Electronic Supplementary Material (ESI) for Polymer Chemistry.

Supporting Information

Elucidating preparation-structure relationships for the morphology evolution during the RAFT dispersion polymerization of N-acryloyl thiomorpholine

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Table S1. Overview of the synthesized polymers and micelles including abbreviations, polymerization conditions and properties.

Exp.	**Polymer**	**T [°C]**	**vol%$_{Diox}$ [%]**	**c [M]**	**M_n [a] [kg mol^{-1}]**	**M_n [b] [kg mol^{-1}]**	**Đ[b]**	**D_H [nm]**	**PDI[c]**	**Morph.[d]**
					PNAM$_n$					
P$_{25}$	PNAM$_{25}$				3.8	4.1	1.10	-	-	-
P$_{50}$	PNAM$_{50}$	70	0	6.5	7.3	7.1	1.06	-	-	-
P$_{100}$	PNAM$_{100}$				14.4	13.1	1.06	-	-	-
					PNAM$_{25}$-*b*-NAT$_n$					
P$_{25}$-1*				0.2		7.5	1.12	31	0.09	S
P$_{25}$-2*			0	0.6		7.5	1.13	46	0.15	S / W
P$_{25}$-3*				1		7.3	1.12	93	0.18	W
P$_{25}$-4				0.2		7.3	1.10	55	0.09	S / W
P$_{25}$-5	PNAM$_{25}$-*b*-PNAT$_{25}$	70	10	0.6	7.7	7.1	1.10	119	0.21	W
P$_{25}$-6				1		7.0	1.08	-	-	bW
P$_{25}$-7				0.2		7.1	1.08	-	-	bW
P$_{25}$-8			25	0.6		7.0	1.08	-	-	bW
P$_{25}$-9				1		7.0	1.08	-	-	bW
P$_{25}$-10			0	0.2		11.3	1.37	P	P	P
P$_{25}$-11	PNAM$_{25}$-*b*-PNAT$_{50}$	70	10	0.2	11.6	11.1	1.17	42	0.06	S / sW
P$_{25}$-12				0.6		10.7	1.16	P	P	P
P$_{25}$-13			25	0.2		10.5	1.08	88	0.06	V
P$_{25}$-14	PNAM$_{25}$-*b*-PNAT$_{60}$	70	10	0.2	13.2	11.8	1.27	79	0.12	S / sW
P$_{25}$-15			10	0.2		12.2	1.27	88	0.12	sW
P$_{25}$-16	PNAM$_{25}$-*b*-PNAT$_{70}$	70		0.6	14.8	12.8	1.24	P	P	P
P$_{25}$-17			25	0.2		13.3	1.16	117	0.11	V
P$_{25}$-18	PNAM$_{25}$-*b*-PNAT$_{75}$	70	10	0.2	15.6	17.3	1.74	P	P	P
P$_{25}$-19		70	25			13.6	1.19	P	P	P
P$_{25}$-20	PNAM$_{25}$-*b*-PNAT$_{100}$	70	25	0.2	19.5	16.1	1.30	P	P	P

Exp.	Polymer	T [°C]	vol%$_{Diox}$ [%]	c [M]	M_n [a] [kg mol^{-1}]	M_n [b] [kg mol^{-1}]	Đ[b]	D_H [nm]	PDI[c]	Morph.[d]
				PNAM$_{25}$-*b*-PNAT$_n$ - 90°C						
P_{25}-21*				0.2		7.5	1.13	44	0.06	S
P_{25}-22*			0	0.6		7.4	1.13	109	0.16	V
P_{25}-23*				1		7.6	1.12	569	0.67	L
P_{25}-24				0.2		7.4	1.10	124	0.07	bW/V/ L
P_{25}-25	PNAM$_{25}$-*b*-PNAT$_{25}$	90	10	0.6	7.7	7.5	1.10	-	-	bW / L
P_{25}-26				1		7.5	1.11	-	-	bW / L
P_{25}-27				0.2		7.1	1.14	220	0.18	bW/V/L
P_{25}-28			25	0.6		7.4	1.10	-	-	bW / L
P_{25}-29				1		7.6	1.10	-	-	bW / L
P_{25}-30				0.2		11.2	1.29	44	0.05	S / sW
P_{25}-31	PNAM$_{25}$-*b*-PNAT$_{50}$	90	0	0.6	11.6	11.6	1.32	P	P	P
P_{25}-32				1		11.0	1.30	P	P	P
P_{25}-33			10	0.2		11.2	1.16	P	P	P
P_{25}-34	PNAM$_{25}$-*b*-PNAT$_{70}$	90	0	0.2	14.8	14.0	1.49	111	0.14	S / sW
				PNAM$_{50}$-*b*-PNAT$_n$						
P_{50}-1				0.2		11.6	1.09	22	0.11	S
P_{50}-2	PNAM$_{50}$-*b*-PNAT$_{30}$	70	0	0.6	12.0	11.5	1.09	20	0.16	S
P_{50}-3				1		11.6	1.09	28	0.13	S
P_{50}-4		50		0.2		13.5	1.32	29	0.09	-
P_{50}-5		90		0.2		13.3	1.15	66	0.20	-
P_{50}-6	PNAM$_{50}$-*b*-PNAT$_{50}$		0	0.2	15.2	14.3	1.22	57	0.13	S / sW
P_{50}-7		70		0.6		14.2	1.24	50	0.18	S / sW
P_{50}-8				1		14.1	1.23	36	0.19	S / sW
P_{50}-9				0.2		13.7	1.13	30	0.06	-
P_{50}-10			10	0.6		14.2	1.13	26	0.12	-
P_{50}-11	PNAM$_{50}$-*b*-PNAT$_{50}$	70		1	15.2	14.4	1.13	23	0.14	S
P_{50}-12				0.2		13.6	1.09	28	0.07	S
P_{50}-13			25	0.6		13.9	1.09	26	0.08	-
P_{50}-14				1		13.8	1.09	24	0.10	S

Exp.	Polymer	T [°C]	vol%$_{Diox}$ [%]	c [M]	M_n [a] [kg mol^{-1}]	M_n [b] [kg mol^{-1}]	Đ[b]	D_H [nm]	PDI[c]	Morph.[d]
P_{50}-15			0	0.2		20.1	1.70	P	P	P
P_{50}-16				0.2		19.4	1.32	60	0.09	S / sW
P_{50}-17			10	0.6		19.6	1.35	55	0.10	S / sW
P_{50}-18	PNAM$_{50}$-*b*-PNAT$_{100}$	70		1	23.0	20.2	1.33	54	0.09	S / sW
P_{50}-19				0.2		19.0	1.16	40	0.04	S
P_{50}-20			25	0.6		19.7	1.16	40	0.05	S
P_{50}-21				1		19.7	1.16	44	0.04	S
P_{50}-22	PNAM$_{50}$-*b*-PNAT$_{200}$		25	0.2	38.7	24.7	1.35	55	0.02	S
					PNAM$_{100}$-*b*-PNAT$_n$					
P_{100}-1				0.2		19.5	1.17	29	0.08	S
P_{100}-2			0	0.6		19.4	1.17	29	0.10	S
P_{100}-3				1		19.1	1.16	28	0.13	S
P_{100}-4				0.2		19.6	1.11	29	0.07	-
P_{100}-5	PNAM$_{100}$-*b*-PNAT$_{50}$	70	10	0.6	22.2	19.5	1.12	30	0.10	-
P_{100}-6				1		19.4	1.12	35	0.09	S
P_{100}-7				0.2		18.9	1.10	28	0.09	-
P_{100}-8			25	0.6		19.5	1.10	32	0.08	-
P_{100}-9				1		19.2	1.10	36	0.07	S
P_{100}-10			0	0.2		25.2	1.43	63	0.10	S / sW
P_{100}-11				0.2		24.1	1.28	40	0.06	S
P_{100}-12			10	0.6		24.0	1.29	37	0.09	S
P_{100}-13	PNAM$_{100}$-*b*-PNAT$_{100}$	70		1	30.1	24.8	1.27	40	0.08	S
P_{100}-14				0.2		23.7	1.18	36	0.03	-
P_{100}-15			25	0.6		24.3	1.18	37	0.06	S
P_{100}-16				1		24.8	1.18	47	0.18	S
PNAT$_{15}$					2.6	2.7	1.11	-	-	-
PNAT$_{30}$					5.0	4.7	1.10	-	-	-
PNAT$_{50}$	PNAT	70	83	2	8.1	7.3	1.08	-	-	-
PNAT$_{100}$					16.0	13.4	1.11	-	-	-

[a] Calculated based on $[M]_0/[CTA]_0 \times$ monomer conversion. [b] Determined by SEC (Eluent: DMAc + 0.21 wt% LiCl, PS-calibration) [c] Determined by DLS measurements of the purified structures (c: 1 mg mL^{-1}). [d] Morphology judgement based on (cryo)-TEM investigations. The morphologies were categorized as follows: spheres (**S**), short worms (**sW**), worms (**W**), vesicles (**V**), lamellae (**L**), and precipitation (**P**). Exp.: experiment, T: reaction temperature, c: monomer concentration, Morph.: morphology. *Samples were previously reported.[1]

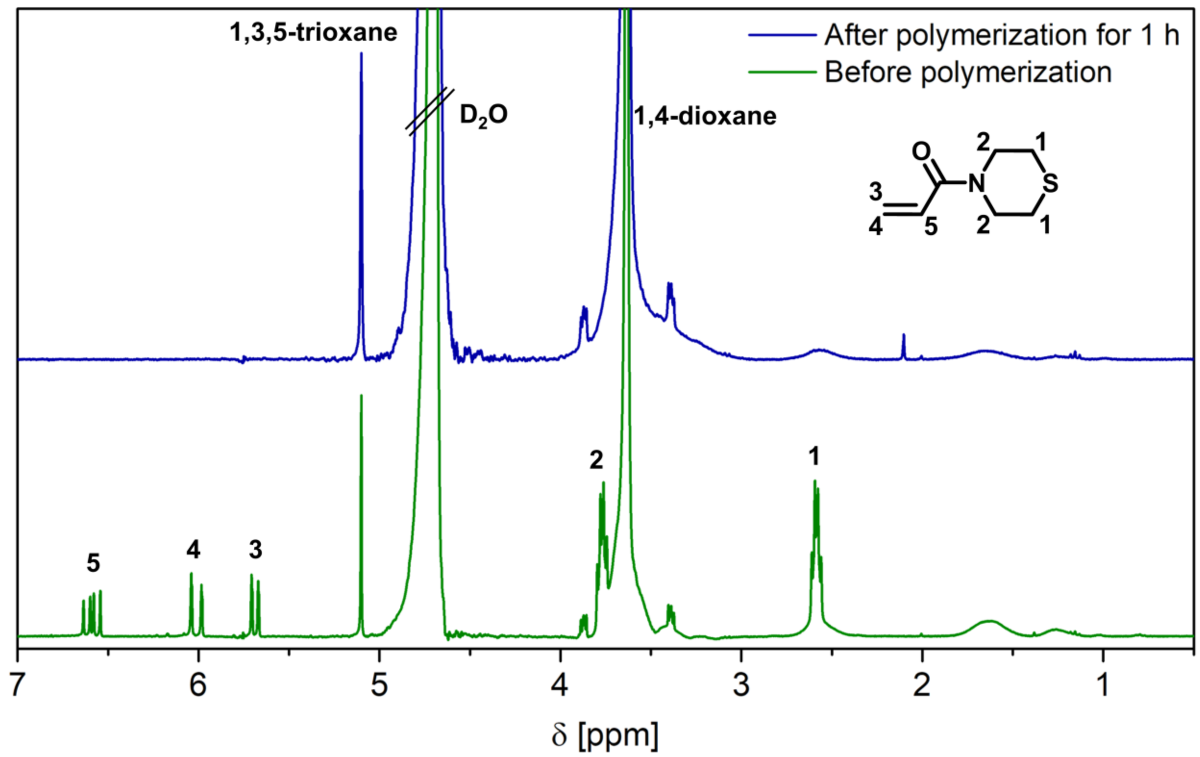
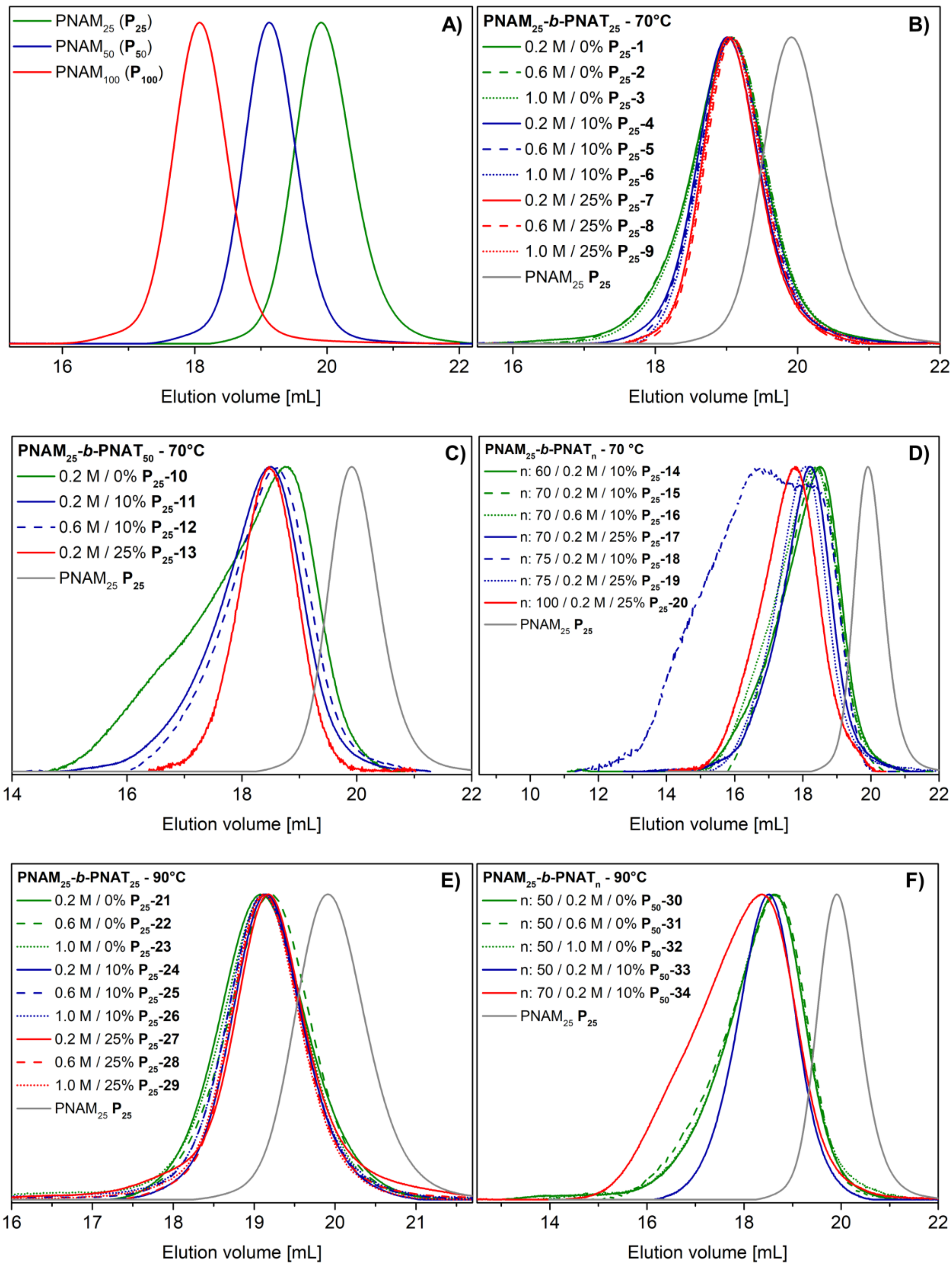


