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论文

原子层沉积法制备巯丙基硅胶及其对溶液中钯(Ⅱ)离子的吸附行为

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摘要:

采用原子层沉积技术, 在3~5 kPa真空和125~150 ℃ 的反应条件下, 使γ-巯丙基三甲氧基硅烷(MPTMS)、γ-巯丙基三乙氧基硅烷(MPTES)和γ-巯丙基二甲氧基甲基硅烷(MPDMMS)3种巯丙基硅烷试剂气化, 并在三乙胺的催化作用下, 分别将其键合于多孔硅胶表面, 制得贵金属钯(Ⅱ)的高效吸附剂. 分别采用FTIR、^{13}C和^{29}Si固体核磁、元素分析、热重分析和氮气吸附-脱附等技术研究了巯基硅胶的键合模式和功能基团键合量. 用分光光度法研究了在pH=3.0条件下水溶液中Pd(Ⅱ)离子在巯基硅胶上的吸附行为. 结果表明, 在MPTMS, MPTES和MPDMMS所修饰的硅胶中, 硅烷试剂的功能基团均以双齿键合结构为主, 表面键合量分别达到2.76, 2.53和2.70 μmol/m^2. 对Pd(Ⅱ)离子的吸附遵从Langmuir等温吸附方程, 饱和吸附量分别达到5.45, 4.21和4.81 μmol/m^2, Pd/S的摩尔比分别为1.44, 1.35和1.39. 原子层沉积法制备的巯丙基硅胶基质钯吸附剂的巯基键合密度和对钯(Ⅱ)离子的吸附容量均比传统的有机溶剂介质法高.

关键词: 原子层沉积; 巯丙基烷氧基硅烷; 多孔硅胶; 钯; 吸附剂

Preparation of Mercaptopropyl Bonded Silica by Atomic Layer Deposition and Its Adsorption of Palladium(Ⅱ) Ions

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Abstract:

Atomic layer deposition was employed for preparation of thiol-functionalized silica as efficient adsorbent of palladium(Ⅱ) ions. Under reaction conditions of 3—5 kPa and 125—150 ℃, trifunctional γ-mercaptopropyltrimethoxysilane(MPTMS), γ-merecaptopropyltriethoxysilane(MPTES) and bifunctional γ-mercaptopropyldimethoxymethylsilane(MPDMMS) were vaporized in an autoclave, and chemically bonded to po-rous silica, respectively. The bonding mode, morphology and thiol group content of the thiol functional silica were characterized by FTIR, solid state ^{13}C and ^{29}Si nuclear magnetic resonance, elemental analysis, TGA, and nitrogen adsorption-desorption analysis. The behavior of the adsorption of Pd(Ⅱ) ions from aqueous solutions at pH=3.0 onto thiol functionalized silica samples was studied by measuring the changes in concentration of Pd(Ⅱ) solution in contact with the silica using spectrophotometry. The results show that bidentate bonding mode is preferred for all three silane reagents and the thiol contents are determined to be 0.84, 0.77 and 0.82 mmol/g, respectively, for MPTMS, MPTES and MPDMMS modified silica. The adsorption of Pd(Ⅱ) ions on MPTMS, MPTES and MPDMMS modified silica could be described by the Langmuir equation with a saturation adsorption capacity of 1.21, 1.04 and 1.14 mmol/g, respectively. The molar ratios of Pd/S were 1.44, 1.35, and 1.39. Thus, both thiol content and adsorption capacity of the materials prepared by atomic layer deposition are higher than those of the bonded silica made by reaction in organic solvent as generally practiced. The present work has demonstrated that atomic layer deposition, characterized by short reaction time, no consumption of organic solvents and high functional group density, is a cost-effective and eco-friendly approach for preparation of silica based palladium scarvengers.

Keywords: Atomic layer deposition; Mercaptopropyl alkoxysilane; Porous silica; Palladium; Absorbent

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