[Supporting Materials]

Near-infrared Light Triggered *in situ* $Cu(DDC)_2$ Complex Formation and Reactive Oxygen Species Amplification Cascade for Cancer Therapy

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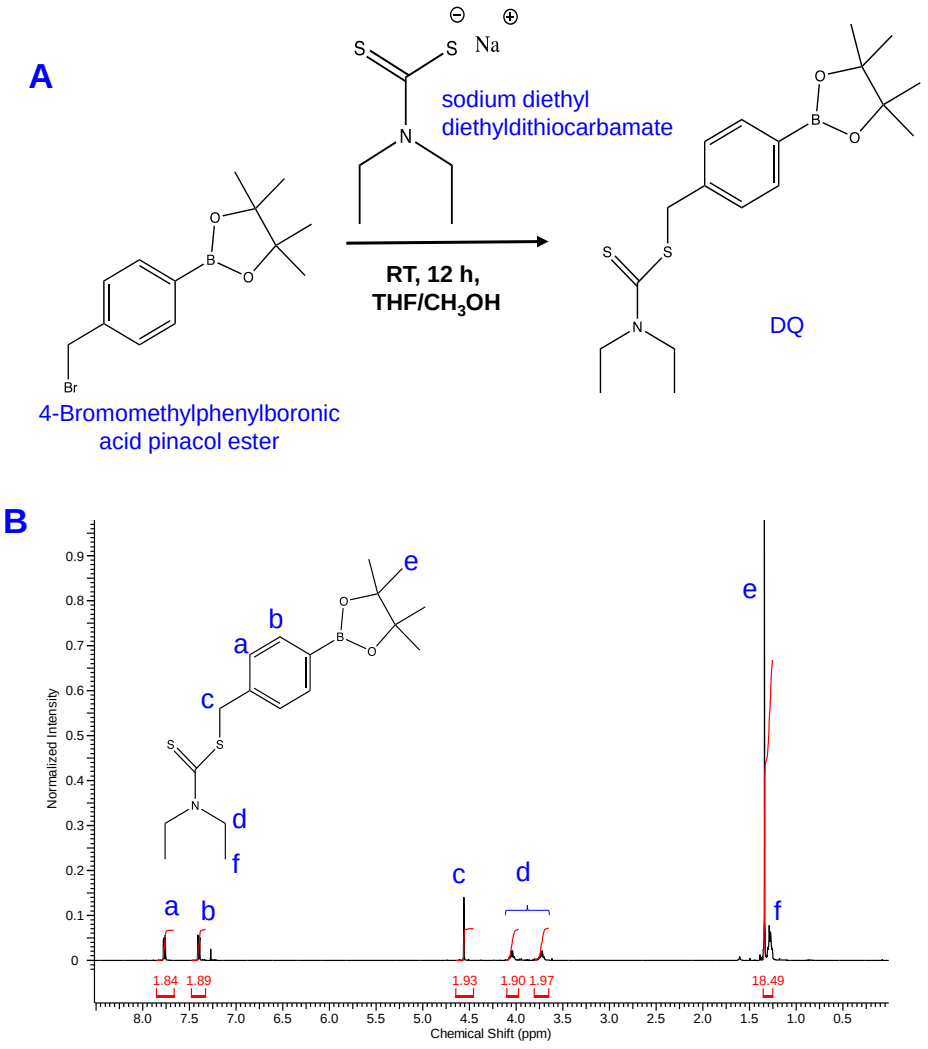

Scheme S1. (A) Synthesis of DQ. 4-Bromomethylphenylboronic acid pinacol ester dissolved in THF was added into sodium diethyl diethyldithiocarbamate trihydrate dissolved in CH_3OH, and reacted at room temperature (RT) for 12 hours. The reaction mixture was dissolved in ethyl acetate, washed with water and brine, concentrated, and purified with a silica column. **(B)** ^{1}H-NMR spectrum of DQ in $CDCl_3$.