Figure S1. NMR overlay of the RAFT dispersion polymerization of NAT using $PNAM_{100}$ as mCTA (70 °C, 0.2 M, 25 vol% 1,4 dioxane, $\mathbf{P_{100}}$**-7**); 1,3,5-trioxane was used as internal standard.


A)
$PNAM_{25}$ (P_{25})
$PNAM_{50}$ (P_{50})
$PNAM_{100}$ (P_{100})
16 18 20 22
Elution volume [mL]
B)
$PNAM_{25}$-*b*-$PNAT_{25}$ - 70°C
0.2 M / 0% P_{25}-1
0.6 M / 0% P_{25}-2
1.0 M / 0% P_{25}-3
0.2 M / 10% P_{25}-4
0.6 M / 10% P_{25}-5
1.0 M / 10% P_{25}-6
0.2 M / 25% P_{25}-7
0.6 M / 25% P_{25}-8
1.0 M / 25% P_{25}-9
$PNAM_{25}$ P_{25}
16 18 20 22
Elution volume [mL]
C)
$PNAM_{25}$-*b*-$PNAT_{50}$ - 70°C
0.2 M / 0% P_{25}-10
0.2 M / 10% P_{25}-11
0.6 M / 10% P_{25}-12
0.2 M / 25% P_{25}-13
$PNAM_{25}$ P_{25}
14 16 18 20 22
Elution volume [mL]
D)
$PNAM_{25}$-*b*-$PNAT_{n}$ - 70 °C
n: 60 / 0.2 M / 10% P_{25}-14
n: 70 / 0.2 M / 10% P_{25}-15
n: 70 / 0.6 M / 10% P_{25}-16
n: 70 / 0.2 M / 25% P_{25}-17
n: 75 / 0.2 M / 10% P_{25}-18
n: 75 / 0.2 M / 25% P_{25}-19
n: 100 / 0.2 M / 25% P_{25}-20
$PNAM_{25}$ P_{25}
10 12 14 16 18 20 22
Elution volume [mL]
E)
$PNAM_{25}$-*b*-$PNAT_{25}$ - 90°C
0.2 M / 0% P_{25}-21
0.6 M / 0% P_{25}-22
1.0 M / 0% P_{25}-23
0.2 M / 10% P_{25}-24
0.6 M / 10% P_{25}-25
1.0 M / 10% P_{25}-26
0.2 M / 25% P_{25}-27
0.6 M / 25% P_{25}-28
1.0 M / 25% P_{25}-29
$PNAM_{25}$ P_{25}
16 17 18 19 20 21
Elution volume [mL]
F)
$PNAM_{25}$-*b*-$PNAT_{n}$ - 90°C
n: 50 / 0.2 M / 0% P_{50}-30
n: 50 / 0.6 M / 0% P_{50}-31
n: 50 / 1.0 M / 0% P_{50}-32
n: 50 / 0.2 M / 10% P_{50}-33
n: 70 / 0.2 M / 10% P_{50}-34
$PNAM_{25}$ P_{25}
14 16 18 20 22
Elution volume [mL]

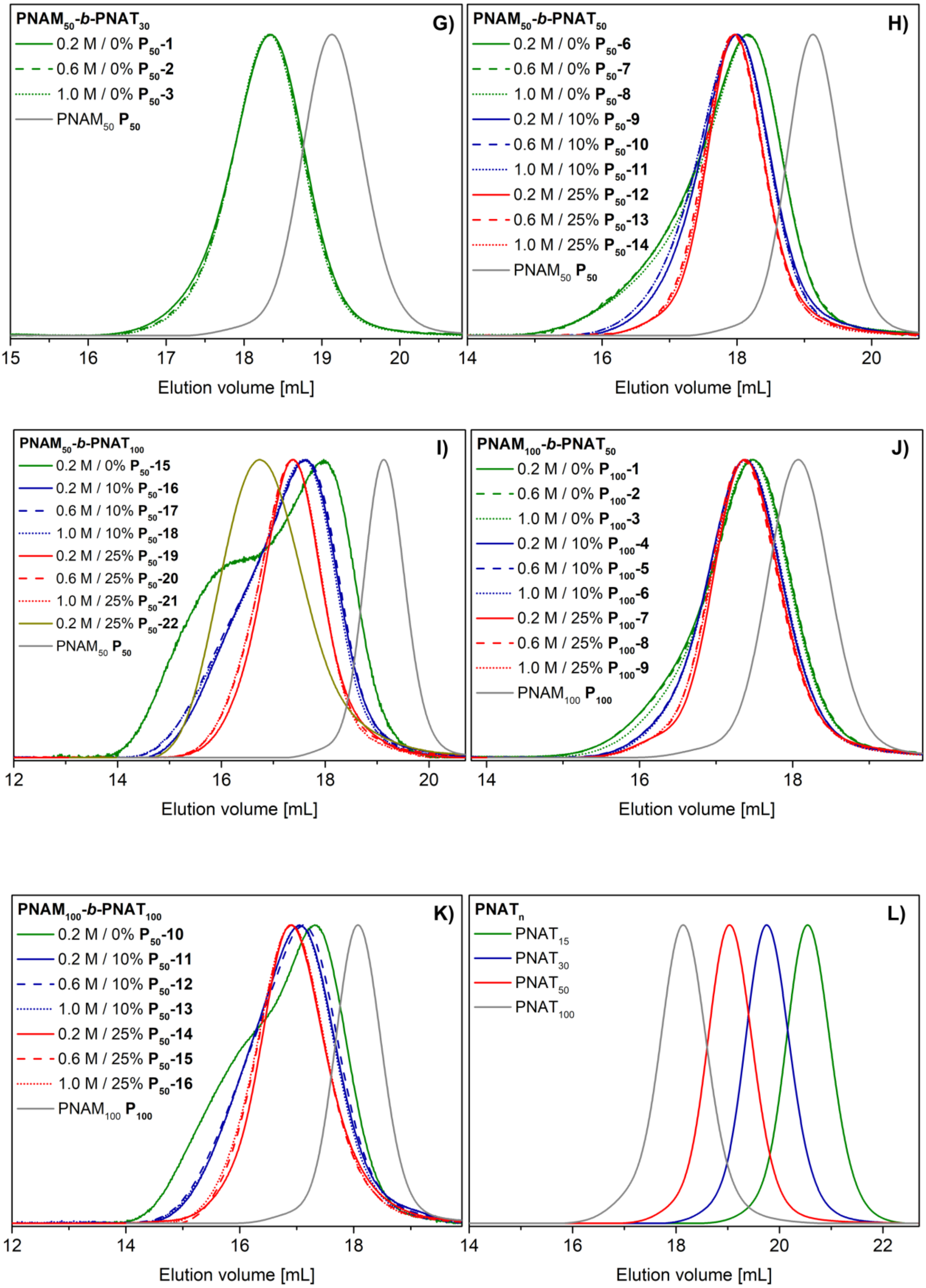


Figure S2. Overview of the SEC curves of the synthesized polymers. (Eluent: DMAc + 0.21% LiCl, PS-calibration).

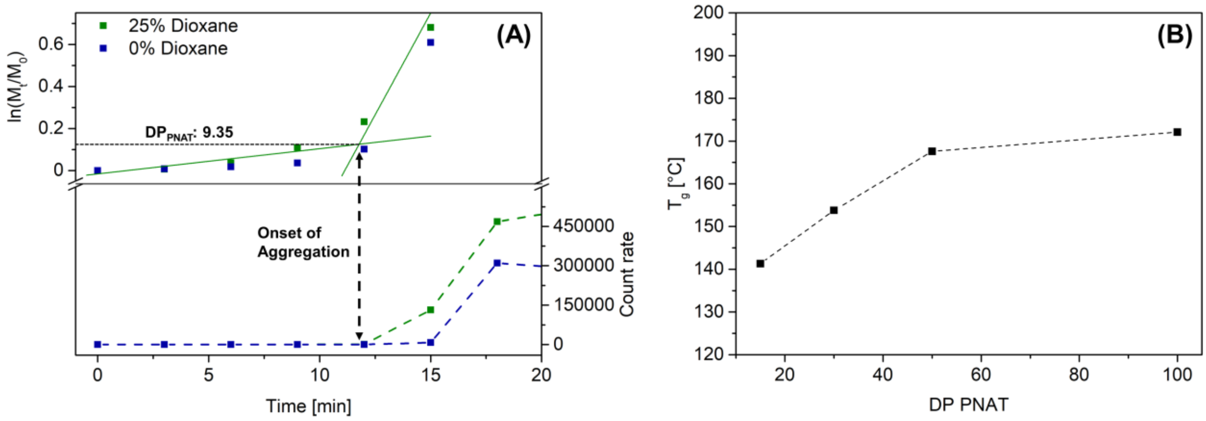
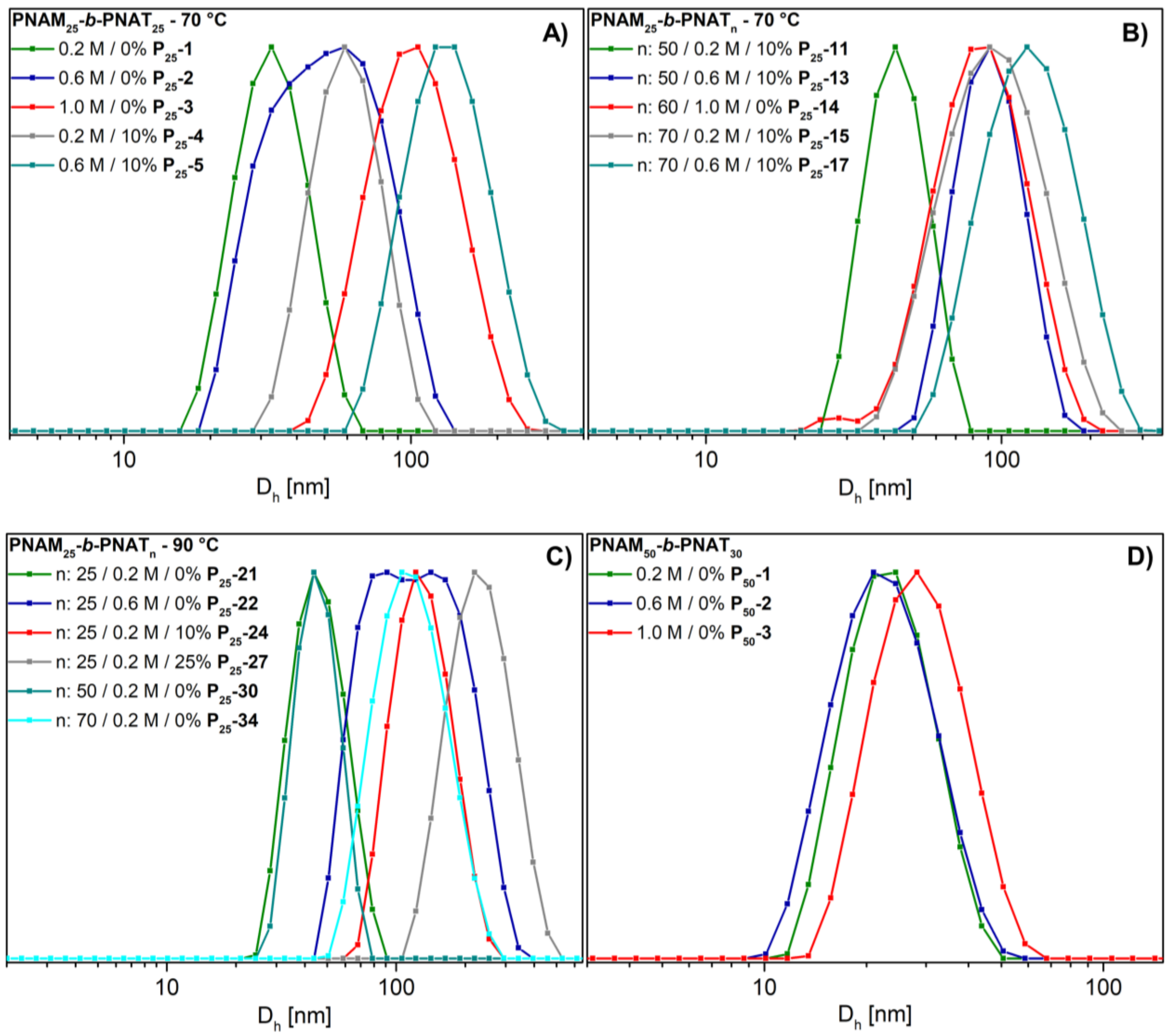


