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阴离子表面活性剂改性水滑石吸附硝基苯的特性研究

夏燕¹, 朱润良^{1,2*}, 陶奇², 刘汉阳¹

(1. 湘潭大学环境工程系, 湘潭 411105; 2. 中国科学院广州地球化学研究所, 广州 510640)

摘要: 以阴离子表面活性剂十二烷基硫酸钠(sodium dodecyl sulfate, SDS)为改性剂, 采用共沉淀法在不同表面活性剂浓度, 0.5~2.0 理论阴离子交换容量(theoretical anion exchange capacity, TAEC)条件下制备了有机水滑石 LDH-DS, 并用 XRD、FT-IR 表征了样品的结构特征。结果表明十二烷基硫酸根成功插层进入 LDH 层间, d_{003} 底面间距由 0.80 nm 增大至 3.98 nm。同时研究了 LDH-DS 对硝基苯的吸附性能, 表明有机水滑石对硝基苯的等温吸附曲线符合线型方程($R^2 > 0.99$), 吸附过程主要为分配作用。随着样品中 SDS 负载量的增加, LDH-DS 对硝基苯的吸附系数 K_d 相应增强, 但有机碳标化的吸附系数 K_{oc} 值基本保持恒定。吸附热力学实验显示吸附过程为吸热反应, 熵增大是吸附过程的驱动力。

关键词: 水滑石; 十二烷基硫酸钠; 硝基苯; 吸附; 热力学

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Sorption of Nitrobenzene to Anionic Surfactant Modified Layered Double Hydroxides

XIA Yan¹, ZHU Run-liang^{1,2}, TAO Qi², LIU Han-yang¹

(1. Department of Environmental Science and Engineering, Xiangtan University, Xiangtan 411105, China; 2. Guangzhou Institute of Geochemistry, Chinese Academy of Sciences, Guangzhou 510640, China)

Abstract: Sodium dodecyl sulfate (SDS) modified MgAl layered double hydroxides (LDHs) were synthesized at different surfactant concentrations (0.5-2.0 TAEC) by the co-precipitation method. The LDH-DS samples obtained were characterized by powder X-ray diffraction and FT-IR spectroscopy. The results showed that SDS was successfully intercalated into the interlayer of the LDH, and the basal spacing was expanded from 0.80 nm to 3.98 nm. The intercalated SDS was considered consistent with a paraffin bilayers arrangement. The sorption of nitrobenzene on LDH-DS was examined, and the results showed that linear model could fit the sorption isotherms well ($R^2 > 0.99$), which implied a partitioning sorption process. The sorption coefficient of nitrobenzene (K_d) on LDH-DS was positively related to the DS^- loading amount, but the organic carbon content normalized sorption coefficient of nitrobenzene (K_{oc}) was shown to remain relatively constant. The sorption thermodynamics results showed that the sorption of nitrobenzene on LDH-DS was an endothermic process, and the increase of entropy was the driving force for the sorption process.

Key words: layered double hydroxides; sodium dodecyl sulfate; nitrobenzene; sorption; thermodynamics

水滑石类层状化合物(layered double hydroxides, LDHs)是一类近年来广受关注的片层状阴离子型黏土。研究表明,将阴离子表面活性剂引入到 LDHs 层间域,能够改变其层间域的空间尺寸和物理化学性质,层间环境由原来的亲水性转变为疏水性,进而能有效地去除水中的疏水性有机污染物^[1,2]。因此,有机改性 LDHs 在水体有机污染控制中具有良好的应用潜力,但要实现其工程化应用仍需进一步研究探明其吸附特征,以提高污染控制效率,降低应用成本。

表面活性剂负载量是影响有机黏土吸附性能的重要因素;考察表面活性剂负载量对有机黏土吸附性能的影响是探测其吸附特征的重要方法^[3,4]。硝基苯作为环境中优先控制的有机物^[5],造成的环境污染问题随着污水处理量的快速增长而愈发明显。为此,本研究用阴离子表面活性剂十二烷基硫酸钠(sodium

dodecyl sulfate, SDS)改性 Mg_4Al-CO_3 型水滑石,考察了 SDS 负载量对改性水滑石吸附硝基苯性能的影响,并探讨了改性水滑石的吸附热力学特征。

