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EFFECT OF GUM TRAGACANTH SIZE REDUCTION ON DTA AND TG BEHAVIOUR AND ITS RELATIONSHIP TO SOLUTION VISCOSITY

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ABSTRACT

The effect of particle size reduction on the hydrocolloid solution viscosity of standardized grades of Persian gum tragacanth have been investigated The DTA and TG techniques used to identify the behaviour of gum on size reduction shows that the structure of the gum changes by fine grinding and the reduction of its solution viscosity could be attributed to the loss of the combined water which binds individual gum molecules by hydrogen bonding. The thermal methods with the relating features are discussed

INTRODUCTION

Gum tragacanth, referred to in this paper as GT, one of the oldest known drugs, is the exudate of the plant genus *Astragalus*, particularly *Astragalus gummifera* [1].

The structure of GT is highly complex and it is not fully elucidated [2]. However, it has been shown that GT is a mixture of polysaccharides consisting of three main components, namely: tragantic acid, a neutral polysaccharide, and a third component which appears to be a glucoside. GT polysaccharides are branched polymers consisting of main chains of α-D-galacturonic acid residue united to 1 : 4-α-glycosidic linkages. To this main chain are attached residues of monosugars in terminal and intermediate positions [3].

GT, upon mild HCl hydrolysis, produces galacturonic acid, sorbose, tagatose, galactose, glucose, mannose, fucose, arabinose, xylose and rhamnose [4]. This highly hydroxyl-containing branched polymer adsorbs large quantities of water and produces highly viscous hydrocolloid solutions.

Gum tragacanth has been used for centuries in pharmaceutical and cosmetic preparations as well as in food, drink, paint, dye, enamel and several other industries. Its wide range of application could be attributed to the proven lack of toxicity and the high viscosity of its hydrocolloid solutions (HCS) at very low concentration which give to its solutions thickening, emulsifying, suspending, and colloid protecting properties, depending upon the conditions of preparation. The viscosity of GT HCS varies from batch to batch and this can be attributed to the nature of the gum and the geographical conditions where it is produced, the gum producing plant species, the variety and grades of the gum used, its physical form and the treatment to which it has been subjected.

The method of solution preparation, even with well homogenized samples, shows, however, a variation in the viscosity of GT HCS at any given concentration due to the above variations in conditions.

One of the most important factors in determining the final viscosity of GT HCS is the particle size variation which has been overlooked by previous investigators. This is now the subject of our investigation using DTA and TG data to indicate structural and chemical changes caused by the particle size variation and which in turn lead to alterations in the viscosity of the GT HCS.

EXPERIMENTAL

Materials

Persian gum tragacanth ribbons (PGTR) and flakes (PGTF) were supplied by The Institute of Standard and Industrial Research of Iran (ISIRI). A synchrono-lectric viscometer, Model R.V.T., Brookfield Engineering Laboratories, U.S.A., was used to determine the viscosity of GT HCS.

A Retschmuhle particle size mill was used for grinding and an Endecott vibrating sieve, Type E.F.L., for gum powder classification. The homogenizer used was an Ultra-Turrax Type PT 1812, Janke and Kunkle. A Du Pont DTA 900 thermal analyzer and TG 950 thermogravimetric analyzer were used in these experiments.

Methods

Persian gum tragacanth is supplied in various grades of ribbons or as flakes. These were found to behave in a similar fashion on particle size reduction and a grade designated as ribbon, No. 3, (PGTR 3) is investigated in this study. PGTR 3 was ground and sieved to particle sizes ranging from 3000 μm to less than 45 μm. A stock solution of 0.5% w/v of PGTR 3 was prepared by swelling overnight each size fraction of PGTR 3 powder in demineralized water and then homogenizing it for 2 min with Ultra-Turbax homogenizer. The homogenized HCS was heated for 6 h at 55°C to produce an air bubble free HCS at the highest viscosity [5]. DTA measurements were carried out under a current of nitrogen at 300 ml min^{-1} using 20 mg of sized powder PGTR 3 and platinum crucible, both for sample and blank, at 20°C min^{-1} heating rate.

