Evaluation of the Stormer Viscosimeter in the Measurement of Certain Pharmaceutical Systems

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The performance of the Stormer viscosimeter was analyzed in detail by the study of the rheological properties of natural gums (acacia and tragacanth) and of synthetic gums (methylcellulose, sodium carboxymethylcellulose, and hydroxyethylcellulose). Two Newtonian systems (glycerin and sucrose solutions) were also investigated. Several different concentrations of each material were studied. There appeared to be an increase in viscosity with increasing rate of shear for most of the systems at the lower concentrations; a level of concentration was eventually reached where all the gum systems exhibited pseudoplastic characteristics and the Newtonian systems true linearity.

THE STORMER viscosimeter is a commercially available Searle type of viscometer described and modified for application to non-Newtonian systems by Fischer (1). The operational features and suggested useful ranges are outlined in several other references (2–5). The instrument has been widely used by investigators in the pharmaceutical sciences for well over a decade (6–12). Durability, ease of operation, and reproducibility of results are largely the strong points of the Stormer which led to its use by the above investigators.

None of the workers reported inconsistencies in the rheological data obtained. The following study was performed largely because of certain unusual results obtained in the measurement of some dilute solutions of hydrocolloids with the Stormer viscosimeter.

The initial inclination to attribute these discrepancies to experimental error was abandoned because of the recurring pattern of the results. Consequently, it was decided to carry out an extensive analysis of the performance of the Stormer viscosimeter in the measurement of various rheological systems.

EXPERIMENTAL

Preparation of Solutions—The substances used in the study, along with the range of concentration of their solutions, are given in Table I. From 7 to 10 solutions of different concentrations were prepared for each substance within the indicated range.

The solutions were prepared in the conventional manner. A Waring blender was used to disperse the gums after they had been moderately hydrated. Heat was employed to speed up solubility in the more concentrated solutions.

Rheological Measurements—A Stormer viscosimeter equipped with the modified cup and bob, as suggested by Fischer (1), was used. A weight hanger with various slotted weights provided the shearing stress. The appropriate instrumental constants, K_v, were determined from calibration data collected using standard oils, and were applied to each point on the rheograms. The apparent viscosity of the various solutions were then calculated from the expression:

$$\eta = K_v \frac{\text{Gm.}}{\text{r.p.m.}}$$

where Gm. represents the shearing stress and r.p.m. the rate of shear. All viscosity measurements were carried out at 20 ± 0.1°.

TABLE I—RANGE OF CONCENTRATION FOR EACH SUBSTANCE USED

Substance	% w/w
Acacia	7.5–45
Tragacanth	0.1–2.0
Methylcellulose	0.4–3.5
Sodium CMC	0.2–4.0
Hydroxyethylcellulose	0.5–2.0
Glycerin	35–90
Sucrose	35–70

RESULTS AND DISCUSSION

Rheograms were constructed for all the solutions by plotting r.p.m. as the ordinate and Gm. as the abscissa. Representative plots at three levels of concentration for each substance are shown in Figs. 1, 3, and 5.

The rheograms in Figs. 1 and 3 were redrawn to show the change in viscosity with rate of shear so that a more thorough analysis of the data could be performed. Figures 2 and 4 show the result of this representation.

The first series of rheograms obtained were those for the acacia solutions. As a result of the data collected, several other systems of known pseudoplastic nature were prepared as indicated in Table I; two Newtonian systems (glycerin and sucrose solutions) were also prepared to provide the basis for a complete comparison of the results.

Figure 1 shows rheograms of each solution at a low level of concentration. Each substance exhibits the same "dilatant" type curve, whereby there is an increase in viscosity with increasing rate of shear. These results can hardly be attributed to isolated experimental discrepancies, since, as can be clearly seen in Fig. 2, a definite pattern is consistent throughout. The instrument was checked as to proper alignment of the cup and bob and for any mechanical defects which might cause discrepancies in the data. No apparent mechanical anomalies were found to exist.

As the concentration of the solutions is increased, the rheograms indicate an over-all decrease and eventual disappearance of this dilatant effect. Figure 4 shows the plots for that level of concentration of each substance above which the so-called dilatant effect no longer exists.

