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Supporting Information

A more Sustainable Synthesis Approach for Cellulose Acetate using the DBU/CO₂ Switchable Solvent System

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Table of Content

1	Calculations	2
3	³¹ P NMR spectra of the phosphorylated CAs	4
4	¹ H NMR spectra of the prepared CAs	. 10
5	¹³ C NMR spectra of CAs for individual DS determination	. 16
6	Thermal Analysis	. 20
7	Mechanical Properties	. 21
8	SEC measurement	. 23
9	Solubility Chart	. 24
10	¹ H NMR spectra "vinegar syndrome"	. 25
11	Deacetylation during work-up procedure	. 27
12	"Green" synthesis of vinyl acetate	. 30
13	Recycling	. 31
14	References	. 34

1 Calculations

DS values of cellulose acetate samples

The degree of substitution (DS) based on ¹H NMR spectroscopy (DS_{1H}) was calculated as shown in Eq. (1).

$$DS_{1H} = \frac{7 \times I_{CH3}}{I_{AGU}}$$
(1)

 I_{CH3} : Integral of the peak of the methyl group in the ¹H NMR spectrum. I_{AGU} : Integral of the peak of the anhydroglucose unit (AGU) in the ¹H NMR spectrum.

The DS based on ³¹P NMR spectroscopy (DS_{31P}) was calculated as reported by Kilpeläinen et al.¹ according to Eq. (2) and (3).

$$DS_{31P} = DS_{max} \times \frac{\frac{1}{OH_S} - \frac{1}{OH_C}}{M_S + \frac{1}{OH_S} - 1}$$
 (2)

$$OH_{\rm S} = \frac{c_{\rm IS} \times V_{\rm IS} \times I_{\rm R}}{1000000 \times m_{\rm S}} \tag{3}$$

 DS_{max} : maximum achievable DS value ($DS_{max} = 3$ for unsubstituted cellulose) OH_S : free hydroxyl groups per weight unit of substrate (mol g⁻¹)

OH_C: free hydroxyl groups per weight unit of cellulose (OH_C = DS_{max}/M_{AGU} =

 $3/162.14 \text{ g mol}^{-1}$)

 $M_{\rm S}$: molecular weight of the substituent, not including the linking oxygen atom between the substituent and the cellulose backbone (g mol⁻¹)

 $c_{\rm IS}$: concentration of the used internal standard (mmol L⁻¹)

 $V_{\rm IS}$: volume of the used internal standard (μ L)

 $I_{\rm R}$: integration ratio of remaining functionalized cellulose hydroxyl groups against internal standard

 $m_{\rm S}$: mass of the sample (mg)

Yield calculation

The yield was calculated using Eq. (4).

yield =
$$\frac{\frac{m_{CA}}{M_{repunit}}}{\frac{m_{cellulose}}{M_{AGU}}}$$
(4)

$$M_{\rm repunit} = M_{\rm AGU} + (M_{\rm S} - 1.01 \,{\rm g \, mol^{-1}}) \times {\rm DS}_{31\rm P}$$
 (5)

 m_{CA} : mass of the cellulose acetate product $m_{cellulose}$: mass of the cellulose educt $M_{repunit}$: average molar mass of the product's repeating unit M_{AGU} : molar mass of the anhydroglucose unit ($M_{AGU} = 162.14 \text{ g mol}^{-1}$)

Molar composition of DMSO/DBU mixture calculation

Calculation of the molar composition of recycled DMSO/DBU mixtures was performed using Eq. (6)–(8).

$$\frac{n_{\rm DBU}}{n_{\rm DMSO}} = \frac{\frac{I_{\rm DBU}}{N_{\rm H,DBU}}}{\frac{I_{\rm DMSO}}{N_{\rm H,DMSO}}} = \frac{I_{\rm DBU} \times N_{\rm H,DMSO}}{N_{\rm H,DBU} \times I_{\rm DMSO}}$$
(6)

$$n_{\rm DMSO} = \frac{m_{\rm total}}{M_{\rm DMSO} + \frac{I_{\rm DBU} \times N_{\rm H,DMSO}}{N_{\rm H,DBU} \times I_{\rm DMSO}} \times M_{\rm DBU}}$$
(7)

$$n_{\rm DBU} = \frac{m_{\rm total}}{M_{\rm DBU} + \frac{I_{\rm DMSO} \times N_{\rm H, DBU}}{N_{\rm H, DMSO} \times I_{\rm DBU}} \times M_{\rm DMSO}}$$
(8)

 n_{DBU} : amount of substance of DBU in the mixture

 n_{DMSO} : amount of substance of DMSO in the mixture

 m_{total} : total mass of the mixture

 M_{DBU} : molar mass of DBU ($M_{\text{DBU}} = 152.24 \text{ g mol}^{-1}$)

