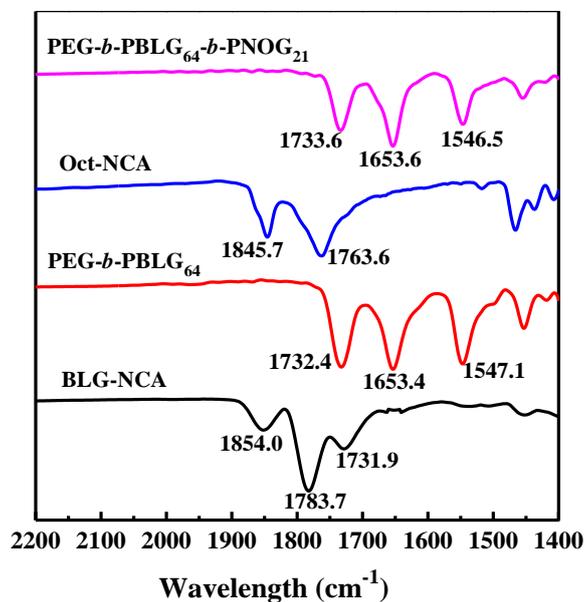
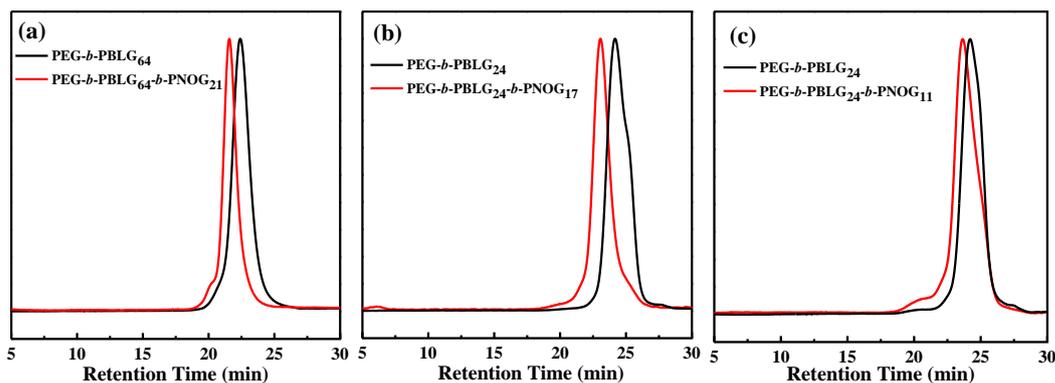


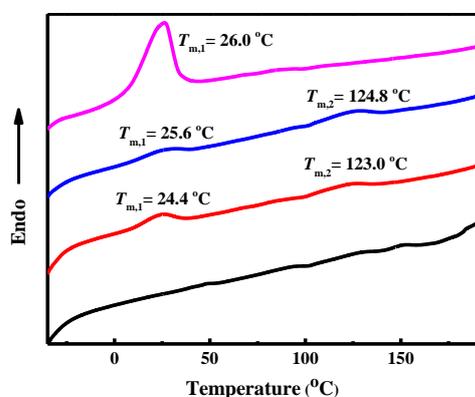
**Figure S1.**  $^1\text{H}$  NMR of (a) BLG-NCA (500 MHz,  $\text{CDCl}_3$ ,  $\delta$ , ppm): 1.86-2.12 (m, 2H), 2.52 (t, 2H), 4.47 (t, 2H), 5.09 (s, 2H), 7.31-7.36 (m, 5H) and (b) Oct-NNCA (500 MHz,  $\text{CDCl}_3$ ,  $\delta$ , ppm): 0.90 (t, 3H), 1.28-1.32 (m, 10H), 1.60-1.64 (m, 2H), 3.41 (t, 2H), 4.10 (s, 2H). \* indicates  $\text{CDCl}_3$ .



