

A Facile Method for Preparing TiO₂ Hollow Spheres

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Abstract: In this work, TiO₂ hollow spheres (THs) with an average diameter of 200 nm were synthesized by molten salt synthesis followed by thermal treatment in air. Carbon black powders were used as both a reactant and reaction template. Morphologies and phase composition of the product powders were examined using SEM and XRD, respectively. The results showed that the as-synthesized products composed mainly of anatase phase, having hollow structure with rough surfaces of the shells due to the presence of TiO₂ nanocrystals. Water contact angle of the product particles were also examined and found that they were hydrophilic.

Keywords: Titanium dioxide, hollow spheres, molten salt synthesis, biomaterials

INTRODUCTION

Titanium dioxide (TiO₂), a semiconductor with a band gap of 3.2 eV, has been widely applied in chemical industry, electronic industry, environmental protection, and medical science [1–2]. In order to improve their performances, various structures, including nanoparticles, nanotubes, and nanorods, have been fabricated successfully. Among these structures, hollow structure has increasingly attracted interest because of its higher specific surface area, lower density, better permeation and stronger light-harvesting capacity compared to solid ones [3–4], which are favored for many applications; catalysis, photonic materials, sensing device, biomedical diagnostics, drug delivery and capsulation [5].

Up to now, many synthetic strategies have been devoted to synthesizing THs, including, sol-gel, pyrolysis, microemulsion and self-assembly method [6–8]. However, these methods always need to add surfactant or organic solvent, which perhaps introduces impurities to the products and increase the cost. In this work, a facile and simple method to produce THs was introduced. Molten salt synthesis (MSS), via a direct reaction of titanium powder with carbon black particles, was simply used to firstly produce core-shell structure of TiC-coated carbon particles (TCBs), followed by thermal treatment of the TCBs in air. Phase composition and morphologies of as-synthesized THs were characterized and the results were discussed.

MATERIALS AND METHODS

Firstly, TCBs were prepared by MSS technique. The synthesis method and starting materials used for the synthesis here were based on our previous work [9], except particle size of the carbon black was approximately 200 nm. Secondly, the obtained TCBs were heated at 500°C for 30 min in air to oxidize TiC shells and remove carbon cores, and finally THs was acquired.

RESULTS AND DISCUSSION

Fig. 1 (a) demonstrates an XRD curve of as-synthesized powders that they composed mainly of anatase phase. A minor proportion of rutile was also observed. No evidences of other impurities or

unreacted TiC were seen, indicating high purity of the product and the complete conversion of TiC to TiO₂ due to reacting with oxygen in air. Fig. 1(b) revealed clearly morphologies and surface structure of the product powders that they were spherical-like shape having diameter of approximately 200 nm with hollow structure. The shells were rough due to the presence of many TiO₂ nanoparticles, having approximately 25 nm in diameter. The as-synthesized THs possessed water contact angle of 65.13° indicating hydrophilicity of these hollow particles.

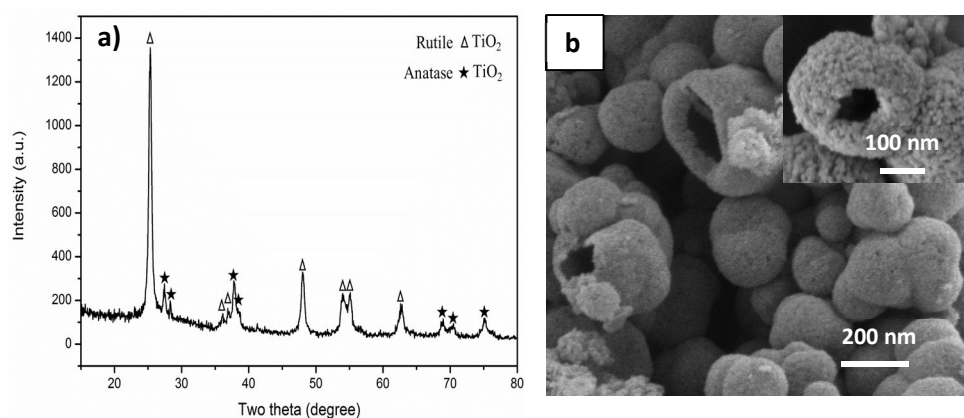


Fig. 1. (a) XRD pattern and (b) SEM image of as-synthesized particles (inset: high magnification image) of as-synthesized hollow TiO₂ spheres after oxidation at 500 °C for 30 min.

CONCLUSIONS

Two mechanisms were involved here: One is template growth mechanism, fabricating core-shell structure of TCBs during MSS, and the other is oxidation of those TCBs to convert TiC shells to TiO₂ and to remove carbon cores. Rough surface with hydrophilic property of the as-synthesized THs suggested their potential applications as catalyst, environmental protection material, and biomaterials, in a particular for inducing of mineralization in bone healing.

REFERENCES

- [1] Ni M, Leung MKH, Leung DYC, Sumathy K. *Renew Sust Energ Rev.*, 2007, **11**(3), 401.
- [2] Manera MG, Cozzoli PD, Curri ML, Leo G, Rella R, Agostiano A, Vasaneli L. *Synth Met.*, 2005, **148**(1), 25.
- [3] Jiang L, Zhong YJ, Li GC. *Mater. Res. Bull.*, 2009, **44**(5), 999.
- [4] Shchukin DG, Caruso RA. *Chem. Matter.*, 2004, **16**(11), 2287.
- [5] Gu Y, Chen L, Li Z, Qian Y, Zhang W. *Carbon.*, 2004, **42**(1), 235.
- [6] Hu YX, Ge JP, Sun YG, Zhang TR, Yin YD. *Nano Lett.*, 2007, **7**(6), 1832.
- [7] Li GL, Kang ET, Neoh KG, Yang XL. *Langmuir Lett.*, 2009, **25**(8), 4361.
- [8] Ren TZ, Yuan ZY, Su BL. *Chem. Phys. Lett.*, 2003, **374**(1) 170.
- [9] Soe HN, Khangkhamano M, Sangkert S, Meesane J, Kokoo R. *Mater. Lett.*, 2018, (In Press)