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Sensitive and Easily Recyclable Plasmonic SERS Substrate based on Ag Nanowire in Mesoporous Silica

Xuefeng Yan, Lingzhi Wang*, Dianyu Qi, Juying Lei, Bin Shen Sen Tapas* and Jinlong Zhang*

Experimental Section

Chemicals:

Tetraethylorthosilicate (TEOS), n-Hexane, Potassium chloride (KCl) were purchased from Shanghai Lingfeng Chemical Reagent Co. Ltd without further purification. Silver nitrate (AgNO3) and FeCl3 • 6H2O was purchased from Shanghai Chemical Reagent. Poly (ethylene glycol)-block-poly(propylene glycol)-block-poly(ethylene glycol) (P123) was purchased from Sigma-Aldrich. Ultrapure water (=18.0 MΩ) purified using the Milipore Milli-Q gradient system

Characterization

Raman spectra were obtained by a Renishaw inVia with a laser of 532 nm and 0.5% strength, samples were arranged on the silica plate. X-ray diffraction (XRD) patterns of the samples were recorded on a Rigaku D/MAX-2550 diffractometer using Cu Kα radiation of wavelength 1.5406 Å, typically run at a voltage of 40 kV and current of 100 mA. UV-visible absorbance spectra were achieved for the dry pressed disk samples using a Scan UV-Vis spectrophotometer (Varian, Cary 500) equipped with an integrating sphere assembly, using BaSO4 as a reflectance sample. Transmission electron microscopy (TEM) images were collected on a JEOL JEM 2010F, electron microscope operated at an acceleration voltage of 200 kV. By utilizing the Barrett−Joyner−Halenda (BJH) model, the pore volumes and pore size distributions were got from the adsorption branches of isotherms.

Preparation of SBA-15

Generally, 8.0 g P123 and 8.8 g KCl were dissolved in 120 mL, 4 M hydrochloric acid solution. After stirring at 38 °C for 2 h, 18 mL TEOS was added into the solution. 2 min later, the solution was kept still for 24 h. After that, the mixture was transferred into an autoclave and hydrothermally treated at 100 °C for 24 h to obtain as-made samples. Then the composites were filtered off and washed by ultrapure water and ethanol. In the end, the sample was calcined at 550 °C for 6 h to remove temples after drying at 60 °C in the vacuum.

Preparation of Ag SBA-15

First, 0.2 g SBA-15 was dried by vacuum at 140 °C with the temperature increasing at 1 °C /min to remove physically adsorbed water. Then the sample was dispersed in 20 mL of n-hexane. 10 min later, 0.2 mL, 1.0 mol/L $AgNO_3$ was added into the solution and stirred for 4 h in the dark. After stirring, the composites were filtered off and dried at 80 °C to remove the liquid. For the synthesis of Ag SBA-15/NW, the powder sample was calcined at 350 °C for 2 h at an increasing rate of 2 °C /min. Ag SBA-15/NS was formed by further calcining Ag SBA-15/NW

at 550 °C for another 2 h or directly calcining the impregnated powder at 550 °C for 2 h.

Preparation of Ag SBA-15 film and Raman detection

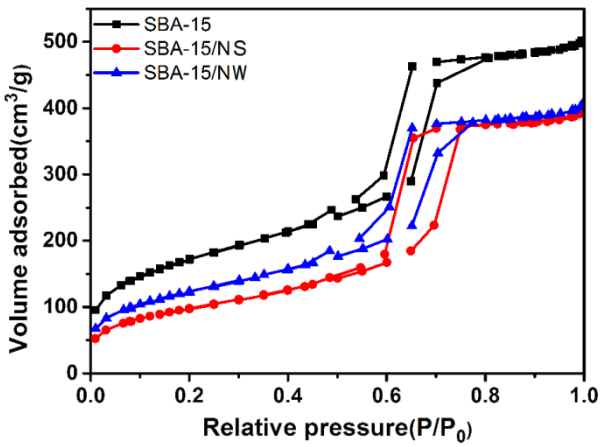
10 mg Ag SBA-15/NS or Ag SBA-15/NW were dispersed in 1 mL ethanol, and sonicated to make it totally dispersed, which was then spin-coated on a glass slide (38 mm × 24 mm). Then the slide was divided into 4 parts and dropped by 10 μ L methylene blue (MB) solution with different concentrations before Raman detection.

Preparation of Ag-AgCl SBA-15/NW and photocatalytic degradation of MB

Ag SBA-15/NW was transformed to Ag@AgCl SBA-15/NW by dropwise adding excessive amount of $FeCl_3$ water solution on the slide or just immersing the slide into the $FeCl_3$ solution to achieve most transformation efficiency from Ag to AgCl. $2*10^{-5}$ M of MB solution was used for the study about the recyclable detection. The substrate adsorbed with MB molecules was illuminated for 10 min by a simulated solar source equipped with a 300 W Xenon lamp for (AM 1.5). The re-adsorption was carried out after MB was completely photocatalytically degraded.

