

煤基聚苯胺对苯酚废水的吸附性能和机理研究

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摘要:为有效去除水体中持久存在、难降解的有机污染物苯酚,采用高能球磨获得褐煤煤粉制备合成煤基聚苯胺,利用扫描电镜、傅里叶变换红外光谱等方法对原煤粉及煤基聚苯胺进行表征。以原煤粉做对照试验,研究了煤基聚苯胺对苯酚模拟废水的吸附性能和机理。结果表明,煤基聚苯胺吸附剂孔结构和分布比原煤粉好,具有较多中孔和微孔结构,有利于增大吸附容量。pH=4时,褐煤原煤粉和煤基聚苯胺去除率最大分别为22.74%和45.32%,因此酸性条件下,2种吸附剂对苯酚溶液的吸附效果较好。煤基聚苯胺对苯酚的去除效果较好,最佳投加量为1 g/L,去除率和饱和吸附量分别为45.32%和32.52 mg/g。煤基聚苯胺吸附剂对苯酚吸附符合 Lagergren 二级吸附动力学模型与 Freundlich 吸附等温模型。煤基聚苯胺吸附剂制备简单,原料易得,具有较好的工业化应用潜力。

关键词:煤基聚苯胺;苯酚;废水;吸附性能;褐煤

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Adsorption properties and mechanism of coal-based polyaniline to phenol wastewater

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Abstract: In order to effectively remove the persistent and refractory organic pollutants phenol in water. Coal-based polyaniline was prepared by lignite coal milled with high energy ball. Various measures such as SEM and FTIR were used to characterize the structure of coal-based polyaniline and raw coal powder. Adsorption properties and mechanism of coal-based polyaniline to phenol solution were studied with raw coal powder as the control experiment. The results show that pore structure and distribution of the coal-based polyaniline adsorbent are better than that of raw coal powder and it has more mesopores and microporous, which is conducive to increase the adsorption capacity. When pH is 4, the removal rate of raw coal powder and coal-based polyaniline are 22.74% and 45.32%, respectively. In acidic conditions, the adsorption effect of the adsorbents on phenol solution are better. The adsorption effect of coal-based polyaniline on phenol solution is better than that of raw coal powder, which optimum dosage is 1 g/L, and the removal rate and adsorption capacity are 45.32% and 32.52 mg/g, respectively. The adsorption of coal-based polyaniline adsorbent on phenol follows the Lagergren second-order adsorption kinetics model and the Freundlich adsorption isotherm model. Coal-based polyaniline adsorbent has good potential for industrial applications due to its simple preparation procedure and easily available raw materials.

Key words: coal-based polyaniline; phenol; wastewater; adsorption; lignite

0 引 言

有机污染物(如苯酚及其衍生物)是废水中最重要的污染物之一,主要来源于天然气和焦炭制造、

制革、纺织、塑料、橡胶、制药、石油等化工行业^[1]。苯酚在水体中存在时间持久,对生物降解具有抗性,超过一定浓度对人和环境中生物体有毒有害^[2],因此成为人们关注的热点。目前,物理吸附法^[3]、化

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学氧化法^[4]、溶剂萃取法^[5]以及光催化法^[6]等已被广泛用于去除苯酚废水,其中物理吸附法工艺操作简单,去除率高,在工业中应用广泛。

聚苯胺(PANI)是一种高分子多胺聚合物,具有稳定性良好、导电率高、成型工艺简单、无毒和原料价廉易得等优点^[7]。PANI及其复合材料由于具有大量的胺和亚胺基团,能够与有机污染物相互作用,去除水溶液中的污染物^[8]。Wang等^[9]通过聚苯胺吸附剂去除水溶液中的鞣酸,最大吸附量为117.65 mg/g,可能是由于鞣酸与聚苯胺之间存在静电相互作用的结果。Belaib等^[10]将聚苯胺涂覆到硅胶上以改变其表面结构来去除水溶液中苯酚,试验证明聚苯胺包覆硅胶等材料的吸附效率高,为消除水溶液中有机污染物如苯酚提供了良好的吸附剂。煤是一种大分子有机矿物岩石,反应活性高且含有丰富的孔隙结构、原料易得,是较为理想的煤基化工和吸附材料^[11]。