TG measurements, using 10 mg sized powder PGTR 3 samples, were carried out at 30°C min^{-1} heating rate and under a current of 300 ml min^{-1} nitrogen up to 700°C, then nitrogen to air.

The viscosity of PGT HCS samples was measured by Brookfield Synchro-Lectric Viscometer, Model LVF, using spindles 2, 3 and 4 and taking the average readings.

RESULTS AND DISCUSSION

The changes in viscosity of PGTR 3 HCS, 0.5% w/v, in relation to the particle size of the gum was measured (see Table 1) and is plotted in Fig. 1.

From the results obtained the viscosity of PGTR 3 HCS seems to decrease with the decrease in particle size below 1 mm average particle diameter For larger particles up to the natural exudate lumps no significant changes in viscosity have been observed. It is re-emphasized here that similar results have been obtained with the other ribbon and flake grades of PGT

The DTA and TG traces of PGTR 3 powders at various particle sizes ranging from 1000 to 45 μm were plotted. As shown in Figs. 2 and 3, and Table 2, the DTA curves show differences in the shape of the curves as a result of changing the particle size while keeping the other variables constant.

The commercial PTG grades contain 10—14% water which is gradually released upon heating, as shown in DTA traces (Fig. 2) and the TG plot (Fig. 3).

It has been found previously by Zahedi and Vedadi [5] that GT releases its adsorbed water by gradual heating up to 105°C but loss of this water does not affect the viscosity of hydrocolloid solutions made from this material. Further heating will cause loss of the combined water up to 150°C, where GT will suffer from drastic reduction in the viscosity of solutions made up from the residue due to polysaccharide degradation, and finally a completely insoluble product is produced. The GT decomposition takes place from 162°C and shows a maximum exothermic decomposition temperature at 259°C.

The destructive effect of heat of GT has also been reported by other investigators, namely Gralen and Karrholm [6], and Levy and Schwartz [7].

TABLE 1

The changes in viscosity of 0.5% w/v PGTR 3 HCS in relation to the particle size of the gum power

Mesh size	Average particle size (μm)	Viscosity (cp)
	10 000	525
	5 000	525
—10 + 30	1 050	525
—30 + 60	302.5	510
—60 + 100	165	497
—100 + 120	135	482
—120 + 150	115	456
—150 + 240	84	434
—240 + 300	58	334

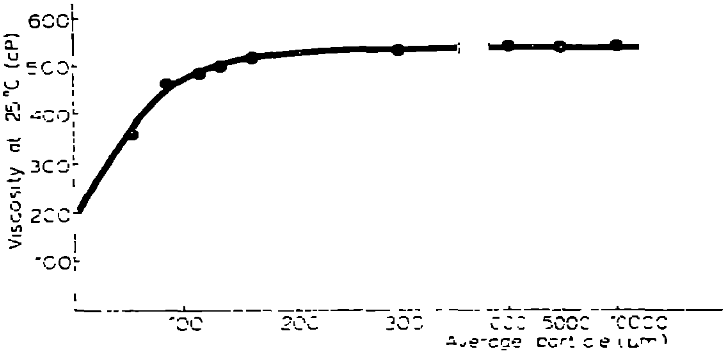

Fig. 1. Changes in the viscosity of 0.5% w/v PGTR 3 in relation to the particle size variations

The particle size reduction increases both the rate of water desorption and also that of the combined water which is probably hydrogen bonded between hydroxyl groups of GT polysaccharides and which are demonstrated to be affected by grinding. The change in the DTA behaviour of both the endothermic water desorption and the latter exothermic thermal degradation clearly shows the effect of grinding on the structure of the gum which in turn affects the viscosity of the PGT hydrocolloid solution. The TG data (Fig. 3) allows the conclusion from DTA traces to be quantitized, namely that desorbed water is increased from samples 4 to 7 on grinding, whilst the combined water content is decreased from 7 to 12%. The loss of desorbed water is clearly a function of the increased area consequent upon particle size reduction.