Photocatalytic degradation of MB in solution

0.10 g of Ag SBA-15/NW after reacted with $FeCl_3$ was dispersed in the 8.0 mL, $1*10^{-5}$ mol/L MB solution. After 0.5 h dark adsorption, the solution was illuminated for 20 min by a simulated solar source equipped with a 300 W Xenon lamp for (AM 1.5). Besides the Raman spectroscopy, UV-Vis absorption spectroscopy was also used for the degradation analysis.



Fiugre. S1 Nitrogen adsorption–desorption isotherms of SBA-15, Ag SBA-15/NS and Ag SBA-15/NW.

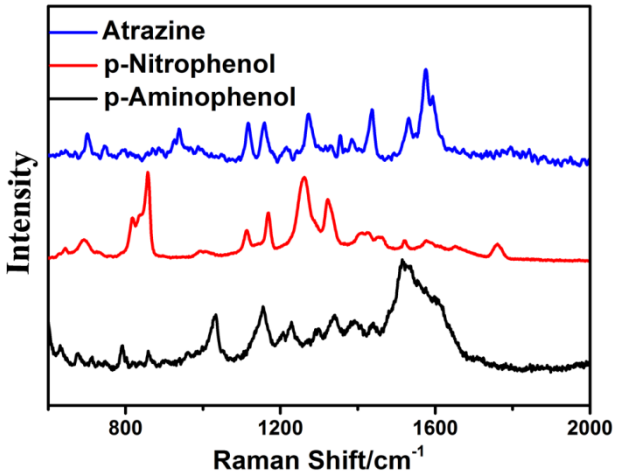


Figure. S2 Raman spectrum of atrazine (1*10^{-5} mol/L), p-nitrophenol (1*10^{-4} mol/L) and p-aminophenol (1*10^{-4} mol/L).

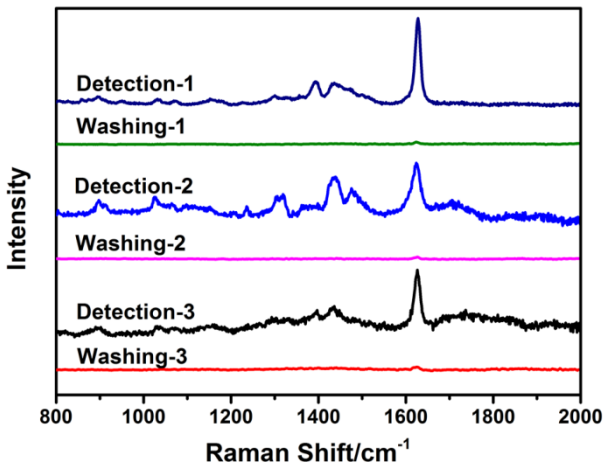


Figure. S3 Recycling SERS detection of 10^{-5} M MB on Ag SBA-15/NW by washing with ethanol.

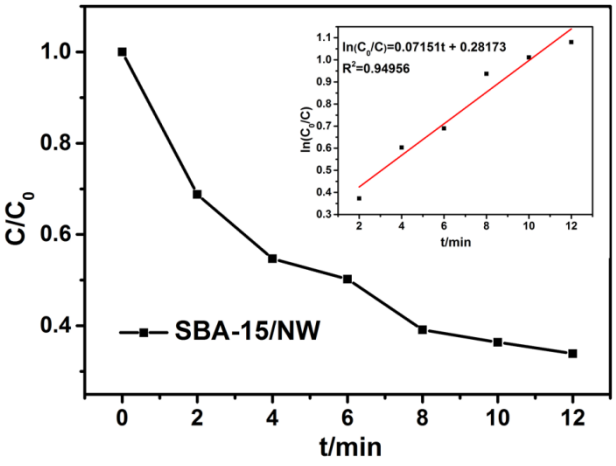


Figure. S4 Photocatalytic degradation curve of MB plotted from UV-Vis absorption spectra and the degradation kinetics curve (Inset). The degradation can be finished within 20 min, which is longer than that observed from SERS detection system. The difference should be caused by the difference between suspension and film photocatalysis systems.

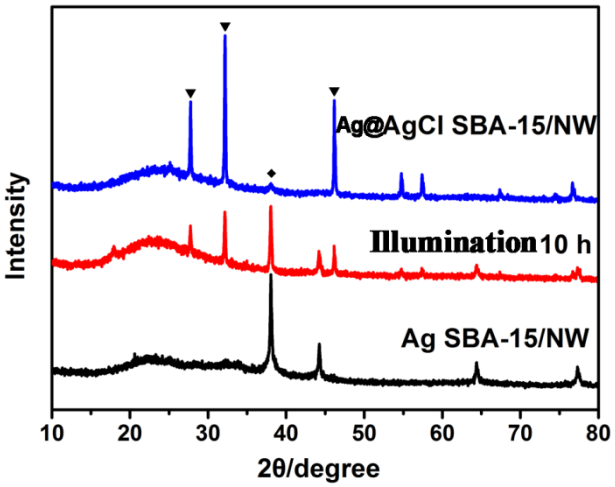


Figure. S5 Wide-angle XRD patterns of Ag SBA-15/NW (black), Ag@AgCl SBA-15/NW before (blue) and after photoillumination (red).