

Low Shear Rate Flow Rheology Study on Carbopol Solutions Using MRI (Magnetic Resonance Imaging)

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Abstract

An analysis of complex (yielding) rheological flow behavior of 0.1 wt%, 0.13 wt%, 0.15 wt% Carbopol solutions has been carried out using MRI velocimetry within the low shear rate region ($10\text{-}4\text{-}10\text{ S}^{-1}$) for gravity driven flow system. For this study, flow kinematics in a pipe was performed at the steady and creeping conditions ($Re < 1$) to get accurate dynamic yield value of Carbopol solutions. Then we show that in this range of shear rates, MRI velocimetry works well using with using the suitable image data acquisition process. Yield stresses were achieved successfully for Carbopol solutions. The less concentrated 0.1 wt% Carbopol solution was found to have the yield stress as 9.8 Pa whereas the more concentrated 0.15 wt% Carbopol solution exhibited higher yield stress value of 54.4 Pa. These are the first measurement of yield stress of Carbopol solutions by this method in the range of $10\text{-}4\text{-}10\text{ S}^{-1}$. The MRI measurements compared well with the measurements of same samples performed on conventional rheometer in the shear rate range of $10\text{-}2\text{-}10\text{ S}^{-1}$. This study provides insights into MRI velocimetry can capture the velocity profile of flows under the creeping condition.

Keywords: Rheology; Carbopol; Yield stress; Pipe flow; MRI

Introduction

Obtained of rheological information from fluid through pipelines flow helps to enhance product quality for industrial processes [1]. The shear viscosity of Newtonian and generalized Newtonian flow can be monitored by MRI based viscometer techniques. Velocity profile imaging can be extracted with the known of pressure drop and pipe dimensions of entire flow using MRI techniques. Conversion of velocity profiles into rheological data is only based on computing relation between shear rate and shear stress data processing during on-line flows (McCarthy) [2]. It can be applied various geometry flow analysis for rheological characterization with some experimental limitation of resolution or quality of velocity data [3].

Accuracy of MRI velocity data implementation is given by Arola et al. [2] and Tozzi et al. [4]. Extracting the velocity profile from the image and calculating the shear rate data presents several challenges for MRI viscometer. The image data need to have sufficient signal-to-noise and sufficient velocity resolution to achieve a desired range of shear rates. The minimum shear rate depends upon the velocity resolution (Arola et al. 1998). After the appropriate velocity resolution is set and the image acquired, a velocity profile is extracted. Shear rates are calculated as a function of radial position by taking the derivative of the velocity profile in MRI rheometer [4].

Zero shear rate or creeping flow rheology has crucial potential for measuring of yield stress of complex fluid. From a practical point of view, yield stress concept has a significant relationship to consumer perception of product quality. Conventional yield stress measurement methods sometimes fail due to accuracy of torque measurement in off-line rheometers [5].

In this study, Carbopol known as a complex fluid (Nyguen and Boger 1992) creeping rheology is investigated by measuring the steady fluid velocity using MRI. Other side of this study is to see the limitation of MRI rheometer to detect the flow in low shear rate region with the consistent resolution of images.

Materials and Methods

Experimental fluids

Cross-linked polyacrylate polymer (Carbopol940, Lubrizol Corporation) was used with different concentrations (0.1%, 0.13%, 0.15% w/w). These solutions were prepared using powder form of Carbopol-940 with mixed with distilled water in a stirred tank for approximately 7 days. The polymer solutions were neutralized with TEA (tri ethanolamine) solution to pH 7.1. The neutralization process is crucial because of the strong dependence of the flow behavior of Carbopol solutions on pH. The neutralization allows the solution to achieve its maximum viscosity since the polymer chains disentangle at this pH.

Flow system

The closed flow loop system used in this study supplies gravity driven flow serving low velocity measurement for MRI imaging. The flow loop is shown schematically in FIG. 1. The test fluid was recirculated using a Moyno pump (Integrated Motor Drive System, Franklin Electric) through a pipe with an internal diameter of 38.1 mm made of PVC pipe connected with stainless steel fitting parts. Pressure drop was obtained at the ends of pipe with a constant length of 1.68 m using pressure transducer (Siemens Company). To ensure fully developed flow in the imaging part of pipe 4.5 m with straight fittings upstream of magnet (between exiting of pump and entrance of pipe section) ($L/d=120$). Flow driven tank height was changed to between 50 cm-150 cm using a lab cart with an adjustable height platform.

