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Ag_3PO_4 photocatalysts loaded on uniform SiO_2 supports for efficient degradation of methyl orange under visible light irradiation

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Experimental

Synthesis of SiO_2 microspheres: SiO_2 microspheres were prepared by a Stöber sol-gel process. In a typical synthesis, a mixed solution containing ethanol (20 mL), concentrated ammonia solution (50 mL, 28 wt%) and distilled water (5 mL) was ultrasonicated fro 5 min and then mechanically stirred for 0.5 h. Subsequently, TEOS solution (5 mL) previously dissolved in ethanol (30 mL) was added dropwise to the above solution under continuous stirring at room temperature. After stirring for 24 h, the product was collected by filtration, washed repeatedly with distilled water and ethanol, and finally dried at 60 ^oC overnight.

Synthesis of Ag_3PO_4/SiO_2 composite photocatalysts: The Ag_3PO_4/SiO_2 composite was prepared by a simple precipitation method where the Ag_3PO_4 was formed via an ion-exchange process between Na_2HPO_4 and $AgNO_3$. In a typical process, 0.2 g as-prepared SiO_2 was dispersed in 20 mL high purity water and sonicated for 0.5 h. Then, a certain amount of $AgNO_3$ solution (0.023 M) was added to the above suspension under violent stirring. Subsequently, Na_2HPO_4 solution (0.014 M) was added dropwise to the above solution. After magnetically stirring for 2 h, the obtained

products were filtered, washed with distilled water and dried at 60 ^{o}C. The resulting materials with different Ag_3PO_4 contents are denoted as x%Ag_3PO_4/SiO_2, where x is the percentage by weight of Ag_3PO_4 (x = 0–30).

Characterization

X-ray diffraction patterns (XRD) were collected on a Rigaku MinFlex II equipped with Cu Kα irradiation. X-ray photoelectron spectra were recorded on an ESCALAB 250 photoelectron spectroscope (Thermo Fisher Scientific) with monochromatic Al Kα radiation (E = 1486.2 eV). Morphologies of the samples were observed by field emission scanning electron microscopy (FE-SEM) (Hitachi SU-8000). A Varian Cary 500 Scan UV/vis system was used to obtain the optical absorption spectra of the samples over a range of 200–800 nm. The specific surface areas of the samples were determined from the nitrogen adsorption data at liquid nitrogen temperature using the Barrett–Emmett–Teller (BET) technique on a Micromeritics ASAP 2000 surface area and porosity analyzer. The photoluminescence spectrum (PL) of the sample was performed on an Edinburgh Analytical Instruments F–4600 coupled with a time correlated single-photo counting system with excitation of 325 nm wavelength incident-light at room temperature. The zeta potentials were determined by dynamic light scattering analysis (Zeta sizer 3000HSA).

Photocatalytic activity

Photocatalytic experiments were performed in an aqueous solution at ambient temperature. A 300 W halogen lamp (Philips Electronics) equipped with a composited cut-off filter (400 nm < λ < 800 nm) was used as the visible light source. The system was cooled by a fan and circulating water to maintain at room temperature. Briefly, 80 mg of photocatalyst was suspended in 80 mL aqueous solution of MO (10 ppm). Prior to irradiation, the suspension was magnetically stirred in dark for

0.5 h to establish an adsorption–desorption equilibrium. A 3 mL aliquot was taken at several minutes intervals during the experiment and centrifuged to remove the powders. The residual concentration of dye was analyzed on a Beijing Puxi TU1801 spectrophotometer. The degradation percentage is reported as C/C_0, where C_0 is the concentration of initial MO, and C represents the corresponding concentration at a certain time interval.

The generation of hydroxyl radicals was investigated by the method of photoluminescence technique with terephthalic acid (PL–TA), in which a basic TA solution including 5×10^{-3} M TA and 0.01 M NaOH was added to the reactor. The collected solution was centrifuged and then measured on a F–4600 type fluorescence spectrophotometer. The excitation wavelength used was 312 nm.

