

DEVELOPMENT OF CELLULOSE ESTERS FOR APPLICATIONS IN ADHESIVES

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Introduction

UV-curing adhesives

UV-curing adhesives

- are often used in the electronic industry, furniture and wood industry, the optical industry, etc.
- different chemical classes are used, e.g. acrylates, epoxies, silicones, etc.
- they have to fulfill many requirements like:
 - fast curing
 - transparency
 - strong adhesion
 - UV-stability
 - weather and ageing resistance
 - ...



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Motivation, challenges & aim

Bioadhesives

Why bioadhesives?

- environmentally friendly (low PCF), as made from renewable resources like **natural polymers** (such as chitosan, cellulose, lignin, gelatin), **oils**, etc.
- can be advantageous in terms of biocompatibility, toxicity and recyclability



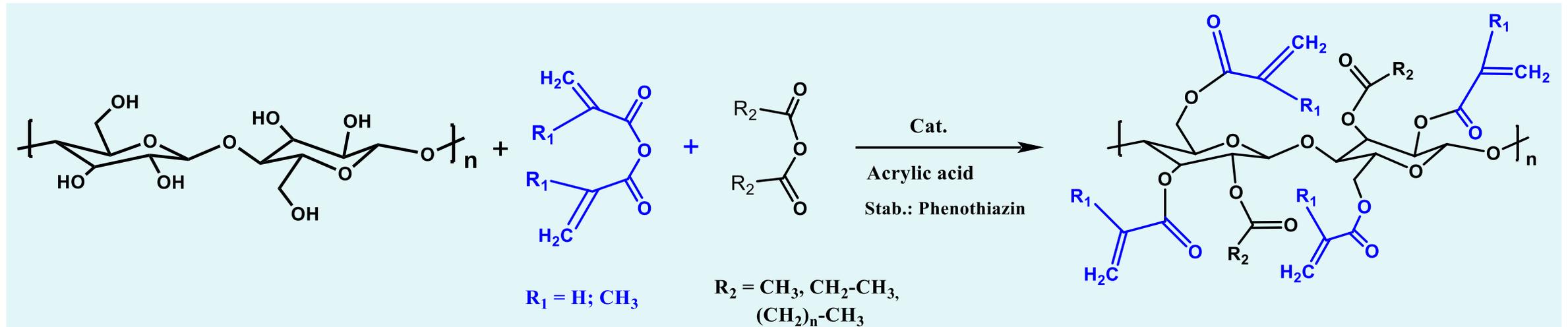
What are the challenges in developing new bioadhesives?

- They need to have similar properties like their petrochemical competitors (including ageing behavior and others)
- The manufacturing process needs to be competitive

Aim: Modification of cellulose for fast UV-curing bioadhesives using industrially scalable processes

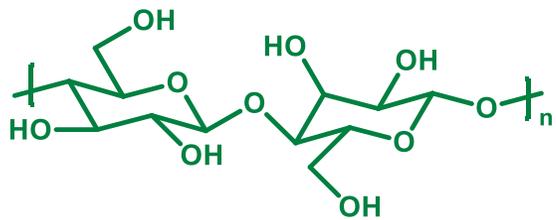
Efficient synthesis of cellulose acrylates / methacrylates

Esterification of wood pulp cellulose

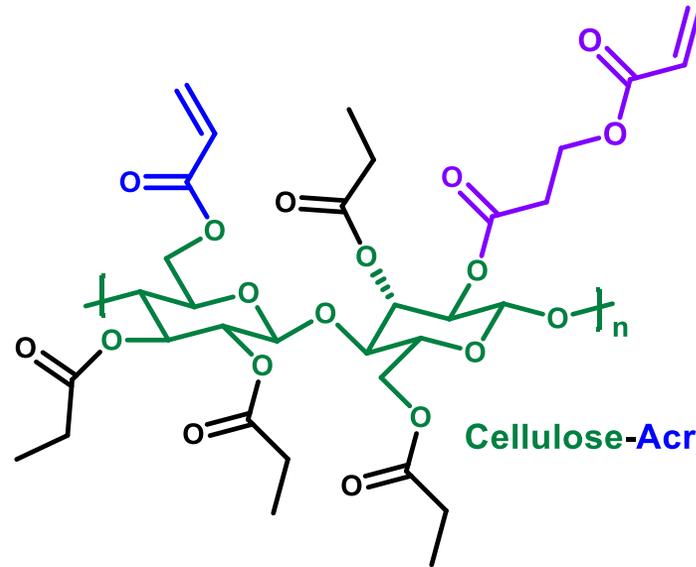
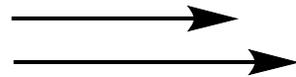


