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改性花生壳对水中镉的动态吸附研究

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摘要: 采用高锰酸钾改性花生壳吸附剂对镉离子进行固定床吸附实验, 考察了床层高度(30~50 cm)、初始离子浓度(0.55~11.00 mg·L⁻¹)、进料流速(15.11~37.00 mL·min⁻¹)等操作参数对镉吸附特性的影响, 同时对吸附穿透曲线进行拟合. 实验结果表明, 改性花生壳固定床对水中镉具有较好的吸附效果, 在吸附操作初期, 吸附柱出水镉离子浓度几乎为 0 (<0.001 mg·L⁻¹), 吸附操作时间根据不同的操作条件可达 2~62 h, 镉离子总去除率均大于 54%. 传质区长度主要受初始离子浓度、进料流速影响. 床层高度的增加使得穿透时间增加, 但传质区长度几乎保持不变; 初始离子浓度和进料流速增加, 穿透时间缩短, 传质区长度增加. 在低浓度条件下, BDST 模型实验穿透曲线拟合效果较好 ($R^2 > 0.99$), 运用该模型能准确预测吸附柱的操作时间.

关键词: 花生壳; 镉; 吸附; 固定床; 穿透曲线

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Dynamic Adsorption of Cadmium (II) in Water on Modified Peanut Shells

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Abstract: Peanut shells modified by potassium permanganate were used as adsorbents for cadmium (II) removal in a fixed bed. The effects of influencing parameters such as bed height (ranging between 30 and 50 cm), initial concentration (ranging between 0.55 and 11.00 mg·L⁻¹) and influent flow rate (ranging between 15.11 and 37.00 mL·min⁻¹) were studied and the corresponding breakthrough curves were obtained. This result indicated that the column packed with modified peanut shells had good adsorption properties for cadmium (II) removal. In the initial adsorption stage cadmium (II) outflow concentration was less than 0.001 mg·L⁻¹. According to different operating conditions the operation time reached 2-62 h and the removal rate was above 54%. The height of mass transfer zone was primarily driven by initial concentration and influent flow rate. The height of mass transfer zone kept substantially unchanged and the breakthrough time increased with the increase of bed height. The height of mass transfer zone increased and the breakthrough time decreased with the elevated initial concentration and influent flow rate. The bed depth service time (BDST) model was used to fit the experiment data resulting in a good effect with $R^2 > 0.99$ under low concentration. The operation time can be accurately predicted using the BDST model.

Key words: peanut shell; cadmium; adsorption; fixed-bed; breakthrough curve

重金属污染的危害关键在于重金属为非降解型污染物, 一旦进入环境将被生物吸收并通过食物链富集, 危害人体健康. 镉是一种毒害性很强的重金属元素, 被广泛应用于电镀、冶炼、染料、电池行业等, 它积存于人体肝或肾脏从而对健康造成危害, 还可导致骨质疏松和软化.

化学沉淀法、电解法、吸附法、离子交换法、膜分离法等传统方法^[1-5]在含镉废水的处理上应用十分广泛, 但仍然存在诸多问题, 如成本高、对于排放标准严格的水源保护区传统处理方法难以达标等. 生物吸附法由于具有效果好、投资少、运作费用低等优点备受人们的关注. 目前国内外报道了很多可用作吸附镉的生物材料, 主要有果皮^[6-8]、椰子壳粉^[9,10]、稻壳^[11,12]、树皮^[13,14]、树枝

叶^[15,16]、改性玉米秸秆^[17]、藻类^[18,19]等, 但对于上述吸附材料的研究更多的是集中在静态实验及吸附机制的探讨上, 且由于其粉末状、机械强度低等缺点, 难以在塔式吸附或者水体原位吸附中直接应用.

经前期实验筛选出一种对镉吸附性能良好的颗粒状改性花生壳吸附剂^[20], 该吸附剂对 Cd²⁺ 的吸附量最大达 43.11 mg·g⁻¹, 采用该吸附剂进行镉的吸附柱实验, 绘制不同操作条件下的吸附穿透曲线并分析该吸附剂对镉的动态吸附特性, 以期为其在

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固定床和河流原位吸附的工业化应用提供理论依据.

1 材料与方 法

1.1 材料与设备

实验用花生壳来源于江西永新县某花生榨油厂,将原花生壳洗净烘干、捣碎,取 10 目筛下和 20 目筛上花生壳颗粒,在常温下经高锰酸钾溶液搅拌浸泡一定时间,取出后用去离子水洗净,放入 60℃ 烘箱烘干备用.

镉溶液采用氯化镉配制,高锰酸钾改性花生壳吸附镉离子在 pH 为 4.5 ~ 6.5 时效果最佳^[20],本实验配制的溶液 pH 为 4.7. 实验所用试剂均为分析纯,镉的浓度采用日立 Z-2000 型塞曼原子吸收分光光度计测定.