1 材料与方法

1.1 样品合成

采用共沉淀法制备水滑石^[6]。将 $Mg(NO_3)_2 \cdot 6H_2O$ 、 $Al(NO_3)_3 \cdot 9H_2O$ 按 $n(Mg)/n(Al)$ 摩尔比为 4.0 溶解于 90 cm³ 去离子水中(溶液 A);取 Na_2CO_3 、NaOH 共同溶解于 100 cm³ 去离子水中(溶液 B)。室温下,用蠕动泵将溶液 A 和 B 逐滴加入 100 cm³ 去离子水中(溶液 C)。滴加过程中用电磁

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作者简介: 夏燕(1986~),女,硕士研究生,主要研究方向新型污水吸附材料, E-mail: ringxy@hotmail.com

* 通讯联系人, E-mail: zhurunliang@gmail.com

搅拌机快速搅拌混合液, 稳定混合溶液 pH 值在 10 左右. 滴加完毕后, 得到的浆液在 60℃ 水浴中陈化 12 h, 离心并用去离子水洗涤, 重复操作至上清液 pH 至中性. 80℃ 烘箱中干燥, 所得产物记为 LDH.

SDS 柱撑的有机水滑石制备步骤同上. 在上述溶液 C 中添加一定量的 SDS, 分别为 LDH 理论阴离子交换容量 (TAEC) 的 0.5、1.0、1.5、2.0 倍, 所得产物记为: LDH- n DS [n 表示 n (SDS) 与 n (LDH)]. 通过空白实验考察了添加有机水滑石后水相中的有机碳含量, 发现表面活性剂解吸非常小, 可以忽略不计.

1.2 吸附实验

称取 0.1 g 有机水滑石于 25 mL 的玻璃离心管中, 然后加入 20 mL 一定质量浓度 (0 ~ 500 mg·L⁻¹) 的硝基苯溶液, 用带特氟隆垫片的盖子密封, 控制在 25℃ 下以 150 r·min⁻¹ 恒温振荡 6 h; 4 000 r·min⁻¹ 离心分离 15 min 后取上清液分析硝基苯的浓度 ($\lambda_{\max} = 268$ nm). 根据硝基苯初始及平衡浓度的差异计算其在有机水滑石上的吸附量. 更改振荡器温度设置 (35℃ 和 45℃), 重复以上吸附实验, 得到不同温度下的等温吸附曲线.

1.3 表征方法

干燥粉末样品的 X-射线衍射分析条件为: CuK α 辐射, 管电压 40 kV, 管电流 300 mA; 2 θ 扫描范围 1° ~ 70°, 扫描速度 4 (°)·min⁻¹, DS/SS 可变狭缝; 测试温度为室温 (约 25℃).

干燥粉末样品的红外吸收光谱分析条件为: BrK 压片, 测试范围 400 ~ 4 000 cm⁻¹, 分辨率 1 cm⁻¹.

总有机碳分析条件为: 采用 680℃ 催化燃烧氧化, 非色散红外检测, 进样 6 ~ 7 min.

2 结果与讨论

2.1 XRD 分析

改性水滑石的 XRD 结果如图 1 所示. 未负载 SDS 的 LDH 样品 d_{003} 值为 0.80 nm, 与文献 [7] 报道的 Mg₄Al-CO₃²⁻-LDH 的值相似 (0.81 nm). 当 SDS 添加到样品合成后, 产物在较低衍射角范围出现了新的衍射峰, 表明了 SDS 进入了水滑石的层间域. 水滑石的 d_{003} 值变化过程为 0.80 nm (LDH) → 0.80 nm (LDH-0.5DS) → 3.13 nm (LDH-1.0DS) → 3.98 nm (LDH-1.5DS) → 3.97 nm (LDH-2.0DS). 其中 LDH-0.5DS 的 d_{003} 值与水滑石前驱体一致, 仍为 0.80 nm, 说明在较低负载量下 DS⁻ 难以进入到水滑

石层间域^[8], 仅负载在有机水滑石外表面. 当添加量达到 2.0TAEC 时, 对应层间距 d_{003} 达到 3.98 nm. 水滑石片层的厚度和 DS⁻ 的长度分别为 0.48 nm 和 2.08 nm^[7], 因此如果 DS⁻ 在水滑石层间采取垂直层板的单层或双层排列时, 其 d 值应分别近似为 2.6 nm 和 4.7 nm^[9]. 但实际 LDH-DS 的 d 值均处于这 2 个数值之间, 由此可推测 DS⁻ 在层间呈斜双层倾斜排列模式 (paraffin-type bilayers arrangement mode^[10,11]).

