



Enhanced thermal- and photo-stability of acid yellow 17 by incorporation into layered double hydroxides

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ABSTRACT

2,5-dichloro-4-(5-hydroxy-3-methyl-4-(sulphophenylazo) pyrazol-1-yl) benzenesulphonate (DHSB) anions, namely acid yellow 17 anions, have been successfully intercalated into Zn–Al layered double hydroxides (LDH) to produce a novel organic-inorganic pigment by a simple method involving separate nucleation and aging steps (SNAS), and the dye-intercalated LDH was analyzed by various techniques, e.g., XRD, SEM, FT-IR, TG–DTA and ICP. The *d*-spacing of the prepared LDH is 2.09 nm. Furthermore, the incorporation of the DHSB aims to enhance the thermal- and photo-stability of the guest dye molecule, for example, the less color change after accelerated thermal- and photo-aging test.

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1. Introduction

Pigments, as one kind of important additives, have been widely used for paints, inks and plastics [1]. According to Ceresana research's report, the worldwide market for inorganic, organic and special pigments had a total volume of ca. 7.4 million tons in 2006 and, furthermore, this volume is expected up to 9.8 million tons in 2016 at an increasing rate of 2.9% per year. Compared with inorganic pigments, organic pigments have brighter color, higher color strength, better transparency and higher chemical stability, but worse light fastness. The worse light fastness of organic pigments mainly results from their poor thermo- and photo-stability. Therefore, it is an important and interesting issue to explore novel pigments with high thermo- and photo-stability as required for practical applications.

Layered double hydroxides (LDH), one class of anionic layered clays, is usually described by a general formula $[M_{1-x}^{2+}M_x^{3+}(\text{OH})_2]^{x+}(A^{n-})_{x/n} \cdot m\text{H}_2\text{O}$ [often abbreviated as $M_y^{2+}M_x^{3+}-A$ LDH, $y=(1-x)/x$, $0.2 < x < 0.33$], where M^{2+} and M^{3+} are di- and trivalent metal cations in the brucite-like host sheet, and A^{n-} is the guest anions in the interlayer galleries [2,3]. Up to date, a lot of synthesis methods have been developed to prepare LDH materials with different chemical composition in the host sheet and various functional guest anions in the interlayer region, for

example, coprecipitation, anion-exchange, hydrothermal and rehydration [2,4]. Usually, the coprecipitation method is used to prepare LDHs containing simple guest anions (e.g., NO_3^- , Cl^-) as the precursor for the anion-exchange method to produce multifunctional LDHs containing complex functional anions [5,6]. On the basis of coprecipitation method, our laboratory has invented a method involving separate nucleation and aging steps (SNAS) to synthesize LDH materials with small particle size and narrow size distribution [7,8]. Compared with others, this method is faster and more facile, particularly more suitable for large batch product. Based on their structure characteristics: adjustable structure, tunable chemical composition and exchangeable interlayer anions, LDH materials have wide applications [9,10] as catalysts [2,11–14], IR absorption materials [15,16], photo functional materials [17–19], flame retardants [20], etc. Thus, it is possible to intercalate coloring dye anions into the interlayer galleries of LDHs to produce novel organic-inorganic composite pigments. These pigments may have brilliant color like organic pigments and relatively high thermo- and photo-stability thanks to the protection by the LDH host sheet.

A few of organic coloring agents (dyes and pigments) have been intercalated into the interlayer galleries of LDH materials, for example, acid blue 9 [21], methyl orange [22], blue dye molecules [23] and chromophore molecules [24]. Most of them have mainly investigated the assembling methods of anions and the corresponding structure. Few researchers, however, have paid special attention to the influence of the LDH structure on the tolerance of the organic anions towards thermal and photo aging

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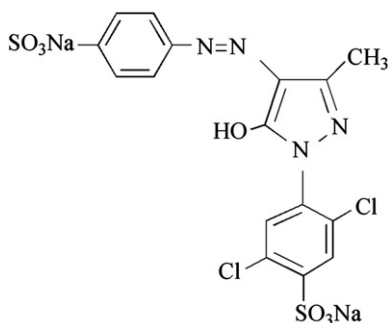


Fig. 1. Chemical structural formula of DDHSB.

[24]. In our group, we have intercalated some pigment and dye guest molecules (e.g., Red 48:2 [25], Red 52:1 [26], Mordant Yellow MY10 [27]) into the LDH materials and found that the LDH structure favors improvement of the thermal and photo-stability.

Disodium 2,5-dichloro-4-(5-hydroxy-3-methyl-4-(sulphophenylazo)pyrazol-1-yl) benzenesulphonate (abbreviated as DDHSB), namely acid yellow 17, one of three basic color dyes, is widely used for textile, paper, paint, medicine and cosmetic, see its chemical structural formula in Fig. 1. In this work, we mainly investigated the direct preparation of the DHSB intercalated LDH by the SNSA method and the corresponding thermal and photo-stability by accelerated aging techniques.

2. Material and methods

2.1. Material

All the chemicals, except for DDHSB, were A.R grade and used as received from Beijing Yili Fine Chemical Reagent Co. without any further purification. DDHSB (98 wt%) was recrystallized three times in water before use. Deionized water with conductivity less than 10^{-6} S cm^{-1} was freshly decarbonated by boiling before use in all of the synthesis and washing steps.

