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废白土炭对印染废水中曙红 Y 和茜素红的吸附性能分析

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摘要 【目的】 文章介绍了废白土炭(SBE@C)对印染废水中曙红 Y(EY)和茜素红(AR)2种典型染料的吸附性能, 并探讨初始 pH 对其吸附能力的影响, 评估其在废水处理中的应用潜力。【方法】 将废白土(SBE)通过高温缺氧热解法制备为废白土炭(SBE@C)。采用吸附动力学和吸附等温线模型对 SBE@C 的吸附性能进行分析。试验中, 引入另一种污染物甲基橙(MO), 组成 3 种染料的复合染料, 通过不同投加量(1、2、3 g/L)的 SBE@C 对复合染料溶液进行吸附试验, 测定不同时间点的吸附量, 并分析初始 pH 对吸附性能的影响。【结果】 试验结果表明, SBE@C 对 EY 和 AR 的吸附符合伪二级动力学模型, 且随着投加量的增加, 吸附容量逐渐下降。EY 在 SBE@C 上的吸附符合 Freundlich 等温线模型, 表现为多层非均质表面吸附; 而 AR 则符合 Langmuir 等温线模型, 表明 AR 的吸附是单层吸附过程。在中性及碱性条件下, SBE@C 对染料溶液有较强的吸附能力。在不同初始 pH 条件下, SBE@C 对 EY 和 AR 的吸附能力表现出较强的 pH 依赖性。在中性及碱性条件下(pH 值=7、9、11), 其吸附能力显著增强, 特别是在 pH 值=7 和 9 时, 吸附容量较高。【结论】 SBE@C 具有良好的吸附性能, 能够有效去除印染废水中的 EY 和 AR 染料。SBE@C 的吸附性能受 pH 影响较大, 在中性和碱性条件下表现出更高的吸附效率。该研究表明, SBE@C 作为一种环保、经济的吸附材料, 具有广泛的应用前景, 特别是在实际废水处理中。

关键词 废白土炭(SBE) 印染废水 吸附 曙红 Y(EY) 茜素红(AR)

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Analysis of Spent Bleaching Earth Carbon for Adsorption Properties of Eosin Y and Alizarin Red in Printing and Dyeing Wastewater

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Abstract [Objective] The paper introduces the adsorption properties of spent bleaching earth carbon (SBE@C) for two typical dyes, eosin Y (EY) and alizarin red (AR), in printing and dyeing wastewater, and explores the effect of initial pH on the adsorption capacity. [Methods] Spent bleaching earth (SBE) was pyrolyzed under anoxic conditions to prepare SBE@C. The adsorption performance was evaluated using adsorption kinetics and isotherm models. In the experiment, methyl orange (MO) was introduced as an additional pollutant to form a multicomponent dye system comprising three dyes. Adsorption tests were conducted on this composite dye solution using different dosages (1, 2, 3 g/L) of SBE@C. The adsorption capacity at different time intervals was measured, and the influence of initial pH on adsorption performance was analyzed. [Results] The result showed that the adsorption of EY and AR by SBE@C followed the pseudo-second-order kinetic model, and the adsorption capacity decreased with increasing dosage. EY adsorption followed the Freundlich model, indicating multi-layer adsorption on a heterogeneous surface, while AR adsorption fitted the Langmuir

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model, suggesting a monolayer adsorption process. Under neutral and alkaline conditions, SBE@ C exhibits strong and adsorption capacity toward dye solutions. The adsorption performance of SBE@ C was pH-dependent, with stronger adsorption observed under neutral and alkaline conditions (pH value was 7, 9, 11), particularly at pH value was 7 and 9. [Conclusion] SBE@ C exhibits excellent adsorption performance for EY and AR dyes in printing and dyeing wastewater. The adsorption capacity is significantly influenced by pH, with optimal performance under neutral and alkaline conditions. This study indicates that SBE@ C is a promising, environmental friendly, and cost-effective adsorbent for wastewater treatment.

