Electronic Supplementary Material (ESI) for Materials Horizons.

Photothermal effect by NIR-responsive excretable ultrasmall-in-nano architectures

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Figure S1

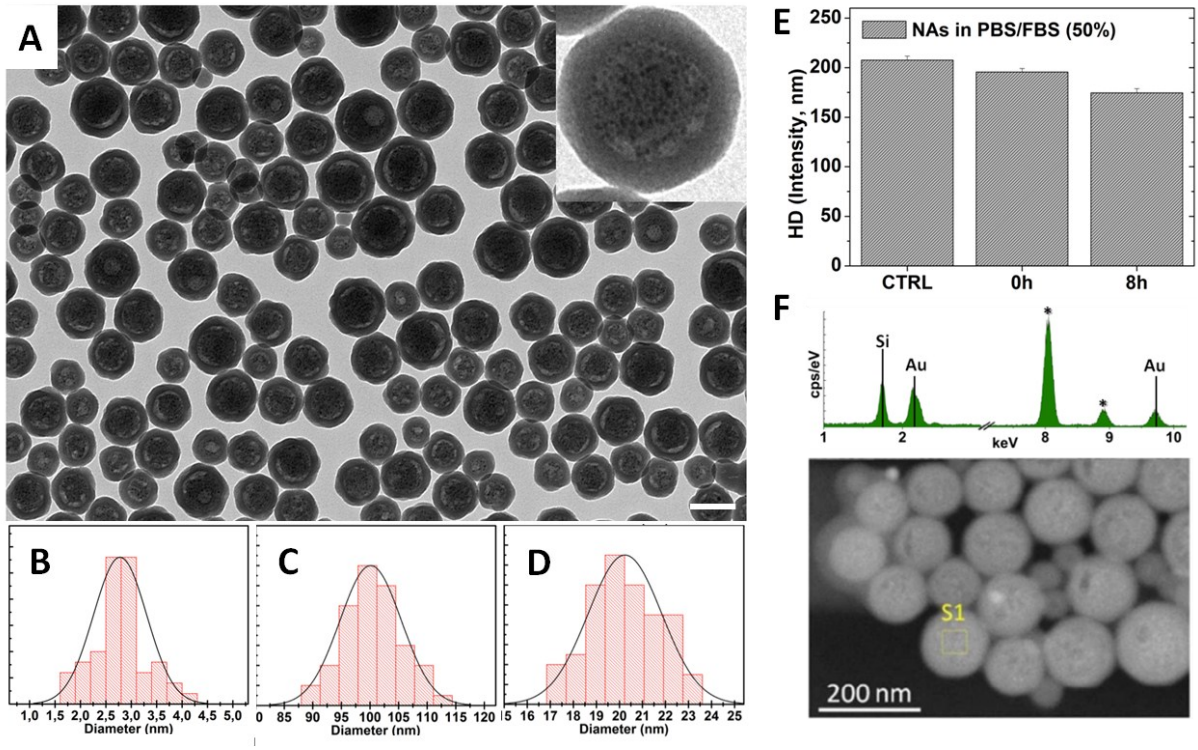

Figure S1: (A) Typical wide-area TEM image of NAs. Scale bar 100 nm. Inset: zoom on one NA. Size histogram of gold USNPs (B), NAs (C), and NAs shell (D) made on at least 100 NAs observed by TEM. (E) DLS measurements on NAs in blood-mimicking solution over 8 hours vs CTRL (NAs in PBS buffer). (F) EDX analysis on NAs revealing the presence of silicon and gold (copper peaks from TEM grid are marked with a black star).