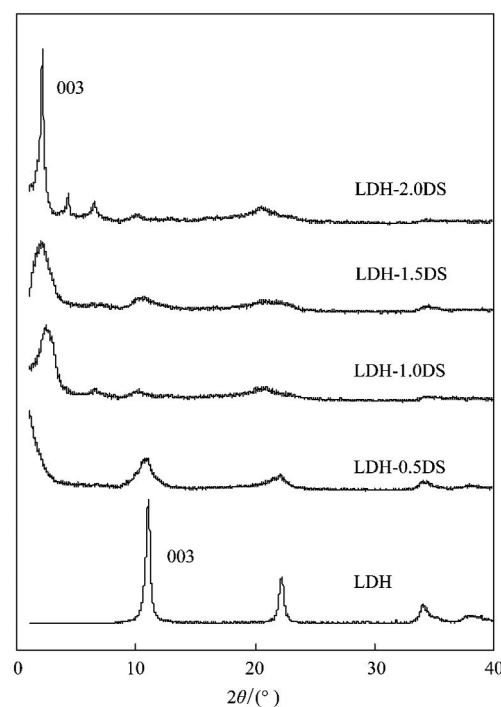


图1 LDH 和 LDH-DS 的 XRD 谱图

Fig. 1 XRD pattern of LDH and LDH-DS

2.2 FT-IR 分析

LDH-DS 的 FT-IR 图谱结果如图 2 所示. LDH-DS 与 LDH 相同的振动峰有: 3 400 ~ 3 500 cm⁻¹ 为羟基的伸缩振动峰, 1 640 cm⁻¹ 为层间自由水分子的弯曲振动峰. 与 LDH 样品相比, LDH-0.5DS 在 1 380 cm⁻¹ 附近的 CO₃²⁻ 特征振动峰明显减弱. 当 SDS 负载量进一步加大, CO₃²⁻ 特征振动峰进一步减弱, 而在 1 230 cm⁻¹, 1 072 cm⁻¹ 处归属 S=O 的对称振动峰和伸缩振动峰逐渐增强^[12]. 此外, LDH-DS 同时出现了 —CH₃ 伸缩振动峰 (2 955 cm⁻¹) 和 —CH₂ 伸缩振动峰 (2 919 cm⁻¹ 和 2 851 cm⁻¹), 以及 1 468 cm⁻¹ 处的 —CH₂ 平面剪式振动峰. 以上 FT-IR 结果也表明 DS⁻ 已经部分取代 CO₃²⁻ 进入水滑石层间域, 与 XRD 的分析结果一致.

2.3 等温吸附曲线与吸附特征

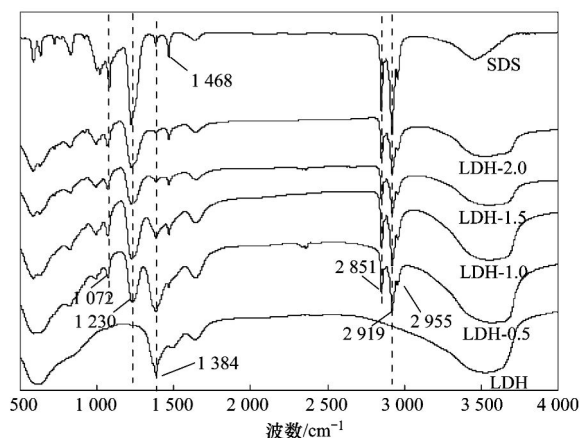


图2 LDH 和 LDH-DS 的 FT-IR 谱图

Fig. 2 FT-IR spectrum of LDH and LDH-DS

图3 为在 $\text{pH} = 8.0$, 25°C 条件下, LDH 和 LDH-DS 对硝基苯的等温吸附曲线。从中可知,未改性的 LDH 对硝基苯的吸附性能极差,而 LDH-DS 对硝基苯的吸附性能比 LDH 有极大提高,且随样品中表面活性剂负载量的增加,吸附性能增强。

