

**Table T1**

synthesized		
$q$ ( $\text{\AA}^{-1}$ )	I(q)	$d$ ( $\text{\AA}$ )
0.49	461	12.81
0.59	1153	10.64
0.62	3225	10.13
0.64	4635	9.81
0.88	320	7.14
0.98	966	6.41
1.01	593	6.22
1.06	354	5.92
1.18	2709	5.32
1.22	2620	5.15
1.24	2296	5.06
1.26	1578	4.98
1.28	1594	4.91
1.36	1310	4.62
1.48	1780	4.24
1.55	1200	4.05
1.63	1870	3.85
1.75	1229	3.59
1.82	507	3.45
1.89	566	3.32
1.95	529	3.22
2.02	513	3.11
2.11	514	2.98
2.23	375	2.82
2.27	375	2.77
2.33	385	2.70
2.46	394	2.55
2.49	378	2.52
2.62	488	2.40
2.68	384	2.34

2.75	334	2.28
2.82	301	2.23
2.91	354	2.16
2.96	319	2.12
3.07	329	2.05
3.13	446	2.01
3.16	414	1.99
3.32	273	1.89
3.39	281	1.85
3.48	268	1.80
3.62	233	1.73

**Table T1:** list in sequence of wavevector, q, and position, d, of all the observed lines, together with their intensities, I(q), of the powder diffraction data of the synthesized sample.

## Table T2

AcN 2%		
$q$ ( $\text{\AA}^{-1}$ )	I(q)	$d$ ( $\text{\AA}$ )
0.53	509	11.85
0.59	225	10.64
0.61	558	10.30
0.64	824	9.81
0.74	130	8.49
0.77	160	8.16
0.79	126	7.95
0.90	274	6.98
0.92	307	6.83
0.98	321	6.41
1.13	440	5.56
1.16	530	5.41
1.18	557	5.32
1.22	634	5.15
1.24	733	5.06
1.26	616	4.98
1.28	504	4.91
1.35	622	4.65

1.37	548	4.58
1.48	619	4.24
1.56	579	4.03
1.59	441	3.95
1.63	646	3.85
1.74	465	3.61
1.83	350	3.43
1.91	304	3.29
1.98	257	3.17
2.04	225	3.08
2.10	215	2.99
2.20	185	2.85
2.33	176	2.70
2.45	155	2.56
2.62	163	2.40
2.68	149	2.34
2.75	153	2.28
2.90	156	2.17
3.04	171	2.07
3.12	183	2.01
3.22	146	1.95
3.32	132	1.89
3.38	124	1.86

**Table T2:** list in sequence of wavevector, q, and position, d, of all the observed lines, together with their intensities, I(q), of the powder diffraction data of the sample prepared in AcN (2%)-H<sub>2</sub>O (98%) mixture.

**Table T3**

AcN 4%		
$q$ ( $\text{\AA}^{-1}$ )	I(q)	$d$ ( $\text{\AA}$ )
0.53	1785	11.85
0.59	1386	10.64
0.61	2971	10.30
0.64	4187	9.81
0.7	756	8.97

0.74	799	8.49
0.77	871	8.16
0.79	789	7.95
0.86	866	7.30
0.9	1118	6.98
0.92	1191	6.83
0.98	1534	6.41
1.18	3078	5.32
1.21	3156	5.19
1.24	3003	5.06
1.28	2225	4.91
1.33	1598	4.72
1.35	2119	4.65
1.37	1797	4.58
1.48	2196	4.24
1.54	1652	4.08
1.56	1848	4.03
1.58	1342	3.97
1.63	2297	3.85
1.74	1644	3.61
1.82	1130	3.45
1.89	1022	3.32
1.91	1047	3.29
2.03	858	3.09
2.11	817	2.98
2.2	698	2.85
2.27	652	2.77
2.33	709	2.70
2.45	643	2.56
2.49	599	2.52
2.62	698	2.40
2.68	614	2.34
2.75	606	2.28
2.83	549	2.22
2.91	637	2.16
2.97	629	2.11

3.05	653	2.06
3.13	702	2.01
3.22	563	1.95
3.32	505	1.89
3.38	487	1.86
3.48	468	1.80
3.62	404	1.73

**Table T3:** list in sequence of wavevector, q, and position, d, of all the observed lines, together with their intensities, I(q), of the powder diffraction data of the sample prepared in AcN (4%)-H<sub>2</sub>O (96%) mixture.

**Table T4**

AcN 6%		
$q$ ( $\text{\AA}^{-1}$ )	I(q)	$d$ ( $\text{\AA}$ )
0.53	630	11.85
0.59	296	10.64
0.61	879	10.30
0.64	1188	9.81
0.7	85	8.97
0.74	121	8.49
0.77	154	8.16
0.79	109	7.95
0.86	117	7.30
0.9	271	6.98
0.92	314	6.82
0.98	355	6.41
1.16	618	5.41
1.17	578	5.37
1.18	735	5.32
1.21	773	5.19
1.24	865	5.06
1.26	663	4.98
1.28	539	4.91
1.35	630	4.65
1.37	526	4.58

1.48	655	4.24
1.55	556	4.05
1.58	399	3.97
1.63	707	3.85
1.74	469	3.61
1.82	318	3.45
1.9	286	3.31
1.96	223	3.20
1.98	238	3.17
2.04	214	3.08
2.11	213	2.98
2.2	184	2.85
2.27	158	2.77
2.33	185	2.70
2.45	161	2.56
2.49	141	2.52
2.62	174	2.40
2.75	147	2.28
2.83	114	2.22
2.9	142	2.17
2.97	121	2.11
3.05	144	2.06
3.12	167	2.01
3.15	154	1.99
3.22	128	1.95
3.32	120	1.89
3.38	116	1.86
3.48	116	1.80
3.62	104	1.73

**Table T4:** list in sequence of wavevector,  $q$ , and position,  $d$ , of all the observed lines, together with their intensities,  $I(q)$ , of the powder diffraction data of the sample prepared in AcN (6%)-H<sub>2</sub>O (94%) mixture.

**Table T5**

AcN 8%
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$q$ ( $\text{\AA}^{-1}$ )	I(q)	$d$ ( $\text{\AA}$ )
0.53	425	11.85
0.59	377	10.64
0.61	1037	10.30
0.64	1557	9.81
0.74	96	8.49
0.77	127	8.16
0.79	98	7.95
0.90	227	6.98
0.92	269	6.83
0.98	446	6.41
1.16	716	5.41
1.18	1013	5.32
1.21	1028	5.19
1.24	986	5.06
1.26	798	4.98
1.28	671	4.91
1.33	481	4.72
1.35	705	4.65
1.37	573	4.58
1.48	773	4.24
1.54	537	4.08
1.56	624	4.03
1.58	400	3.97
1.63	829	3.85
1.74	553	3.61
1.82	316	3.45
1.89	304	3.32
1.91	303	3.29
1.94	254	3.24
1.96	259	3.20
1.98	257	3.17
2.04	239	3.08
2.12	239	2.96
2.2	185	2.85
2.27	167	2.77

2.33	191	2.70
2.45	183	2.56
2.49	163	2.52
2.62	214	2.40
2.68	175	2.34
2.75	170	2.28
2.83	141	2.22
2.9	176	2.17
2.97	182	2.11
3.05	178	2.06
3.12	213	2.01
3.22	155	1.95
3.32	137	1.89
3.38	134	1.86
3.48	131	1.80
3.61	115	1.74

**Table T5:** list in sequence of wavevector, q, and position, d, of all the observed lines, together with their intensities, I(q), of the powder diffraction data of the sample prepared in AcN (8%)-H<sub>2</sub>O (92%) mixture.