Figure S2

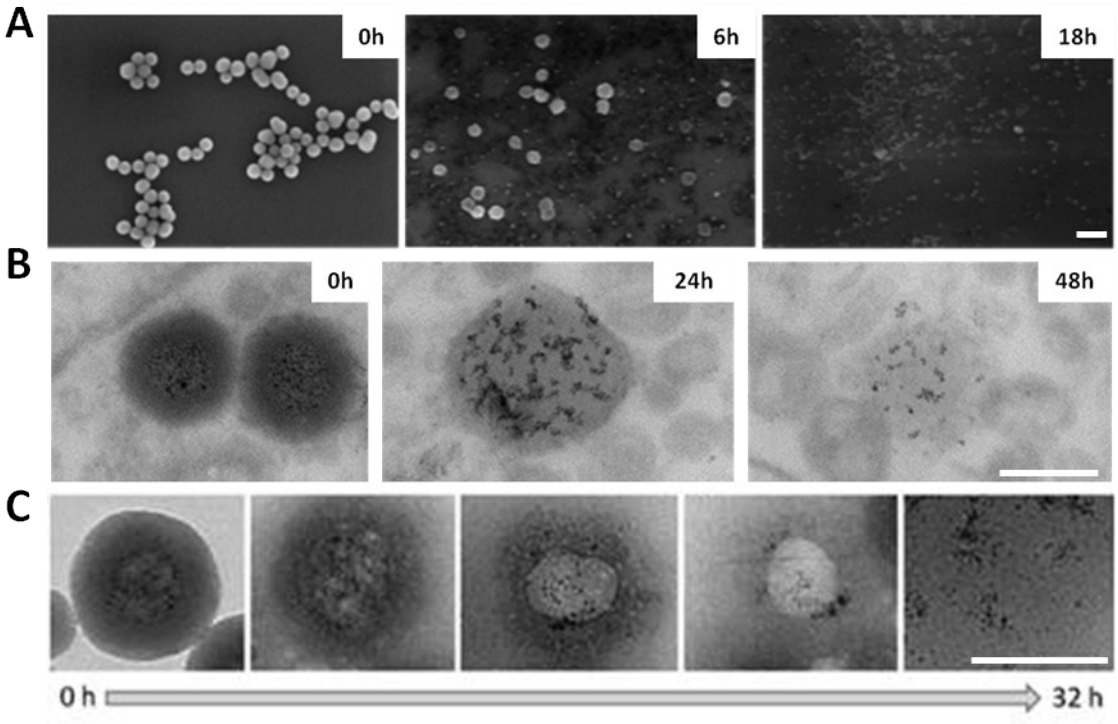

Figure S2: Electron microscopy imaging of NAs biodegradation in (B) full human serum, (B) 2D-cultured Mia PaCa-2 cells, (C) full human blood. Scale bars: 100 nm

Figure S3

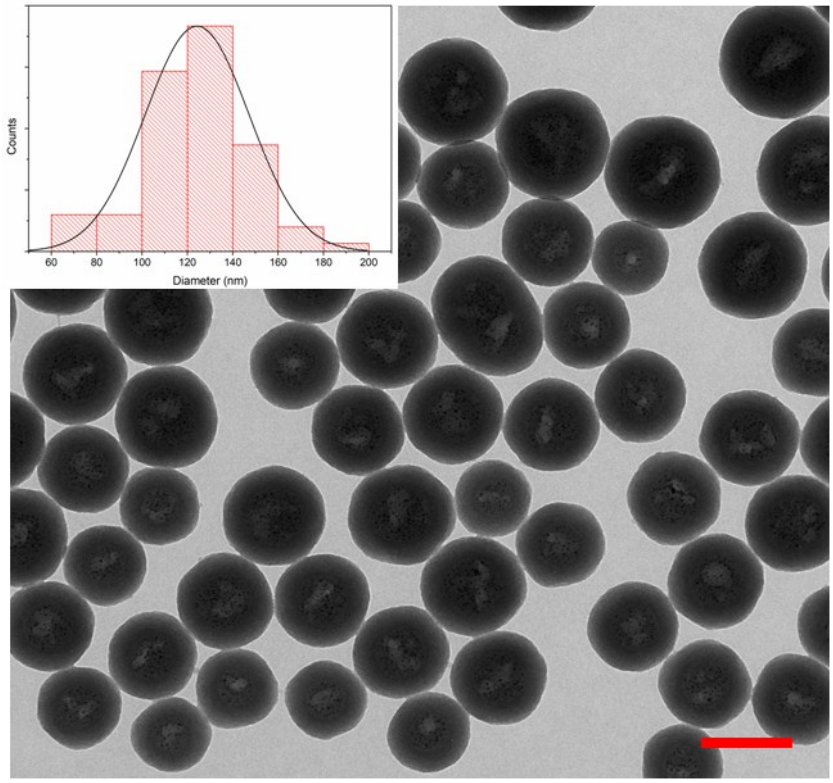

Figure S3: TEM image of *t*NAs. Scale bar 100nm. The inset shows the size histogram of *t*NAs calculated on n=151 nanoparticles.