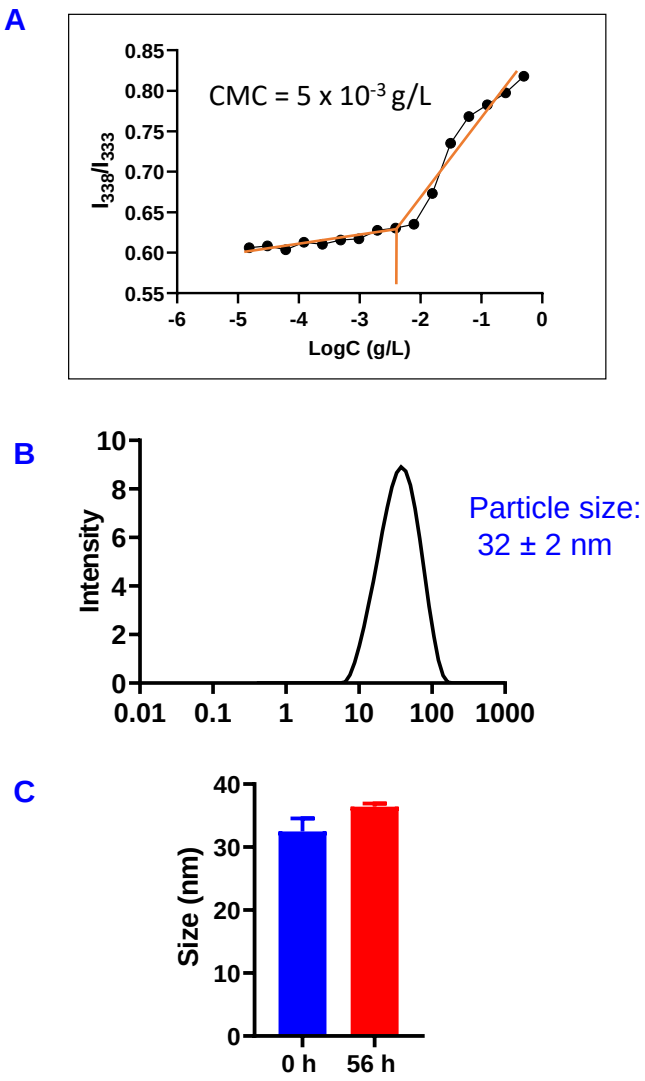

Figure S2. **(A)** Critical Micelle Concentration (CMC) of PEG-PLA micelle. **(B)** Particle size. **(C)** Stability in serum-containing PBS.

