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Facile synthesis of tunable polymer spheres for encapsulation pigment application

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ABSTRACT

Polymer spheres with tunable particle size were synthesized by hydrothermal method derived from sucrose. Afterwards, zircon was coated on their surface to form the zircon encapsulated carbon ($C@ZrSiO_4$) black pigment after calcination. The corresponding relationship between the particle size of polymer spheres and the chromaticity of $C@ZrSiO_4$ pigments was investigated. The results revealed that the chromatic performance of $C@ZrSiO_4$ pigment can be significantly improved by decreasing the colorant size and the relationship between them was approximately linear. The pigment exhibits deep black hue, as L^* = 41.04, a^* = 1.01, b^* = 3.22, with the colorant size of 123 nm. Moreover, it also presents excellent thermal and chemical stability, indicating its potential candidate for ceramic decoration.

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1. Introduction

Ceramic pigment is a widely used coloring agent for glazes and ceramic bodies. With the development of the inkjet printing technology on the ceramic decoration, it has attracted a great deal of attention in the Cyan-Magenta-Yellow-Black (CMYK) printing system, which draws higher demand in the quality and aesthetic properties of ceramic pigments [1]. Nowadays, commercial black pigments are almost cobalt based oxides with spinel structure, e.g., $(Ni,Fe)(Fe,Cr)_2O_4$, $(Fe,Co)(Fe,Cr)_2O_4$ and $(Fe,Mn)(Fe,Mn)_2O_4$. Therefore, their disadvantages, i.e. high-cost, environment harmfulness and impure color, are limited their practical application. Carbon is a potential candidate owing to its high tinting strength, low cost and environment friendliness. Unfortunately, it is easily oxidized at high temperature, which hinders its utilization in ceramic decoration. In recently, it was reported that coating a dense and transparent zircon layer on its surface can improve its thermal and chemical stability greatly. Therefore, many research studies have been carried out on this topic. For example, Q.B. Chang *et al.* succeeded in synthesizing the zircon encapsulated carbon (i.e. $C@ZrSiO_4$) pigment by a sol-gel-spraying method at 1300 °C [2]. F. Zhao *et al.* prepared $C@ZrSiO_4$ gray pigment via the sol-gel modified method, using inorganic salt and carbon black powders as starting materials [3]. Particularly, organic materials, i.e. phenolic resin [4], cotton fiber [1] and tetraethoxysilane [5], were suggested to replace the carbon black powders to prepare the black encapsulation pigment. Due to a number of functional groups on the surface, the organics brought about better chromatic performance than the traditional colorant. However, the reports about the relationship between the colorant and the chromaticity of the encapsulation pigment were rare. In this paper, we synthesized the $C@ZrSiO_4$ encapsulation pigments via a non-hydrolytic sol-gel method, using the polymer spheres derived carbon as colorant. The effects of polymer spheres size on the phase composition, morohology and chromatic value of $C@ZrSiO_4$ encapsulation pigment were also investigated.

2. Experimental

Polymer spheres were prepared by hydrothermal method [6]. Briefly, sucrose aqueous solution was transferred into a Teflon-lined autoclave and maintained 180 °C for 5 h. The product was collected by centrifuging and washed with ethanol and water, respectively. The $ZrSiO_4$ sol was prepared by non-hydrolytic sol-gel (NHSG) method [1]. For $ZrSiO_4$ coating, the as-prepared polymer spheres (6 wt%) were dispersed in 1 M $ZrSiO_4$ sol by ultrasonication for 15 min. The mixture was dried at 110 °C in vacuum, and calcined at 900 °C for 2 h in N_2 atmosphere. Finally, the $C@ZrSiO_4$ pigments were achieved after recalcining at 700 °C for 2 h in air to eliminate the uncoated polymer spheres. The samples were characterized by X-ray diffraction (XRD, Bruker D8 Advance), scanning electron microscope (SEM, JSM-6700F), high-resolution

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transmission electron microscopy (HRTEM, JEM-2010), fourier transform infrared spectroscopy (FT-IR, Nicolet 5700), UV-visible (Lambda850), and colorimeter (WSD-3C).