Figure S4

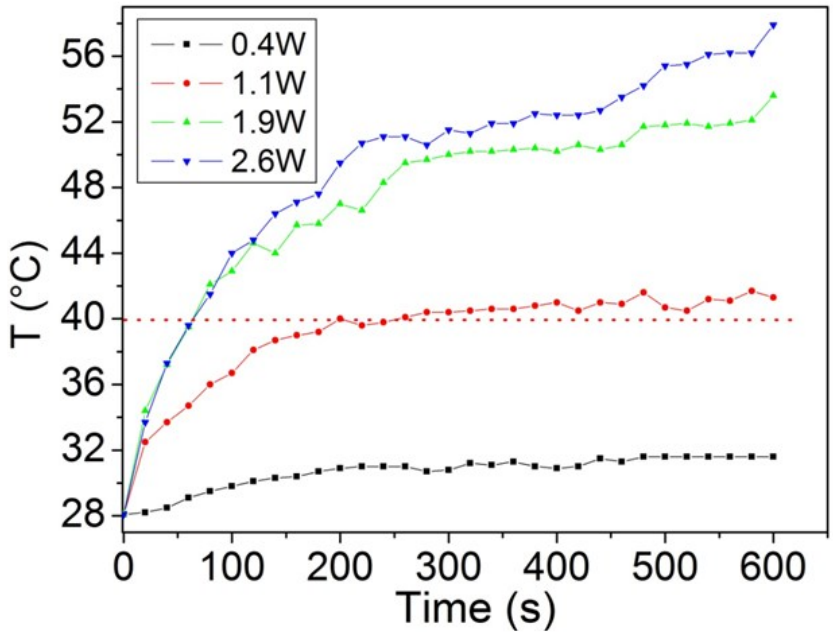

Figure S4: Temperature trend of colloidal dispersion of *t*NAs upon irradiation at varying laser output power for 10 minutes.

Figure S5

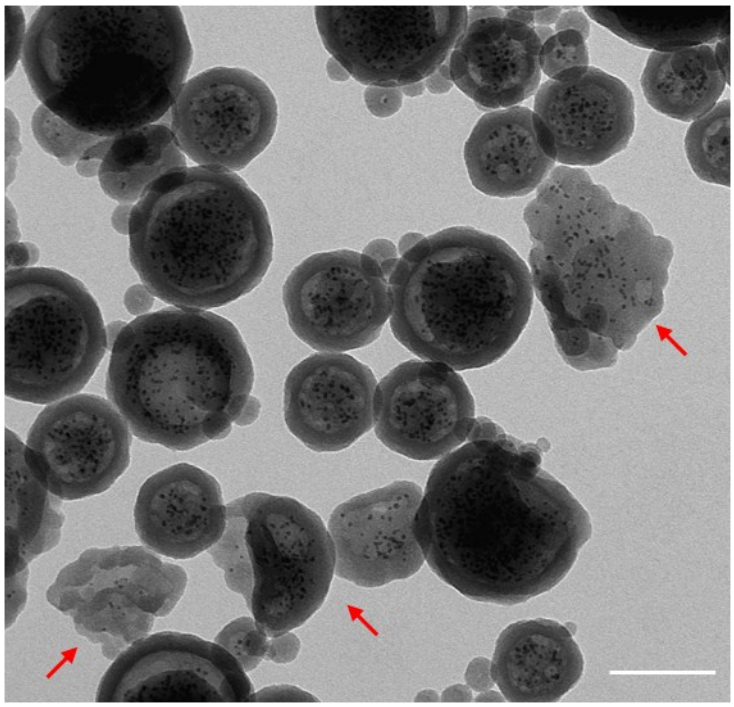

Figure S5: TEM images of *t*NAs after 4 PT cycles at laser power 2.6 W. Red arrows highlights *t*NAs that underwent structural breakage most probably for the pressure caused by the high temperature reached in the core. Scale bar: 100 nm.

