

# **PEGylated cationic nanoassemblies based on triblock copolymers to combine siRNA therapeutics with anticancer drugs**

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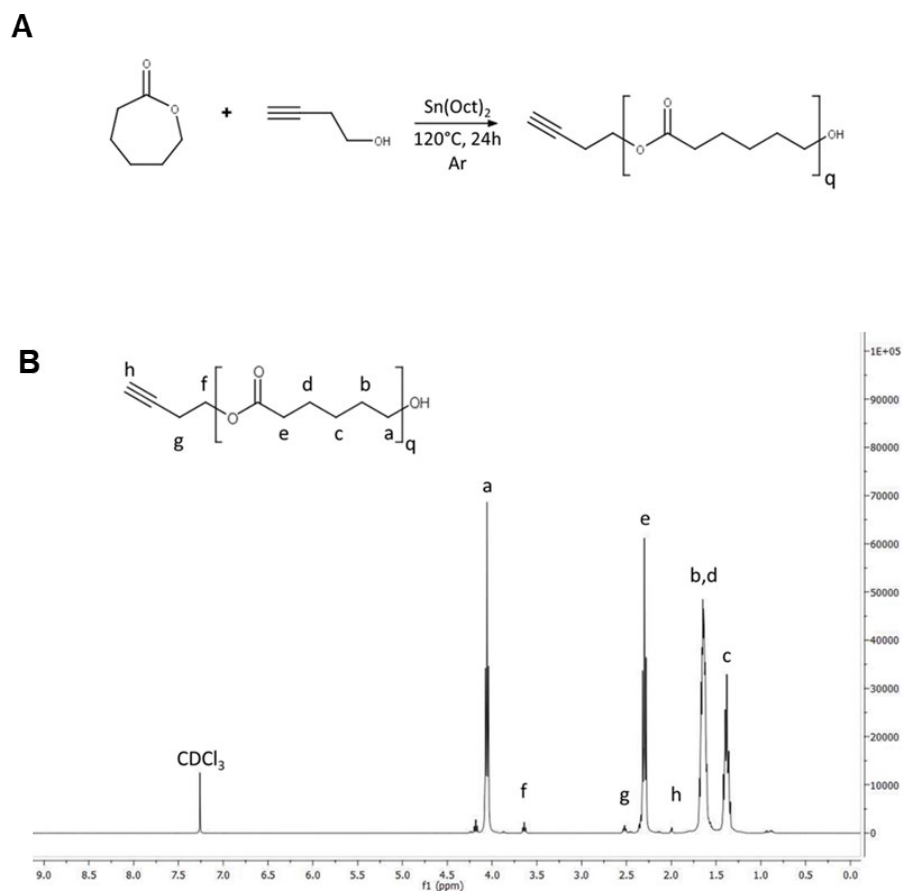
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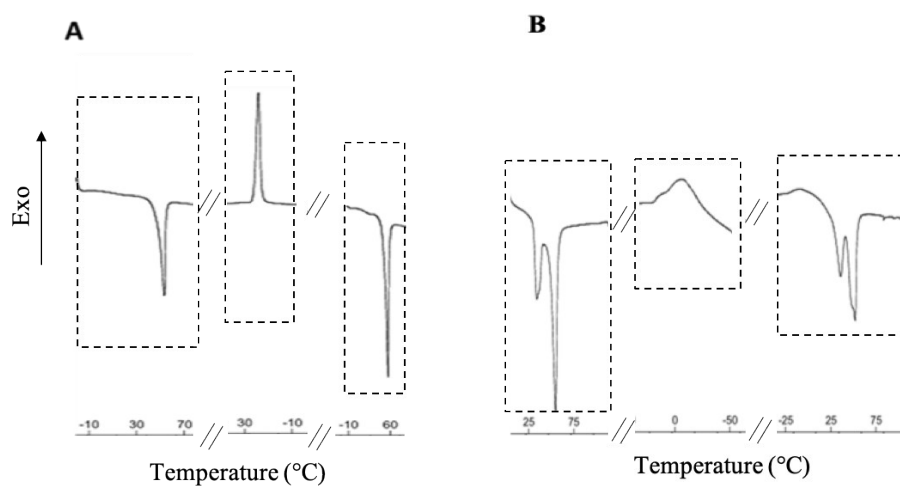
**Table S1.** Molecular weight (Da) and polydispersity index (PDI) of mPEG, mPEG-pDMAEMA-N<sub>3</sub> and butynyl-PCL