 $M_{\rm DMSO}$: molar mass of DMSO ($M_{\rm DMSO} = 78.13 \,\mathrm{g \, mol^{-1}}$)

 I_{DBU} : integral of the DBU signal (1.73–1.62 ppm) in the ¹H NMR spectrum

 I_{DMSO} : integral of the DMSO signal (2.70–2.30 ppm) in the ¹H NMR spectrum

 $N_{\rm H,DBU}$: number of protons expected for the DBU signal (1.73–1.62 ppm) in the ¹H NMR spectrum

 $N_{\rm H,DMSO}$: number of protons expected for the DMSO signal (2.70–2.30 ppm) in the ¹H NMR spectrum

E-Factor calculation

$$E \operatorname{Factor} = \frac{m_{\operatorname{Educts}} + m_{\operatorname{Solvent losses}} - m_{\operatorname{Product}}}{m_{\operatorname{Product}}}$$
(9)

 m_{Educts} : mass of the educts

 $m_{\text{Solvent losses}}$: mass of solvent losses (including *i*PrOH, DMSO and DBU) m_{Product} : mass of the desired product

3 ³¹P NMR spectra of the phosphorylated CAs

Low residual amounts of MeOH did not evaporate from CA-1 under high vacuum (0.02 mbar) and interfered with the signal for DS_{31P} determination. Peak deconvolution was used to correct the determined integral. The integration ratio using the data from peak deconvolution was calculated: $I_{R,CA-1} = 12.86$.



180 178 176 174 172 170 168 166 164 162 160 158 156 154 152 150 148 146 144 142 140 138 136 134 132 130 128 126 δ / ppm

Figure S1 ³¹P NMR (CDCl₃) spectrum of the phosphorylated CA-1. Peak deconvolution: blue lines in expanded view (calculated: $DS_{31P}=1.04$).

Table S1 Calculated area of the peaks in the ³¹P NMR spectrum by peak deconvolution for sample CA-1.

Peak	δ / ppm	Area / arb. unit
1	151.31	63598710.84
2	147.59	4674350.87
3	147.26	822819323.69



180 178 176 174 172 170 168 166 164 162 160 158 156 154 152 150 148 146 144 142 140 138 136 134 132 130 128 126 δ/ppm

Figure S2 ³¹P NMR (CDCl₃) spectrum of the phosphorylated CA-2 (calculated: DS_{31P}=1.47).



180 178 176 174 172 170 168 166 164 162 160 158 156 154 152 150 148 146 144 142 140 138 136 134 132 130 128 126 δ/ppm

Figure S3 31 P NMR (CDCl₃) spectrum of the phosphorylated CA-3 (calculated: DS_{31P}=1.73).



180 178 176 174 172 170 168 166 164 162 160 158 156 154 152 150 148 146 144 142 140 138 136 134 132 130 128 126 : δ / ppm

Figure S4 ³¹P NMR (CDCl₃) spectrum of the phosphorylated CA-4 (calculated: DS_{31P}=2.18).



180 178 176 174 172 170 168 166 164 162 160 158 156 154 152 150 148 146 144 142 140 138 136 134 132 130 128 126 δ/ppm

Figure S5 ³¹P NMR (CDCl₃) spectrum of the phosphorylated CA-5 (calculated: DS_{31P}=2.50).



180 178 176 174 172 170 168 166 164 162 160 158 156 154 152 150 148 146 144 142 140 138 136 134 132 130 128 126 δ/ppm

Figure S6 ³¹P NMR (CDCl₃) spectrum of the phosphorylated CA-6 (calculated: $DS_{31P}=2.52$).



Figure S7 ³¹P NMR (CDCl₃) spectrum of the phosphorylated CA-7 (calculated: DS_{31P}=2.97).



180 178 176 174 172 170 168 166 164 162 160 158 156 154 152 150 148 146 144 142 140 138 136 134 132 130 128 126 δ/ppm

Figure S8 ³¹P NMR (CDCl₃) spectrum of the phosphorylated CA-FP (calculated: $DS_{31P}=2.44$).



