SUPPORTING INFORMATION

O-nitrobenzyl-*alt*-(phenylethynyl)benzene copolymer-based nanoaggregates with highly efficient two-photon-triggered

degradable property via a FRET process

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Figure S1. ¹H NMR of the monomer 3, 3'-((2, 5-diethynyl-1, 4-phenylene) bis (oxy)) bis (propan-1-ol). CDCl₃ was used as the solvent.



Figure S2. ¹H NMR of the monomer (2-nitro-1, 3-phenylene) bis (methylene) bis (4-bromobenzoate). CDCl₃ was used as the solvent.



Figure S3. ¹H NMR of the monomer 1, 3-phenylenebis (methylene) bis (4-bromobenzoate). CDCl₃ was used as the solvent.



Figure S4. ¹³C NMR of the monomer 3, 3'-((2, 5-diethynyl-1, 4-phenylene) bis (oxy)) bis (propan-1-ol). CDCl₃ was used as the solvent.



Figure S5. ¹³C NMR of the monomer (2-nitro-1, 3-phenylene) bis (methylene) bis (4-bromobenzoate). CDCl₃ was used as the solvent.

the solvent.

Figure S7. ¹H NMR spectra of the photodegradable polymer Poly(ONB-alt-PEB), CDCl₃ was used as the solvent

Figure S8. GPC traces of the Poly(ONB-alt-PEB) (A), and Poly (ONB-alt-PEB)-g-PEG (B)

Figure S9. Open aperture Z-scan data of poly(ONB-*alt*-PEB)-*g*-PEG for the TPA cross sections obtained by using 800 nm femtosecond pulses.

	Mn	PDI
no irradiation	14877	1.72
380 nm irradiation for 1 h	7840	2.2
800 nm irradiation for 1 h	7499	2.01

Table S1. M_n and PDI of Poly (ONB-alt-PEB)-g-PEG before and after 380 nm and 800 nm irradiation for 1 h.

Figure S10. ¹H NMR spectra of the Poly(OB-*alt*-PEB)-*g*-PEG before (black) and after 380 nm (blue) and 800 nm (red) irradiation for 1 h., CDCl₃ was used as the solvent.

	Mn	PDI
no irradiation	21059	1.8
380 nm irradiation for 1 h	22558	1.5
800 nm irradiation for 1 h	20102	1.75

Table S2. M_n and PDI of Poly (OB-*alt*-PEB)-*g*-PEG before and after 380 nm and 800 nm irradiation for 1 h.