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Synthesis of polysaccharide derivatives bearing pyridine *N*-oxide groups and their use as asymmetric organocatalysts

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ABSTRACT

Novel amylose and cellulose derivatives bearing pyridine *N*-oxide groups were synthesized, and their performance as asymmetric organocatalysts was investigated. The amylose derivatives bearing 3-pyridyl *N*-oxide groups enantioselectively catalyzed the allylation of benzaldehyde by allyltrichlorosilane with different enantiomeric excesses (ee; 13–32% ee), depending on the degree of substitution of the pyridine *N*-oxide. In contrast, the corresponding cellulose derivatives showed the opposite enantioselectivity with much lower ee (2–11% ee). These results suggest that the higher-order structures of polysaccharides play an important role in the enantioselective allylation of benzaldehyde.

1. Introduction

Materials development using environmentally friendly and sustainable resources has been attracting considerable attention in many fields. Polysaccharides such as amylose and cellulose are considered strong candidates in this regard because they are the most abundant and renewable resources on the Earth. In addition, native polysaccharides are optically active and can discriminate among enantiomers, although their recognition abilities are not sufficient for practical purposes [1,2]. Polysaccharides have hydroxy groups and can be easily derivatized by reactions with suitable reagents to afford benzoate and phenylcarbamate derivatives [3–5]. These polysaccharide derivatives are known to show high chiral recognition abilities for many racemates when they are used as chiral stationary phases (CSPs) for high-performance liquid chromatography (HPLC) [6–10]. At present, nearly 90% of chiral compounds can be successfully separated using polysaccharide-based CSPs [11]. In contrast to the successful use of polysaccharide derivatives as CSPs for enantioseparation, their use as chiral catalysts for asymmetric reactions has been limited to reports from a few research groups [12–16].

Interest in asymmetric organocatalysts is growing because of their advantages over metal-based catalysts, including their cost effectiveness, stability in air, and low environmental toxicity [17–19]. Amine *N*-oxides are known to be promising organocatalysts with Lewis-base properties [20–25]. To date, a wide variety of chiral amine *N*-oxides have been successfully used as chiral organocatalysts in asymmetric reactions such as the allylation of aldehydes [26–30], cyanosilylation of carbonyl and imine compounds [31], aldol-type reactions [32,33], and desymmetrization of *meso*-epoxides [34,35]. However, to the best of our knowledge, amylose and cellulose have never been used as chiral scaffolds for asymmetric organocatalysts [13,36]. In the present study, we synthesized novel amylose and cellulose derivatives bearing pyridine *N*-oxide groups (**1**–**3** in Fig. 1) and investigated their abilities as asymmetric organocatalysts for the allylation of benzaldehyde **4** using allyltrichlorosilane **5**.

2. Experimental

2.1. Materials

Amylose (DP∼300) and Chiralcel OD-H (25 × 0.46 cm ID) were kindly supplied by Daicel Chemical Industries (Tokyo, Japan). Cellulose (Avicel, DP∼200) was purchased from Merck (Darmstadt, Germany). *p*-Toluoyl chloride and isonicotinic acid *N*-oxide were from Tokyo Kasei (Tokyo, Japan). Allyltrichlorosilane was obtained from Aldrich (Milwaukee, WI, USA). Benzaldehyde, nicotinic acid *N*-oxide, tetrabutylammonium iodide, thionyl chloride, and *N*-ethyldiisopropylamine were purchased from Wako (Osaka, Japan). Pyridine and dichloromethane were purchased from Kanto (Tokyo, Japan) as anhydrous solvents. Acid chlorides of nicotinic acid *N*-oxide and isonicotinic acid *N*-oxide were prepared from the

