One-Step Hydrothermal–Electrochemical Route to Carbon-Stabilized Anatase Powders

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Black carbon-stabilized anatase particles were prepared by a simple one-step hydrothermal–electrochemical method using glucose and titanium citrate as the carbon and titanium source, respectively. Morphological, chemical, structural, and electrochemical characterizations of these powders were carried out by Raman spectroscopy, Fourier-transform infrared spectroscopy, x-ray diffraction, scanning electron microscopy, and cyclic voltammetry. It was revealed that 200-nm carbon/anatase TiO_2 was homogeneously dispersed, and the powders exhibited excellent cyclic performance at high current rates of 0.05 V/s. The powders are interesting potential materials that could be used as anodes for lithium-ion batteries.

Key words: Carbon, anatase, complex, hydrothermal–electrochemical method, electrochemical

INTRODUCTION

Titanium dioxide exists in three crystal phases: anatase, rutile, and brookite. Brookite is the least studied of the three because it is the most difficult to obtain in pure form. The rutile phase has the highest refractive index and ultraviolet absorption among the titania phases, thus being employed in pigments, paints, and ultraviolet absorbents.^{1,2} Because of its excellent photocatalytic activity, chemical stability, and high activity, the anatase phase is an effective material for applications such as photocatalysis, optical coatings, dye-sensitized solar cells, and lithium-ion batteries.^{3–7} Fine crystalline anatase can also be used as a safe anode material in lithium-ion batteries due to its higher Li-insertion potential (1.5 V to 1.8 V versus Li⁺/Li) in comparison with commercialized carbon anode materials. However, its low intrinsic electrical conductivity and poor cycling performance have limited its application. These deficiencies could be avoided by using carbon-stabilized anatase particles.^{8,9} Thin carbon layers can alleviate the aggregation and crystal growth of TiO₂ particles, suppress the phase transformation from anatase to rutile at high temperatures, and also increase the electrical conductivity of anatase particles.¹⁰ Carbon-stabilized anatase has been prepared directly by using anatase as a titanium source and organic contaminants as a carbon source through decomposition of the organic contaminants to form carbon.^{10–15} However, to the best of our knowledge, direct, one-step synthesis of C/anatase complex materials from mixtures of organic carbon and titanium solution has not been reported so far.

In this paper, we report the preparation of an anatase/carbon complex obtained by a hydrothermal-electrochemical method in one step. Anatase/C materials were synthesized under mild aqueous conditions. The anatase/C obtained in this way could be dispersed well in aqueous solution, and there were functional groups on its surface, which could facilitate further modification in future applications.

EXPERIMENTAL PROCEDURES

Ta substrates with dimensions of $10 \text{ mm} \times 10 \text{ mm} \times 0.1 \text{ mm}$ and 99.9% purity were mechanically polished and degreased with acetone using an ultrasonic cleaner. Titanium citrate and glucose of

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