1.2 实验方法

1.2.1 吸附柱实验

固定床吸附实验采用自制装置. 固定床装置如图 1 所示. 吸附柱材料为玻璃材质,规格为 ϕ 20 mm \times 1 000 mm. 把改性好的花生壳吸附剂(密度为 $0.49 \text{ g}\cdot\text{cm}^{-3}$)装入吸附柱,并加入 2 L 去离子水浸润使吸附剂填充更均匀,再将特定浓度的镉溶液通过蠕动泵自上而下恒速加入吸附柱中,定时检测流出液浓度.

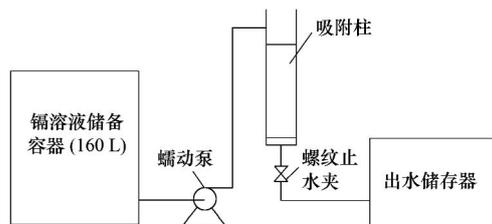


图 1 实验装置示意

Fig. 1 Experimental set-up

1.2.2 吸附柱参数计算

空床停留时间(EBRT)是衡量吸附柱操作成本的参数之一,空床停留时间减少,吸附传质区缩短,床层得以更充分利用,但吸附柱的处理率减小. 空床停留时间由式(1)计算:

$$\text{EBRT} = \frac{V_b}{Q} \quad (1)$$

从吸附柱开始操作到穿透终点吸附柱吸附镉的总量 M_{ad} 可由穿透曲线与初始浓度的直线所围成的积分面积进行计算,计算式如式(2). 改性花生壳吸附剂的动态吸附量 q 由式(3)计算.

$$M_{\text{ad}} = \frac{Q}{1\,000} \int_0^{t_e} (c_0 - c_e) dt \quad (2)$$

$$q = \frac{M_{\text{ad}}}{m} = \frac{M_{\text{ad}}}{\rho_0 V_b} \quad (3)$$

从吸附柱开始操作到穿透终点流过的镉总量 $M_{\text{total}} = c_0 Q t / 1\,000$, 则吸附柱对镉的总去除率 R 由式(4)可计算得出:

$$R = \frac{M_{\text{ad}}}{M_{\text{total}}} \quad (4)$$

吸附区内剩余吸附容量分率:

$$f = \frac{\int_{t_b}^{t_e} (c_0 - c_e) dt}{c_0 (t_a - t_b)} \quad (5)$$

吸附床层的传质区长度^[21]:

$$H_{\text{MTZ}} = \frac{c_0 Q}{q \rho_0 A} (t_e - t_b) \quad (6)$$

式中, Q 为进水流量 ($\text{L}\cdot\text{h}^{-1}$); m 为吸附柱填充吸附剂质量 (g); c_0 为初始镉离子浓度 ($\text{mg}\cdot\text{L}^{-1}$); c_e 为出水中镉离子浓度 ($\text{mg}\cdot\text{L}^{-1}$); V_b 为吸附柱床层体积 (cm^3); t_b 为穿透时间 (h); t_e 为穿透终点时间 (h); ρ_0 为床料密度 ($\text{g}\cdot\text{cm}^{-3}$); A 为吸附柱横截面积 (cm^2).

1.2.3 穿透曲线的模型拟合

BDST (bed depth service time) 模型是最普遍地应用于固定床吸附的简化模型之一^[22~24]. 应用该模型可以预测在不同的进料流速、床层高度、进料流速等操作条件下的吸附操作时间.

$$t = \frac{N_0}{c_0 v} z - \frac{1}{K c_0} \ln \left(\frac{c_0}{c} - 1 \right)$$

式中, c_0 为进水初始浓度 ($\text{mg}\cdot\text{L}^{-1}$); c 为出水浓度 ($\text{mg}\cdot\text{L}^{-1}$); K 为吸附速率常数 [$\text{L}\cdot(\text{mg}\cdot\text{h})^{-1}$]; N_0 为最大吸附容量 ($\text{mg}\cdot\text{L}^{-1}$); z 为吸附柱高度 (cm); v 为进水线速度 ($\text{cm}\cdot\text{h}^{-1}$); t 为吸附时间 (h).

2 结果与分析

2.1 吸附柱参数计算

在实验浓度范围内,改性花生壳吸附柱对水中镉离子具有较好的吸附效果,在吸附操作初期,吸附柱出水镉离子浓度几乎为 0 ($< 0.001 \text{ mg}\cdot\text{L}^{-1}$),吸附操作时间根据不同的操作条件可达 2 ~ 62 h,镉离子总去除率均大于 54%.