Figure S6

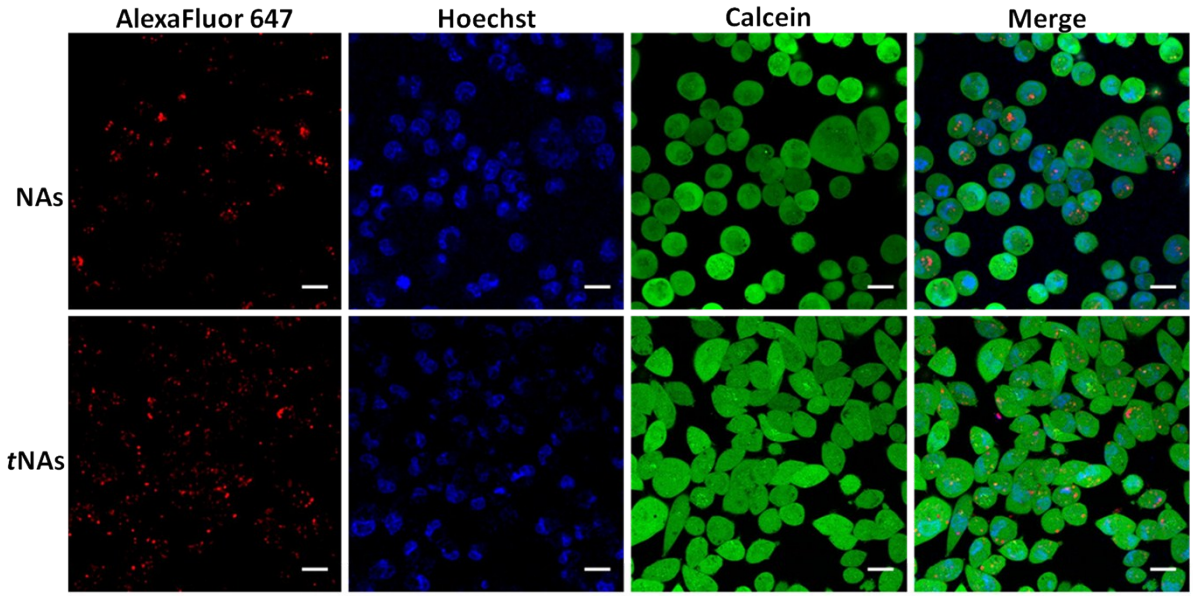

Figure S6: Confocal fluorescence images of the cellular uptake assessment for NAs and *t*NAs on 2D cultured MIA PaCa-2 cells. Red, blue and green LUTs represent AlexaFluor 647 from nano-architectures, Hoechst and Calcein, respectively. Scale bars: 10 μm.

Figure S7

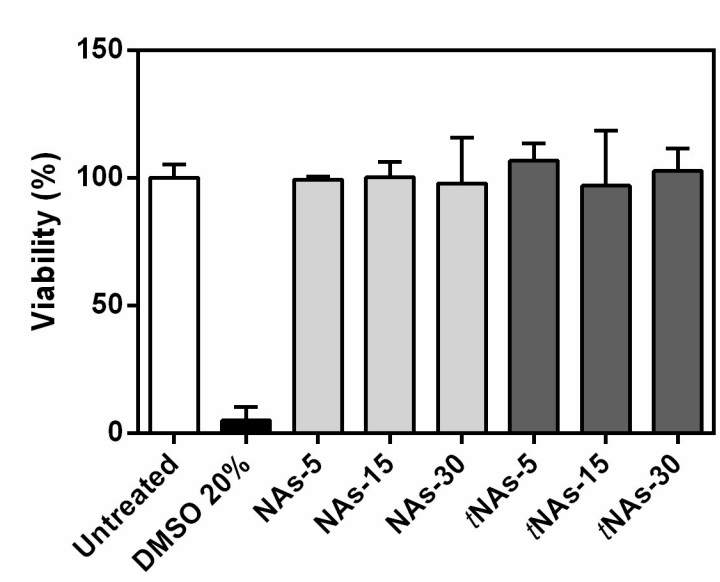

Figure S7: WST-8 viability assay on NAs and *t*NAs incubated in Mia PaCa-2 cells at increasing amount of nano-architectures (5, 15 and 30 μg).