**Table T6**

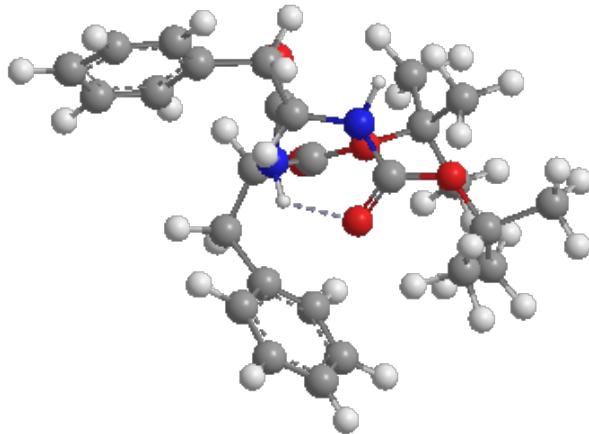
AcN 10%		
$q$ ( $\text{\AA}^{-1}$ )	I(q)	$d$ ( $\text{\AA}$ )
0.48	145	13.08
0.50	195	12.56
0.52	214	12.08
0.57	228	11.02
0.59	293	10.64
0.61	764	10.30
0.64	1080	9.81
0.74	126	8.49
0.77	137	8.16
0.79	132	7.95
0.84	153	7.48
0.90	190	6.98

0.92	198	6.83
0.98	352	6.41
1.00	274	6.28
1.05	212	5.98
1.07	214	5.87
1.14	409	5.51
1.18	727	5.32
1.22	747	5.15
1.24	709	5.06
1.26	584	4.98
1.28	599	4.91
1.33	436	4.72
1.35	561	4.65
1.37	514	4.58
1.48	651	4.24
1.54	511	4.08
1.55	538	4.05
1.63	658	3.85
1.64	620	3.83
1.74	491	3.61
1.82	319	3.45
1.89	310	3.32
1.91	294	3.29
1.94	281	3.24
1.98	253	3.17
2.03	233	3.09
2.12	219	2.96
2.20	176	2.85
2.27	165	2.77
2.33	167	2.70
2.45	155	2.56
2.49	149	2.52
2.54	139	2.47
2.62	179	2.40
2.68	154	2.34
2.75	152	2.28

2.83	145	2.22
2.91	164	2.16
2.97	165	2.11
3.06	172	2.05
3.12	190	2.01
3.23	150	1.94
3.32	136	1.89
3.38	131	1.86
3.48	124	1.80
3.62	112	1.73

**Table T6:** list in sequence of wavevector, q, and position, d, of all the observed lines, together with their intensities, I(q), of the powder diffraction data of the sample prepared in AcN (10%)-H<sub>2</sub>O (90%) mixture.

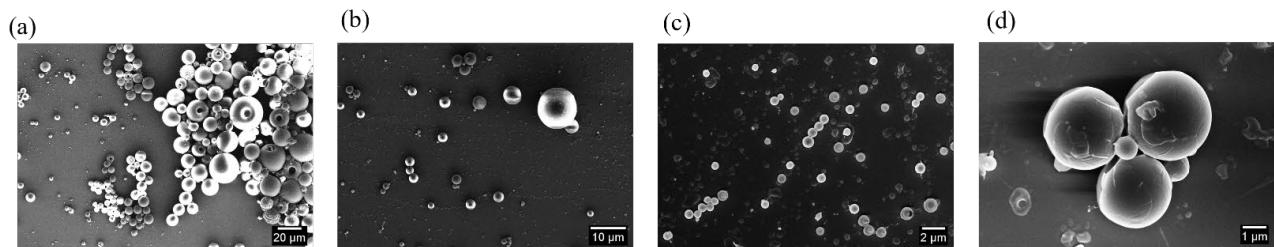
## Figure S1



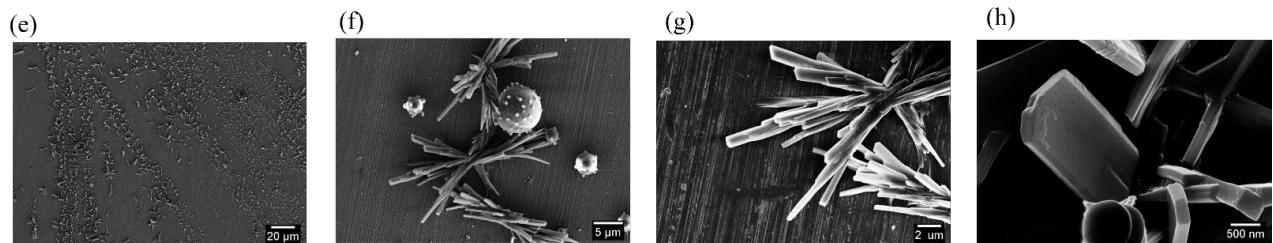
**Figure S1:** Ball-and stick model of Boc-FF molecule.