- Synthesis process is analogous to the industrial production of cellulose triacetate
- No use of toxic or chlorine containing reagents, no harmful byproducts, atom-efficient procedure
- Variation of structural features possible (**degree of functionalization, molecular weight, solubility ...**)

Synthesis of cellulose acrylate diacrylates from wood pulp

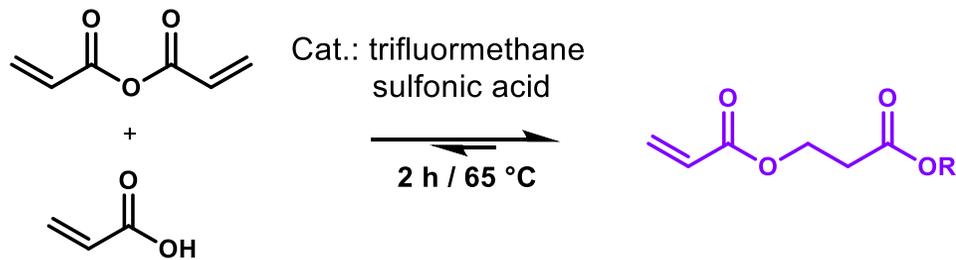


Wood pulp



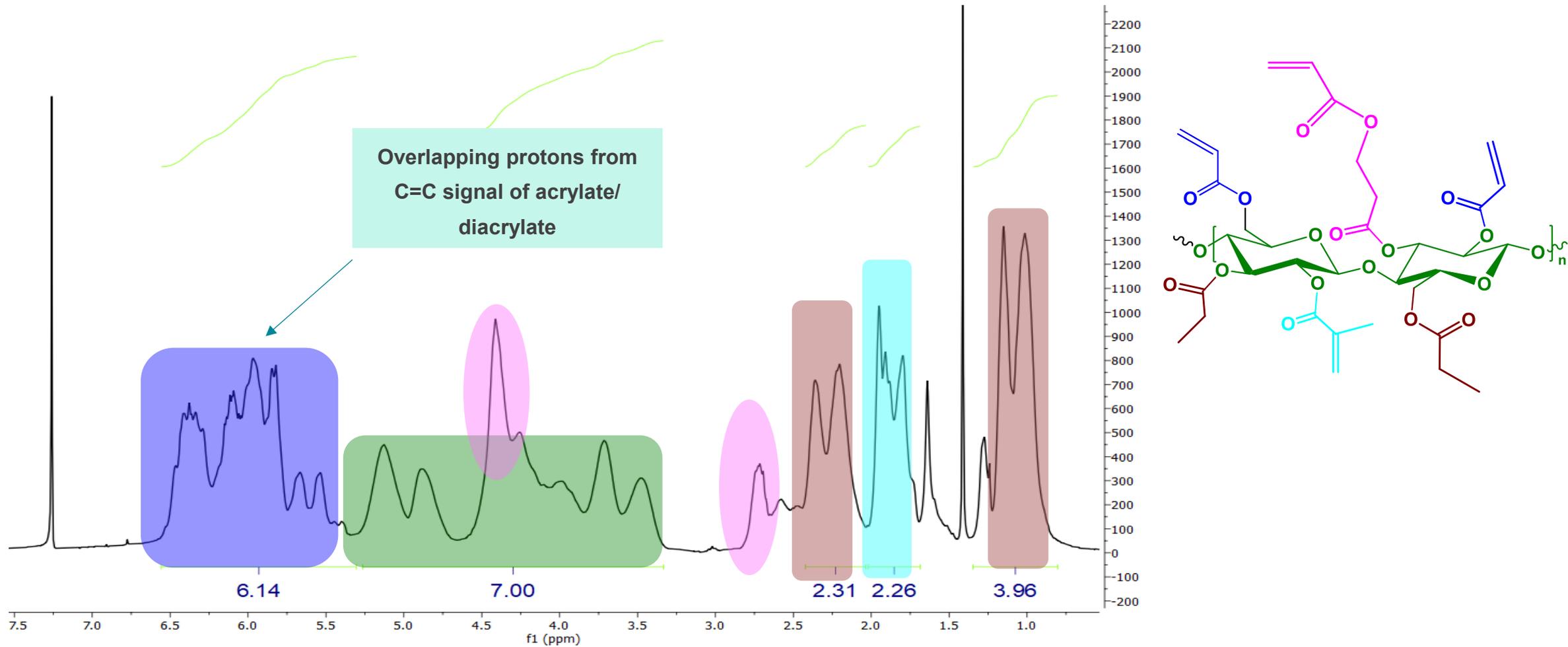
Cellulose-Acrylate-Diacrylate-Propionate

