

## Research Report:

# RHEOMETRY OF POLYACRYLIC ACID (PAA) POLYMERS TO DETERMINE THIXOTROPIC BEHAVIOUR

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**Abstract:** This report details the rheological testing performed to determine whether **polyacrylic acid (PAA) polymers- Carbopol 934 and Carbopol 974P-** exhibit **thixotropic behaviour**.

**Problem Statement:** Carbopol 934 and Carbopol 974P find use in **bio-adhesives, muco-adhesives and other drug delivery systems**. Their rheological properties (which vary with chemical structure) are the key to usage in such applications. For instance, **time-dependent viscosity at fixed shear stress/rates (or thixotropy)** is of use in such applications. Rheological characterization is the key to quantification and design of drug delivery vehicles utilizing these materials. Accurate and logical use of rheometry can determine these properties as is shown below.

**Materials & Methods:** Carbopol 934 and Carbopol 974P were received in the form of fine powders (Bangalore Fine Chemicals). They were prepared into **2.5% w/w solutions in distilled water using continuous magnetic stirring**; final pH ranged from 2-3. They were then neutralized using **0.1N Sodium Hydroxide (NaOH) solution** until a pH of 7 was obtained.

Rheometry was performed on the **Anton Paar Physica MCR 301 Rheometer** (Department of Chemical Engineering, IIT Hyderabad) using the **PP50 (Parallel Plate, 50mm diameter)** as the **primary measuring system**. The tests were also repeated with the CP75 (Cone and Plate, 75mm diameter) measuring system. The *software* used to control the settings of the Rheometer was **RHEOPLUS/32 V3.61 21006012-33056**.

## Results:

For a given a sample to be characterized, one of the early tests used in its characterization is the 'Flow test' wherein the **apparent viscosity is measured at continuously varying shear rates**. Results of the flow test for the two PAA polymers are given in Figure 1. This led to the conclusion that **both materials exhibit shear-thinning, Non-Newtonian behaviour**.

