Title	Plant mucilages. XXXIX. a representative mucilage, "hibiscus-mucilage SL" from the leaves of hibiscus syriacus
Sub Title	
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Publisher	共立薬科大学
Publication year	1986
Jtitle	共立薬科大学研究年報 (The annual report of the Kyoritsu College of Pharmacy). No.31 (1986.) ,p.39- 40
JaLC DOI	
Abstract	
Notes	抄録
Genre	Technical Report
URL	https://koara.lib.keio.ac.jp/xoonips/modules/xoonips/detail.php?koara_id=AN00062898-00000031-0039

Plant Mucilages. XXXIX. A Representative Mucilage, "Hibiscus-Mucilage SL" from the Leaves of *Hibiscus syriacus*

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We now report the isolation and structural investigation of a representative mucilage from the leaves of *Hibiscus syriacus* L., as the second example of the mucilages from plants of the genus *Hibiscus*.

The fresh leaves were homogenized and extracted with cold water. The crude mucilage was precipitated from the extract by addition of ethanol. The solution of the crude mucilage was applied to a column of DEAE-Sephadex A-25 (carbonate form). After elution with 0.2 M ammonium carbonate, the eluate with 0.5 M ammonium carbonate was dialyzed and purified by successive gel chromatography on Sephadex G-15, Sephacryl S-300 and Sephadex G-25.

The mucilage gave a single spot on zone electrophoresis with glass-fiber paper, and in addition, it gave a single peak on gel chromatography with Sepharose CL-4B. The mucilage had $[\alpha]_D^{23}+57.7°$, and its aqueous solution gave the high intrinsic viscosity value of 26.5 at 30°. Gel chromatography with standard dextrans gave a value of about 2,000,000 for the molecular weight. The name "Hibiscus-mucilage SL" is proposed for this substance.

As component sugars of the mucilage, rhamnose, galactose, galacturonic acid, and glucuronic acid were identified. Quantitative determination showed that the mucilage contained 31.6% rhamnose, 4.8% galactose, 38.0% galacturonic acid, and 19.0% glucuronic acid, and that their molar ratio was 8.0 : 1.1 : 8.0 : 4.0. Determination of protein content was carried out and a value of 1.8% was obtained.

Methylation of the original mucilage and the carboxyl-reduced derivative was performed with methylsulfinyl carbanion and methyl iodide in dimethyl sulfoxide. The fully methylated products were hydrolyzed, and the hydrolyzates were converted into the partially methylated alditol acetates.

GLC-MS revealed derivatives of 3,4-di-*O*-methyl-L-rhamnose, 3-*O*-methyl-L-rhamnopyranose, and 2,3,4,6-tetra-*O*-methyl-D-galactopyranose as the products in a molar ratio of 7.5 : 0.9 : 1.0 from the original mucilage. Alditol acetates of 3,4-di-*O*-methyl-L-rhamnopyranose, 3-*O*-methyl-L-rhamnopyranose, 2,3,4,6-tetra-*O*-methyl-D-glucopyranose, 2,3,4,6-tetra-*O*-methyl-D-galactopyranose, 2,3,6-tri-*O*-methyl-D-galactopyranose, and 2,6-di-*O*-methyl-D-galactopyranose were identified in a molar ratio of 7.4 : 0.9 : 3.5 : 0.9 : 4.6 : 4.0

* 本報告は *Chem. Pharm. Bull.*, **34**, 4133—4138 (1986) に発表.

from the carboxyl-reduced product.

The mucilage was partially hydrolyzed with dilute sulfuric acid, and then neutralized and treated with Dowex 50W (H^+). The eluate with water was applied to a column of DEAE-Sephadex A-25 (formate form). In addition to small amounts of the component monosaccharides, five oligosaccharide (I to V) were obtained by stepwise elution with dilute formic acid, then purified by rechromatography. Based on the results of component suger analysis and a comparison of their chromatographic properties, their ^{1}H-NMR spectra, and their values of specific rotation with those of authentic samples, I to V were identified.

By a comparison of the ^{13}C-NMR spectra of the original mucilage and the product obtained by the acid treatment, it can be concluded that the anomeric signal at δ 107.57, which is observed only in the former, is due to β-D-galactose residues.

Based on the accumulated evidence described above, it may be concluded that the polysaccharide moiety of the mucilage contains the units shown in Chart 1.

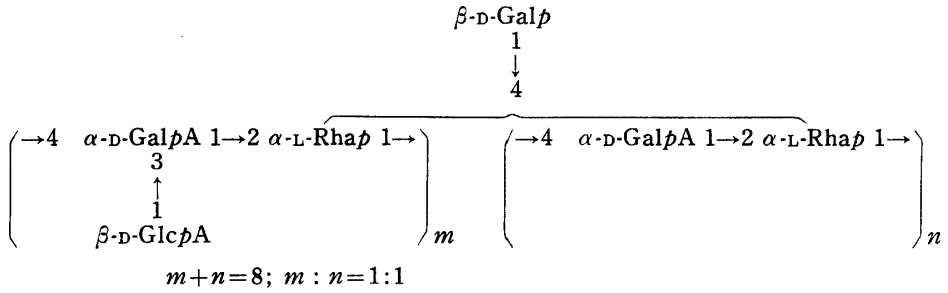

Chart 1. A Possible Structural Fragment of the Polysaccharide Moiety of Hibiscus-Mucilage SL