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Research Article

RHEOLOGICAL PARAMETER ESTIMATION OF CMC-WATER SOLUTIONS USING MAGNETIC RESONANCE IMAGING (MRI)

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ABSTRACT

In this study, the application of Magnetic Resonance Imaging (MRI) rheometry on the measurement of complex fluid Carboxymethyl cellulose (CMC)-water solutions (0.5%, 1.0%, 1.5%, 2.0% w/w) flow was described. Depending on CMC concentration, Power law or Herschel-Bulkley models gave the best fit according to MRI and conventional rheometer (CVO) results. Power Law model was valid for 0.5% and 1.0% CMC ($R^2=0.9993$ - $R^2=0.9987$ and $R^2=0.9983$ - $R^2=0.9985$ respectively by MRI and CVO). On the other hand, 1.5% and 2.0% CMC solutions flow were well described by Herschel-Bulkley model ($R^2=0.9994$ - $R^2=0.9996$ and $R^2=0.9986$ - $R^2=0.9981$ respectively by MRI and CVO). The MRI measurements agreed well with the CVO measurements.

Keywords: *Magnetic Resonance Imaging, Conventional Rheometer, CMC, Rheology*

1. INTRODUCTION

Offline methods for rheological measurements such as cylindrical coquette, cone and plate geometries (conventional rheometries) generally used for the study of fluid motion in shear. However, obtained results from these types of geometries need to be verified with suitable online or inline methods. Especially, many industrial processes, such as extrusion, transfer processes involve established or developing flows in pipes or tubes. Therefore, online techniques based on the measurement of the velocity profile in a pipe flow using Magnetic Resonance Imaging (MRI), which is a non-invasive method, and simultaneously determining the pressure drop, are promising for use a product quality or rheology control tool during the fluid flow. Magnetic resonance imaging (MRI) can be used as a viscometer, based on analysis of a measured velocity profile of fluid flowing in a tube coupled with a simultaneous measurement of the pressure drop driving the flow (Arola *et al.*, 1997 and Arola *et al.*, 1999).

This type of measurement is well suited for rheological characterization of non-Newtonian fluids (Choi *et al.*, 2002 and Tozzi *et al.*, 2012).

To evaluate shear viscosity in tube (or capillary) flow, an incompressible fluid must undergo steady pressure-driven flow in the laminar regime. The conservation of linear momentum, which equates pressure forces to viscous forces, provides the relationship between the shear stress, σ , and radial position, r :

$$\sigma(r) = \frac{-\Delta P}{2L} r \quad (1)$$

where ΔP is the pressure drop over the tube length L . The shear rate is obtained at the same radial position using the velocity profile obtained from a flow image. The expression for the shear rate in tube flow is:

$$\gamma(r) = \frac{dV(r)}{dr} \quad (2)$$

Where V is the axial velocity. Using Equations 2 and 3, the apparent viscosity η is determined by the ratio of shear stress to shear rate:

$$\eta(r) = \frac{\sigma(r)}{\gamma(r)} \quad (3)$$

Graphical User Interface (GUI) programs are used to analyze data and display rheological results (Choi *et al.*, 2005 and Tozzi *et al.*, 2012). Main step in the data processing procedure include calculating the shear stress as a function of radial position in the pipe, processing the velocity profile image to obtain a velocity profile, calculating the shear rate as a function of radial position from the velocity profile, and generating the rheogram by plotting the shear stress against the shear rate (Arola *et al.*, 1997, Callaghan 1999 and Tozzi *et al.*, 2012). Calculating the shear stress is straightforward as in Equation 1.

In this study, Carboxymethyl cellulose (CMC) was used as test fluid. CMC is widely used as thickener

especially in food and pharmaceutical industries (Benchabane and Bekkour, 2008). This is also known as complex fluid due to no linear relationship between stress and shear rate in simple shear during the flow.

2. MATERIALS AND METHODS

2.1. Materials

The CMC, with nominal molecular weight of 250,000 g/mol was supplied from Sigma. Aqueous solutions of CMC were prepared by dissolving the appropriate amount of CMC powder in distilled water. The high CMC concentration solutions (0.5%, 1.0%, 1.5%, 2% w/w.) were prepared by using water heated at 50 °C by gentle stirring with the sufficient time < 24 h.

Online and offline measurements were performed with an MRI (Magnetic Resonance Imaging) at Food and Science Technology Department at University of California, Davis, USA using flow loop depicted in Fig. 1. At 25 °C, MRI Flow Imaging Tests were done for 0.5, 1, 1.5, 2% (w/w) CMC solutions to determine rheological constitutive equations parameters. Inlet diameter of PVC tube was 38.1 mm. The test fluid was recirculated using Moyno pump (Integrated Motor Drive System, Franklin Electric) Pressure drop was obtained at the ends of pipe with a constant length of 1.68 m using pressure transducer (Siemens Company).

