

**Supporting Information**

**Self-Assembly of 2D Gold Nanoparticle Superlattice in a Polymer Vesicle  
Layer Driven by Hydrophobic Interaction**

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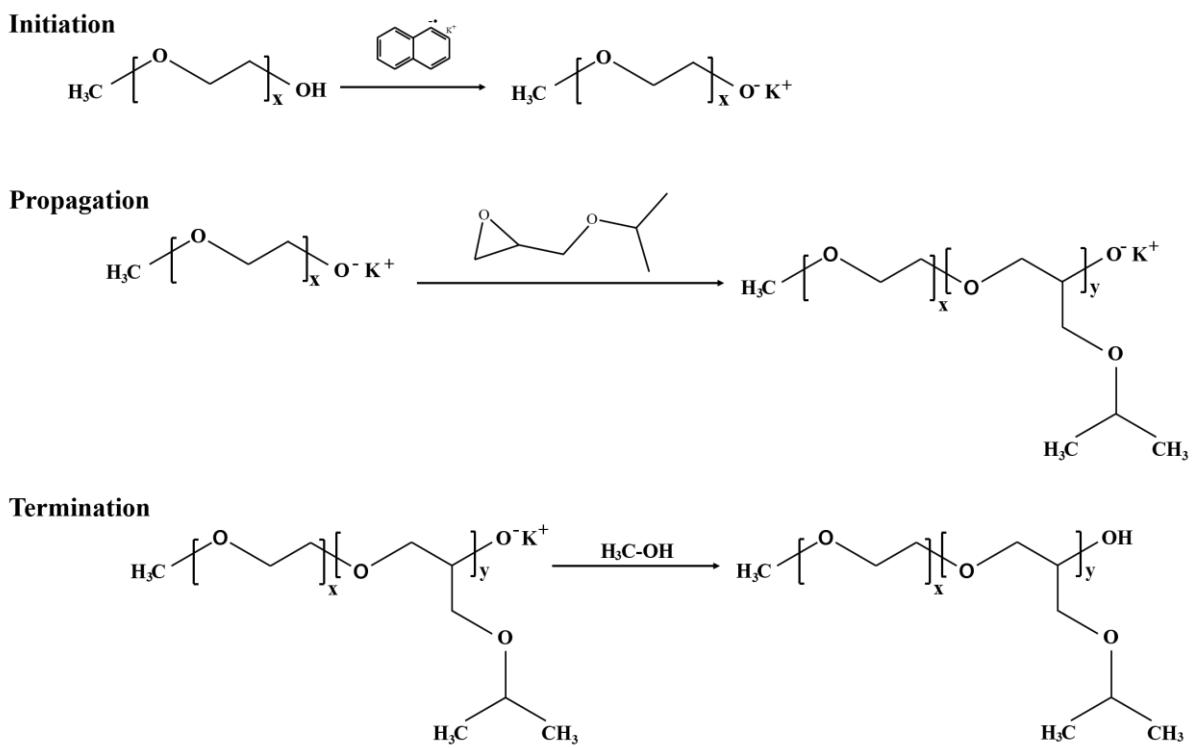