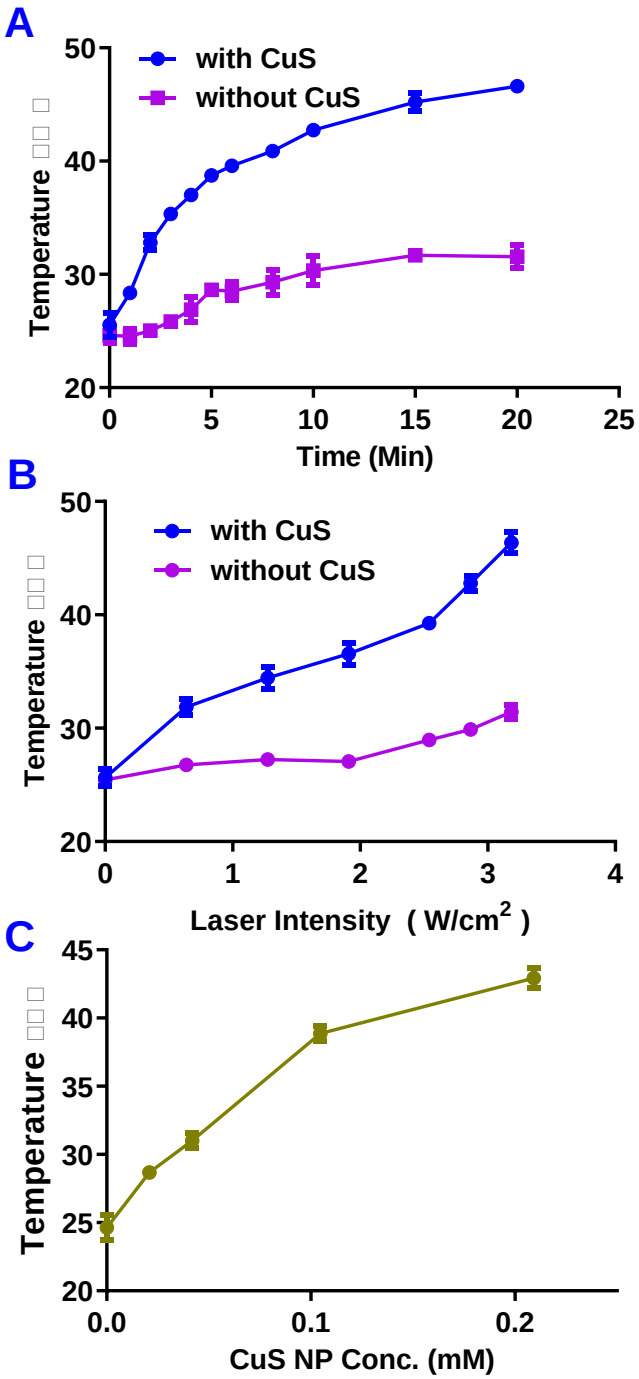

Figure S3. Photothermal study of CuS NPs. (**A**) The effects of NIR laser treatment time on the temperature change in CuS NP sample and pure water control. (NIR laser intensity: 2.54 W/cm^2, CuS NP concentration: 0.1 mM.) (**B**) The effects of NIR laser intensity on the temperature change in CuS NP sample and pure water control. (NIR laser treatment time: 5 min. CuS NP concentration: 0.1 mM.) (**C**) The effect of CuS NP concentration on the temperature change. (NIR laser intensity: 2.54 W/cm^2; NIR laser treatment time: 5 min.) Results are mean ± SD (n=3).

