

Synthesis and Characterization of Polyrotaxane based on Mono-6-Tosyl- β -Cyclodextrin

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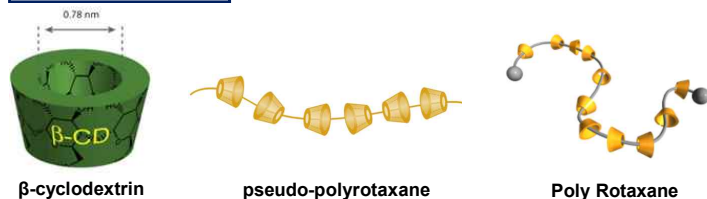
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Abstract

β -Cyclodextrin(β -CD) is a type of cyclic oligosaccharide used in various fields such as drug delivery, gene therapy, catalyst, chemical sensor, and liquid crystal. Mono-6-deoxy-6-(p-tolylsulfonyl)- β -cyclodextrin (TsO- β -CD) was synthesized by replacing one hydroxyl group among 21 hydroxyl groups in β -CD under various pH conditions using p-toluenesulfonyl chloride(p-TsCl), and used to synthesis polyrotaxane by threading TsO- β -CD on PPG-diamine with a molecular weight of 2000, followed by end capping using Trt-Gly-OH, N-Hydroxysuccinimide (NHS), ethylene dichloride (EDC). The synthesized CD derivatives and polyrotaxane were characterized by ¹H NMR and FTIR spectroscopies.

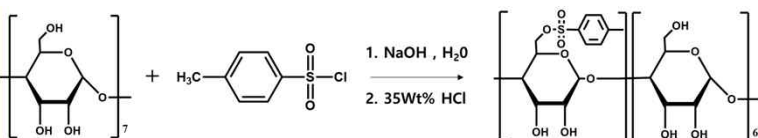
Introduction



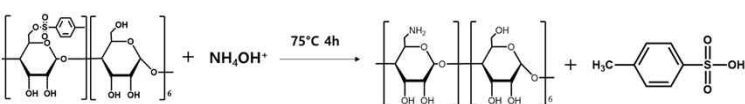
β -CD is a cyclic oligosaccharide composed of 7 glucose units. TsO- β -CD is produced through the tosylation reaction. TsO- β -CD host-guest interaction allows PPG chains to enter. End groups are capped with macromolecular substances.

Experimental

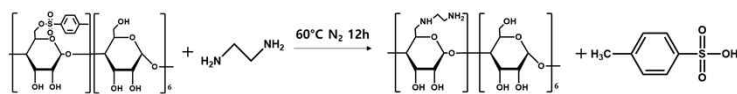
Synthesis of mono-6-deoxy-6-(p-tolylsulfonyl)- β -cyclodextrin (TsO- β -CD)



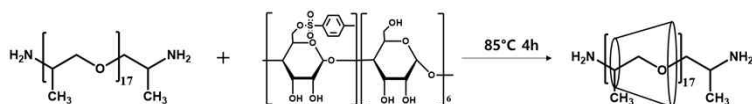
Synthesis of mono-6-deoxy-6-amino- β -cyclodextrin (NH₂- β -CD)



Synthesis of mono-6-deoxy-6-aminoethylamino- β -cyclodextrin (Et- β -CD)



Synthesis of mono-6-deoxy-6-(p-tolylsulfonyl)- β -cyclodextrin pseudo-polyrotaxane (TsPPR)

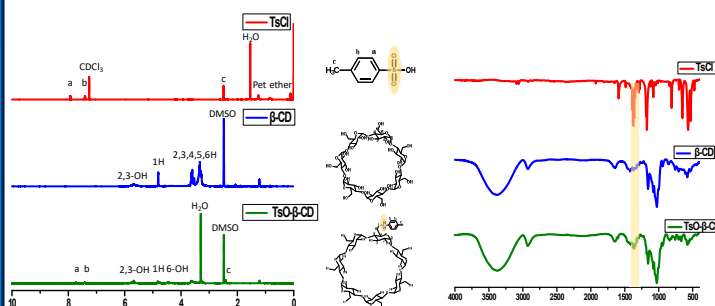


Mole ratio of TsO- β -CD, NH₂- β -CD and Et- β -CD TsPPR

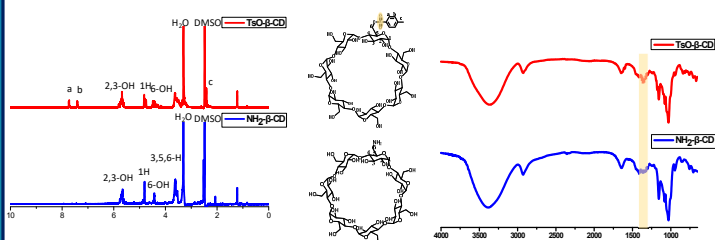
Material	β -CD	P-TsCl	Material	TsO- β -CD	NH ₄ OH	Material	TsO- β -CD	EDA	Material	TsO- β -CD	PPGN
Mole ratio	1	1.8	Mole ratio	1	1116	Mole ratio	1	664	Mole ratio	8	1

Results and Discussion

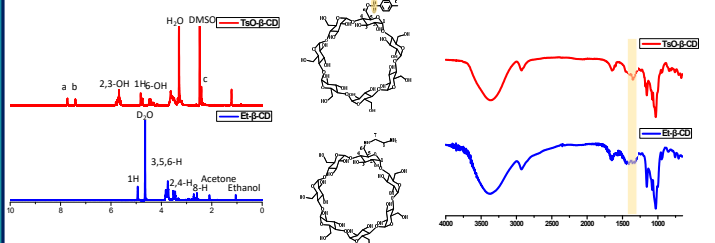
TsO- β -CD



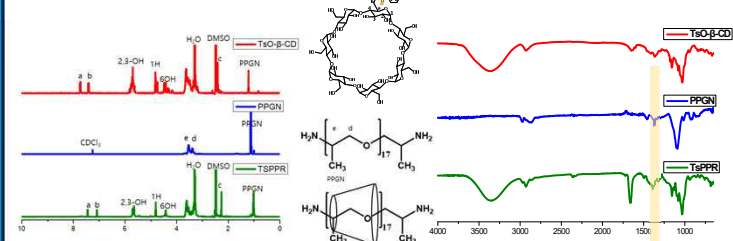
NH₂- β -CD



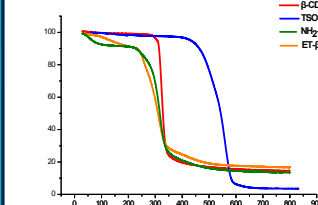
Et- β -CD



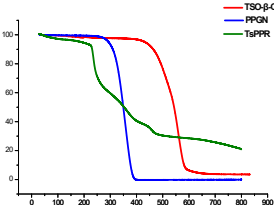
TSPPR



TGA of CD derivatives



TGA of TSPPR



Conclusion

TsO- β -CD was synthesized from β -CD and p-TsCl, and one of 21 hydroxyl groups was successfully converted. TsO- β -CD was synthesized by replacing the tosyl group with an amine group using ammonia water or changing the tosyl group using EDA. TsO- β -CD and PPG diamine were Threaded successfully. TsO- β -CD, TSPPR, NH₂- β -CD and Et- β -CD were synthesized and confirmed by FT-IR and ¹H-NMR spectroscopies.