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Fabrication of highly dispersed NiTiO₃@TiO₂ yellow pigments with enhanced NIR reflectance



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ABSTRACT

The highly dispersed NiTiO $_3$ @TiO $_2$ yellow pigments with core-shell structure were prepared through calcinations of precursors obtained from the precipitation of Ni $^{2+}$ on the surface of TiO $_2$ particles. The synthesized pigments were characterized by XRD, SEM, TEM, UV-vis-IR spectroscopy. The results showed that the pigments were consisted of TiO $_2$ core and outer ilmenite NiTiO $_3$ shell. UV-vis diffuse reflectance spectroscopy results showed the enhance reflection peak at \sim 580 nm of the NiTiO $_3$ @TiO $_2$ pigments result in a better yellow color than that of the pure NiTiO $_3$ pigments. Compared with pure NiTiO $_3$ pigments, the NiTiO $_3$ @TiO $_2$ pigments also presented a much higher NIR reflectance. The mechanism analyzed results indicated that the reason was attributed to the special core-shell structure. The solar reflective coating colored by the NiTiO $_3$ @TiO $_2$ pigments reduced the inner surface temperatures of glass plates by approximately 8.7 °C.

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1. Introduction

Intense solar radiation can potentially raise temperature inside buildings owing to its exterior surfaces absorb solar energy, such unwanted heating increases cooling loads [1]. The heat producing region of the solar spectrum lies in the NIR range (700–2500 nm) corresponding to 52% of the solar radiation energy, whereas 5% can be assigned to UV (200–400 nm), and 43% to the visible range (400–700 nm) [2]. Thus, the highly NIR reflective "cool" coating can reflect a significant fraction of the solar energy that arrives as NIR radiation [3], thereby reducing the heat gain at exterior surface of building. Consequently, the "cool" coating can improve internal thermal comfort in building without air conditioning.

Recently, the inorganic pigments with highly NIR reflection are widely used as cool materials for building roofs and facades [4,5]. ${\rm TiO_2}$, a white pigment with a high solar reflectance of about 87%, is currently regarded as the best pigment for coating materials. However, the nonwhite pigments become increasingly necessary because the aesthetics of color coatings are usually preferred. Zhang [6] reported a convenient method to prepare color "cool" coating by mixing color pigments with ${\rm TiO_2}$ pigments. Due to ${\rm TiO_2}$ pigment with higher NIR reflection, the NIR reflectance of the color pigments was enhanced. However, the color intensity of

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color pigments was reduced. Therefore, it is necessary to improve the NIR reflectance of color pigments and simultaneously hold up or enhance their color intensity. The core-shell structure, with an inner core encapsulated by outer shell, is considered a good platform for the fabrication of high performance function materials [7,8]. The core-shell structure not only can improve chemical-physical performance of function materials, but also can effectively prevent interaction between the inner core and environment.

Nickel titanate (NiTiO₃), a traditional yellow pigment, receive considerable interesting owing to its highly visible opacity and high solar reflectance in near-infrared (NIR) radiation [3,9]. In particular, incorporation of NiTiO₃ into TiO₂ can be expected to enhance their NIR reflectance. In this paper, we synthesized highly dispersed NiTiO₃@TiO₂ yellow pigments with core-shell structure through sol-precipitation methods. The thermal insulation properties of coatings colored with the NiTiO₃@TiO₂ yellow pigments were investigated.

