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Layered yttrium hydroxide composite as supersensitive fluorescent sensor on Fe(III) ions

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ABSTRACT

The synthesized HN/OS-LYH composite (HN: 3-hydroxy-2-naphthoic acid, OS: 1-octane sulfonate, LYH: layered yttrium hydroxide) demonstrates tuned luminescence performance and sensitive recognition capability for Fe³⁺. In solid state, the HN/OS-LYH presents a green emission of 505 nm, with 40 nm blue shift relative to the 545 nm green emission of free HN-Na salt; under delamination in formamide (FM), the HN/OS-LYH displays a blue emission of 480 nm in contrast to a 515 nm emission of HN⁻ in FM solution. The delaminated HN/OS-LYH exhibits excellent recognition capability towards Fe³⁺ with distinct fluorescence quenching. For Fe³⁺, a much large quenching constant of 3.85×10^3 M⁻¹ and an extremely low detection limit of 1.14×10^{-8} M are achieved. The HN/OS-LYH is expected to be a "turn-off" type fluorescent sensor for Fe³⁺ detection. This work may provide new ideas for design of novel LRH composites which can be applied on fluorescent sensoring.

1. Introduction

Layered materials have attracted comprehensive attention for rich interlayer chemistry [1] and multiple applications such as ion exchangers, sensors, catalysts, and photochemical materials [2]. Layered rare-earth hydroxides (LRH) are a new kind of layered compounds that have recently appeared. When combined with the inorganic LRH layers, structural stability of the organics is generally enhanced, and fluorescence performance can be improved due to the synergetic effect of host layers and organics [3–5]. So far, there are many reports with regards to LRH-chromophore hybrids [3–14], however, most of them emphasise on the luminescence property of individual organic chromophore or inorganic RE^{3+} , rather than the synergetic interactions of the including components. We have developed a convenient method for delamination of LRH materials [15–17], for which the readily available colloidal suspensions would expand the application range of the LRH materials.

In addition, following the extensive applications in environmental and physiological aspects, chemosensors that can detect metal ions are of great significance [18–20]. Fluorescent chemosensing has been highly valued by researchers due to the low cost, rapid response, and desirable real-time monitoring [21,22]. But there are have been rare reports on LRH composites acting as chemsensors. Byeon's group [23] developed an inner filter effect (IFE) which enabled the LYH:**x**Tb **(LYH is the abbreviation of layered yttrium hydroxide)** to work as fluorescent sensor to detect Cr(VI). They also used organic spacer-modified LGdH: Eu to recognize vanadate anions [24]. These recognition effects are realized through luminescence change of RE³⁺ rather than organics. However, there are few studies on fluorescence sensing performance of organic-LRH hybrids.

Iron is a key element in most organisms such as plants and animals and performs an important role in biochemical processes [25,26]. Fe³⁺ overload or deficiency could induce various disorders, for example anemia, hemochromatosis, heart failure, liver damage, diabetes and Parkinson's disease [27]. Relying on fluorescence 'turn-on' [28,29] or 'turn-off' [30,31] mechanisms, recognition of Fe³⁺ from co-existing ions can be realized, for example, the naphthalimide and coumarin based 'turn-on' fluorescence chemosensor for detecting Fe³⁺ [32]. Fan et al. [33] presented fluorescent off-on enantiomers utilizing rhodamine derivatives for Fe³⁺ imaging in living cells. In addition, Dy-Zn MOF working as turn-off chemosensor for Fe³⁺ had also been reported [34]. There are few researches on the identification of Fe³⁺ using intercalation compounds. Thanks to the protection of the host layers normally regarded as inorganic macromolecules, the chemical stability of the organic luminophors in the interlayers can be enhanced. Also, inorganic

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Fig. 1. XRD patterns of NO₃-LYH precursor (a) and composites of OS-LYH (b), HN/OS-LYH (c). The *d*-values are given in nanometers.

nanoscale layers are regularly present in a colloidal state, which could uniformly contact with the solution systems of metal ions to be identified, thus improving the recognition effect. We recently demonstrated a FLN/OS-LRH composite which exhibited fluorescence turn-off chemosensoring for detecting Fe(III) [35]. So far, there is still a lack of research on the recognition of Fe³⁺ by LRH composites.

