Synthesis of nanosized Y-type TiOPc by a high gravity method

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Oxotitanium phthalocyanine (TiOPc) is an ideal photoconductive material and electrophotographic photoreceptor since it exhibits remarkable absorption and photosensitivity properties in the visible and near-infrared wavelength region, which lead to the generation of free carriers with a high quantum yield [1]. It was reported that Y-type TiOPc had the most excellent photosensitive properties among five typical crystal forms of TiOPc [2–7].

In conventional routes, TiOPc of other crystal forms are usually synthesized and then transformed to Y-type TiOPc by processes such as thermomechanical, solvent, acid treatment and mechanical grinding. Solvent treatment is preferred among these processes due to its convenience and high efficiency where TiOPc is subjected to crystal transformation in the presence of water, aromatic compounds, alcohol, ketone, ether and their derivatives. Since Chen *et al*. [8] reported that photoconductivity of TiOPc depended on particle size, many works have been directed to obtaining nanosized Y-type TiOPc by different methods.

In this paper, Y-type TiOPc is synthesized under high gravity environment created by a rotating packed bed (RPB) reactor, which can greatly intensify micromixing and mass transfer between fluids. Advantages of the high gravity method in synthesizing nanosized inorganic materials have been reported in our previous works [9, 10].

The experimental set-up for the synthesis of Y-type TiOPc is shown in Fig. 1. Ten grams of raw TiOPc, synthesized and purified according to the literature [11], was dissolved in 200 ml of sulfuric acid (98%, wt.) under stirring and then filtrated to obtain a purified TiOPc solution. The crystal transformation of TiOPc was conducted in a RPB installation, where 3000 ml of ethanol, 300 ml of *n*-butylalcohol and 60 ml of chlorobenzene were added into a solvent tank and kept circulation between RPB and the solvent tank via pumping. Meanwhile, ice water was pumped through the system to maintain the reaction temperature below 10 °C. Sulfuric acid solution of TiOPc was added dropwise into RPB from a flask to mix vehemently with the solvents in the packing part of RPB to form Y-type TiOPc. Finally, the as-prepared Y-type TiOPc was filtrated and washed with deionized water, acetone and hexane, and subsequently dried at 100 °C for 1 hr and 40 °C for 4 hr to obtain nanosized Y-type TiOPc powder.

Fig. 2 shows X-ray diffraction (XRD) (Siemens D500D, Germany) pattern of the as-prepared Y-type TiOPc. The most intense diffraction peak is observed at $2\theta = 27.3°$, which is in agreement with the main peak of Y-type TiOPc [12, 13]. Three other peaks appearing at $2\theta = 9.6°$, $14.7°$ and $24.1°$ also correspond to Y-type TiOPc structure. The results reveal that Y-type TiOPc can be obtained by the high gravity method.

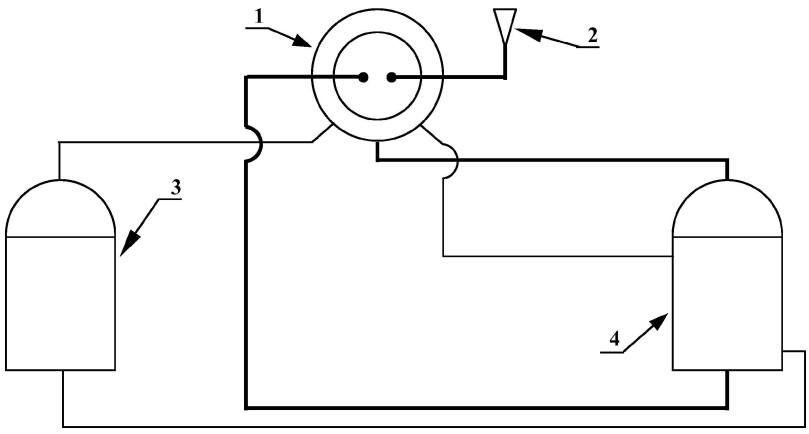

Figure 1 Experimental set-up: (1) RPB reactor, (2) flask filled with TiOPc raw materials, (3) ice water tank, (4) solvent tank.

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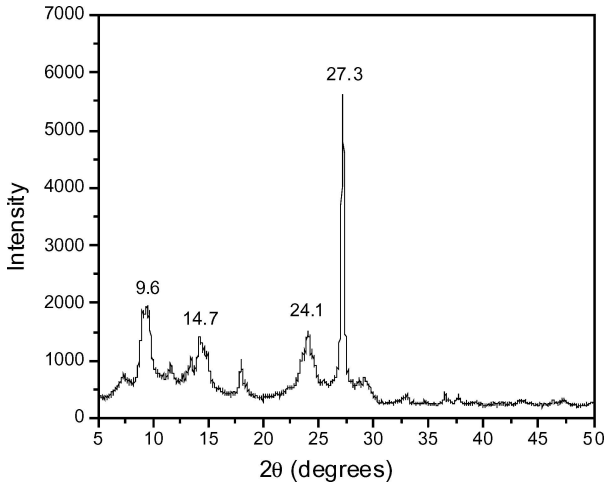

Figure 2 XRD pattern of Y-type TiOPc synthesized by the high gravity method.

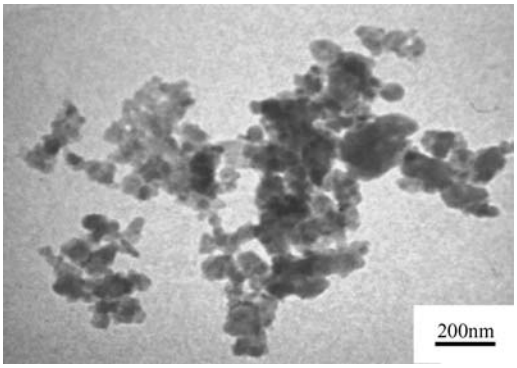

Figure 3 TEM image of Y-type TiOPc synthesized by the high gravity method.

Fig. 3 demonstrates the transmission electron microscope (TEM) (Hitachi-800, Japan) image of Y-type TiOPc, indicating that Y-type TiOPc synthesized in RPB has a size distribution of 30–50 nm with slight aggregation. BET surface area (Micromeritics ASAP 2010 Analyzer, USA) of the same sample was measured as 52.3 m^2/g. From TEM and BET results we reach the conclusion that nanosized Y-type TiOPc can be synthesized in a RPB reactor.

In summary, a novel route of synthesizing nanosized Y-type TiOPc by a high gravity method is presented in this paper. Y-type TiOPc with an average particle size of 30–50 nm was fabricated using a RPB reactor. This method may be promising for the mass production of Y-type TiOPc thanks to low cost and convenience.

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