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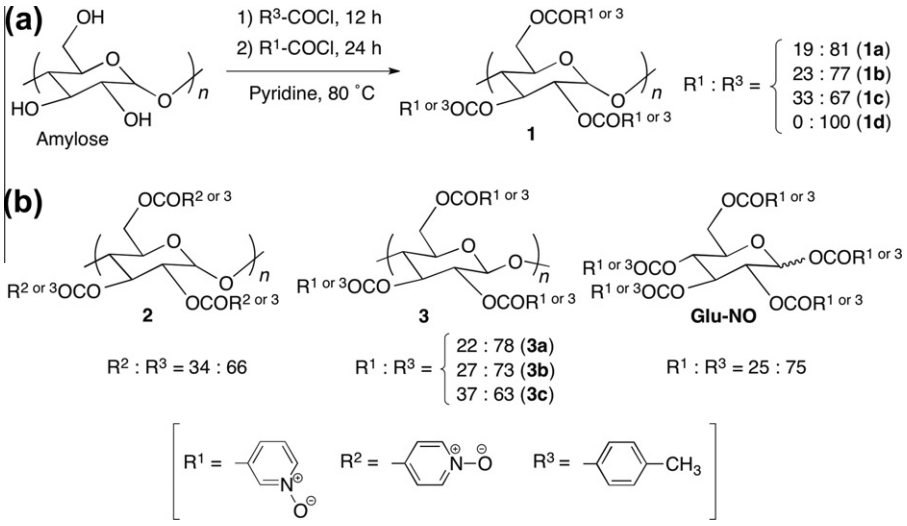

Fig. 1. Synthesis of amylose (**1**) derivatives bearing 3-pyridyl *N*-oxides (a) and structures of amylose (**2**), cellulose (**3**), and glucose (**Glu–NO**) derivatives bearing pyridine *N*-oxide groups (b).

corresponding carboxylic acid through a reaction with thionyl chloride at 80 °C.

2.2. Synthesis of polysaccharide derivatives ***1–3*** *and glucose derivative* ***Glu–NO***

2.2.1. Synthesis of ***1b***

Amylose (DP∼300, 0.50 g, 3.09 mmol) was first dispersed in pyridine (15 ml) and was allowed to react with *p*-toluoyl chloride (0.93 ml, 7.04 mmol) for 20 h at 80 °C. Then, the remaining hydroxy groups were treated with an excess of the acid chloride of nicotinic acid *N*-oxide (1.11 g, 7.05 mmol) for 28 h at 80 °C. After the reaction, the target amylose derivative **1b** (1.14 g, 71%) was obtained by the usual work-up. ^{1}H NMR (DMSO-d_6, 80 °C, 500 MHz) δ 1.8–2.4 (6.9H, br, Ph-CH_3), 3.7–6.2 (7H, br, glucose protons), 6.7–9.6 (12H, br, aromatic) ppm. IR (KBr): 1719 cm^{-1} ($\nu_{C=O}$). Elemental analysis calcd. (%) for $C_{28.62}H_{25.93}N_{0.69}O_{8.69}\cdot 0.7H_2O$: C, 64.71; H, 5.19; N, 1.82. Found: C, 64.78; H, 5.11; N, 1.88.

2.2.2. Synthesis of ***1a***

This compound was synthesized by a procedure similar to that of **1b**. ^{1}H NMR ($CDCl_3$, 55 °C, 500 MHz) δ 1.8–3.2 (8.3H, br, Ph-CH_3), 3.5–6.5 (7H, br, glucose protons), 6.8–8.9 (12H, aromatic) ppm. IR (KBr): 1725 cm^{-1} ($\nu_{C=O}$). Elemental analysis calcd. (%) for $C_{28.85}H_{26.27}N_{0.58}O_{8.58}\cdot 0.9H_2O$: C, 64.86; H, 5.30; N, 1.49. Found: C, 64.72; H, 5.16; N, 1.53.

2.2.3. Synthesis of ***1c***

This compound was synthesized by a procedure similar to that of **1b**. ^{1}H NMR (DMSO-d_6, 80 °C, 500 MHz) δ 1.8–2.5 (6.0H, br, Ph-CH_3), 3.5–5.8 (7H, br, glucose protons), 6.8–8.9 (12H, aromatic) ppm. IR (KBr): 1719 cm^{-1} ($\nu_{C=O}$). Elemental analysis calcd. (%) for $C_{28.02}H_{25.03}N_{0.99}O_{8.99}\cdot 0.6H_2O$: C: 63.46; H: 4.99; N: 2.61. Found: C, 63.39; H, 5.07; N, 2.65.