Keywords spent bleaching earth carbon (SBE) printing and dyeing wastewater adsorption eosin Y (EY) alizarin red (AR)

废白土(SBE)是油脂精炼脱色后失去活性的白土,含有大量油脂(20%~40%)、金属杂质和其他有机物^[1]。SBE 位列我国“国家危险废物清单”(HW06)^[2],常见处理方法有焚烧、填埋等^[3],但会造成二次污染。SBE 暴露于大气中易引发残油的氧化,氧化反应会引起 SBE 自燃,存在火灾隐患^[4]。因此,亟需一种能够安全处理和高效利用废白土的方法。

Loh 等^[5]将 SBE 与一些农业和棕榈油加工副产品共同堆肥,可以作为高效的生物有机肥料;Eliche-Quesada 等^[6]报道了 SBE 作为砖的成孔剂和硅前驱体的可行性;Suhartini 等^[7]将废白土制为煤饼以代替煤炭。

除上述用途之外,还可将 SBE 制备为吸附剂,去除废水中的污染物。Wambu 等^[8]对 SBE 进行酸活化,吸附溶液中 Cu(Ⅱ),去除率可达 98%;Mana 等^[9]用十六烷基三甲基溴化铵(CTAB)对 SBE 改性,去除染料酸性黑 10B,最大吸附容量为 100 mg/g;Tang 等^[10]采用水热法改性 SBE,对亚甲基蓝、Pb(Ⅱ) 和盐酸四环素的吸附容量分别为 271.28、180.90 mg/g 和 308.21 mg/g。

综上所述,SBE 能够通过改性转化为有效的水处理吸附剂,但制备水处理吸附剂所用的水热法、酸碱及表面活性剂改性法等存在二次污染、产量低、不易操作等缺点,因此实现 SBE 的简单高效、绿色经济处理是其再利用的关键。

曙红 Y(EY)是水溶性山叮类阴离子染料,其分子式为 $C_{20}H_6Br_4Na_2O_5$,红棕色粉末,分子式如图 1(a)所示。Henry Caro 通过荧光素的溴化反应发现了 EY,同年被用于制造业和纺织业^[11]。皮肤接触 EY 会导致红肿疼痛,眼睛接触可导致角膜的损害^[12],人体摄入会损坏肝肾等重要器官,并且 EY 的代谢产物在本质上也是致癌物^[13]。茜素红(AR)是水溶性蒽醌类阴离子染料,其分子式为 $C_{14}H_7NaO_7S \cdot H_2O$,橙黄色或黄棕色粉末,其分子式

如图 1(b)所示。AR 广泛应用于纺织、化妆品、造纸等行业,具有剧毒性、诱变性、致癌性,威胁人类健康^[10],人体吸入易引起呼吸系统疾病、肺气肿或尘肺病;体内摄入可能引起胃炎和高铁血红蛋白症^[14~16]。以上 2 种染料均为典型的印染废水污染物,目前处理染料污染物的方法有超生增电化学氧化法^[17]、生物分解法^[18]和吸附法^[19~21]等。与其他方法相比,吸附法具有价格低廉、操作简单、适用范围广泛等优点,是目前去除水体中有害物质的重要手段。

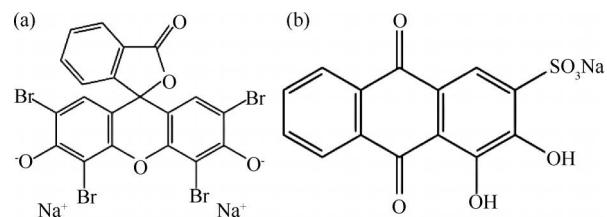


图 1 (a)EY 与(b)AR 的化学结构

Fig. 1 Chemical Structure of (a) EY and (b) AR

本研究将 SBE 制备为废白土炭(SBE@ C)吸附剂,选取印染废水中 2 种典型的染料(EY 和 AR),做单一吸附试验,并进一步引入另一种常见染料甲基橙(MO)组成复合染料,并分析初始 pH 对印染废水吸附性能的影响。