2. Experimental

The NiTiO₃@TiO₂ yellow pigments with core-shell structure were prepared by a two step process. In a typical synthesis, 5 mL of TBOT were added dropwise to the mixture solution of 300 ml of ethanol and 1.0 mL of KCl aqueous solution (0.4 mM) with continuous stirring. Having been stirred for 2 h, the obtained white suspension was collected by centrifugation and then dispersed into 200 mL distilled water. Subsequently, a desired amount of Ni

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 $(NO_3)_2 \cdot 6H_2O$ and $CO(NH_2)_2$ were added to the obtained suspension. After fully dissolved, the suspension was continuous stirred for 6 h under the 80 °C water bath. Then, cooling down to room temperature, the precipitates were collected by centrifugation and rinsed sequentially with ethanol and water for several times. The green powders were obtained by drying the precipitates at

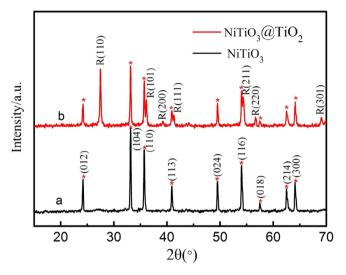


Fig. 1. XRD patterns of as prepared pure NiTiO $_3$ and the NiTiO $_3$ @TiO $_2$ pigments calcined at 750 °C. * and *R* denote the ilmenite NiTiO $_3$ and rutile TiO $_2$ respectively.

 $60 \, ^{\circ}\text{C}$ overnight. Finally, the NiTiO₃@TiO₂ yellow pigments was obtained by calcinations the powders at 750 $^{\circ}\text{C}$ for 3 h. For a comparison, the pure NiTiO₃ yellow pigments were prepared by sol-gel route according to the literature [9]. None of the samples were further milled or sieved.

The phases of the products were characterized by XRD (D/max-2200, Japan) using Cu Ka radiation (λ = 0.154 nm). The morphology analyses were performed by scanning electron microscopy (JSM-6700F, JEOL Japan) operated at 5 kV and transmission electron microscopy (JEM 2010, JEOL, Japan) operated at 200 kV. UV–vis diffuse reflectance spectroscopy and Infrared diffuse reflectance spectroscopy of samples were performed on ultraviolet–visible–infrared (UV–vis-IR) spectroscopy (Cary 5000, Varian, Australia) with an integrating; BaSO₄ was used as a reference sample.

To investigate the thermal insulation properties of the solar reflective coatings colored with yellow pigments, 0.2 g pigments were dispersed in 10 g polyurethane (PU) paint with ultrasonic treatment for 5 min to obtain well-distributed colored paints. The circular colored coating was obtained by curing the paint in a 9 cm Petri dish. The thermal insulation effects of the colored coating were measured by an indigenously designed device. The schematic of the measuring device was shown in Fig. 1s, and the detail experimental produce of thermal insulation test were described in supplementary information.

3. Results and discussion

Fig. 1 displays XRD patterns of as-prepared pure NiTiO₃ and NiTiO₃@TiO₂ pigments after calcinations. As shown in curve a, all

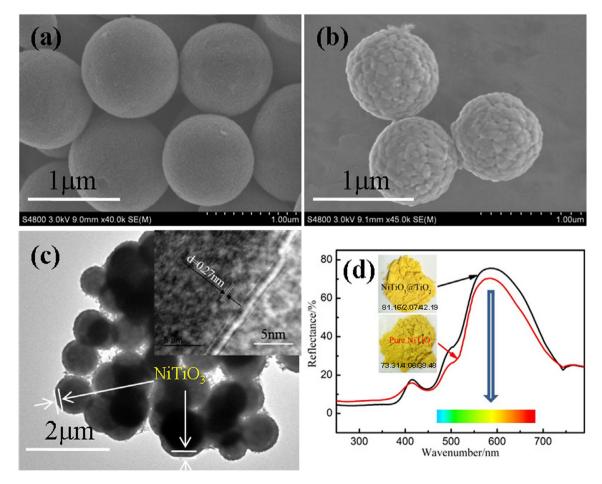


Fig. 2. SEM and TEM images of as-prepared TiO₂ (a) and NiTiO₃@TiO₂ yellow pigments (b and c); and UV-vis diffuse reflectance spectroscopy of pure NiTiO₃ and the NiTiO₃@TiO₂ pigments (d). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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