本文综合煤的纳米孔结构及大分子结构特征,以高能球磨处理后的褐煤煤粉为原料,与聚苯胺经引发聚合制得煤基聚苯胺吸附剂,研究其对苯酚模拟废水的吸附和机理。

1 试验

1.1 试验材料和仪器

试验所用褐煤采自内蒙古鄂尔多斯。

主要化学试剂:苯胺,分析纯,天津化学试剂三厂;盐酸,分析纯,西安化学试剂厂;过硫酸铵溶液(APS),分析纯,国药集团化学试剂有限公司;苯酚,天津盛奥化学试剂有限公司;4-氨基安替比林,分析纯,华东师范大学化工厂;去离子水。

主要试验仪器:VIS-7220型紫外可见分光光度计(北京瑞利分析仪器公司);DF-101S集热式恒温加热磁力搅拌器(陕西太康生物科技有限公司);HH-6数显恒温水浴振荡器(常州国华电器有限公司);PHS-3C型精密pH计(上海雷磁仪器厂);Quanta 200型扫描电镜(荷兰FEI公司);TENSOR II型傅里叶变换红外光谱仪(布鲁克光谱仪器公司)。

1.2 煤基聚苯胺的制备

煤基聚苯胺参照文献^[12]制备。室温下,将一定量经高能球磨过筛后的褐煤煤粉和经减压蒸馏后的精制苯胺(An)与一定体积的盐酸(2 mol/L)混匀,搅拌溶胀一定时间后,将过硫酸铵溶液(APS)加入其中引发聚合(APS与An摩尔比为1:1);再将

产物用去离子水反复冲洗至滤液呈无色,所得固体置于真空干燥箱60℃下干燥至恒重。样品如图1所示。



图1 煤基聚苯胺样品

Fig. 1 Coal-based polyaniline sample

1.3 测试分析方法

溶液中苯酚测定采用4-氨基安替比林分光光度法。

1.4 吸附试验

吸附动力学:向100 mL质量浓度为1 g/L的苯酚溶液中加入1g煤基聚苯胺吸附剂或原煤粉,25℃下分别震荡10、30、60、90、120、150、180 min后过滤,测定苯酚溶液的浓度。

吸附热力学:取100 mL质量浓度分别为100、200、300、500、800、1 000 mg/L的苯酚溶液,加入1g煤基聚苯胺吸附剂或原煤粉,25℃下震荡1 h后过滤,测定苯酚溶液的浓度。

pH值及投加量对吸附的影响:取100 mL质量浓度为1 g/L的苯酚溶液,加入1g煤基聚苯胺吸附剂或原煤粉,用0.1 mol/L的HCl与NaOH溶液调节不同的pH值后,常温震荡1 h过滤,测定苯酚溶液的浓度;向同体积同浓度的苯酚溶液中加入不同量的煤基聚苯胺吸附剂或原煤粉,常温下震荡1 h后过滤,测定苯酚溶液的浓度。

2 试验结果与讨论

2.1 原煤粉和煤基聚苯胺吸附剂的分析表征

2.1.1 原煤粉性质

试验所用褐煤的工业分析和元素分析见表1。

表1 褐煤的工业分析和元素分析

Table 1 Proximate and ultimate analysis of lignite

工业分析/%				元素分析/%			
M_{ad}	A_{ad}	V_{ad}	FC_{ad}	C_{ad}	H_{ad}	N_{ad}	O_{ad}
14.94	8.78	33.87	42.41	73.95	6.56	1.33	18.16

由表1可知,褐煤的挥发分及氢、氮、氧含量较高,说明褐煤的化学结构中含有多种活性官能团,化学反应性较好^[13]。

2.1.2 扫描电镜分析

褐煤原煤粉和煤基聚苯胺吸附剂放大不同倍数的扫描电镜照片如图2所示。制得的煤基聚苯胺吸附剂为颗粒状固体,平均粒径为2~3 mm。由图2可知,与原煤粉相比,煤基聚苯胺吸附剂的颗粒孔隙有所减少,这是由于原煤粉颗粒与聚苯胺颗粒在溶胀聚合时将原来的部分孔隙覆盖填充。由图2(c)和图2(d)看出,煤基聚苯胺吸附剂形成了较多絮状体且具有相当多的孔隙结构,更有利于吸附污染物。因此煤基聚苯胺吸附剂的孔结构和分布比原煤粉好,有利于增大吸附容量。