2.2. Preparation of ZnAl-DHSB-LDH

ZnAl-DHSB-LDH was directly synthesized by the SNSA method [7,8]. Typically, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (11.90 g, 0.04 mol), $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (7.50 g, 0.02 mol) and DDHSB (8.26 g, 0.015 mol) were dissolved in CO_2 -free deionized water to make a mixed salt solution A (100 mL); NaOH (4.80 g, 0.12 mol) was dissolved in CO_2 -free deionized water to form an alkali solution B (100 mL). Solutions A and B were simultaneously and slowly added into a colloid mill at a rotor speed of 3000 rpm and mixed for another 2 min. Then, the resulting slurry was aged at the refluxing temperature (102°C) for 8 h under a N_2 stream. The final product was collected by centrifuging (4200 rpm for 5 min each time), washing with water, ethylene glycol and N,N-dimethylformamide until the solution without color, and drying at 80°C to constant weight.

2.3. Analysis and characterization

Powder X-ray diffraction (XRD) measurements were performed on Shimadzu XRD-6000 X-ray powder diffractometer (Cu $K\alpha$ radiation, $\lambda=0.154$ nm) from 3° to 70° with a step length of 0.02° at a scanning rate of $0.02^\circ \text{ s}^{-1}$. High-resolution transmission electron microscopy (HRTEM) was carried out on a JEOL JEM-2100 microscope operated at 200 kV (point resolution, 0.19 nm and line resolution, 0.14 nm). FT-IR spectra were recorded on a

Bruker Vector 22 Fourier transfer infrared spectrophotometer using the KBr pellet method with a ratio of sample/KBr of 1:100 by weight. Thermogravimetry and differential thermal analysis (TG-DTA) curves were obtained on a PCT-IA instrument in the range of 30 – 700°C at a rate of $10^\circ\text{C min}^{-1}$ under air. Diffuse reflectance UV–vis absorbance spectra were collected on Shimadzu UV-2501PC with an integrating sphere attachment in the range of 200 – 800 nm using BaSO_4 as the reference. Elemental analysis was carried out on a Shimadzu ICPS-7500 inductively coupled plasma (ICP) emission spectrometer using concentrated nitric acid (65 wt%) as the dissolving agent. The color difference (ΔE) of materials after thermal- and photo-aging treatment was determined in terms of CIE 1976 $L^*a^*b^*$ with a TC-P2A automatic colorimeter [25].

3. Results and discussion

3.1. Structure and morphology of ZnAl-DHSB-LDH

Fig. 2 displays XRD patterns of ZnAl-DHSB-LDH. Here, the first three Bragg reflection peaks locate at 4.22° (2.09 nm), 8.45° (1.05 nm) and 12.66° (0.70 nm)/2-theta (d -value), respectively. One may note a simple multiple between these three d -values, for example, $2.09 \text{ nm} \approx 2 \times 1.05 \text{ nm} \approx 3 \times 0.70 \text{ nm}$ showing a typically layered structure like the LDH [2]. It is worthy to note no XRD pattern for ZnAl- NO_3 -LDH, indicating that the SNSA method is available for direct preparation of the LDH materials containing relatively complex anions besides simple anions, e.g., CO_3^{2-} [7,8] and NO_3^- [15]. The cell parameters are estimated based on structural features of LDH materials: $a=2d_{110}=0.303$ nm and $c=3d_{003}=6.27$ nm. Here, remind that both of 110 and 113 Bragg reflections are usually overlapped for the intercalated LDHs as observed in ref. [28,29]. We separate the broad peak from 60° to $62.7^\circ/2\theta$ to two peaks using Gauss function: $60.76^\circ/2\theta$ for 110 and $61.57^\circ/2\theta$ for 113, see the inset graph in Fig. 2. The calculated a value is in agreement with those reported in the literature for Zn_2Al LDH [28] since the a value mainly depends on the composition of the brucite-like host sheet.

Besides, Fig. 3 shows the HRTEM micrographs of the intercalated LDH. One observes the average particle size ca. 50 nm with narrow size distribution. Moreover, the basal spacing of ZnAl-DHSB LDH is detected from the average distance between the parallel fringes as marked in Fig. 3B and C. The evaluated value of ca. 2.25 nm (11.26 nm/5) is in good agreement with the d_{003} value of 2.09 nm determined by XRD.

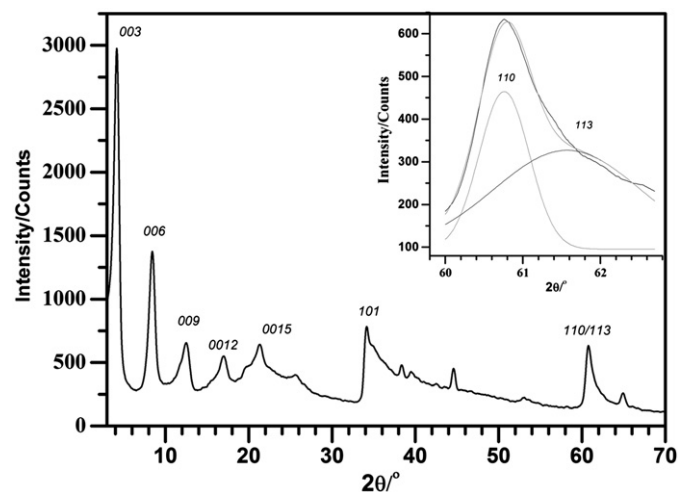


Fig. 2. Powder X-ray diffraction (XRD) patterns of ZnAl-DHSB-LDH.

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