## **Electronic Supplementary Information**

## **Electrochemical Synthesis of Polymer Microgels**

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Figure S1. IR spectrum of the purified PNIPAM microgels.

Sample ID Operator ID Elapsed Time Mean Diam. Rel. Var. Skew RmsError	1.8V- yst 00:06 160.1 0.000 0.100 1.725	-NIPAM- 3:50 I (nm) ) 51e-02	5			50 0 50.0		Diame	500.0					
								Diame	ter (mn)					
d	G(d)	C(d)	d	G(d)	C(d)	d	G(d)	C(d)						
144.55	0	0	163.39	39	98	184.68	0	100						
146.17	0	0	165.22	6	100	186.75	0	100						
147.80	0	0	167.07	0	100	188.84	0	100						
149.46	0	0	168.94	0	100	190.96	0	100						
151.13	0	0	170.83	0	100	193.10	0	100						
152.83	0	0	172.74	0	100	195.26	0	100	Print Window					
154.54	0	0	174.68	0	100	197.45	0	100						
156.27	30	9	176.64	0	100	199.66	0	100	Copy For Spreadsheet					
158.02	67	30	178.61	0	100	201.89	0	100						
159.79	100	62	180.61	0	100	204.15	0	100	Copy to Clipboard					
161.58	76	86	182.64	0	100	206.44	0	100	Close					

Figure S2. A screenshot showing DLS size distribution of PNIPAM microgels.



**Figure S3.** Temperature-dependent  $< D_h >$  of PNIPAM microgels.



**Figure S4.** Typical cyclic voltammograms of (a) NIPAM ( $1.0 \times 10^{-3}$  mol) and (b) MBAAm ( $5.0 \times 10^{-5}$  mol) in pure CH<sub>3</sub>CN (20.0 mL).

Sample ID Operator ID Elapsed Time Mean Diam. Rel. Var. Skew RmsError	2.3V- wu 00:10 615.0 0.000 0.007 1.227	-AAm-1 ):03 ) (nm) ) 7 76e-03				100 50 0 50.0	5000.0 Diameter (nm)					
	0(4)	0(4)		0(4)	0(4)		0(4)	0(4)				
- ° —	_G( <u>a</u> )	(0)		G( <u>a</u> )	(a)		(d)	(a)				
595.48	0	0	610.74	17	4	622.58	0	100				
596.73	0	0	611.81	36	11	624.75	0	100				
597.97	0	0	612.88	64	23	626.06	0	100				
599.22	0	0	613.95	89	40	627.37	0	100				
600 47	0	0	615.02	100	59	628.68	0	100				
601 73	0	ō	616.09	90	76	629.99	ō	100	Print Window			
602.98	ō	ō	617 17	64	89	631.31	ō	100				
604 24	0	ŏ	618 25	37	96	632.62	ő	100	Copy For Spreadsheet			
605.50	ň	ň	619.33	17	00	633.05	ň	100				
606.77	ŏ	ŏ	620.41	6	100	635.27	ő	100	Copy to Clipboard			
609.67	ĕ	1	621.49	ő	100	636.60	ő	100				
000.07	Ŭ.,		021.10			000.00			Close			

Figure S5. A screenshot showing DLS size distribution of PAAm microgels.



Figure S6. IR spectrum of the purified PAAm microgels.



**Figure S7.** TEM images of PAAm microgels synthesized at a potentiostat of (a) +2.5 V and (b) + 2.6 V *versus* Ag/AgCl, respectively. All microgels were harvested after 3hs' reaction.µm

(a)									(b)									
Sample ID 2.   Operator ID w   Elapsed Time 01   Mean Diam. 55   Rel. Var. 0.   Skew 0.   RmsError 3.	D 2.5V-AAm-1 rD Wu Time 00:10:06 am. 552.3 (nm) 0.000 0.245 r 3.7277e-02				Diame	5000.0 ter (nm)	Sample ID Operator ID Elapsed Time Mean Diam. Rel. Var. Skew RmsError	ID 2.6V-AAm-1 or ID wu d Time 00:11:07 Jiam. 529.1 (nm) r. 0.000 0.160 or 3.5647e-02			100 0 0 50.0 D				Diame	5000.0 ter (nm)		
d G(d)   500.61 0   507.04 0   513.57 0   520.17 0   526.86 0   533.64 0   540.51 57   547.46 92   554.50 100   568.86 22	t) C(d) 0 0 0 0 0 0 17 45 76 93 100	d 576.18 583.59 591.09 598.70 606.40 614.20 622.10 630.10 638.21 638.21 646.42 654.73	G(d) 0 0 0 0 0 0 0 0 0 0 0 0 0	C(d) 100 100 100 100 100 100 100 100 100 10	d 663.15 671.68 689.02 689.07 697.94 706.92 716.01 725.22 734.55 734.55 744.00 753.57	G(d) 0 0 0 0 0 0 0 0 0 0 0 0 0	<b>C(d)</b> 100 100 100 100 100 100 100 100 100 10	Print Window Copy For Spreadsheet Copy to Clipboard	d 483.12 489.26 499.54 501.77 508.15 514.61 521.15 527.77 534.47 541.27 548.14	G(d) 0 0 0 0 31 72 100 82 42 11	C(d) 0 0 0 9 30 60 84 97 100	d 555.11 562.16 569.30 576.54 583.86 591.28 598.80 606.41 614.11 621.91 629.82	G(d) 0 0 0 0 0 0 0 0 0 0 0 0 0 0	C(d) 100 100 100 100 100 100 100 100 100 10	d 637.82 645.93 654.13 662.44 670.86 679.39 688.02 696.76 705.62 714.58 723.66	G(d) 0 0 0 0 0 0 0 0 0 0 0 0 0 0	C(d) 100 100 100 100 100 100 100 100 100 10	Print Window Dopy For Spreadsheet Copy to Clipboard

**Figure S8.** Typical screenshots showing DLS size distribution of PAAm microgels, which were synthesized at a potentiostat of (a) +2.5 V and (b) +2.6 V *versus* Ag/AgCl, and harvested after 3hs' reaction.



Figure S9. A screenshot showing DLS size distribution of PAA microgels.



Figure S10. IR spectrum of the purified PAA microgels.