The alteration in the combined water content upon reduction of particle size is, however, a consequence of a lower average molecular weight, as may be inferred from viscosity measurements.

TABLE 2

The area and peak height of DTA traces for the endothermic desorption of adsorbed and combined water and exothermic degradation—decomposition of 2- mg PGTR 3 at various particle sizes

Particle size (µm)	Endothermic peak area (mm^2)	Endothermic peak ht. (mm)	Exothermic peak area (mm^2)	Exothermic peak ht (mm)
1000<	550	18	220	18
1000—500	800	32	264	20
500—250	1000	40	320	38
250—150	1385	53	242	25
150—100	1250	50	240	25
100— 75	1250	52	230	25
75— 63	1210	55	225	25
63— 45	1150	46	138	21

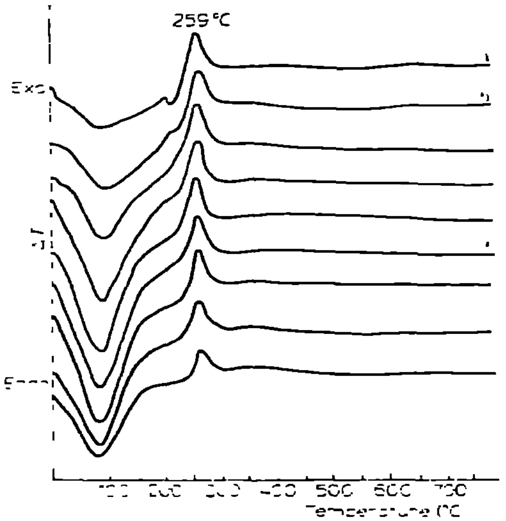

Fig 2. DTA curves of Persian gum tragacanth ribbon 3. Du Pont instrument, 20 mg sample, heating rate = 20°C min^{-1} in N_2 at 300 ml min^{-1} Particle size (μm): a = 1000<, b = 1000—500, c = 500—250, d = 250—150, e = 150—100, f = 100—75, g = 75—63, h = 63—45, i = 45>.

Considering the chemical structure of GT, i.e. a highly hydroxylated branch polymer, and the traganthic acid as its main constituent, it may be noted that a chemical structure has been proposed by James and Smith [3]. It would seem possible that water binds the molecules of GT together by hydrogen bridging with hydroxyl groups of GT polysaccharide. The grinding may partially rupture this hydrogen bonding so that individual GT molecules of lower average molecular weight are released. This means that the loss of some proportion of the combined water is an intramolecular chain cleavage

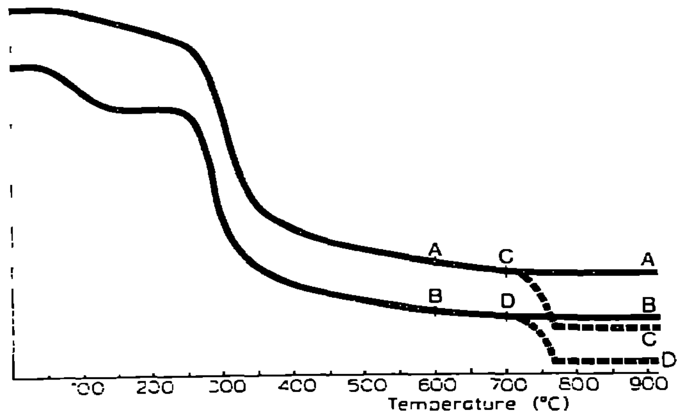

Fig 3. TG curves of Persian gum tragacanth ribbon 3. Du Pont instrument, 10 mg sample, heating rate = 30°C min^{-1} in N_2 at 300 ml min^{-1} Particle size (μm): A = 1000< in N_2. B = 45> in N_2, C = 1000<, D = 45> in air.

or chain scission process, whilst the majority is a chain stripping process. The latter will produce the endothermic process whilst the former would possibly contribute towards the exothermic peak.

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