2.2.4. Synthesis of ***2***

This compound was synthesized by a procedure similar to that of **1b**. ^{1}H NMR (DMSO-d_6, 80 °C, 500 MHz) δ 1.8–2.4 (6.9H, br, Ph-CH_3), 3.8–6.0 (7H, br, glucose protons), 6.7–9.5 (12H, br, aromatic) ppm. IR (KBr): 1723 cm^{-1} ($\nu_{C=O}$). Elemental analysis calcd. (%) for $C_{27.93}H_{24.90}N_{1.03}O_{9.03}\cdot 0.8H_2O$: C, 62.89; H, 5.01; N, 2.68. Found: C, 62.81; H, 5.03; N, 2.76.

2.2.5. Synthesis of ***3a***

This compound was synthesized by a procedure similar to that of **1b**. ^{1}H NMR (DMSO-d_6, 80 °C, 500 MHz) δ 1.8–2.7 (9.8H, br, Ph-CH_3), 2.8–5.7 (7H, br, glucose protons), 6.5–9.0 (12H, br, aromatic) ppm. IR (KBr): 1727 cm^{-1} ($\nu_{C=O}$). Elemental analysis calcd. (%) for $C_{28.69}H_{26.04}N_{0.65}O_{8.65}\cdot 0.2H_2O$: C, 65.98; H, 5.10; N, 1.77. Found: C, 66.00; H, 5.09; N, 1.76.

2.2.6. Synthesis of ***3b***

This compound was synthesized by a procedure similar to that of **1b**. ^{1}H NMR ($CDCl_3$, 55 °C, 500 MHz) δ 1.9–2.6 (8.3H, br, Ph-CH_3), 2.8–5.6 (7H, br, glucose protons), 6.5–8.9 (12H, aromatic) ppm. IR (KBr): 1729 cm^{-1} ($\nu_{C=O}$). Elemental analysis calcd. (%) for $C_{28.40}H_{25.60}N_{0.80}O_{8.80}\cdot 0.3H_2O$: C, 65.01; H, 5.03; N, 2.16. Found: C, 64.97; H, 5.14; N, 2.15.

2.2.7. Synthesis of ***3c***

This compound was synthesized by a procedure similar to that of **1b**. ^{1}H NMR (DMSO-d_6, 80 °C, 500 MHz) δ 1.8–2.5 (7.5H, br, Ph-CH_3), 3.5–6.1 (7H, br, glucose protons), 6.6–9.9 (12H, aromatic) ppm. IR (KBr): 1731 cm^{-1} ($\nu_{C=O}$). Elemental analysis calcd. (%) for $C_{27.75}H_{24.62}N_{1.13}O_{9.13}\cdot 1.1H_2O$: C, 61.83; H, 5.02; N, 2.88. Found: C, 61.75; H, 4.97; N, 2.99.

2.2.8. Synthesis of ***Glu–NO***

This compound was synthesized by a procedure similar to that of **1b**. ^{1}H NMR ($CDCl_3$, 55 °C, 500 MHz) δ 2.1–2.5 (11.3H, br, Ph-CH_3), 4.3–4.8 and 5.6–6.4 (7H, br, glucose protons), 7.0–8.7 (20H, aromatic) ppm. IR (KBr): 1719 cm^{-1} ($\nu_{C=O}$). Elemental analysis calcd. (%) for $C_{43.47}H_{38.21}N_{1.27}O_{12.27}\cdot 0.3H_2O$: C, 66.93; H, 5.01; N, 2.28. Found: C, 66.94; H, 5.07; N, 2.29.