Figure S8

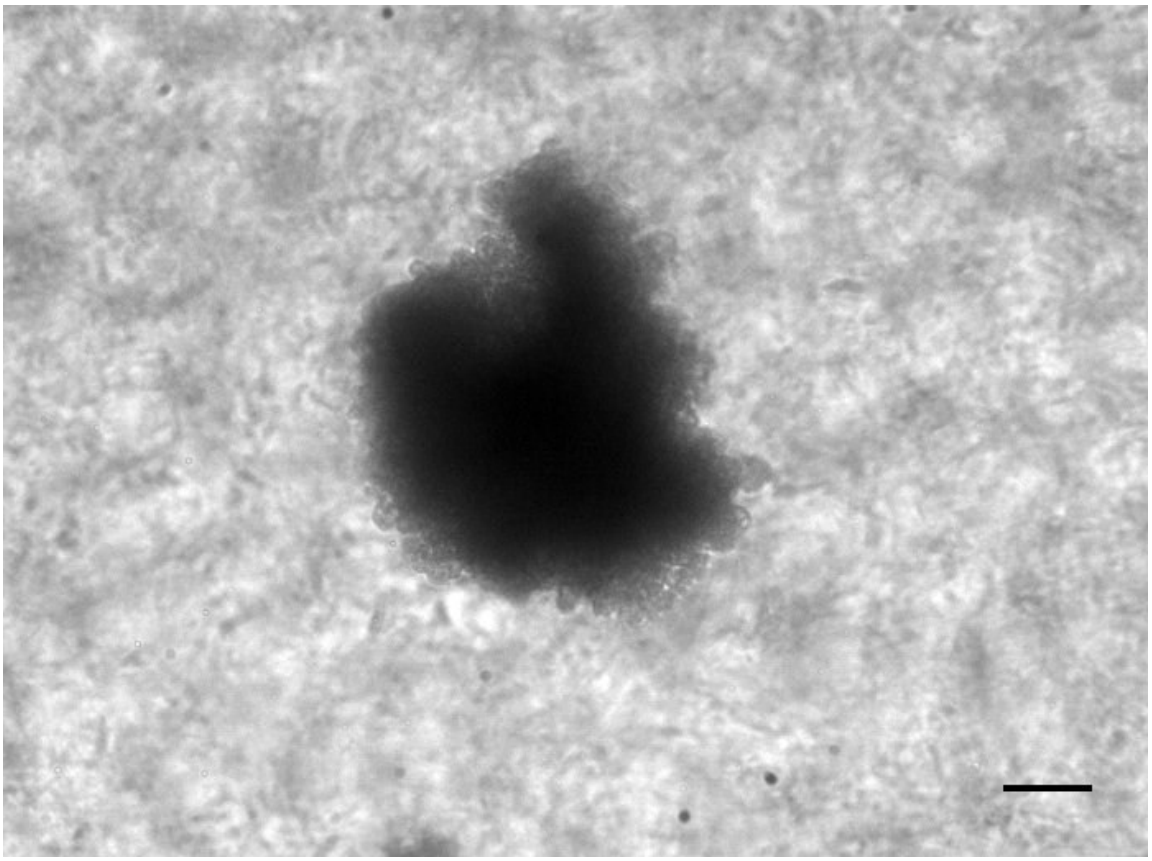

Figure S8: Example of a spheroid used for 3D photothermal experiments. Bright field image taken with a light transmission microscope of a 3D structure in a round bottom 96-wells plate. Scale bar: 100 μm.