180 178 176 174 172 170 168 166 164 162 160 158 156 154 152 150 148 146 144 142 140 138 136 134 132 130 128 126 δ/ppm

Figure S9 ³¹P NMR (CDCl₃) spectrum of the phosphorylated CA-HET (calculated: $DS_{31P}=2.96$).



180 178 176 174 172 170 168 166 164 162 160 158 156 154 152 150 148 146 144 142 140 138 136 134 132 130 128 126 δ/ppm

Figure S10 ³¹P NMR (CDCl₃) spectrum of the phosphorylated CA-REC (calculated: $DS_{31P}=2.32$).



180 178 176 174 172 170 168 166 164 162 160 158 156 154 152 150 148 146 144 142 140 138 136 134 132 130 128 126 δ/ppm

Figure S11 ³¹P NMR (CDCl₃) spectrum of the phosphorylated CA-REC2 (calculated: $DS_{31P}=2.42$).

4 ¹H NMR spectra of the prepared CAs

To all ¹H NMR spectra of CAs, which were measured in DMSO- d_6 , trifluoroacetic acid (TFA) was added to shift the water signal downfield out of the relevant area of the AGU signals (3.0–5.5 ppm).



Figure S12 ¹H NMR (DMSO- d_6 + TFA) spectrum of CA-1.

CA-1: Yield: 91% ATR-IR (cm⁻¹): 3032–3675 ν (O-H), 2819–2991 ν (C-H), 1728 ν (C=O), 1370 methyl ν (C-H), 1229 ester ν (C-O), 1023 AGU ν (C-O). ¹H NMR (400 MHz, DMSO-*d*₆) $\delta_{\rm H}$ ppm: 2.78–5.30 (m, AGU, 7H), 1.69–2.27 (m, Ac_{6,2,3}, 9H).



Figure S13 ¹H NMR (DMSO- d_6 + TFA) spectrum of CA-2.

CA-2: Yield: 91% ATR-IR (cm⁻¹): 3123–3689 ν (O-H), 2828–2998 ν (C-H), 1733 ν (C=O), 1371 methyl ν (C-H), 1228 ester ν (C-O), 1032 AGU ν (C-O). ¹H NMR (400 MHz, DMSO-*d*₆ + TFA) $\delta_{\rm H}$ ppm: 2.79–5.51 (m, AGU, 7H), 1.72–2.26 (m, Ac_{6,2,3}, 9H)



Figure S14 ¹H NMR (DMSO- d_6 + TFA) spectrum of CA-3.

CA-3: Yield: 90% ATR-IR (cm⁻¹): 3104–3685 ν (O-H), 2819–3007 ν (C-H), 1736 ν (C=O), 1371 methyl ν (C-H), 1228 ester ν (C-O), 1035 AGU ν (C-O). ¹H NMR (400 MHz, DMSO-*d*₆ + TFA) $\delta_{\rm H}$ ppm: 2.73–5.39 (m, AGU, 7H), 1.50–2.21 (m, Ac_{6,2,3}, 9H)



Figure S15 ¹H NMR (DMSO- d_6 + TFA) spectrum of CA-4.

CA-4: Yield: 83% ATR-IR (cm⁻¹): 3213–3662 ν (O-H), 2813–3025 ν (C-H), 1736 ν (C=O), 1366 methyl ν (C-H), 1219 ester ν (C-O), 1028 AGU ν (C-O). ¹H NMR (400 MHz, DMSO-*d*₆ + TFA) $\delta_{\rm H}$ ppm: 2.80–5.39 (m, AGU, 7H), 1.52–2.23 (m, Ac_{6,2,3}, 9H)



Figure S16 ¹H NMR (DMSO- d_6 + TFA) spectrum of CA-5.

CA-5: Yield: 90 % ATR-IR (cm⁻¹): 3266–3661 ν (O-H), 2824–3046 ν (C-H), 1736 ν (C=O), 1369 methyl ν (C-H), 1216 ester ν (C-O), 1030 AGU ν (C-O). ¹H NMR (400 MHz, DMSO-*d*₆ + TFA) $\delta_{\rm H}$ ppm: 2.90–5.49 (m, AGU, 7H), 1.42–2.28 (m, Ac_{6,2,3}, 9H)



Figure S17 ¹H NMR (CDCl₃) spectrum of CA-6.

CA-6: Yield: 99% ATR-IR (cm⁻¹): 3421–3677 ν (O-H), 2828–3041 ν (C-H), 1741 ν (C=O), 1371 methyl ν (C-H), 1223 ester ν (C-O), 1037 AGU ν (C-O). ¹H NMR (400 MHz, CDCl₃) $\delta_{\rm H}$ ppm: 2.76–5.44 (m, AGU, 7H), 1.78–2.26 (m, Ac_{6,2,3}, 9H)



Figure S18 ¹H NMR (CDCl₃) spectrum of CA-7.

