0.019293
Wet 20	0.005283	0.000943	7.56E-05	1.80E-06	95.811	1.2533	4.3705	0.19726	36.666	0.77665	2.738	0.019301
Wet 21	0.0048075	0.00096	7.44E-05	1.84E-06	9.83E+01	1.3291	4.749	0.22255	38.108	0.84912	2.6777	0.018315
Wet 22	0.0036039	0.000952	4.97E-05	2.19E-06	110.82	2.537	6.1786	0.44873	47.907	1.6163	2.5232	0.016708

S11 – SASVIEW FITTING OUTPUTS: WATER TEMPERATURE

PMF fitting outputs (gray = fixed values):

	scale	scale_err	background	background_err	xi1	xi1_err	xi2	xi2_err	d1	d2	d2_err	sld_polymer	sld_nonsolvent
Wet01	0.051643	0.001582	0.024979	0.000843	9.0888	0.21313	168.52	3.4381	2	2.078	0.008943	1.75	6.4
Wet02	0.040147	0.0013938	0.012366	0.00082	8.8269	0.24773	170.8	4.0327	2	2.0722	0.010147	1.75	6.4
Wet03	0.032985	0.0012266	0.012528	0.000889	7.7342	0.27767	153.09	3.5775	2	2.0719	0.011019	1.75	6.4
Wet04	0.034335	0.00071407	0.003347	0.001018	5.9689	0.21176	437.09	28.3	2	2.0748	0.006195	1.75	6.4
Wet05	0.038188	0.00095696	0.038378	0.000931	7.6277	0.21572	425.88	28.823	2	2.056	0.007365	1.75	6.4
Wet06	0.029757	0.00065676	0.022349	0.0011	5.5827	0.23885	268.48	8.5408	2	2.1191	0.006562	1.75	6.4
Wet07	0.043974	0.0012562	0.016988	0.000795	9.3094	0.21649	382.85	22.809	2	2.0362	0.008306	1.75	6.4

DL fitting outputs:

	Chi2	background	background_err	A_scale	A_scale_err	A_cor_length	A_cor_length_err	B_lorentz_scale_1	B_lorentz_scale_1_err	B_lorentz_length_1	B_lorentz_length_1_err	B_lorentz_exp_1	B_lorentz_exp_1_err
Wet 01	2.5274	0.0150	0.001044	9.37	1.93E-05	91.341	1.0169	4.3947	0.18754	34.727	0.72599	2.6812	0.019192
Wet 02	2.6275	0.0035979	0.001006	6.77	1.80E-05	93.839	1.3212	3.7711	0.19925	36.635	0.9566	2.6224	0.021607
Wet 03	2.1737	0.0061326	0.001065	6.11	1.71E-05	84.776	1.1919	2.4986	0.14745	32.974	1.0064	2.6009	0.028561
Wet 04	3.5694	-0.0082309	0.001051	5.05	1.36E-05	139.01	2.6754	6.7488	0.43935	52.412	1.8467	2.317	0.016208
Wet 05	3.6776	0.025104	0.001041	4.73	1.39E-05	1.47E+02	3.2459	7.9435	0.49822	54.574	1.7688	2.3872	0.015138
Wet 06	3.1287	0.0071824	0.000864	1.31	1.14E-05	409.85	192.07	54.796	12.573	141.62	14.873	2.2326	0.0066
Wet 07	4.4302	0.0034785	0.000945	4.94	1.34E-05	144.05	2.9642	7.9746	0.42262	51.989	1.3716	2.4862	0.014182