

Design of visible light sensitive (Fe, Ti) - containing mesoporous silica photocatalyst using a photo-assisted deposition (PAD) method

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The transition metal oxide moieties in metallosilicate catalysts are considered to be highly dispersed at the atomic level and also well-defined catalysts which exist in the specific structure of the framework. These transition metal oxide moieties such as tetrahedral coordinated titanium oxide moieties incorporated and isolated within silica matrices of zeolite and mesoporous silica have been named as “single-site photocatalysts” . Various methods for nanoparticles preparation based on chemical reduction, sonication, -ray radiolysis, UV irradiation, thermal decomposition, vapor deposition, electrochemical synthesis, and microwave assisted synthesis, have been developed . All of the above methods are in principle more useful to produce colloids and clusters in the nanoscale. In order to develop the efficient and practical catalysts, a novel strategy is required to provide nano-sized metallic nanoparticles with well-controlled size and dispersability on solid support . Together with such strategy, it is also desired to manipulate the single-site photocatalyst band gap to acquire single-site photocatalyst ability to work under UV and/or visible light radiation. Under UV and/or visible irradiation, these single-site photocatalysts could form charge transfer excited state which can show the highly active and selective photocatalytic performance. Although the previous studies for the utilization as photocatalyst

have mainly been focused on their photocatalytic activity, the coupling of single-site photocatalyst together with the conventional catalysts such as nano-sized metal catalysts has not been investigated so far. In the present study, nano-sized Fe metal has been deposited on the Ti-containing mesoporous silica (Ti-HMS) using the photo-assisted deposition (PAD) method with the aim to control the band gap of Ti-HMS and to produce more efficient single-site photocatalyst.

The crystalline phase of Ti-HMS, and PAD-Fe/Ti-HMS nanoparticles were determined by powder XRD and the obtained patterns are shown in Fig. 1. All the patterns show the existence of the specific peak of hexagonal mesoporous silica (HMS) in the low angle at around 2–3°. These results suggest that the mesoporous structure remained after the Fe doping for Ti-HMS by the photo-assisted deposition. The X-ray diffraction patterns of Fe doped Ti-HMS exhibit no peak corresponding to crystalline phase at the higher angle, indicating that Fe and Ti-oxide moieties are highly dispersed in the mesoporous structure, while no other phases are formed.

The TEM images of PAD-Fe/Ti-HMS samples are shown in Fig. 2. It can be seen from TEM micrographs that there is a continuous reduction in the particle size of Ti-HMS with increment of Fe wt %. The reduction of Ti-HMS particle size takes place due to doping effect where

the Fe is linked to the Ti-HMS surface and consequently prevents the growth of its particles. Furthermore, the Fe nanoparticles were evenly distributed within Ti-HSM matrix and this can be noticed with different Fe wt %. This is attributed to, in PAD method, Fe can be only deposited on the highly dispersed Ti-oxide in the framework of silica and Fe can be anchored by Ti-oxide.

It is clarified that the band gap of Ti-HMS can be well controlled by doping with Fe at different wt %. X-ray diffraction patterns confirm that with Fe doping for Ti-HMS, the mesoporous structure was maintained. DR-UV suggested the insertion of Fe within Ti-HMS intra-framework till 2 wt %; thereafter with increment of Fe wt% the Fe-TiO₂ extra-framework begin to be formed. DR-UV also indicates the gradual narrowing of the band gap of Ti-HMS by doping with 1-4 wt% Fe. Finally TEM micrographs showed the homogenous distribution of Fe within Ti-HMS matrix.

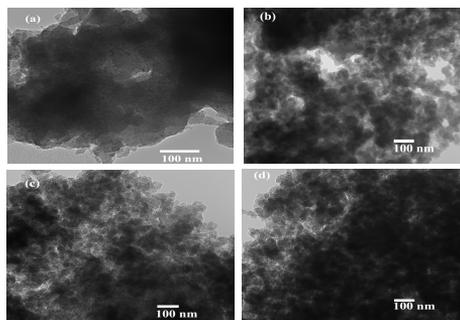


Fig.2. The TEM micrographs of the PAD-Fe/Ti-HMS samples after H₂ treatment at 473 K.

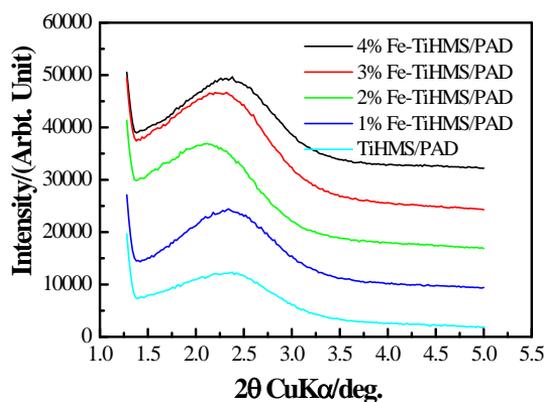


Fig.1. XRD patterns of the PAD-Fe/Ti-HMS samples.