Journal of Magnetic Resonance 286 (2018) 30–35

Contents lists available at ScienceDirect

Journal of Magnetic Resonance

journal homepage: www.elsevier.com/locate/jmr

Validation of a low field Rheo-NMR instrument and application to shear-induced migration of suspended non-colloidal particles in Couette flow

A.A. Colbourne [a,b], T.W. Blythe [a], R. Barua [a], S. Lovett [b], J. Mitchell [b,*], A.J. Sederman [a], L.F. Gladden [a]

[a] Magnetic Resonance Research Centre, Department of Chemical Engineering and Biotechnology, University of Cambridge, West Cambridge Site, Philippa Fawcett Drive, Cambridge CB3 0AS, UK

[b] Schlumberger Gould Research, High Cross, Madingley Road, Cambridge CB3 0EL, UK

ARTICLE INFO

Article history:
Received 24 October 2017
Revised 17 November 2017
Accepted 18 November 2017
Available online 21 November 2017

Keywords:
Rheo-NMR
Low field
Non-colloidal particles
Particle migration
Couette flow
Process monitoring

ABSTRACT

Nuclear magnetic resonance rheology (Rheo-NMR) is a valuable tool for studying the transport of suspended non-colloidal particles, important in many commercial processes. The Rheo-NMR imaging technique directly and quantitatively measures fluid displacement as a function of radial position. However, the high field magnets typically used in these experiments are unsuitable for the industrial environment and significantly hinder the measurement of shear stress. We introduce a low field Rheo-NMR instrument (^{1}H resonance frequency of 10.7 MHz), which is portable and suitable as a process monitoring tool. This system is applied to the measurement of steady-state velocity profiles of a Newtonian carrier fluid suspending neutrally-buoyant non-colloidal particles at a range of concentrations. The large particle size (diameter > 200 μm) in the system studied requires a wide-gap Couette geometry and the local rheology was expected to be controlled by shear-induced particle migration. The low-field results are validated against high field Rheo-NMR measurements of consistent samples at matched shear rates. Additionally, it is demonstrated that existing models for particle migration fail to adequately describe the solid volume fractions measured in these systems, highlighting the need for improvement. The low field implementation of Rheo-NMR is complementary to shear stress rheology, such that the two techniques could be combined in a single instrument.

1. Introduction

Transport of suspended non-colloidal solid particles is an important process in many industries, including oil and gas production [1] where the controlled placement of a cement slurry [2] and conveyance of solids by drilling fluids [3] are critical for safe and successful operations. Determining the hydrodynamic properties of non-colloidal large particle (diameter > 200 μm) suspensions is a challenge. These complex fluids are not amenable to measurement in narrow-gap rheometric devices due to particle jamming, and violate the narrow-gap approximation [4] in wide-gap geometries such as the cylindrical Taylor-Couette cell [5], where additionally the local rheology may be a function of radius. Nuclear magnetic resonance rheology (Rheo-NMR) measures the spatial variation in velocity and concentration of a sample under shear [6,7] and gives direct access to local shear rate $\dot{\gamma}$ in the wide-gap Couette geometry through the relation

$$\dot{\gamma}(r) = r\frac{\partial}{\partial r}\left(\frac{v}{r}\right), \tag{1}$$

where v is the tangential velocity at radius r [8]. In the case of liquids containing suspended non-colloidal particles, the sample evolves over time due to shear-induced radial migration of particles, plus centrifugal radial migration and gravity-driven vertical migration depending on the density mismatch between the solid and liquid components [9–13]. The ability of Rheo-NMR to probe inhomogeneous and opaque liquid systems makes it ideal as a tool for routine process monitoring and quality control in the industrial environment. A commercial Rheo-NMR device is available, designed to fit in the vertical bore of a high field superconducting magnet [14]. However, the installation of high field magnets in a factory or remote location is impractical due to the limitations of cost, safety (strong magnetic fields), and maintenance (requirement for cryogens). Low field permanent magnets, or recently commercialized cryogen-free superconducting magnets, offer lower cost, safer alternatives. Permanent magnet systems also offer the advantage of portability [15]. Low field magnets have been used previously to provide relaxation time measurements of liquid samples in conjunction with shear rheology measurements [16–18]. The usual

