Rheological Characterization of Carbopol 940 in Steady Shear and Start-up Flow Fields

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ABSTRACT

Using a strain-controlled rheometer, rhelogical characterization of carbopol 940 has been made in steady shear and start-up flow fields. In this study, aqueous carbopol 940 solutions with different concentrations were used to provide a fundamental information in application of pharmaceutical and cosmetic areas.

INTRODUCTION

Carbopols are polymers of acrylic acid cross-linked with polyalkenyl ethers or divinyl glycol.[1] Depending upon the degree of cross-linked and manufacturing conditions, various grades of carbopols are available. Generally, carbopols have a great water sorption property. They swell in water up to 1000 times their original volume and 10 times their original diameter to form a gel when exposed to a pH environment above 4.0 to 6.0.[1-2]

Carbopols, as an anionic hydrogel, are widely used to improve the rheological properties of thickening systems.[3] Their exceptionally good optical clarity and thickening power make carbopols very effective and economical. After neutralization, they are used for emulsification, stabilization and rheological control in cosmetic and pharmaceutical industries. They are also used to control the release of medicaments from time-release tablets or from entrapped systems and known to be a highly efficient thickener by forming a networked microgel structure in aqueous solutions. In personal care, home care, pharmaceutical, and cosmetic areas, carbopol dispersions and gels are useful vehicles for many functional ingredients and are found in dozens of everyday products, including toothpastes, tile cleaners, bath gels, and artificial tears.[4] Recently, they are also used for their mucoadhesive properties and a relevant amount of work has been done on the bioadhesive potential of carbopol polymers.

Carbopol 940 is a cross-linked polyacrylate polymer. It is an extremely efficient rheology modifier capable of providing high viscosity and forms sparkling clear water or hydroalcoholic gels and creams.[5]

In spite of such a wide use of carbopols in pharmaceutical and cosmetic industries, only a little research has been performed on their rheological characterization.

The objective of this study is to investigate the rheological properties of carbopol 940 in steady shear flow fields and start-up flow fields. This work would be able to provide an important information in the study of rheological attribution of carbopol in application of pharmaceutics and cosmetics.

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EXPERIMENTAL SECTION

Preparation of Carbopol Solutions

The carbopol 940 sample used in this study was kindly provided from the Lubrizol Corporation (USA).

In this study, aqueous carbopol solutions with different concentrations of 0.3, 0.4 and 0.5 wt% were prepared by constant stirring using propeller-type variable-speed homogenizer for 6 hr with a rotational speed of 600 rpm at room temperature. Then, it was neutralized by adding TEA at 1:1 equivalent ratio of TEA : carbopol and this system was further constantly stirred for 3 hr with a rotational speed of 1000 rpm at room temperature. In order to complete dissolution of polymer, the prepared solutions were kept at rest at room temperature for 24 hr prior to conducting the rheological measurements.

Rheological Measurements

The rheological properties of prepared carbopol 940 solutions were measured using a strain-controlled rheometer [Advanced Rheometric Expansion System (ARES), Rheometric Scientific, USA] equipped with a parallel-plate fixture with a radius of 12.5 mm and a gap size of 2.0 mm. All measurements were performed at a fixed temperature of 20 ℃.

In steady shear flow fields, experiments were performed over a wide range of shear rates from 0.025 to 1000 1/s with a logarithmically increasing scale.

The transient rheological behavior on start-up of steady shear flow was investigated at a number of shear rates of 0.025, 0.05, 0.1, 0.25, 0.5, 1.0, 2.5, 5, 10, 25, 50, and 100 1/s.

Before the carbopol solutions were loaded, the two parallel-plates were covered with sandpaper in order to remove a wall slippage between the test material and the plates. In all measurements, a fresh sample solution was used and rested for 15 min after loading to allow material relaxation and temperature equilibration.

RESULTS AND DISCUSSION

Figure 1 shows the steady shear stress for carbopol 940 solutions with different concentrations. For all concentrations of carbopol solutions, the steady shear stress tends to level off and approach a limiting constant value as a decrease in shear rate towards zero at low range of shear rates, indicating that these polymer systems exhibit a finite magnitude of yield stress. It is also observed that the larger values of yield stress are obtained with increasing carbopol concentration.

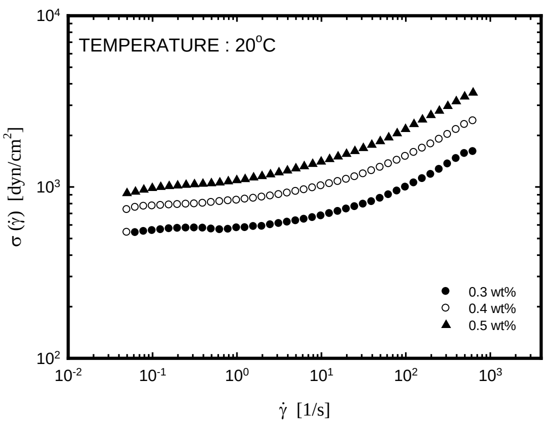

Figure 1. Steady shear stress as a function of shear rate for carbopol 940 solutions with different concentrations.

