THE EVALUATION OF TRAGACANTH BY MEANS OF THE APPARENT VISCOSITY DETERMINED IN A STANDARD U-TUBE VISCOMETER

PART II.—WHOLE GUM

BY W. P. CHAMBERS

From the Laboratory of Damancy & Co., Ltd.

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IN A PREVIOUS COMMUNICATION[1] a method was suggested for the routine comparison of the apparent viscosities of powdered tragacanth by the use of a U-tube viscometer.

It was found that in order to use this type of instrument the sample of mucilage must be relatively homogeneous, a condition that was arrived at by the combined application of heat treatment and mechanical homogenisation. Using the U-tube viscometer it was not found possible to attain homogeneity by heat treatment alone without reducing the viscosity of the sample to figures that bore but little relation to the original viscosity; and as the object was to exhibit the gum at a point as near as possible to the maximum viscosity, both heat and mechanical treatment were found to be necessary. The problems involved in the evaluation of the whole gum are essentially those encountered in the powder, but modified to some extent by the intractable nature of the flake.

EXPERIMENTAL

An examination of a number of commercial samples of whole gum showed great diversity in size, thickness, texture and colour; while attempts to prepare mucilages from flake, without preliminary treatment and within those limits which are known to be without deleterious action on the viscosity, resulted in complete failure. That mucilages can be prepared from whole gum by treatment with boiling water or by immersion in a water-bath over a sufficiently long period is, of course, recognised, but as previous experiments show, such a method of ensuring homogeneity does not yield comparable results when determinations are carried out in a U-tube viscometer. It has long been recognised that the quality of tragacanth is adversely affected when the whole gum is reduced to powder by the customary commercial methods, and this has usually been thought to be due to mechanical destruction or to the heat created during grinding; but whatever the cause, it is advisable that both these attributes to a reduction of viscosity be maintained at the lowest level.

The method as finally adopted consists of "kibbling" in a mortar a suitable quantity of a representative sample of the flake until it passes a No. 30 sieve, after which the sample is thoroughly mixed. Attempts at preparing mucilages from this treated material, although an advance over the whole gum, resulted in products which, even after standing

for several days, showed large aggregations of undispersed gum. The problem of effecting "solution" of the gum was finally solved by the use of a mechanical stirrer. A simple Pyrex all-glass stirrer operated by compressed air was found to be very satisfactory. The addition of alcohol prior to the addition of water serves to keep the particles of gum sufficiently separated until the stirrer can be introduced into the flask. In the absence of alcohol, aggregates of gum are formed which are not usually dispersed by subsequent stirring. Fairly good dispersion is usually effected in less than one hour's stirring.

It soon became obvious that the employment of the same technique as that used for the powder, namely, allowing to stand at room temperature for 48 hours, would not result in full hydration of the gum. In addition, most samples contained a proportion of woody debris in amounts that would probably block the jet of the homogeniser. The mucilage, therefore, after standing for about 24 hours, was "cleaned" by passing it twice through a No. 100 sieve by means of reduced pressure. It was usually necessary to assist the mucilage through the sieve by means of a very small stencil brush, finally removing any mucilage adherent to the under surface of the sieve by means of a spatula. For this purpose it is desirable to use a piece of apparatus which can easily be dismantled and a suitable combination may be made from a Phœnix filter funnel, diameter $3\frac{1}{2}$ inches, and a Sifting Investigator outfit carrying B.S.S. sieves of $3\frac{7}{8}$ inches diameter. With a view to accelerating the rate of hydration and studying the effect of maintaining mucilages at different temperatures, a series of samples were stored in an oven operating at controlled temperatures. Table I shows the effect of such treatment on four different gums.

TABLE I

THE EFFECT ON THE VISCOSITY (TIMES OF FLOW IN SEC.) OF 0·5 PER CENT. MUCILAGES, PREPARED FROM WHOLE GUM, OF MAINTAINING THEM AT VARIOUS TEMPERATURES

Period	48 hours	24 hours	48 hours	48 hours	48 hours	48 hours
Temperature °C.	Room	40°	40°	50°	60°	70°
1	243, 283 258	272, 260 342	351, 355, 374 422, 367, 361	447, 451, 450 478, 439, 430	421, 408, 412 418, 423, 439	189, 246, 171 237, 210
2	2500	2734	2520, 3022	3300, 2820	1805, 1735	925
3	3840	3720	5015, 4800	3670, 4080	2440, 2930	813
4	1735	2160	2280, 2290	2100, 2040	1680, 1765	814

From these experiments it emerges that, in general, using a U-tube viscometer, maximum hydration is not attained by allowing the mucilage to stand at room temperature for 48 hours. The viscosity is seen to rise until a maximum is reached around 40°C., above which a falling off occurs, while a temperature of 70°C. has a very marked effect in reducing the viscosity. It may reasonably be assumed, therefore, that in employing an oven temperature of 40°C. for 48 hours, the hydration

is thereby accelerated to an approximate maximum and at the same time such temperature is without undue significance in its destructive effect on the viscosity.

As the strengths of the mucilages made from whole gum were the same as those previously used in experiments on the powder, namely, 0·5 per cent., it was at once apparent in the samples so far examined that the viscosities obtained when using whole gum were far higher than those encountered in the case of the powdered drug. Using a No. 3 U-tube viscometer (K = 0·306) it is unusual, at a strength of 0·5 per cent., to find times of flow for the powdered gum in excess of 1000 sec., yet in the case of whole gum times of the order of 2000 to 5000 sec. are quite common.