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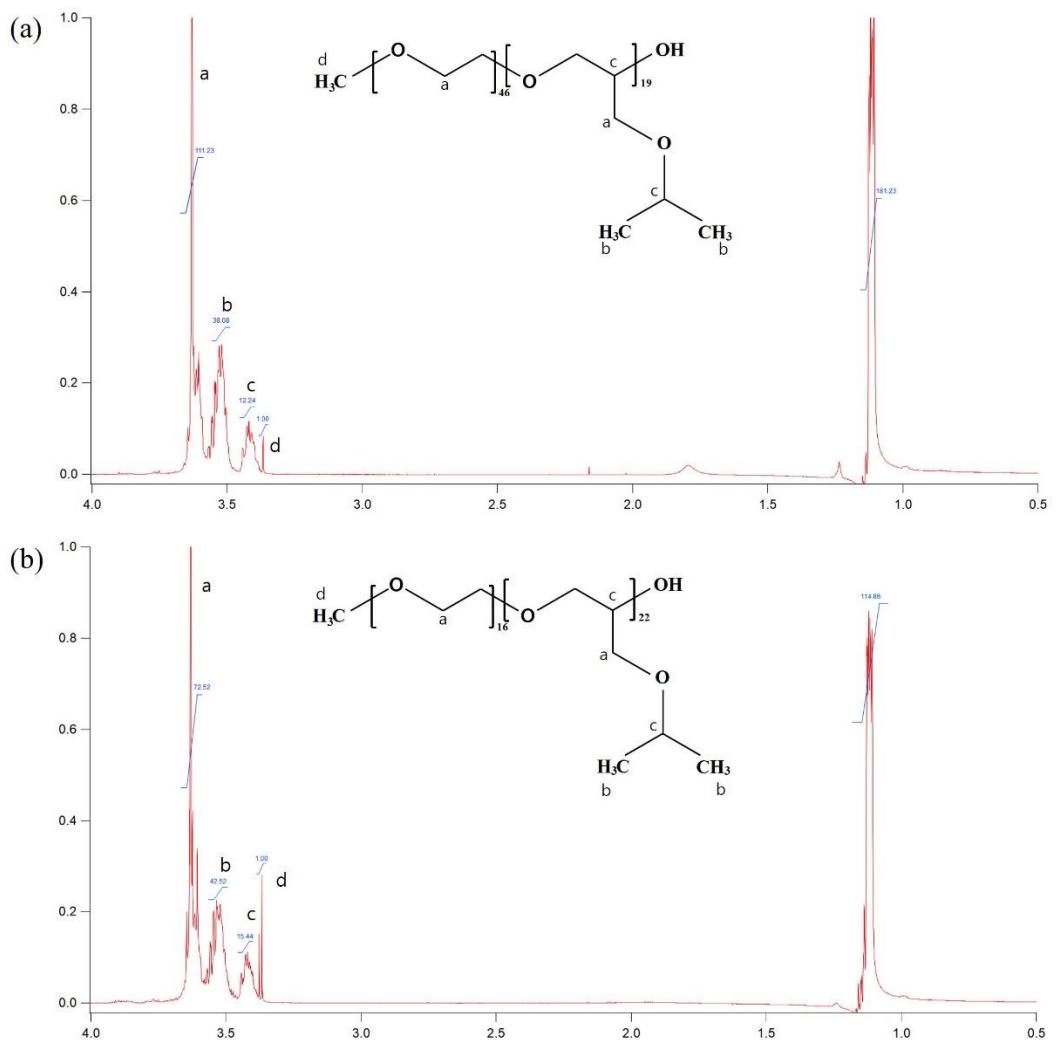
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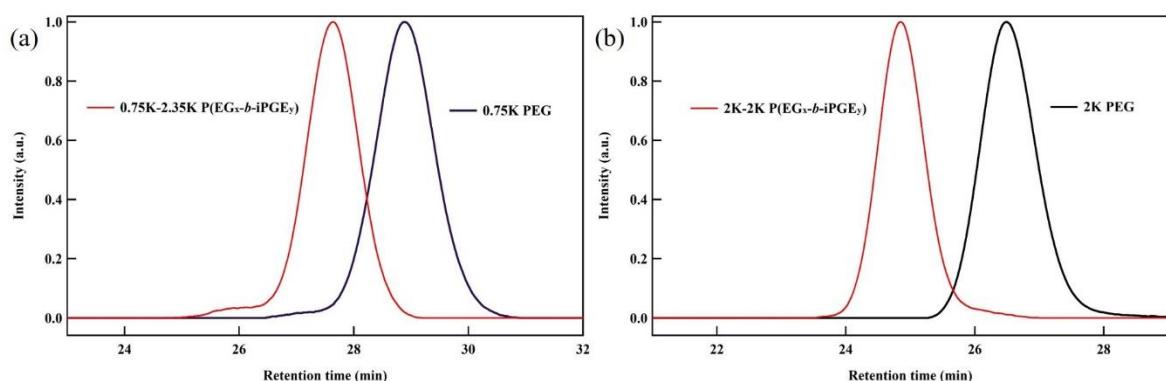
Figures S1 to S4  
Tables S1 to S2