The 3-hydroxy-2-naphthoic acid (HN) is a small fluorescent organic molecule which has been utilized to detect aluminium [36], copper [37], and vanadium [38]. We want to figure out if the HN can be used to identify iron. Moreover, when the HN is combined with certain inorganic host layers such as LYH, its luminescence performance may be changed or sometimes improved. Thus we synthesize the HN/OS-LYH composite to check its luminescence performance and the capability to recognize iron ions. Contrary to the 'turn-on' type fluorescence chemosensor for Al³⁺ [39], the HN/OS-LYH shows a 'turn-off' recognition character for Fe³⁺, with a large quenching constant of 3.85×10^3 M⁻¹ and an extremely low detection limit of 1.14×10^{-8} M. This describes a novel example of LRH composites applied in fluorescence sensoring to identify Fe³⁺.

2. Experimental sections

2.1. Synthesis of NO3-LYH, OS-LYH and HN/OS-LYH and delamination

NO₃-LYH and OS-LYH were prepared referring to the literatures [5, 40], with the details shown in supplementary materials. Intercalation of HN/OS into LYH was performed via an ion exchange reaction. The 1:1 stoichiometric NaOH was mixed with HN to make it deprotonated beforehand, and then the OS was added. Into the aqueous solution with a 1.29 mmol of HN + OS (HN:OS molar ratio: 1:4), 0.43 mmol of NO₃-LYH powders were added. The mixture was transferred into a Teflon-autoclave (solution volme is 80 ml) and reacted at 70 °C. After reaction for 24 h, the as-formed HN/OS-LYH precipitates were filtered, water-washed, and dried at vacuum at 50 °C over night.

For the delamination of the HN/OS-LYH, 0.05 g of sample was dispersed into FM (20 mL) and treated with mechanical shaking (\sim 2 h). A transparent colloidal suspension was then obtained, indicating that the HN/OS-LYH was exfoliated.



Fig. 2. FT-IR spectra of HN (a), OS (b), NO_3 -LYH (c), and HN/OS-LYH composite (d).

2.2. Synthesis of sodium salt of HN (HN-Na)

For the preparation of sodium salt of HN (HN-Na), equal molar equivalent of NaOH was mixed with the HN to remove its protons. After complete dissolution of HN and NaOH, the solvents were evaporated slowly. During the solvent evaporation process, solid product was precipitated out gradually. After filtration, the HN-Na salt was collected, which can be used to test the fluorescence property.

2.3. Ion recognition experiments of HN/OS-LYH

For recognition experiments, nine metal ions $(Mg^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Pb^{2+}, Cd^{2+}, Hg^{2+} and Fe^{3+})$ were selected as target ions. The ion solutions were got from dissolving their corresponding nitrate salts. To the colloidal suspension (3 ml), the aqueous solution (1 ml) of the metal ion with certain concentrations were added. Then fluorescence emission spectrum of the mixtures were tested.

3. Results and discussion

3.1. Characterization of the prepared samples

XRD patterns of NO₃-LYH, OS-LYH and HN/OS-LYH were detected. As depicted in Fig. 1a, the series of (00*l*) diffractions at 0.89, 0.45 and 0.29 nm indicate a basal spacing (d_{basal}) of 0.89 nm, consistent with that reported for NO₃-LYH [41]. For the OS-LYH (Fig. 1b), the (00*l*) diffractions at 2.01, 1.01, 0.67, 0.51, 0.41 nm shows a 2.01 nm d_{basal} . The diffractions of HN/OS-LYH are similar to those of OS-LYH, with a close d_{basal} of ~2.0 nm (Fig. 1c). This illustrates the interlayer space of HN/OS-LYH is mainly supported by the OS, possibly due to the higher content and longer size of OS. As seen all samples contain a peak located at 0.31 nm, which belongs to (220) reflection of LRH layers [41,42]. This means that the layer structure does not change during ion exchange process.