Figure 2 represents the shear rate dependence of the steady shear viscosity for carbopol 940 solutions with different concentrations. For all concentrations of carbopol solutions, while the Newtonian region is not observed at low shear rates, the steady shear viscosity is sharply decreased as an increase in shear rate, demonstrating that these polymer systems exhibit a marked shear-thinning behavior.

Figure 3 illustrates the shear stress on start-up of steady shear flow at shear rate of 25 1/s for carbopol 940 solutions with different concentrations. In the case of ordinary polymer solutions, the stress is rapidly increased (stress overshoot phenomenon) at an initial stage and then, followed by a stress decay behavior until reaching the steady state of stress value. However, no stress overshoot phenomenon is observed for carbopol solutions and a perfect equilibrium state of stress value is not reached. Such an unexpected behavior may be explained by analyzing the results shown in Figure 4.

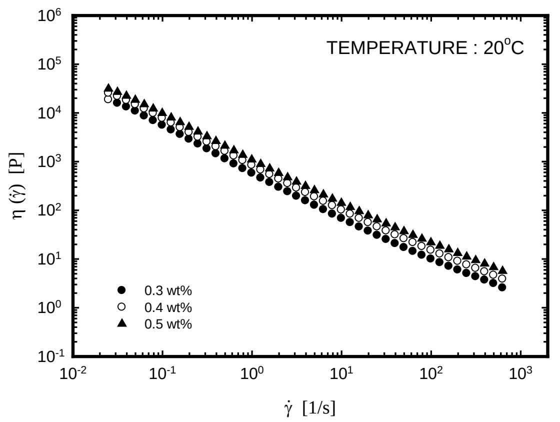

Figure 2. Shear rate dependence of steady shear viscosity for carbopol 940 solutions with different concentrations.

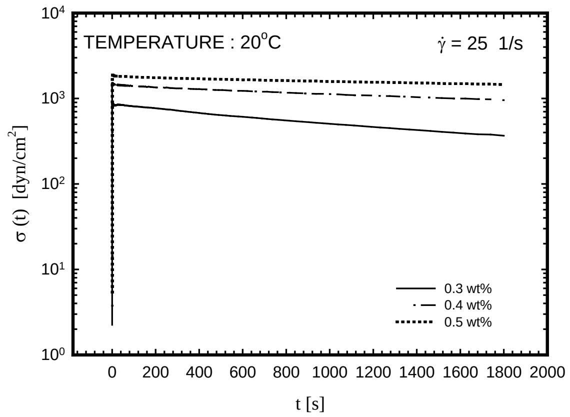

Figure 3. Start-up flow behavior with time for carbopol 940 solutions with different concentrations at shear rate of 25 1/s.

Figure 4 displays the angular frequency dependence of the storage modulus and loss modulus for 0.3 wt% carbopol 940 solution. While the storage modulus is almost independent on angular frequency, the loss modulus is gradually decreased as an increase in angular frequency. This causes a continuous stress decay behavior in start-up of steady shear flow fields.

In Figure 5, transient flow behavior on start-up of steady shear flow with several constant shear rates for 0.3 wt% carbopol 940 solution is illustrated. A continuously slow stress decay behavior is observed for all shear rates imposed even though the stress value is progressively increased as an increase in applied shear rate.

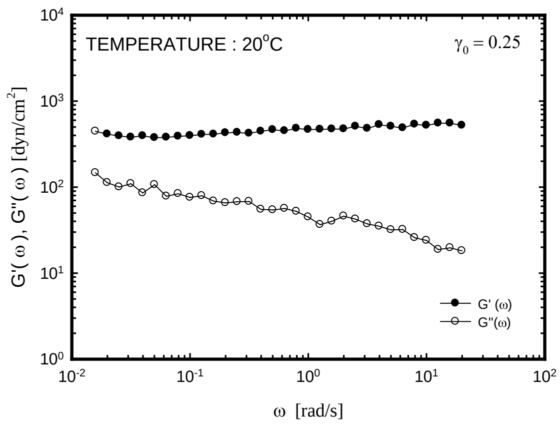

Figure 4. Frequency dependence of storage modulus and loss modulus for 0.3 wt% carbopol 940 solution.

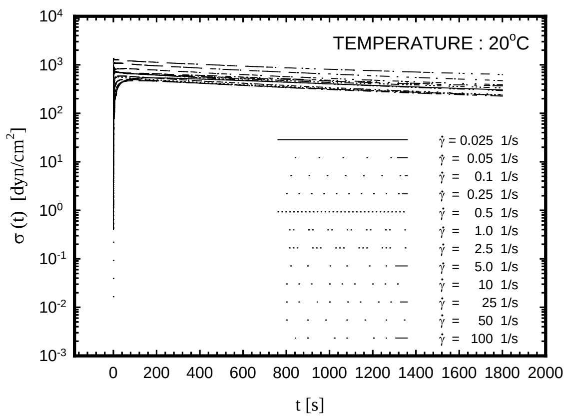

Figure 5. Start-up flow behavior with time for 0.3 wt% carbopol 940 solution at various shear rates.

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