在实验浓度范围内,硝基苯在 LDH-DS 上的等温吸附曲线均显示为线性,可以用线性方程拟合:

$$Q = K_d \cdot c_e \quad (1)$$

式中, Q 为吸附剂的吸附量 ($\text{mg} \cdot \text{g}^{-1}$), K_d 为吸附剂对硝基苯的吸附系数 ($\text{mL} \cdot \text{g}^{-1}$), c_e 为吸附质的平衡浓度 ($\text{mg} \cdot \text{L}^{-1}$)。从等温吸附线的斜率计算得到 K_d 值,该值越大,表明吸附剂的吸附能力越强。以上各有机水滑石吸附硝基苯的线性拟合结果以及计算所得的 K_d 值列于表1。线性等温吸附线经常见诸于阳离子表面活性剂改性矿物黏土对有机物的吸附过程^[13~15]。这类吸附过程通常被认为是分配作用,有

表1 等温吸附曲线的回归数据及吸附参数

Table 1 Regression data of sorption isotherms and partition coefficients

吸附剂	线性方程	R^2	$K_d/\text{mL} \cdot \text{g}^{-1}$	$f_{\text{oc}}/\%$	$K_{\text{oc}}/\text{mL} \cdot \text{g}^{-1}$
LDH-0.5DS	$y = 0.0372x$	0.9954	39.5	20.3	195
LDH-1.0DS	$y = 0.0511x$	0.9975	51.1	25.5	200
LDH-1.5DS	$y = 0.0633x$	0.9978	63.3	27.6	229
LDH-2.0DS	$y = 0.073x$	0.9977	73.0	32.4	225

有机碳标化的吸附系数 $K_{\text{oc}} (= K_d/f_{\text{oc}})$ 可以反映有机黏土中表面活性剂的利用效率,并反映表面活性剂负载量对有机黏土吸附特征的影响^[18,19]。因此,计算所得各 LDH-DS 的 K_{oc} 值,结果如表1所示。随着 SDS 负载量增加,硝基苯的 K_{oc} 值基本保持相对恒定,变化不明显, K_{oc} 值的变化范围基本稳定在 $200 \text{ mL} \cdot \text{g}^{-1}$ 左右。这与有机阳离子黏土对疏水性有机物的吸附规律不同^[3,20]。对于有机膨润土,大量研究报

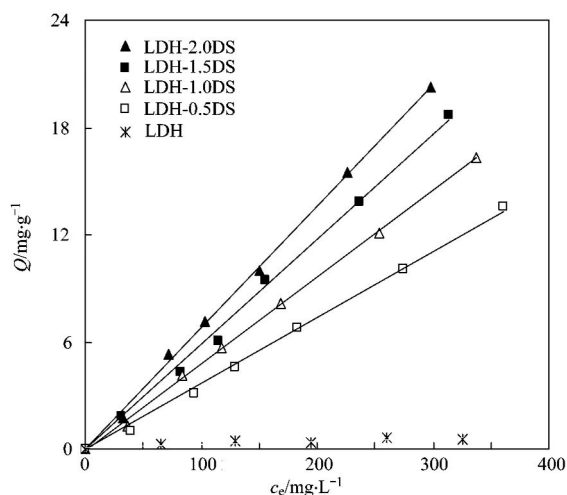


图3 LDH 和 LDH-DS 对硝基苯的等温吸附曲线

Fig. 3 Sorption isotherms of nitrobenzene on LDH and LDH-DS

机黏土层间表面活性剂形成的有机相是分配作用的主要介质^[13]。这一机制同样可以解释硝基苯在 LDH-DS 上的吸附:由于 DS^- 在其层间形成了疏水有机相,从而对硝基苯产生分配吸附作用。