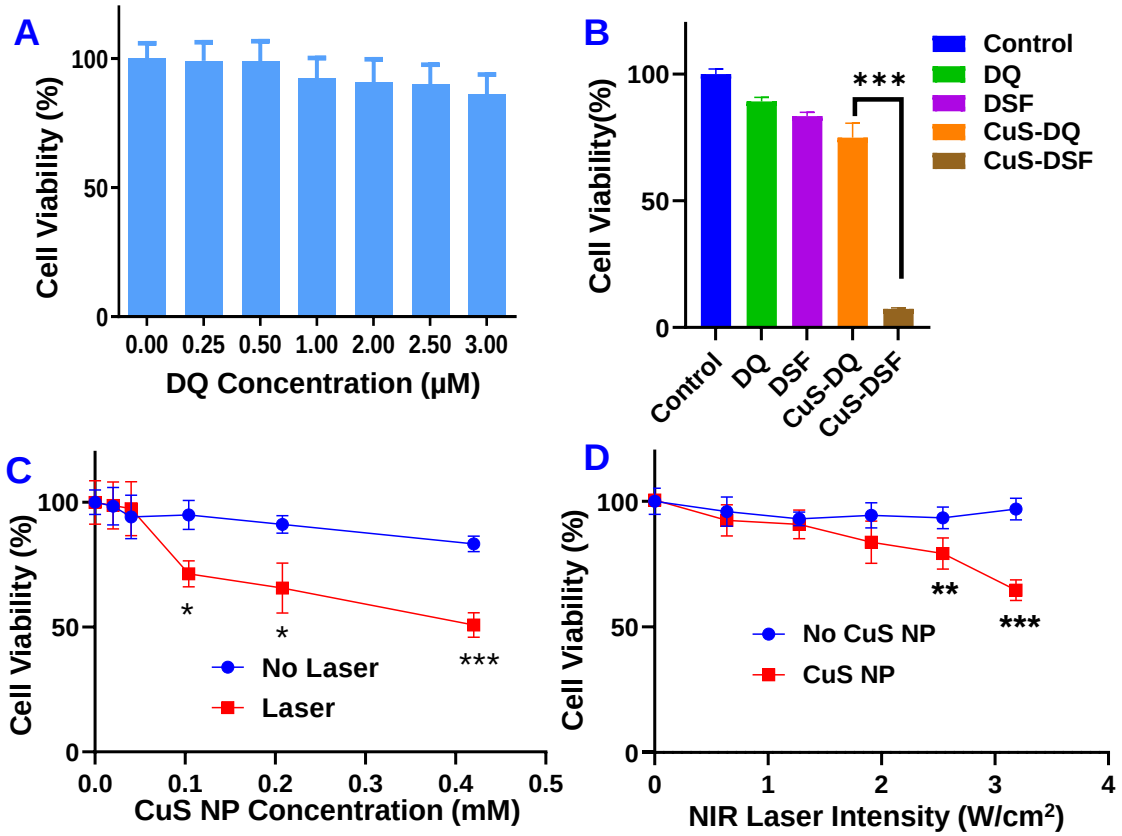

Figure S4. (A) The effects of DQ micelles on 4T1 cell viability as determined with the MTT assay. (**B**) The effects of DQ (2 μM), DSF (1 μM), DQ (2 μM) + CuS (0.1mM), and DSF (1 μM) + CuS (0.1mM) on 4T1 cell viability as determined with the MTT assay. Effects of (**C**) CuS NP concentration and (**D**) NIR laser intensity on 4T1 cells viability as determined with the MTT assay. (Results are mean ± SD, n=3, * $P < 0.05$, ** $P < 0.01$, *** $P < 0.001$)