Figure S3. A) Semilogarithmic plot of the monomer conversion and the derived count rate vs. reaction time of the PISA of PNAM25-*b*-PNAT70 for 0 and 25 vol% 1,4-dioxane at 70 °C and 0.2 M. B) Dependency of the T_g of PNAT homopolymers on the DP, determined by DSC measurements.



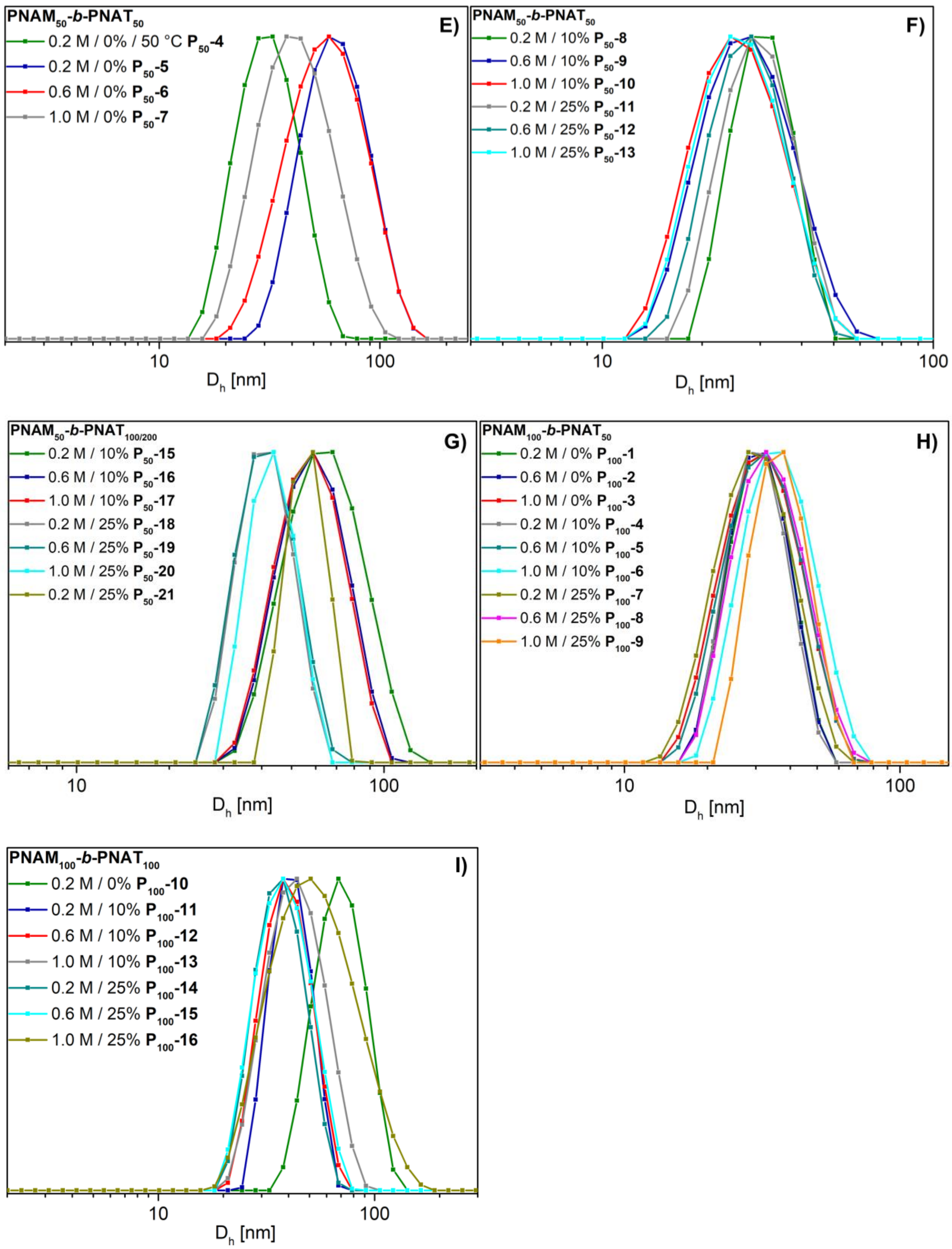


Figure S4. Overview of intensity weight size distributions of the prepared micelles determined by DLS (1 mg mL^{-1} in H_2O).

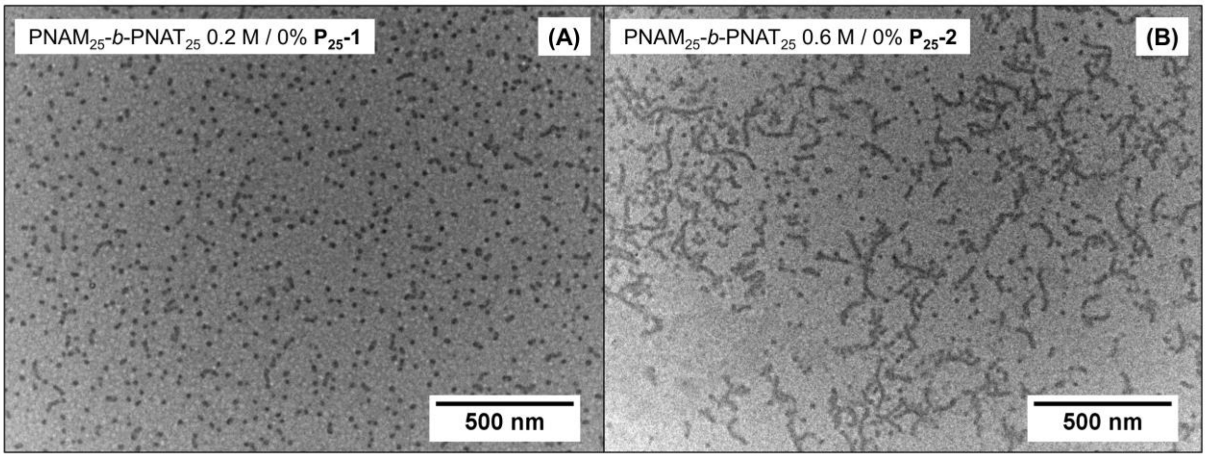


Figure S5. Exemplary dry-TEM images of PNAM$_{25}$-*b*-PNAT$_{25}$ micelles after dialysis. A) Prepared with 0 vol% 1,4-dioxane at 0.2 M and 70 °C. B) Prepared with 0 vol% 1,4-dioxane at 0.6 M and 70 °C.

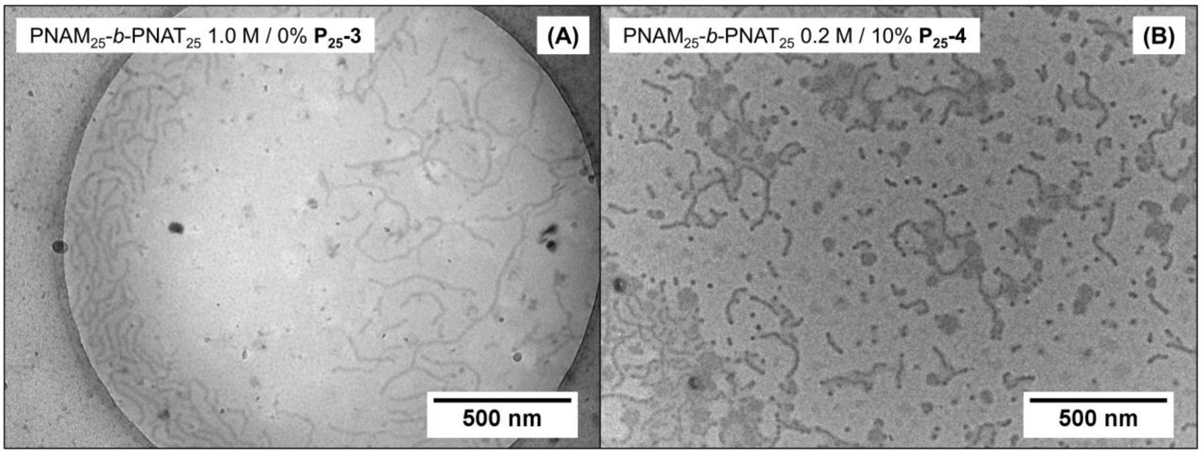


Figure S6. Exemplary TEM images of PNAM$_{25}$-*b*-PNAT$_{25}$ micelles after dialysis. A) Cryo-TEM, prepared with 0 vol% 1,4-dioxane at 1.0 M and 70 °C. B) Dry-TEM, prepared with 10 vol% 1,4-dioxane at 0.2 M and 70 °C.

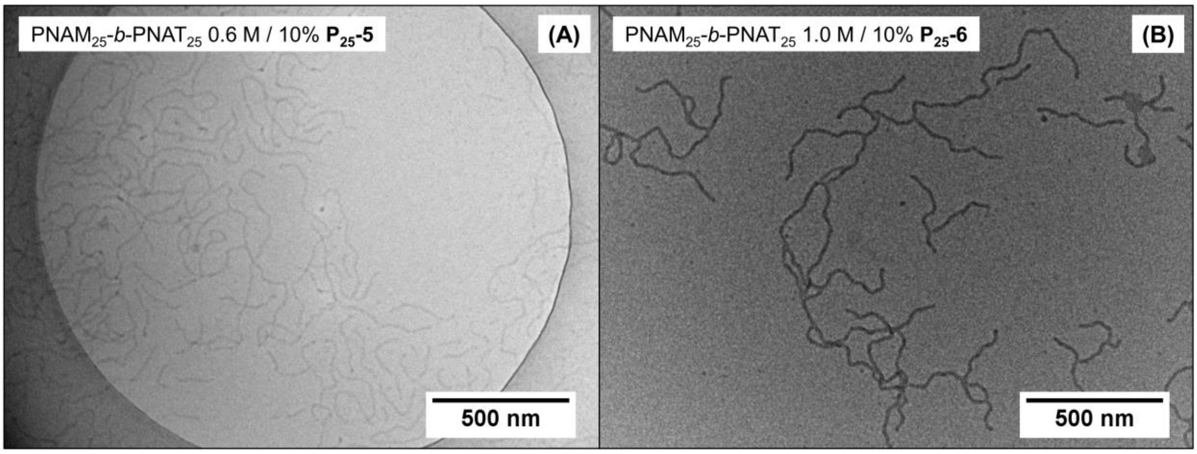


Figure S7. Exemplary TEM images of PNAM$_{25}$-*b*-PNAT$_{25}$ micelles after dialysis. A) Cryo-TEM, prepared with 10 vol% 1,4-dioxane at 0.6 M and 70 °C. B) Dry-TEM, prepared with 10 vol% 1,4-dioxane at 0.6 M and 70 °C.

