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REGIOSELECTIVE SYNTHESIS OF CELLULOSE CARBAMATE-BASED CHIRAL CATION EXCHANGERS FOR ENANTIOMER RESOLUTION BY HPLC

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Introduction

Chirality is a basic feature in the structure and chemistry of all living matter. Due to the asymmetric nature, chiral compounds exhibit different properties (*e.g.* *D*-asparagine with sweet taste, *L*-asparagine with bitter taste, *S*-limonene with lemon flavor, and *R*-limonene with orange flavor). Enantiomers can be chromatographically separated (*e.g.* by high-performance liquid chromatography) if the stationary phases themselves are chiral. Since Hesse and Hagel, the pioneers in the successful application of cellulose triacetate as chiral stationary phases in 1976, different polysaccharides and their derivatives have been exploited and employed as chiral stationary phases due to their high enantiomer separation power.

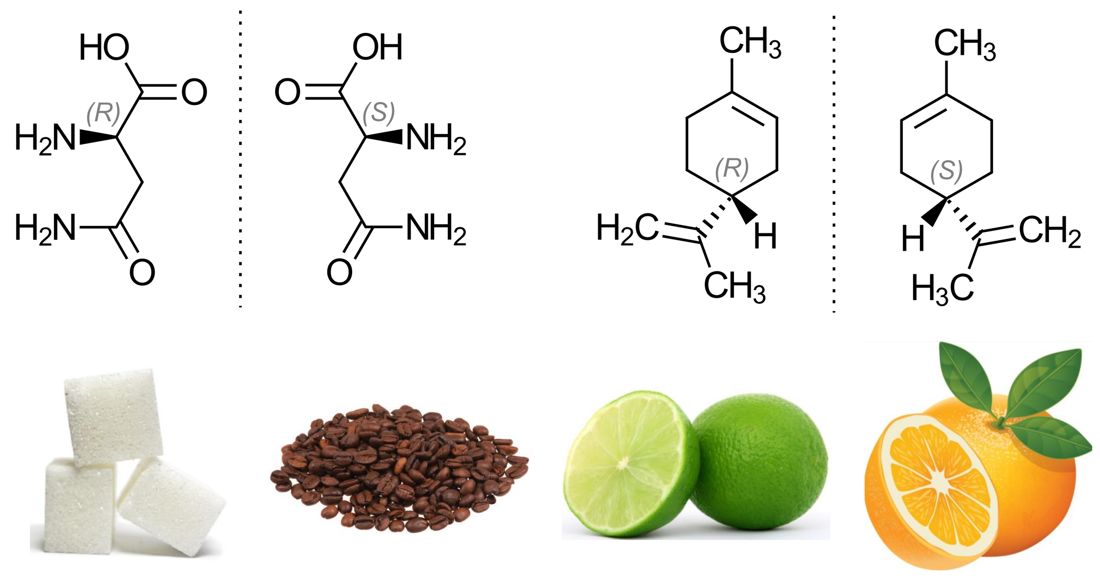

Fig. 1. The chemical structures of asparagine (left) and limonene (right)

Aim – Objectives

The purpose of this project is to synthesize cellulose derivatives as chiral separator phases and to evaluate their enantiomer separation performance in high-performance liquid chromatography (HPLC).

