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活性焦对水中汞的吸附性能

李志超^{1, 2, 3} 兰华春¹ 武利园¹ 刘会娟^{1*} 曲久辉¹

(1. 中国科学院生态环境研究中心, 北京 100085; 2. 中国科学院大学, 北京 100049;
3. 审计署驻广州特派员办事处, 广州 510623)

摘要 采用活性焦作为吸附剂, 通过静态吸附实验, 研究了活性焦对水中汞的吸附特性, 并初步探讨了其吸附机理。活性焦对汞的吸附可用拟二级动力学模型描述; 在 pH 为 5 时能达到对 Hg(Ⅱ)的最大吸附容量, 在不同离子强度下均能保证对 Hg(Ⅱ)有较高的去除率; 据 Langmuir 吸附等温线模型计算出活性焦对 Hg(Ⅱ)的饱和吸附容量可达 412.1 mg/g。结合红外光谱、Zeta 电位测试的结果, 可推测活性焦对 Hg 的吸附过程是物理吸附和化学吸附综合作用的结果。活性焦是一种成本低、效果显著且稳定的吸附剂, 有望在含汞废水处理中发挥重要作用。

关键词 活性焦 吸附 废水 Hg FTIR Zeta 电位

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Adsorptive behaviors of activated coke towards aquatic mercury

Li Zhichao^{1,2,3} Lan Huachun¹ Wu Liyuan¹ Liu Huijuan¹ Qu Juhui¹

(1. Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, Beijing 100085, China;
2. University of Chinese Academy of Sciences, Beijing 100049, China;
3. CNAO's Guangzhou Resident Office, Guangzhou 510623, China)

Abstract Activated coke was facilitated as an adsorbent for the mercury wastewater and characterized via static adsorption experiments. The adsorption process was found to be best described by pseudo-second-order model. The maximum adsorption capacities occurred at pH 5, and high removal can be ensured despite variation of ionic strength. The Langmuir maximum adsorption capacity for Hg(Ⅱ) was 412.1 mg/g. It can be drawn from the FTIR, Zeta-potentials analysis that the adsorption of Hg(Ⅱ) on activated coke result from both physical and chemical effects. And activated coke is thus inferred to be an economic, effective and stable adsorbent, which would favor the treatment of mercury effluent.

Key words activated coke; adsorption; wastewater; Hg removal; FTIR; Zeta-potentials

人们普遍认为, 汞是毒性最大的重金属之一^[1,2], 研究表明, 汞可对中枢神经系统、肺、肾和染色体造成永久性伤害^[3]。汞的来源可分为天然源和人工源, 其中人工源主要来自氯代烷烃、纸浆、化肥等工业的废弃物^[4]。而在众多除汞方法中, 吸附法兼具适用性和经济性^[5-8]。活性炭因为具有很高的比表面积^[5,6], 经常用作吸附剂处理水中的重金属^[5,9-12]。目前活性炭已有很成熟的商业化应用, 但是还是存在着价格过高的问题^[13,14], 因此寻求一种来源更广和性能更好的吸附剂就显得尤为必要。

活性焦是以褐煤为主要原料研制出的一种外观为暗黑色的多孔含碳物质, 是没有得到充分干馏或活化的活性炭类吸附剂。与活性炭相比, 活性焦的机械强度更高, 更耐受循环和装卸过程^[15,16], 而且其成本与活性炭相比大幅度降低, 因此引起了研究

人员的广泛兴趣。活性焦的比表面积要低于活性炭, 其孔结构主要是由大孔和介孔组成的, 微孔的比例相对少一些, 更有利于溶液相在吸附剂内部的迁移和扩散^[17,18]。活性焦对较多污染物具有很好的吸附效果, 已经广泛应用于工业废气处理中^[15,16,19-22], 在污水处理中的应用也逐渐引起人们的重视^[18,23-25]。本文旨在研究活性焦吸附去除废水中的汞的效果和机理, 以期为水中汞的去除提供技术参考。

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作者简介: 李志超(1987~), 男, 硕士研究生, 主要从事吸附水处理技术研究工作。E-mail: lizhichao210@163.com