CA-7: Yield: 99% ATR-IR (cm⁻¹): 3433–3706 ν (O-H), 2836–3056 ν (C-H), 1738 ν (C=O), 1369 methyl ν (C-H), 1219 ester ν (C-O), 1037 AGU ν (C-O). ¹H NMR (400 MHz, CDCl₃) $\delta_{\rm H}$ ppm: 3.20–5.45 (m, AGU, 7H), 1.75–2.47 (m, Ac_{6,2,3}, 9H)



Figure S19 ¹H NMR (CDCl₃) spectrum of CA-FP.

CA-FP: Yield: 91% ATR-IR (cm⁻¹): 3148–3687 ν (O-H), 2802–3032 ν (C-H), 1738 ν (C=O), 1369 methyl ν (C-H), 1216 ester ν (C-O), 1032 AGU ν (C-O). ¹H NMR (400 MHz, CDCl₃) $\delta_{\rm H}$ ppm: 3.02–5.45 (m, AGU, 7H), 1.65–2.14 (m, Ac_{6,2,3}, 9H)



Figure S20 ¹H NMR (CDCl₃) spectrum of CA-HET.

CA-HET: Yield: 91% ATR-IR (cm⁻¹): 2826–3048 ν (C-H), 1738 ν (C=O), 1366 methyl ν (C-H), 1214 ester ν (C-O), 1032 AGU ν (C-O). ¹H NMR (400 MHz, CDCl₃) $\delta_{\rm H}$ ppm: 5.07 (t, H₃, 1H), 4.79 (t, H₂, 1H), 4.28–4.50 (m, H_{1,6}, 2H), 3.98–4.09 (m, H₆, 1H), 3.71 (t, H₄, 1H), 3.46–3.56 (m, H₅, 1H), 2.12 (s, Ac₆, 3H), 2.01 (s, Ac₂, 3H), 1.94 (s, Ac₃, 3H).



Figure S21 ¹H NMR (DMSO- d_6 + TFA) spectrum of CA-REC.

CA-REC: Yield: 95% ATR-IR (cm⁻¹): 3190–3665 ν (O-H), 2809–3018 ν (C-H), 1740 ν (C=O), 1370 methyl ν (C-H), 1225 ester ν (C-O), 1034 AGU ν (C-O). ¹H NMR (400 MHz, CDCl₃) $\delta_{\rm H}$ ppm: 2.76–5.58 (m, AGU, 7H), 1.22–2.28 (m, Ac_{6,2,3}, 9H)



Figure S22 ¹H NMR (DMSO- d_6 + TFA) spectrum of CA-REC2.

CA-REC2: Yield: 99% ATR-IR (cm⁻¹): 3272–3702 ν (O-H), 2824–3040 ν (C-H), 1740 ν (C=O), 1370 methyl ν (C-H), 1227 ester ν (C-O), 1039 AGU ν (C-O). ¹H NMR (400 MHz, CDCl₃) $\delta_{\rm H}$ ppm: 2.83–5.47 (m, AGU, 7H), 1.51–2.28 (m, Ac_{6,2,3}, 9H)

5 ¹³C NMR spectra of CAs for individual DS determination

Peak deconvolution was performed using the software *MestReNova v12.0.0*, Method: quantitative GSD with 10 improvement cycles, peaks type: positive and negative, refinement level: 4 (20 fitting cycles).



Figure S23 ¹³C NMR (inverse gated decoupling, DMSO- d_6) spectrum of CA-3 with peak deconvolution: blue lines in expanded view.

Table S2 Calculated area of the carbonyl peaks via peak deconvolution for sample CA-3 $(DS_{31P} = 1.73)$.

Peak	δ / ppm	Area / arb. unit
76	170.80	186801.54
73	169.95	164767.94
72	169.57	51928.79



Figure S24 ¹³C NMR (inverse gated decoupling, DMSO- d_6) spectrum of CA-4 with peak deconvolution: blue lines in expanded view.

Table S3 Calculated area of the carbonyl peaks via peak deconvolution for sample CA-4 $(DS_{31P} = 2.18)$.					
Peak δ / ppmArea / arb. unit					
76	170.79	151430.37			
76	85063.47				
7 ₃ 169.85 226520.76					
72 169.47 105902.05					



Figure S25 ¹³C NMR (inverse gated decoupling, DMSO- d_6) spectrum of CA-5 with peak deconvolution: blue lines in expanded view.

