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研究论文

四配位硅单体及其共聚物的制备和结构表征

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摘要 研究了直接从无定形二氧化硅出发, 与乙二醇、氢氧化钾反应, 生成高反应活性的五配位硅钾化合物, 并以此为原料与含活泼氯的3-氯丙烯反应制备出含双键官能团的四配位硅单体. 讨论了合成单体的条件如温度、反应时间、反应物浓度、溶液pH值及溶剂等因素的影响. 然后以该四配位硅单体与甲基丙烯酸甲酯(MMA)在偶氮二异丁腈(AIBN)作引发剂下进行自由基聚合得到支链含硅共聚物. 并借助于红外光谱(IR)、核磁共振(^{13}C和^{1}H, ^{29}Si)、能谱元素分析对合成的单体进行了结构表征; 用红外光谱(IR)、热失重谱(TG)、差示扫描量热谱(DSC)、凝胶渗透色谱法(GPC)等现代测试手段对支链含硅共聚物进行了结构表征及热性能分析.

IR表明四配位硅单体在1646 cm^{-1}处是C＝C的伸缩振动吸收峰, 在共聚物中此峰消失; TG表明共聚物在249.6 ℃才开始失重, 552 ℃有机部分失重完毕; GPC分析表明共聚物的数均分子量为8.7万.

关键词 配位硅化合物 含硅聚合物 自由基溶液聚合

分类号

Synthesis and Structure Analysis of Tetra-coordinated Sili-con Monomer and Branch Silicon Copolymer

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Abstract In this paper, penta-coordinated silicon compound with high reactivity was prepared by the reaction of silica with ethylene glycol and potassium hydroxide. This penta-coordinated silicon then reacted with allyl chloride to give a tetra-coordinated silicon monomer. The effects of conditions such as temperature, reaction time, reagent concentration, solvent and pH value on reaction were discussed. Then branch silicon copolymer between the tetra-coordinated silicon monomer and methyl methacrylate (MMA) was gained through the free radical solution polymerization using AIBN as an initiator. The structure of monomer was analyzed by the modern measuring methods, including FTIR, NMR (^{13}C, ^{1}H and ^{29}Si), and elemental analysis of energy diffraction spectrum (EDS). The structure and thermal property of branch silicon copolymer were analyzed by IR, TG, DSC and GPC. The IR spectrum of tetra-coordinate silicon monomer indicated an absorption band for C＝C at 1646 cm^{-1}, but the copolymer exhibited no absorption band at 1646 cm^{-1}. TG analysis showed the copolymer began losing weight at 249.6 ℃ and ended at 550 ℃. GPC analysis displayed that the mo-lecular weight of the copolymer was 87000.

Key words coordinated-silicon containing-silicon polymer free radical solution polymerization

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