* 通讯联系人, E-mail: hjiu@rcees.ac.cn

1 材料与方法

1.1 实验材料

试剂:活性焦从国家电力科学院获得,活性焦经100目筛分,并于0.01 mol/L HNO₃中振荡72 h后用去离子水洗至pH中性,80℃下烘干15 h,于干燥器中保存备用。

Hg(NO₃)₂为分析纯,购自姜堰环球试剂厂;HNO₃、HCl、H₂SO₄、KOH为优级纯,KNO₃、KCl、KBH₄、KBr、KBrO₃、盐酸羟胺、为分析纯,购自国药集团(Sinopharm Chemical Reagent Co., Ltd.)。测定Hg(Ⅱ)所采用的标准溶液购自国家标准中心(National Research Center for Certified Reference Materials, China)。实验过程所用水均为超纯水(18.2 MΩ cm),用Milli-Q净化设备制得。

Hg(Ⅱ)的储备液(100 mg/L)是通过将定量的Hg(NO₃)₂溶于超纯水中制取,于避光处保存,于使用前进行稀释,即为实验中所用含汞溶液。冷原子荧光法测汞所用氧化剂为KBrO₃-KBr溶液,取2.784 g KBrO₃和10 g KBr溶于1 L超纯水而得;还原剂为KBH₄-KOH溶液,取0.1 g KBH₄溶于1 L 0.2% KOH溶液而得;过量的氧化剂用数滴100 g/L盐酸羟胺去除。

1.2 实验仪器

溶液pH用pH计(Orion 3 Star pH benchtop, Thermo Scientific)测定。Hg(Ⅱ)的浓度采用冷原子荧光法测定,原子荧光光谱仪型号为AF-610B(Beijing Rayleigh Analytical Instrument Co., China)。

使用ASAP 2000比表面积分析仪(Micromeritics Co., USA),傅里叶变换红外光谱(Bruker TENSOR 27 FTIR, USA),Zeta电位仪(DelsaNano C, Beckman Coulter Ltd., U. S. A.),透射电子显微镜(H-7500,日本日立公司),元素分析仪(Vario Macro, Germany)对活性焦进行表征。

1.3 吸附实验

吸附动力学实验:准确量取400 mL Hg初始浓度为1 mg/L的Hg(Ⅱ)溶液(离子强度为10 mM KNO₃,pH值为5)于800 mL烧杯中,加入40 mg活性焦。烧杯用PVC膜封住,以防止汞的挥发,用磁力搅拌器以150 r/min的速度搅拌18 h,在预定时间采样后用0.45 μm滤膜过滤,滤液置于塑料离心管中密封好冷冻保存,以待后续检测。

pH和离子强度影响实验:配制Hg浓度为1

mg/L的Hg(NO₃)₂溶液,其离子强度分别为1 mmol/L、10 mmol/L、100 mmol/L或1 mol/L KNO₃。溶液的初始pH用HCl或NaOH调节到3、5或8。称取50 mL溶液于玻璃磨口三角瓶中,添加5 mg吸附剂,将三角瓶加盖封好,置于恒温摇床中25℃下振荡,速度为150 r/min,15 h后取样分析。

吸附等温线实验:配制了一系列Hg初始浓度在0~30 mg/L内的Hg(NO₃)₂溶液(pH 5,离子强度10 mmol/L KNO₃)。各取100 mL溶液,置于玻璃磨口三角瓶中,并投加5 mg吸附剂。其余步骤与pH和离子强度影响实验相同。

所有实验均做平行实验,以确保实验数据的准确性。

2 结果与讨论

2.1 活性焦的性质

碳是构成活性焦最主要元素,作为活性焦的骨架存在^[17]。元素分析测定活性焦的C、N和H 3种元素的质量组分分数分别为34.10%、0.32%和0.54%。由图1和图2可知,活性焦呈现不规则的多孔结构,介孔(2~50 nm)和大孔(>50 nm)结构发达,此外还有少量的微孔结构(0~2 nm)。经BET模型计算得到活性焦的比表面积为193.7 cm⁻¹,平均孔径为2.8 nm,孔容为0.18 cm³/g。在吸附过程中,吸附剂的孔径分布是经常需要考虑的因素^[26]。介孔结构作为通道,更有利于溶液相在吸附剂内部的迁移和扩散,使Hg(Ⅱ)更易进入微孔结构中,进而被吸附^[27],因而在吸附过程扮演了很重要的角色。

