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To improve the photocatalytic activity of ZnO in semiconductor photocatalysis, ZnO-SnO₂ coupled catalysts and Ag/ZnO-SnO₂ catalysts with different Ag contents (1–5%) were prepared through the coprecipitation method. The samples were characterized by X-ray diffraction, UV-Vis diffuse reflectance spectroscopy, specific surface area, and transmission electron microscopy combined with energy dispersive spectroscopy. The degradation rate of methyl orange shows that the photocatalytic activity of ZnO-SnO2 coupled catalyst prepared at pH 7 was higher than that of the coupled catalyst prepared at pH 10. After Ag was loaded on the surface of coupled catalysts prepared at pH 7, the photocatalytic performance of catalysts was greatly improved. The optimum Ag loading amount was found to be around 3%. The activity of 3%Ag/ZnO-SnO₂ catalyst was higher than that of the pure ZnO catalyst and the ZnO-SnO₂ coupled catalyst prepared at pH 7 by 84 and 88%, respectively. Our study demonstrates that the combination of noble metal loading and the semiconductor coupling on the surface of a semiconductor catalyst would be an effective way to improve the photocatalytic activity of catalysts.