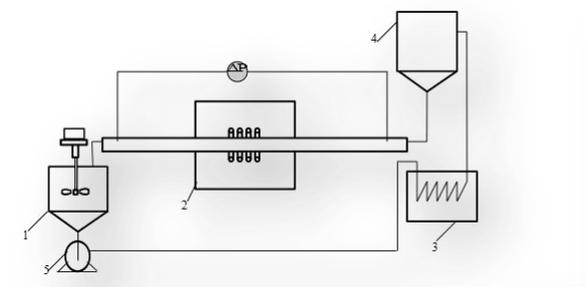


FIG. 1. Components of the flow system: 1. Storage tank, 2. Imaging magnet 3. Temperature bath 4. Flow driven tank, 5. Moyno pump (positive displacement pump).

Magnetic resonance imaging rheometer

Velocity profiles and rheological measurements were detected using MRI under pulsed gradient spin echo sequence (PGSE) (Arola et. al 1998) on an Aspect Imaging 1 Tesla permanent magnet (Aspect Imaging, Shoham, Israel) at Food and Science Technology Department at University of California, Davis, USA. Imaging parameters are adjusted to capture steadily imaging files. Slice of thickness (mm) gives thickness of shell or slice of normal direction of flow. Thicker slice improves the signal intensity during imaging. Field of view (FOV) is related to the thickness of vertical direction of flow approach to the signal. Velocity Sweep Width (VSW) is adjusted with respect to velocity of flow and decides to the velocity resolution of image. They are critical parameters to capture velocity of flow accurately. The slice of thickness and field of view were 30 mm and 50 mm respectively. The radial and velocity encodings used 256 steps. Small velocity sweep width values (VSW) to capture accurate low velocity flow imaging through the pipe are used as listed in TABLE 1. The radio frequency of coil was a solenoid with four turns, encasing a cylindrical volume 60 mm in diameter and 60 mm long. System temperature was kept constant at 22.2°C using temperature controller and heat exchanger. Steady state shear flow experiment parameters were used shown in TABLE 1. The Reynolds number was smaller than unity.

TABLE 1. Some typical example of experimental parameters used in imaging process.

Carbopol Conc. (%)	VSW (cm/s)	Q (l/min)	<i>Re</i>
0.10	9	1.235	0.370
0.13	6	0.864	0.020
0.15	5	0.746	0.015

Results and Discussion

Measured velocity MRI images for 0.15% Carbopol solutions for no flow and axial velocity flow condition are given in FIG. 2 and FIG. 3. MRI no-flow condition is acquired prior to initiating flow and also has same signal for 0.1% and 0.13% Carbopol solutions. The signal intensity depicts the axial velocity as a function of radial position. The vertical bright region in the center of no-flow image spans the width of the pipe which indicates the position of pipe walls. The bright region position in the center of the image for no-flow condition at voxel number 256 is for axial velocity $v=0$. The Carbopol velocity profiles for 0.15% are shown in FIG. 3a-3d at different volumetric flow rates. As the flow rate is increased, the signal intensity is positioned more to the right as expected with higher velocities. At 0.25 l/min, the image in FIG. 3a exhibits a

more blunted velocity profile for VSW value of 5 cm/s. It is sufficient to capture small shear rate ranges for the flow. Plug like shape velocity profile expands as volumetric flow rate is increased as in FIG. 3b-3d. Carbopol solutions do not exhibit slip velocity at these flow rates as seen in FIG. 3.

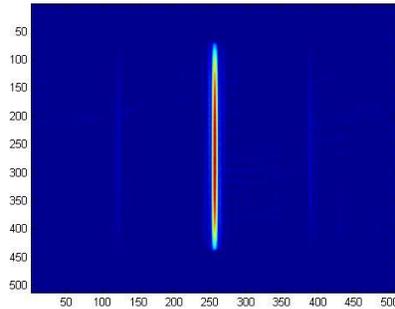


FIG. 2. No flow condition of solutions (no wall slip).

