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Author	友田, 正司(Tomoda, Masashi) 佐藤, 訓子(Sato, Noriko) 松村, 剛(Matsumura, Go)
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Plant Mucilages. XX. The Location of *O*-Acetyl Groups in Paniculatan and the Influence of Deacetylation on Its Physical Properties *

MASASHI TOMODA, NORIKO SATOH, and GŌ MATSUMURA **

友田正司, 佐藤訓子, 松村 剛

The isolation and the structural feature of the mucous polysaccharide, named paniculatan, from the inner bark of *Hydrangea paniculata* SIEB. have been reported in the previous papers of this series. The polysaccharide contains 2.0 % of *O*-acetyl groups. The present work was undertaken to elucidate the location of *O*-acetyl groups. This paper is also concerned with the influence of its deacetylation on the physical properties of the polysaccharide as a native polymer.

The polysaccharide was exhaustively treated with methyl vinyl ether in the presence of *p*-toluenesulfonic acid in dimethyl sulfoxide. After conversion of the free hydroxyl groups to 1-methoxyethyl ethers, the derivative was deacetylated, then methylated with methyl iodide and silver oxide in *N,N*-dimethylformamide. The resultant product was subjected to acid hydrolysis, and the final products were analyzed by GLC-MS after conversion into alditol acetates. 1, 2, 4, 5-Tetra-*O*-acetyl-3-mono-*O*-methyl L-rhamnitol was detected and identified. The result of GLC showed that the molar ratio of 3-mono-*O*-methyl-L-rhamnose and L-rhamnose was 1.0 : 1.4. Basing on this result, it can be concluded that approximately 40 % of L-rhamnopyranosyl residues are present as 3-*O*-acetyl derivative in the molecule of paniculatan.

Paniculatan was treated with 0.1 N sodium hydroxide for 10 min at room temperature. The solution was neutralized, dialyzed, then lyophilized.

The deacetylated paniculatan was shown to be homogeneous in both ultracentrifugal analysis and gel chromatography with Sephadex G-200. Its solubility in water increased more than two times that of paniculatan, in contrast to the case of the deacetylated lily glucomannans. Its aqueous solution gave the intrinsic viscosity of 14.2 at 30°. This value and that of the original polysaccharide correspond to a ratio of 1 : 3.8.

The measurement of osmotic pressure gave the value of 14260 as the molecular weight of the ammonium salt of the deacetylation product. The value and that of the ammonium salt of paniculatan correspond to an approximate ratio of 1 : 7.6. However, we now found that the ammonium salt of paniculatan contained only 0.72 % of *O*-acetyl groups. Thus even the treatment with very dilute ammonium hydroxide resulted in the partial deacetylation of the original polysaccharide. Therefore, the mo-

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**昭和大学薬学部。

lecular weights of the original and the deacetylated polysaccharides were reinvestigated by the measurement of sedimentation equilibrium. The results gave the values of 545900 and 41950 as the molecular weights of paniculatan and its deacetylation product, respectively. In this case the value of the deacetylation product and that of paniculatan correspond to a ratio of 1 : 13.

The deacetylation of paniculatan caused the remarkable lowering of the values in both the molecular weight and the viscosity of aqueous solution. From this fact, it is possible to derive the following two sorts of inference. First, in the case of the treatment with dilute sodium hydroxide, the possibility of the direct degradation of a polymer with alkali hydroxide is undeniable. However, as the second possibility, the result that even the treatment with very dilute ammonium hydroxide caused the release of about two thirds of acetyl groups in the native polysaccharide and the lowering of the molecular weight suggests a significant role of *O*-acetyl groups in the polysaccharide in the association of a macromolecule. Further investigation of these problems is now under progress.

It is possible to conclude that one of the causes of the considerable difference in both values of molecular weight of paniculatan by the sedimentation equilibrium method and the osmotic pressure method is due to the partial dissociation of a macromolecule owing to the release of the majority of acetyl groups. And all the results also provide the evidence that paniculatan belongs to a polydispersed polysaccharide.