2.3. Allylation of benzaldehyde using allyltrichlorosilane catalyzed by polysaccharide derivatives bearing pyridine N-oxide groups

Tetrabutylammonium iodide (443 mg, 1.2 mmol), catalyst (0.1 mmol based on pyridine *N*-oxide), allyltrichlorosilane (0.17 ml, 1.2 mmol), and *N*-ethyldiisopropylamine (0.83 ml, 5.0 mmol) were dissolved in dichloromethane (2.0 ml) and cooled to 0 °C. Benzaldehyde (0.10 ml, 1.0 mmol) was added, and the resulting mixture was stirred for 48 h at 0 °C. The reaction was quenched by the addition of aqueous saturated $NaHCO_3$, and the reaction system was poured

into methanol to remove the catalyst. The supernatant was concentrated and extracted with ethyl acetate (3 × 10 ml). The combined organic layer was washed with brine (10 ml), dried over Na_2SO_4, and evaporated under reduced pressure. The crude product was purified by silica gel chromatography with hexane–ethyl acetate (20/1, v/v) as the eluent. The enantiomeric excess (ee) of the isolated product **6** was determined by chiral HPLC analysis on Daicel Chiralcel OD-H (hexane-2-propanol (99/1, v/v); flow rate 0.8 ml/min; temperature 20 °C; λ 220 nm): t_R = 27.2 min, t_S = 34.4 min. ^{1}H NMR ($CDCl_3$, rt, 500 MHz) δ 2.10 (s, 1H), 2.5–2.6 (m, 2H), 4.7–4.8 (m, 1H), 5.1–5.2 (m, 2H), 5.7–5.9 (m, 1H), 7.2–7.4 (m, 5H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$, rt): δ 43.8, 73.4, 118.3, 125.9, 127.6, 128.4, 134.6, 144.0 ppm. Elemental analysis calcd. (%) for $C_{10}H_{12}O$: C, 81.04; H, 8.16. Found: C, 79.99; H, 8.34.

2.4. Instruments

The ^{1}H NMR (500 MHz) spectra were taken in $CDCl_3$ at 55 °C and DMSO-d_6 at 80 °C using a JEOL ECA-500 spectrometer (JEOL, Tokyo, Japan). IR spectra were obtained using a JASCO FT-IR-620 spectrometer as a KBr pellet. Ultraviolet (UV) and circular dichroism (CD) spectra were measured in chloroform in a 0.1 mm quartz cell using a JASCO Ubset-55 and a JASCO J-720 spectrometer, respectively. Chromatographic experiments were performed using a JASCO PU-980 Intelligent HPLC pump equipped with UV (JASCO 970-UV) and polarimetric (JASCO OR-990) detectors (JASCO, Tokyo, Japan) at 20 °C. A solution of a chiral compound was injected into the chromatographic system by a Rheodyne Medel 7125 injector (Rheodyne, Rohnert Park, CA, USA).

3. Results and discussion

3.1. Synthesis of polysaccharide derivatives *1*–*3*

The amylose derivatives **1a**–**c** bearing 3-pyridyl *N*-oxide groups at the 2, 3, and 6-positions of the glucose units were synthesized by the sequential additions of *p*-toluoyl chloride and the acid chloride of nicotinic acid *N*-oxide, as shown in Fig. 1a. The content of the pyridine *N*-oxide groups could be controlled by the amount of *p*-toluoyl chloride that was used in the initial step. Derivative **1d**, without pyridine *N*-oxide groups, was also prepared for comparison [37]. The introduction of the pyridine *N*-oxide groups was confirmed by ^{1}H NMR and elemental analysis of the products. In addition, the corresponding cellulose derivatives **3a**–**c** and an amylose derivative **2** bearing 4-pyridyl *N*-oxide groups were synthesized in a similar way (Fig. 1b). The D-glucose derivative **Glu–NO** was also prepared as a model compound consisting of a monosaccharide.

