

CHEMICAL CONSTITUTION OF THE GUM FROM 'BOSWELLIA SERRATA.'

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As very little is known of the chemical constitution of the gum occurring in the gum-oleo-resin of *Boswellia serrata* some exploratory experiments were made, in the course of other related investigations,¹ in the hope of throwing some light on the subject.

The work proved difficult and tedious and the results are not as complete as could be wished. As, however, it appears unlikely that the writer will be in a position to pursue the work in the near future, it has been thought desirable to place the observed facts on record, so far as they go.

The only available information with regard to the chemical constitution of *Boswellia* gum results from the work of Tschirch and Halbey,² who state, that it consists of arabic acid, bassorin and a bitter principle.

In the experiments to be described, the general method followed was that employed by O'Sullivan³ in his investigations on the constitution of gums, viz., gentle hydrolysis with acid and investigation of the acid and sugar resulting from this process.

EXPERIMENTAL.

The gum used for the experimental work was prepared from the gum-oleo-resin by thorough extraction with 90 per cent. alcohol in a Soxhlet apparatus. The gum so prepared was soluble in water to the extent of only 0.5 per cent. The ash consisted mainly of calcium carbonate and amounted to 5.2 per cent. of the dry gum.

The solubility being so low, the two methods used by O'Sullivan to determine the homogeneous character of the product, viz., fractional precipitation of the gum solution with alcohol, and measurement of the optical activity of these fractions, could not be used. An attempt to reduce the ash by adding hydrochloric acid to the gum mucilage, and subsequent dialysis of the mixture did not meet with much success; moreover, no marked reduction of nitrogen-content

¹ Fowler and Malandkar, *This Journal*, 1925, 8A, 221.

² *Arch Pharm.*, 1898, 236, 487.

³ *J. Chem. Soc.*, 1884, 45, 41; 1891, 59, 1029.

followed extraction with dilute alcohol. The gum was therefore hydrolysed in the following manner :—

The powdered gum (100 gms.) was rubbed in a mortar with 3 per cent. sulphuric acid (800 c.c.). The mucilage was heated on the water-bath for eight hours in a round-bottomed flask, fitted with an air-condenser. The liquid was filtered, heated to boiling, neutralised with well-washed barium carbonate, again filtered and the filtrate evaporated to small bulk under diminished pressure. By a systematic precipitation with 90 per cent. alcohol, decantation, evaporation and reprecipitation with 90 per cent. alcohol, the sugar solution was obtained free from the barium salt of the gum-acid. The barium salt was dissolved in water and freed from sugar in a similar manner by precipitation, solution and reprecipitation.

From 100 gms. of the gum containing about 16 per cent. of water, about 10 gms. insoluble in alcohol and about 5 gms. soluble in alcohol were obtained; the residual gum amounted to about 30 gms. Very little material could be washed out of the barium sulphate precipitate by boiling water. No appreciable amount of gas could be observed as formed during hydrolysis. The loss of 40 per cent. of gum can only be explained by retention in the barium sulphate precipitate, as indicated by the fact that a portion of the precipitate after washing, still charred on ignition.

Examination of the alcohol-insoluble product of hydrolysis.—This was obtained as a thick brown syrup, consisting of the barium salt of the gum-acid. An aqueous solution of this salt when decolorised with animal charcoal was optically inactive, and reduced Fehling's solution to a very slight extent. It did not give a precipitate with silver nitrate, mercuric chloride, or lead acetate solution. The free acid could not be isolated by adding hydrochloric acid to the aqueous solution of the salt and subsequent dialysis of the mixture, nor could it be dissolved by ether.

The syrupy precipitate of the barium salt was rubbed with alcohol to remove as much water as possible. The lumps were then dried *in vacuo* over sulphuric acid for a few days. On drying a portion of this in a steam-oven the weight did not become constant but decreased on heating with simultaneous darkening. The heating was therefore stopped when the decrease in weight, after half an hour's heating, amounted to less than one mgm. The organic matter was then burnt off and the barium in the ash determined in the usual way ($\text{BaO} = 8.26$ per cent. of the dry salt).