Figure 5 shows the rheograms depicting the performance of the Stormer viscosimeter in the measure-

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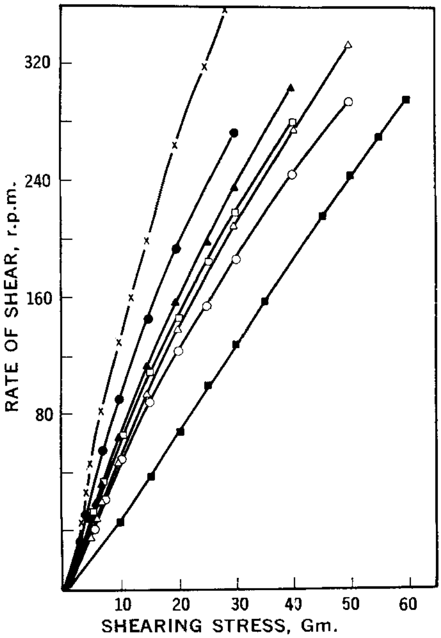

Fig. 1—Rheograms of aqueous solutions of low concentration. Key: ×, *0.15% tragacanth;* ●, *10.00% acacia;* ▲, *0.40% sodium carboxymethylcellulose;* □, *63.50% glycerin;* △, *0.70% methylcellulose;* ○, *51.50% sucrose;* ■, *0.50% hydroxyethylcellulose.*

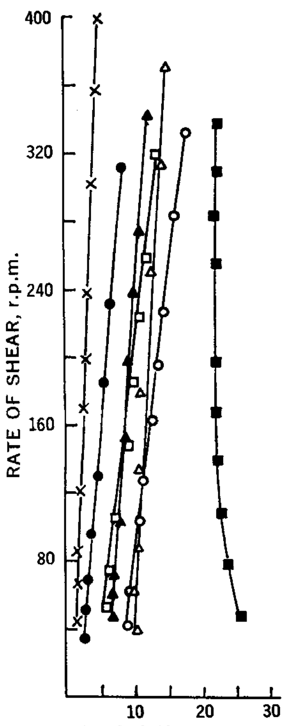

Fig. 2—Viscosity–shear rate dependence of aqueous solutions. Key: ×, *0.15% tragacanth;* ●, *10.00% acacia;* ▲, *0.40% sodium carboxymethylcellulose;* □, *63.50% glycerin;* △, *0.70% methylcellulose;* ○, *51.50% sucrose;* ■, *0.50% hydroxyethylcellulose.*

ment of the gum systems at the highest level of concentration of each, as indicated in Table I. All the

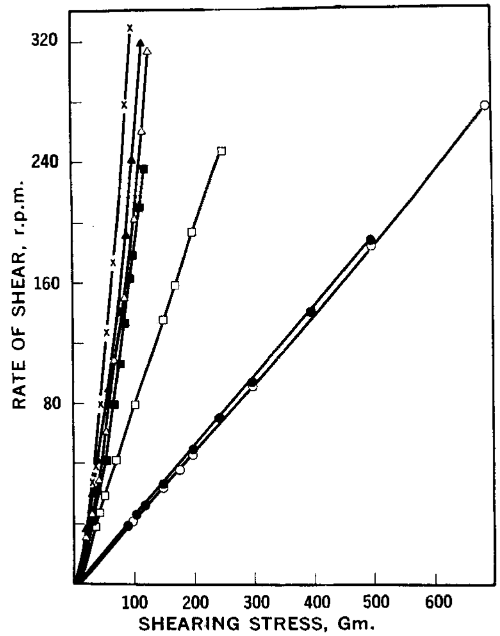

Fig. 3—Rheograms of aqueous solutions of concentration above which no dilatant effect exists. Key: ×, *0.95% tragacanth;* ●, *35.00% acacia;* ▲, *1.25% sodium carboxymethylcellulose;* □, *88.00% glycerin;* △, *1.30% methylcellulose;* ○, *70.00% sucrose;* ■, *0.75% hydroxyethylcellulose.*