在较低负载量时,阴离子表面活性剂并没有进入水滑石的层间,仅被吸附在水滑石层板上暴露在体系外表面或端面部分。随着负载量的增加, DS^- 进入水滑石层间域形成疏水有机相,一方面层间有机碳含量增大,加强了 DS^- 和硝基苯之间的疏水效应;另一方面负载量的增加使 DS^- 在层间排列方式发生变化,从 0.5DS 过渡到 2.0DS 样品, LDH-DS 的层间域增大,这也为硝基苯在层间的储存提供了更多空间^[16],从而使有机水滑石吸附能力有所提高, K_d 值增大^[17]。

道其吸附疏水性有机物的 K_{oc} 值随表面活性剂负载量增加而呈现先增大后减小的变化规律^[3,20]。Zhu 等^[3] 认为过高的有机阳离子堆垛密度使有机膨润土吸附性能降低。而有机水黑云母由于其较高电荷密度使表面活性剂处于较高堆垛密度, K_{oc} 随着样品中 CTMA⁺ 负载量的增大呈现先减小后趋向稳定的规律^[3]。有机水滑石 K_{oc} 值的变化规律不同于以上 2 种,表明黏土载体以及烷基链的差异对有机物吸附性

能的重要影响,而表面活性剂 SDS 的堆垛密度也有可能 是导致其吸附规律变化的重要原因。

2.4 吸附热力学研究

图 4 为 $\text{pH} = 8.0$ 时, 25、35 和 45℃ 条件下

LDH-DS 对硝基苯的吸附曲线。各等温吸附曲线在 实验浓度范围内均呈线性。4 种负载量不同的 LDH-DS 显示出的规律一致: 随着温度的升高, LDH-DS 对硝基苯的吸附性能明显增强。