**Figure S1.** Synthesis of the P(EG<sub>x</sub>-b-iPGE<sub>y</sub>) diblock copolymer.

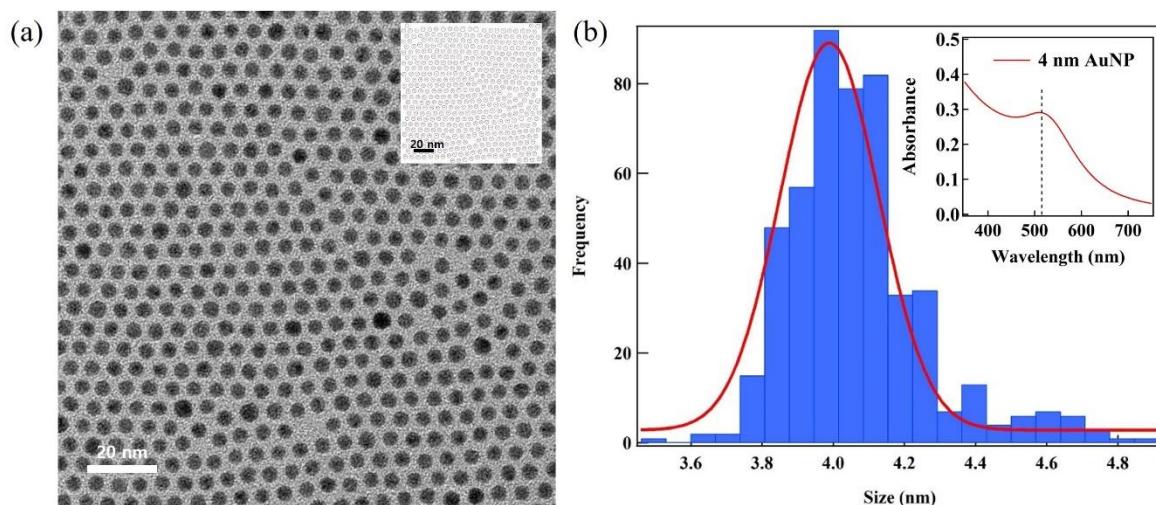


**Figure S2.** <sup>1</sup>H-NMR spectra of the P(EG<sub>x</sub>-*b*-iPGE<sub>y</sub>) diblock copolymer. (a) 0.75K-2.35K P(EG<sub>x</sub>-*b*-iPGE<sub>y</sub>) diblock copolymer and (b) 2K-2K P(EG<sub>x</sub>-*b*-iPGE<sub>y</sub>) diblock copolymer.



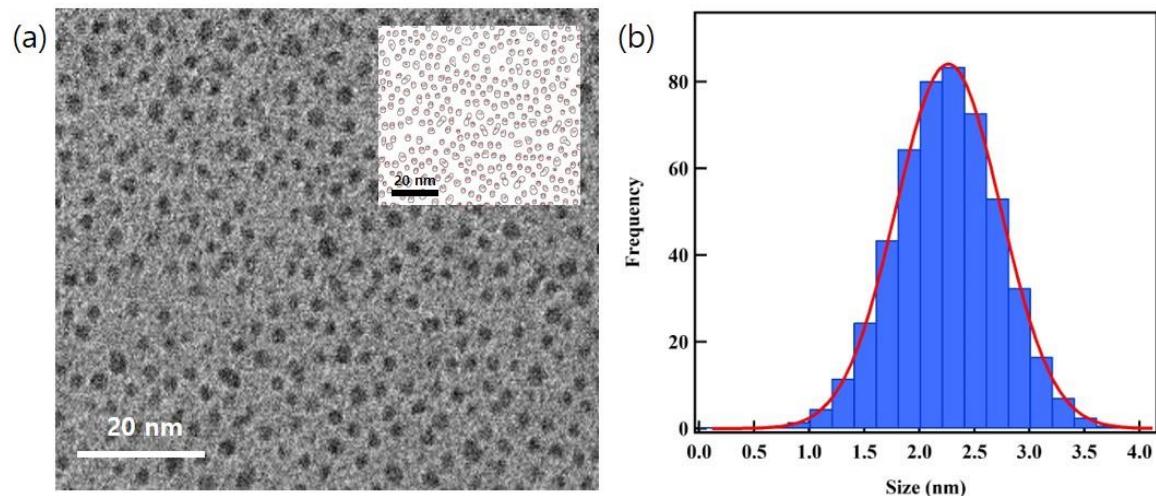
**Figure S3.** GPC traces of (a) 0.75K PEG and 0.75K-2.35K P(EG<sub>x</sub>-*b*-iPGE<sub>y</sub>) diblock copolymer and (b) 2K PEG and 2K-2K P(EG<sub>x</sub>-*b*-iPGE<sub>y</sub>) diblock copolymer in dimethylformamide

(DMF)