Polymer	Mn (theoretical)	Mn ( <sup>1</sup> H NMR)	Mn (GPC)	Mw (GPC)	PDI (GPC)
mPEG <sub>2k</sub>	-	-	1870	2240	1.20
mPEG <sub>5k</sub>	-	-	4980	5826	1.17
mPEG <sub>2k</sub> pDMAEMA <sub>5.6k</sub> -N <sub>3</sub>	6000*	5660*	7720	10036	1.32
mPEG <sub>5k</sub> pDMAEMA <sub>15k</sub> -N <sub>3</sub>	15000*	14934*	19853	24022	1.21
PCL <sub>750</sub>	750	762	753	895	1.18
PCL <sub>4k</sub>	4000	4161	4376	5443	1.24

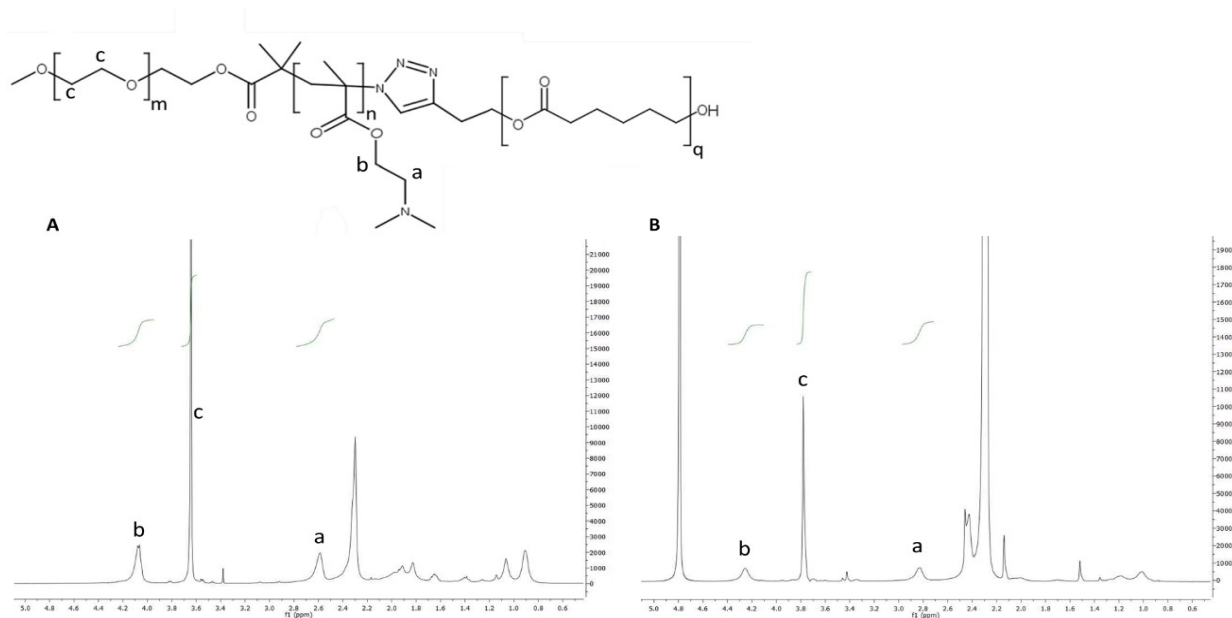
\* molecular weights refer to pDMAEMA block