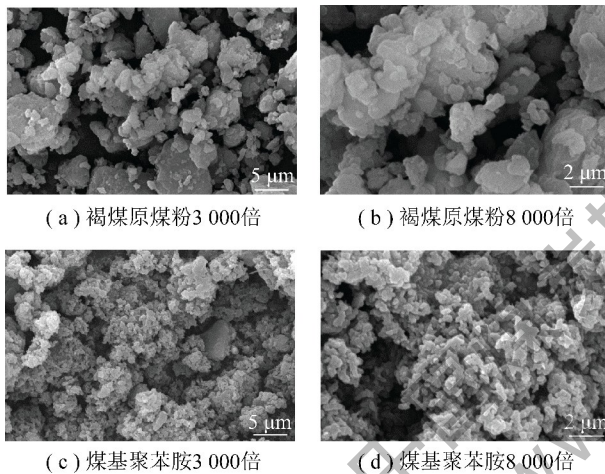


图2 褐煤原煤粉和煤基聚苯胺的 SEM

Fig. 2 SEM of lignite coal power and coal-based polyaniline

2.1.3 红外光谱分析

用傅里叶变换红外光谱仪对褐煤原煤粉和煤基聚苯胺吸附剂进行分析,采用KBr压片法。褐煤原煤粉及煤基聚苯胺的红外光谱如图3所示。

由图3可知,3 401 cm^{-1} 为 N—H 吸收峰,说明将聚苯胺掺杂到褐煤中得到的煤基聚苯胺中掺杂了胺基的作用^[9]; 1 650 cm^{-1} 为 C=O 吸收峰;对比原煤粉和煤基聚苯胺,1 432 cm^{-1} 为 C—H 键的伸缩振动^[14]; 1 283 cm^{-1} 为苯环中的 C—H 吸收峰;1 095 cm^{-1} 为 C—OH 的吸收峰;这些吸收峰的出现表明聚苯胺已经被掺杂到褐煤中。

2.2 煤基聚苯胺吸附剂对苯酚溶液的吸附作用

2.2.1 吸附动力学

不同时间原煤粉和煤基聚苯胺吸附剂对苯酚

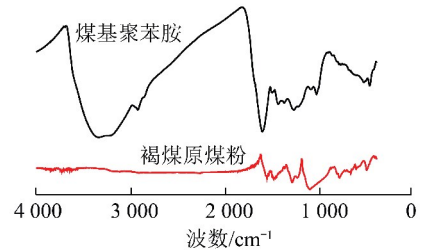


图3 褐煤原煤粉和煤基聚苯胺的红外光谱

Fig. 3 FT-IR of lignite coal power and coal-based polyaniline

溶液的吸附效果如图4所示。由图4可以看出,原煤粉和煤基聚苯胺吸附剂的吸附量均随时间的增加而增加,80 min后趋于稳定并逐渐达到吸附平衡,且煤基聚苯胺吸附剂对苯酚溶液的吸附效果比原煤粉的吸附效果好,吸附量分别为27.49、13.60 mg/g 。

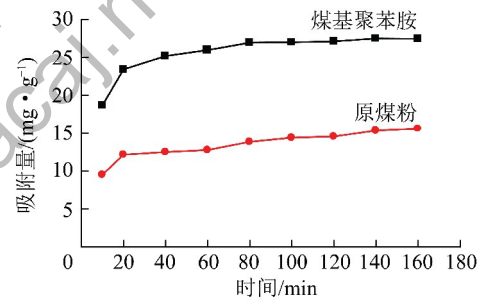


图4 不同时间原煤粉与煤基聚苯胺吸附苯酚的效果

Fig. 4 Adsorption amount of phenol on raw coal power and coal-based polyaniline in different time

常用的吸附速率方程有以下2种:

Lagergren 一级吸附动力学方程式^[15]为

$$\lg(q_e - q_t) = \lg q_e - \frac{k_1}{2.303} t \quad (1)$$

二级吸附动力学方程式^[16]为

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

式中, q_e 为平衡吸附量, mg/g ; q_t 为 t 时刻的吸附量, mg/g ; k_1 为一级吸附速率常数, L/min ; k_2 为二级吸附速率常数, $\text{g}/(\text{mg} \cdot \text{min})$ 。