2.2 操作方式对穿透曲线的影响

2.2.1 不同吸附柱高度对穿透曲线的影响

初始浓度为 $2 \text{ mg}\cdot\text{L}^{-1}$, 进料流速为 $8.39 \text{ cm}\cdot\text{min}^{-1}$, 在不同填料高度下测定高锰酸钾改性花

生壳吸附柱对 Cd^{2+} 的吸附性能,绘制的穿透曲线如图 2 所示. 吸附柱高度分别为 30、40、50 cm 时,吸附柱穿透时间分别为 2.17、12.40 和 16.86 h,随着吸附柱高度的增加,吸附质与吸附材料的接触时间

增加,提高了吸附材料对镉离子的吸附量,穿透时间推迟,但传质区长度和穿透曲线形状几乎无变化,这是因为吸附平衡和传质扩散速率不随吸附柱高度的变化而变化.

表 1 不同操作条件下吸附柱的参数计算结果¹⁾

Table 1 Parameter results under different operating conditions

c_0 / $\text{mg}\cdot\text{L}^{-1}$	v / $\text{cm}\cdot\text{min}^{-1}$	z / cm	EBRT / min	t_b / h	t_e / h	$R/\%$	$f/\%$	H_{MTZ} / cm
2.00	8.39	30	3.57	2.17	40.67	62.69	39.42	28.29
2.00	8.39	40	4.77	12.40	52.00	72.33	36.33	27.49
2.00	8.39	50	5.96	16.86	60.00	70.03	40.50	29.96
0.55	8.39	40	4.77	49.09	180.27	79.68	30.71	22.71
11.00	8.39	40	4.77	5.38	43.00	54.26	57.45	39.40
2.00	4.81	40	8.31	62.00	124.69	83.16	34.09	13.55
2.00	11.82	40	3.39	2.06	40.00	63.95	38.00	38.34

1) 本实验取出水浓度为 $0.001 \text{ mg}\cdot\text{L}^{-1}$ 时的时间为吸附穿透时间 t_b ,出水浓度为初始浓度的 95% 时的时间为吸附穿透终点时间 t_e ^[25,26], t_b 、 t_e 由线性插值法求得

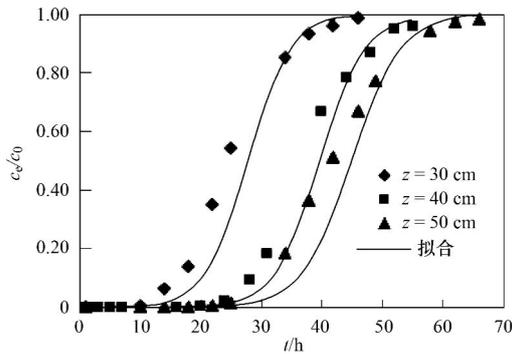


图 2 床层高度对穿透曲线的影响

Fig. 2 Effects of bed height on the breakthrough curves

2.2.2 不同进料流速对穿透曲线的影响

进料流速是吸附柱操作中的重要参数,它直接影响吸附剂与吸附质接触时间,从而影响吸附的传质速率. 实验选取 4.81 、 8.39 、 $11.82 \text{ cm}\cdot\text{min}^{-1}$ 共 3 个流速,在初始浓度为 $2 \text{ mg}\cdot\text{L}^{-1}$,吸附柱高度为 30 cm 条件下测绘的穿透曲线如图 3 所示. 当进料流速由 $4.81 \text{ cm}\cdot\text{min}^{-1}$ 增加到 $8.39 \text{ cm}\cdot\text{min}^{-1}$ 和 $11.82 \text{ cm}\cdot\text{min}^{-1}$ 时,吸附柱的穿透时间分别由 62.00 h 减少至 12.40 h 和 2.06 h,这是由于随着进料流速的增加吸附剂与吸附质之间的接触时间减少,传质区长度增加,穿透时间缩短. 进料流速降低,吸附剂与吸附质之间的接触时间增加,固定床层的利用率增加,因而固定床的剩余吸附容量分率呈下降的趋势.

2.2.3 不同初始浓度对穿透曲线的影响

吸附柱高度为 40 cm,进料流速为 $8.39 \text{ cm}\cdot\text{min}^{-1}$,不同初始浓度时吸附柱的穿透曲线如图 4. 当初始浓度为 0.55 、 2.00 、 $11.00 \text{ mg}\cdot\text{L}^{-1}$ 时吸附

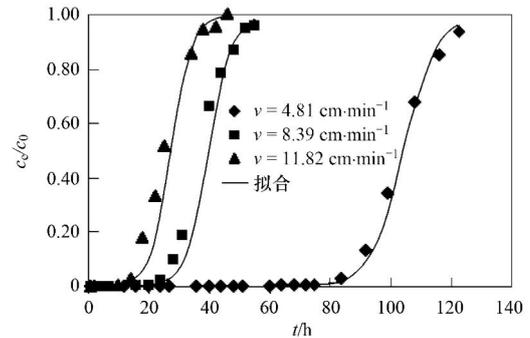


图 3 进料流速对穿透曲线的影响

Fig. 3 Effects of influent flow rate on the breakthrough curves

柱穿透时间分别为 49.09、12.40、5.38 h,初始浓度增加,吸附剂单位时间吸附的镉离子量增加,因而吸附柱达到穿透点的速度更快. 随着初始浓度的增加,传质区长度增加,穿透曲线形状变陡,床层利用率降低,固定床剩余吸附容量分率增加.