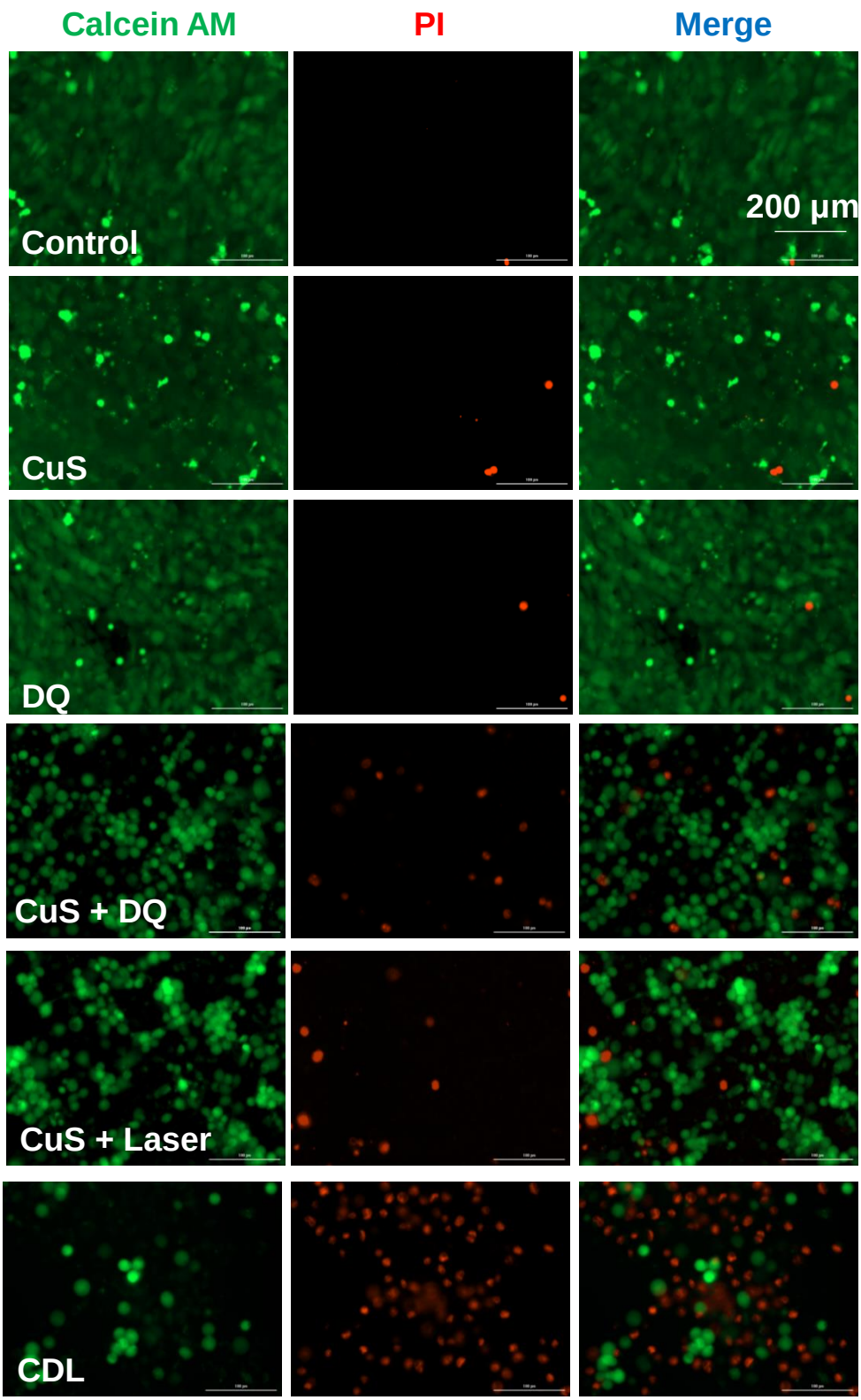

Figure S5. The viability of 4T1 cells receiving different treatments were determined with the Calcein-AM/PI straining. (CuS, 0.1 mM; DQ, 2 μM; Laser, 2.54 W/cm^2 for 5 minutes.)