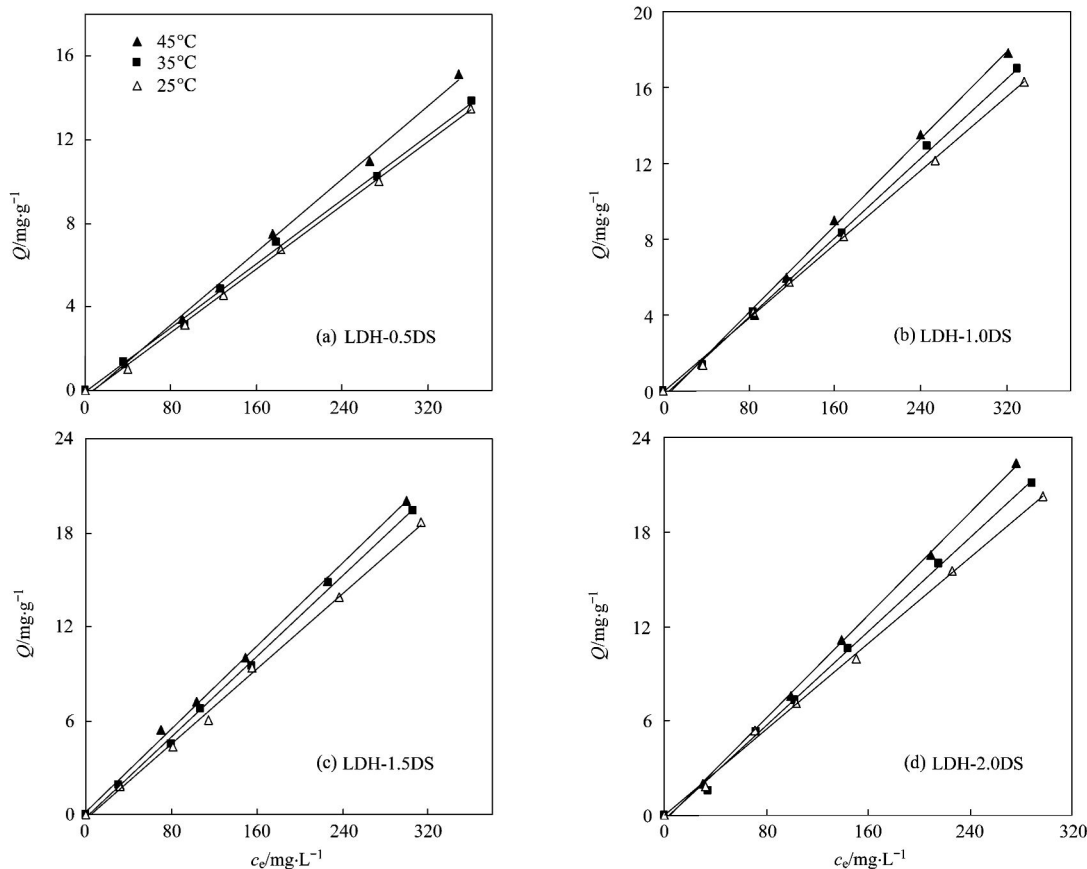


图 4 不同温度下 LDH-DS 对硝基苯的等温吸附曲线

Fig. 4 Sorption isotherms of nitrobenzene on LDH-DS at 25, 35 and 45°C

利用以上 K_d 随温度的变化数据和下列热力学方程^[21]可以计算吸附过程的焓变、熵变和自由能, 这些热力学参数可以反映温度对于吸附过程的影响。

$$\Delta G^\theta = -RT \ln K_d \quad (2)$$

$$\Delta G^\theta = \Delta H^\theta - T\Delta S^\theta \quad (3)$$

$$\ln K_d = -\frac{\Delta H^\theta}{RT} + \frac{\Delta S^\theta}{R} \quad (4)$$

式中, ΔH^θ 为标准吸附热, ΔS^θ 为吸附的标准熵变,

ΔG^θ 为吸附的标准自由能改变量, R 为摩尔气体常数 $[8.314 \text{ J} \cdot (\text{mol} \cdot \text{K})^{-1}]$, T 为热力学温度, K_d 为吸附系数。通过采用线性回归的方法, 用 $\ln K_d$ 对 $1/T$ 作图, 由斜率及截距可以求得 ΔH^θ 及 ΔS^θ , 相关参数结果列于表 2。

表 2 结果显示 ΔH^θ 均为正值, 说明吸附过程表现为焓阻碍效应。随温度升高, 硝基苯吸附量增加, 反应为吸热过程; 焓变较小 ($3 \sim 6 \text{ kJ} \cdot \text{mol}^{-1}$) 表明吸附过程中并无化学键力 ($63 \sim 84 \text{ kJ} \cdot \text{mol}^{-1}$)^[22] 和配

表 2 LDH-DS 吸附硝基苯的热力学参数

Table 2 Gibbs free energy, enthalpy, entropy changes associated with nitrobenzene by LDH-DS

吸附剂	$\Delta G^\theta / \text{kJ} \cdot \text{mol}^{-1}$			$\Delta H^\theta / \text{kJ} \cdot \text{mol}^{-1}$	$\Delta S^\theta / \text{J} \cdot (\text{mol} \cdot \text{K})^{-1}$
	298 K	308 K	318 K		
LDH-0.5DS	-12.80	-13.23	-13.67	3.85	42.98
LDH-1.0DS	-14.93	-15.43	-15.93	5.34	50.11
LDH-1.5DS	-15.11	-15.62	-16.13	5.03	50.73
LDH-2.0DS	-16.11	-16.65	-17.19	5.69	54.07

位键力(约 $40 \text{ kJ} \cdot \text{mol}^{-1}$)^[22]. ΔS^θ 均为正值,说明吸附反应是以熵推动为主^[23]. 焓变和熵变结果显示吸附过程是基于分配作用^[24]. ΔG^θ 均小于 0,说明吸附质倾向于从溶液到吸附剂表面,吸附过程为自发进行. 但吸附反应的自由能由于受吸附焓和吸附熵的双重影响,随温度的升高而降低, ΔG^θ 绝对值随着温度的升高而增大,说明在研究的温度范围内,其自发性随着温度的升高而增强^[25].

3 结论

SDS 改性的水滑石对硝基苯有较好的吸附效果,吸附容量远远大于未改性 LDH. 改性水滑石吸附等温线呈线性,可用线性方程拟合,表明硝基苯在表面活性剂改性 LDH 上的吸附是分配作用所致. 随着负载量的增加,吸附系数 K_d 值逐渐增大,而有机碳标化的吸附系数 K_{oc} 值保持相对恒定,说明有机阴离子的负载量对疏水性有机物的吸附有重要影响. 热力学实验表明吸附过程为吸热反应,以熵增加推动为主,其吸附自发性随着温度的升高而增强.

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