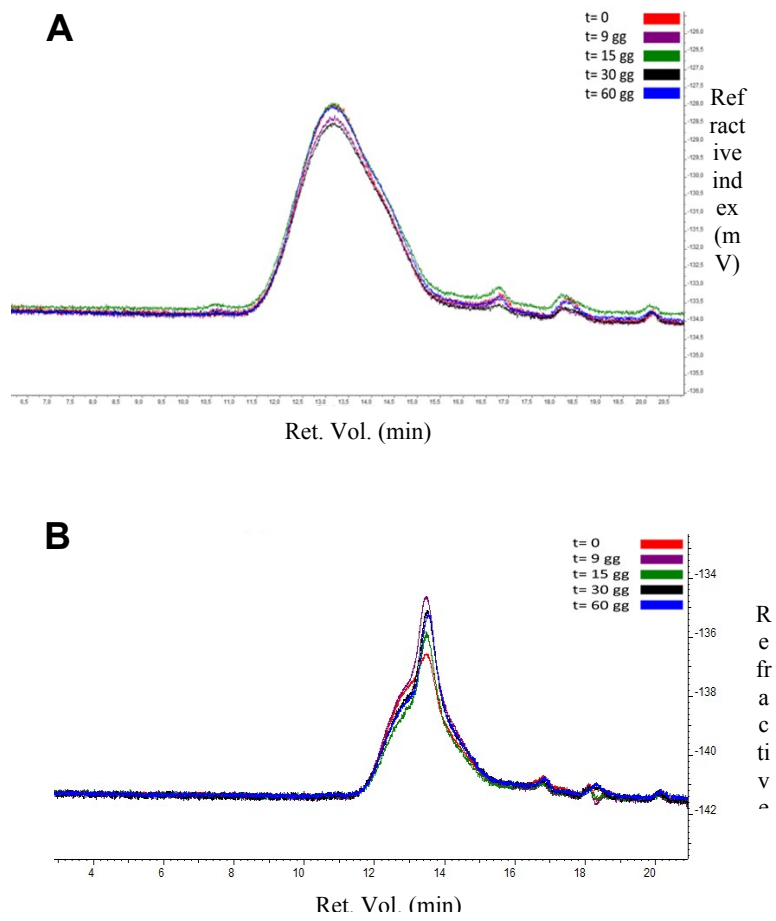
**Figure S1.** A) Polymerization of  $\epsilon$ -CL initiated by 3-butyn-1-ol); B) <sup>1</sup>H-NMR spectrum of butynyl-PCL<sub>4k</sub>



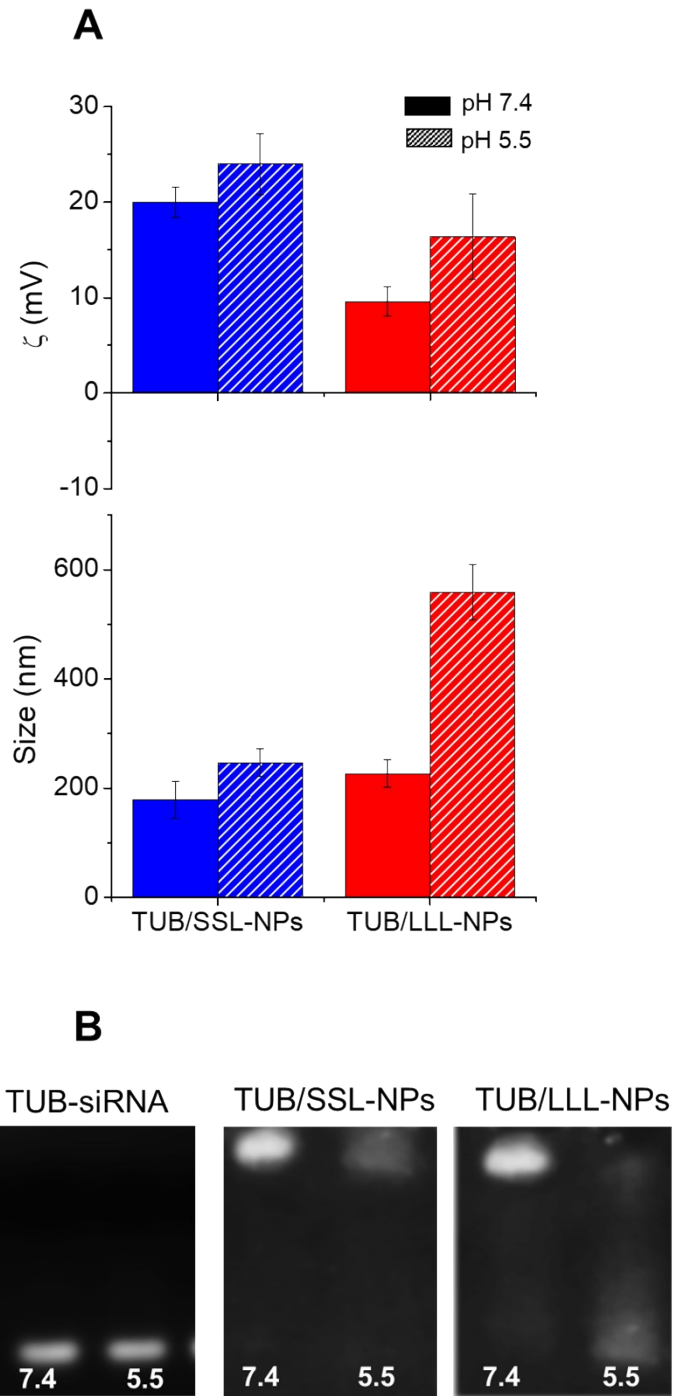
**Figure S2.** DSC thermograms of LLS (A) and SSL (B) copolymers showing first melting, crystallization from the melt and second melting peaks.