3.2. Circular dichroism spectra of polysaccharide derivatives *1* and *3*

The CD and UV spectra of amylose (**1a** and **1b**) and cellulose (**3a** and **3b**) derivatives bearing 3-pyridyl *N*-oxide groups are shown in Fig. 2. Because **1c** and **3c** were insoluble in chloroform, their spectra could not be measured. The CD spectral patterns of the amylose derivatives were similar to each other, although the wavelengths and the peak intensities depend on the degree of substitution. The cellulose derivatives showed opposite Cotton effects to those of the amylose derivatives in the absorption region because of aromatic chromophores. This result indicates that the pendant 3-pyridyl *N*-oxide as well as 4-methylbenzoate of both **1** and **3** seems to be arranged in opposite chiral environments, probably because of the higher-order structures of the polysaccharides.

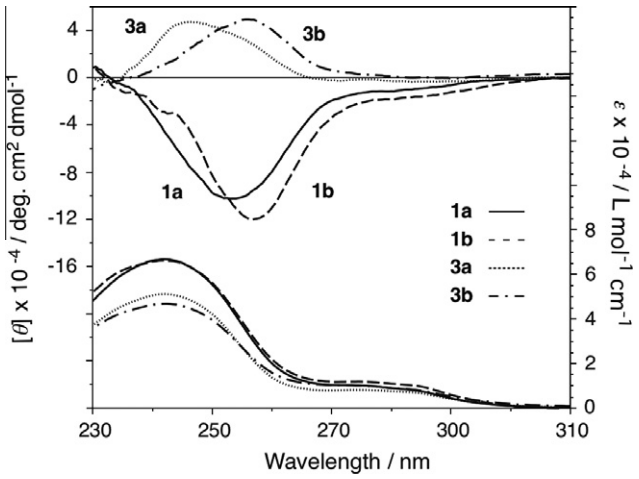

Fig. 2. CD (upper) and UV (lower) spectra of amylose (**1a** and **1b**) and cellulose (**3a** and **3b**) derivatives in chloroform at room temperature. (Cell length: 0.1 mm, concentration: 2.0×10^{-3} M based on glucose units).

3.3. Enantioselective allylation catalyzed by amylose derivatives *1* bearing pyridine *N*-oxide groups

The pyridine *N*-oxide-bound amylose derivatives **1a**–**c** were used as organocatalysts for the asymmetric addition of allyltrichlorosilane **5** to benzaldehyde **4**; this allylation is often used as a test reaction for new chiral nucleophilic catalysts. As described in the literature [26], **4** (1.0 mmol) was reacted with **5** (1.2 mmol) in the presence of the amylose derivatives **1a**–**c** (0.1 mmol based on pyridine *N*-oxide), $Bu_4N^+I^-$ (1.2 mmol), and *N*-ethyldiisopropylamine (^{i}Pr$_2$NEt) (5.0 mmol) in dichloromethane (CH_2Cl_2) (2.0 mL) for 48 h at 0 °C (Fig. 3). After purification of the crude product by silica-gel chromatography with hexane/ethyl acetate (20/1, v/v) as the eluent, the ee of the isolated 1-phenyl-3-buten-1-ol **6** was determined by chiral HPLC analysis on Daicel Chiralcel OD-H.

Table 1 summarizes the results of the enantioselective allylation of **4** with **5** using the amylose derivatives **1a**–**c** as catalysts. The amylose derivatives **1a**–**c** (entries 1–3) bearing 3-pyridyl *N*-oxide groups catalyzed the reaction to give (*R*)-**6** in moderate yields (47–62%) with different ee values (13–32% ee), depending on the degree of substitution of the pyridine *N*-oxide. Conversely, **1d** (entry 4), without pyridine *N*-oxide groups, showed no catalytic activity in this reaction, as expected. When the amount of pyridine *N*-oxide was increased from 19% (**1a**, entry 1) to 23% (**1b**, entry 2), both the yield and enantioselectivity improved from 47% and 13% ee to 62% and 32% ee, respectively. However, a further increase in the amount of 3-pyridyl *N*-oxide caused the enantioselectivity to deteriorate (24% ee; **1c**, entry 3). On the other hand, when the D-glucose-based catalyst (**Glu–NO**) was used for the allylation reaction, instead of the amylose derivatives (entry 9), a poor and opposite enantioselectivity (8% ee, *S* configuration) was observed for **6**. These results indicate that the enantioselectivity obtained from the amylose-based catalysts (**1**) was attributable to higher-order structures, such as one-handed helical structures, and that a slight difference in their higher-order structures could influence the enantioselectivity in this allylation reaction.