将图4所得的试验数据用 Lagergren 一级吸附速率方程和二级吸附速率方程进行拟合,拟合结果见表2。由表2可知,原煤粉和煤基聚苯胺吸附剂对苯酚吸附行为更符合二级动力学模型,具体结果如图5所示。

表2 原煤粉与煤基聚苯胺吸附苯酚的动力学模型参数

Table 2 Kinetic model parameters for adsorption of phenol on raw coal power and coal-based polyaniline

吸附剂	$q_e/$ ($\text{mg} \cdot \text{g}^{-1}$)	Lagergren 一级动力学模型			二级动力学模型		
		k_1/min^{-1}	$q_e/(\text{mg} \cdot \text{g}^{-1})$	R^2	$k_2/(\text{g} \cdot (\text{mg} \cdot \text{min})^{-1})$	$q_e/(\text{mg} \cdot \text{g}^{-1})$	R^2
原煤粉	13.60	0.087 3	7.45	0.873	0.061 6	15.26	0.995
煤基聚苯胺	27.49	0.013 4	13.82	0.965	0.035 3	30.96	0.999

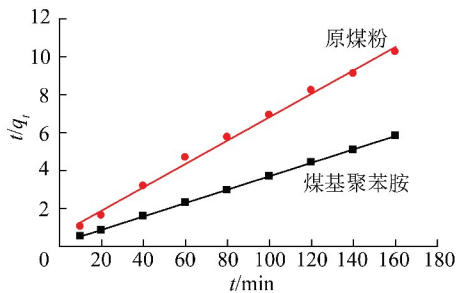


图5 原煤粉与煤基聚苯胺吸附苯酚的二级动力学曲线

Fig. 5 Pseudo second-order curves for adsorption of phenol on raw coal power and coal-based polyaniline

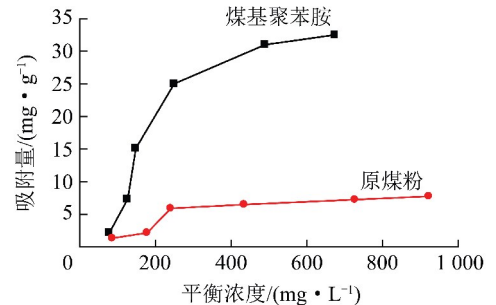


图6 原煤粉与煤基聚苯胺吸附苯酚溶液的吸附等温线

Fig. 6 Adsorption isotherms of phenol on raw coal power and coal-based polyaniline

2.2.2 吸附等温线

褐煤原煤粉和煤基聚苯胺吸附剂对苯酚溶液的吸附等温线如图6所示。由图6可知,煤基聚苯胺吸附剂的吸附效果远好于原煤粉,且煤基聚苯胺和原煤粉的最大吸附量为分别为32.52、7.74 mg/g。

Langmuir 吸附等温式^[17]为

$$\frac{1}{q_e} = \frac{1}{Q^0} + \frac{1}{bQ^0 C_e} \quad (3)$$

Freundlich 吸附等温式^[18]为

$$\lg q_e = \lg K_F + \frac{1}{n} \lg C_e \quad (4)$$

式中, Q^0 为单分子层吸附时单位质量吸附剂的饱和吸附量,mg/g; C_e 为平衡时溶液中剩余吸附质的量,mg/L; b 、 K_F 为常数; n 为与温度等有关的常数。

对试验所得数据运用 Langmuir 吸附等温式和 Freundlich 吸附等温式进行拟合,拟合结果见表3和图7。由表3可知,原煤粉、煤基聚苯胺吸附剂对苯酚的吸附等温线更符合 Freundlich 吸附等温式。

表3 原煤粉与煤基聚苯胺吸附苯酚的等温式参数

Table 3 Langmuir and Freundlich isotherm constants for phenol adsorption on raw coal power and coal-based polyaniline

吸附剂	Langmuir 等温线参数			Freundlich 等温线参数		
	$Q^0/(\text{mg} \cdot \text{g}^{-1})$	$b/(\text{L} \cdot \text{mg}^{-1})$	R^2	K_F	n	R^2
原煤粉	7.74	0.001 09	0.985	0.060 0	0.635	0.993
煤基聚苯胺	32.52	0.002 99	0.972	0.001 8	1.245	0.990