Methods

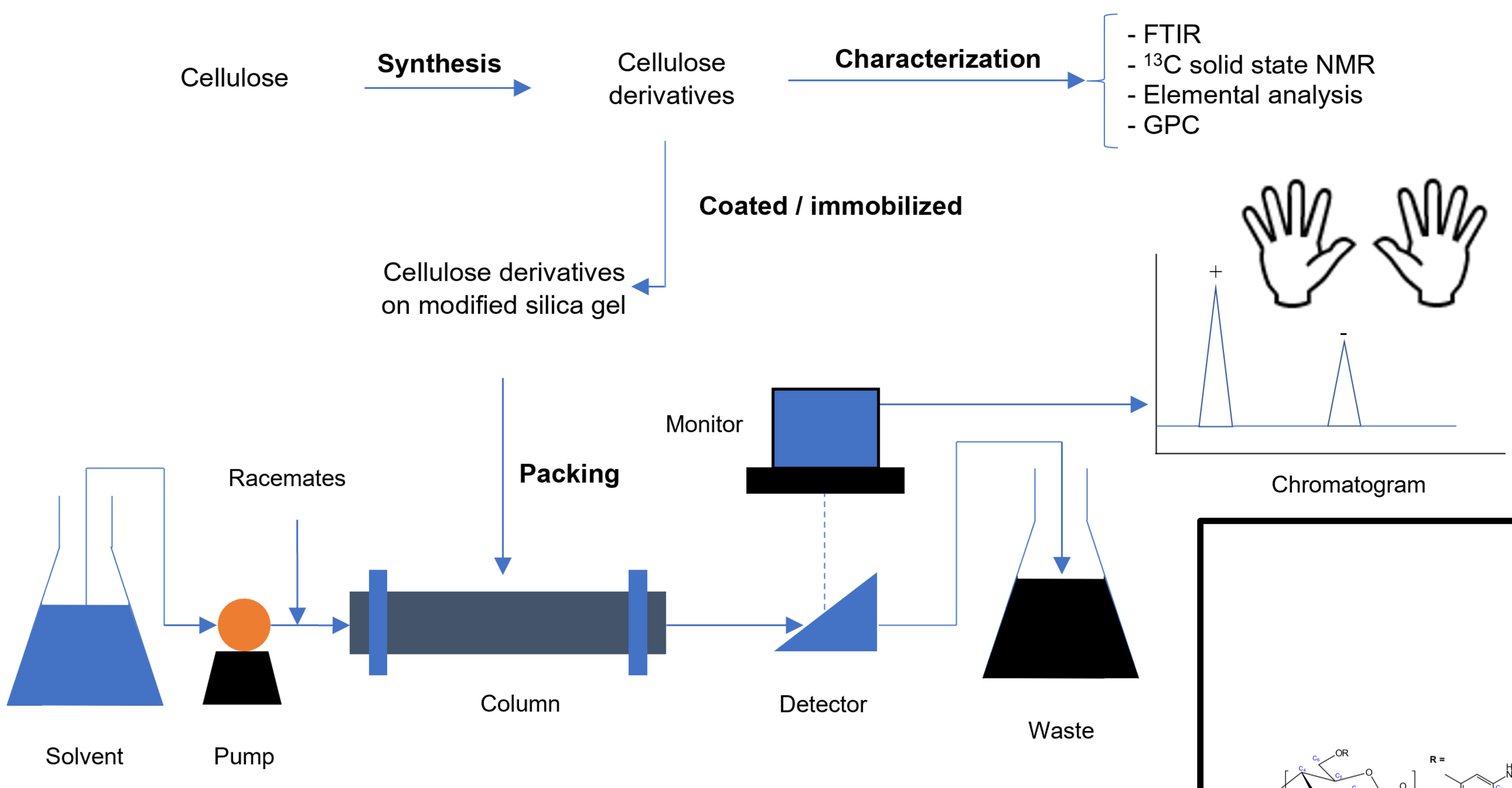

Fig. 2. Experimental setup

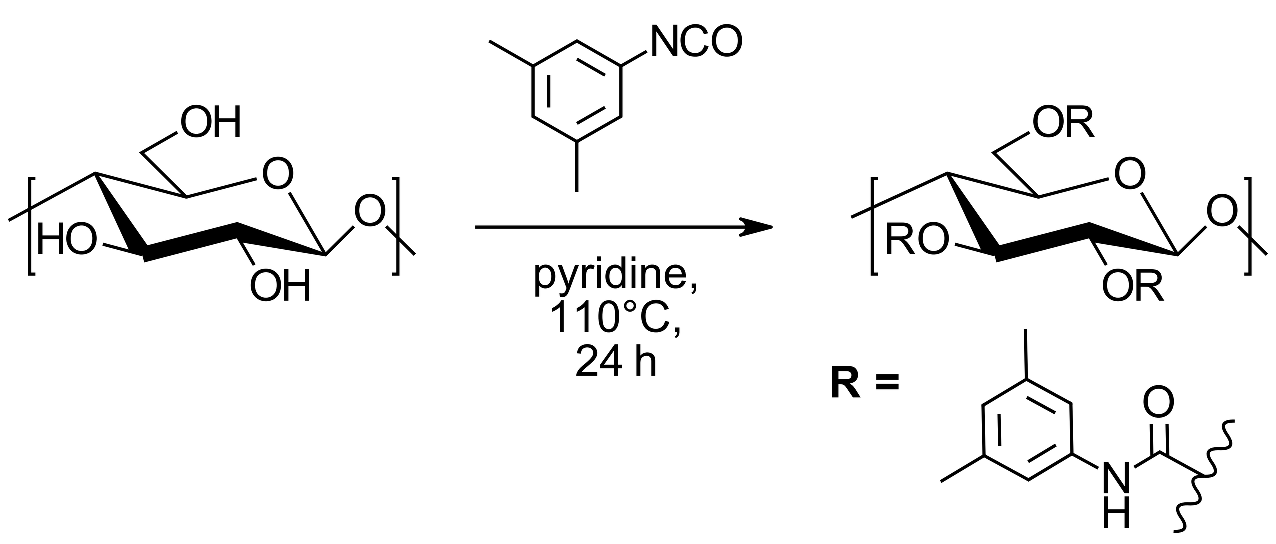

Fig. 4. Synthesis of cellulose *tris*-(3,5-dimethylphenyl carbamate)

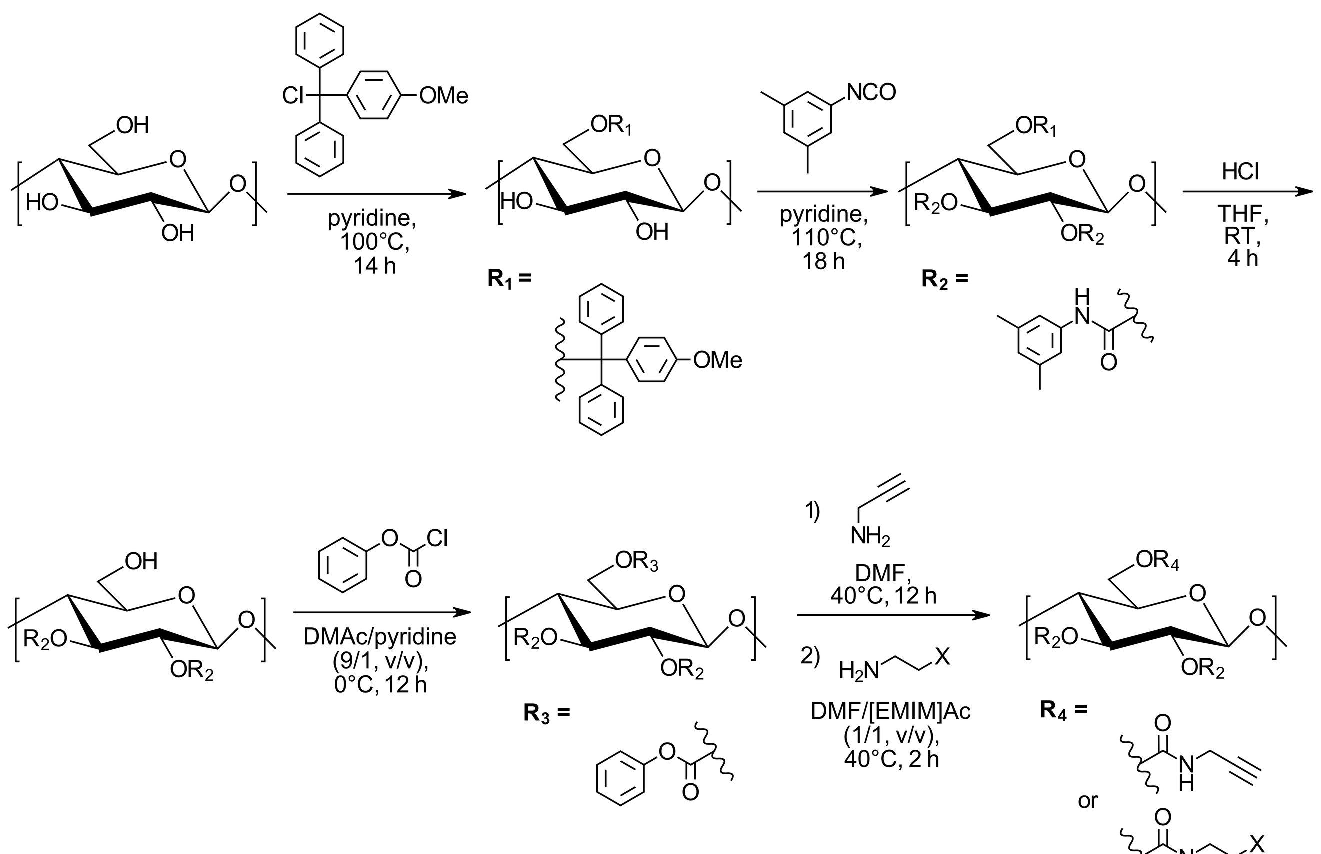

Fig. 3. Regioselective carbamoylation by aminolysis with propargyl amine, taurine, and β-alanine