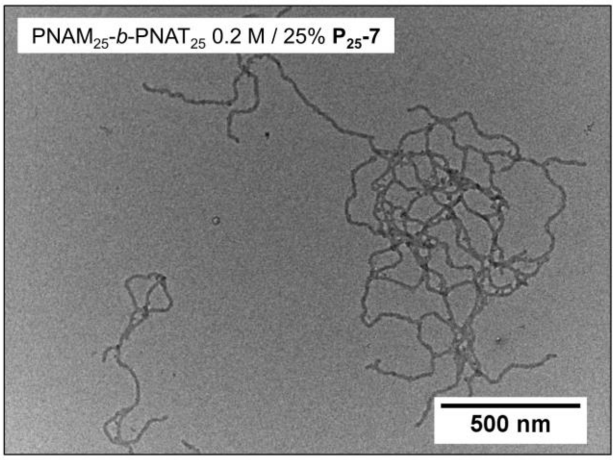


Figure S8. Exemplary dry-TEM images of PNAM$_{25}$-*b*-PNAT$_{25}$ micelles after dialysis, prepared with 25 vol% 1,4-dioxane at 0.2 M and 70 °C.

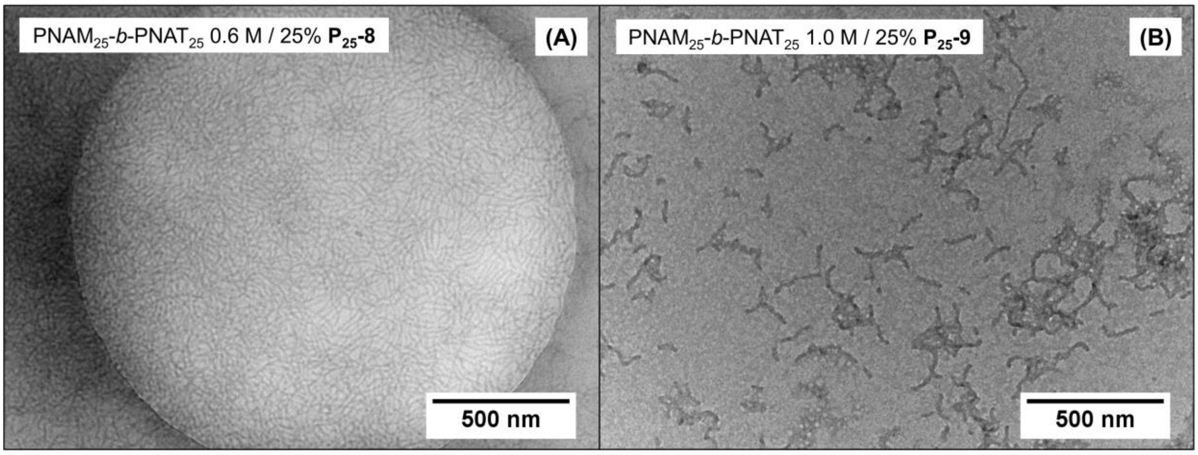


Figure S9. Exemplary TEM images of PNAM$_{25}$-*b*-PNAT$_{25}$ micelles after dialysis. A) Cryo-TEM, prepared with 25 vol% 1,4-dioxane at 0.6 M and 70 °C. B) Dry-TEM, prepared with 25 vol% 1,4-dioxane at 0.6 M and 70 °C.

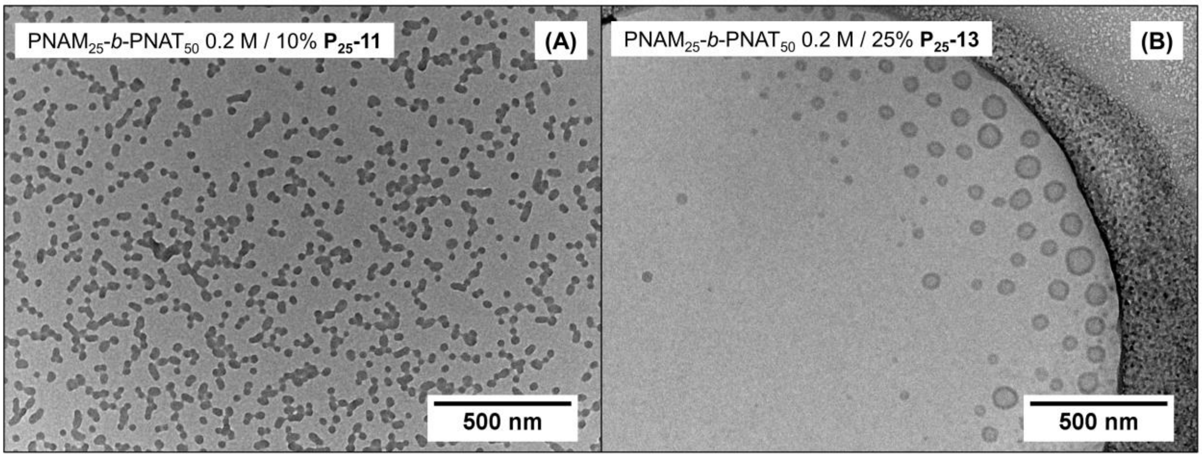


Figure S10. Exemplary TEM images of micelles after dialysis. A) Dry-TEM of PNAM$_{25}$-*b*-PNAT$_{50}$ prepared with 10 vol% 1,4-dioxane at 0.2 M and 70 °C. B) Cryo-TEM of PNAM$_{25}$-*b*-PNAT$_{50}$ prepared with 25 vol% 1,4-dioxane at 0.2 M and 70 °C.

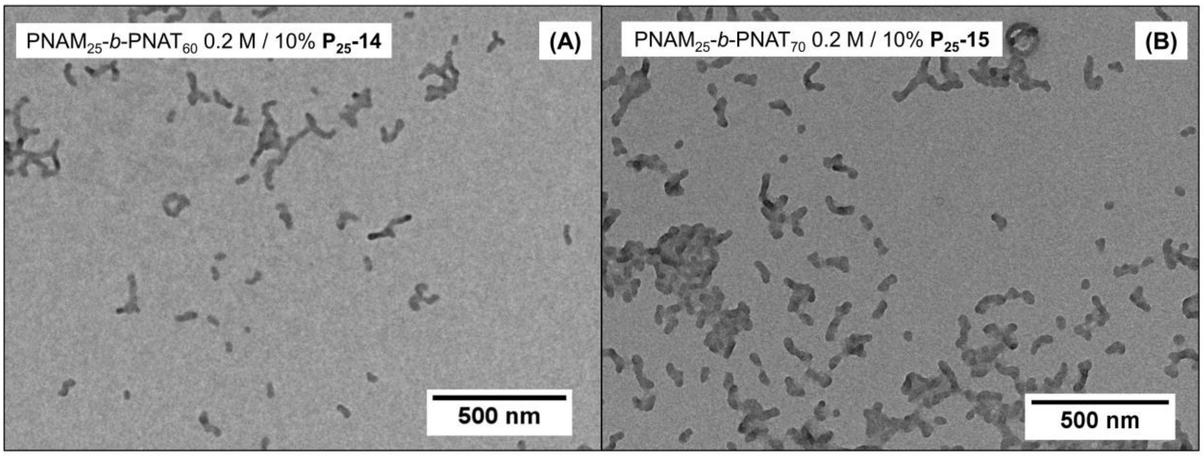


Figure S11. Exemplary dry-TEM images of micelles after dialysis. A) $PNAM_{25}$-*b*-$PNAT_{60}$, prepared with 10 vol% 1,4-dioxane at 0.2 M and 70 °C. B) $PNAM_{25}$-*b*-$PNAT_{70}$ prepared with 10 vol% 1,4-dioxane at 0.2 M and 70 °C.

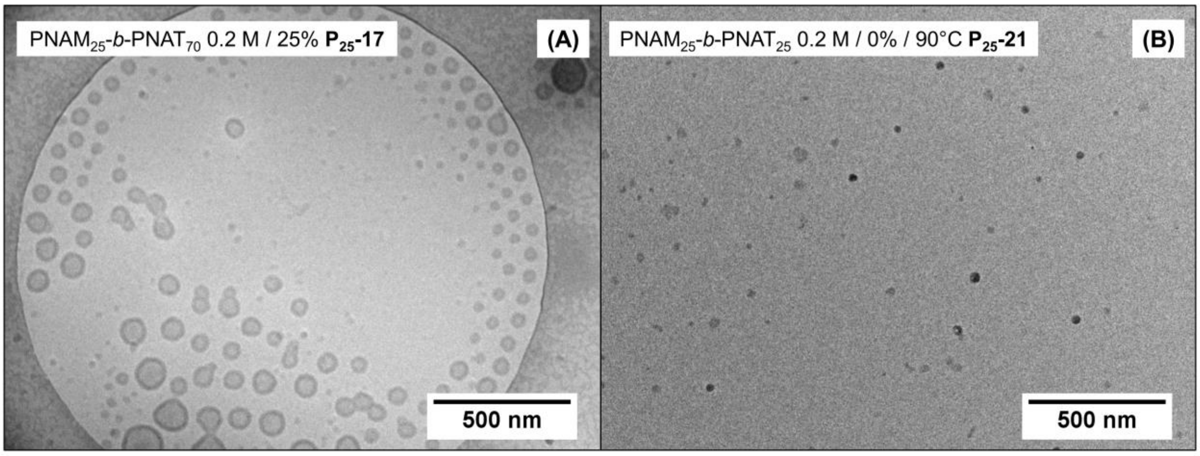


Figure S12. Exemplary TEM images of micelles after dialysis. A) Cryo-TEM of $PNAM_{25}$-*b*-$PNAT_{70}$ prepared with 25 vol% 1,4-dioxane at 0.2 M and 70 °C. B) Dry-TEM of of $PNAM_{25}$-*b*-$PNAT_{25}$ prepared with 0 vol% 1,4-dioxane at 0.2 M and 90 °C.

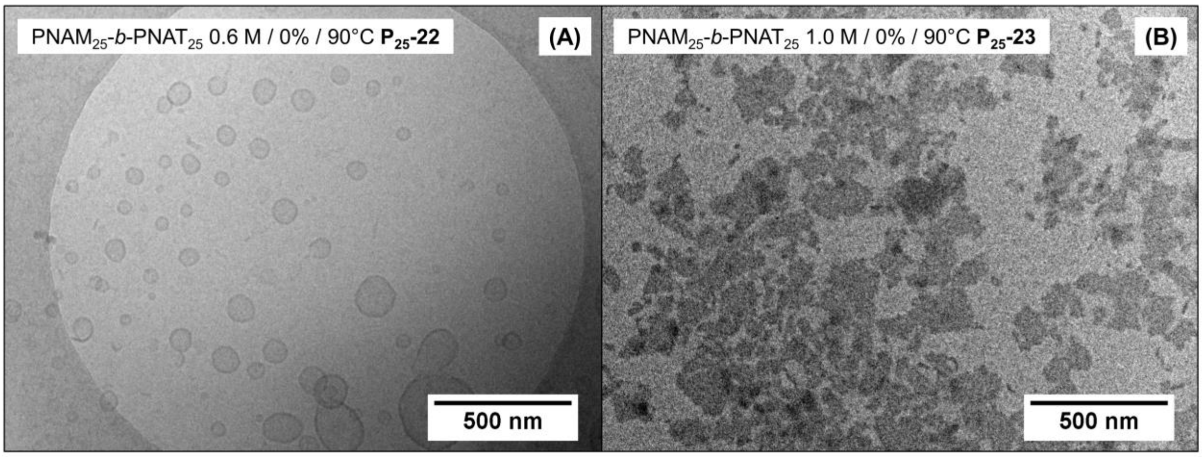


Figure S13. Exemplary TEM images of micelles after dialysis. A) Cryo-TEM of $PNAM_{25}$-*b*-$PNAT_{25}$ prepared with 0 vol% 1,4-dioxane at 0.6 M and 90 °C. B) Dry-TEM of $PNAM_{25}$-*b*-$PNAT_{25}$ prepared with 0 vol% 1,4-dioxane at 1.0 M and 90 °C.