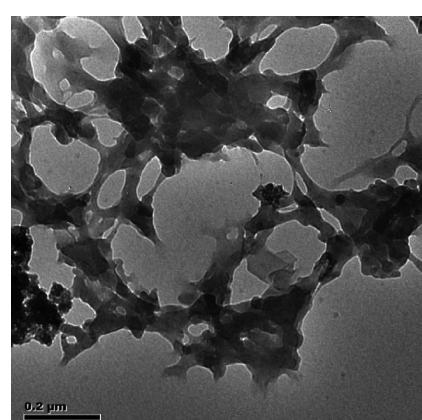


图1 活性焦的TEM图像

Fig. 1 TEM image of activated coke

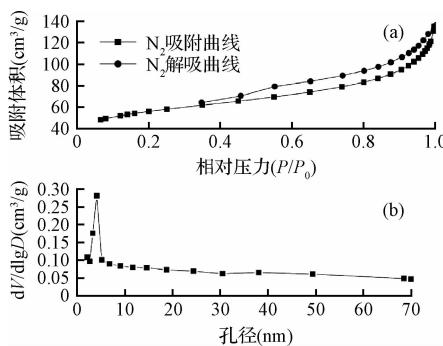


图 2 活性焦的 N_2 吸附/解吸曲线及
BJH 孔径分布曲线

Fig. 2 N_2 adsorption/desorption curve and BJH pore diameter distribution curve of activated coke

2.2 活性焦的吸附性能

2.2.1 吸附动力学

研究吸附动力学可以更深入地了解吸附剂与吸附质反应的路径和机理^[14]。对此进行研究,吸附剂量 0.1 g/L, pH 5, 离子强度 10 mM KNO_3 , 搅拌速度 160 r/min。如图 3 所示,活性焦对于不同初始浓度的 Hg 的吸附过程类似,都可分为 2 个阶段,分别为快速反应阶段和缓慢平衡阶段。在第 1 阶段,活性焦对 Hg 的吸附量快速上升,以 Hg(II) 的初始浓度为 1.0 mg/L 时为例,活性焦在吸附的前 200 min 的吸附量可达到平衡吸附量的 90% 以上。一般认为,这一阶段受控于 Hg(II) 离子由溶液向吸附剂表面活性位点的迁移过程^[28,29],而吸附开始阶段表

面浓度与平均吸附相浓度之间较高的差值则促进了这一迁移过程^[13]。此外活性焦的多孔性结构可为 Hg(II) 进入其表面(或其大孔表面)提供便捷的通道^[17],也会对吸附过程产生有利的影响。第 2 阶段 Hg(II) 的吸附速率较慢,并逐渐趋近平衡。由于吸附性表面及大孔活性位点都已被占据^[9],Hg(II) 的吸附转向孔内及微孔结构的表面。此时, Hg(II) 的吸附过程主要受孔内/表面扩散过程控制^[13]。

采用拟一级、拟二级动力学模型^[14,30]对吸附数据进行拟合,结果如表 1 所示。可以看出,用拟二级动力学模型拟合的相关系数较高,说明活性焦对 Hg(II) 的吸附为二级反应,有化学过程的参与^[14,30]。实验发现,活性焦对 Hg(II) 的吸附均可在 480 min 内达到平衡。在以下实验中为了确保吸附均能达到平衡,设定搅拌时间为 15 h。