## Figure S2

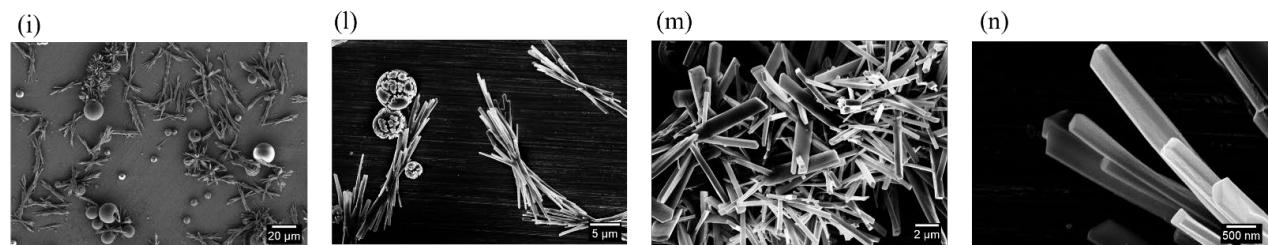
**AcN(2%)-H<sub>2</sub>O(98%)**



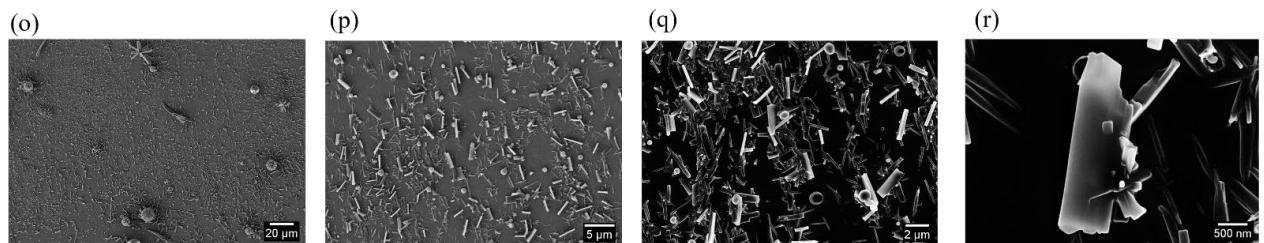
**AcN(4%)-H<sub>2</sub>O(96%)**



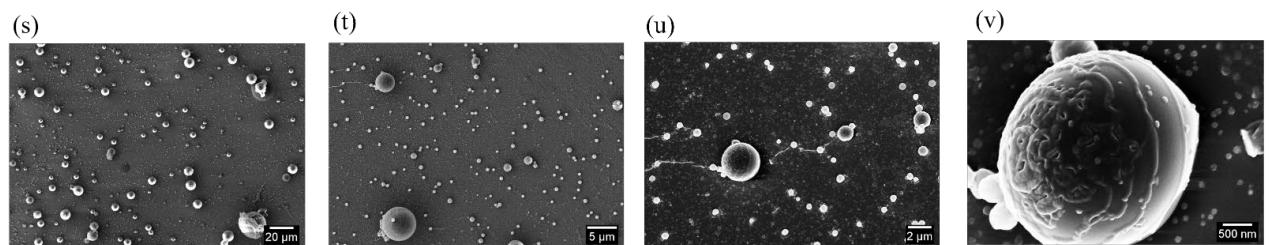
**AcN(6%)-H<sub>2</sub>O(94%)**



**AcN(8%)-H<sub>2</sub>O(92%)**



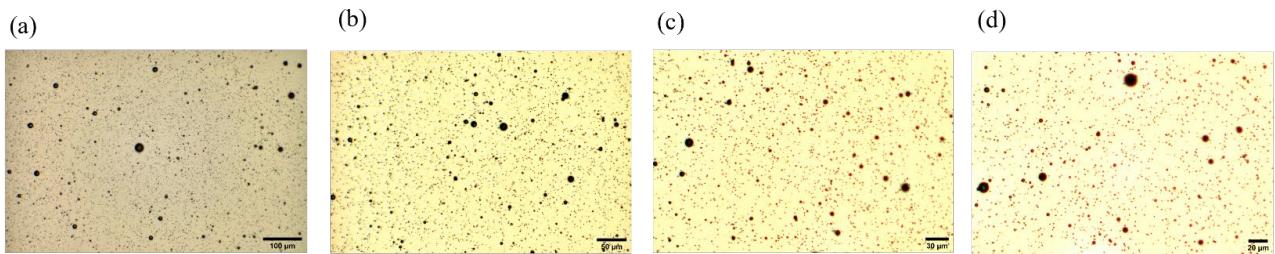
**AcN(10%)-H<sub>2</sub>O(90%)**



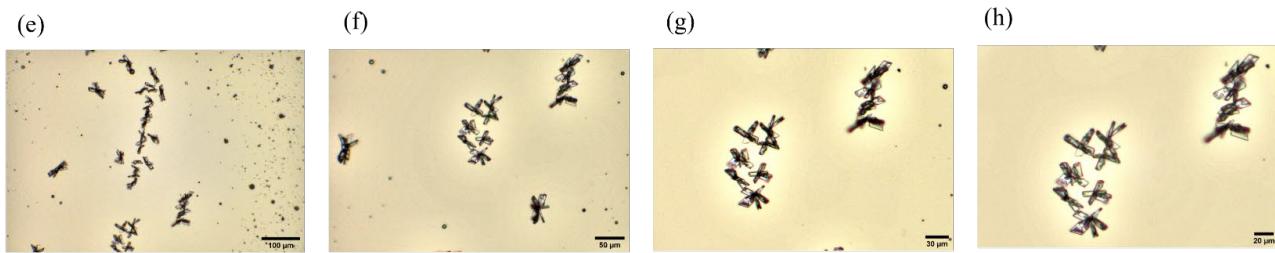
**Figure S2:** SEM images of Boc-FF 1.1mM aggregates formed from the colloidal suspensions at 25°C in the different solvent mixtures at different magnifications (1k, 5k, 10k and 50k).