Results

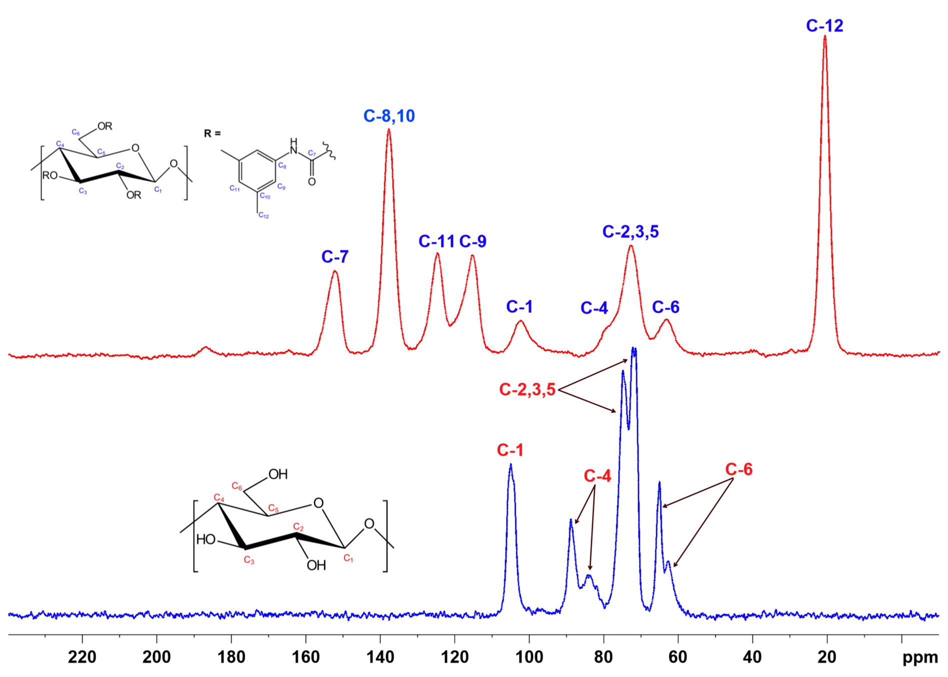

Fig. 5. Solid state ^{13}C NMR spectra of cellulose *tris*-(3,5-dimethylphenyl carbamate) *vs.* microcrystalline cellulose

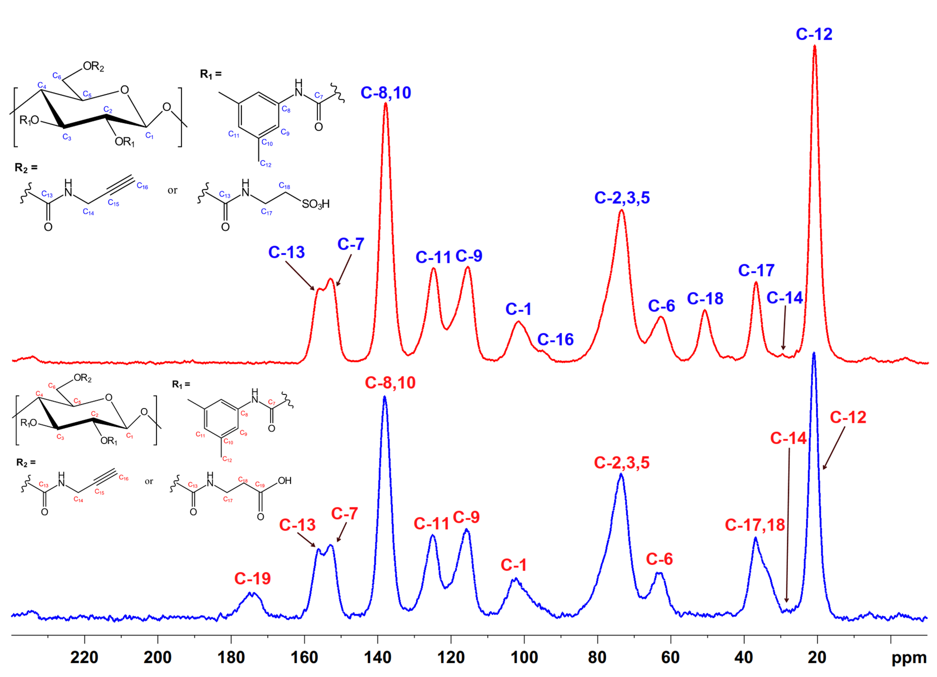

Fig. 6. Solid state ^{13}C NMR spectra of cellulose derivatives after aminolysis with propargyl amine, taurine and β-alanine