1 材料与方法

1.1 试验仪器与药剂

本试验采用的主要试剂有 MO($C_{14}H_{14}N_3O_3S$)、EY ($C_{20}H_6Br_4Na_2O_5$)、AR ($C_{14}H_7NaO_7S$)、盐酸(HCl)和氢氧化钠(NaOH),均购于上海麦克林生化科技有限公司(分析纯)。

试验采用的仪器如表 1 所示。

1.2 材料制备

SBE 原料由河南阳光油脂集团(河南郑州)提供。将 SBE 在 105 °C 下干燥 24 h,使用研钵研磨,过 100 目筛使其粒径小于 150 μm。放入管式炉中在氮气气氛下热解,控制氮气流速为 150 mL/min。

表 1 试验仪器
Tab. 1 Experimental Instruments

仪器	型号	生产商
磁力搅拌器	78-1	上海华燕医疗器械有限公司
标准 pH 计	PHS-3C	上海精密科学仪器有限公司
电子天平	HZK-FA110	福州华志科学仪器有限公司
双光束紫外可见分光光度计	TU-1900	北京普析通用仪器有限责任公司
程控箱式电炉	BCD-85	广东奥马电器股份有限公司

热解温度为 800 ℃, 恒温 2 h, 升温速率为 8 ℃/min。降至室温后, 取出晾干, 并标记为 SBE@ C。

1.3 试验及分析方法

1.3.1 吸附动力学

分别配制 20 mg/L 的 EY 和 AR 溶液 500 mL, 置于锥形瓶中, 加入 SBE@ C(1、2、3 g/L), 室温下置于磁力搅拌器上, 转速为 150 r/min。测定不同反应时间(0~180 min)样品的吸附量。

1.3.2 吸附等温线

将不同初始质量浓度(5~35 mg/L)的 EY 和 AR 溶液 100 mL 分别置于锥形瓶中, 加入 SBE@ C(1 g/L), 操作步骤同上。在平衡状态下, 测定 SBE@ C 对污染物的吸附量。

1.3.3 溶液初始 pH 的影响

在 500 mL 锥形瓶中加入 3 种初始质量浓度均为 20 mg/L 的染料溶液(MO、EY、AR), 使用 0.1 mol/L HCl 和 NaOH 调节染料溶液的初始 pH(pH 值 = 3、5、7、9、11), 加入一定量的 SBE@ C(1 g/L), 室温下置于磁力搅拌器上, 转速为 150 r/min。测定

不同初始 pH 下的平衡吸附量。

2 结果与讨论

2.1 吸附动力学

图 2 显示的是利用一级动力学模型^[22]、伪二级动力学模型^[23]和颗粒内扩散模型^[24]对 SBE@ C 吸附 EY 和 AR 的吸附动力学数据拟合结果。

一级动力学模型计算如式(1)。

$$q_t = q_e \times (1 - e^{-k_1 t}) \quad (1)$$

伪二级动力学模型计算如式(2)。

$$q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t} \quad (2)$$

颗粒内扩散模型计算如式(3)。

$$q_t = k_3 t^{0.5} + b \quad (3)$$

其中: q_t —吸附容量, mg/g;

q_e —平衡吸附容量, mg/g;

t —反应时间, min;