In-situ diacrylate formation

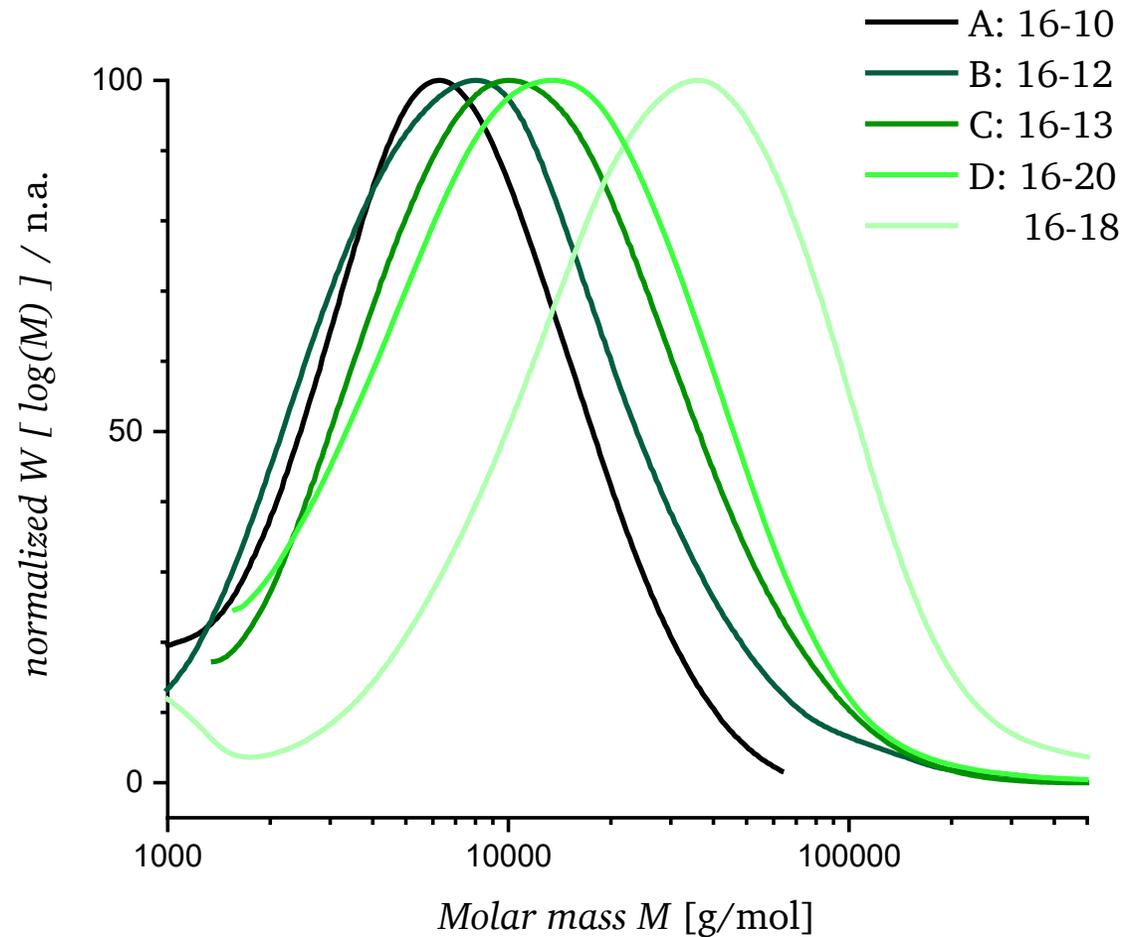


- ✓ Product is well-soluble and compatible with various comonomers
- ✓ Using of low-cost wood pulp as starting material
- ✓ Synthesis conducted in 300 g scale, further upscaling planned