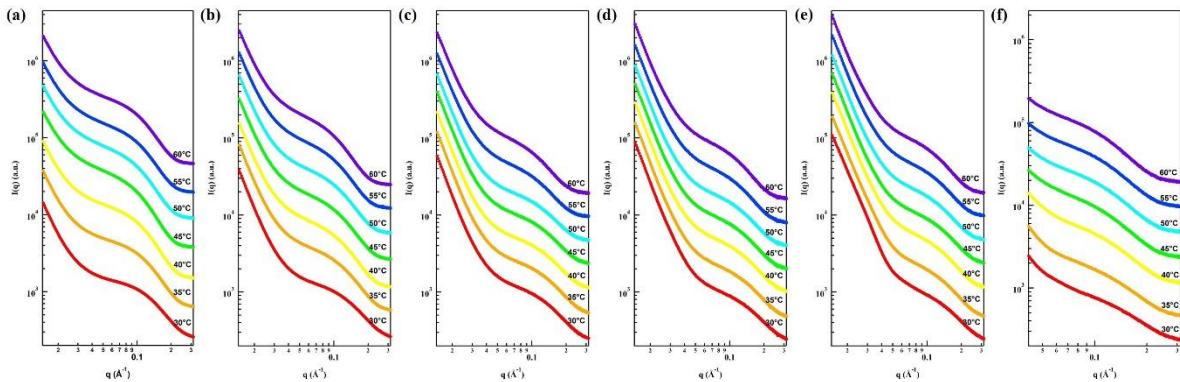
**Figure S4. Transmission electron microscopy (TEM) analysis of the synthesized AuNPs.**

(a) TEM image (The average size is 4.00 nm ( $\pm 0.20$  nm), and the polydispersity is 0.05.) and (b) size distribution of the gold nanoparticles (AuNPs). The inset in (b) shows the ultraviolet-visible (UV-vis) spectrum of the pristine AuNP.

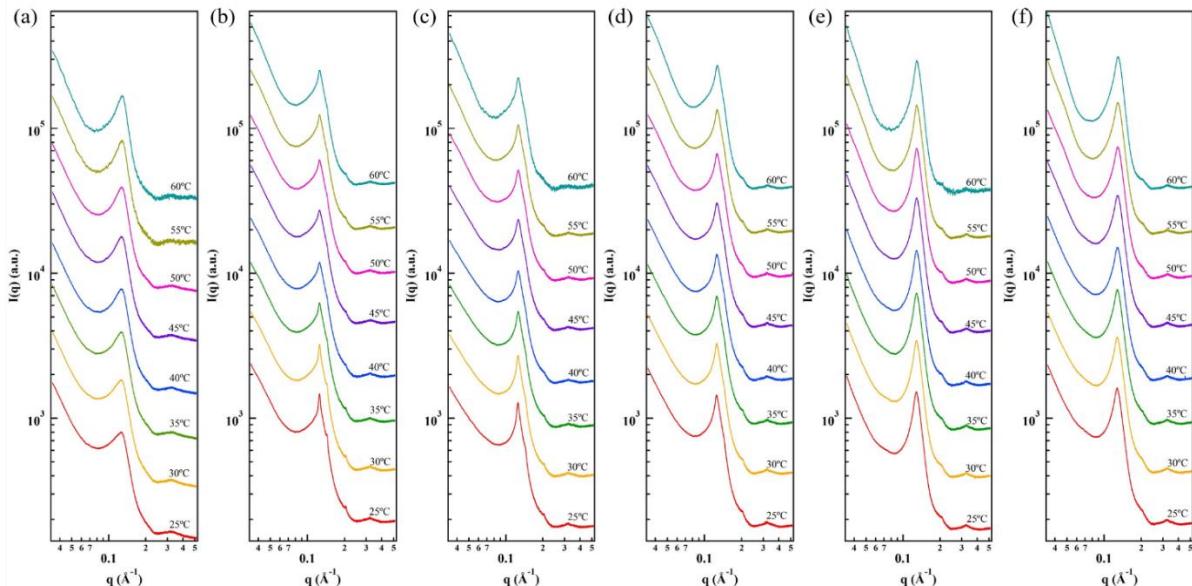


**Figure S5. Transmission electron microscopy (TEM) analysis of the synthesized AuNPs.**

(a) TEM image (The average size is 2.15 nm ( $\pm 0.47$  nm), and the polydispersity is 0.22.) and (b) size distribution of the gold nanoparticles (AuNPs).