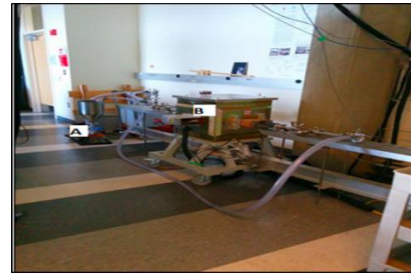


Fig. 1. Flow loop setup for CMC testing A) Positive displacement pump B) MRI magnet

2.2. Methods

In Fig. 2, flow image for an example of 0.5% CMC flow, can be seen with data processing window. The velocity profile is used to obtain shear rate distribution, while the pressure drop is used to calculate the shear stress distribution. By taking the ratio of these quantities at a radial position, local viscosity can be obtained within the shear rate range in the flow, zero at the center, and maximum at the wall, within minutes. There is not observed slip effect on the wall as in Fig. 2.

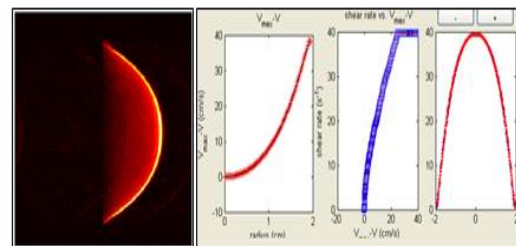


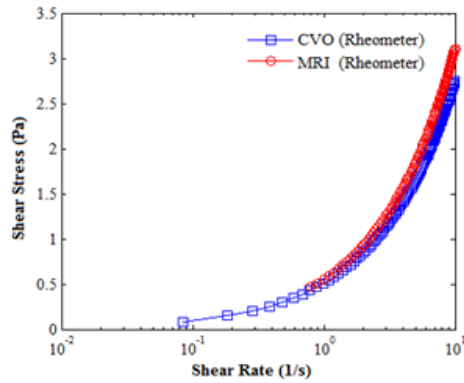
Fig. 2. MRI Image for 0.5% CMC

Fig. 2 shows the flow curves of the CMC solutions at different concentrations. Instrument CVO rheometer (Bohlin Instruments) with a cone and plate rheometer (with a cone angle 4° and diameter 40 mm) at 25 °C was used for offline measurement. A steady state shear rate ramp from 0.085 to 10 s⁻¹ was performed in logarithmic mode with 10 points/ decade.

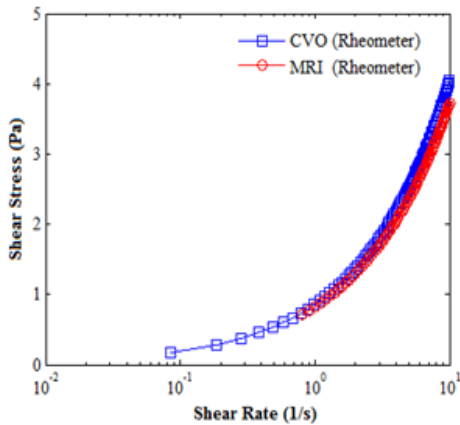
3. RESULTS AND DISCUSSIONS

For MRI measurements at the different pump speed of flow loop and also measured using a conventional technique and the agreement between the results is satisfactory shown in Figure 3. MRI measurement results of CMC solutions are also listed in the Table 1 with changing pump speed of flow loop shown in Fig. 3.

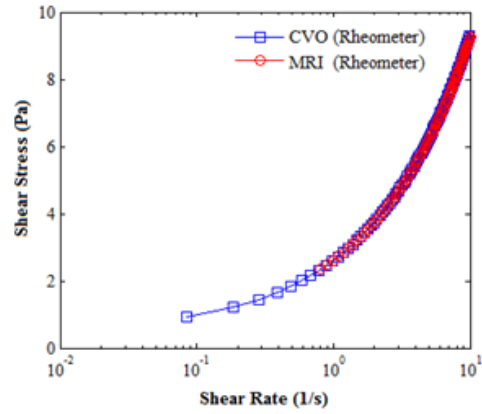
All obtained rheograms for different CMC solutions are as listed in Table 1. Rheological properties are independent of flow velocity. Hence, zero shear viscosities are nearly constant during the flow. As Reynolds number and concentration of flow increased, fluid shear stress acting on the pipe wall also increased as seen in Table 1.