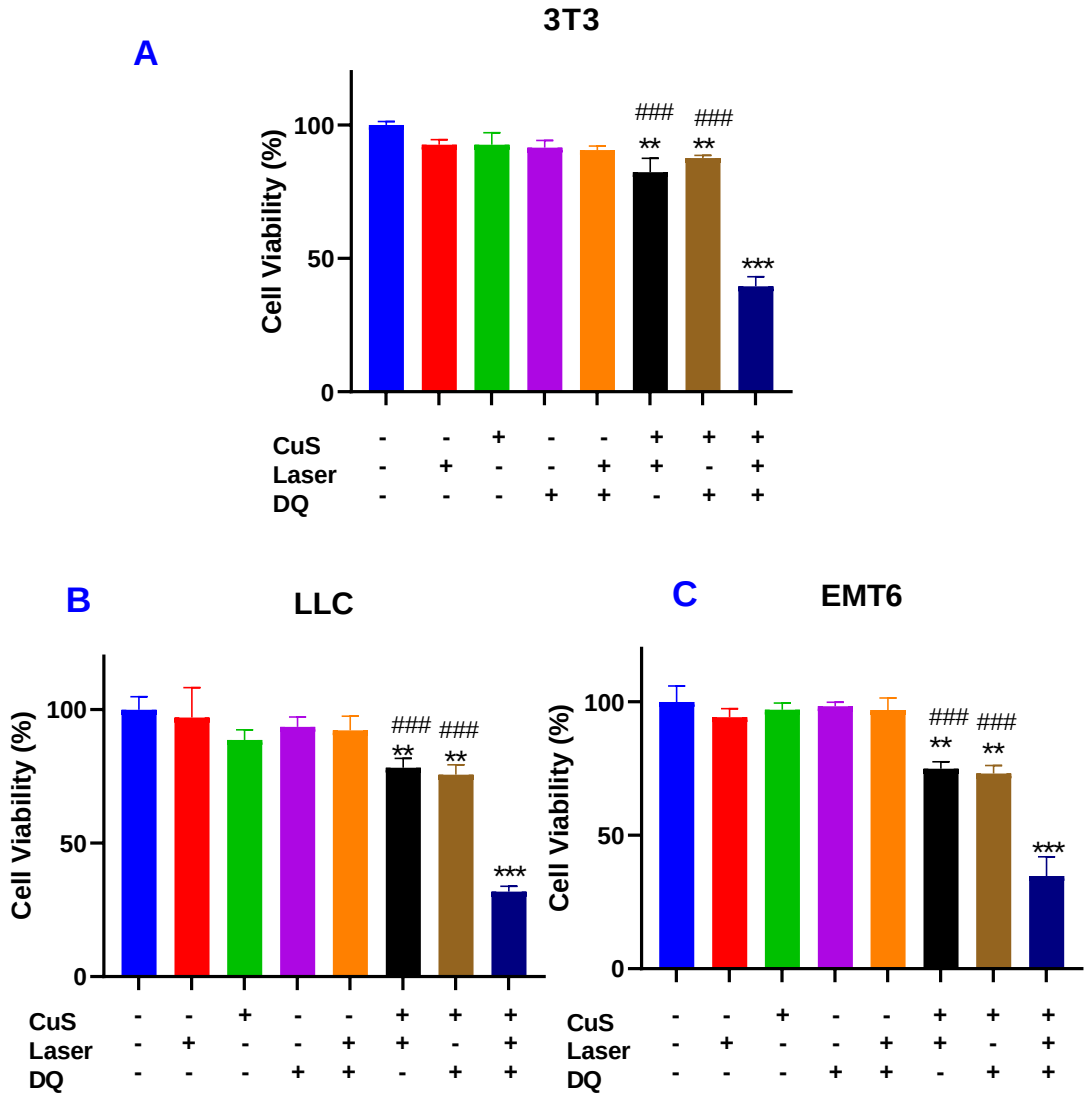

Figure S6. The viability of cells was determined with the MTT assay. (A) 3T3 cells. **(B)** LLC cell. **(C)** EMT6 cells. (CuS, 0.1mM; DQ, 2 μM; Laser, 2.54 W/cm^2 for 5 minutes. Data are presented as the mean ± SD, n = 3, * P < 0.05, ** P < 0.01, *** P < 0.001 compared with the negative control group; # # P < 0.01, # # # P < 0.001, compared with CDL treatment group).

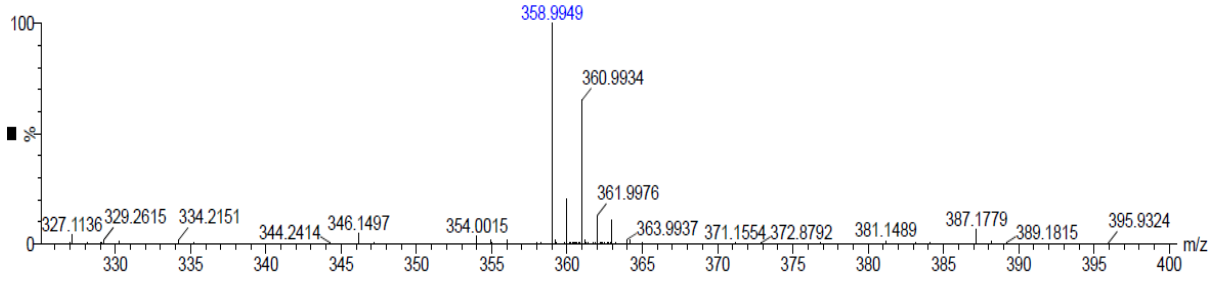

Figure S7. $Cu(DDC)_2$ formed in cells treated with CDL combination therapy was detected by LC/MS.