Table S4 Calculated area of the carbonyl peaks via peak deconvolution for sample CA-5 $(DS_{31P} = 2.50)$.					
Peak δ / ppm Area / arb. unit					
76	208230.47				
7 ₃ 169.84 222476.37					
7 ₂ 169.49 156993.97					



Figure S26 13 C NMR (inverse gated decoupling, CDCl₃) spectrum of CA-7 with peak deconvolution: blue lines in expanded view.

Table S5 Calculated area of the carbonyl peaks via peak deconvolution for sample CA-7 $(DS_{31P} = 2.97)$.					
Peak δ / ppmArea / arb. unit					
7 ₆ 170.34 17208.14					
7 ₃ 169.86 16753.88					
7 ₂ 169.42 18172.41					

6 Thermal Analysis



Figure S27 TGA analysis of CA-1 to CA-7.



Figure S28 DSC analysis of CA-1 to CA-7, second heating cycle.

7 Mechanical Properties



Figure S29 Tensile strength measurements of the cellulose acetate films prepared by solvent casting.



Figure S30 Linear region of three tensile strength measurements of CA-7 with the respective linear fit through the origin.



Figure S31 Linear region of three tensile strength measurements of CA-6 with the respective linear fit through the origin.



Figure S32 Linear region of three tensile strength measurements of CA-5 with the respective linear fit through the origin.

8 SEC measurement

Further SEC analyses were performed in DMAc/LiBr on a PL-SEC 50 Plus Integrated System with a PLgel 5 μ m bead-size guard column (50 × 7.5 mm) followed by three PLgel 5 μ m Mixed C columns (300 × 7.5 mm) and a differential RI detector. *N*,*N*-dimethylacetamide (DMAc) enriched with 0.03 wt% lithium bromide (LiBr) was used as eluent at a temperature of 50 °C and a flow rate of 1.0 mL min⁻¹. The samples were prepared with a concentration between 1.0-2.0 mg mL⁻¹ and 100 μ L were injected to the system. A poly(methyl methacrylate) standard was used for extrapolation of the number average molar mass (*M*_n).



Figure S33 SEC traces (DMAc/LiBr) of cellulose acetate synthesized by the heterogeneous acetylation using acetic anhydride (CA-HET, $DS_{31P} = 2.96$) and the homogeneous route using vinyl acetate (CA-7, $DS_{31P} = 2.97$).

The signal at a retention time of 38 min for CA-7 was not observed in SEC measurements on the Tosoh EcoSEC HLC-8320 with HFIP/KTFAc as eluent. Higher average molecular weight M_n was also observed on this system for CA-7 ($M_n = 49$ kDa) compared to CA-HET ($M_n = 21$ kDa).

9 Solubility Chart

= soluble		= insoluble
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Entry	CHCl ₃	CH ₂ Cl ₂	Acetone	DMSO	H ₂ O
CA-1					
CA-2					
CA-3					
CA-4					
CA-5					
CA-6					
CA-7					
CA-FP					

10 ¹H NMR spectra "vinegar syndrome"



Figure S34 ¹H NMR (CDCl₃) spectra of CA-7 before and after 2.5 months storage in a closed screw cap vial. Calculated $DS_{1H}(CA-7) = 2.94$, after 2.5 months: $DS_{1H}(CA-7) = 2.96$.



Figure S35 ¹H NMR (CDCl₃) spectra of CA-HET before and after 2.5 months storage in a closed screw cap vial. Peak deconvolution: blue lines in expanded view. Calculated DS_{1H}(CA-HET) = 2.92, after 2.5 months: DS_{1H}(CA-HET) = 2.04 (30.1% deacetylation).

Table S6 Calculated area of the peaks by peak deconvolution for sample CA-HET in the ¹H NMR spectrum after 2.5 months storage.

Peak	δ / ppm	Area / arb. unit
86	2.13	96971684.94
a (acetic acid -CH ₃)	2.09	111740815.02
82	2.01	124179862.14
83	1.94	86141829.25

11 Deacetylation during work-up procedure



Figure S36 ¹H NMR (DMSO- d_6 + TFA) spectra of one homogeneous CA synthesis batch precipitated from iPrOH (top), EtOH (middle) and MeOH (bottom) with subsequent stirring for 30 min.

