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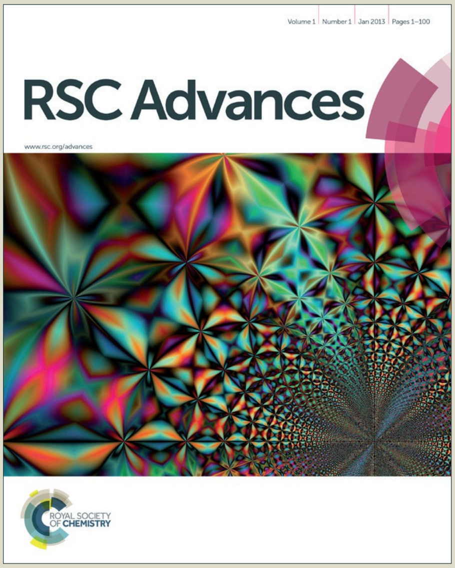

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Anionic Metal-Organic Framework for High-Efficiency Pollutant Removal and Selective Sensing of Fe(III) Ions†

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An anionic metal-organic framework, $(DMA)_2[Y_9(\mu_3\text{-}OH)_8(\mu_2\text{-}OH)_3BTB_6]_n\cdot(solv)_x$ (DMA = dimethylamine cation and BTB = 1,3,5-benzene(tris)benzoate), (gea-MOF-1), was a novel rare earth (RE) Nonanuclear MOF with an unusual gea topology. It could be used as adsorbent material for efficiency removing cationic dye MB^+. This MOF not only can emit strong fluroescence in the MeOH suspension, but also serve as a fluorescent probe for selectively detecting Fe^{3+} among other metal ions through luminescent emission quenching.

With the development of chemical industry, poisonous chemicals, like toxic heavy metal ions and organic small molecules, are increasingly released from industrial provision and other human activities, which have a negative impact on human health and environment.[1-3] Particularly, organic dyes have both brachychronic and chronisch toxicity effects on human health, which may lead to transgenation, cancer, endocrine disrupting and other serious diseases.[4] As indispensable industrial products, organic dyes have been generally used in paper-making, textile industry etc.[5] Therefore, it is extremely important to resolve the environmental contamination caused by organic dyes. Up to now, several approaches were presented to solve the problems, such as advanced oxidation, adsorption, photocatalysis, membrane filtration and coagulation.[6] Among them, adsorption is well known to be one of the strategies because of low cost, high efficiency and environmentally friendly.[7] For this matter, many common adsorbents, such as zeolites, active carbon and polymeric materials, have been extensive applied to remove organic dye contaminants.[8] Although these common adsorbents can easily adsorb multifarious mixed organic dyes, they are usually inferior to selectively separating objective organic dye wastes.[9] Therefore, it is desirable to prepared novel materials which could separate target selectively from mixed dyes wastes. In addition, iron element is essential for human body. The superfluous or lack amounts of Fe^{3+} in body fluids may cause a potentially deadly or hereditary disease such as hemochromatosis and iron-deficiency anemia. Therefore, the detecting of Fe^{3+} with a selective and accurate manner is crucial.

Porous metal-organic frameworks (MOFs) emerge attractive applications in the areas of gas storage, separation, luminescence, drug delivery and heterogeneous catalysis owing to their diverse structural topologies and functional sites.[10] So far, ionic MOFs can be applied in adsorption and separation of organic dyes.[2,11] For example, Zhao et al. constructed a series of mesoporous positive MOFs, which serve as a platform for the anion exchange-based separation process to capture anionic organic dyes.[2e] Our group also synthesized anionic In^{III}-MOFs for adsorption and separation of methylene blue, which act as chromatographic column immobile phases to separate smaller size cationic organic dyes.[2f] Most of the reported MOFs are utilized to separate smaller size organic dyes based on their size exclusion, which is excellent for highly selective separation organic dyes.[2f] Meanwhile, RE(III)-based MOFs are outstanding candidates as fluorescence probes materials for heavy metal ions sensing.[12]

