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^{13}C-NMR Spectra of the Alditol-form Oligosaccharides having the Fundamental Structural Units of the Malvaceae Plant Mucilages and a Related Polysaccharide*

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In our previous reports, the structural features of eight mucilages from plant sources in the Malvaceae family, *i. e.*, the roots of *Abelmoschus manihot*, *Althaea officinalis*, *Abelmoschus glutinotextilis*, *Althaea rosea*, and *Abelmoschus esculentus*, the immature fruit of *Abelmoschus esculentus*, and the leaves of *Althaea officinalis* and *Althaea rosea*, have been elucidated. The main part of most of them is made up of a component unit having the structure (1→4)-[*O*-β-(D-glucopyranosyluronic acid)-(1→3)]-*O*-α-(D-galactopyranosyluronic acid)-(1→2)-*O*-α-L-rhamnopyranose in common, while Okra-mucilage F has no branch at the D-galacturonic acid residues in the main chain. Carbon-13 nuclear magnetic resonance (^{13}C-NMR) spectroscopy is a useful tool for the structural determination of carbohydrates. In this paper, the ^{13}C-NMR data for five oligosaccharides and a polysaccharide having the fundamental structural units of the above-mentioned plant mucilages are presented.

The five oligosaccharides (I to V) were obtained by partial hydrolysis of the mucilages. To avoid the complexity of the spectra based on α- and β-anomeric forms of each reducing end unit, the oligosaccharides were treated with sodium borohydride to give the corresponding alditol forms (I-ol to V-ol in Chart 1.).

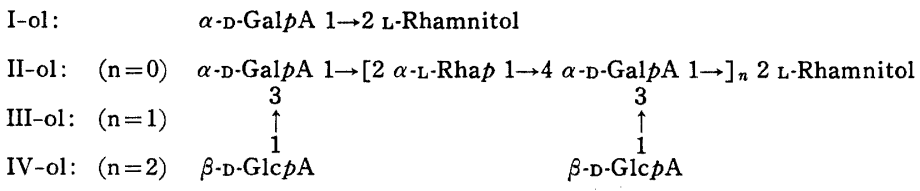

Chart 1. Structural Features of Reduced Oligosaccharides I-ol, II-ol, III-ol, IV-ol, and V-ol

The variations of chemical structure are reflected by the signals in the region of 77.82 to 106.99 ppm, which are mainly attributable to the anomeric carbon and the carbons involved in glycosidic linkages.

The C-1 signals for the glycosidic carbon of α-L-rhamnopyranose appeared at 101.19 to 101.42 ppm ; the corresponding figures were 100.09 to 100.62 ppm for α-D-galactopyranosyluronic acid and 106.39 to 106.99 ppm for β-D-glucopyranosyluronic acid. Thus, the glycosidic carbon resonances are divided into three groups.

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The downfield displacements of the C-2 signal of 2-*O*-α-(D-galactopyranosyluronic acid)-L-rhamnonitol in I-ol and II-ol were 7.22 and 7.35 ppm, compared with the C-2 signal of L-rhamnitol, and those of 2-*O*-α-(D-galactopyranosyluronic acid)-L-rhamnose units in III-ol, IV-ol and V-ol were 8.88 to 9.03 ppm, compared with α-L-rhamnopyranose. The downfield displacements of the C-3 signal of 3-*O*-β-(D-glucopyranosyluronic acid)-D-galacturonic acid units in II-ol, III-ol, IV-ol, and V-ol were 10.14 to 10.43 ppm, compared with the D-galacturonic acid unit in I-ol, and those of the C-4 signal of 4-*O*-α-L-rhamnopyranosyl-D-galacturonic acid units in III-ol, IV-ol, and V-ol were 4.41 to 5.17 ppm, compared with the D-galacturonic acid unit in II-ol.

The methyl signals (C-6) of rhamnitol and rhamnose were separately observed in the region of 21.34 to 21.37 ppm and at 19.14 ppm in I-ol, II-ol, and III-ol, and the carboxyl signals (C-6) of galacturonic acid were separately observed in the region of 174.95 to 175.72 ppm. Those of glucuronic acid were also separately observed in the region of 175.41 to 176.47 ppm in the same oligosaccharides. However, in the nonasaccharide (IV-ol) and dodecasaccharide (V-ol), some of the carbon signals having the same position numbers showed overlapping.

The ^{13}C-NMR spectrum of Althaea-mucilage OL from the leaves of *Althaea officinalis* is shown. Structural studies indicated that the mucilage is mainly composed of the fundamental trisaccharide (II) repeating unit with 1% acetyl groups and *ca.* 3% protein. The assignment of the signals was done by comparing the data with those of the oligosaccharides described above. The signals at 178.57 and 177.36 ppm are assigned to C-6 carbons of glucuronic acid and galacturonic acid residues, and the signals at 100.94 and 79.98 ppm are assigned to C-1 and C-2 carbons of α-1→2 linked L-rhamnose residues. The signals at 106.47 and 100.80 ppm are assigned to C-1 carbons of β-D-glucuronic and α-D-galacturonic acid residues, and those at 81.28 and 78.70 ppm to the C-3 and C-4 glycosyl-linked carbons in the α-D-galacturonic acid residues. In addition to the methyl signal of rhamnose at 19.30 ppm, the signals at 21.79 ppm and 176.36 ppm are attributable to the methyl and carbonyl carbons in the acetyl groups.

The results described above indicate that ^{13}C-NMR spectroscopy can provide useful information about the structures of plant mucilages in the Malvaceae family if the mucilages show good solubility.