## Figure S3

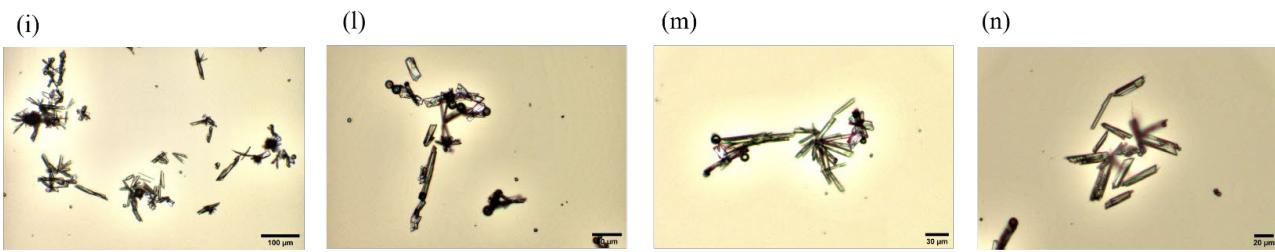
**AcN(2%)-H<sub>2</sub>O(98%)**



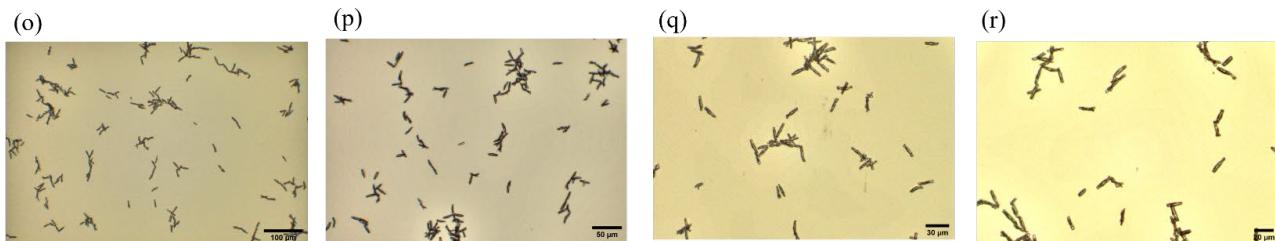
**AcN(4%)-H<sub>2</sub>O(96%)**



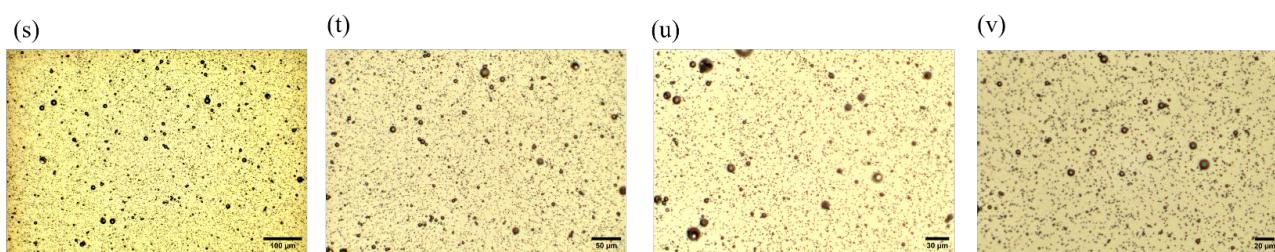
**AcN(6%)-H<sub>2</sub>O(94%)**



**AcN(8%)-H<sub>2</sub>O(92%)**

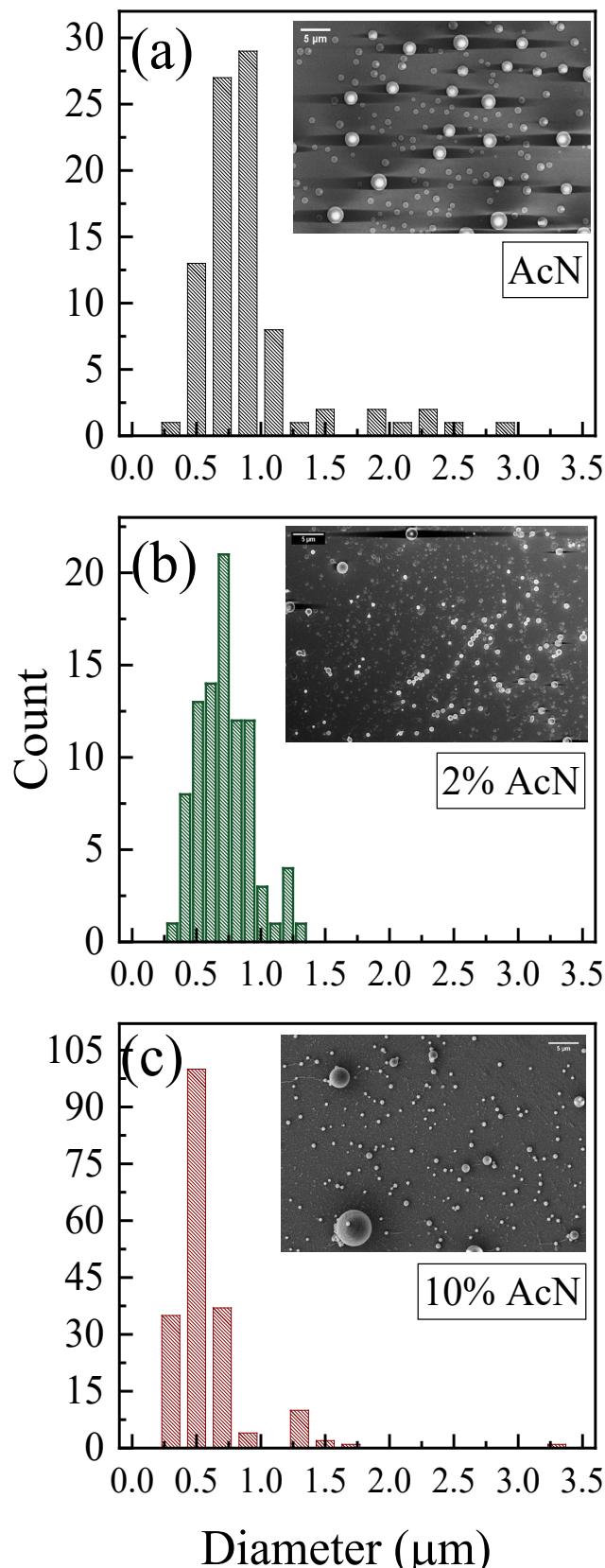


**AcN(10%)-H<sub>2</sub>O(90%)**



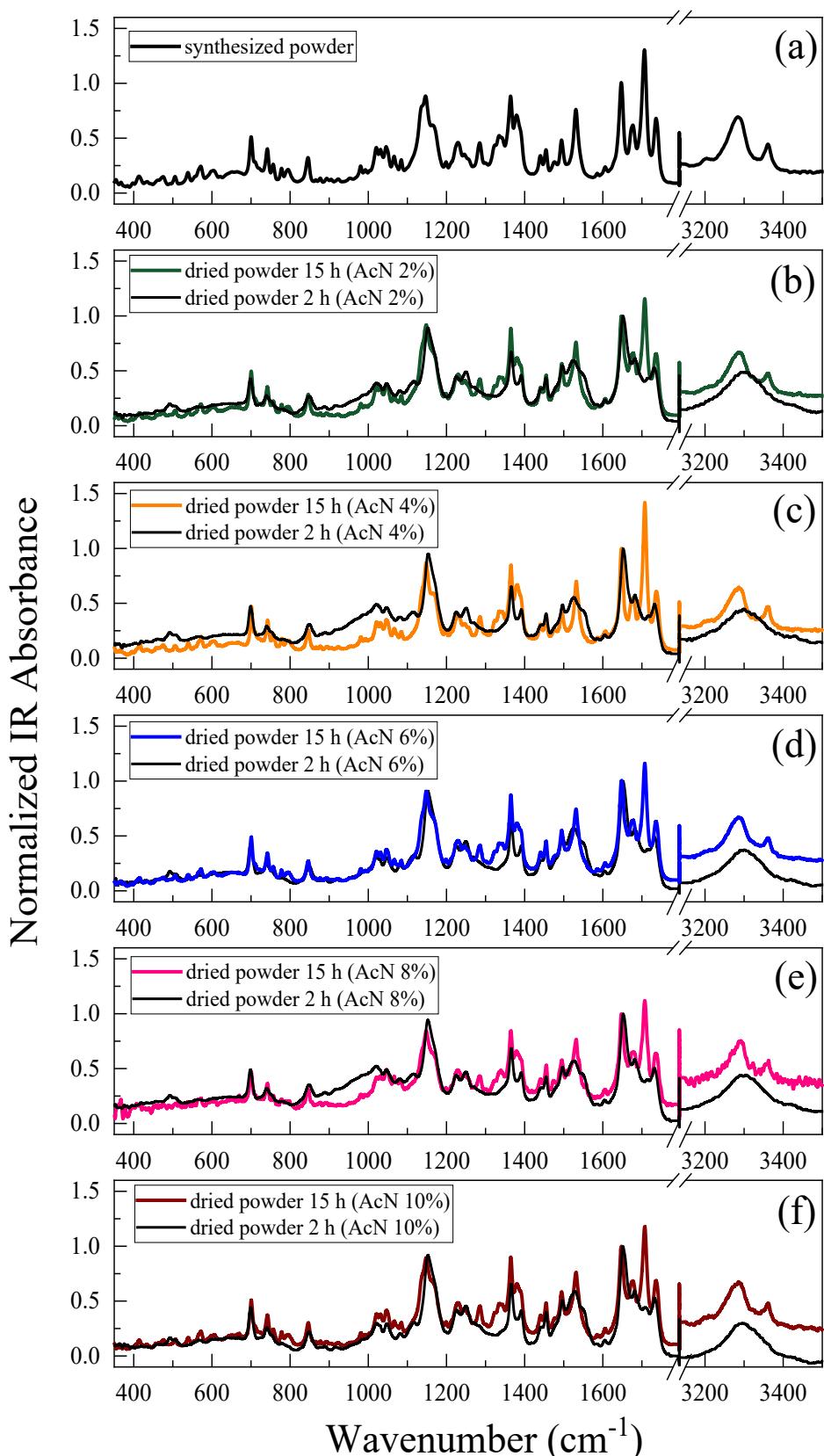
**Figure S3:** OM images of Boc-FF 1.1mM aggregates formed from the colloidal suspensions at 25°C in the different solvent mixtures at different magnifications (400, 600, 800 and 1k).

