Electronic Supplementary Material (ESI) for Nanoscale.
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Supporting information

Experimental section

Cell culture.

HeLa cells were maintained in a flask containing RPMI-1640 (Life Technologies) supplemented with 10% fetal bovine serum (Life Technologies), penicillin and streptomycin (Life Technologies) at 37°C in a humidified atmosphere containing 5% CO_2. Cell number was determined with a Petroff Hausser cell counter (USA).Cells were cultured in 6-well plates and grown to 80% confluency for 48 h before incubation with the probes. To stimulate or inhibit expression of ATP in vivo, the cell medium was mixed with etoposide or oligomycin immediately before sensing.

Cytotoxicity assay.

The cytotoxicity of dimers was determined using the CCK-8 assay. Briefly, after seeding in 96-well plates and culturing overnight, the cells were incubated with 100 μL culture medium containing dimer probes for different times. Control cells were incubated with 100 μL culture medium without probes. After washing twice with PBS, CCK-8 (10 μL) was added to each well. After incubation at 37°C for 1.5 h, the absorbance (450 nm) was recorded by a microplate reader. Cell viability was determined as described by the manufacturer.

Evaluation of cytotoxicity due to the Drug.

Cells were seeded in 96-well plates and incubated with 10 and, 5 μg mL^{-1} oligomycin, not treated, and treated with 59 and, 88 μg mL^{-1} etoposide for 1 h. After washing twice with PBS, CCK-8 (10 μL) was added to each well and incubated at 37°C for 1.5 h. The absorbance (450 nm) was recorded using a microplate reader. Cell viability was determined as described by the manufacturer.

Table S1. DNA and ATP aptamer sequences

Sequence number	5’-3’ (the underlined nucleotides are phosphorothioate-modified)
Linker DNA	ACT CAT CTG TGA AGA GA ACC TGG GGG AGT ATT GCG GAG GAA GGT
5’-SH DNA for large GNP	TTT TTT TTT CCC AGG TTC TCT
5’-SH DNA for small GNP	TCA CAG ATG AGT TTT TTT TTT
Mismatch sequence	ACT CAT CTG TGA AGA GA TCG TGG GCG AGT ATT GCG TAG GAA GAT

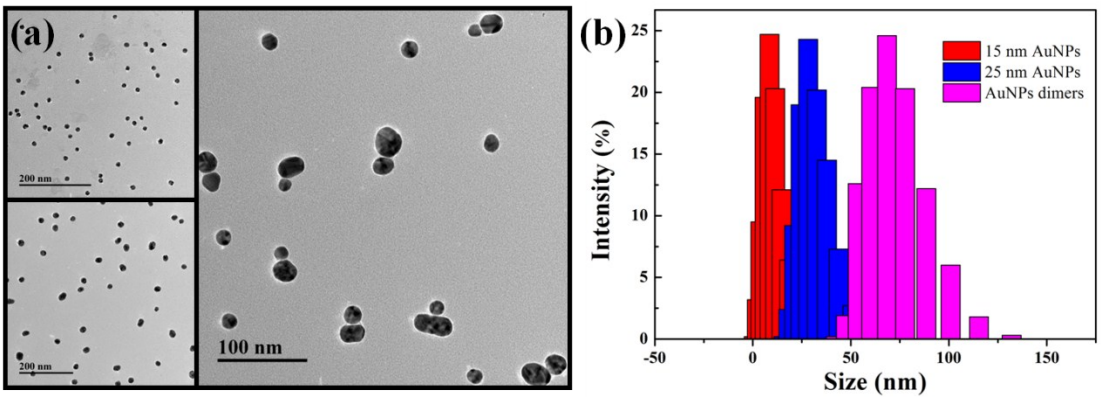

Figure S1. (a) Representative TEM images of 10nm, 25nm GNPs and GNP heterodimers. (b) DLS characterization of the GNPs and the prepared GNP dimers.