Figure S9

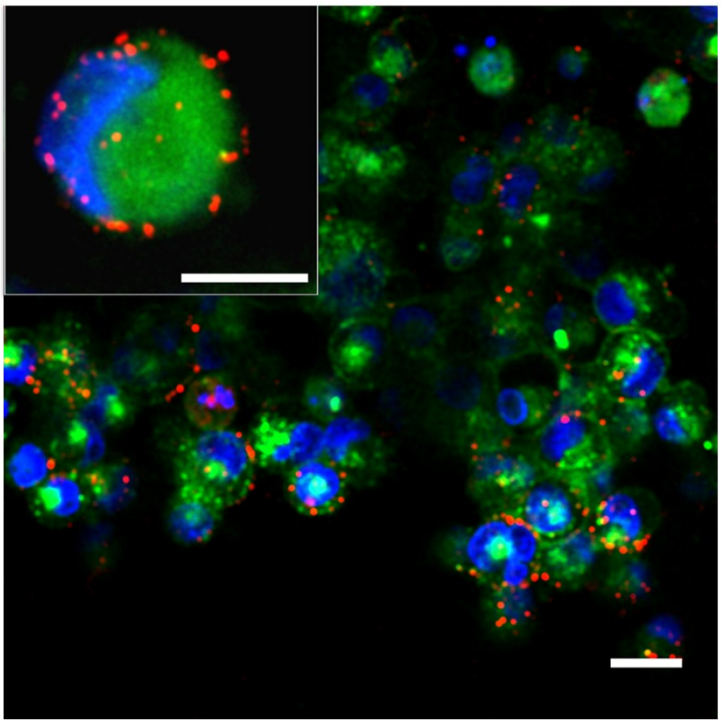

Figure S9: Confocal images of 3D MIA PaCa-2 model after 2 hours of incubation with NAs. Red, blue and green LUTs represent AlexaFluor 647 from *t*NAs, Hoechst 33342, and CellMask green plasma membrane stain, respectively. Scalebar: 20 µm. Inset: NAs on a single cell. Scalebar: 10 µm.

Figure S10

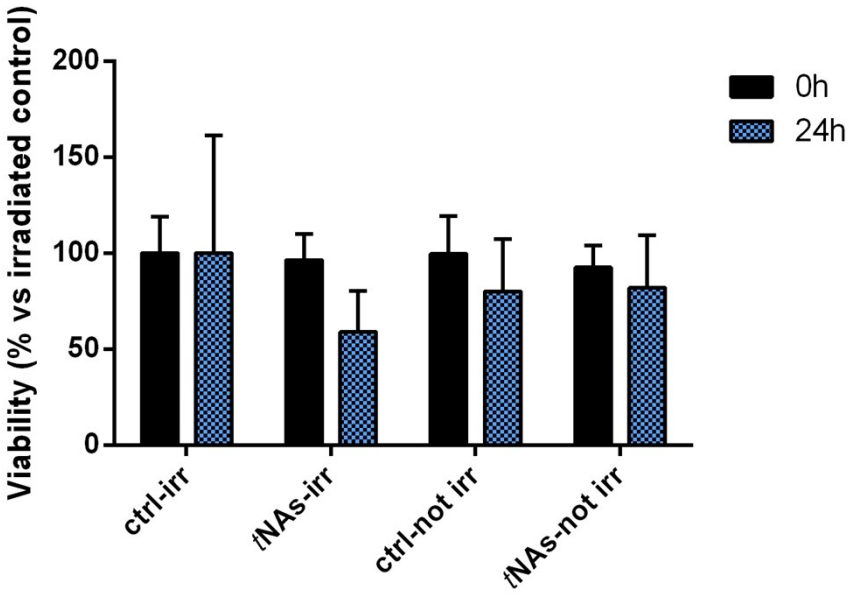

Figure S10: 3D viability assay on 3D models. For each time point, viability of each spheroids was measured using CellTiter-Glo® 3D Cell Viability Assay. Results are reported as the percentage of viability respect to the irradiated control (ctrl-irr). Data represent the average of three independent experiments. Error bars represent the standard error.