**Figure S4**



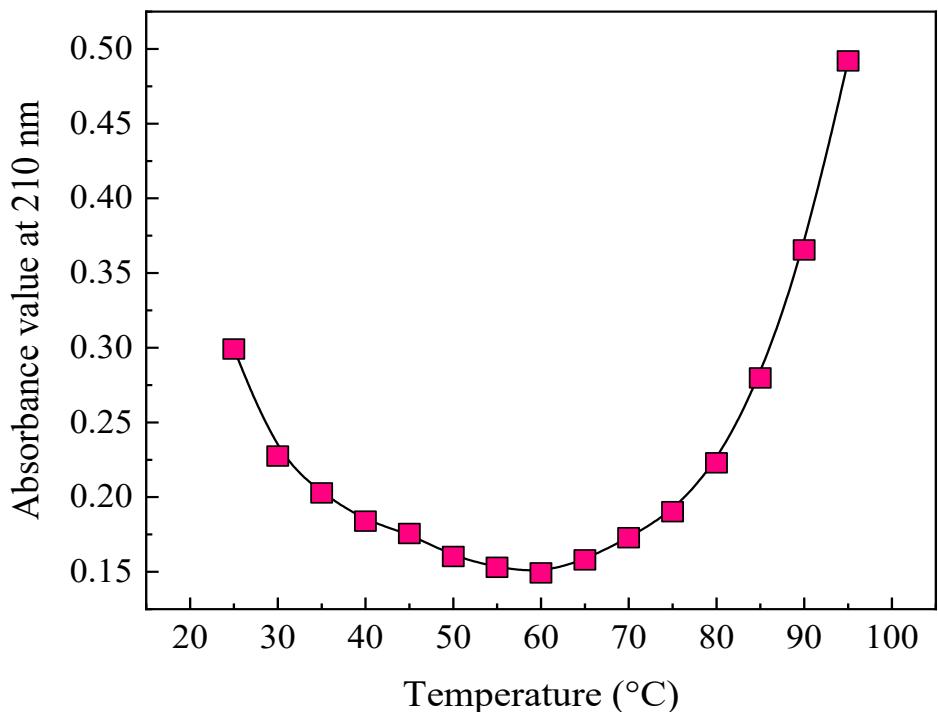
**Figure S4:** dimensional distribution of Boc-FF dry powders of the AcN solution (a) and of the colloidal suspensions in 2% (b) and 10% (c) AcN mixtures. Inserts show the SEM image acquired using 5 k magnification, that have been used to calculate the dimensional distributions.

**Figure S5**



**Figure S5:** FTIR spectra of Boc-FF synthesized powder (a) and powders formed from the colloidal suspensions (b-f) that are dried for 15 h (coloured lines) and 2 h (black lines), recorded in ATR configuration.