**Figure S6.** The SAXS experiments on various temperatures of the 2.15 nm AuNP-P( $\text{EG}_x$ -*b*- $\text{iPGE}_y$ ) mixtures. (a) is the 2.15 nm AuNPs-P( $\text{EG}_{46}$ -*b*- $\text{iPGE}_{19}$ ) / P( $\text{EG}_{16}$ -*b*- $\text{iPGE}_{22}$ ) (P(46-19) 0.1 % / P(16-22) 0.0 %) mixture. (b) is the 2.15 nm AuNPs-P(46-19) 0.1 % / P(16-22) 0.1 % mixture. (c) is the 2.15 nm AuNPs-P(46-19) 0.1 % / P(16-22) 0.2 % mixture. (d) is the 2.15 nm AuNPs-P(46-19) 0.1 % / P(16-22) 0.3 % mixture. (e) is the 2.15 nm AuNPs-P(46-19) 0.1 % / P(16-22) 0.4 % mixture. (f) is the 2.15 nm AuNPs-P(46-19) 0.1 % / P(16-22) 0.5 % mixture.



**Figure S7.** The SAXS experiments on various temperatures of the AuNP-P( $\text{EG}_x$ -*b*- $\text{iPGE}_y$ ) mixtures. (a) is the AuNPs-P( $\text{EG}_{46}$ -*b*- $\text{iPGE}_{19}$ ) / P( $\text{EG}_{16}$ -*b*- $\text{iPGE}_{22}$ ) (P(46-19) 0.1 % / P(16-22) 0.0 %) mixture. (b) is the AuNPs-P(46-19) 0.1 % / P(16-22) 0.1 % mixture. (c) is the AuNPs-P(46-19) 0.1 % / P(16-22) 0.2 % mixture. (d) is the AuNPs-P(46-19) 0.1 % / P(16-22) 0.3 % mixture. (e) is the AuNPs-P(46-19) 0.1 % / P(16-22) 0.4 % mixture. (f) is the AuNPs-P(46-19) 0.1 % / P(16-22) 0.5 % mixture.

**Table S1.** Scattering length densities

Molecule	Scattering Length Density (SLD) ( $\times 10^{-6} \text{ \AA}^{-2}$ )
Poly(Ethylene Oxide) (PEO)	<b>0.419</b>
iso-Propyl Glycidyl Ether (iPGE)	<b>0.317</b>
Gold Nanoparticle (AuNP)	<b>4.667</b>
D <sub>2</sub> O	<b>6.335</b>

**Table S2.** The information of the vesicle core and layer calculated by model fitting. The vesicle formed by (a) P(46-19) / P(16-22) mixture, and (b) AuNP- P(46-19) / P(16-22) mixture.

(a)	P(46-19) / P(16-22) mixture		
Polymer concentration	Core diameter (nm)	Thickness of the polymer outer layer (nm)	Thickness of the polymer inner layer (nm)
P(46-19) 0.1 % / P(16-22) 0.0 %	-	-	-
P(46-19) 0.1 % / P(16-22) 0.1 %	133.1	8.0	3.2
P(46-19) 0.1 % / P(16-22) 0.2 %	145.9	6.9	3.1
P(46-19) 0.1 % / P(16-22) 0.3 %	149.6	5.7	2.9
P(46-19) 0.1 % / P(16-22) 0.4 %	151.7	5.2	2.8
P(46-19) 0.1 % / P(16-22) 0.5 %	151.7	5.2	2.8

AuNPs-P(46-19) / P(16-22) mixture					
With AuNPs (dominated > 97 %)				Without AuNPs	
Polymer concentration	Core diameter (nm)	Thickness of the polymer outer layer (nm)	Thickness of the gold inner layer (nm)	Core diameter (nm)	Thickness of the polymer bilayer (nm)
P(46-19) 0.1 % / P(16-22) 0.0 %	-	-	-	-	-
P(46-19) 0.1 % / P(16-22) 0.1 %	132.7	5.1	3.6	129.2	10.1
P(46-19) 0.1 % / P(16-22) 0.2 %	152.0	4.9	3.6	143.0	9.8
P(46-19) 0.1 % / P(16-22) 0.3 %	155.9	3.7	3.1	145.9	9.2
P(46-19) 0.1 % / P(16-22) 0.4 %	154.5	3.7	3.0	148.4	8.5
P(46-19) 0.1 % / P(16-22) 0.5 %	167.6	3.4	2.8	150.6	8.3