* Corresponding author.
E-mail address: JMitchell16@slb.com (J. Mitchell).

https://doi.org/10.1016/j.jmr.2017.11.010
1090-7807/

Rheo-NMR experiment lacks a direct measure of shear stress although custom systems have been proposed that combine NMR and torque sensors [19,20], albeit still based on superconducting magnets with limited industrial application. Rheo-NMR has been applied to the study of many complex samples, including shear-banding materials [21–23], polymer solutions [24,25], worm-like micelles [26,27], liquid crystalline materials [19,28–31], and colloidal particle suspensions [32,33]. Shear-induced migration of non-colloidal particles has been studied by NMR imaging in Couette [34–36] and pipe flow [37,38].

The migration of particles in neutrally-buoyant suspensions in non-uniform shear flows has been described with a variety of models. Diffusive-type models describe the migration process directly as a diffusive process, with particle fluxes directed by gradients in shear rate and particle concentration [39,40]. Models of the suspension-balance type describe migration as resulting from gradients in normal stress of the suspension [41,42]. In recent years, researchers have been successful in modeling the behavior of dense suspensions using frictional rheology models [43] similar to those previously used for dry granular flow [44]. These can be shown to be equivalent in some cases to suspension balance models [45], but have been generalized successfully to describe additional physics such as suspensions in yield-stress fluids [46] and compaction beyond the jamming limit [45,38].

In this paper we use a low field permanent magnet imaging system to measure velocity and concentration profiles in a wide-gap Couette cell. The hardware and experimental procedures are described in Section 2. Measurements of large non-colloidal particle suspensions in a Newtonian carrier fluid are demonstrated, and the results compared to high field Rheo-NMR acquisitions at matched shear rates in Section 3. For illustration we compare our experimental data to the model of Morris and Boulay [42]. An assumption of this model, as with the other models described above, is that the suspension is locally Newtonian. However, disregarding issues of particle migration, many suspensions exhibit non-Newtonian behavior even at moderate volume fractions and where inertial and Brownian effects are negligible [47]. We observe such effects in our system and subsequently a departure from the predictions of the suspension-balance model.

2. Experimental

2.1. Low field Rheo-NMR

The low field NMR system comprised a $B_0 = 0.25$ T permanent magnet (Laplacian, UK) of SmCo planar disc poles enclosed in a rectangular iron yoke of dimensions $52 \times 55 \times 48$ cm and approximate mass 150 kg. The field strength corresponded to a resonance frequency of 10.7 MHz for ^{1}H. Thermal stability of the magnet was maintained at 28 °C by active heating and cooling. The vertical bore rf resonator had an inner diameter of 53 mm and active damping feedback pre-amplifier (MRF Innovations, UK) for improved response time and signal-to-noise ratio (SNR). The rf power was supplied by a 500 W CW amplifier (Tomco, Australia). Magnet homogeneity was improved by manual adjustment of an 8 channel shim current supply unit (Resonance Reasearch Inc., USA; model MXD-8). Planar gradient coils driven by high-performance audio amplifiers (AE Techron, USA; model 7224) provided a maximum gradient strength of $g_{max} = 0.125$ T m^{-1} (x and y axes) and $g_{max} = 0.14$ T m^{-1} (z axis). The water cooled gradient plates maintained the sample temperature at 25°C. The NMR Experiments were controlled by a DRX-TCP spectrometer (Oxford Instruments, UK) with custom pulse sequences and digital filters [48] optimized for imaging.