3. Results and discussion

A schematic drawing for the encapsulation process of C@$ZrSiO_4$ pigment is shown in Fig. 1(a). The polymer spheres were prepared by hydrothermal method using sucrose as raw material. Meanwhile, $ZrSiO_4$ sol was prepared by NHSG process. After thermal treatment, the dense $ZrSiO_4$ layer was coated on the surface of the polymer spheres derived carbon to achieve the encapsulation pigment. The TEM image of the polymer spheres (Fig. 1(b)) indicates that it can be delicate controlled about 100 nm. After calcining, it was carbonized and then occluded in the matrix of $ZrSiO_4$ (Fig. 1(c)). The HRTEM image in Fig. 1(d) shows that the shell has high crystallinity and its interplanar spacing are about 0.43 nm and 0.33 nm, close to the {1 0 1} and {2 0 0} lattice spacing of $ZrSiO_4$, respectively [1]. The main chemical compositions of the

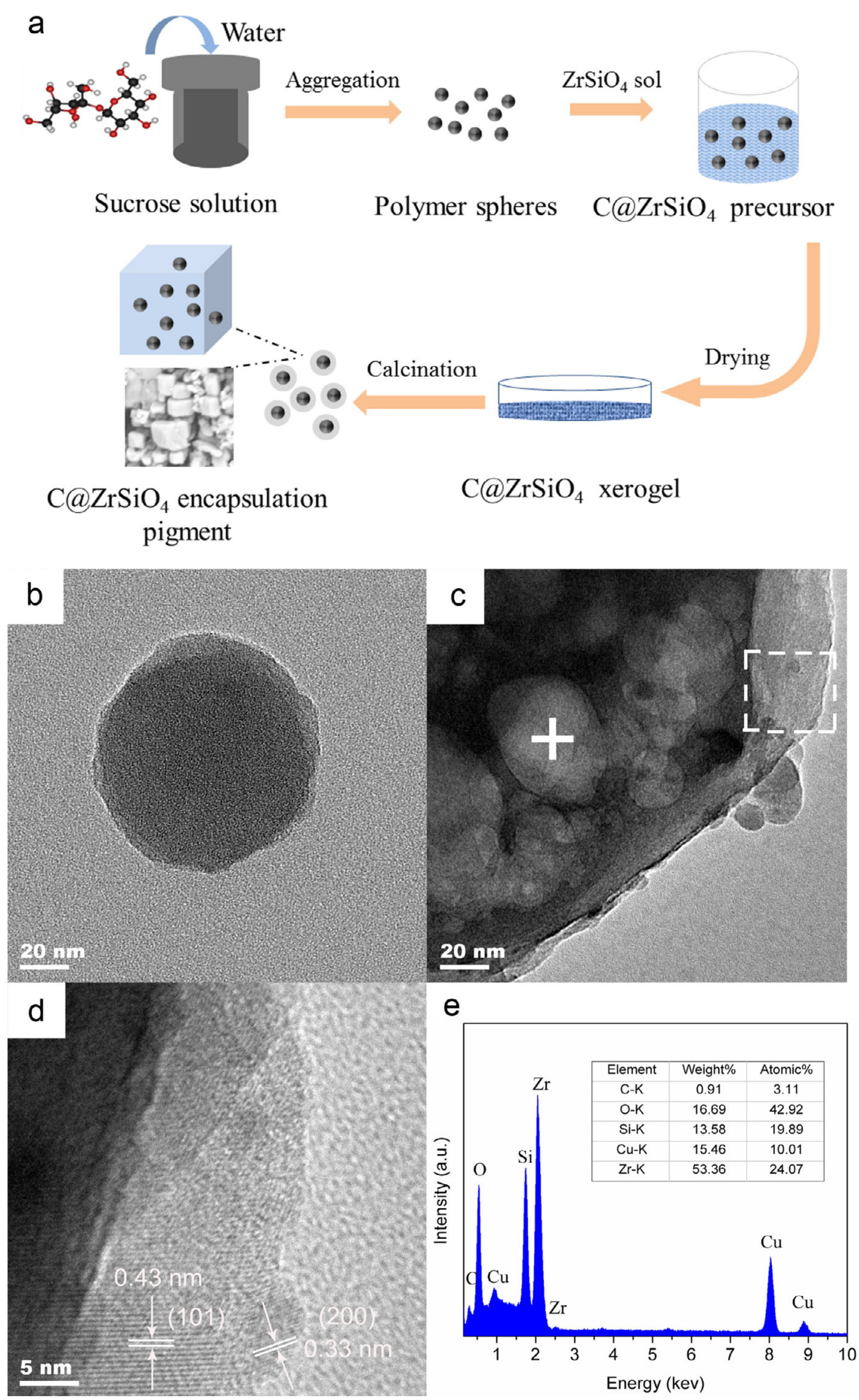

Fig. 1. (a) Schematic illustration of the $ZrSiO_4$ encapsulated carbon process, TEM images of (b) polymer spheres prepared at 180 °C for 5 h with 0.25 mol·L^{-1} sucrose solution, (c) and (d) C@$ZrSiO_4$ pigment, (e) EDS spectrum of the corresponding part taken from c.

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