Results

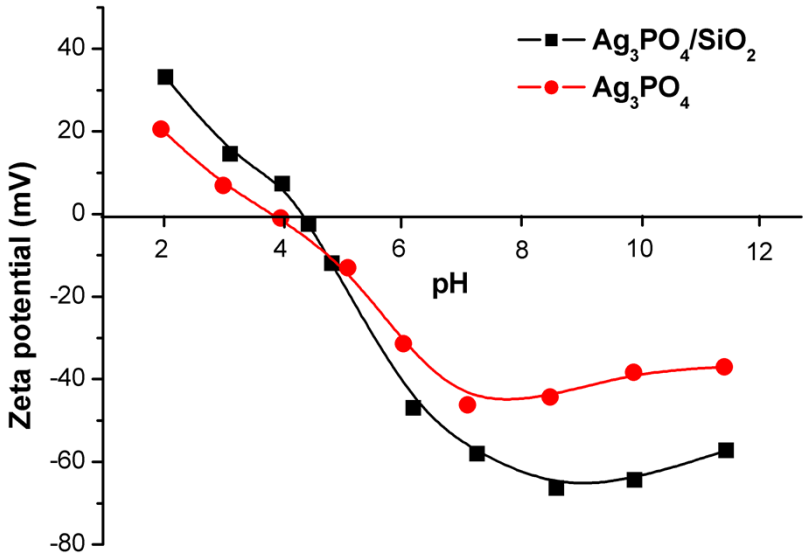

Fig. S1 The zeta potentials of Ag_3PO_4 and 20%Ag_3PO_4/SiO_2 composite as a function of pH in deionized water.

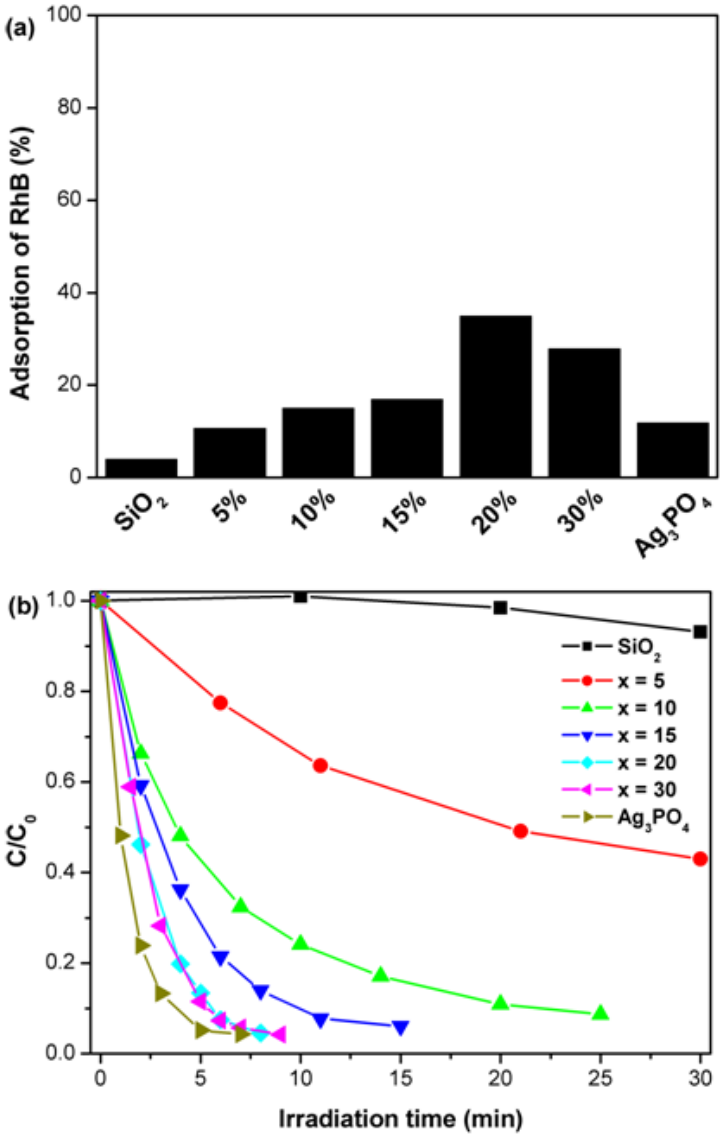

Fig. S2 The adsorption (a) and degradation (b) curves of RhB over different photocatalysts under visible light irradiation.