Preparation and evaluation of silica xerogel monolithic column

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Abstract Using potassium silicate as silicon source, formamide as catalyst, a series of silica xerogel monolithic columns with different consistencies were prepared. The column bed would not rupture and collapse during drying at high temperatures. This is the biggest advantage compared with the inorganic monolithic columns using alkoxy silane as precursor. The effect of the modulus of potassium silicate on the physical structure of the monolithic column was investigated. The monolithic silica columns were characterized by scanning electron micrograph (SEM) and nitrogen adsorption. The relationship between column pressure and flow rate was evaluated. The column efficiency for anthracene was tested. The breakthrough curve for toluene was studied. The results showed that the column bed could maintain good stability at high temperatures, high column pressures and high flow rates. The column efficiency of 41 400 plates/m was achieved for anthracene. The column capacity for toluene was 61 ng.

Key words inorganic monolithic column, xerogel, stability, potassium silicate, formamide

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1

1.1


Table 1  BET surface areas and BJH adsorption average pore diameters of silica xerogel monolithic columns with different moduli.

<table>
<thead>
<tr>
<th>Modulus</th>
<th>BET surface area $m^2/g$</th>
<th>BJH adsorption average pore diameter/nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.8</td>
<td>49.55</td>
<td>7.09</td>
</tr>
<tr>
<td>3.0</td>
<td>65.28</td>
<td>5.24</td>
</tr>
<tr>
<td>3.5</td>
<td>93.35</td>
<td>3.86</td>
</tr>
</tbody>
</table>

2

2.1

2.1.1

2.1.2

2.1.3
Fig. 1  Scanning electron micrographs (SEM) of silica xerogel monolithic columns of different moduli

Fig. 2  a) SEM and b) microscope images of column calcined at 300 °C

2.1.4 3.0 cm  10 cm

2.2  C₁₈  3.0 µL/min

2.3  C₁₈  2 mg/L

2.4  5 µL/min  10 cm  10 µL

2.5  5 µL/min  100 µL

2.6  5 µL/min  110 µL

2.7  10 µL/min  10 µL

2.8  10 µL/min  40 µL

2.9  10 µL/min  100 µL

2.10  GC-MS  3 min

2.11  GC-MS  3 min

2.12  GC-MS  3 min
严逢川,等:干凝胶无机整体柱的制备及评价

图 1 整体柱的柱压与流速之间的关系

图 2 整体柱对硫脲和蒽的分离谱图

接口温度: 
扫描方式: 
质量范围: 
扫描速度: 
根据图 1 和表 5 中的结果进行计算,得出该整体柱对甲苯的吸附量为 57.0 μg。

结论
本文对干凝胶法制备无机整体柱的条件进行了优化,在不同模数下得到了一系列致密度不同的无机整体柱;其最大的优点在于制备过程中不受高温的影响,即使在高温的条件下,色谱柱依然具有很强的稳定性。相对于以烷氧基硅烷为前驱体采用溶胶凝胶法制备整体柱的方法,本方法具有制备工艺简单、不需要对毛细管壁进行预处理等特点。不同模数整体柱的平均孔径大小不同,可以适应不同分析研究的需要,其在微柱分离分析中将有一定的应用前景。

表 2 甲苯在 C_{18} 整体柱上的突破曲线

<table>
<thead>
<tr>
<th>Elution volume/μL</th>
<th>Mass concentration of toluene in eluent/μg/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>6130</td>
</tr>
<tr>
<td>20</td>
<td>15.4</td>
</tr>
<tr>
<td>30</td>
<td>9.8</td>
</tr>
<tr>
<td>40</td>
<td>3.72</td>
</tr>
</tbody>
</table>

Table 2 Elution of toluene adsorbed on a C_{18} monolithic column by methanol

3 1. Ye F G; Han Y Y; Zhao S L. Journal of Instrumental Analysis 2008; 27(6): 670
3. Wang Y; Li F; Yin; Zhao T B et al. Petroleum Processing and Petrochemicals 2006; 37(5): 53

Fig. 5 Breakthrough curve of toluene on a C_{18} monolithic column

C_{18} 61 ng/L