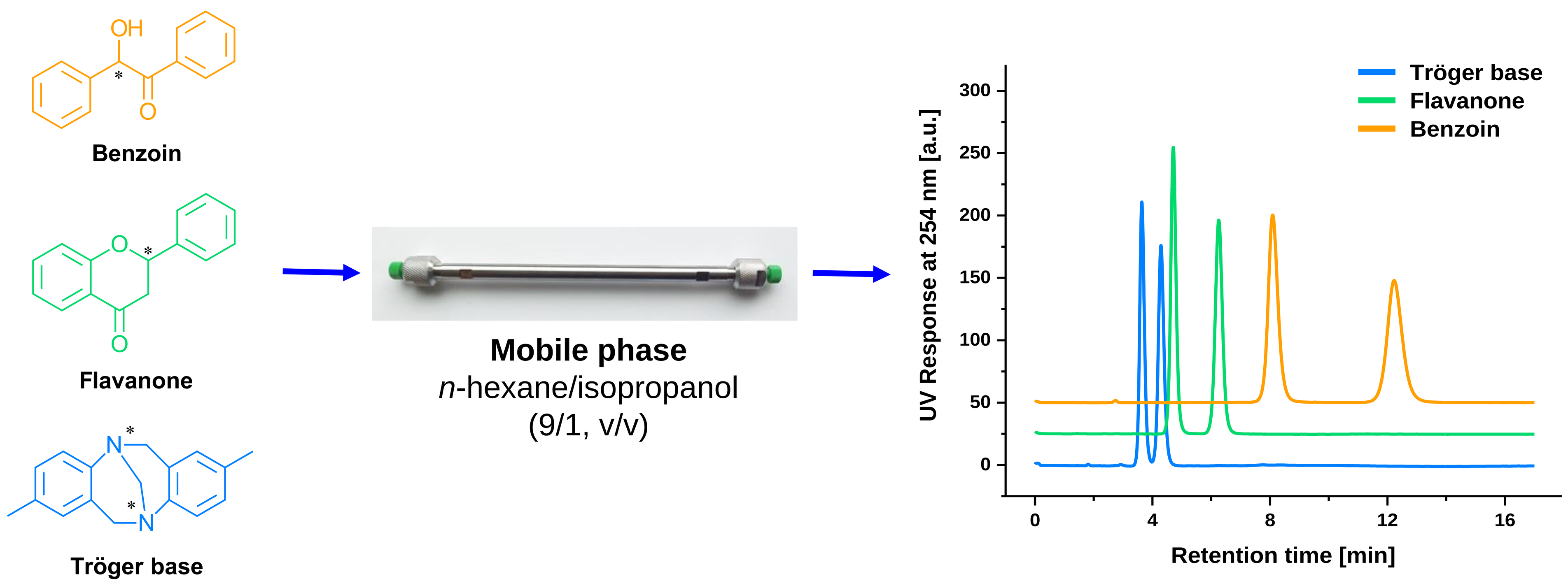

Fig. 7. The chemical structures of the analytes, a prepared HPLC column, and enantiomer separation in case of cellulose *tris*-(3,5-dimethylphenyl carbamate) as chiral selector

Conclusion and Outlook

- Successful synthesis and full characterization of a reference chiral selector and newly developed chiral selectors.
- High enantiomer separation performance of the reference chiral selector.
- Continuing evaluation of enantiomer separation performance of the newly developed chiral selectors.

References

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[2] Hettegger, H.; Lindner, W; Rosenau, T. (2020): Derivatized polysaccharides on silica and hybridized with silica in chromatography and separation – a mini review. In: Rauter, A.P.; Christensen, B.; Somsak, L.; Kosma, P.; Adamo, R. (Eds.), Recent Trends in Carbohydrate Chemistry: Synthesis, Structure and Function of Carbohydrates, 492; Elsevier, Amsterdam, The Netherlands; ISBN 9780128174678

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