2.2.3 pH 值对吸附效果的影响

不同 pH 值下褐煤原煤粉和煤基聚苯胺对苯酚溶液的吸附效果如图8所示。由图8可知,2种吸附剂对苯酚溶液的去除率随 pH 值的增大先增大后减小。pH=4 时,褐煤原煤粉和煤基聚苯胺最大去除率分别为 22.74% 和 45.32%;因此在酸性条件下,2种吸附剂对苯酚溶液的吸附效果较好;随碱性的增强,吸附性能变差,由于苯酚溶液会发生水解易与 OH^- 结合,并与吸附剂生成吸附物,稳定性降低,

因此吸附量减小。

2.2.4 投加量对吸附效果的影响

不同投加量下褐煤原煤粉和煤基聚苯胺对苯酚溶液的吸附效果如图9所示。由图9可知,相同投加量时,煤基聚苯胺对苯酚的去除效果较好;随着吸附剂投加量的增加,2种吸附剂对苯酚溶液的去除率随之增加,但吸附剂对苯酚溶液的单位吸附量减小,且原煤粉作为吸附剂时吸附量的减小速度较快。这是因为随着原煤粉和煤基聚苯胺投加量的增加,

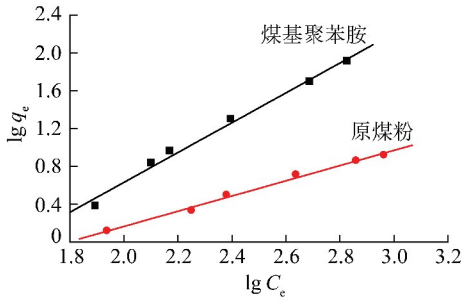


图7 原煤粉与煤基聚苯胺吸附苯酚的 Freundlich 吸附等温线

Fig. 7 Freundlich isotherms for phenol on raw coal power and coal-based polyaniline

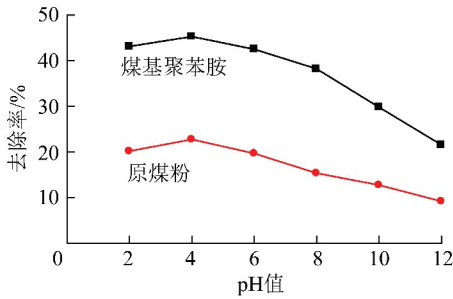


图8 不同 pH 值下原煤粉与煤基聚苯胺吸附苯酚的效果

Fig. 8 Adsorption amount of phenol on raw coal power and coal-based polyaniline in different pH values

其提供的活性基团增加,与有机离子结合的机会也增加,但一定浓度下的溶液离子固定不变,因此其互相争夺有机离子的几率增加,单位吸附量反而减小,因此煤基聚苯胺吸附剂的最佳投加量为 1 g,此时去除率和饱和吸附量分别为 45.32% 和 32.52 mg/g。

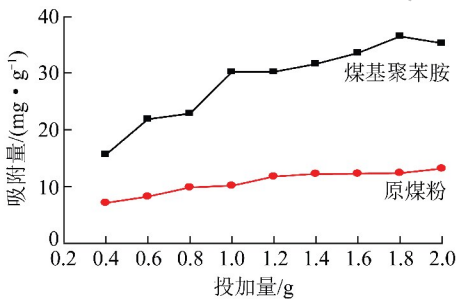


图9 不同投加量下原煤粉与煤基聚苯胺吸附苯酚的效果

Fig. 9 Adsorption amount of phenol on raw coal power and coal-based polyaniline in different dosages

3 结 论

1) 煤基聚苯胺吸附剂孔结构和分布比原煤粉好,具有较多中孔和微孔结构,利于增大吸附容量。

2) 煤基聚苯胺吸附剂对苯酚模拟废水的吸附

效果明显高于原煤粉。在 80 min 时达到吸附平衡,煤基聚苯胺和原煤粉吸附量分别为 27.49、13.60 mg/g。煤基聚苯胺最佳投加量为 1 g/L, pH=4 时,煤基聚苯胺对苯酚溶液的吸附效果较好,去除率为 45.32%。

3) 煤基聚苯胺吸附剂对苯酚模拟废水的吸附符合 Freundlich 等温线方程,其单层饱和吸附容量 32.52 mg/g。吸附动力学符合 Lagergren 二级吸附动力学模型。

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