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研究论文

可用于色谱固定相的介孔氧化硅球材料的合成

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摘要 采用非离子型嵌段高分子表面活性剂$EO_{20}PO_{30}EO_{20}$ (P65)为结构导向剂, 正硅酸乙酯为硅源, 在酸性介质中, 静置法制备了微米级介孔氧化硅球. 通过改变合成温度、反应时间或者无机盐KCl的加入量, 可以调节介孔氧化硅球的直径(9.0～17.6 μm); 加入1,3,5-三甲苯(TMB)或者调节水热温度, 可以调节介孔氧化硅球的孔径(2.3～4.8 nm). 采用X射线衍射(XRD)、N_2吸附-脱附、扫描电镜(SEM)、激光散射粒度分布和对溶菌酶的吸附等方法, 对介孔氧化硅球的结构、孔性质、形貌、吸附性质等进行了表征. 实验发现, 孔径较小的介孔氧化硅球(≤4.3 nm)对溶菌酶的吸附不明显(≤42 mg/g), 而孔径(4.8 nm)大于溶菌酶直径的材料对溶菌酶有较大的吸附量(192 mg/g), 说明孔径均匀可调的介孔氧化硅球材料可以很好地用作体积排阻色谱柱的固定相.

关键词 介孔氧化硅 形貌控制 嵌段共聚物 合成 溶菌酶

分类号

Synthesis of Ordered Mesoporous Silica Spheres with Controllable Diameter in Micrometer

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Abstract A new procedure for the synthesis of mesoporous silica spheres with controlled particle size (9.0～17.6 μm) and pore size (2.3～4.8 nm) was developed. It was based on the hydrothermal syntheses by mixing nonionic surfactant $EO_{20}PO_{30}EO_{20}$ (P65) and TEOS under static acidic conditions. The size of spheres was controlled by adjusting the synthesis temperature, the synthesis time, and the amount of added KCl. The pore size of the spheres was controlled by adding the 1,3,5-trimethylbenzene (TMB) or changing the hydrothermal temperature and characterized by nitrogen adsorption-desorption analysis, whereas the size of the spheres was analyzed by scanning electron microscopy (SEM) and further confirmed by the laser scattering particle size distribution analyzer. The bio-immobilization ability of such mesoporous silica spheres was characterized by protein adsorption analysis. Mesoporous silica spheres with the largest pore size (4.8 nm) among all materials under investigation showed the highest specific immobilization amount of lysozyme (192 mg/g), while the spheres with smaller pore size (≤4.3 nm) have markedly low immobilization amount (≤42 mg/g). These mesoporous silica spheres with diameter ～10 μm in size may be readily used as a good substrate in high performance liquid chromatography (HPLC).

Key words mesoporous silica morphology control block copolymer synthesis lysozyme

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