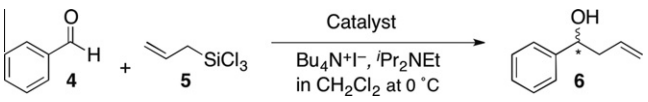

Fig. 3. Enantioselective allylation of benzaldehyde (**4**) using allyltrichlorosilane (**5**) catalyzed by polysaccharide (**1**–**3**) and glucose (**Glu–NO**) derivatives.

Table 1
Enantioselective allylation catalyzed by polysaccharide (**1**–**3**) and glucose (**Glu–NO**) derivatives.[a]

Entry	Catalyst	DS (%)[b]	Yield (%)[c]	ee (%)[d]	Configuration[e]
1	**1a**	19	47	13	*R*
2	**1b**	23	62	32	*R*
3	**1c**	33	59	24	*R*
4	**1d**	0	0	–	–
5	**2**	34	37	<1	n.d.
6	**3a**	22	70	11	*S*
7	**3b**	27	64	2	*S*
8	**3c**	37	59	8	*S*
9	**Glu–NO**	25	55	8	*S*

[a] Reactions were carried out at 1 mmol scale in CH_2C1_2 (2 mL) with allyltrichlorosilane (1.2 equiv.), *N*-oxide (10 mol%), $Bu_4N^+I^-$ (1.2 equiv.), and $_iPr_2NEt$ (5.0 equiv.) for 48 h at 0 °C.
[b] Degree of substitution of pyridine *N*-oxide determined by elemental analysis.
[c] Yield of the isolated product.
[d] Determined by chiral HPLC on Chiralcel OD-H (hexane-2-propanol (99/1, v/v)).
[e] Absolute configuration of the predominant isomer assigned by the comparison of the sign of optical rotation.

3.4. Influence of the position of the N-oxide group on enantioselective allylation

In contrast to the 3-pyridyl *N*-oxide-bound amylose derivative **1**, the corresponding 4-pyridyl *N*-oxide-bound amylose derivative **2** showed almost no enantioselectivity (entry 5). It seems likely that the catalytically active *N*-oxide groups in **2** may not be situated in a suitable chiral environment for chirality induction in the allylation of benzaldehyde because the groups are located away from the polysaccharide backbone. These results suggest that to achieve high enantioselectivity, the reaction should occur in the chiral groove formed by the higher-order structures of the amylose derivatives.

*3.5. Enantioselective allylation catalyzed by cellulose derivatives **3** bearing pyridine N-oxide groups*

The cellulose derivatives **3a**–**c** also catalyzed the allylation reaction in moderate yields (59–70%; entries 6–8), but the enantioselectivities were much lower than those obtained with the corresponding amylose derivatives **1a**–**c**. Interestingly, the absolute configurations of the predominant enantiomers obtained with the amylose-based catalysts were opposite to those obtained with the cellulose-based catalysts. As described in the CD spectral analysis, 3-pyridyl *N*-oxides in the amylose and cellulose derivatives seem to be in opposite chiral environments because of their higher-order structures, and the opposite environments may result in the different enantioselectivities in the allylation reactions.

4. Conclusions

We present a facile synthesis of amylose and cellulose derivatives bearing various amounts of pyridine *N*-oxide groups, and their use as organocatalysts for the enantioselective allylation of benzaldehyde with allyltrichlorosilane. The present results indicate that the higher-order structures that are inherent in each polysaccharide derivative significantly affect the efficiency of chiral induction in asymmetric reactions. We anticipate that a more efficient polysaccharide-based asymmetric organocatalyst may be created by an appropriate combination of polysaccharides and catalytically active groups as a result of the synergistic effects of the chiralities of the constituent units and higher-order structures, such as one-handed helical structures.

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