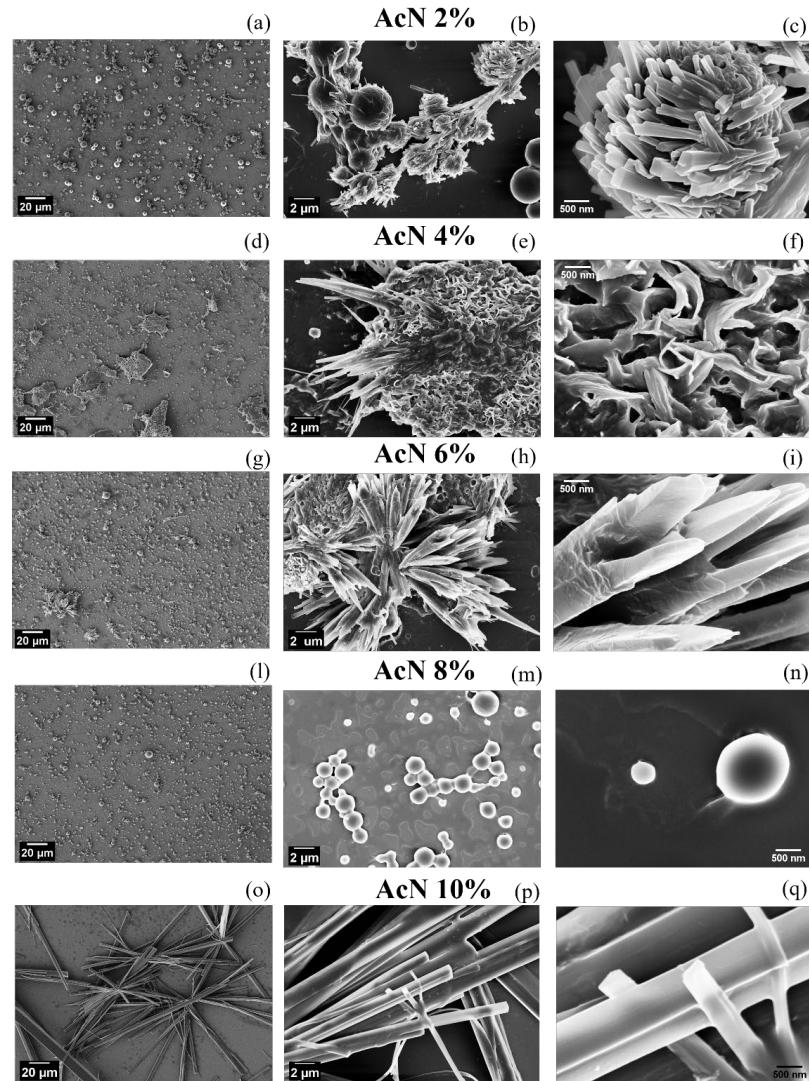
**Figure S6**



**Figure S6:** UV-Vis absorption values at 210 nm, of 4% AcN Boc-FF colloidal suspension

reported as a function of temperature

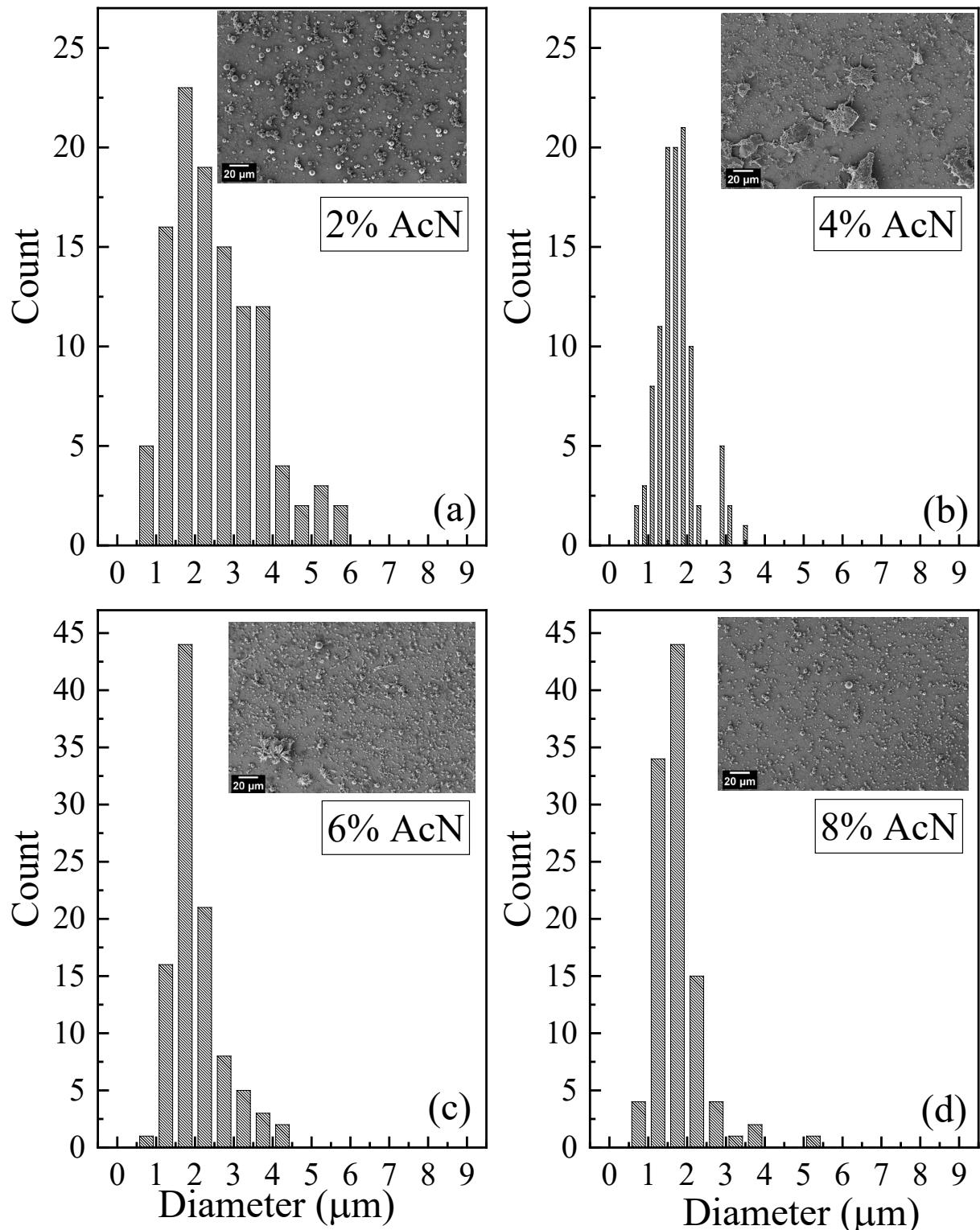
## Figure S7



**Figure S7:** SEM images of Boc-FF 1.1mM aggregates formed at 85°C in the different solvent mixtures.

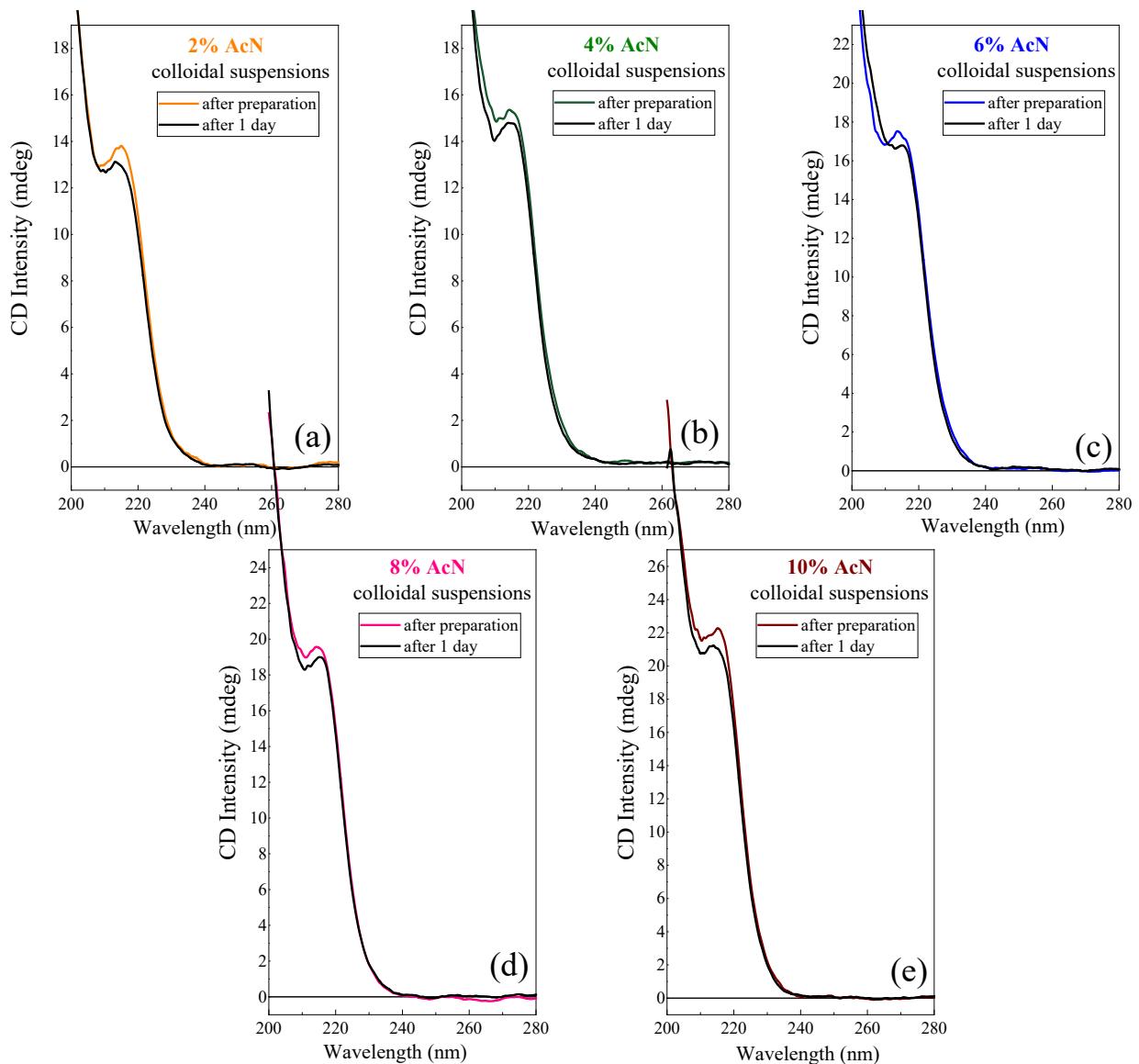
Images are acquired with 1 k magnification (a, d, g, l, o) the scale bar is 20  $\mu\text{m}$ , with 10 k magnification (b, e, h, m, p) the scale bar is 20  $\mu\text{m}$  and with 50 k magnification (c, f, i, n, q) the scale bar is 500 nm.