Characterization via $^1\text{H-NMR}$



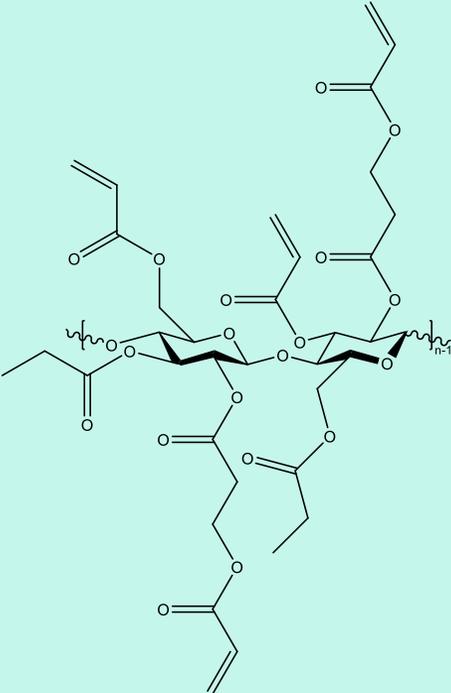
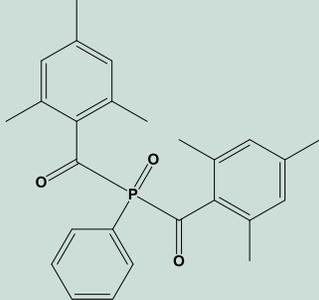
Targeted synthesis of cellulose esters with graduated molecular weights

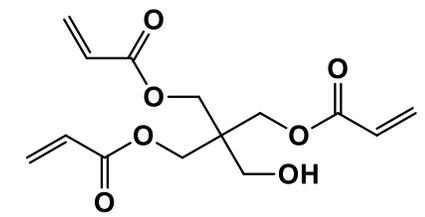


- ✓ Solubility of cellulose esters and their compatibility with comonomers depends on the molecular weight
- ✓ Molecular weight is dependent on the reaction conditions applied
- ✓ Synthesis of ester with very different molecular weights possible

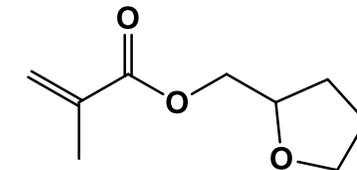
Co-monomer selection

Solubility and transparency screening

CeAc	Co-monomer 1	Co-monomer 2	Photoinitiator
	1,4 BDDMA	-	 <p>Irgacure 819</p>
	1,4-BDDA	-	
	DTMPTA	-	
	PETIA	-	
	THFMA	-	
	1,4-BDDMA	THFMA	
	1,4-BDDA	THFMA	
	PETIA	1,4-BDDA	
	PETIA	Tetrahydrofurfuryl methacrylate	
	HEA	-	
	HEA	THFMA	
	HEMA	THFMA	
	EHA	THFMA	
	Isobornyl acrylate	THFMA	
	Lauryl acrylate	THFMA	
	Lauryl methacrylate	THFMA	
	1,10-Decadiol diacrylate	THFMA	
	Tri(propylene) glycol diacrylate	THFMA	
Dipentaerythritol penta-/hexa-acrylate	THFMA		