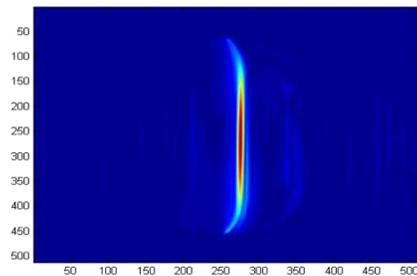


FIG. 3a. 0.25 l/min.

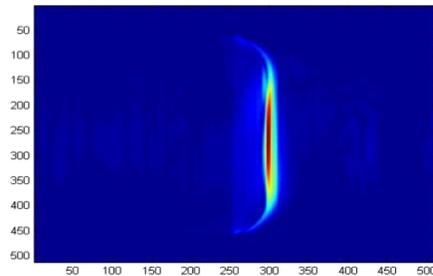


FIG. 3b. 0.5 l/min.

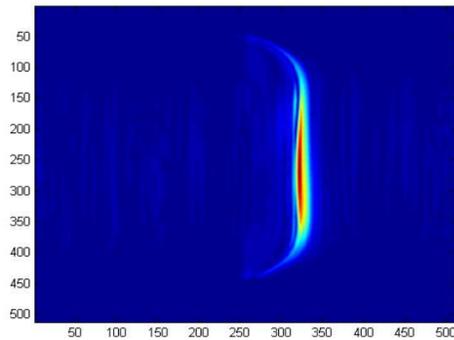


FIG. 3c. 0.75 l/min.

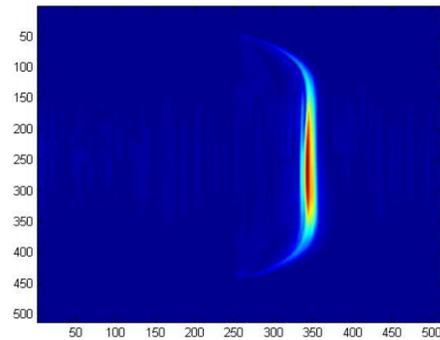


FIG. 3d. 1.0 l/min.

FIG. 3. Measured velocity profile 0.15% Carbopol solutions with different flow rates.

FIG. 4a-4c show measured velocity profiles for Carbopol solutions with no-slip at wall ($r=-0.02$ and $r=0.02$). They are given as function of pipe radius, r . The flow is unidirectional flow and velocity field is simply given by $V=V(r)$ for different pressure drop measurements in FIG. 4a-4c. Velocity magnitude is small enough to observe low shear rate flow. Maximum

velocity is reached for 0.1% Carbopol solution as expected in FIG. 4a. Velocity data exhibits apparent plug-like behavior. The minimum shear rate data point was determined at the lowest rate investigated shown in all FIG. 4a-4c.

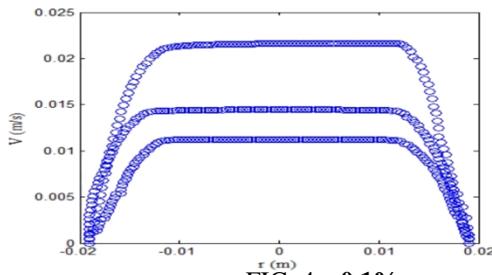


FIG. 4a. 0.1%.

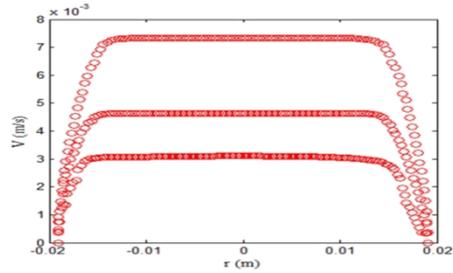


FIG. 4b. 0.13%.

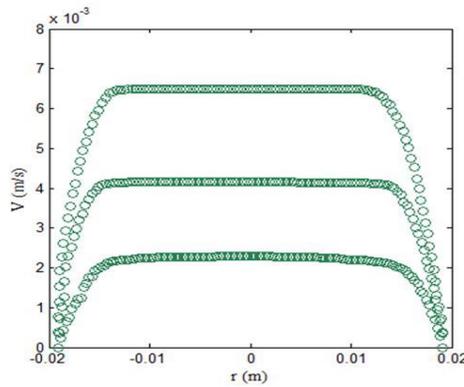


FIG. 4c. 0.15%.

FIG. 4. Velocity profiles for Carbopol solutions.