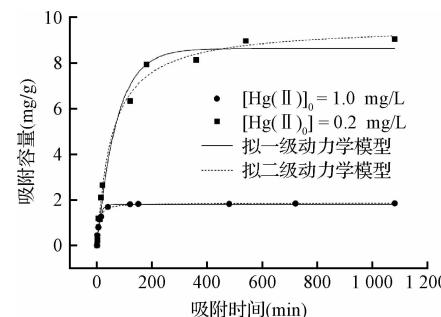


图 3 活性焦吸附 Hg(II) 的动力学曲线

Fig. 3 Hg(II) adsorption kinetics curve by activated coke

表 1 吸附动力学拟合参数

Table 1 Parameters of adsorption kinetics calculated from two models

[Hg(II)] ₀ (mg/L)	拟一级动力学模型 $q_t = q_e (1 - e^{-k_1 t})$			拟二级动力学模型 $q_t = k_2 q_e^2 t / (1 + k_2 q_e t)$		
	q_e (mg/g)	k_1 (min ⁻¹)	R^2	q_e (mg/g)	k_2 (min ⁻¹)	R^2
0.2	1.79	0.101	0.969	1.86	0.092	0.988
1.0	8.62	0.014	0.985	9.64	0.002	0.994

2.2.2 pH 和离子强度的影响

图 4 所示为不同 pH 及离子强度时活性焦对 Hg 吸附的影响。Hg(II) 的初始浓度为 1.0 mg/L, 吸附剂投加量 0.1 g/L, 搅拌速度 160 rpm, 平衡时间 15 h。溶液 pH 分别为 3、5 和 8。所采用离子强度调节剂为 KNO_3 , 离子强度分别为 1 mmol/L、10 mmol/L、100 mmol/L 和 1 mol/L。对于活性焦来说,各个离子强度下吸附容量随 pH 变化的趋势是相似

的。活性焦对 Hg 的吸附容量在 pH 小于 5 时随 pH 升高而上升,最大值出现在 pH 5 处,pH 继续升高,吸附容量反而下降。

图 5 所示为 Hg(II) (1.0 mg/L) 在不同 pH 下的形态分布模拟结果。可以看出,在 pH < 5 时,Hg(II) 的形态主要为 3 种,即 $\text{Hg(OH)}_2(\text{aq})$ 、 Hg^{2+} 和 Hg(OH)^+ 。随着 pH 升高, $\text{Hg(OH)}_2(\text{aq})$ 所占比例逐渐升高,直至 pH > 5 时, $\text{Hg(OH)}_2(\text{aq})$ 成为单一

的主导形态。在低 pH 时, 大量存在的 H^+ 会与带正电的 Hg^{2+} 和 $\text{Hg}(\text{OH})^+$ 在结合位点上产生竞争^[31]。同时 pH 越低, 越有利于活性焦表面的质子化, 也就越削弱和减少带负电的位点^[29]。这种情况下, 活性焦和带正电形态的 $\text{Hg}(\text{II})$ 之间会产生静电斥力, 阻止它们靠近吸附剂和溶液之间的界面^[31]。溶液 pH 升高, 会增强静电引力, 减弱 H^+ 的竞争, 因此吸附容量上升。当 pH 超过 5 后, Hg 的水解作用成为主导, 大量水解产物覆盖于活性焦表面, 会对活性焦的吸附性能产生不利影响^[32], 使得吸附容量下降。

在 pH 3~5 范围内, 在 $\text{Hg}(\text{II})$ 形态中占主导的为 $\text{Hg}(\text{OH})_2(\text{aq})$, Hg^{2+} 和 $\text{Hg}(\text{OH})^+$, 与活性焦的作用为内层配合^[33,34]。这些离子覆盖在吸附剂表面, 会带来大量正电荷。而离子强度增加的话, 会对这些正电荷有一定保护作用^[34], 从而会提高吸附容量。而 pH 5~8 范围内, $\text{Hg}(\text{II})$ 的水解产物占主导, 与活性焦的作用为外层配合。离子强度增加, 会削弱外层配合的静电力, 使吸附容量降低。

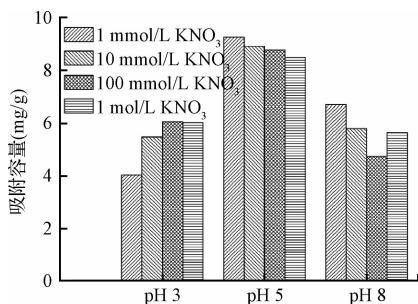


图 4 不同 pH 及离子强度对活性焦吸附 $\text{Hg}(\text{II})$ 的影响

Fig. 4 Influence of pH and ionic strength on $\text{Hg}(\text{II})$ adsorption by activated coke

