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***Original Paper***

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**INTRODUCTION**

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**EXPERIMENTAL**

**Materials**

The starting materials, fructose (D-(-)-type, Sigma­Aldrich, 99%), silver nitrate (AgNO3, AR, QRëCTM), titanium tetraisopropoxide (TTIP, Sigma­Aldrich, 97%) and absolute ethanol (EtOH, HmbG® Chemicals) were used as the precursor for carbon microspheres, silver, and TiO2 microspheres, respectively.

**Synthesis of Ag/TiO2 microspheres**

The Ag/TiO2 microspheres, where the location of Ag nanoparticles are attached on the external surface of hollow TiO2, were prepared by deposition-precipitation method using urea and previously synthesized hollow anatase TiO2. The synthesis of hollow TiO2 spheres is given in details in reference ([Baharvand *et al.*, 2014](#_ENREF_5)). TiO2 (0.1 g) was added to a 10 mL aqueous solution containing AgNO3 (Ag 1 wt/wt %) and 0.25 g urea. The suspension was then refluxed at 80 °C and kept at this temperature for 4 h under continuous stirring. The sample was then collected by centrifugation, washed repeatedly with deionized water, dried for 12 h at 80 °C and then calcined at 300 °C for 4 h.

**Synthesis of Ag@TiO2 microspheres**

The Ag@TiO2 particles, silver loaded hollow anatase TiO2 particles with the location of Ag are inside the hollow TiO2 structure is by template method. 2.4 mL of aqueous solution AgNO3 was added dropwise into 40 mL of aqueous fructose solution (0.5 M) under vigorous stirring. After stirring for 20 min, the solution was transferred and sealed in a Teflon-sealed autoclave. The autoclave was placed in an oven at 160 °C for 5 h, yielding Ag@C microspheres. 2.6 mL of TTIP was added to a colloidal suspension of Ag@C under vigorous stirring and then the mixture was aged under ambient conditions for one day. The product was collected by centrifugation and washed with ethanol, then dried at 60 °C in an oven. Ag@TiO2 were finally obtained after the sample was calcined at 600 °C for 3 h in a furnace.



**Fig. 1** Schematic diagram for preparation of TiO2@Ag and Ag/TiO2 microspheres.

**RESULTS AND DISCUSSION**

**Physicochemical properties of Ag@TiO2 and Ag/TiO2 microspheres**

FESEM images of Ag@TiO2 and Ag/TiO2 microspheres are shown in Fig. 2. It can be seen from the Fig. 2(a) that the synthesized particles are spherical in shape with an average diameter of about 536 nm. The white arrow in Fig. 2(a) shows partly broken sphere, thus confirming that the spheres are hollow inside. Fig. 2(b) shows Ag/TiO2 spheres of about 670 nm in average diameter with a rough surface.



**Fig. 2** FESEM images of (a) Ag@TiO2 and (b) Ag/TiO2 microspheres.

Further information on the elemental composition of the samples can be provided by EDX analysis. Fig. 4 shows the EDX spectrum of synthesized Ag@TiO2 while the elemental composition of Ag@TiO2 sample is summarized in Table 1. The EDX results confirmed the presence of Ti, O and Ag elements in the microspheres. The spectrum (Fig. 5) shows that Ag/TiO2 microspheres consist of Ti, O and Ag elements with the corresponding percentages given in Table 2. The distribution of Ag in heterostructure was confirmed by the EDX elemental mapping as shown in Fig. 6. The EDX elemental mapping clearly shows the presence of Ag particles on TiO2, which should correspond to the darker small spots in HRTEM image (Fig. 3(d)).

The amount of Ag in Ag@TiO2 and Ag/TiO2 samples was determined by XRF measurement. Table 3 and 4 illustrate Ag content in the prepared samples. Experimental Ag mass % measured by XRF technique is in good agreement with the calculated one, revealing the effectiveness of the current preparation methods.

**Table 3** Elemental analysis by XRF for Ag@TiO2 microspheres.

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| --- | --- |
| **Component** | **Mass %** |

|  |  |
| --- | --- |
| SiTiO2TiAgMgAl | 0.165.333.31.30.10.1 |

**Evaluation of photocatalytic activity**

The photocatalytic activity of Ag modified hollow anatase TiO2 microspheres in the environment was tested out in the photodegradation of imazalil sulphate and its imazalil sulphate photodegradation efficiency was compared with that of commercial TiO2. The photodegradation efficiency was calculated using the Eq. (3).

*Photodegradation efficiency (%) = (1- Ct/C0) × 100 (3)*

Where C0 is the concentration of the compound before illumination and Ct is the concentration of solution after time t. Fig. 12 shows the photodegradation efficiency of imazalil sulphate. The photodegradation efficiency was in the following order: Ag/TiO2 > hollow anatase TiO2 > Ag@TiO2 > commercial anatase TiO2. The highest efficiency was shown by Ag/TiO2 photocatalyst. 5 h of irradiation resulted in degradation of 27.9% for imazalil sulphate. From Fig. 12, it can be seen that the presence of Ag nanoparticles on the surface of hollow TiO2 gave a more efficient photodegradation than the other photocatalysts, which demonstrates the efficiency of Ag nanoparticles in reducing the recombination rate of the electron-hole pair. Even though the prepared photocatalyst contains Ag in the metallic form, it can act as an electron sink ([Wang *et al.*, 2013](#_ENREF_43)). When the reaction mixture is irradiated by UV, the photoexcited electrons from the valence band of TiO2 gets captured by the Ag, allowing the generated hole to decompose the pesticides ([Chiarello *et al.*, 2008](#_ENREF_10); [Wang *et al.*, 2013](#_ENREF_43)). Electron migration from the conduction band of TiO2 to the metallic Ag particles is feasible since the Fermi level of TiO2 is higher than that of Ag metal ([Wang *et al.*, 2013](#_ENREF_43)). When the quantity of Ag is little and the photogenerated electrons effectively migrate to Ag, better separation of electrons and holes would be accomplished. The role of Ag is to increase separation of the electron-hole pair, decreasing recombination rate of the electron-hole pair and in turn, enhanced the photocatalytic efficiency of the Ag/TiO2 photocatalyst.

**CONCLUSION**

Hollow anatase TiO2 containing Ag, in the different location, has been successfully synthesized. This was proven by the images obtained using TEM. Apart from that, the existence of Ag was also confirmed by XRF and EDX. DR UV–Vis spectra showed the existence of absorbance peak for Ag at around 404–650 nm. The Ag’s particles size was measured by TEM. The results showed that the size of Ag particles inside the hollow anatase TiO2 sample was larger (45 nm) than its particles size when it was located outside (9–20 nm).

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