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分离工程

超声波协同反应-结晶耦合单离脱氢枞酸

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摘要

以歧化松香和2-乙醇胺为原料、乙醇为溶剂、超声波协同反应-结晶制备脱氢枞酸乙醇胺盐，经萃取、结晶、重结晶、酸化制得脱氢枞酸。通过正交优化实验，考察了溶剂浓度、反应温度、反应时间、超声波功率、搅拌转速对脱氢枞酸收率的影响，确定最佳单离条件为：反应时间50 min,反应温度35℃,超声波功率500 W，溶剂浓度50%，搅拌转速400 r•min^{-1}，在该操作条件下脱氢枞酸的收率达55.37%，纯度达99.53%。并探讨了脱氢枞酸胺化反应-结晶过程相态变化对反应平衡和选择性的影响，当反应温度分别为35℃和70℃时，胺化反应是在非均相和均相下进行，所得脱氢枞酸产品的纯度为99.53%和95.66%。采用GC、GC-MS、元素分析、UV、FT-IR、熔点仪和旋光仪对脱氢枞酸产品进行了分析鉴定，实验值与文献值吻合。

关键词

歧化松香 脱氢枞酸 超声波 反应-结晶

分类号

Isolation of dehydroabietic acid by reaction-crystallization coupled with ultrasound wave

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Abstract

The preparation of 2-aminoethanol salt of dehydroabietic acid from disproportionated rosin by reaction-crystallization coupled with ultrasound wave was studied by using disproportionated rosin and 2-aminoethanol as raw materials, ethanol as reaction solvent, and pure dehydroabietic acid could be obtained by extraction, crystallization, recrystallization and acidification.The effects of concentration of ethanol, reaction temperature, reaction time, ultrasound intensity and agitating velocity on the yield of dehydroabietic acid were investigated by orthogonal experiment, and the optimal conditions for isolation were obtained as follows: reaction time 50 min, reaction temperature 35℃, ultrasound power 500 W, concentration of ethanol 50%, agitating velocity 400 r•min^{-1}.Under these conditions, the yield of dehydroabietic acid was 55.37%, and the purity was 99.53%.At the same time, the effect of the conversion of phase state during the aminating reaction-crystallization on the chemical equilibrium and selectivity was investigated.When reaction temperatures were 35℃ and 70℃, respectively, the reaction was heterogeneous and homogeneous, and the purities of dehydroabietic acid prepared were 99.53% and 95.66%.The dehydroabietic acid prepared was analyzed and confirmed with GC, GC-MS, elemental analysis, UV, FT-IR, melting point instrument and polarimeter as well, and the results were in good agreement with the values in literature.

Key words

disproportionated rosin dehydroabietic acid ultrasound wave reaction-crystallization

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