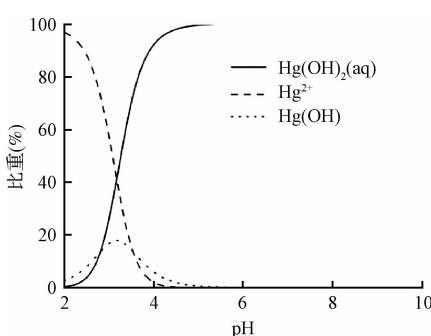


图 5 $\text{Hg}(\text{II})$ 在不同溶液 pH 下形态分布曲线

Fig. 5 $\text{Hg}(\text{II})$ speciation as a function of solution pH

2.2.3 吸附等温线

图 6 所示为活性焦对 Hg 的吸附等温线。所述

实验条件为 pH 5, 温度 25°C, 离子强度为 10 mM KNO_3 , 吸附剂投加量 0.05 g/L, 搅拌速度 160 r/min, 平衡时间 15 h。

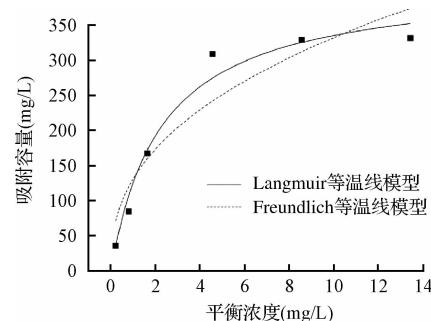


图 6 活性焦对 $\text{Hg}(\text{II})$ 的吸附等温线

Fig. 6 $\text{Hg}(\text{II})$ adsorption isotherm by activated coke

分别用 Langmuir 和 Freundlich 等温线模型^[27,35]来拟合实验数据, 得到的参数列于表 2。可以看出, Langmuir 等温线模型可以较好地拟合活性焦对 Hg 的吸附, 其最大吸附容量是 412.1 mg/g。而 Jenny 等利用石油裂解副产物制得的活性炭对 Hg 的吸附容量可达 112 mg/g^[36], Stenphen 和 Kadirlvelu 利用生物质制得的活性炭对 Hg 的饱和吸附容量分别为 94.4 mg/g 和 55.6 mg/g^[37,38], Gomez 等^[10]利用 SO_2 和 H_2S 在高温下对活性炭进行改性后, 对 Hg 的吸附容量达到 345.8 mg/g。本研究所用活性焦对 Hg 的吸附容量活性焦可与活性炭相媲美, 加之其大大降低的成本^[17], 为实际应用在含 Hg 废水的工艺中提供了广阔的前景。

表 2 活性焦吸附等温线拟合参数

Table 2 Parameters of adsorption isotherm calculated from two models

Langmuir model			Freundlich model		
$Q = Q_{\max}LC/(1 + LC)$	$Q = FC^{1/n}$		F	N	R^2
Q_{\max} (mg/g)	L (L/mg)	R^2			
412.1	0.44	0.969	12.2	1.4	0.868

2.3 吸附机理初探

为研究活性焦吸附 $\text{Hg}(\text{II})$ 的机理, 利用红外光谱研究了吸附 $\text{Hg}(\text{II})$ 前后表面的官能团的变化, 结果如图 7 所示。活性焦的谱图中, 最显著的是 $3550 \sim 3250 \text{ cm}^{-1}$ 和 $1260 \sim 1000 \text{ cm}^{-1}$ 的 2 处吸收谱带, 分别代表醇类—OH、C—O 的伸缩振动^[39]。饱和烷烃的 C—H 的对称与不对称伸缩振动在

