Data Article

Solid-state relaxation NMR dataset for a water-soluble \( \beta-\{1 \rightarrow 3, 1 \rightarrow 6\}\)-glucan from \textit{Aureobasidium pullulans} and schizophyllan from \textit{Schizophyllum commune}

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Abstract

We report the solid-state nuclear magnetic resonance (NMR) relaxation dataset for a triple helix and a random structure of water-soluble \textit{Aureobasidium pullulans} \( \beta-(1 \rightarrow 3, 1 \rightarrow 6)\)-o-glucan (APG) and those of schizophyllan from \textit{Schizophyllum commune} (SPG), obtained by the Bruker BioSpin 500 MHz NMR spectrometer. These data include solid-state proton spin-lattice relaxation in the rotating frame (\(T_1R\)) and \(^{13}\)C spin-lattice relaxation (\(T_{1C}\)) of these two \( \beta-\{1 \rightarrow 3, 1 \rightarrow 6\}\)-glucans, which are related to the subject of article in \textit{International Journal of Biological Macromolecules}, entitled “Characterization of the secondary structure and order –disorder transition of a \( \beta-\{1 \rightarrow 3, 1 \rightarrow 6\}\)-glucan from \textit{Aureobasidium pullulans}” [1]. Data can help to investigate the structural characterization of the structural polysaccharides.

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DOI of original article: https://doi.org/10.1016/j.ijbiomac.2019.11.018.

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1. Data

The presented data include the $T_{1\text{pH}}$ and $T_{1\text{C}}$ relaxation NMR data of a triple helix and a random structure of APG and those of SPG. $^{13}$C NMR spectra of triple helix and a random structure of APG and those of SPG acquired by inserting $\delta^1$H spin-lock times that range from 0 to 15 ms during the $T_{1\text{pH}}$ experiments (Figs. S1–S4), and the $^{13}$C peaks were integrated for the following regions: C1 (110–96 ppm), C3 of the (1→3)-β-glucosyl main-chain (96–83 ppm), C6 (66–56 ppm), and other carbon resonances (83–66 ppm). The resulting integration values for each region as functions of $\delta^1$H spin-lock time are summarized in Table 1, which were fitted to the following mono-exponential function:

$$I_t = I_0 \exp(-t / T_{1\text{pH}})$$

where $I_t$ is the measured integral value at $\delta^1$H spin-lock time $t$ and $I_0$ is the initial intensity ($t = 0$) of the $^{13}$C magnetization. The $T_{1\text{pH}}$ values for the four $^{13}$C region of each sample could be determined by the fitting curves [1].

A series of $^{13}$C NMR spectra for a triple helix and a random structure of APG and those of SPG recorded with 10 relaxation delays that range from 0 to 60 s during the $T_{1\text{C}}$ experiments (Figs. S5–S8). As described for the $T_{1\text{pH}}$ experiments, the four spectral regions were integrated (Table 2), and the
resulting integration values for each region as functions of $^{13}\text{C}$ relaxation delay were fitted to the following mono-exponential function:

$$I_t = I_0 \exp(-t / T_{1C})$$

where $I_t$ and $I_0$ are defined as for Eq. (1). The $T_{1C}$ values for the four $^{13}\text{C}$ regions for each sample could be determined by the fitting curves [1].

2. Experimental design, materials, and methods

APG was kindly provided from Itochu Sugar Co. (Japan), which was prepared according to a previously reported method [2–4]. SPG was purchased from InvivoGen (USA). Triple helical and single random coil structures of APG were prepared by dissolving 250 mg of APG in 50 mL of deionized water and DMSO, respectively, at 298 K for 3 d followed by lyophilization. In a method similar to that used to prepare the triple helical and single random coil structures of APG, lyophilization of SPG dissolved in water or DMSO for 3 d provided the triple helical and single random coil structures of SPG, respectively [1].

