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USE OF SELECTIVE MOF FORMATION FOR SEPARATION OF α-, β- AND γ-CYCLODEXTRINS

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Cyclodextrins (CD's) are cyclic oligosaccharides widely used in pharmaceutical, food and cosmetic industries as excipients and stabilizers. They are produced by enzymatic transformation of starch, obtained from renewable green sources (potato, corn, etc.). This process is unselective and typically leads to a formation of a mixture of CD's with various numbers of glucose subunits in the cycle (6 for α, 7 for β and 8 for γ). One of approaches to their separation is a selective formation of a metal-organic framework (MOF), where a CD molecule serves as a ligand [1].
In order to create an efficient, sustainable and scalable separation process behavior of various CD mixtures was investigated under MOF crystallization conditions. In all cases crystallization of MOF was carried out in the presence of Na2CO3 in aq. MeOH. The precipitated material was investigated by powder X-ray diffractometry and obtained results are summarized on ternary diagram (Figure 1).

Fig. 1. Ternary diagram of crystalline pro¬ducts obtained from various compo¬si¬ti¬ons of β-CD, γ-CD and Na2CO3 with dif¬fe¬rent mass ratios (border lines between phases are put for clarity, they are not accurate).
α-CD remained in solution in a wide range of concentrations (10-90 mass% from the total mass of CD's). It was excluded and a variable amount of Na2CO3 was included in the diagram instead. The least soluble β-CD dominates in the solid phase when its amount in solution is >50 mass%; while in the 20-50 mass% range a mixture β-CD + γ-CD-Na MOF is precipitated. Low concentrations of Na2CO3 (<10 mass%) lead to precipitation of γ-CD·MeOH solvate with a low yield. At a higher concentration of Na2CO3 (>40 mass%) γ-CD-Na is formed, but it is contaminated with Na2CO3. The most efficient crystallization of pure γ-CD-Na was achieved in 10-40 mass% range of Na2CO3 with <20 mass% of β-CD.
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References:
[1] Limketkai, B. N.; Botros, Y. Y. Methods to Isolate Cyclodextrins. US2017058306A1, 02.03.2017.

Primary authors: ŠUBINS, Kirills (Latvian Institute of Organic Synthesis); KRŪKLE-BĒRZIŅA, Kristīne (Latvian Institute of Organic Synthesis); BEĻAKOVS, Sergejs (Latvian Institute of Organic Synthesis)

Presenter: KRŪKLE-BĒRZIŅA, Kristīne (Latvian Institute of Organic Synthesis)

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