The remaining portion of the barium salt was hydrolysed with 2.5 per cent. sulphuric acid for four hours in the proportion of 10 gms. of salt to 100 c.c. of acid; the latter was then removed with

barium carbonate paste, the barium salt and sugar being purified by the method already described. The salt obtained in this way was much darker in colour than the one from which it was prepared. The percentage of barium oxide was found to be 9.48 on the dry weight of salt. A very small quantity of sugar was obtained which, from the crystalline appearance of the phenylosazone, appeared to be arabinose. A further hydrolysis for four hours more yielded a barium salt containing 10.84 per cent. BaO, together with a trace of sugar too small for determination.

This production of an acid gradually decreasing in molecular weight shows that *Boswellia* gum resembles those investigated by O'Sullivan.

Alcohol-soluble product of hydrolysis.—The sugar soluble in alcohol was very hygroscopic. It was dissolved in 90 per cent. alcohol and evaporated under reduced pressure to a very thick syrup which did not crystallise even after a month *in vacuo* over sulphuric acid.

An aqueous solution was slightly sweet, and gave furfural when distilled with hydrochloric acid. The phenylosazone had the appearance of arabinosazone under the microscope and melted at 156° after purification with boiling alcohol and animal charcoal.

The specific rotation of the substance was 62.9° and the cupric oxide reducing power $K = \frac{100 \times 2.1}{2.205} = 95.2$.

An attempt was made to crystallise arabinose by seeding the syrupy solution of the sugar with arabinose crystals; although some crystals formed in this way, they were difficult to separate from the accompanying hygroscopic mass, and the quantity formed was very small. Since no trace of glucosazone was observed among the osazone crystals in the original syrup it was concluded that glucose was absent.

The presence of arabinose was confirmed by preparing the diphenylhydrazone; it resembled under the microscope the hydrazone prepared from pure arabinose and consisted of white needles melting at 195–196° as compared with 198° for the pure substance.

Further proof of the presence of arabinose was obtained by examining the crystals which separated from the syrup after about six months *in vacuo* over sulphuric acid. These crystals could be separated by rubbing the mass with absolute alcohol, in which the crystals were insoluble. The separated crystals were dried in the steam oven. The specific rotation was found to be 104.6°, the m. p. of the crystals 158°, and the m.p. of the phenylosazone 159°, figures which agree fairly well with arabinose.

Galactose.—The presence of galactose in the original syrup was proved by the production of mucic acid by heating with nitric acid; it melted at 215° . Thus the syrup contains galactose, but considering the amount of the phenylosazone precipitate, it is probable that galactose is present as galactan rather than as the free substance.

Xylose.—The presence of xylose was proved by Bertrand's reaction with bromine and cadmium carbonate, which gave the characteristic boat-shaped crystals of the double bromide and xylonate of cadmium, $(C_5H_9O_6)_2Cd$, $CdBr_2 + 2H_2O$. A control observation with 0.2 gm. of xylose showed a large amount of precipitate, and the crystals under the microscope resembled those obtained from the syrup. The low yield from the syrup showed that xylose was present only in small amount.

Total pentoses.—A measure of the total pentoses present in terms of arabinose was obtained by distilling 1 gm. of the dry material with 100 c.c. of hydrochloric acid (sp. gr. 1.06), in the manner described by Browne.¹ The furfural in the distillate was determined according to Ling and Nanji's² method by precipitation with a standard solution of phenylhydrazine, excess of which was determined by titration with standard iodine and thiosulphate solutions.

The amount of pentoses, calculated as arabinose, was about 65 per cent. of the syrup. From the quantity of arabinose crystals, separated as described in a former paragraph, it would not appear that the amount of arabinose is as high as 60 per cent. The percentage of xylose appeared to be but small. It is possible, therefore, that some strongly hygroscopic pentose, such as lyxose, may be present.

Fermentation of the syrup.—Fermentation tests with various species of yeast were practically negative, confirming what might be expected from the character of the sugars present.

CONCLUSION.

The gum appears to resemble true gums in that it yields sugars like arabinose, xylose and galactose, on hydrolysis. There is some evidence that acids of diminishing molecular weights can be produced from the original gum-acid by successive gentle hydrolysis.

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¹ *Handbook of Sugar Analysis.*

² *Biochem. J.*, 1921, 15, 466.