Solid-state $T_{1\text{ph}}$ and $T_{1C}$ experiments were performed at 298 K using a Bruker AVIII500 spectrometer (Bruker BioSpin GmbH, Germany) equipped with a 4 mm dual-tuned MAS probe according to methods previously reported [5,6]. To determine $T_{1\text{ph}}$ values, Cross-polarization (CP)/MAS $^{13}\text{C}$ NMR spectra were recorded by inserting $^1\text{H}$ spin-lock times of 0.5, 1, 2, 3, 4, 8, 10, and 15 ms prior to CP, and MAS frequency,

| Sample                  | Spin-lock time/ms | C1     | C3 (main-chain) | C2,3,4,5 | C6     |
|-------------------------|-------------------|--------|-----------------|----------|--------|
| APG (triple helix)      | 0                 | 0.163  | 0.112           | 0.640    | 0.085  |
|                         | 0.5               | 0.143  | 0.097           | 0.574    | 0.075  |
|                         | 1                 | 0.128  | 0.089           | 0.512    | 0.068  |
|                         | 2                 | 0.104  | 0.072           | 0.412    | 0.053  |
|                         | 3                 | 0.080  | 0.058           | 0.330    | 0.042  |
|                         | 4                 | 0.065  | 0.043           | 0.263    | 0.035  |
|                         | 8                 | 0.023  | 0.016           | 0.111    | 0.012  |
|                         | 10                | 0.013  | 0.007           | 0.074    | 0.006  |
|                         | 15                | 0.002  | −0.002          | 0.022    | 0.004  |
| APG (random structure)  | 0                 | 0.165  | 0.086           | 0.646    | 0.103  |
|                         | 0.5               | 0.150  | 0.074           | 0.585    | 0.091  |
|                         | 1                 | 0.129  | 0.069           | 0.519    | 0.082  |
|                         | 2                 | 0.105  | 0.051           | 0.412    | 0.068  |
|                         | 3                 | 0.084  | 0.041           | 0.336    | 0.055  |
|                         | 4                 | 0.068  | 0.027           | 0.271    | 0.040  |
|                         | 8                 | 0.025  | 0.012           | 0.117    | 0.018  |
|                         | 10                | 0.021  | 0.007           | 0.074    | 0.009  |
|                         | 15                | 0.005  | 0.004           | 0.032    | 0.000  |
| SPG (triple helix)      | 0                 | 0.173  | 0.124           | 0.599    | 0.105  |
|                         | 0.5               | 0.153  | 0.112           | 0.544    | 0.094  |
|                         | 1                 | 0.140  | 0.098           | 0.483    | 0.081  |
|                         | 2                 | 0.110  | 0.080           | 0.389    | 0.068  |
|                         | 3                 | 0.092  | 0.071           | 0.315    | 0.056  |
|                         | 4                 | 0.074  | 0.055           | 0.257    | 0.044  |
|                         | 8                 | 0.028  | 0.022           | 0.115    | 0.017  |
|                         | 10                | 0.018  | 0.013           | 0.073    | 0.010  |
|                         | 15                | 0.005  | 0.000           | 0.028    | 0.000  |
| SPG (random structure)  | 0                 | 0.169  | 0.120           | 0.600    | 0.111  |
|                         | 0.5               | 0.146  | 0.104           | 0.526    | 0.096  |
|                         | 1                 | 0.124  | 0.087           | 0.456    | 0.084  |
|                         | 2                 | 0.095  | 0.067           | 0.345    | 0.064  |
|                         | 3                 | 0.070  | 0.047           | 0.265    | 0.049  |
|                         | 4                 | 0.053  | 0.041           | 0.207    | 0.040  |
|                         | 8                 | 0.013  | 0.010           | 0.070    | 0.010  |
|                         | 10                | 0.008  | 0.002           | 0.039    | 0.004  |
|                         | 15                | 0.001  | −0.002          | 0.007    | 0.002  |
contact time, acquisition time, and repetition time were set to 10 kHz, 2 ms, 15 ms, and 4 s, respectively. The $T_{1\rho H}$ values for the specific $^{13}$C resonance regions were integrated to obtain the $T_{1\rho H}$ curves, which were fitted to Eq. (1). The $T_{1C}$ experiments were performed using the Torchia method [7]. The spectra were recorded at relaxation delays of 0.1, 0.5, 1, 2.5, 5, 7.5, 10, 30, and 60 s, and the specific $^{13}$C resonance regions were integrated to obtain $T_{1C}$ curves, which were fitted to Eq. (2). The chemical shifts were calibrated by assigning the value of 176.03 ppm to the carbonyl carbon of the external standard D-glycine.

**Funding**

This work was, in part, supported by the Japan Society for Promotion of Science (JSPS) [grant number JP16K05802] (H.K.).

**Conflict of Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.
Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.dib.2019.104993.

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