3 000~2 800 cm⁻¹有强烈吸收,其对称弯曲振动也在1 383 cm⁻¹处有一单一吸收峰^[40,41]。通过对比活性焦在吸附Hg(Ⅱ)后的谱图,发现活性焦表面C—H的吸收(3 000~2 800 cm⁻¹, 1 383 cm⁻¹)在吸附Hg后变得更加尖锐,之前的文献中也有类似报道^[40]。这说明C—H可能作为静电吸附位点与Hg(Ⅱ)结合,从而将之吸附在活性焦表面。而众所周知的是,活性焦是一种由煤或其他生物质炼制而来的碳基材料,所以其中广泛存在的C—H可在Hg(Ⅱ)的吸附过程中发挥重要作用。

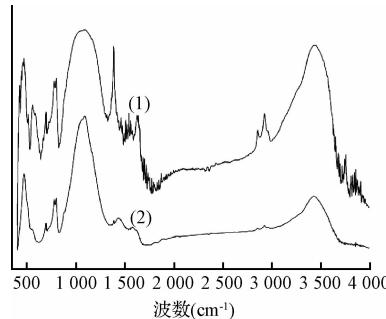


图7 活性焦对Hg(Ⅱ)吸附前(1)和吸附后(2)的红外光谱图像

Fig. 7 FTIR spectrum of activated coke before (1) and after (2) Hg(Ⅱ) adsorption

用Zeta电位法分析吸附剂在吸附Hg(Ⅱ)前后的表面电特性随pH变化的情况,所用空白溶液为超纯水,Hg(Ⅱ)溶液浓度为1.0 mg/L,吸附剂投加量为0.1 g/L,结果如图8所示。活性焦在低pH下表现出正电位,而当pH升高到一定程度后则转为负电位。活性焦的等电点在pH 5左右,与文献报道活性炭的等电点接近^[42]。这与活性焦与活性炭均为热裂解后的碳基产品^[5,17]不无关系。这类碳基产品表面含有丰富的官能团和π键,从而具有酸碱两性^[42]。吸附剂的Zeta电位会随pH增长会显著降低,说明固液界面吸附羟基是造成界面负电性的主要原因^[43]。对Hg(Ⅱ)的吸附会使吸附剂的Zeta电位升高,是因为吸附剂表面吸附了Hg(Ⅱ)之后正电荷积累的原因。同时,还发现在弱酸性至弱碱性的pH范围内发生了电性的改变。在此pH范围内,吸附剂表面接近中性,而此时溶液中的Hg(Ⅱ)趋近固液界面是没有静电作用力支持的^[43]。也就是说,还有Hg(Ⅱ)与吸附剂表面的专性吸附参与其中。化学吸附的作用使得吸附剂表面不仅能发生电性中和,还能继续积累Hg(Ⅱ)进而发生电性改

变^[44]。这说明活性焦对于Hg(Ⅱ)的吸附过程不仅仅是简单的静电吸附作用,还包含专性的化学吸附过程。这与活性焦本身含有的羟基等基团有关。

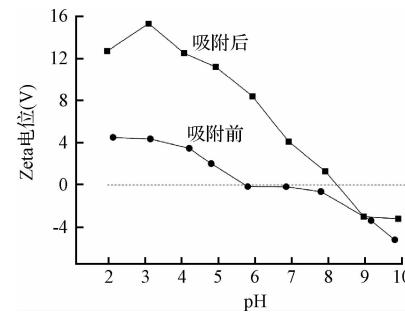


图8 吸附Hg(Ⅱ)前后的活性焦在不同溶液pH下的Zeta电位变化曲线

Fig. 8 Zeta-potentials of activated coke before and after Hg(Ⅱ) adsorption as a function of solution pH

3 结 论

(1)活性焦具有以介孔为主的多孔性结构,表面存在多种基团,有利于对Hg(Ⅱ)的吸附。

(2)活性焦对于Hg(Ⅱ)的吸附过程可在480 min内达到平衡,较符合拟二级动力学模型,为二级反应。

(3)活性焦对于Hg(Ⅱ)的吸附最优pH在5左右,对于Hg(Ⅱ)的饱和吸附容量可达412.1 mg/g,在含碳类吸附剂中具有很大优势。

(4)活性焦作为一种水处理吸附剂,因其较大的吸附容量、稳定的去除效果、较高的机械强度和低廉的成本,有望在含汞废水等一系列重金属废水的处理中发挥重要作用。

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