Pentaerythritol triacrylate

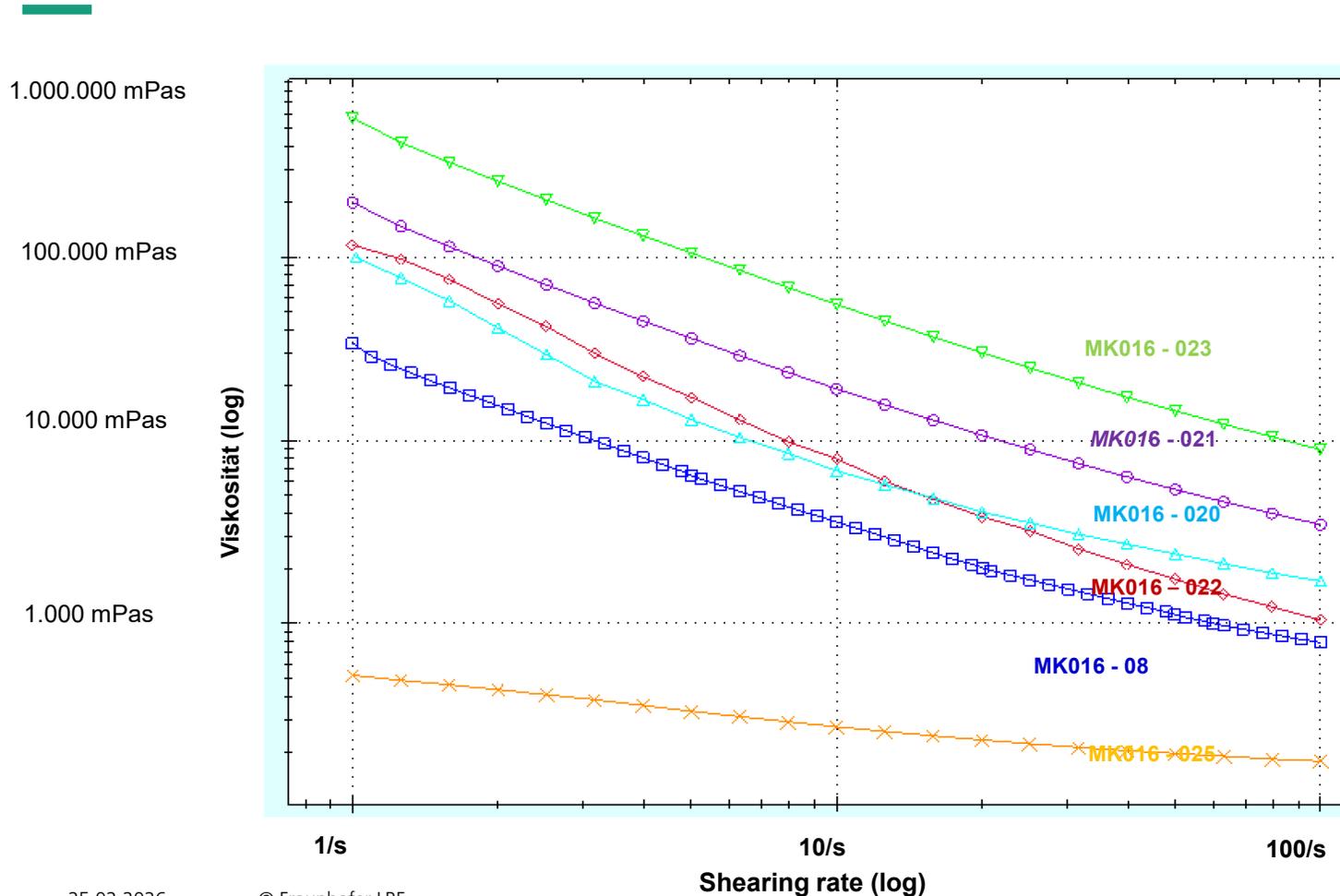


Tetrahydrofurfuryl methacrylate

Features: low viscosity, good adhesion, low shrinkage, high hardness, good balance of mechanical properties **AND** partially bio-based

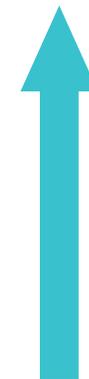
Investigations of various cellulose-acrylate-diacrylate-propionates

Rheological tests: 25 wt.% solution in HEMA (hydroxyethyl methacrylate)