Porous materials $(DMA)_2[Y_9(\mu_3\text{-}OH)_8(\mu_2\text{-}OH)_3BTB_6]_n\cdot(solv)_x$ (DMA = dimethyl amine cation and BTB = 1,3,5-benzene(tris)benzoate) **gea-MOF-1** were synthesized according to the previously reported lietrature[13] and confirmed by Powder X-ray diffraction (PXRD) patterns (Fig. S1, ESI†). This porous MOF material exhibits the potential for gas-storage and heterogeneous catalysis applications. In this work, we found anionic **gea-MOF-1** can rapidly adsorb cationic dyes Methylene Blue (MB^+) with a smaller size based on the cation exchange, but hardly adsorb anionic Methyl Orange (MO^-), as well as neutral Sudan I (SD^0) and cationic dyes Methylene

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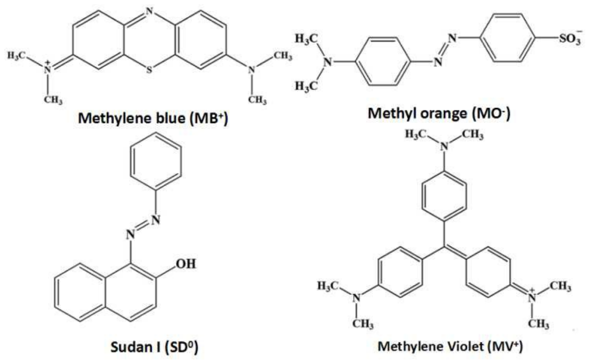
† Electronic Supplementary Information (ESI) available: Experimental details, PXRD, UV-Vis absorption spectrum, luminescence spectra. DOI: 10.1039/x0xx00000x



Scheme 1 Chemical structures of MB^+, MO^-, SD^0 and MV^+.

Violet (MV^+) (Scheme 1) with larger size from dimethyl formamide (DMF) solution. Surprisingly, gea-MOF-1 can preferentially adsorb MB^+ from the mixed organic dyes in the DMF solution. Moreover, **gea-MOF-1** also displays excellent luminescent property, which can act as luminescent probe of Fe^{3+}.

As shown in Fig. 1, **gea-MOF-1** display pillared hexagonal (hxl) layers structure along the *c* axis, creating one-dimensional channels with the windows sizes of 12.834 Å×9.386 Å. DMA^+ cations reside in the hxl channel.

According to the relative references[2e,14], the charge characteristic and size effect of the organic dye molecules play crucial roles during the process of dye adsorption or separation.[2e] Due to the highly porous feature and anionic characteristic of **gea-MOF-1**, we investigate its potential capacity for adsorption and separation of organic dye molecules from DMF solution. In the adsorption study, three organic dyes with different charges have been chosen: MB^+, MO^- and SD^0, and dissolve them into DMF (5×10^{-5} M), respectively, which characters the analogical relative molecular mass and size but different charges peculiarity (Table S1, ESI†). The as-synthesied **gea-MOF-1** was soaked in the fresh as-prepared DMF solution of MB^+, MO^- and SD^0. The capacity of **gea-MOF-1** for capturing dyes from DMF solution was detected by UV-vis absorption spectroscopy at certain time intervals. As shown in Fig. 2a, the Abs peak value of the supernate declined gradually up to 210 min, suggesting that almost all of the MB^+ in the supernatants was removed by the **gea-MOF-1**. Exactly the opposite, the Abs peak values of MO^- (Fig. S2, ESI†) and SD^0 (Fig. S3, ESI†) solutions were unchanged at all. Moreover, further exploration on the selective separation of MB^+ from the mixtures of MB^+/MO^- and MB^+/SD^0 solutions were implemented. As demonstrated in Fig. 3b and Fig. 3c, the results were as anticipatory: only the MB^+ in the mixture solutions was removed by the **gea-MOF-1** and the solutions finally showed the colors of MO^- and SD^0. Spectroscopic evaluations of the supernatants revealed that **gea-MOF-1** can highly efficiently capture organic cationic dyes, whereas organic anionic and neutral dyes can not be adsorbed by **gea-MOF-1**. At last, the color of the **gea-MOF-1** changed from transparency to blue. This phenomenon was ascribed to the anionic peculiarity of the **gea-MOF-1** framework, in which the free DMA^+ present in the channel can be exchanged with organic cationic dyes MB^+. Therefore, **gea-MOF-1** can remove organic cationic dyes among the neutral and positively charged organic dyes through ion-exchange process. The organic dye molecules with semblable sizes but different charges could be selectively separated by **gea-MOF-1** through ion-exchange processes.