It would thus appear that the whole gum is a far superior article, as judged by apparent viscosity, than the generally available commercial powder. The knowledge that the gum yields a product of inferior quality when ground leads naturally to the assumption of different standards for the two forms; and this fact renders the use of the U-tube viscometer more rational than otherwise would be the case. In Table II are recorded a few results showing the figures for times of flow of mucilage of whole gum, when partly ground and when fully ground, and although the complete history of the gums was not fully known, the figures nevertheless suggest that a reduction of viscosity may be expected on grinding.

TABLE II

THE EFFECT OF GRINDING ON THE VISCOSITY (TIMES OF FLOW IN SEC.) OF 0·5 PER CENT. HOMOGENEOUS MUCILAGES PREPARED FROM WHOLE GUM, PARTLY GROUND AND FULLY GROUND GUM

Sample	Whole Gum	No. 80 Powder	No. 140 Powder
1	1657	1302	840
2	300	205	121
3	167	172	98
4	2830	—	1830
5	2760	—	1962
6	286	—	183

Strengths of mucilage that are applicable to the powder are, when applied to the whole gum, found to yield times of flow that are altogether too time-consuming for routine comparisons, and it is necessary to reduce the strength of the flake to yield figures that are comparable to those obtained when using the powder. A reduction in weight from 1·0 g. to 0·75 g. results in times of flow for the flake which are of the order of those obtained for the powder. The effect of reducing the strength of the mucilage is seen to be roughly proportional over the range of concentrations examined (Table III).

Method.—Kibble a representative sample of whole gum in a mortar until it passes a No. 30 sieve. To 0·75 g., accurately weighed, contained in a 300-ml. conical flask, add 5 ml. of alcohol (95 per cent.) followed

by 200 ml. of water, swirling the contents of the flask during the addition. Immerse the flask up to the neck in a water-bath maintained at from 40° to 50°C., insert a suitable mechanical stirrer and stir at a speed sufficient to maintain movement of the particles for about one hour. Remove from the bath, stopper the flask and place for a period

TABLE III

THE EFFECT OF VARIATION IN THE PERCENTAGE STRENGTHS OF GUM ON THE VISCOSITY (TIMES OF FLOW IN SEC.)

Concentration	0·25 per cent.	0·33 per cent.	0·375 per cent.	0·50 per cent.	Ratios
1	163	455	694	3000	1 : 2·8 : 4·3 : 18·4
2	165	460	838	3700	1 : 2·8 : 5·1 : 22·6
3	129	296	525	2300	1 : 2·3 : 4·1 : 17·8

of 24 hours in an oven maintained at 40°C. Attach a reflux condenser and immerse the flask up to the neck in a bath of boiling water for 5 minutes, swirling the contents of the flask vigorously for 5 seconds at the end of the first, second, third and fourth minutes. Remove from the source of heat and allow to cool to about 40°C. with occasional shaking and replace in the oven for 3 or 4 hours. Remove from the oven and by means of reduced pressure pass the contents of the flask as completely as possible through a No. 100 sieve twice. Replace in the oven until about 48 hours have elapsed from the time of commencement of the assay, after which pass the mucilage 3 times through a Q.P. homogeniser using a uniform and even pumping speed of about one complete stroke per sec. Determine the apparent viscosity (time of flow in seconds) at 20°C. in a No. 3 B.S. U-tube viscometer. The results of the application of this method to a number of samples of flake tragacanth are recorded in Table IV.

TABLE IV

THE RESULTS OF THE EVALUATION OF WHOLE GUM SAMPLES BY DETERMINATION OF THE APPARENT VISCOSITY (TIMES OF FLOW IN SEC.) IN A NO. 3 B.S. U-TUBE VISCOMETER

Sample	A	B	C	D	E	F	G	H	I
	637	709	45·0	126	377	572	740	145	657
	661	645	44·0	132	370	600	805	143	674
	665	690	46·0	129	382	579	827	142	702
	623	660	45·2	126	373	566	785	142	730
	643	675	45·5	131	350	576	720	144	712
	657	730	45·1	120	390	586	790	141	700
Mean	648	685	45·1	127	374	580	778	143	696
Standard deviation per cent.	± 2·5	± 4·6	± 1·47	± 3·4	± 3·7	± 2·1	± 5·2	± 1·1	± 3·8

Results of viscosity determinations on whole gum are seen to be considerably more variable than those on the powdered drug, and this is thought to be caused by a more variable rate of hydration resulting from the use of a kibbled product, which presents a surface to the hydrating medium that is only a fraction of that presented by a fairly uniform fine powder; in addition to the methodic errors the operative errors are also greater.

SUMMARY

1. A method is described for the routine comparison of the apparent viscosity of whole gum.

2. When determined in a U-tube viscometer, mucilages prepared from whole gum reach maximum hydration, and therefore maximum viscosity, after storage for 48 hours at a temperature of 40°C. With shorter periods at lower temperatures hydration is not complete and the viscosity is below the maximum, while with higher temperatures over the same period, although hydration is complete, the viscosity is reduced by virtue of the destructive effect of the higher temperatures.

3. At the same concentration, mucilages of whole gum yield higher viscosities than when commercially powdered, thereby indicating a loss of quality mainly due to grinding.

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REFERENCE

1. Chambers. *Quart. J. Pharm. Pharmacol.*, 1948, **21**, 44.