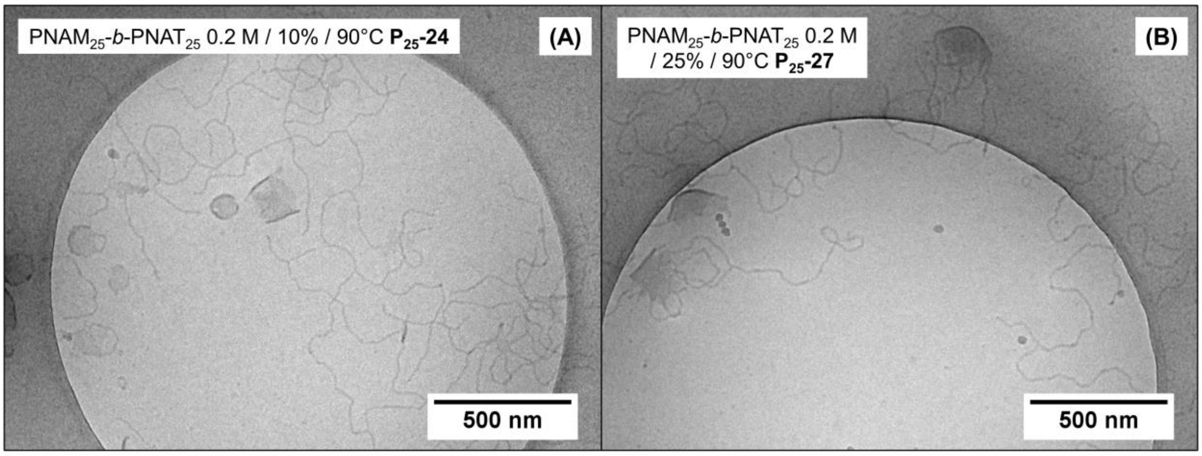


Figure S14. Exemplary TEM images of $PNAM_{25}$-*b*-$PNAT_{25}$ micelles after dialysis. A) Cryo-TEM prepared with 10 vol% 1,4-dioxane at 0.2 M and 90 °C. B) Cryo-TEM prepared with 25 vol% 1,4-dioxane at 0.2 M and 90 °C.

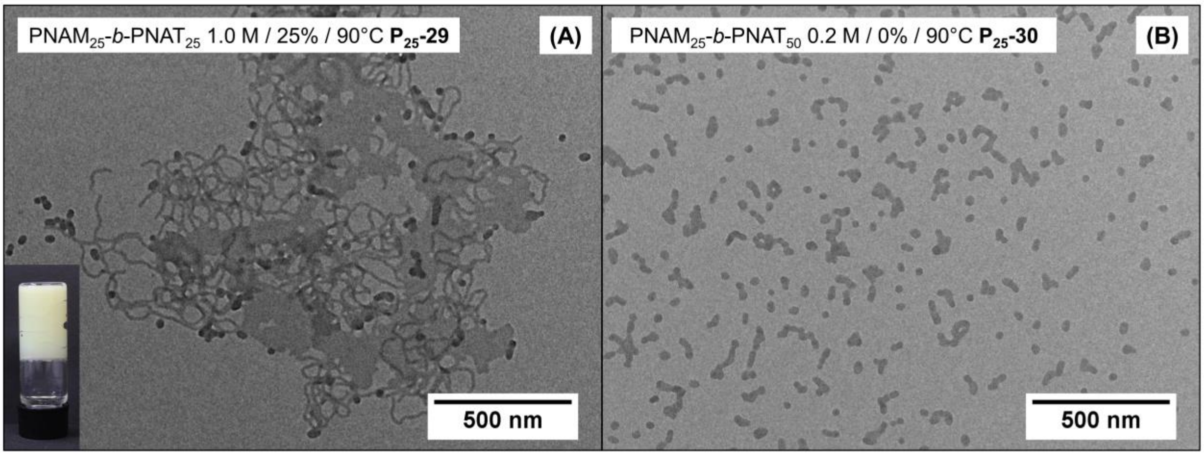


Figure S15. Exemplary TEM images of the micelles after dialysis. A) Dry-TEM of $PNAM_{25}$-*b*-$PNAT_{25}$ Prepared with 25 vol% 1,4-dioxane at 1.0 M and 90 °C (the inset shows a corresponding photograph of the reaction solution). B) Dry-TEM of $PNAM_{25}$-*b*-$PNAT_{50}$ prepared with 0 vol% 1,4-dioxane at 0.2 M and 90 °C.

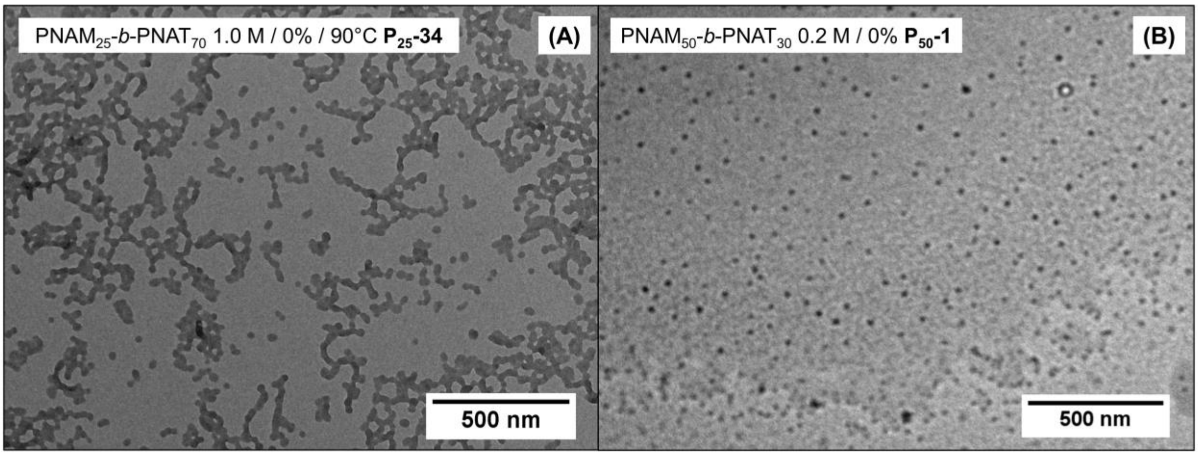


Figure S16. Exemplary TEM images of micelles after dialysis. A) Dry-TEM of $PNAM_{25}$-*b*-$PNAT_{70}$ prepared with 0 vol% 1,4-dioxane at 1.0 M and 90 °C. B) Dry-TEM of $PNAM_{50}$-*b*-$PNAT_{30}$ prepared with 0 vol% 1,4-dioxane at 0.2 M and 70 °C.

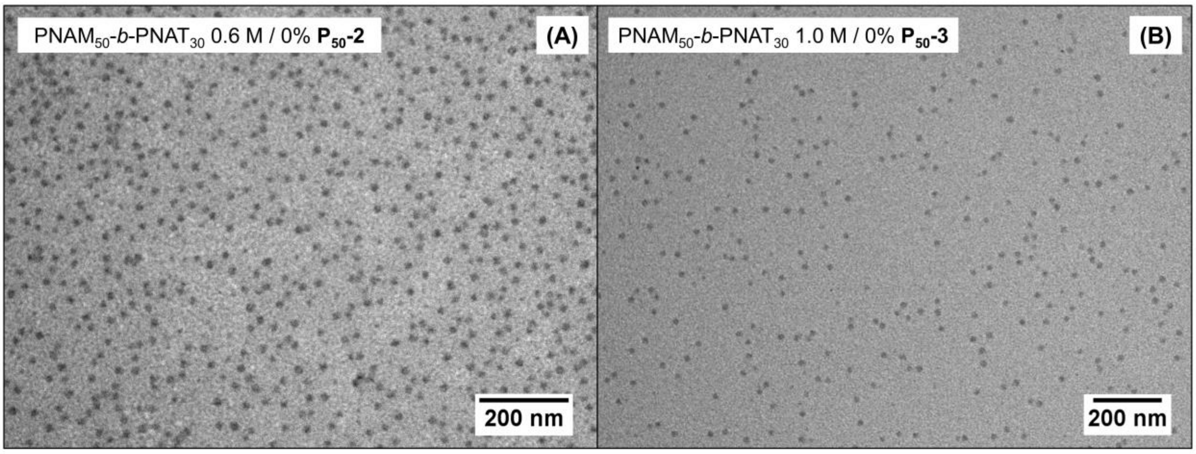


Figure S17. Exemplary dry-TEM images of micelles after dialysis. A) PNAM$_{50}$-*b*-PNAT$_{30}$ prepared with 0 vol% 1,4-dioxane at 0.6 M and 70 °C. B) PNAM$_{50}$-*b*-PNAT$_{30}$ prepared with 0 vol% 1,4-dioxane at 1.0 M and 70 °C.

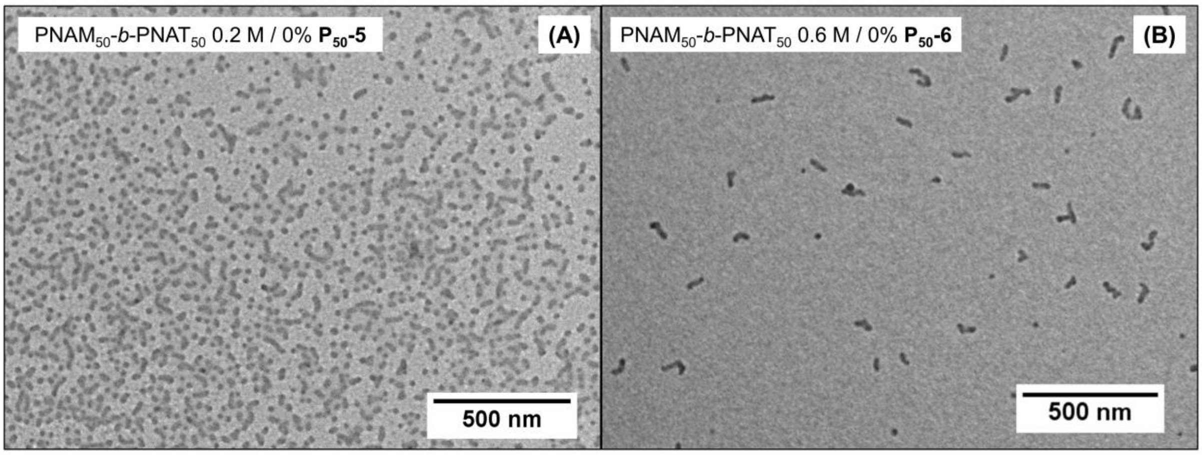


Figure S18. Exemplary dry-TEM images of micelles after dialysis. A) PNAM$_{50}$-*b*-PNAT$_{50}$ prepared with 0 vol% 1,4-dioxane at 0.2 M and 70 °C. B) PNAM$_{50}$-*b*-PNAT$_{50}$ prepared with 0 vol% 1,4-dioxane at 0.6 M and 70 °C.

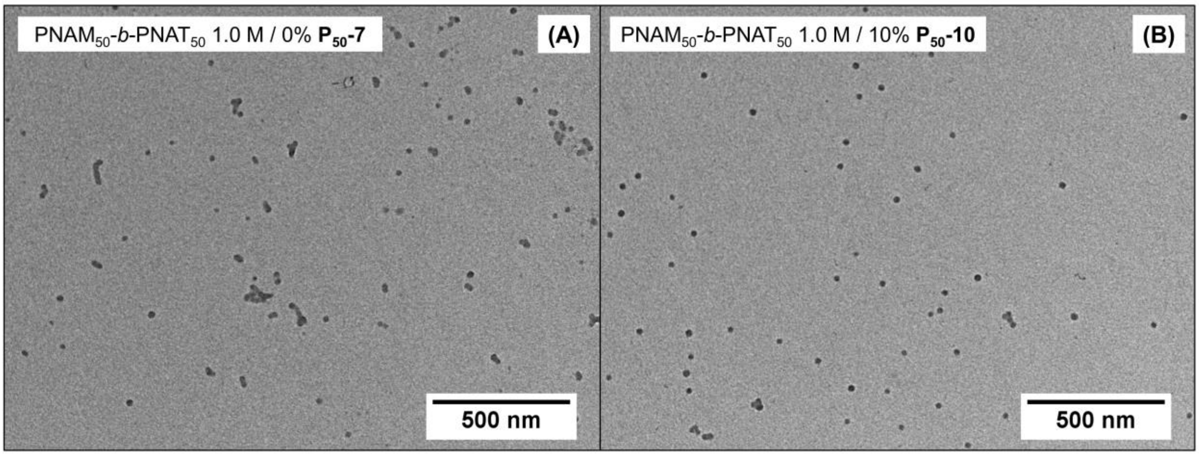


Figure S19. Exemplary dry-TEM images of $PNAM_{50}$-*b*-$PNAT_{50}$ micelles after dialysis. A) Prepared with 0 vol% 1,4-dioxane at 1.0 M and 70 °C. B) Prepared with 10 vol% 1,4-dioxane at 1.0 M and 70 °C.

