
聚乙二醇脱氢枞氧基缩水甘油基醚合成工艺研究

摘 要：以脱氢枞醇为起始原料，首先与环氧乙烷作用生成脱氢枞醇聚氧乙烯醚，然后在 $\text{BF}_3 \cdot (\text{C}_2\text{H}_5)_2\text{O}$ 的催化下，与环氧氯丙烷（ECH）加成生成 3-氯-2-羟丙基聚乙二醇脱氢枞氧基醚，最后在 NaOH 存在下环化得聚乙二醇脱氢枞基缩水甘油基醚。用红外光谱仪表征了原料、中间体及产物的结构中的特征官能团，盐酸-丙酮法测定了产物中环氧值含量，正交实验方法优化了聚乙二醇脱氢枞基缩水甘油基醚的合成条件。结果表明，以 $\text{BF}_3 \cdot (\text{C}_2\text{H}_5)_2\text{O}$ 为催化剂催化合成聚乙二醇脱氢枞基缩水甘油基醚时，其最佳条件为：开环反应的温度为 65°C ，反应时间 6.0h， $n(\text{ECH})/n(\text{脱氢枞醇聚氧乙烯醚})$ 为 2.5， $m(\text{BF}_3 \cdot \text{Et}_2\text{O})/m(\text{脱氢枞醇聚氧乙烯醚})$ 为 5.0%；在此条件下，聚乙二醇脱氢枞基缩水甘油基醚环氧值含量达到 88.48%。

关键词：脱氢枞醇聚氧乙烯醚；聚乙二醇脱氢枞基缩水甘油基醚；正交实验

Study on the synthesis technology of polyethylene glycol dehydroabieoyl glycidyl ether

Abstract: With dehydroabieol as the starting material, dehydroabieol polyoxyethylene ether was first formed by the interaction with ethylene oxide, and then, under the catalysis of BF_3 -ethyl ether, it was added with epichlorohydrin to form 3-chloro-2-hydroxypropyl polyethylene glycol dehydroabiethoxyether. Finally, in the presence of NaOH, the cyclization of polyethylene glycol dehydroabiethoyl glycidyl ether was obtained. The characteristic functional groups in the structure of raw materials, intermediates and products were characterized by infrared spectrometer, the content of epoxy in the products was determined by hydrochloric acid-acetone method, and the synthesis conditions of polyethylene glycol dehydroabieoyl glycidyl ether were optimized by orthogonal experiment. The results showed that the optimum conditions for the synthesis of polyethylene glycol dehydroabietic glycidyl ether with BF_3 -ether as catalyst were as follows: the addition reaction temperature and time were $65\text{ }^\circ\text{C}$ and 6.0h respectively, the molar ratio of epichlorohydrin to dehydroabietic polyoxyethylene ether was 2.5, and the amount of BF_3 -ether was 5.0% of the mass of dehydroabietic polyoxyethylene ether. Under these conditions, the content of epoxy value of peg dehydroabietic glycidyl ether was 88.48%.

Key words: Dehydroabieol polyoxyethylene ether; Polyethylene glycol dehydroabietic glycidyl ether; Orthogonal test

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