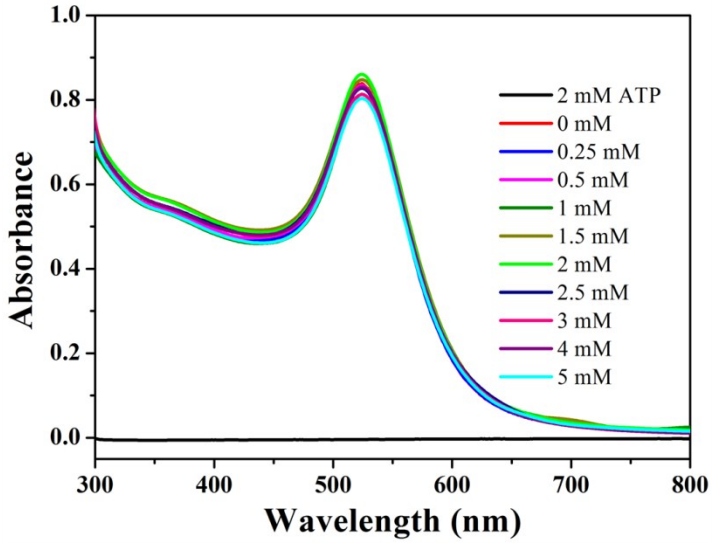

Figure S2. Model UV-vis profile of heterodimers in PB buffer in the presence of different amount of ATP.

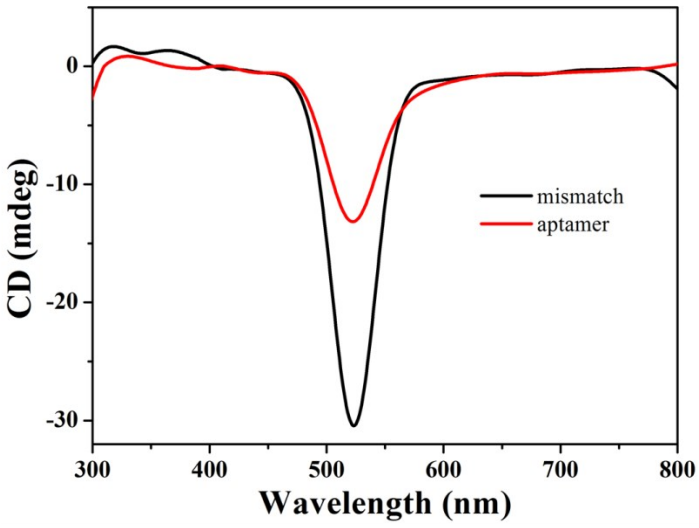

Figure S3. CD spectra of mismatch-dimers (black) and aptamer-dimers (red) treated with ATP (2 mM).

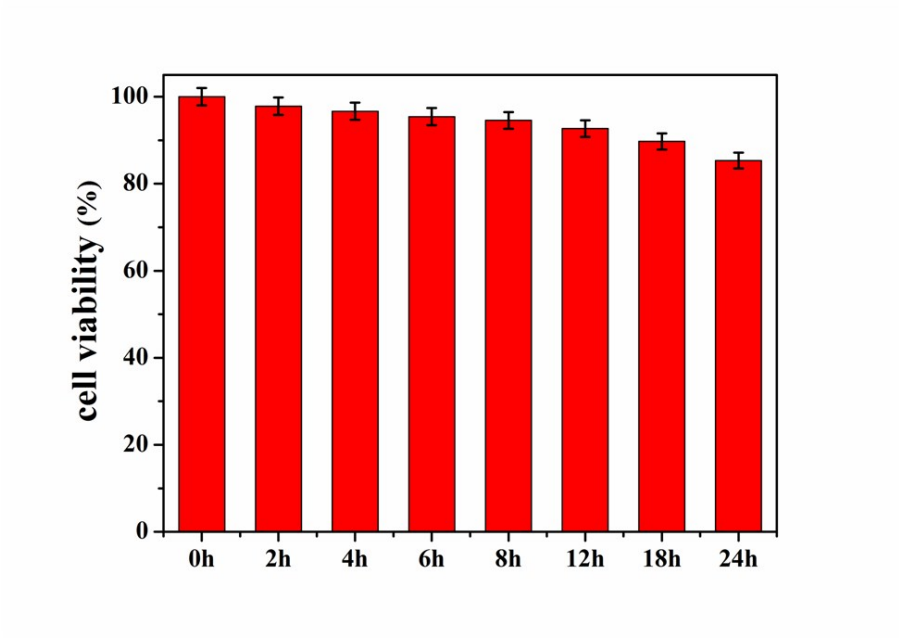

Figure S4. Viability of HeLa cells (100 μL, 1.0×10^6 mL^{-1}) after incubation with culture medium containing aptamer-dimers for different times.