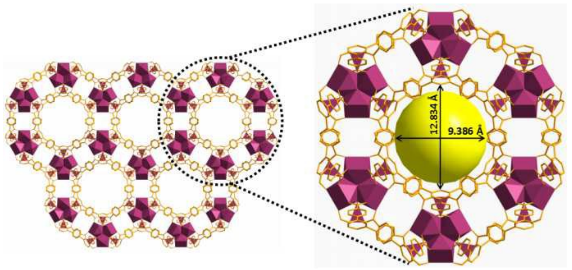


Fig. 1 Illustration of the pillaring of hxl layers in the gea-MOF-1, creating one-dimensional channels.

We also investigate the effect of size on absorption of cationic dyes. Two organic cationic dyes with different molecular sizes were chosen for candidate: MB^+ and MV^+. They are in uniform charges characteristic but different molecular sizes: MV^+ (4.00 Å × 16.32 Å) presents larger appearance than MB^+ (4.00 Å × 7.93 Å) along x and y directions (Table S1, ESI†). Typically, the fresh as-synthesied samples of **gea-MOF-1** were immersed into DMF solutions of MB^+ and MV^+, respectively. As shown in Fig. 2a and Fig. S4, ESI†, the concentration of smaller dye declined sharply. However, the concentration of the larger one almost no changed. The size selective effect was also tested under mixed solution of MB^+/MV^+. As shown in Fig 2d, only the characteristic peak of MB^+ decreased significantly, meanwhile, the solution changed from blue-violet to purple. The above results shows that only MB^+ can be exchanged by the **gea-MOF-1** and MV^+ can't finish the cation-exchange process with **gea-MOF-1** owing to its larger size. From the above experiments, **gea-MOF-1** might be a admirable adsorbent for efficient and selective removal of smaller size cationic dyes.

Within the dye adsorption and release process, the stability and reversibility of the crystal are also a crucial. The experiments of MB^+ release are also implemented. MB^+@gea-MOF-1 sample was immersed into the saturated $NaNO_3$ DMF solution and the concentration change of MB^+ in the supernate was monitored by UV–visible spectra. As shown in Fig. S5, ESI†, the Abs peak value of supernate increased gradually. This phenomenon is ascribed to the cation exchange process that

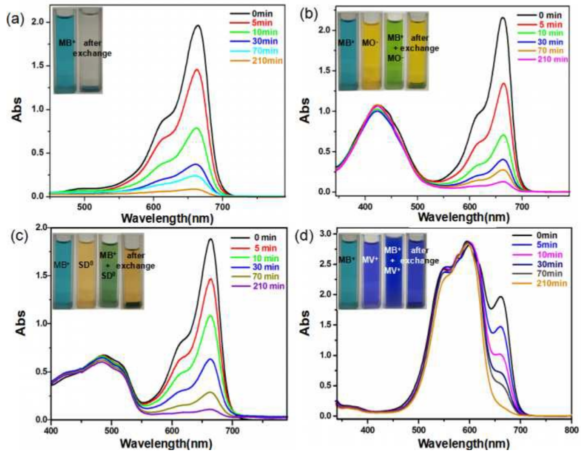


Fig. 2. UV-vis spectra of DMF solutions of equimolar dyes in the presence of gea-MOF-1 monitored with time: (a) MB^+, (b) MB^+/MO^-, (c) MB^+/SD^0, (d) MB^+/MV^+. The photographs show the colors of the dye solutions and the crystalline samples of gea-MOF-1, before and after ion-exchange for 210 min.