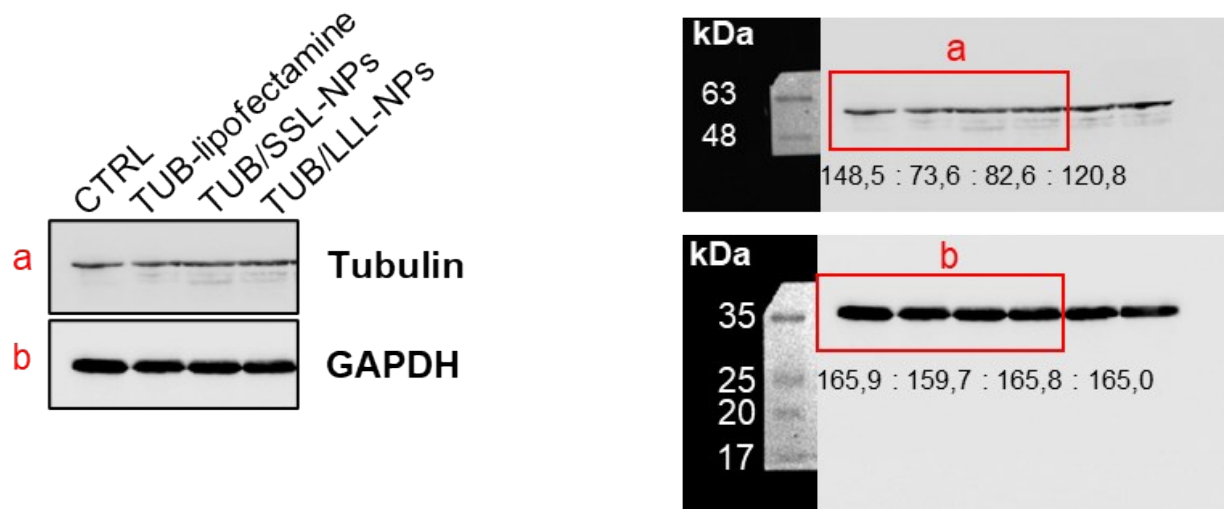
**Figure S3.**  $^1\text{H-NMR}$  spectrum of SSS-NPs in  $\text{CDCl}_3$  (A) and  $\text{D}_2\text{O}$  (B). In evidence signals relative to  $\text{O-CH}_2\text{-CH}_2\text{-N}$  (a) and  $\text{O-CH}_2\text{-CH}_2\text{-N}$  (b) protons of DMAEMA block, and  $\text{O-CH}_2\text{-CH}_2\text{-O}$  (c) protons of mPEG.



**Figure S4.** Hydrolytic degradation analyzed through gel permeation chromatography (refractive index detector) of SSL-NPs (A) and LLL-NPs (B).



**Figure S5.** Stability of siRNA/NP complexation in PBS at pH 7.4 and 5.5. (A) Size, polydispersity index and  $\zeta$  of NPs complexed with TUB-siRNA at N/P ratio 10. B) siRNA complexation as evaluated by the gel retardation assay.



**Figure S6.** Full length western blots of Figure 7 with antibodies against indicated proteins. GAPDH was used as loading control. Mean densitometric values of three independent experiments are reported.