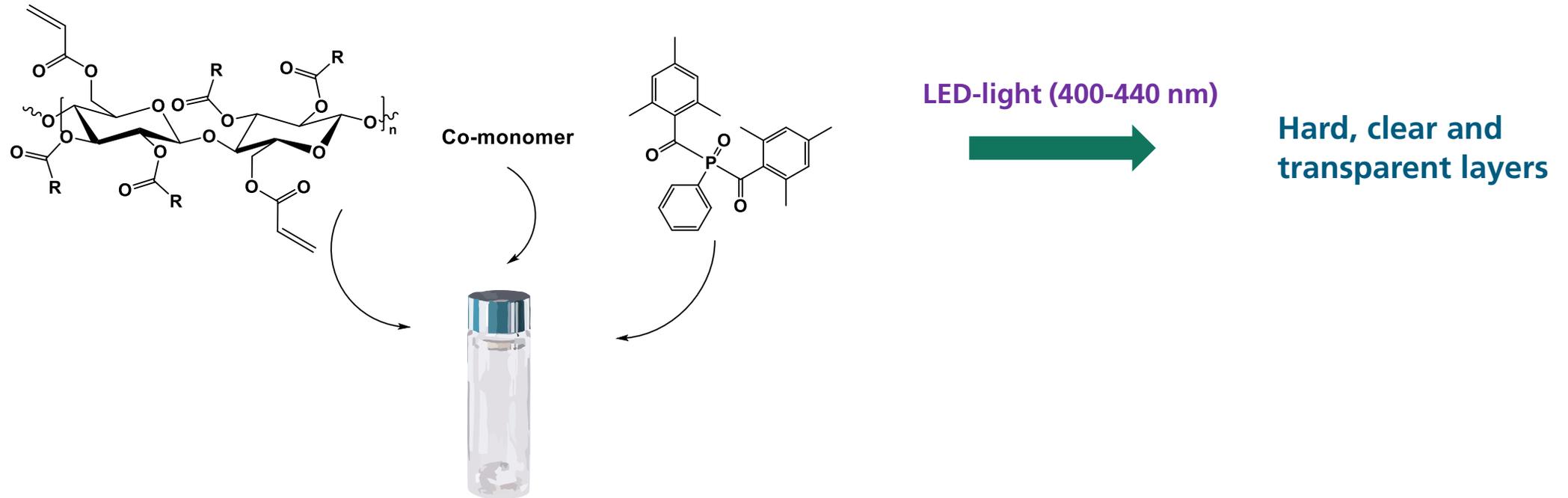
Viscosity

Shearing rate



- ✓ Viscosity decreases with increasing shearing rate
- ✓ The structural characteristics of cellulose esters strongly influence the viscosity
- ✓ Viscosity can be adapted in dependence of requirements

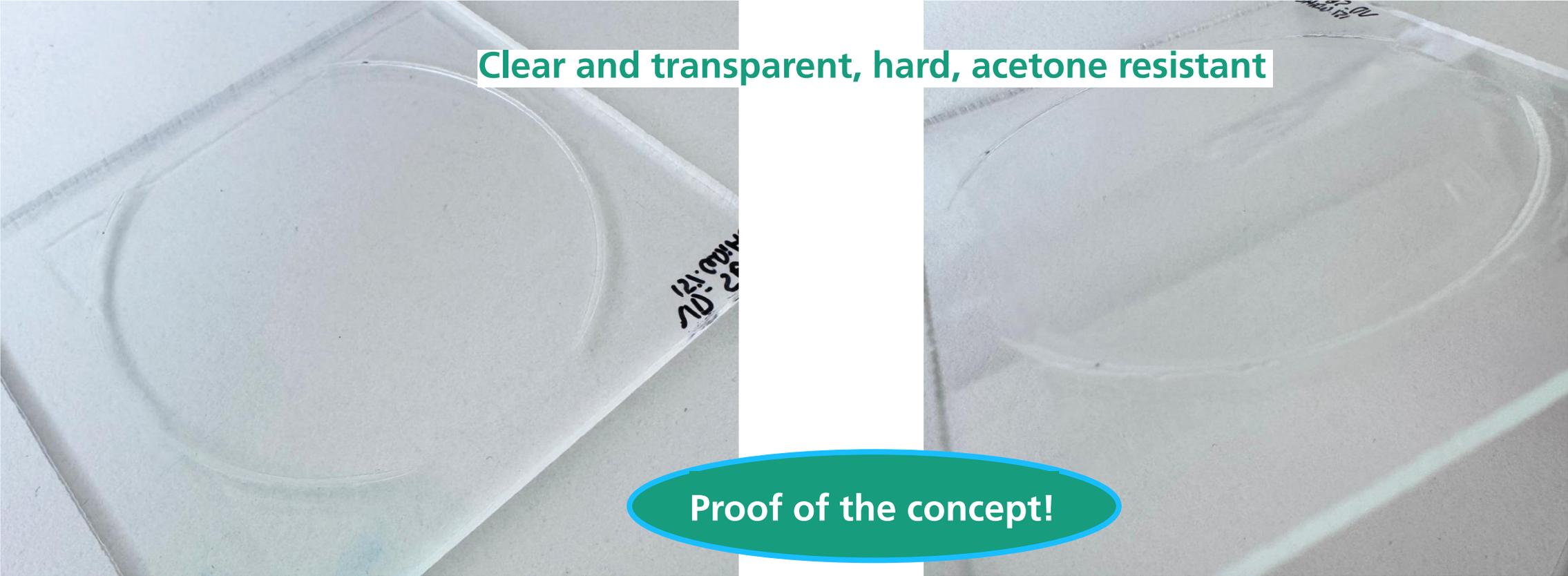
LED-light curing



The light curing process was thoroughly investigated using a near-infrared spectroscopy and ultrasonic reflectometry