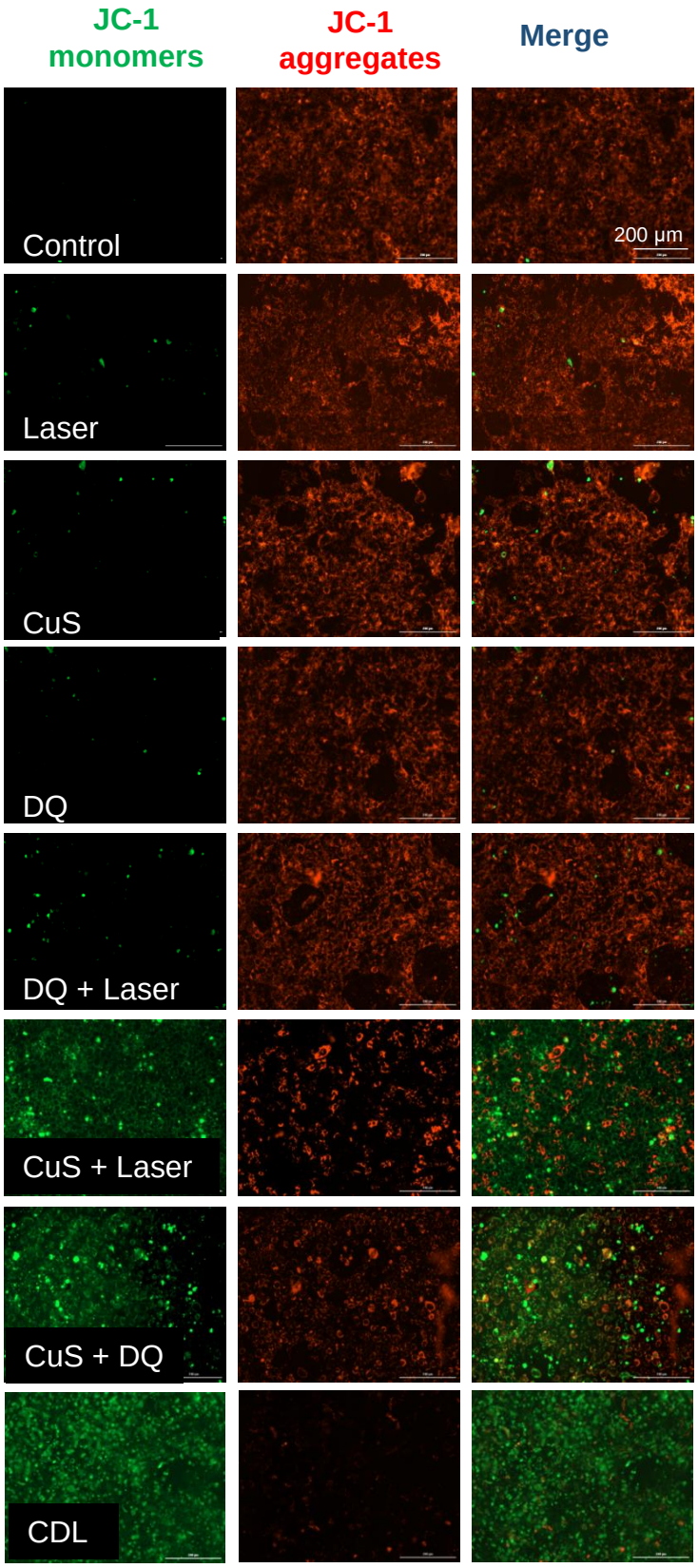

Figure S8. The MMP of 4T1 cells receiving different treatments were determined with the JC-1 staining. (CuS, 0.1 mM; DQ, 2 μM; Laser, 2.54 W/cm^2 for 5 minutes.)