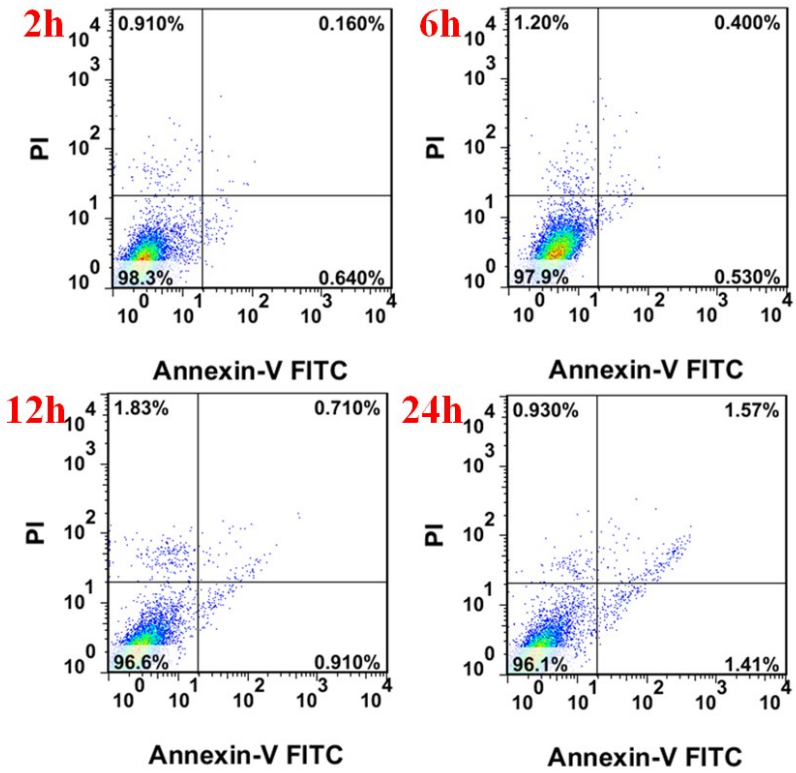

Figure S5. Flow cytometric detection of HeLa cells (100 μL, 1.0×10^6 mL^{-1}) after incubation with culture medium containing aptamer-dimers for different times.

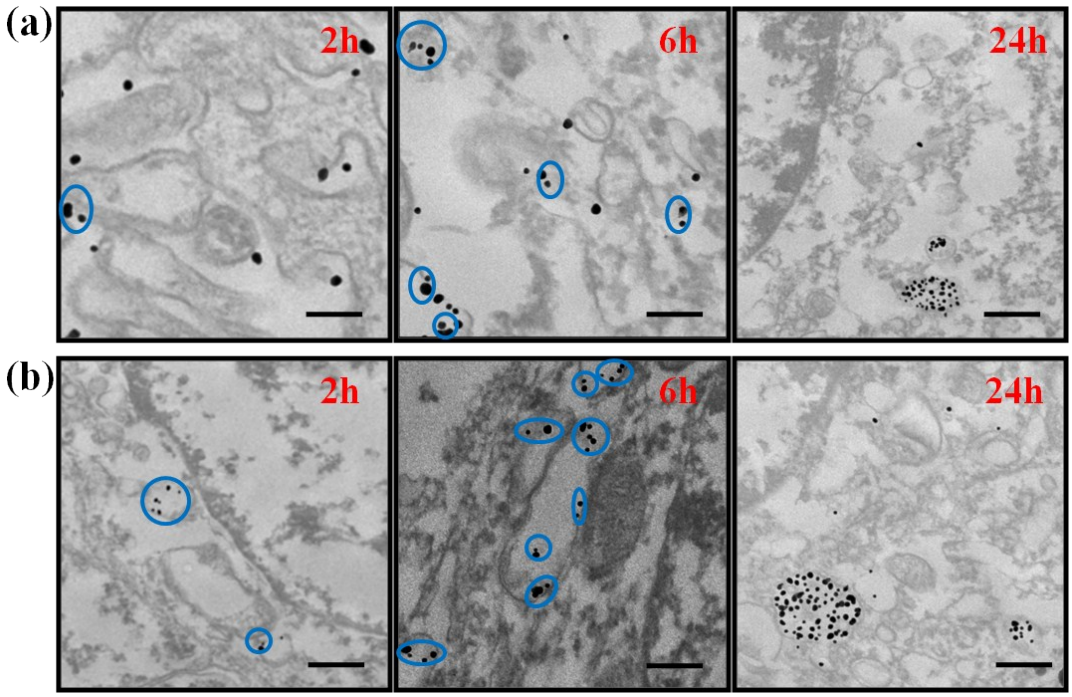

Figure S6. Bio-TEM of HeLa cell after incubated with (a) aptamer-dimers and (b) mismatch-dimers in different times. scale bars 100nm.