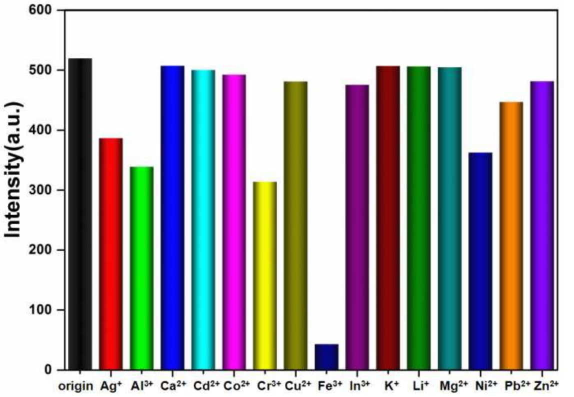


Fig. 3 Room-temperature luminescent intensity of gea-MOF-1 at 375 nm in methanol suspension of gea-MOF-1 upon addition of various metal ions (λ_{ex} = 290 nm).

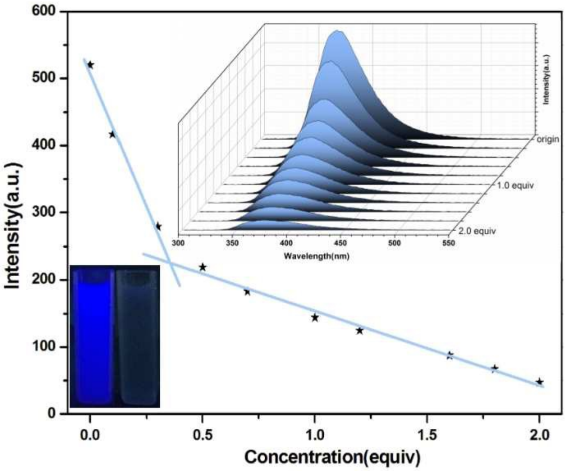


Fig. 4 Emission spectra of the gea-MOF-1 in methanol at room temperature in the presence of different equiv. of Fe^{3+} ion with respect to gea-MOF-1, respectively (λ_{ex} = 290 nm). Inset: photograph showing the change of the original fluorescence of methanol suspension of gea-MOF-1 (left) and the decreased luminescence upon the addition of Fe^{3+} ion in the methanol suspension of gea-MOF-1 (right).

Na^+ entered into the pore of **gea-MOF-1** replacing MB^+. The PXRD of **gea-MOF-1** after release investigation confirmed the host skeleton of **gea-MOF-1** material did not change(Fig. S6 and S7, ESI†).

With a view to the potential applications of RE^{III}-MOFs as luminescent sensor materials in biological systems, we also studied the solid state photoluminescent properties of H_3BTB and **gea-MOF-1**, as shown in Fig. S8 and S9, ESI†. Excited at 284 nm and 290 nm at room temperature, H_3BTB and **gea-MOF-1** reveal the emission peak at 385 nm and 375 nm, respectively, which can be tentatively attributed to π-π* transition of the intraligand.[15] The test of **gea-MOF-1** for the recognizing of metal ions were also be studied. $M(NO_3)_n$ (M^{n+} = Ag^+, Li^+, Cd^{2+}, Al^{3+}, Pb^{2+}, Fe^{3+} Cr^{3+}, Zn^{2+}, Mg^{2+} Ca^{2+}, Co^{2+}, Cu^{2+}, In^{3+}, Ni^{2+} and K^+) were added into the methanol suspension of **gea-MOF-1** at room temperature, respectively. As shown in Fig. 3, the luminescence intensity of the methanol suspension of **gea-MOF-1** has the strongest quench effect with Fe^{3+} ion adding in. However, with Ag^+, Al^{3+}, Ni^{2+} and Cr^{3+} adding in, the emission intensity of **gea-MOF-1** was weakened in a small degree, and others metal ions had inappreciable effect on the photoluminescence intensity of **gea-MOF-1**, indicating **gea-MOF-1** could selectively detect Fe^{3+} ion through luminescent quenching. Furthermore, the luminescence emission intensity of **gea-MOF-1** declined gradually *via* addition of 0.1 - 2.0 equiv of Fe^{3+} ions (Fig. 4) until the photoluminescence was completely quenched when 2.0 equiv of Fe^{3+} ion was introduced into the methanol suspension of **gea-MOF-1**. Meanwhile, according to the previous references,[16] we investigated the selectivity to Fe^{3+} ion among other metal ions (Fig. S10 and S11, ESI†) as demonstrated by introduction of Li^+, Cd^{2+}, Pb^{2+}, Zn^{2+}, Mg^{2+} Ca^{2+}, Co^{2+}, Cu^{2+}, In^{3+} and K^+ into the system. As a result, it indicate that **gea-MOF-1** can selectively probe Fe^{3+} among other metal ions.