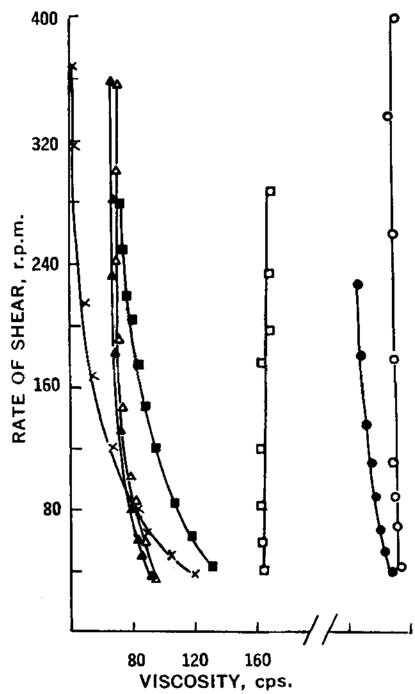

Fig. 4—Viscosity–shear rate dependence of aqueous solutions. Key: ×, *0.95% tragacanth;* ●, *35.00% acacia;* ▲, *1.25% sodium carboxymethylcellulose;* □, *88.00% glycerin;* △, *1.30% methylcellulose;* ○, *70.00% sucrose;* ■, *0.75% hydroxyethylcellulose.*

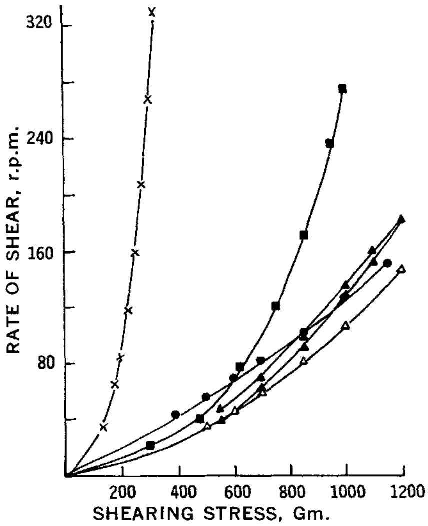

Fig. 5—Rheograms of aqueous gum solutions of high concentration. Key: ×, *1.8% tragacanth;* ●, *45% acacia;* ▲, *4.0% sodium carboxymethylcellulose;* △, *3.5% methylcellulose;* ■, *2.0% hydroxyethylcellulose*

systems are cleary pseudoplastic in nature, except for sodium carboxymethylcellulose which exhibits a slight degree of thixotropy. No apparent problems are evident at this level of concentration with the instrument used.

From all of the results obtained, unless one cares to argue the case for actual dilatancy of all the systems at low concentrations, there are some interesting inferences which can be made.

It is conceivable that all gums used are pseudoplastic throughout the entire range of shear at all concentrations, and that there is an inherent "dilatant" producing effect with the Stormer which shifts all systems in that direction at lower concentrations. This effect then becomes less pronounced as the concentrations are increased and the true pseudoplastic nature of all the hydrocolloids eventually becomes predominant rendering the dilatant appearance imperceptible.

The other possibility is that these hydrocolloids are Newtonian in nature at the lower concentrations and the dilatant behavior simply indicates that the Stormer cannot be used effectively for systems having a viscosity lower than 20 cps. As the concentration is increased the systems become truly pseudoplastic and the Stormer performance is satisfactory at all rates of shear. This approach would explain the glycerin rheograms, since they exhibit Newtonian behavior only after a certain concentration is reached. Glycerin solutions, incidentally, should not be used to calibrate the Stormer viscosimeter in order to obtain the instrumental constants, K_v, because of their hygroscopic nature.

SUMMARY

1. Various hydrocolloids at several concentrations were studied using the Stormer viscosimeter.

2. Two Newtonian systems (glycerin and sucrose solutions) at various concentrations were also studied rheologically.

3. Results showed some interesting features not usually attributed to these systems: (*a*) there appeared to be a dilatant effect for all systems at low concentrations; (*b*) there seemed to be a minimum concentration for all gum systems, above which pseudoplastic behavior is observed throughout the entire range of shear rate.

4. The Stormer viscosimeter appeared to perform well in the rheological measurements of the more concentrated gum systems.

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