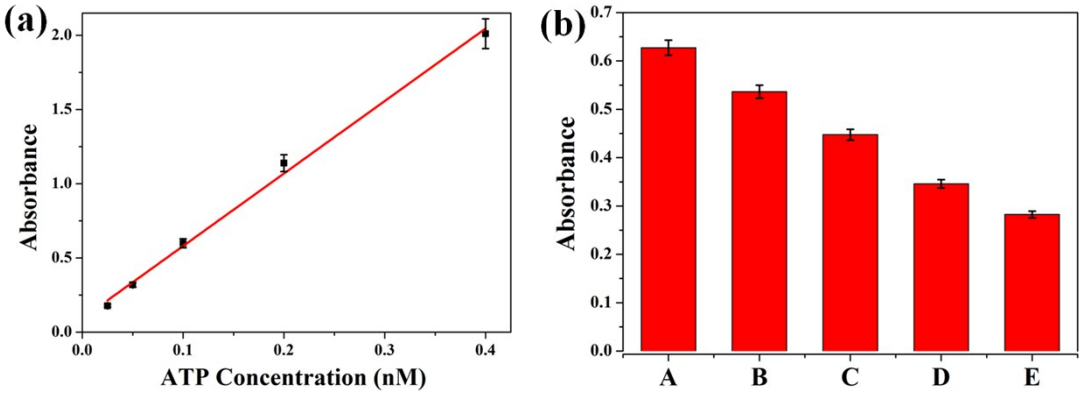

Figure S7. (a) Standard curve for detection of ATP concentration in solution with the ELISA kit. (b) Absorbance of ATP ELISA kit for cell extracts collected from HeLa cells(5 ×10^7 cells) respectively treated with 10, 5 μg mL^{-1} oligomycin(inhibitor),untreated ,and treated with 59,88 μg mL^{-1} etoposide(promoting reagent) (from A to E).

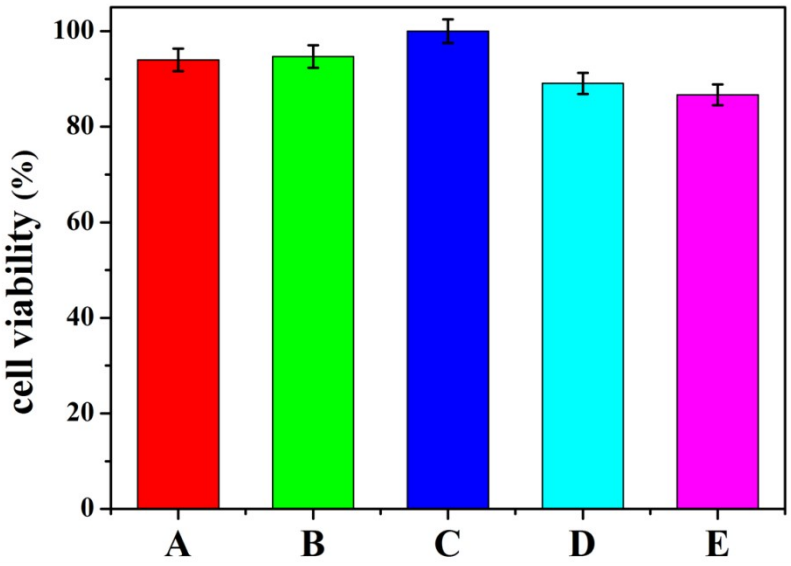

Figure S8. Viability of HeLa cells (100 µL, 1.0×10^6 mL^{-1}) after incubation with (A)10, (B) 5 µg mL^{-1} oligomycin, (C) untreated, and treated with (D) 56, (E) 88 µg mL^{-1} etoposide.