Table S7 Calculated DS_{1H} and deacetylation for a CA precipitated from three different antisolvents. No deacetylation assumed for iPrOH because no iPrOAc was found in the supernatant as shown in **Figure S37**.

Antisolvent	DS_{1H}	Deacetylation / %
iPrOH	2.43	_
EtOH	2.12	12.8
MeOH	1.73	28.8



Figure S37 ¹H NMR (CDCl₃) spectra of the supernatant of a purified CA sample ($DS_{1H} = 2.43$) in a solution of 3 Eq. DBU (per AGU) and MeOH (top), EtOH (middle), iPrOH (bottom). The samples were stirred for 12 h at r.t. The formation of methyl acetate (top) and ethyl acetate (middle) was found whereas no formation of isopropyl acetate (bottom) was observed.



Figure S38 ATR-IR spectra of a purified CA sample ($DS_{1H} = 2.43$, black) and this sample stirred in iPrOH (red), EtOH (blue), and MeOH (green) with DBU for 12 h at r.t., filtered and dried.

12 "Green" synthesis of vinyl acetate



Scheme S1 Industrial synthesis of vinyl acetate from fossil ressources (red) and possibilities from renewable ressources (green).²⁻⁶

13 Recycling



Scheme S2 Flow scheme for the homogeneous cellulose acetylation and recycling process. All recycling ratios given in % are related to the used starting material, respectively.



Figure S39 ¹H NMR (CDCl₃) spectrum of the redistilled DMSO/DBU mixture of the first cycle.

Table S8 Used and recovered substances	for the synthesis of a larg	ger batch CA (2.5 Eq VA,
60 °C, 240 min).		

Substance	Used	Recovered	Recycling ratio		
DMSO	126.68 g	121.88 (+ 1.95) ^a g	96.2 (+ 1.5) ^a %		
DBU	11.34 g	9.87 g	87.0%		
iPrOH	364.08 g	336.44 (+ 23.62) ^a g	92.4 (+ 6.5) ^a %		
VA	5.31 g	-	-		
Acetaldehyde	-	0 g	0%		
$DS_{1H} = 2.29$, $DS_{31P} = 2.32$, Yield: 94.8%, Conversion VA: 91.6%					

^a In brackets: DMSO and iPrOH in the intermediate (mixed) fraction. Composition of DMSO/iPrOH calculated from the integrals in the ¹H NMR spectrum.

25.57 g of the intermediate mixed iPrOH/DMSO fraction was recovered, composed of 23.62 g iPrOH and 1.95 g DMSO. The composition of this fraction was calculated analogously to the composition of the DMSO/DBU fraction (Eq (6)–Eq (8)).

5.09 g distillation residue remained in the flask, which is expected to mainly consist of degradation products of DBU and DMSO (loss of DMSO+DBU: 4.32 g). Side reactions of acetaldehyde or minor degradation products of cellulose are also possible.

Calculated E-factor for the whole procedure: 1.92 (23.62 g iPrOH and 1.95 g DMSO from the mixed fraction was taken into account, as the mixed fraction was added to the iPrOH fraction).



Figure S40 ¹H NMR (CDCl₃) spectrum from the redistilled DMSO/DBU mixture of the second cycle.

Table S9 Used and	ecovered substances from a second cycle for the synthesis of CA-REC2
(3.0 Eq VA, 60 °C, 1	240 min).

Substance	Used	Recovered	Recycling ratio	
DMSO	32.28 (+ 1.21) ^b g	31.41 (+ 0.76) ^a g	93.8 (+ 2.3) ^a %	
DBU	2.82 g	2.47 g	87.5%	
iPrOH	171.23 (- 1.21) ^b g	160.08 (+ 3.11) ^a g	94.2 (+ 1.8) ^a %	
VA	1.59 g	-	-	
Acetaldehyde	-	0 g	0%	
$DS_{1H} = 2.40$ Yield: 99.4% Conversion VA: 80.0%				

^a In brackets: DMSO and iPrOH in the intermediate (mixed) fraction. Composition of DMSO/iPrOH calculated from the integrals in the ¹H NMR spectrum. ^b In brackets: Corrections based on the DMSO contained in the recovered iPrOH.



Figure S41 Pie chart of the partial E-factors for the synthesis of CA in the second recycling cycle.



Figure S42 ¹H NMR (CDCl₃) spectrum of the acetaldehyde byproduct with unreacted vinyl acetate and DMSO captured in a -50 °C cold trap attached to the reaction flask.

14 References

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