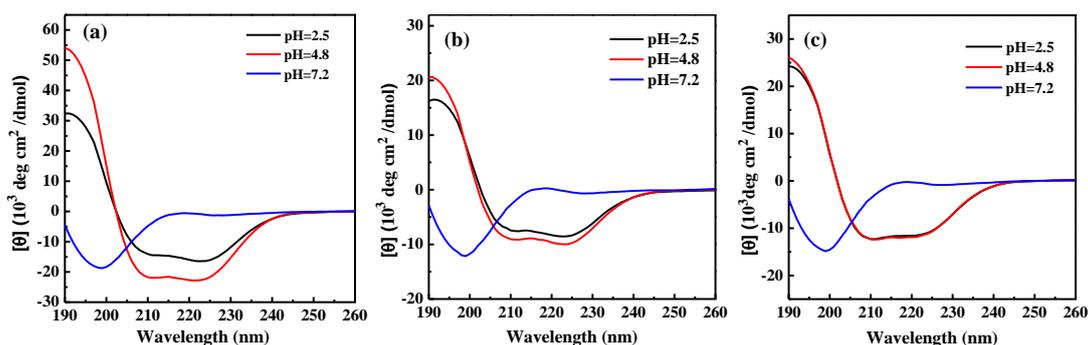
**Figure S2.** FTIR spectrums of BLG-NCA, Oct-NNCA, PEG-*b*-PBLG and PEG-*b*-PBLG-*b*-PNOG.



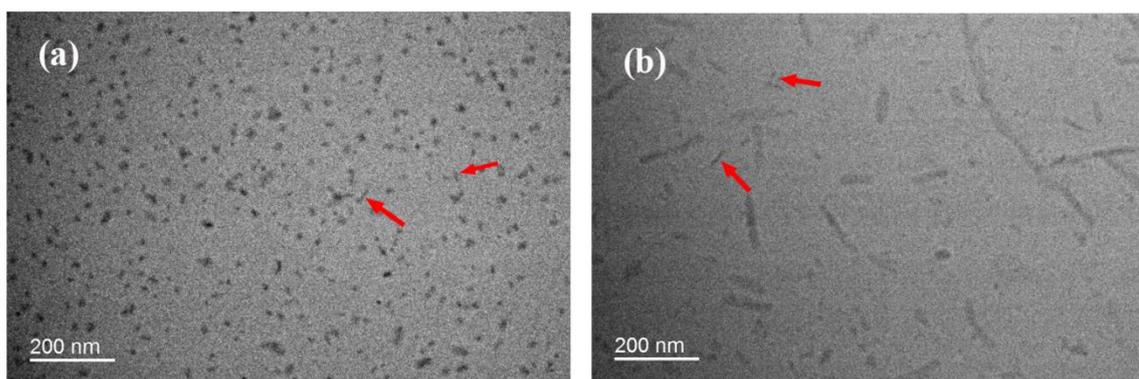
**Figure S3.** Representative GPC chromatograms of (a) PEG-*b*-PBLG<sub>64</sub> and PEG-*b*-PBLG<sub>64</sub>-*b*-PNOG<sub>21</sub>; (b) PEG-*b*-PBLG<sub>24</sub> and PEG-*b*-PBLG<sub>24</sub>-*b*-PNOG<sub>17</sub>; (c) PEG-*b*-PBLG<sub>24</sub> and PEG-*b*-PBLG<sub>24</sub>-*b*-PNOG<sub>11</sub>.



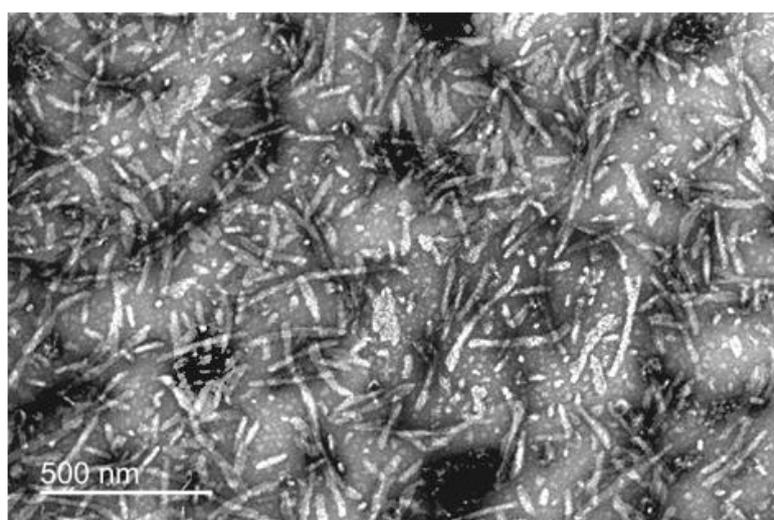
**Figure S4.** The DSC thermograms of all the triblock copolymers: black curve represents PEG-*b*-PGA<sub>64</sub>-*b*-PNOG<sub>21</sub>; pink curve represents PEG-*b*-PGA<sub>24</sub>-*b*-PNOG<sub>11</sub>; blue curve represents PEG-*b*-PGA<sub>24</sub>-*b*-PNOG<sub>17</sub>; red curve represents PEG-*b*-PGA<sub>42</sub>-*b*-PNOG<sub>17</sub>.