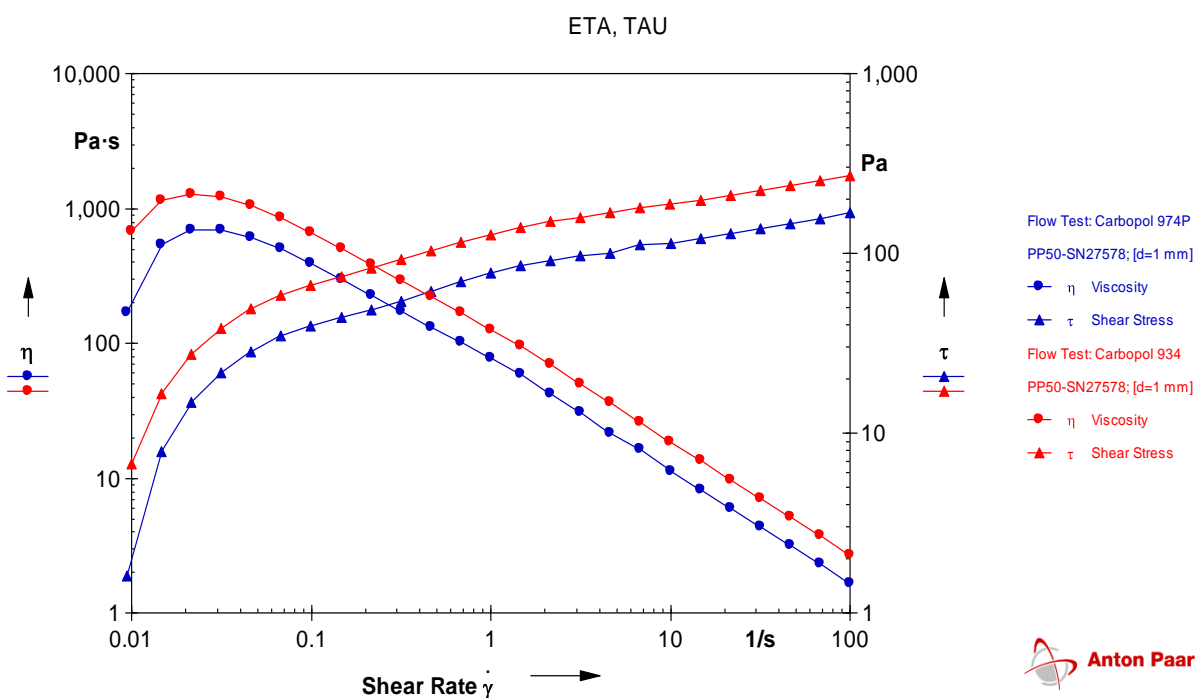


Figure 1: Flow Test of Carbopol 934 and Carbopol 974P

Having determined this basic rheological characteristic, we then proceeded to further characterize the samples using the 'Constant shear rate test' wherein the **apparent viscosity is measured at a fixed value of shear rate**. The results of this test (shown in Figures 2a & 2b) are critical to determine whether a sample exhibits thixotropic behaviour or not: **if the measured viscosity decreases with time, then the sample is said to be thixotropic**; else, it is not. It is observed that both **Carbopol 974P and Carbopol 934 exhibit thixotropic behaviour**. Further, **Carbopol 934 exhibits a higher viscosity than Carbopol 974P** for the range of shear strain under study.

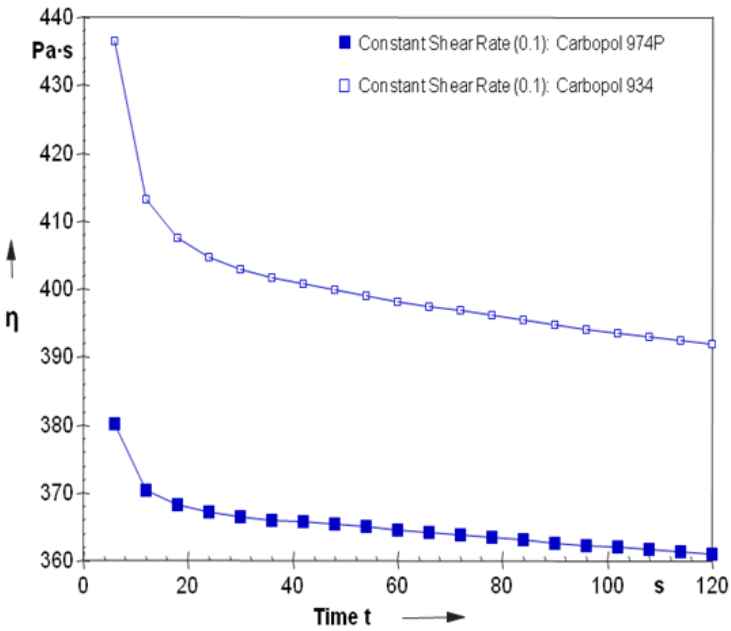


Figure 2 (a): Constant Shear rate test at 0.1 sec-1 for both Carbopol 934 and Carbopol 974P