LED light curing

under N₂, at room temperature with blue LED light (405 nm) for 25 s



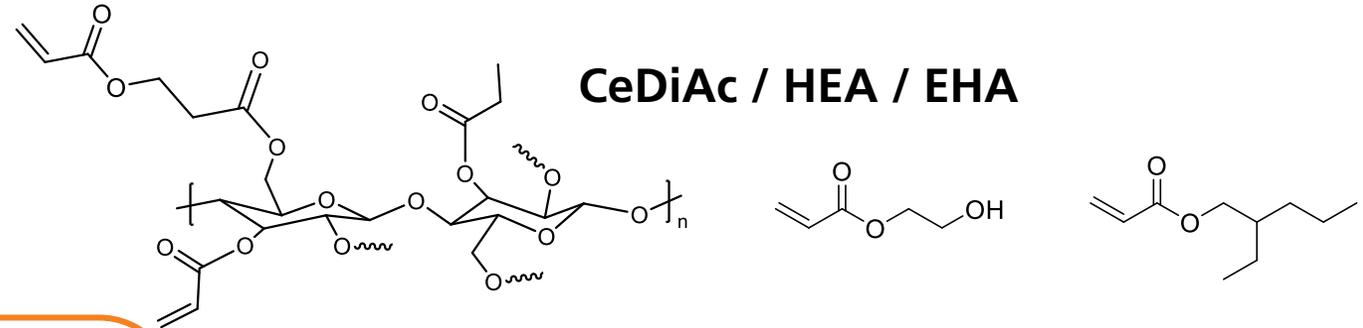
Clear and transparent, hard, acetone resistant

Proof of the concept!

Formulation contains celuose acrylate / methacrylate and suitable comonomers (preferably biobased ones)

LED light curing

Mechanical properties in dependence of structural parameters



Module	20/80/00	20/60/20	20/50/30	20/40/40	20/30/50	M_n/M_w	DA	#
G_n / GPa	2.8 ± 0.9	1.7 ± 0.1	1.71 ± 0.6	1.2 ± 0.4	0.52 ± 0.01	4 kDa	50%	A
L_n / GPa	16.0 ± 0.4	12 ± 2	10 ± 2	8 ± 1	4.5 ± 0.2	9 kDa		
G_n / GPa	3.2 ± 0.5	1.4 ± 0.02	2.7 ± 0.2	0.8 ± 0.1	0.4 ± 0.3	5 kDa	20%	B
L_n / GPa	10 ± 0.8	8.6 ± 1	6.2 ± 0.2	4.6 ± 0.4	3.8 ± 0.5	14 kDa		
G_n / GPa	2.0 ± 0.2	1.4 ± 0.2	1.2 ± 0.07	1.9 ± 0.3	0.5 ± 0.07	7 kDa	45%	C
L_n / GPa	10 ± 0.06	6.8 ± 0.4	6.2 ± 0.5	4.0 ± 0.6	3.9 ± 0.2	18 kDa		
G_n / GPa	2.4 ± 0.2	1.7 ± 0.1	2.1 ± 0.2	1.13 ± 0.03	0.8 ± 0.1	8 kDa	41 %	D
L_n / GPa	16.8 ± 0.9	14.5 ± 3	11.2 ± 2	6.7 ± 0.6	5 ± 0.5	24 kDa		

Summary & outlook

Summary

- Novel class of cellulose acrylates introduces as binder for biobased adhesives
- Effect of substitution pattern and molecular weight on relevant properties (solubility in comonomers, transparency and hardness) studied
- Cellulose-acrylate-diacrylate-propionate with co-monomers show promising properties

Outlook

- Application-related tailoring of cellulose acrylates
- Upscaling of synthesis (current status ca. 300 g)
- Search for cooperation opportunities

■ Thank you very much
for your attention!



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