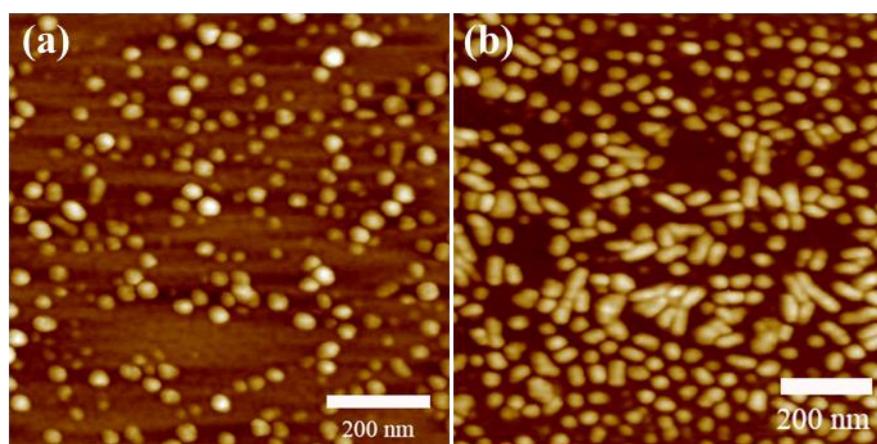
**Figure S5.** CD spectrum of (a) PEG-*b*-PGA<sub>64</sub>-*b*-PNOG<sub>21</sub>; (b) PEG-*b*-PGA<sub>24</sub>-*b*-PNOG<sub>17</sub>; (c) PEG-*b*-PGA<sub>24</sub>-*b*-PNOG<sub>11</sub> at different pH.



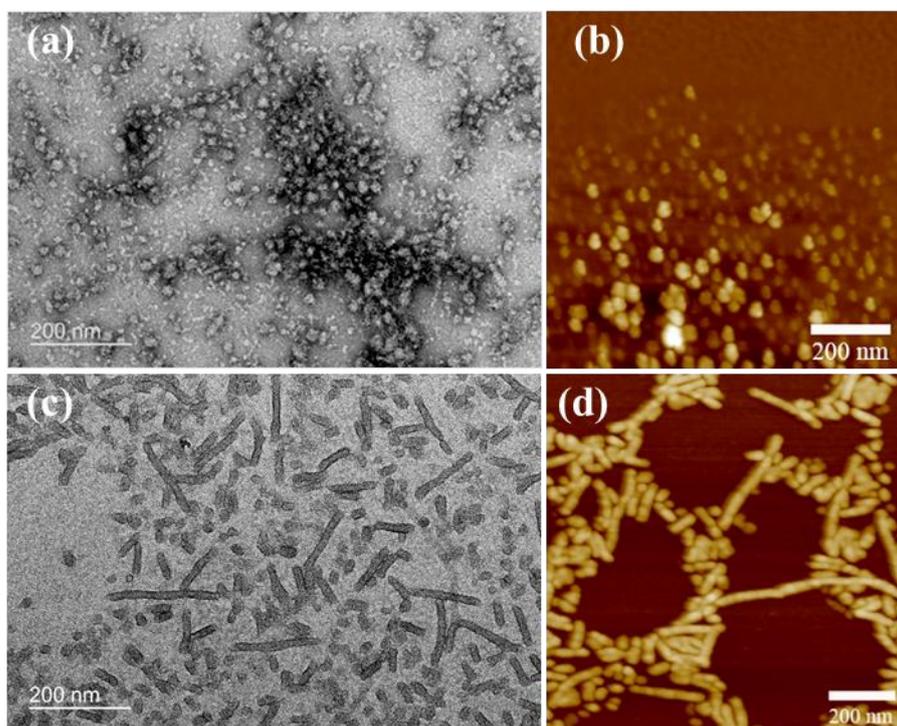
**Figure S6.** Cryo-TEM micrographs of (freshly prepared) 0.2 wt % PEG-*b*-PGA<sub>42</sub>-*b*-PNOG<sub>17</sub> in aqueous solution at (a) pH 7.2 and (b) pH 4.8. The arrows indicate the self-assemblies that lie orthogonal to the plane.