**Figure S8**



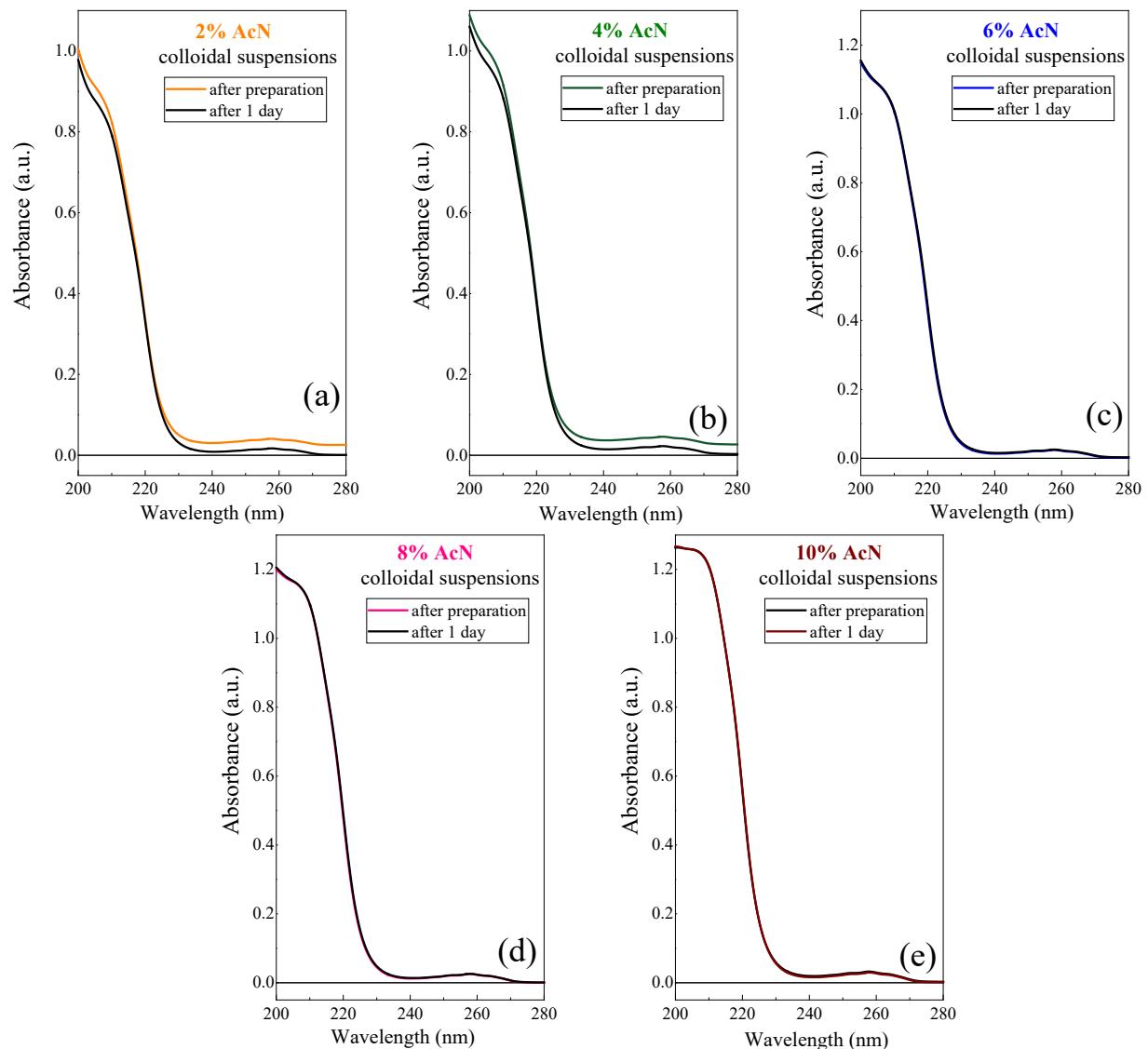
**Figure S8:** dimensional distribution of Boc-FF aggregates in 2% (a), 4% (b), 6% (c) and 8% (d) AcN mixtures. Inserts show the SEM image acquired using 1 k magnification, that have been used to calculate the dimensional distributions.

## Figure S9



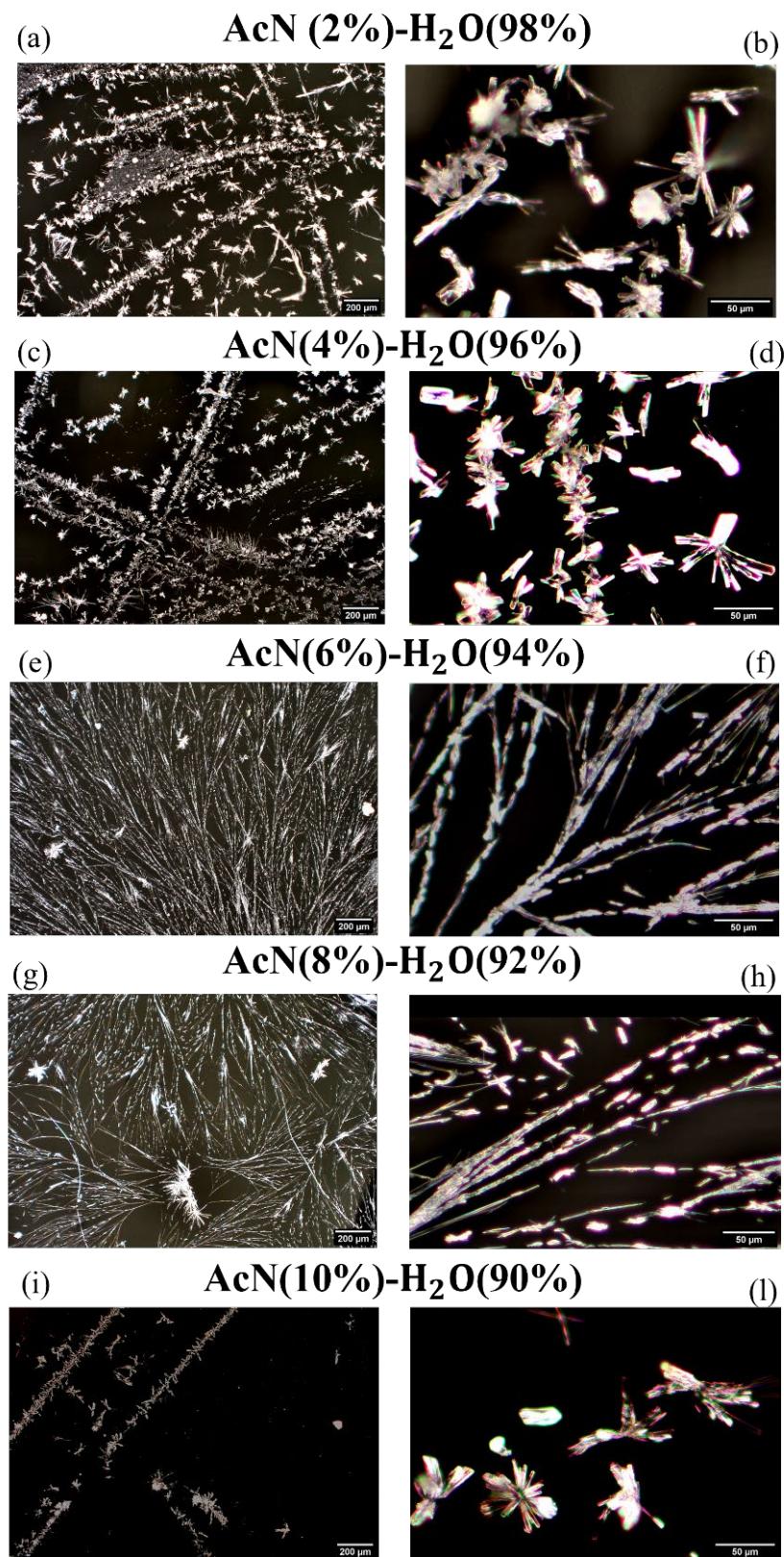
**Figure S9:** comparison of the CD spectra recorded at 25°C, of the colloidal suspensions prepared directly at 85°C immediately after the cooling down (coloured curves) and the day after (black curves).