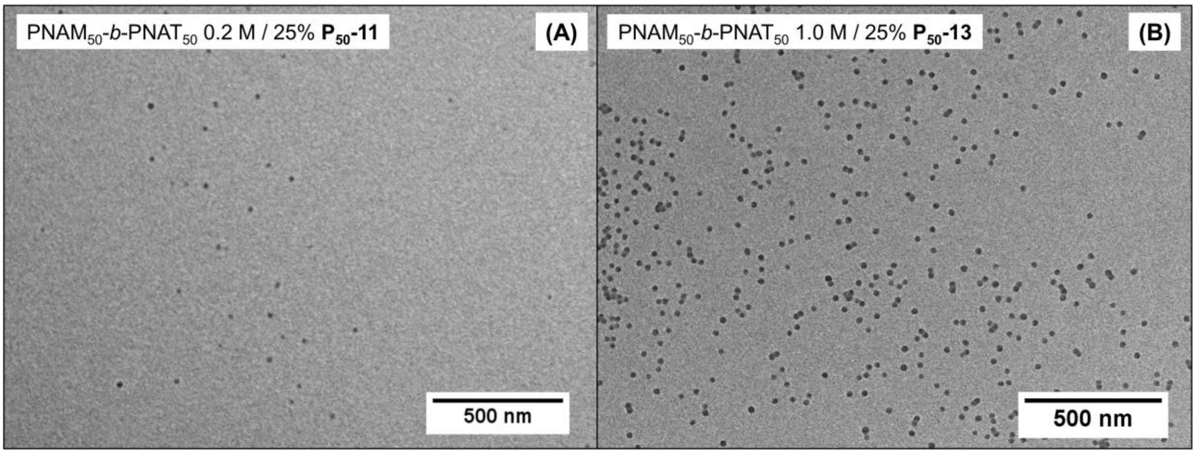


Figure S20. Exemplary dry-TEM images of $PNAM_{50}$-*b*-$PNAT_{50}$ micelles after dialysis. A) Prepared with 25 vol% 1,4-dioxane at 0.2 M and 70 °C. B) Prepared with 25 vol% 1,4-dioxane at 1.0 M and 70 °C.

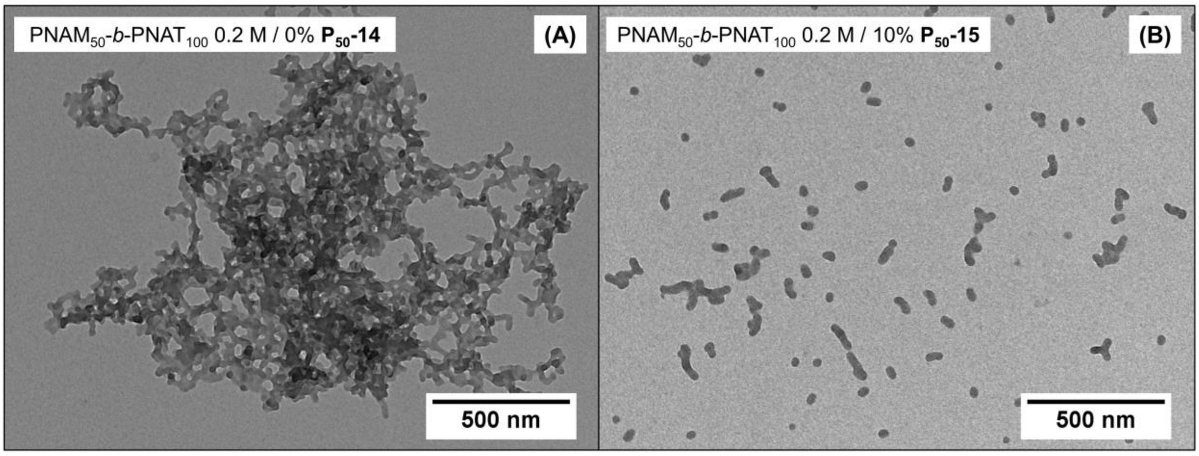


Figure S21. Exemplary dry-TEM images of micelles after dialysis. A) PNAM$_{50}$-*b*-PNAT$_{100}$ prepared with 0 % 1,4-dioxane at 1.0 M and 70 °C. B) PNAM$_{50}$-*b*-PNAT$_{100}$ prepared with 10 vol% 1,4-dioxane at 0.2 M and 70 °C.

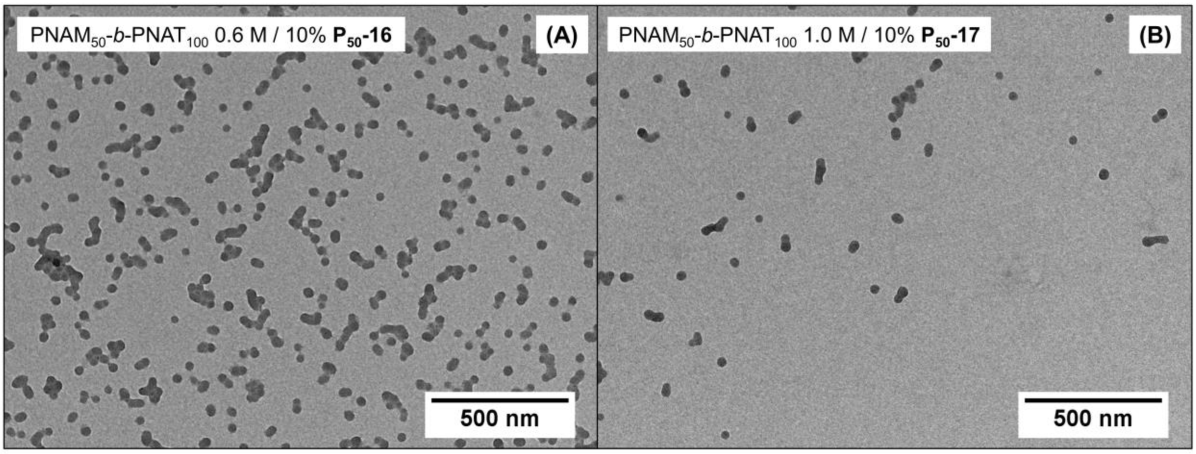


Figure S22. Exemplary dry-TEM images of micelles after dialysis. A) PNAM$_{50}$-*b*-PNAT$_{100}$ prepared with 10 vol% 1,4-dioxane at 0.6 M and 70 °C. B) PNAM$_{50}$-*b*-PNAT$_{100}$ prepared with 10 vol% 1,4-dioxane at 1.0 M and 70 °C.

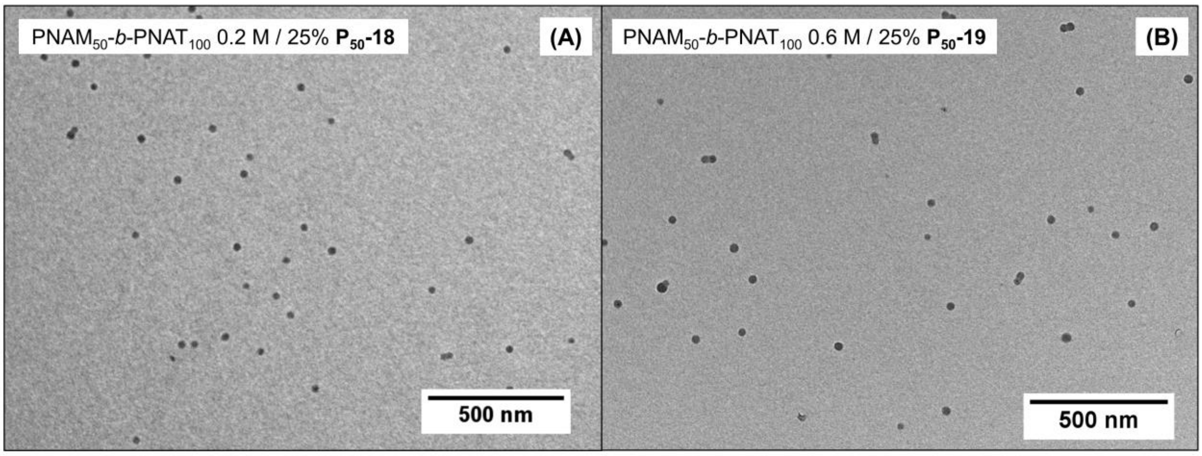


Figure S23. Exemplary dry-TEM images of PNAM$_{50}$-*b*-PNAT$_{100}$ micelles after dialysis. A) Prepared with 25 vol% 1,4-dioxane at 0.2 M and 70 °C. B) Prepared with 25 vol% 1,4-dioxane at 0.6 M and 70 °C.

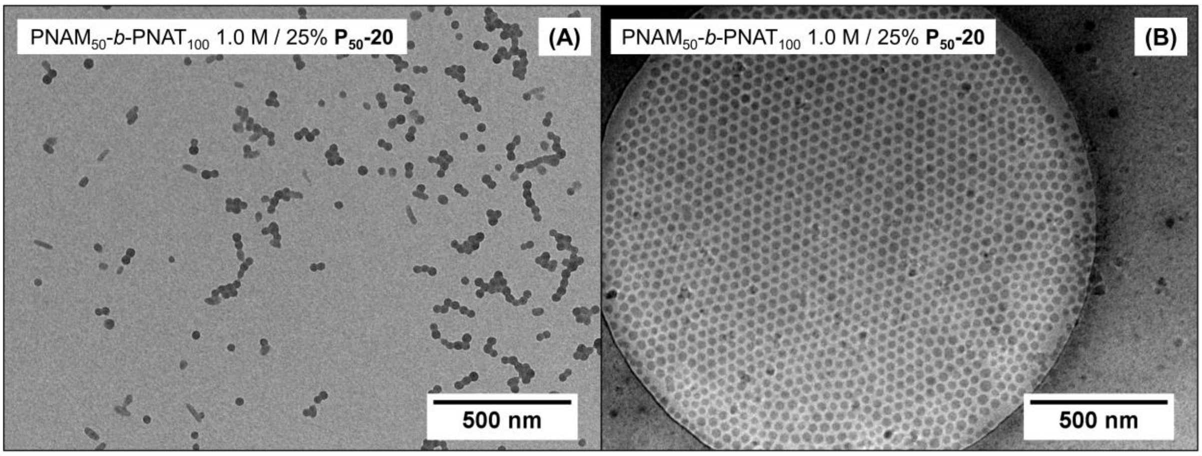


Figure S24. Exemplary TEM images of PNAM$_{50}$-*b*-PNAT$_{100}$ micelles after dialysis. A) Dry-TEM, prepared with 25 vol% 1,4-dioxane at 1.0 M and 70 °C. B) Cryo-TEM, prepared with 25 vol% 1,4-dioxane at 1.0 M and 70 °C.

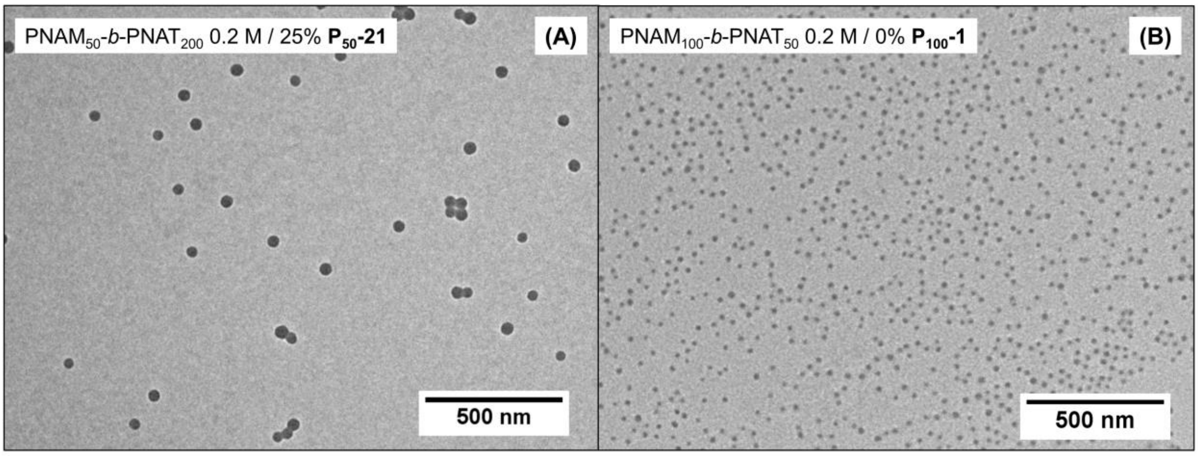


Figure S25. Exemplary dry-TEM images of micelles after dialysis. A) Dry-TEM of PNAM$_{50}$-*b*-PNAT$_{200}$ prepared with 25 vol% 1,4-dioxane at 0.2 M and 70 °C. B) Dry-TEM of PNAM$_{100}$-*b*-PNAT$_{50}$ prepared with 0 vol% 1,4-dioxane at 0.2 M and 70 °C.