Spatially resolved concentration and velocity profiles were obtained using a standard spin-echo imaging sequence [49]. Gaussian rf pulses of duration $t_p = 1024$ μs were used to provide a vertical slice of 30 mm full-width half-maximum (FWHM) and a horizontal slice of 5 mm FWHM; the rf power was adjusted to provide $\pi/2$ and π pulses as required. For the velocity encoding, three gradient increments were used $(-g_v, 0, +g_v)$ to provide a robust calculation of phase shift. The gradient amplitude was scaled according to the Couette rotation rate to provide the same maximum phase shift in all experiments. The velocity-encoding gradient pulses, placed symmetrically about the refocusing rf pulse, had duration $\delta = 1.5$ ms and the observation time was $\Delta = 11.0$ ms. The frequency-encoded read gradient provided a field of view FOV = 59 mm over 256 pixels, such that the spatial resolution was $\Delta x = 229$ μm with 56 pixels across the Couette gap. The measured profiles extended over the full diameter of the Couette cell, providing antisymmetric velocity components on either side of the rotating center body. The magnitudes of the y velocities were averaged at the absolute $|x|$ coordinates (taking $x = 0$ to be the center of the Couette geometry), to provide $|\overline{v_y}|$ as a function of radius. A recycle time of 500 ms was used between scans, and 256 repeat scans were summed to improve the SNR, resulting in an acquisition time of approximately 2 min per gradient increment.

A custom Rheo-NMR Couette device was constructed for the low field magnet. The outer cylindrical body (stator) was gun-drilled from a rod of polyamide-imide (Torlon; Solvay, Belgium) to provide a high-precision machined part. The inner cylindrical body (rotor, or "bob") was machined from polyether ether ketone (PEEK). The Couette geometry had an outer radius of $r_o = 20.3$ mm and an inner radius of $r_i = 7.5$ mm, such that the gap was $r_o - r_i = 12.8$ mm with a gap ratio of $(r_o - r_i)/r_o = 0.63$. The total bob length was 252 mm. Rotation of the bob was achieved using a 12 V d.c. motor with the speed determined by manual adjustment of the voltage supply; an optical encoder in the motor was used to confirm the rotation rate. Rotation rates ranged from Ω = 3–40 rpm; the rotation rate was limited to a maximum of $\Omega = 15$ rpm when measuring Newtonian liquids due to the formation of Taylor vortexes at higher shear rates.

2.2. High field Rheo-NMR

A $B_0 = 7$ T vertical bore superconducting magnet, corresponding to 300 MHz for ^{1}H, was used for the high field Rheo-NMR studies. The imaging experiments were controlled by a Bruker DMX300 spectrometer. A bird-cage rf coil of inner diameter 25 mm was employed. Imaging gradients up to $g_{max} = 0.987$ T m^{-1} were available on all three axes. The sample temperature was maintained at 20 °C. A motion-compensated imaging sequence was used with Gaussian rf pulses of duration $t_p = 512$ μs, providing a vertical slice of 4 mm FWHM and a horizontal slice of 1 mm FWHM. A reliable phase shift measurement was achieved with just two gradient increments (0 and $+g_v$) due to the SNR and stability of the high-field instrument. The velocity-encoding gradient pulses had duration $\delta = 1.5$ ms and the observation time was $\Delta = 11.8$ ms. The frequency-encoded read gradient provided a FOV = 28 mm over 256 pixels, such that the spatial resolution was $\Delta x = 113$ μm, with 53 pixels across the Couette gap. As with the low field data, $|\overline{v_y}|$ was obtained as a function of radius from the antisymmetric velocity components on either side of the rotating center body. A recycle delay of 6 s was used and 4 repeat scans were summed, with an acquisition time of less than 1 min per profile.

A commercial Rheo-NMR device was used (Magritek, New Zealand and Bruker BioSpin, Germany), designed to fit the vertical bore of the high field magnet. Full details of this device are given in [8]. The radius of the stator $r_o = 9.5$ mm and the radius of the rotor