Fig. 2 depicted FT-IR spectra of the samples. For unprotonated HN (Fig. 2a), the band at 3284 cm⁻¹ belongs to O–H vibration — [43], and those at 1665/1467 cm⁻¹ correspond to C–O vibration of –COOH.



Fig. 3. SEM images of NO₃-LYH precursor (a, a') and HN/OS-LYH composite (b, b').



Fig. 4. Emission spectra of (a) HN-Na ($\lambda_{ex} = 394$ nm) and (b) HN/OS-LYH ($\lambda_{ex} = 415$ nm) in (A) solid state and (B) delamination state in FM ($\lambda_{ex} = 390$ nm). (A') and (B') are corresponding photographs of (a) and (b) under 365 nm UV irradiation.

The 1516 cm⁻¹ band is ascribed to C— C vibration of naphthalene ring [44]. For OS (Fig. 2b), two bands at 1199/1067 cm⁻¹ are characteristic absorptions of SO₃⁻ [45]. In Fig. 2c, the 1384 cm⁻¹ band of NO₃⁻ proves successful synthesis of NO₃-LYH [46]. In Fig. 2d, the NO₃⁻ band at 1384 cm⁻¹ becomes unobservable, suggesting substitution of NO₃⁻ by HN⁻ and OS in the HN/OS-LYH. The SO₃⁻ vibrations observed at 1171/1049 cm⁻¹ signify the presence of OS in the composite. The **absorption bands at** 1596/1401 cm⁻¹ can be assigned to $-COO^-$, for which the red shift of **the wavelengths** relative to **the 1665/1467 cm⁻¹ of** -COOH results from the deprotonation. The hydrogen bonding interactions of $-COO^-$ with layer -OH groups also decrease in vibration frequency.

SEM observations were employed for the NO₃-LYH and HN/OS-LYH as shown in Fig. 3. The NO₃-LYH precursor (Fig. 3a,a') exhibits plate-like morphology with a nearly regular hexagon shape, and some crystals grow into columnar or flower-like aggregates. The HN/OS-LYH (Fig. 3b, b') basically maintains the morphology of NO₃-LYH. The coincidence of morphology of the HN/OS-LYH composite with the NO₃-LYH precursor means a topotactic intercalation process of the organics into the LYH interlayers.

3.2. Luminescence performance of HN/OS-LYH in solid state and delaminated state

Emission spectra of HN-Na salt and HN/OS-LYH in solid state were depicted in Fig. 4A. For HN-Na, at the 394 nm excitation ($\lambda_{ex} = 394$ nm, with the excitation spectra shown in Fig. S1a), a green emission is observed at 545 nm (see Fig. 4A-a). For HN/OS-LYH (Fig. 4A-b and Fig. S1b, $\lambda_{ex} = 415$ nm), though it is still a green emission, the peak position appears at 505 nm, with a 40 nm blue-shift, and the luminescence intensity is much enhanced in comparison to HN-Na. It is noted that in the testing, the solid dosage of HN-Na and HN/OS-LYH composite is equal, which means a much less HN amount in the HN/OS-LYH. Intermolecular aggregation of HN can be prevented by the LYH layers and interlayered OS [47], thus causing the increased luminescence intensity. Meanwhile, OS and LYH layers together induce the blue shift of emission wavelength. The same green color range but different emission position of the two samples is depicted in the CIE 1931 chromaticity diagram (Fig. 4A').