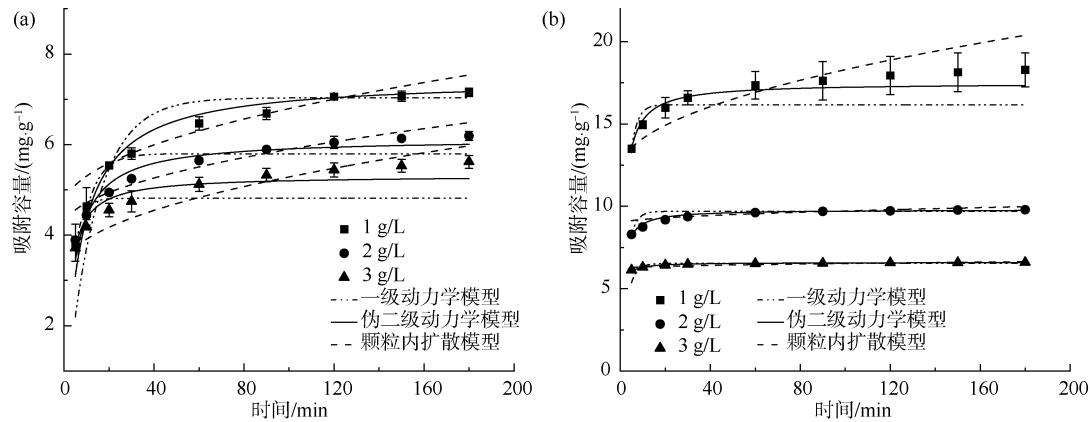
k_1 —一级动力学模型的速率常数, min^{-1} ;

k_2 —伪二级动力学模型的速率常数, $\text{g}/(\text{mg} \cdot \text{min})$;

k_3 —颗粒内扩散模型的速率常数, $\text{mg}/(\text{g} \cdot \text{min}^{1/2})$;

b —边界层厚度相关常数, mg/g。

如图 2 所示, 投加量越大, SBE@ C 的吸附容量越小, 其原因可能是投加量增加, 为污染物提供了更



注: $C_{\text{EY}} = 20 \text{ mg/L}$, $C_{\text{AR}} = 20 \text{ mg/L}$, 体积 = 500 mL, 转速 = 150 r/min, 温度 = 25 ℃。

图 2 (a) EY 和(b) AR 的吸附动力学曲线

Fig. 2 Adsorption Kinetic Curves of (a) EY and (b) AR

多吸附位点,而污染物的浓度未增加,使其只占据部分活性位点,吸附容量降低^[25],平衡时间随之缩短。如图2(a)所示,1、2、3 g/L的SBE@C对EY的吸附容量在180 min时分别为7.16、6.19、5.62 mg/g。如图2(b)所示,1、2、3 g/L的SBE@C对AR的吸附容量在180 min时分别为18.29、9.97、6.59 mg/g。投加量为3 g/L时,SBE@C对EY的吸附在30 min达到平衡;投加量为3 g/L时,SBE@C对EY的平

衡吸附容量为5.62 mg/g。相较EY,SBE@C对AR是一个快速吸附过程,且吸附容量最高,可达18.29 mg/g。

表2是SBE@C吸附AR和EY的动力学模型拟合参数,由表2可知,伪二级动力学模型($R^2 > 0.92$)比一级动力学模型($R^2 > 0.60$)更符合该反应过程,表明SBE@C吸附染料的动力学过程均由化学吸附主导^[26]。

表2 AR 和 EY 的动力学模型拟合参数
Tab. 2 Fitting Parameters of Kinetic Models for AR and EY

目标污 染物	投加量/ (g·L ⁻¹)	一级动力学模型		伪二级动力学模型		颗粒内扩散模型	
		k_1/min^{-1}	R^2	$k_2/$ [g·(mg·min) ⁻¹]	R^2	$k_3/$ [mg·(g·min ^{1/2}) ⁻¹]	R^2
AR	1.0	0.358 3	0.863	0.038 6	0.980	0.611 0	0.837
	2.0	0.378 0	0.763	0.103 1	0.970	0.076 1	0.579
	3.0	0.344 5	0.697	0.323 5	0.992	0.031 4	0.906
EY	1.0	0.075 0	0.890	0.019 0	0.976	0.217 7	0.924
	2.0	0.153 0	0.607	0.041 4	0.921	0.172 4	0.885
	3.0	0.294 6	0.787	0.087 3	0.954	0.201 1	0.922

