

论文

反应性梯形聚氢基倍半硅氧烷合成方法的改进

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摘要 对反应性梯形聚氢基倍半硅氧烷 (H-T) 的合成方法进行了改进. 首先利用硅羟基与硅氯基之间的脱氯化氢缩聚来代替以前采用的硅羟基脱水缩聚反应, 进一步提高了作为梯撑的对苯二胺之间氢键在聚合反应中的模板作用. 另外利用三甲基氯硅烷与对苯二胺梯撑的聚硅氧烷中间体的末端硅羟基进行封端反应, 从而保证在脱除对苯二胺梯撑模板分子过程中避免进一步的无规缩合导致支化或交联, 得到的反应性梯形聚氢基倍半硅氧烷 (H-T) 的规整性有所改善. 热分析结果表明与单链聚二甲硅氧烷的 T_g (-123°C) 相比, 其 T_g 高达 117.0°C , 证明这种梯形高分子具有刚性链结构. 尤其是 ^{29}Si -NMR 谱中代表梯形主链上硅原子 ($\text{SiO}_{3/2}$) 峰的基线宽度 $\Delta=5$, 而采用硅羟基间脱水缩合方法得到聚合物的 $\Delta=8\sim 10$. 表明该反应性梯形聚氢基倍半硅氧烷 H-T 的规整性得到了明显的提高. 而且, 该聚合物又是第一个可溶性、反应性、纯梯形主链无机高分子, 它可以进一步通过硅-氢侧基接枝反应制备不同类型的梯形无机主链功能高分子.

关键词 [梯形聚有机硅倍半硅氧烷](#) [梯形聚氢基倍半硅氧烷](#) [自组装](#) [逐步偶联聚合](#)

分类号

AN IMPROVEMENT IN THE PREPARATION OF THE REACTIVE LADDERLIKE POLYHYDROSILSESQUIOXANE

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Abstract An ordered reactive ladderlike polyhydrosilsesquioxane (H-T) was successfully synthesized via a modified "stepwise coupling polymerization" process, which included two improvements in synthetic method. First, dehydrochlorination polycondensation between Si—OH and Si—Cl groups during polycondensation reaction is employed instead of the dehydration between Si—OH groups used before, which improves the H-bonding template effect of the bridged-phenylene diamine. Meanwhile, trimethylchlorosilane is used to block the terminal silanol groups, avoiding the further intermolecular crosslinking of the phenylene diamine-bridged ladderlike macromolecular intermediates. Consequently, the regularity of H-T has been improved, and compared with a very low T_g (-123°C) of the single-chained polydimethylsiloxane, a significantly higher T_g (117.0°C) of H-T in DSC measurement confirms that the ordered reactive H-T macromolecule possesses comparatively stiff main chain. Above all, the peak of $\text{HSiO}_{3/2}$ (-84.8) with the baseline width (Δ) of about 5 in ^{29}Si -NMR spectrum which is much narrower than that of the polysilsesquioxane ($\Delta=8\sim 10$) prepared by the usual dehydration condensation method shows that the regularity of H-T is obviously improved. Furthermore, H-T is the first soluble, reactive and purely inorganic ladderlike macromolecule, which can be grafted through hydrosilylation reaction and others to generate functional LPSs with different side groups.

Key words [Ladder-like polyorganosilsesquioxane \(LPS\)](#) [Polyhydrosilsesquioxane](#) [Self-assembly](#) [Stepwise coupling polymerization \(SCP\)](#)

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