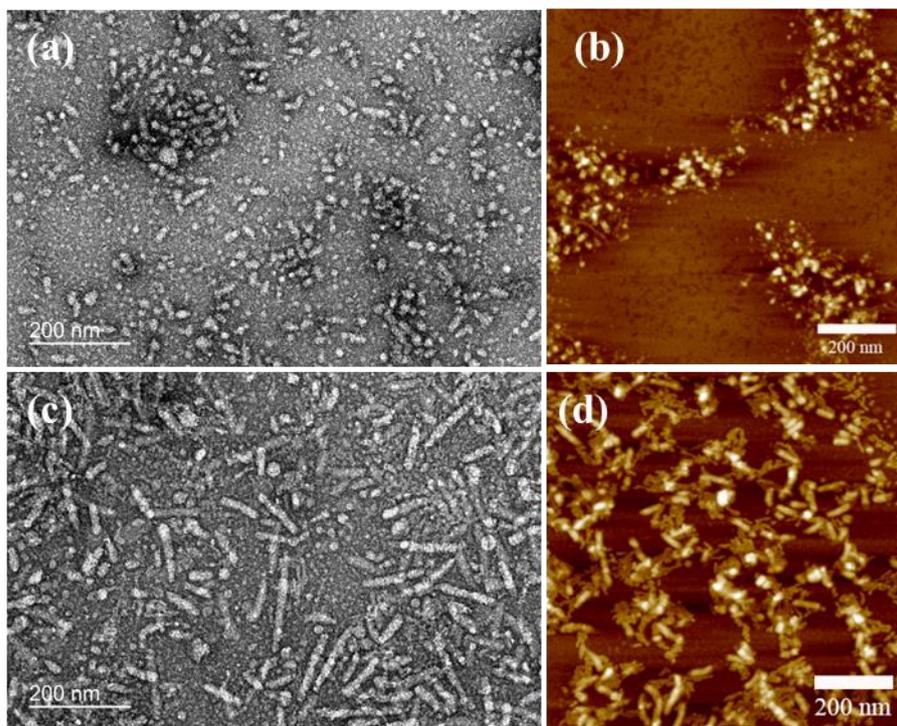
**Figure S7.** TEM image of PEG-*b*-PGA<sub>42</sub>-*b*-PNOG<sub>17</sub> at pH 2.5 at concentration of 2 mg/mL.



**Figure S8.** AFM image of (a) PEG-*b*-PGA<sub>64</sub>-*b*-PNOG<sub>21</sub> at pH 4.8 and (b) PEG-*b*-PGA<sub>24</sub>-*b*-PNOG<sub>17</sub> at pH 7.2 at concentration of 2 mg/mL.



**Figure S9.** (a) TEM image and (b) AFM image of PEG-*b*-PGA<sub>64</sub>-*b*-PNOG<sub>21</sub> at pH 7.2 at concentration of 2 mg/mL. (c) TEM image and (d) AFM image of PEG-*b*-PGA<sub>24</sub>-*b*-PNOG<sub>17</sub> at pH 4.8 at concentration of 2 mg/mL.



**Figure S10.** TEM image of PEG-*b*-PGA<sub>24</sub>-*b*-PNOG<sub>11</sub> at (a) pH 7.2 and (c) pH 4.8 at concentration of 2 mg/mL; AFM image of PEG-*b*-PGA<sub>24</sub>-*b*-PNOG<sub>11</sub> at (b) pH 7.2 and (d) pH 4.8 at concentration of 2 mg/mL.

**Table S1.** The characteristics of the assemblies of all the triblock copolymers at different pH.

Samples	Structure Type		Height (nm)	
	pH 7.2	pH 4.8	pH 7.2	pH 4.8
PEG- <i>b</i> -PBLG <sub>64</sub> - <i>b</i> -PNOG <sub>21</sub>	disk	disk	5.2±0.4	9.8±0.6
PEG- <i>b</i> -PBLG <sub>42</sub> - <i>b</i> -PNOG <sub>17</sub>	disk	sheet	5.9±0.4	10.2±0.4
PEG- <i>b</i> -PBLG <sub>24</sub> - <i>b</i> -PNOG <sub>17</sub>	sheet	sheet	9.2±0.6	9.8±0.6
PEG- <i>b</i> -PBLG <sub>24</sub> - <i>b</i> -PNOG <sub>11</sub>	disk	sheet	6.0±0.3	8.0±0.5

\*For statistical analysis, ~ 40 nanodisks or nanosheets were traced to determine the size.