

材料化学工程与纳米技术

3,3'-二磺化-4,4'-二氟二苯砜二钠盐的合成与表征

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摘要

以工业级4,4'-二氟二苯砜(DFDPS)为原料,利用升华方法进行纯化处理后,采用发烟硫酸直接磺化,通过改变反应物计量比、反应温度、反应时间等参数,系统研究了3,3'-二磺化-4,4'-二氟二苯砜二钠盐(SDFDPS)的制备方法。采用HPLC、UV、¹H NMR及FTIR对磺化产物结构及纯度进行了表征,由此得到最佳的磺化条件为:反应物摩尔比(SO₃:DFDPS)为3.0:1,在110℃下反应20 h。在此反应条件下的磺化产物中未发现单磺化产物以及未磺化的原料DFDPS。经过乙醇/水两次重结晶后,总收率达到75%。以合成的SDFDPS为原料合成了磺化度60%的磺化聚芳砜醚聚合物,该聚合物具有较高的相对黏度,同样也表明了SDFDPS的高纯度。

关键词

[磺化反应](#) [二氟二苯砜](#) [磺化聚芳砜醚](#) [质子交换膜](#)

分类号

Synthesis and characterization of 3,3'-disulfonated-4,4'-difluorodiphenyl sulfone disodium salt

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Abstract

3,3'-Disulfonated-4,4'-difluorophenyl sulfone (SDFDPS) was synthesized from 4,4'-difluorophenyl sulfone (DFDPS) with industrial grade. The starting material DFDPS was purified by sublimation before use. The reaction condition was optimized by varying the molar ratio of SO₃ to DFDPS, reaction temperature and reaction time. The purity and yield of the product was determined by using HPLC, UV, ¹H NMR and FTIR. The optimized reaction condition was found as that the molar ratio of SO₃ to DFDPS is 3.0:1 with a reaction temperature of 110℃ for 20 h, under which neither monosulfonated product nor DFDPS residue was detected. The product SDFDPS was obtained with a total yield of 75% upon recrystallization twice from ethanol/water. Poly(arylene ether sulfone) copolymer with 60% sulfonation degree prepared from the synthesized SDFDPS shows a high relative viscosity, which also indicates the high purity of the synthesized SDFDPS.

Key words

[sulfonation reaction](#) [difluorodiphenyl sulfone](#) [sulfonated poly\(arylene ether sulfone\)](#) [proton exchange membrane](#)

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