2.2.4 各操作因素对固定床传质区长度的影响比较

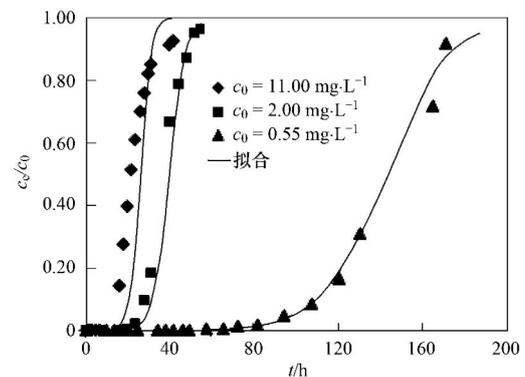


图 4 初始浓度对穿透曲线的影响

Fig. 4 Effects of initial concentration on the breakthrough curves

为了考察床层高度、初始离子浓度和进料流速对吸附穿透特性影响程度的大小,采用传质区长度随各操作因素变化的相应平均变化率即 $|\Delta y/\Delta x|$ 来衡量^[16],计算得出传质区长度对床层高度、处理离子浓度、进料流速的平均变化率分别为 0.08、2.07、3.53,可以看出床层高度的变化对传质区长度的影响较小,初始离子浓度和进料流速对传质区长度影响较大,其中进料流速对传质区长度的影响最大。

2.3 穿透曲线的模型拟合

在不同操作条件下 BDST 模型的穿透曲线拟合参数见表 2。从中可知,BDST 模型得出的穿透曲线与实验数据所得穿透曲线相关性较好,实验所得穿

透时间与模型计算出的理论穿透时间相差不大,这说明 BDST 模型能够较好地预测镉离子在高锰酸钾改性花生壳吸附剂上的穿透特性。溶液初始浓度为 $0.55 \text{ mg}\cdot\text{L}^{-1}$ 时,模型拟合得出的可决系数 R^2 更高,理论穿透时间与实际穿透时间的误差更小,即 BDST 模型更适用于低浓度镉离子的固定床吸附过程模拟,这是由于 BDST 模型是基于表面吸附而建立的,未考虑吸附剂的内扩散作用,而在低浓度情况下,吸附剂的内扩散作用可忽略不计。图 2~4 显示拟合穿透曲线与实际穿透曲线相比,吸附中间段实际出水浓度比理论出水浓度偏高,这说明吸附过程内扩散缓慢。

表 2 不同操作条件下穿透曲线拟合参数

Table 2 Fitting parameters under different operating conditions

c_0 / $\text{mg}\cdot\text{L}^{-1}$	v / $\text{cm}\cdot\text{min}^{-1}$	z / cm	R^2	N_0 / $\text{mg}\cdot\text{L}^{-1}$	K / $\text{L}\cdot(\text{mg}\cdot\text{h})^{-1}$	理论 t_b / h	实际 t_b / h
2.00	8.39	30	0.949 7	933.14	0.14	1.19	2.17
2.00	8.39	40	0.965 9	1 000.72	0.13	11.50	12.40
2.00	8.39	50	0.963 9	905.81	0.12	14.08	16.86
0.55	8.39	40	0.994 5	898.88	0.13	49.85	49.09
11.00	8.39	40	0.886 1	3 262.27	0.04	2.81	5.38
2.00	4.81	40	0.995 6	1 500.62	0.09	63.14	62.00
2.00	11.82	40	0.953 4	965.95	0.15	1.82	2.06

3 结论

(1)在实验浓度($0.55 \sim 11 \text{ mg}\cdot\text{L}^{-1}$)范围内,经改性花生壳固定床吸附后水中镉离子浓度低于 $0.001 \text{ mg}\cdot\text{L}^{-1}$,吸附操作时间根据不同的操作条件可达 2~62 h,改性花生壳是一种潜在的镉离子吸附剂,可用于塔式吸附和固定床原位修复水中的镉离子。

(2)床层高度、进料流速、初始离子浓度均会影响动态吸附过程,随着进料流速、初始浓度的增加,传质区长度增加,穿透时间缩短,其中进料流速对吸附区长度的影响程度最大。

(3)在低浓度情况下 BDST 模型能成功拟合改性花生壳吸附柱吸附镉的过程,运用该模型能预测吸附操作时间,可为实际应用提供理论基础。

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