## **Supporting Information**

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**Figure S1.** Oil-water separation of the 3D Au NPs/nickel foam. The as-prepared 3D Au NPs/nickel foam was placed in the bottom of the syringe. (a)-(c) chloroform/water separation experiment (water at the top of the syringe); (a')-(c') benzene/water separation experiment (water at the bottom of the syringe).



**Figure S2.** (a) A SERS spectrum of 10<sup>-3</sup> M pyrene on the 1-octadecanethiol modified 3D Au NPs/nickel foam substrate and (b) a Raman spectrum of 1-octadecanethiol modified 3D Au NPs/nickel foam substrate.



**Figure S3.** (a) A SERS spectrum of 10<sup>-3</sup> M 1-naphthol on the 1-octadecanethiol modified 3D Au NPs/nickel foam substrate and (b) a Raman spectrum of 1-octadecanethiol modified 3D Au NPs/nickel foam substrate.



**Figure S4.** (a) A Raman spectrum of 1-naphthol solid powder, (b)-(e) SERS spectra of 1-naphthol with different concentrations by using the modified substrates:  $10^{-3}$ ,  $10^{-4}$ ,  $10^{-5}$  and  $10^{-6}$  M , (f) A Raman spectrum of the modified substrate without 1-naphthol.



**Figure S5.** SERS intensity at 1385 and 712 cm<sup>-1</sup> versus the concentration of pyrene on the 1-octadecanethiol modified 3D Au NPs/nickel foam substrate. Each error bar means the standard deviation at five different detected positions randomly.



**Figure S6.** (a) The photograph of the 3D hydrophobic SERS substrate. (b) The simple oil-water separation device of the 3D hydrophobic SERS substrate. (c) The process of the adsorption of PAHs using the simple oil-water separation device for the detection of PAHs. A stands for the entrance of the analytes; B stands for the position of the 3D hydrophobic SERS substrate; C stand for the exit of organic solvents; D stands for the exit of aqueous solution with salt or contaminant.



**Figure S7.** SERS spectra of pyrene molecules in the absence and presence of various kinds of metal ions with concentrations of  $10^{-3}$ ,  $10^{-4}$  and  $10^{-5}$  M.



**Figure S8.** SERS spectra of pyrene molecules in the tap water with concentration of  $10^{-5}$  M. SERS spectra a, b, c and d, e, f show at different positions in sample 1 and sample 2, respectively.



Figure S9. SERS spectra of 1-naphthol molecules in real environment (the tap water) with concentration of  $10^{-5}$  M. SERS spectra a, b, c and d, e, f show at different positions in sample 1 and sample 2, respectively.

Pyrene /10 <sup>-5</sup> M	Wavenumber /cm <sup>-1</sup>	407	1240	1405
Sample 1	AVG	112.215	161.504	117.412
	RSD	3.10	0.98	2.03
	Recovery percent	109.2%	98.0%	94.4%
Sample 2	AVG	106.552	156.092	92.760
	RSD	3.57	2.61	3.23
	Recovery percent	115.0%	99.2%	101.2%

**Table S1.** The recovery percent of pyrene  $(10^{-5} \text{ M})$  in the tap water. The average values and RSD come from three different detected positions randomly in each sample.

1-naphthol /10-5 M	Wavenumber/cm <sup>-1</sup>	712	1385
Sample 1	AVG	75.741	97.826
	RSD	2.97	2.16
	Recovery percent	98.4 %	109.2 %
Sample 2	AVG	77.010	103.571
	RSD	2.44	1.12
	Recovery percent	97.4 %	106.6 %

**Table S2.** The recovery percent of 1-naphthol  $(10^{-5} \text{ M})$  in the tap water. The average values and RSD come from three different detected positions randomly in each sample.