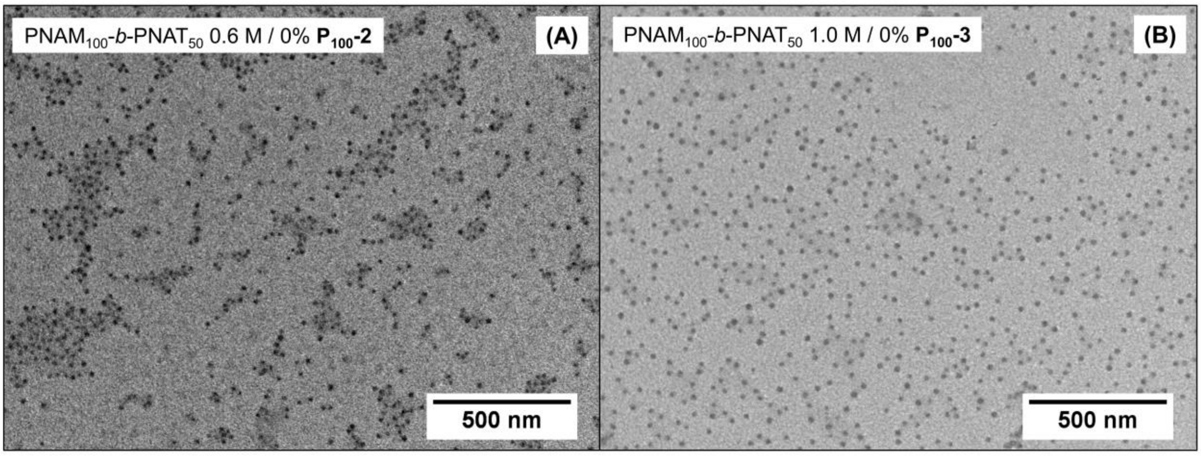


Figure S26. Exemplary TEM images of PNAM$_{100}$-*b*-PNAT$_{50}$ micelles after dialysis. A) Dry-TEM, prepared with 0 vol% 1,4-dioxane at 0.6 M and 70 °C. B) Dry-TEM, prepared with 0 vol% 1,4-dioxane at 1.0 M and 70 °C.

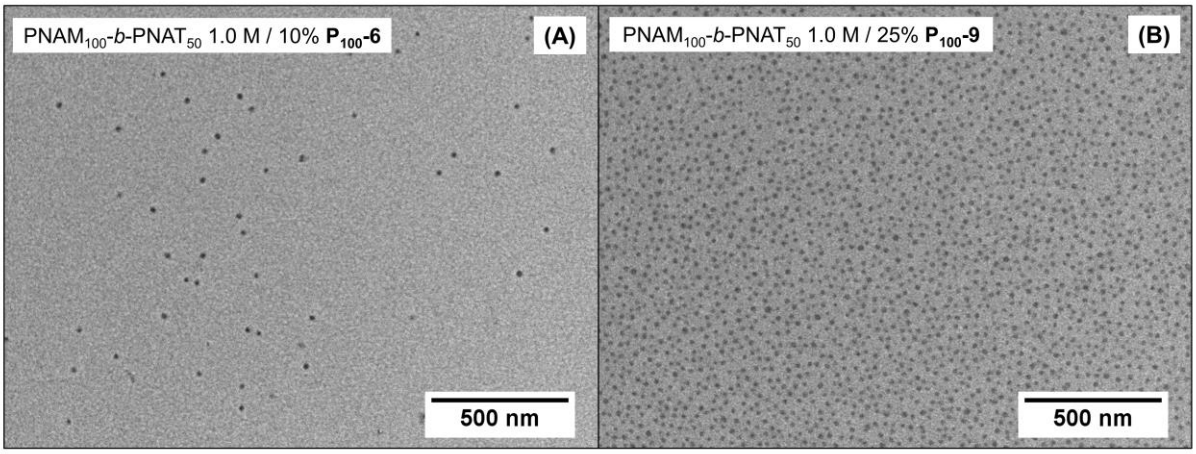


Figure S27. Exemplary dry-TEM images of micelles after dialysis. A) $PNAM_{100}$-*b*-$PNAT_{50}$ prepared with 10 vol% 1,4-dioxane at 1.0 M and 70 °C. B) $PNAM_{100}$-*b*-$PNAT_{50}$ prepared with 25 vol% 1,4-dioxane at 1.0 M and 70 °C.

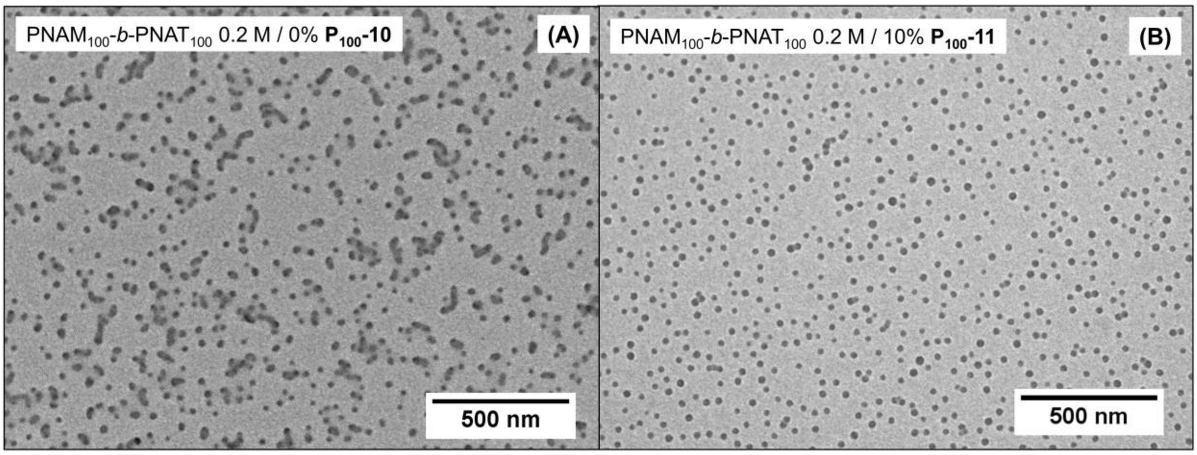


Figure S28. Exemplary dry-TEM images of the $PNAM_{100}$-*b*-$PNAT_{100}$ micelles after dialysis. A) Prepared with 0 vol% 1,4-dioxane at 0.2 M and 70 °C. B) Prepared with 10 vol% 1,4-dioxane at 0.2 M and 70 °C.

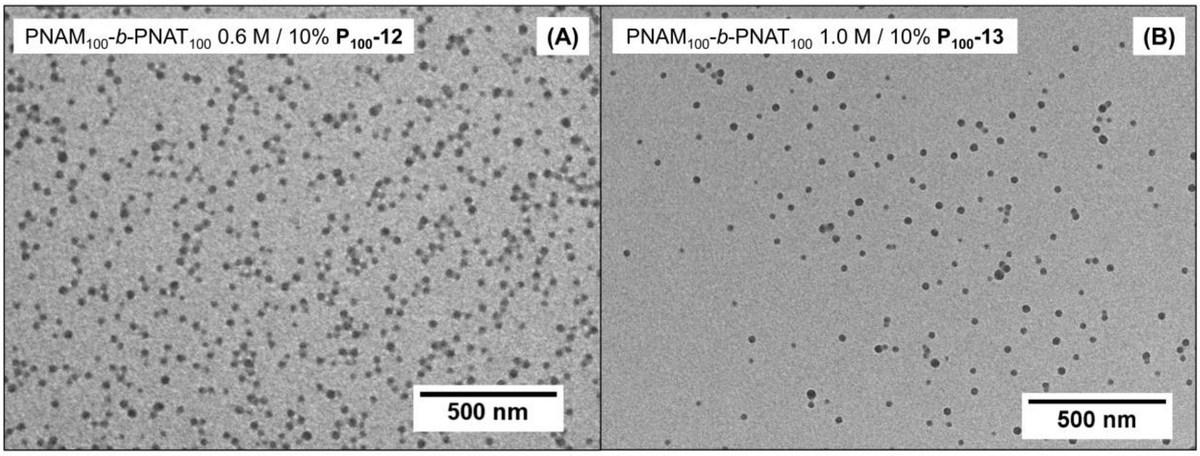


Figure S29. Exemplary dry-TEM images of $PNAM_{100}$-*b*-$PNAT_{100}$ micelles after dialysis. A) Prepared with 10 vol% 1,4-dioxane at 0.6 M and 70 °C. B) Prepared with 10 vol% 1,4-dioxane at 1.0 M and 70 °C.

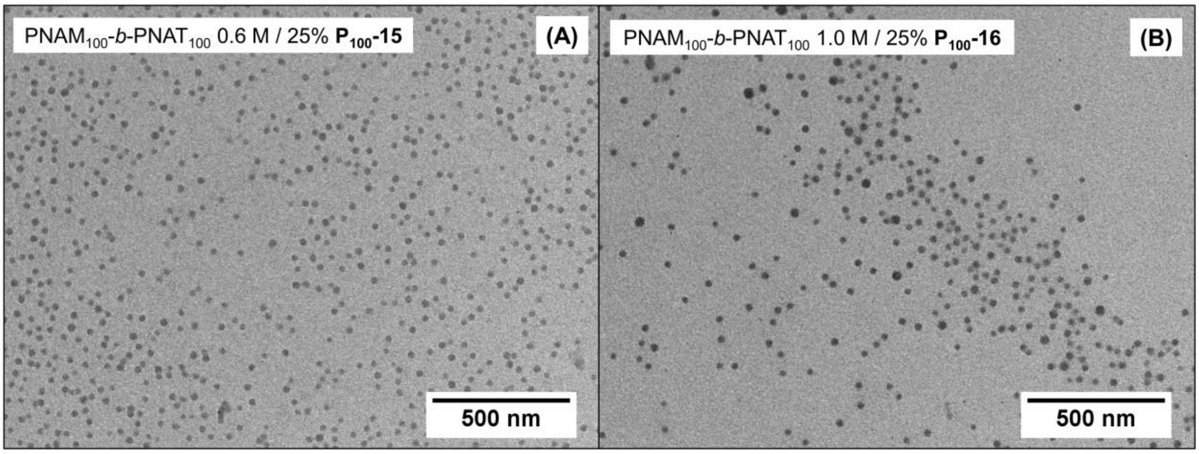


Figure S30. Exemplary dry-TEM images of $PNAM_{100}$-*b*-$PNAT_{100}$ micelles after dialysis. A) Prepared with 25 vol% 1,4-dioxane at 0.6 M and 70 °C. B) Prepared with 25 vol% 1,4-dioxane at 1.0 M and 70 °C.

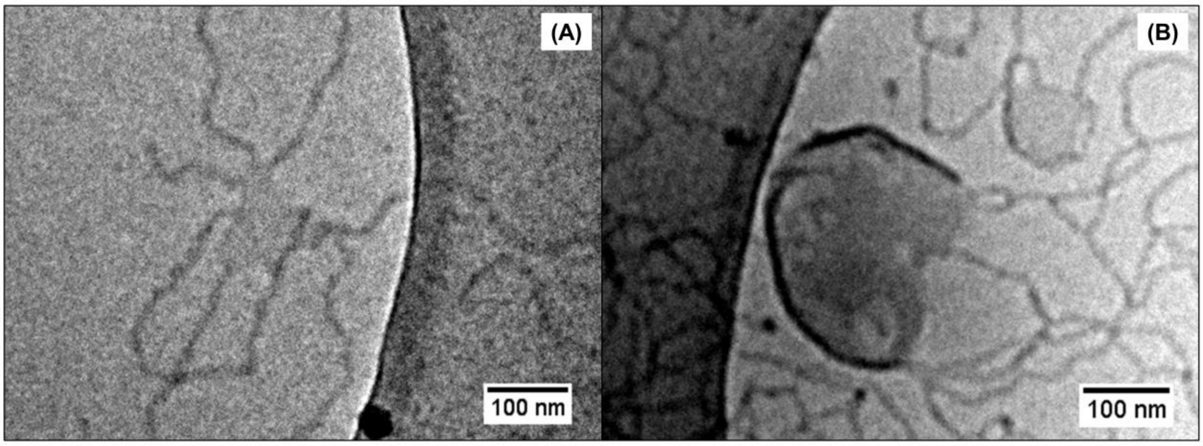


Figure S31. Exemplary cryo-TEM images of intermediate structures. A) Partially coalesced worms 'octopi-like', **P$_{25}$-24**. B) Half-closed vesicles, also called 'jellyfish', **P$_{25}$-27**.