## Figure S10



**Figure S10:** comparison of the UV-Vis spectra recorded at 25°C, of the colloidal suspensions prepared directly at 85°C immediately after the cooling down (coloured curves) and the day after (black curves).

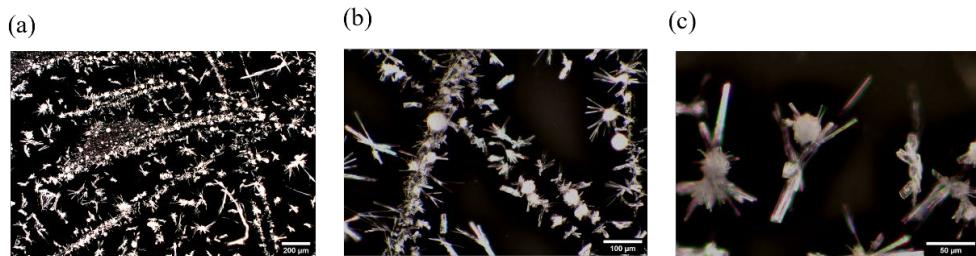
## Figure S11



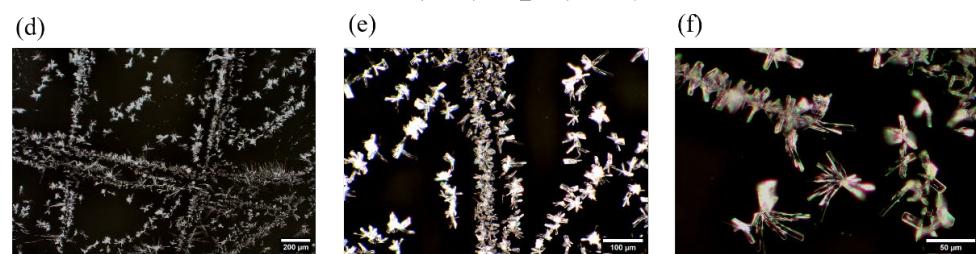
**Figure S11:** OM images of Boc-FF 1.1mM aggregates formed from the colloidal suspensions at 25°C in the different solvent mixtures, after a long evaporation time. Microscopy images are acquired with 140 magnification (a, c, e, g, i) the scale bar is 200  $\mu\text{m}$  and with 1000 magnification (b, d, f, h, l) the scale bar is 50  $\mu\text{m}$

## Figure S12

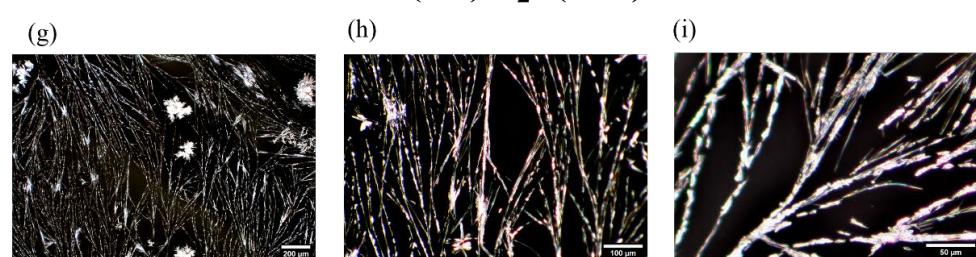
**AcN(2%)-H<sub>2</sub>O(98%)**



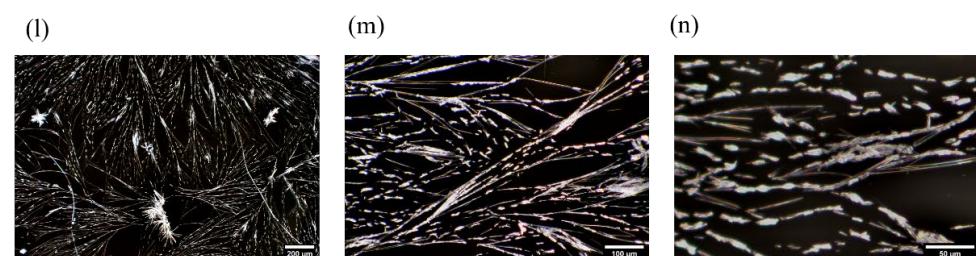
**AcN(4%)-H<sub>2</sub>O(96%)**



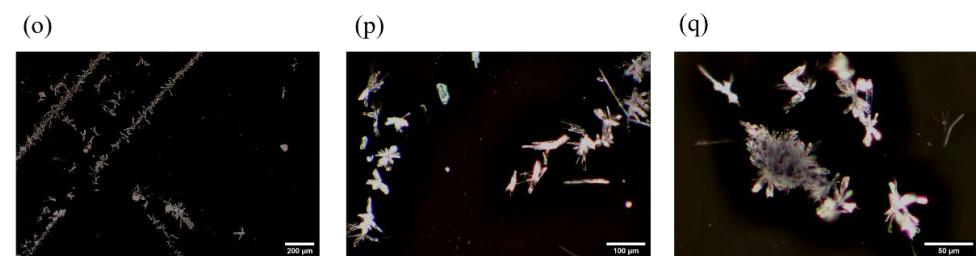
**AcN(6%)-H<sub>2</sub>O(94%)**



**AcN(8%)-H<sub>2</sub>O(92%)**



**AcN(10%)-H<sub>2</sub>O(90%)**



**Figure S12:** OM images of Boc-FF 1.1mM aggregates formed from the colloidal suspensions at 25°C in the different solvent mixtures, after a long evaporation time. Microscopy images are acquired with 140, 800 and 1k magnification.