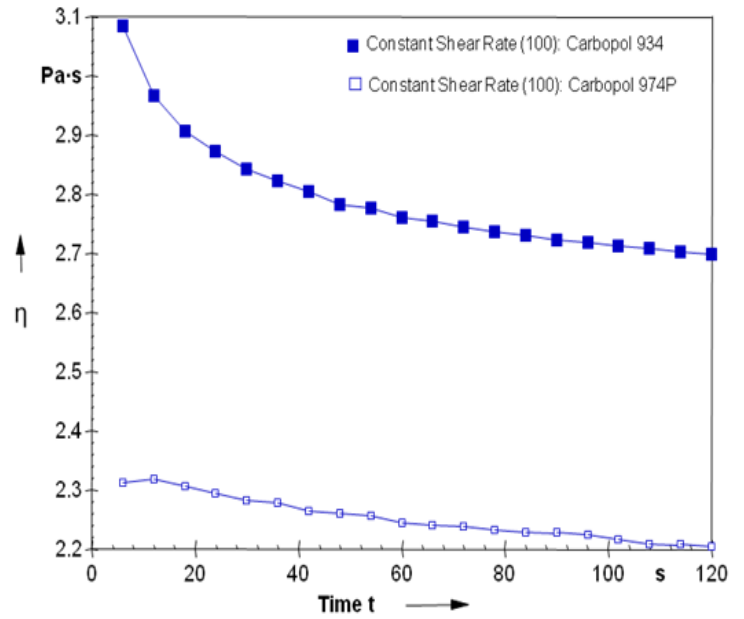


Figure 2 (b): Constant Shear rate test at 100 sec-1 for both Carbopol 934 and Carbopol 974P

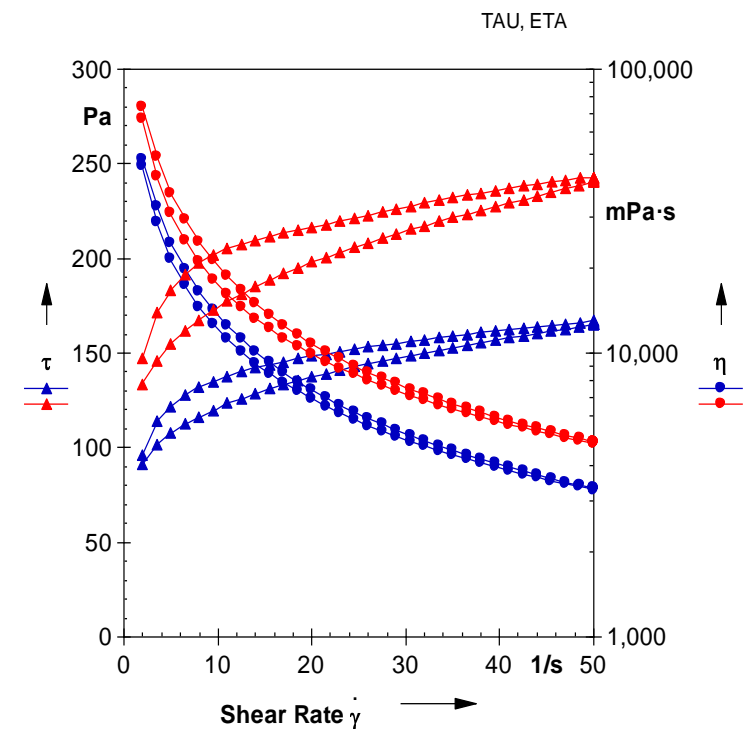


Figure 3: Hysteresis Test of both Carbopol 934 and Carbopol 974P

In order to confirm that the results obtained in Figure 2 are repeatable, and that both samples are indeed thixotropic, it is necessary to perform a ‘Hysteresis test’ wherein the shear rate increases steadily to a pre-set fixed value, and then decreased at the same rate to the initial value. The larger the enclosed area of the hysteresis loop, stronger is the time-dependent behaviour of the materials. Hence, no hysteresis loop is observed for time-independent fluids. Conversely, a hysteresis loop is obtained for fluids having time-dependent nature. The results shown in Fig. 3 show both *Carbopol 974P* and *934* having finite hysteresis loops confirming that they are indeed thixotropic.



**Conclusions:** The rheometric testing involving the Flow Test, The Constant Shear test and The Hysteresis test has yielded data from which it can be said that the given PAA Gel Polymer Samples, Carbopol 934 and Carbopol 974P exhibit time dependent nature or thixotropy, and are also shear-thinning. Further properties of PAA gels should be assessed using creep, oscillatory and sweep studies.

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