Rheograms are given for three different Carbopol concentrations determined using in-line and off-siline methods as depicted in FIG. 5. The square symbol lines represent the values obtained with the off-line conventional rheometer. The circles correspond to MRI velocimetry data. For conventional methods, shear rates start from 0.085 S^{-1} . MRI velocimetry data match closely with the off-line data for all concentrations. Instrument CVO rheometer (Bohlin Instruments) with a cone and plate rheometer (with a cone angle 4° and diameter 40 mm) at 22°C was used for offline measurement. A steady state shear rate ramp from 0.085 S^{-1} to 10 S^{-1} was performed in logarithmic mode with 10 points/decade.

Many rheological measurements at different flow rate conditions shear stress/shear rate data are obtained as shown in FIG. 5. The lower shear rate is limited by the velocity resolution of flow image because no data are acquired on shear rates below the minimum calculated velocity resolution for MRI method. For this study the minimum shear rate range is on the order of 10^{-4} S^{-1} in TABLE 1. It must be consistent with measured experimental shear rate data to estimate of the quality of shear stress and shear rate data. The minimum shear rate can be calculated using the relationship between non dimensionalized minimum shear rate and resolution of velocity data (Arola et al. 1998). The results are obtained using rescaled smoothing process pre-

described by Tozzi et al. 2012. This rescaling feature also helps to eliminate artifacts that can occur in the velocity profiles during data acquisition process performed using MATLAB. It removes potential sources of error which can be resulted from image acquisition. Accurate dynamic yield values estimates can be obtained from this process and are shown in FIG. 5a-5c. Lower yield is achieved for lower concentration of Carbopol as in FIG. 5a. From FIG. 5a-5c, yield stresses are found as 9.8, 22.8 and 54.4 Pa which corresponds to the intersection of shear stress value when approaching shear rate is nearly zero. As Carbopol concentration increased, yield stress gets larger as expected. These results are also in good agreement with those reported by Coussot et al. (2009).

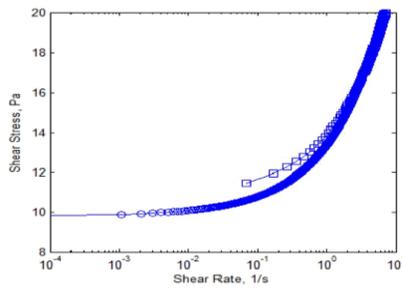


FIG. 5a. **0.1%.**

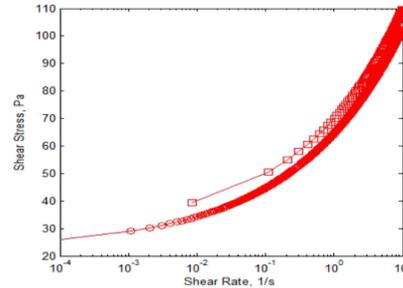


FIG. 5b. **0.13%.**

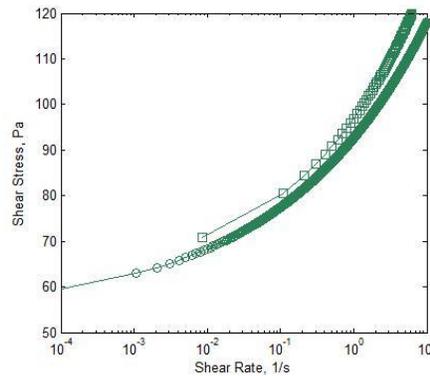


FIG. 5c. **0.15%.**

FIG. 5. Rheograms for Carbopol solutions.

Conclusion

The combination of MRI velocity measurements with a pressure drop measurements permits a relationship between shear rate and shear stress to be developed and thus yields a rheological measurements. An experimental investigation of the creeping flow of 0.1%, 0.13% and 0.15% w/w concentrations of Carbopol solutions in MRI as a process monitoring rheometer was presented. The following conclusions can be drawn from the results of the experiments:

- MRI flow imaging at low Reynolds number are readily obtained from the gravity driven experimental flow design.

- MRI velocimetry is an effective tool for obtaining accurate yield stress data for Carbopol solutions at low shear rate ranges.
- An earlier developed rescaled smoothing method is useful for creeping flow image acquisition.
- Carbopol solutions do not exhibit slip velocity at these low Reynolds numbers.

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