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Structure dependent thermo-reversible dissolution of organic molecules based on β-cyclodextrin complexes and its application in preparetive-scale separation of xylene isomers

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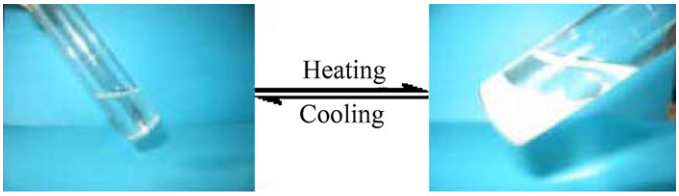
HIGHLIGHTS

- An unusual structure dependent thermo-reversible precipitation–dissolution behavior was reported.
- Varieties of small organic molecules could be precipitated/dissolved in the presence of β-CD by heating/cooling in DMF.
- They can form organic molecules–β-CD complex by heating.
- Different organic molecule–β-CD complex precipitates corresponded to different temperature.
- This behavior was successfully applied for the separation of xylene isomer mixtures in DMF solution.

GRAPHICAL ABSTRACT

Varieties of small organic molecules can be precipitated in DMF by β-CD in the form of inclusion complex at different temperatures by a molecular recognition thermo-reversible process. The finding was successfully applied for the separation of xylene isomer mixtures in a preparation-scale in DMF solution.



The thermo-reversible dissolution of organic molecule-β-CD complex precipitates

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ABSTRACT

This paper describes an interesting structure dependent thermo-reversible dissolution of organic molecules in the presence of β-cyclodextrin (β-CD) in N, N-dimethyllformamide (DMF). β-CD could influence the solubility of dissolved organic molecules, resulting in the precipitation of organic molecule–β-CD complex from the original transparent solution as the temperature is increasing, while the precipitate will be dissolved again upon cooling. Different organic molecule–β-CD complex precipitates corresponded to different turbid temperatures (T_t). No precipitates were observed solely for either organic molecules or β-CD in DMF when the temperature increased, indicating the structure dependent recognition of β-CD to organic molecules. This molecular recognition behavior had been successfully applied in the separation of xylene isomers in preparative scale.

1. Introduction

Normally, organic molecules dissolve very well in DMF, particularly as the temperature increases [1–4]. Cyclodextrins (CDs, including α-, β-, γ-, etc.) with hydrophobic pockets and hydrophilic exteriors can complex with various organic quest molecules through supramolecular interactions [5,6]. Most of the organic molecules can bind CDs at room temperature (r.t.) in many non-aqueous solvents, such as dimethyl sulfoxide (DMSO), DMF, acetonitrile, etc., to form supramolecular complex in solution [7–15]. The stability of the complex is relevant to the structure of the guest molecules and the CDs. It also depends on the external environments, such as the temperature, the components of the solvent media, etc. Generally, when the temperature or the organic

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