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## **Supplementary Information**

## Fabrication Optimization and Application of 3D Hybrid SERS Substrates

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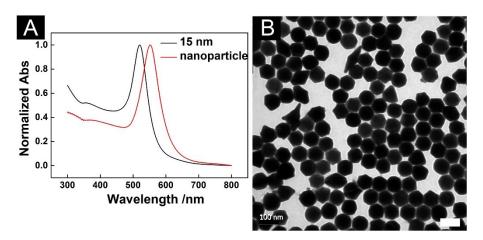


Fig. S1 (A) The UV-vis-IR absorption spectra of Au nanoparticles with sizes of 15.98  $\pm$  0.64 nm (black) and 79.09  $\pm$  3.28 nm (red). (B) TEM images of the Au nanoparticles with a size of 79.09  $\pm$  3.28 nm. The scale bar is 100 nm.

Table S1. The nanogap spacing between nanoparticles of 2D substrates composed of AuNS of different diameters

AuNS	15.98 ± 0.64	29.82 ± 1.35	45.68 ± 1.99	54.67 ± 2.61	64.76 ± 2.46
size	nm	nm	nm	nm	nm
Distance of	6.04 ± 1.28	3.31 ± 1.04	4.44 ± 1.06	3.57 ± 0.82	3.65 ± 0.70
nanogap	nm	nm	nm	nm	nm

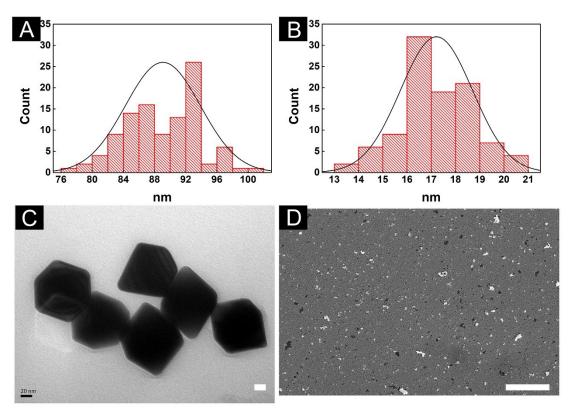


Fig. S2 (A) The distribution of the length of AuNR. (B) The distribution of the diameter of AuNR. (C) TEM of AuNO with the scale bar of 20 nm. (D) SEM of AuNO with the scale bar of 10  $\mu$ m.

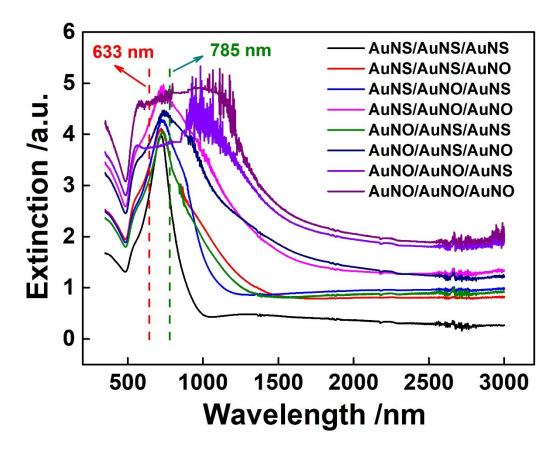


Fig. S3 The UV-Vis-NIR spectra of the substrates with different combination of (1) AuNS/AuNS/AuNS, (2) AuNS/AuNS/AuNO, (3) AuNS/AuNO/AuNS, (4) AuNS/AuNO/AuNO/AuNO, (5) AuNO/AuNS/AuNS, (6) AuNO/AuNS/AuNO, (7) AuNO/AuNO/AuNO, (8) AuNO/AuNO/AuNO.

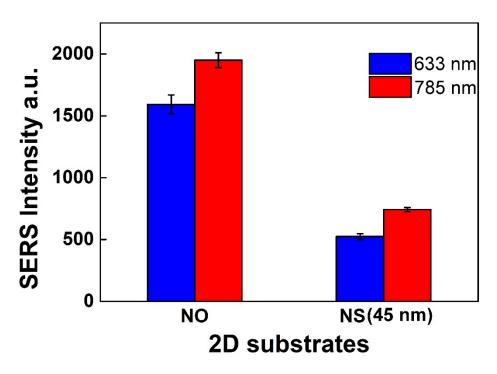


Fig. S4 Histogram of SERS intensity of monolayer AuNO and AuNS planar substrates. The SERS intensity at 1080 cm<sup>-1</sup> of 4-MBA were used for comparing the performance.

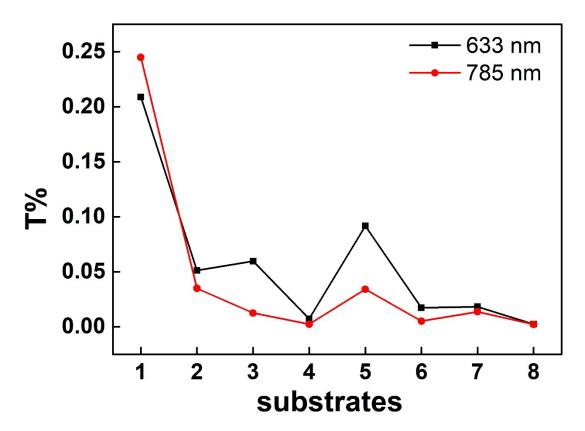


Fig. S5 Light transmittance of the substrates (1) AuNS/AuNS/AuNS, (2) AuNS/AuNS/AuNO, (3) AuNS/AuNO/AuNS, (4) AuNS/AuNO/AuNO, (5) AuNO/AuNS/AuNS, (6) AuNO/AuNS/AuNO, (7) AuNO/AuNO/AuNO, (8) AuNO/AuNO/AuNO at wavelengths of 633 and 785 nm, respectively.