In order to better understand the mechanism of luminescence quenching with Fe^{3+} adding in. The PXRD of the sample after sensing of Fe^{3+} was measured. The pattern indicated that this quenching phenomenon has no relation with the crystallographic alteration, and that the detection process of **gea-MOF-1** in the methanol solution would not induce the framework collapse(Fig. S12, ESI†). Furthermore, Inductively Coupled Plasma optical emission spectroscopy (ICP-OES) measurement was also carried out to monitor the amount change of Y and Fe elements during the sensing process. The ICP-OES results showed that the amount of Fe element was much less than that of Y (Table S2, ESI†), indicating that the luminescent quenching effect should not arise from a cation-exchange process.[17] Last, according to the UV-Vis absorption spectrum (see Fig. S13, ESI†), the strong absorption of the Fe^{3+} methanol solution was in the range of 200 - 500 nm, while the other metal ions methanol solution did not overlap the absorption range, thus, the following two possible mechanisms may account for the origin of the high selectivity and sensitivity towards Fe(III). Firstly, there exists the competition of absorption of excitation wavelength (290 nm) energy between the Fe^{3+} ions methanol solution and the **gea-MOF-1**. Such competitive adsorption will significantly decrease the transfer of excitation energy. Secondly, there exists a complete overlap between the absorption spectrum of the Fe^{3+} methanol solution and the broad emission band at 375 nm of **gea-MOF-1**. By increasing Fe^{3+} ion concentration, the color of **gea-MOF-1** suspension would be gradually deepened and the luminescence intensity of **gea-MOF-1** suspension declined gradually. Based on these pieces of evidence, we then suggest that the competitive absorption mechanism may account for the selective quenching response of **gea-MOF-1** towards Fe^{3+}.[18]

In summary, anionic **gea-MOF-1** containing 1D channels exhibits excellent size-selective adsorption of organic cationic dyes MB^{+} among MO^{-}, SD^{0} and MV^{+} *via* cation exchange process. Furthermore, the **gea-MOF-1** could be utilized as potential luminescent probe of Fe^{3+}, which shows significantly quenching effect in Fe^{3+} among other metal cations. Therefore, RE-based MOFs have great potential application to develop MOF-based multifunctional materials.

Acknowledgements

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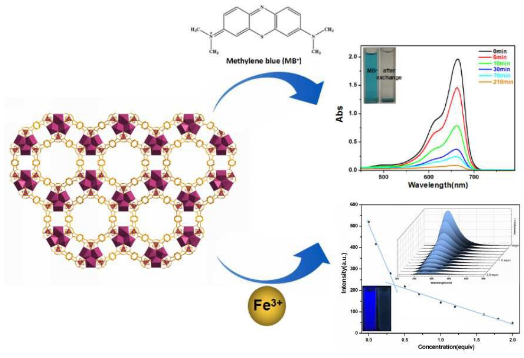
SYNOPSIS TOC.



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An anionic metal-organic framework, $(DMA)_2[Y_9(\mu_3\text{-OH})_8(\mu_2\text{-OH})_3BTB_6]_n\cdot(solv)_x$ (DMA = dimethylamine cation and BTB = 1,3,5-benzene(tris)benzoate), (gea-MOF-1), was a novel rare earth (RE) Nonanuclear MOF with an unusual gea topology. It could be used as adsorbent material for efficiency removing cationic dye MB^+. This MOF not only can emit strong fluroescence in the MeOH suspension, but also serve as a fluorescent probe for selectively detecting Fe^{3+} among other metal ions through luminescent emission quenching.