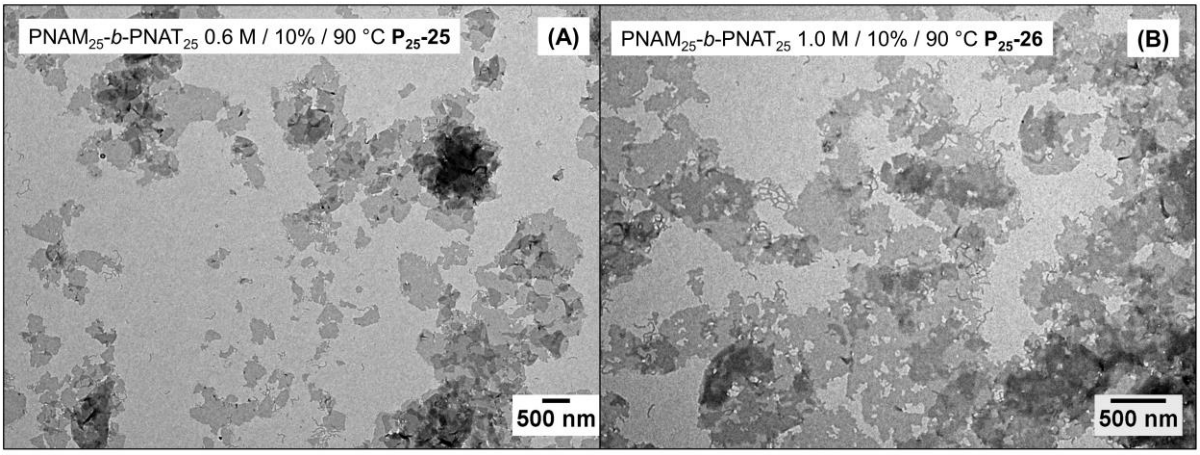


Figure S32. Exemplary dry-TEM images of PNAM$_{25}$-*b*-PNAT$_{25}$ nanostructures after dialysis. A) Prepared with 10 vol% 1,4-dioxane at 0.6 M and 90 °C. B) Prepared with 10 vol% 1,4-dioxane at 1.0 M and 90 °C.

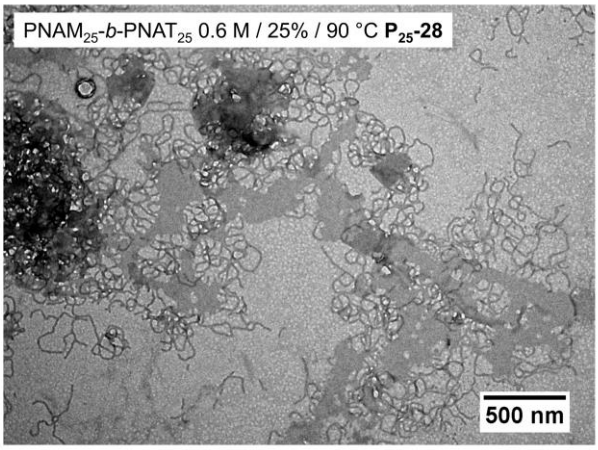


Figure S33. Exemplary dry-TEM images of PNAM$_{25}$-*b*-PNAT$_{25}$ nanostructures after dialysis, prepared with 25 vol% 1,4-dioxane at 0.6 M and 90 °C.

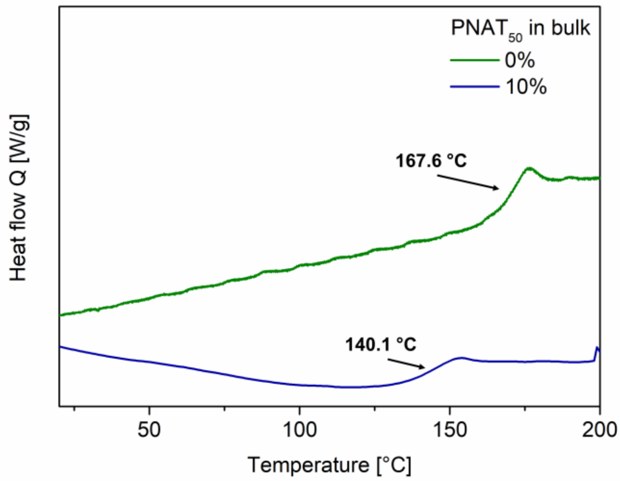


Figure S34. Comparison of the glass transition determined by DSC of pure $PNAT_{50}$ and $PNAT_{50}$ swelled in 1,4-dioxane.

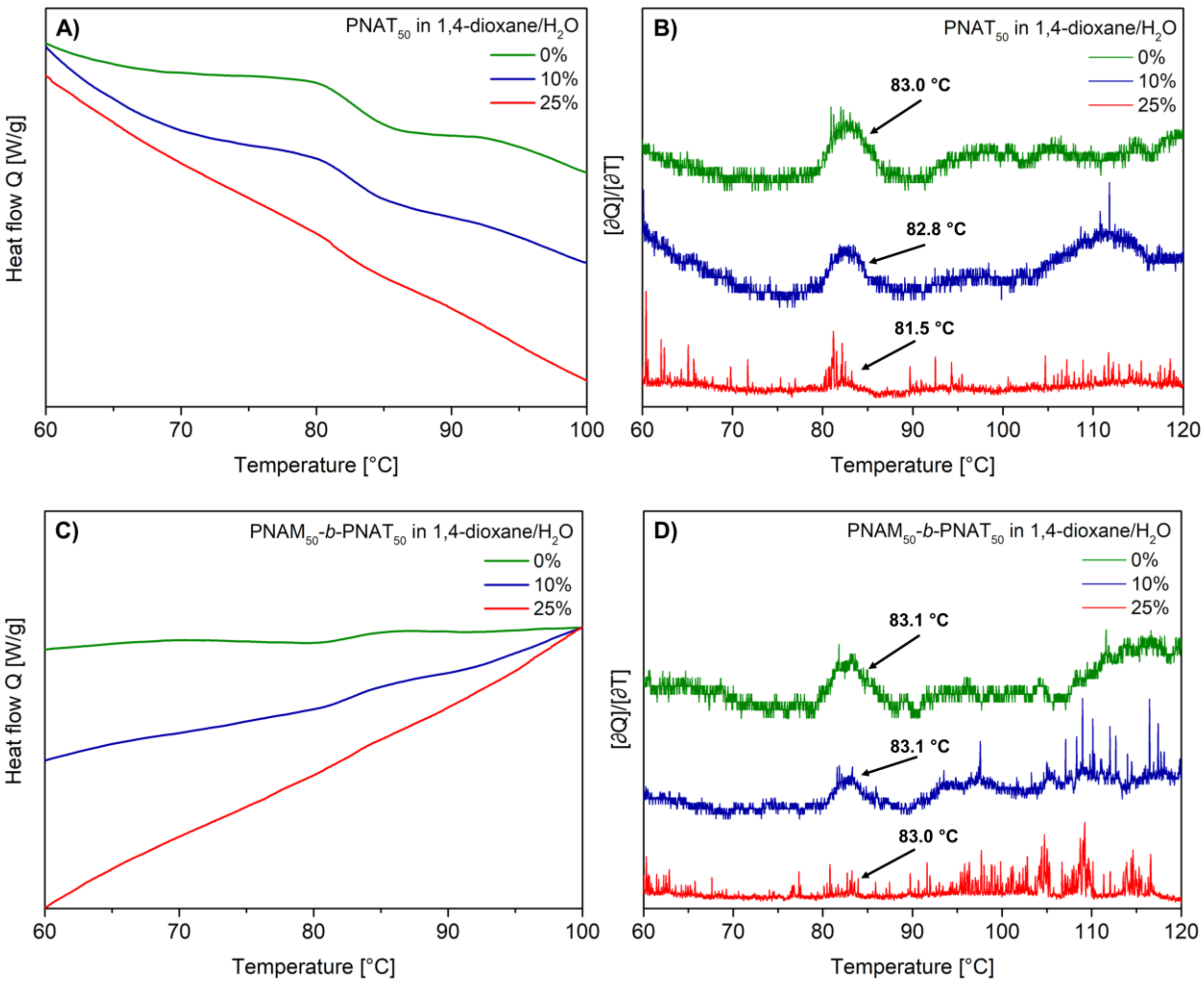


Figure S35. DSC analysis of aqueous polymer suspension with varying 1,4-dioxane content. A) Overlay of heating cycles of aqueous suspensions of $PNAT_{50}$ polymers (50 µL, 50 mg mL^{-1}). B) 1st derivative of the heat flow vs. temperature of aqueous suspensions of $PNAT_{50}$ polymers (50 µL, 50 mg mL^{-1}). C) Overlay of heating cycles of aqueous suspensions of

PNAM$_{50}$-*b*-NAT$_{50}$ micelles (50 µL, 200 mg mL^{-1}). D) 1st derivative of the heat flow vs. temperature of aqueous suspensions of PNAM$_{50}$-*b*-NAT$_{50}$ micelles (50 µL, 200 mg mL^{-1}).

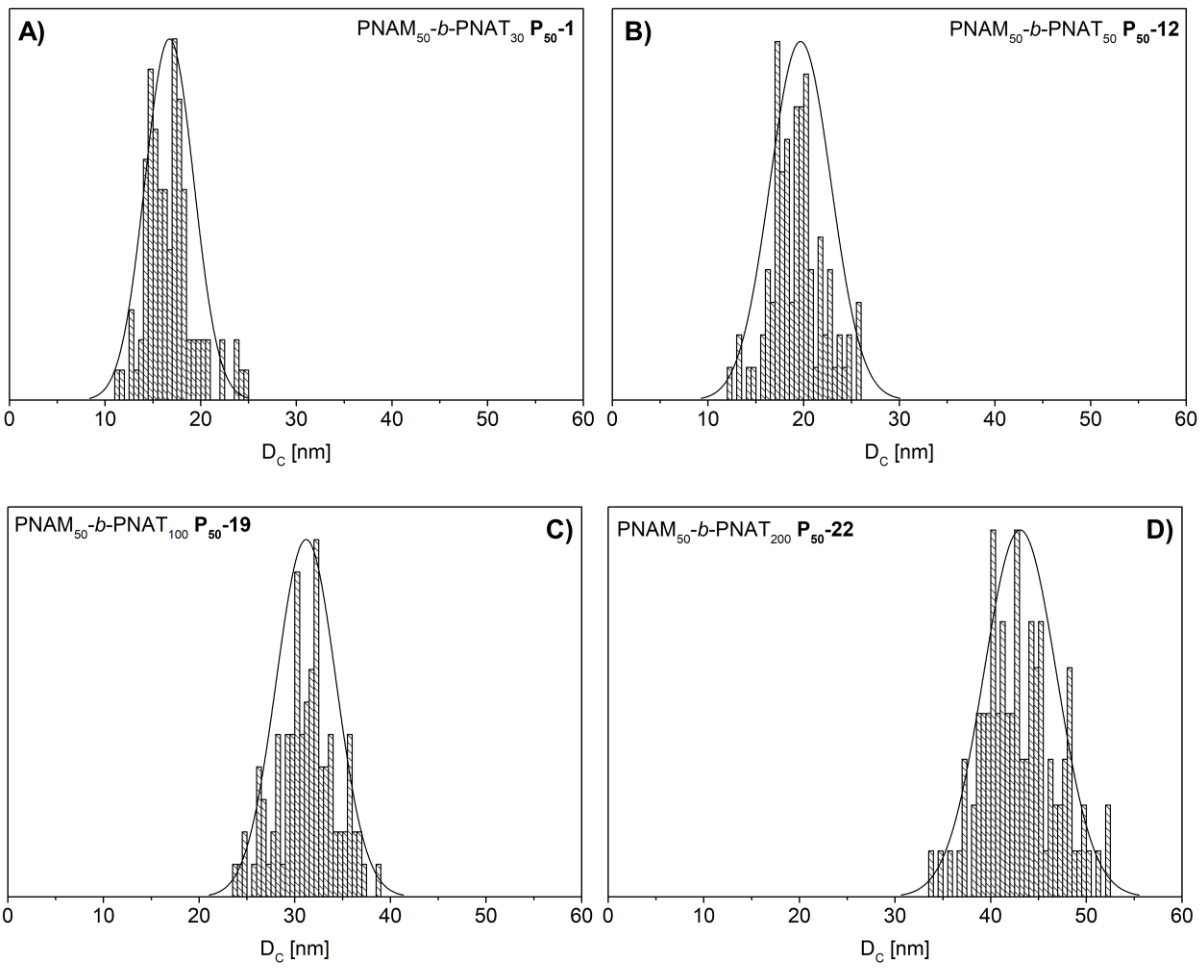


Figure S36. Size distribution of the core diameter determined by graphical analysis of ≥ 100 micelles in TEM images. A) Histogram of sample **P$_{50}$-1**. B) Histogram of sample **P$_{50}$-12**. C) Histogram of sample **P$_{50}$-19**. D) Histogram of sample **P$_{50}$-22**.

[1] F. H. Sobotta, M. Kuchenbrod, S. Hoeppener and J. C. Brendel, *Nanoscale*, **2020**, *12*, 20171-20176.