Luminescence behavior of HN-Na solution and HN/OS-LYH colloid in FM were investigated. The HN/OS -LYH colloid displays a blue emission at 480 nm (Fig. 4B-b), forming striking contrast to the green luminescence (505 nm) of the composite in solid state (Fig. 4A-b) as well as the green emission (515 nm) of free HN⁻ in FM solution (Fig. 4B-a). This marked blue shift identifies the role of LYH host layers and interlayered OS. Moreover, the delamination of the HN/OS-LYH offers much free space for HN surrounding positively-charged LYH layers (loose contact), giving the opportunity of OS to play its role in terms of luminescence regulation. Also, the delamination process may also promote the appearance of blue emission. The blue shift phenomenon was reported in DDS-AQS/LDH [48]. As we know surfactants may reduce aggregation of chromophores [49]. Here the OS acts as a diluter for the luminous organic anions (here is HN) and thus prevents them from aggregation which may reduce the fluorescence [47,50,51]. The CIE 1931 chromaticity diagram and inserted photos in Fig. 4B' present the transition from green to blue color of the two samples, coinciding with the emission spectrum. Here we see the CIE1931 chromaticity diagrams enable a clearer view of the emitted color of related samples. The chromaticity diagram mainly divides three color areas, that is, red, blue and green. In the present work, in solid state, although the emissions of HN-Na (545 nm) and the HN/OS-LYH composite (505 nm) both belong to green luminescence, their emission wavelengths are different (with 40 nm change), for which the corresponding position can be displayed in the chromaticity diagram. Moreover, the green emission (515 nm) of the HN-Na solution and the blue emission (480 nm) of the exfoliated HN/OS-LYH can be well distinguished and observed in the chromaticity



Fig. 5. Emission spectra of HN/OS-LYH colloid mixed with various metal ions (λ_{ex} =355 nm). For measurements, 3 ml of composite colloid mixed with 1 ml of aqueous solutions containing metal ions in a 1000 ppm concentration. The 'None' case is a control experiment, for which 3 ml of composite colloid was mixed with 1 ml of pure water.



Fig. 6. Fluorescence intensity (525 nm) of HN/OS-LYH colloidal suspension with the addition of Fe³⁺ and other metal ions ($\lambda_{ex} = 355$ nm). The luminous intensity of Fe³⁺ is too low to be displayed.

diagram. Based on integrating sphere technique [52], the PL quantum yield of the HN/OS-LYH composite was determined to be 23.20 %, which is much larger than that (4.86 %) of the HNA-Na [39]. The increased quantum yield of the composite demonstrates the significant function of the LYH layers for improvement of the luminescence performance of HN.

3.3. Ion recognition of HN/OS-LYH composite

Luminescence performance was studied for HN/OS-LYH mixed with aqueous solutions containing metal ions at certain concentrations. As shown in Fig. 5, red shift of emission from 480 nm to 525 nm is found after addition of aqueous solutions of metal ions, which may be because of the H₂O entrance. To detect the effect of solvents, emission spectra of HN-Na in H₂O, FM and their mixtures were measured (Fig. S2). In pure FM, water and different proportional FM+H₂O, emission wavelengths of 515, 530, and 515-530 nm were observed. As shown in Fig. 5, fluorescence intensity varied in the cases with various types of metal ions. The fluorescence was remarkably quenched by Fe^{3+} , forming sharp contrast to other metal ions. This may originate from the interactions between Fe³⁺ and HN/OS-LYH composite. As known Fe³⁺ may coordinate with the hydroxyl and carboxylate groups of HN, and radiative transition can be disrupted, which causes the quenching of fluorescence. The highly effective recognition capability towards Fe³⁺ verifies an admirable fluorescent sensoring. The high selectivity may be due to strong affinity of Fe³⁺ with HN. Meanwhile, paramagnetic Fe³⁺ has



Fig. 7. Emission spectra of HN/OS-LYH colloidal suspension with addition of Fe^{3+} at various concentrations (0–1000 ppm) (3 ml colloid +1 ml aqueous solution).



Fig. 8. Linear relationship between fluorescence intensity ratio (I₀/I) at 525 nm of HN/OS-LYH colloid and Fe³⁺ concentration (mmol/L) in the range of 0.018 – 0.18 mmol/L (1-10 ppm). The luminescence intensities used for plotting I/I₀ are the *y*-coordinate values in Fig. 7.

strong coordination interactions with the oxygen ligand-containing HN, which may lead to the change of charge distribution of HN [53]. For clarity, I bar diagram (Fig. 6) was used to show the relative magnitude of fluorescence intensity for the cases with varied metal ions.