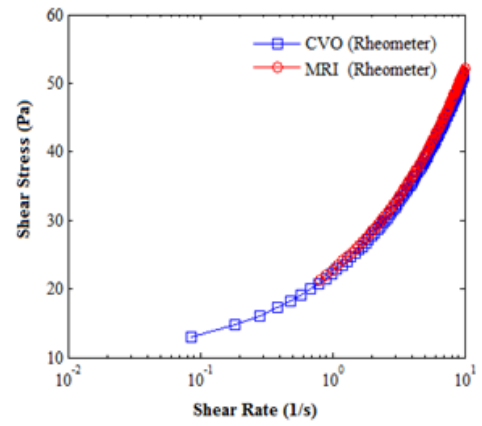
(a)



(b)



(c)



(d)

Fig. 3. Flow curves of shear stress versus shear rate for CMC- water for various mass fractions (a) 0.5% CMC (b) 1.0% CMC (c) 1.5% CMC (d) 2.0% CMC at 25 °C

Rheological parameters for CMC solutions are also listed in Table 2. Depending on CMC concentration, Power law or H.Bulkley models give the best fit according to MRI flow result using Equations 4 and 5 respectively. Power Law model is valid for 0.5% and 1.0% CMC. On the other hand, 1.5% and 2.0% CMC solutions flow are well described by Herschel–Bulkley model.

$$\sigma = K\gamma^n \quad (4)$$

$$\sigma = \sigma_0 + K\gamma^n \quad (5)$$

Table 1. MRI flow measurement for CMC-water solutions

| | Pump Speed (rpm) | V(m/s) | Re | Wall stress (Pa) | Zero Shear Viscosity (Pa.s) |
|---------|------------------|--------|--------|------------------|-----------------------------|
| 0.5%CMC | 330 | 0.023 | 2.176 | 4.371 | 0.412 |
| | 400 | 0.039 | 4.020 | 4.840 | 0.411 |
| | 700 | 0.078 | 8.840 | 7.590 | 0.411 |
| | 1000 | 0.121 | 14.445 | 11.730 | 0.413 |
| 1.0%CMC | 1500 | 0.196 | 25.013 | 15.240 | 0.415 |
| | 330 | 0.032 | 1.620 | 7.230 | 1.002 |
| | 430 | 0.043 | 2.312 | 9.350 | 1.003 |
| | 600 | 0.060 | 3.390 | 12.780 | 1.001 |
| | 1000 | 0.122 | 7.870 | 20.670 | 1.002 |
| | 1500 | 0.193 | 13.590 | 28.605 | 1.001 |
| 1.5%CMC | 330 | 0.035 | 0.970 | 16.800 | 2.001 |
| | 460 | 0.047 | 1.400 | 21.560 | 1.989 |
| | 600 | 0.058 | 1.850 | 27.770 | 2.012 |
| | 1000 | 0.094 | 3.300 | 42.540 | 2.014 |
| | 1500 | 0.183 | 7.690 | 55.550 | 2.001 |
| 2.0%CMC | 330 | 0.031 | 0.180 | 53.681 | 9.012 |
| | 500 | 0.053 | 0.360 | 77.670 | 9.022 |
| | 800 | 0.077 | 0.580 | 95.431 | 8.912 |
| | 1000 | 0.095 | 0.760 | 106.071 | 8.993 |
| | 1500 | 0.147 | 1.340 | 127.350 | 8.912 |

In Equations 4 and 5, consistency index, K, and power law index, n, and yield stress, σ_0 , data values are obtained from shear stress v.s. shear rate data using online (MRI Rheometry) method and offline (CVO Rheometry) method. R^2 values of the fittings are also satisfactory. As CMC concentration increased, yield stress gets larger and elastic forces dominates the viscoelastic flow medium (Nguyen & Boger, 1992).

Table 2. Rheological Parameters for CMC-water solutions

| | MRI Rheometer | CVO Rheometer | Goodness of the fit R^2 (MRI- CVO) |
|---------|---------------------------------------|---------------------------------------|--------------------------------------|
| 0.5%CMC | K=0.550 n=0.753 | K=0.512 n=0.730 | 0.9993-0.9987 |
| 1.0%CMC | K=0.825 n=0.653 | K=0.863 n=0.670 | 0.9983-0.9985 |
| 1.5%CMC | $\tau_0=0.436$ K=2.176 n= 0.607 | $\tau_0=0.424$ K=2.640 n=0.608 | 0.9994-0.9996 |
| 2.0%CMC | $\tau_0=9.054$ K=14.731 n=0.495 | $\tau_0=9.150$ K=13.120 n=0.507 | 0.9986-0.9991 |

4. CONCLUSION

MRI velocity measurements with a pressure drop measurements allows a relationship between shear rate and shear stress and yields a rheological parameters measurements. A rheological investigation of CMC flow of 0.5%, 0.10%, 0.15%, 0.20% w/w concentrations in

MRI and CVO was presented. The following conclusions can be highlighted from the results of the study:

- Rheological parameters are independent of flow conditions.
- 0.5% and 1.0% w/w CMC are suited with Power law model. 1.5% and 2.0% CMC w/w solutions flow are well described by Herschel–Bulkley model.
- Online and offline measurement results are good agreement with each other.
- MRI flow imaging is suitable for evaluations of rheological parameters of CMC solutions even in high concentration of 1.5 and 2.0% w/w CMC.

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