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Plant Mucilages. XL. A Representative Mucilage, "Hibiscus-mucilage SF," from the Flower Buds of *Hibiscus syriacus**

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The white flower bud of *Hibiscus syriacus* L. is an Oriental crude drug (Japanese name, Mokukinka) used as a demulcent and antidiarrheic. We have now isolated a representative mucilage from the white flower buds of this plant. Its properties and structural features are reported here.

The fresh buds were homogenized and extracted with cold water. The crude mucilage was precipitated from the extract by addition of ethanol, then dissolved again in water. The solution was treated with sodium lauryl sulfate and sodium chloride. After centrifugation, the supernatant obtained was poured into acetone. The precipitate obtained was dissolved in water and the solution was dialyzed and purified by gel chromatography with Sephadex G-25, then the eluate was lyophilized.

The mucilage gave a single spot on zone electrophoresis with glass-fiber paper, and it gave a single peak on gel chromatography with Sephacryl S-400. Further, it gave a clear band on polyacrylamide gel disk electrophoresis. Both the periodate-Schiff reagent and the Coomassie blue reagent revealed the band in the same position. The mucilage had $[\alpha]_D^{24} + 30.0^\circ$ (0.1% HN_4OH , $c=0.1$), and its aqueous solution gave the high intrinsic viscosity value of 26.0 at 30°C . Gel chromatography with standard dextrans gave a value of about 1050000 for the molecular weight. The name "Hibiscus-mucilage SF" is proposed for this substance.

As component sugars of the mucilage, rhamnose, galactose, galacturonic acid, and glucuronic acid were identified. The $^1\text{H-NMR}$ spectrum showed a signal at δ 2.13, suggesting the presence of *O*-acetyl groups. The molar ratio of rhamnose : galactose : galacturonic acid : glucuronic acid : *O*-acetyl is 36 : 36 : 33 : 22 : 36. Determination of protein content was 8.1%.

Methylation of the original mucilage and the carboxyl-reduced derivative was performed with methylsulfinyl carbanion and methyl iodide in dimethyl sulfoxide. The 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-rhamnose, 2,3,4,6-tetra-*O*-methyl-D-galactose, and 2,3,6-tri-*O*-methyl-D-galactose as the products in a molar ratio of 1 : 1 : 1 : 1 from the original mucilage. Alditol acetates of 3,4-di-*O*-methyl-L-rhamnose, 3-*O*-methyl-L-rhamnose, 2,3,4,6-tetra-*O*-methyl-D-

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glucose, 2,3,4,6-tetra-*O*-methyl-D-galactose, 2,3,6-tri-*O*-methyl-D-galactose, and 2,6-di-*O*-methyl-D-galactose were identified in a molar ratio of 18 : 18 : 22 : 18 : 29 : 22 from the carboxyl-reduced product.

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

All galactose residues were liberated from the mucilage under the conditions of partial hydrolysis described above. In conjunction with the results of methylation analysis, this finding suggests that a half of the rhamnose residues in the backbone chain possesses a 1→4 galactosyl galactose chain at position 4.

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

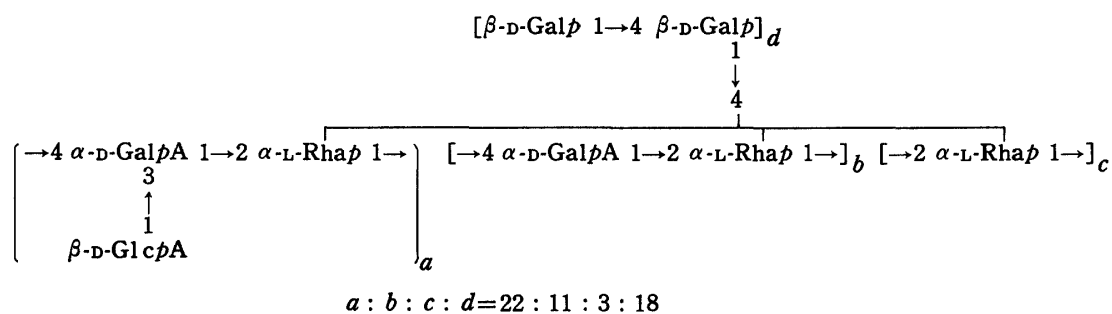


Chart 1. A Possible Structural Fragment of the Polysaccharide Moiety of Hibiscus-mucilage SF