We also measured the emission spectra of HN/OS-LYH contacted with solutions of Fe^{3+} at varied concentrations (0–1000 ppm). From Fig. 7, following the increase of Fe^{3+} concentration, the fluorescence of

the mixture has been gradually weakened and finally quenched, demonstrating a sensitive sensing for Fe³⁺. As displayed in Fig. 8, emission intensity exhibits a linear relationship with Fe³⁺ concentration $(4.5 \times 10^{-7} - 1.79 \times 10^{-4} \text{ M})$. At low concentration range of 0-0.2 mmol/L (0–10 ppm), quenching coefficient (K_{sv}) can be determined from the slope of titration curve, based on the modified Stern-Vollmer equation [54]:

$$I_0/I = 1 + K_{sv}[M]$$

where I/I₀: fluorescence intensity with/without metal ions (here is Fe³⁺), K_{sv} : quenching constant, [M]: Fe³⁺ concentration. Based on the above equation, we get the K_{sv} value of $3.85 \times 10^3 \text{ M}^{-1}$. From the linear relationship, the detection limit is determined to be 1.14×10^{-8} M. This value is much lower than maximum contaminant level of drinking water based on the U. S. EPA [55–58]. The K_{sv} of the present HN/OS-LYH is larger than those other reported materials for Fe³⁺ recognition [35, 59–61], for example, K_{sv} of $2.26 \times 10^2 \text{ M}^{-1}$ by pyrene-doped electrospun PMMA-PVC fibers [59], and K_{sv} of $3.96 \times 10^2 \text{ M}^{-1}$ by polyvinyl chloride-graft-polystyrene copolymers [60]. The detection limit of 1.14×10^{-8} M is extremely lower than the values of other reported materials [35,62–64] such as 1.2×10^{-6} M for N-doped carbon quantum dots [62], 10^{-7} M for hexahomotrioxacalix [3]arene [63], 7.03×10^{-3} M and 8.76×10^{-3} M for PP and PS, respectively [64], and 2.58×10^{-8} M for FLN/OS-LRH [35].

Considering that there are many heteroions co-existing in actual wastewater, interference of other metal ions to Fe^{3+} sensor is worthy of careful study. To 3 ml of the HN/OS-LYH colloid, the solution with mixed ions (1000 ppm for each ion) was added. As shown in Fig. 9, for the cases containing Fe^{3+} mixed with other ions, the fluorescence intensity presents a slight enhancement compared with that with only Fe^{3+} , but the degree of enhancement seems to be small. This demonstrates that other ions have almost no interference for the Fe^{3+} recognition. All these results verify the superior recognition capability of the HN/OS-LYH composite towards Fe^{3+} .

4. Conclusions

In conclusion, co-intercalation of HN and OS into LYH via an ion exchange reaction generates the HN/OS-LYH composite which exhibits tuned luminescence and ion recognition performance. In solid state, HN/OS-LYH composite displays a green emission (505 nm) while a blue emission (480 nm) in delaminated state. The blue shift of HN/OS-LYH composite in comparison to free HN-Na salt results from the confined effect of LYH layers, dilution function of OS as well as the intermolecular interactions between HN and OS. The delaminated colloid of HN/OS-LYH composite is highly selective to Fe³⁺ over other metal ions. It is a good turn-off chemosensor for Fe³⁺ exhibiting an extremely low detection limit of 1.14×10^{-8} M and a much large quenching constant of 3.85



Fig. 9. (A) Contrast of luminescence intensity of HN/OS-LYH colloid and its mixtures with single and mixed metal ions (concentration is 1000 ppm), (B) Enlarged view of luminescence intensity of Fe³⁺-existing systems after the red bars of (A) are omitted.

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 \times 10³ M⁻¹. The excellent recognition selectivity may be due to the strong affinity between Fe³⁺ and HN with oxygen as the ligand. This work opens a new way for the fabrication of novel LRH materials that can be utilized on fluorescence chemosensoring.

Author statement

Shulan Ma and Zupei Liang conceived and directed the whole research study. Huiqin Yao supervised all of the experiments. Jian Li and Feifei Su synthesized the samples and carried out most characterizations and experimental meaurements. All the authors discussed the results and participated in writing of the manuscript.

Declaration of Competing Interest

The authors declare no conflict of interest.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.materresbull.2020 .111135.

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