2.2 吸附等温线

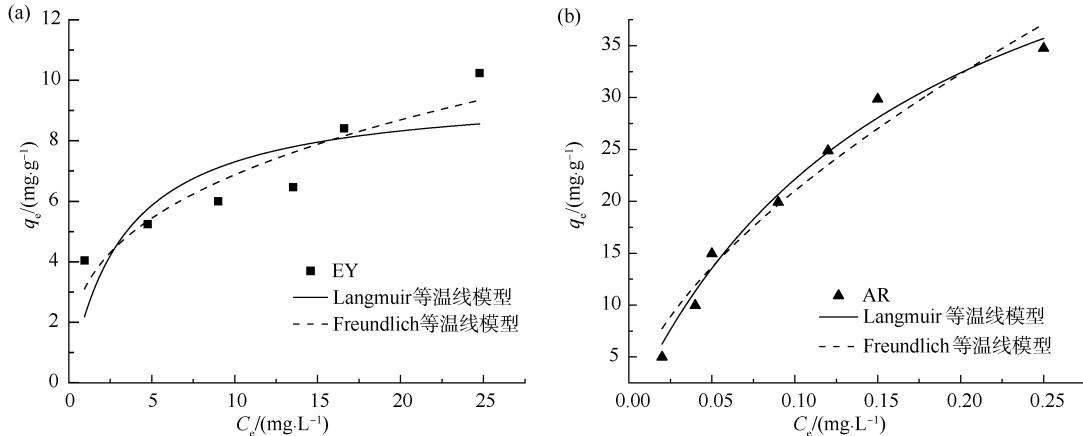
图3为Langmuir^[27]和Freundlich等温线模型^[28]对SBE@C吸附EY和AR的拟合结果。

Langmuir等温线模型计算如式(4)。

$$q_e = \frac{Q_0 K_L C_e}{1 + K_L C_e} \quad (4)$$

Freundlich等温线模型计算如式(5)。

$$q_e = K_F C_e^{1/n} \quad (5)$$



注: $C_{\text{EY}} = C_{\text{AR}} = 5 \sim 35 \text{ mg/L}$, 投加量 = 1 g/L, 体积 = 100 mL, 转速 = 150 r/min, 温度 = 25 °C, 反应时间 = 24 h。

图3 (a) EY 和 (b) AR 在 SBE@C 上的吸附等温线

Fig. 3 Adsorption Isotherms of (a) EY and (b) AR at SBE@C

其中: Q_0 ——最大吸附容量, mg/g;

K_L ——Langmuir 常数, L/μg;

C_e ——平衡时污染物质量浓度, mg/L;

K_F ——Freundlich 常数, $(\text{mg}/\text{kg}) \cdot (\text{L}/\text{mg})^{1/n}$ 。

图3为吸附容量 q_e 与平衡浓度 C_e 之间的关系曲线。如图3所示,随着初始浓度的增加,平衡浓度上升,SBE@C对2种染料的吸附容量均不断升高,当初始质量浓度增加至35 mg/L时,EY和AR的吸

附容量分别为 10.24 mg/g 和 34.75 mg/g 。这可能是因为高浓度污染物使染料与吸附剂间作用力增大,从而增大 SBE@ C 的吸附容量^[29]。结果显示, EY 在 SBE@ C 上的吸附更符合 Freundlich 等温线模型,其 R^2 为 0.862,表明 SBE@ C 对 EY 的吸附可能在多层非均质表面。AR 在 SBE@ C 上的吸附更符合 Langmuir 模型($R^2=0.982$),说明 AR 可能是单层吸附占主导^[30]。

2.3 零电荷点(pH_{PZC})分析

当溶液中决定电位离子的浓度为某一特定值时,固体表面上的净电荷等于 0,两相(固/液)之间由自由电荷引起的电位差也为 0, pH_{PZC} 即为此时溶液中决定电位离子的浓度。材料的表面电荷也是影响吸附性能的重要指标。使用 Zhu 等^[31]报道的方法测定 SBE@ C 的 pH_{PZC} ,结果如图 4 所示。SBE@ C 的 pH_{PZC} 为 11.73,即当 pH 值小于 11.73 时,SBE@ C 表面带正电荷,当 pH 值大于 11.73 时,则带负电荷。该结果与之前的本课题组研究有所差异^[32],可能是因为 SBE 的批次不同导致所制备的 SBE@ C 的 pH_{PZC} 也差别较大。

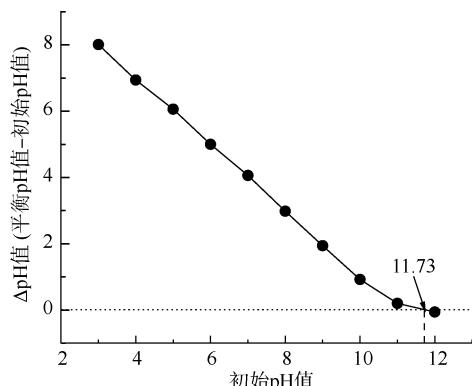


图 4 SBE@ C 的 pH_{PZC}

Fig. 4 pH_{PZC} of SBE@ C

2.4 溶液初始 pH 的影响

在实际印染废水中,通常存在不同浓度的其他离子。为真实模拟印染废水中复杂染料种类,在此引入新的染料污染物 MO,对比 SBE@ C 对不同染料的吸附性能。图 5 是不同初始 pH 对 SBE@ C 吸附性能的影响。结果表明,当 pH 值为 3 时,SBE@ C 对 MO、EY 和 AR 3 种染料的吸附容量均达最低值,分别为 4.21 、 4.00 mg/g 和 4.00 mg/g 。而当初始 pH 值为 7、9 和 11 时,吸附容量基本没有变化,说明在中性和碱性条件下,SBE@ C 对染料溶液均有较好的吸附性能。

液仍有较好的吸附性能,这将有利于其在实际废水中的应用。SBE@ C 的 pH_{PZC} 为 11.73,即当 pH 值小于 11.73 时,SBE@ C 表面带正电荷。酸性条件下吸附容量降低,可能是因为溶液酸度过高,与 SBE@ C 表面带的正电荷相排斥,所以吸附容量下降^[33]。

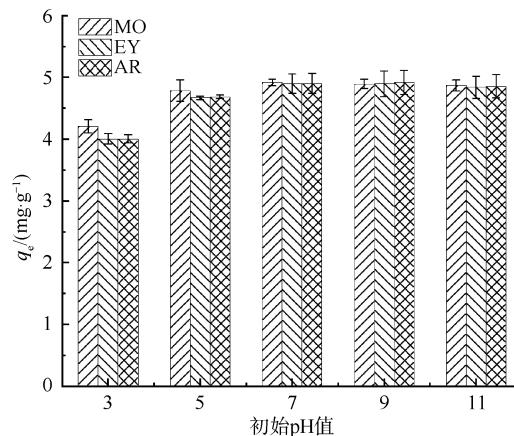


图 5 溶液初始 pH 值对 SBE@ C 吸附的影响

Fig. 5 Effect of Initial pH Value of Solution on Adsorption of SBE@ C

3 结论

(1) 吸附动力学表明:随着投加量的增加,吸附容量逐渐降低;SBE@ C 对 EY 和 AR 2 种染料的吸附动力学更符合伪二级动力学模型,表明 SBE@ C 吸附染料的动力学过程均由化学吸附决定。

(2) 吸附等温线表明:EY 在 SBE@ C 上的吸附更符合 Freundlich 模型,表明其吸附可能在多层非均质表面;AR 更符合 Langmuir 模型,表明 AR 是单层吸附占主导。

(3) 当溶液初始 pH 值为 7、9、13 时,SBE@ C 对 MO、EY 和 AR 的吸附容量相较于 pH 值为 3、5 时有所下降,表明在中性和碱性条件下,SBE@ C 对染料溶液均有较好的吸附性能。

(4) SBE@ C 的 pH_{PZC} 为 11.73,表明当 pH 值小于 11.73 时,SBE@ C 表面带正电荷,当 pH 值大于 11.73 时,带负电荷。此外,当溶液酸度过高时染料分子呈酸性,与 SBE@ C 表面带的正电荷相排斥,导致吸附容量下降。

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