VELOCITY AND CONCENTRATION STUDIES OF FLOWING SUSPENSIONS BY NUCLEAR MAGNETIC RESONANCE IMAGING

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Abstract

Nuclear magnetic resonance imaging (NMRI) techniques were developed to study concentrated suspension flows. Some of the proposed tasks were completed and others partly completed before the funding was terminated. The tasks completed were 1) materials selection for imaging of both particle and fluid components, 2) pipe flow measurements, and 3) flows in complex geometries. The task tackled with good progress is to develop rapid imaging techniques by analog compensation of eddy currents generated by the gradient pulses and real-time image reconstruction from the rapidly obtained data. The most suitable combination of materials arrived at is pharmaceutical beads in silicon oil. Their relaxation times T_1 are sufficiently different to permit imaging the two components separately. The pipe flow experiment used 3 mm, neutrally buoyant, plastic particles, up to 40% by volume, in 80-90W transmission oil flowing in a 5 cm diameter pipe. A series of distances ranging from 60 cm to 6 m downstream from a commercial mixer was studied. The flow is fully developed at 6 m and the velocity and concentration profiles agree with the earlier lower resolution experiments. The eddy current compensation scheme works well for two channels and is being extended to eight channels including the uniform field compensation term. In addition, we have implemented a rapid reconstruction hardware that processes and displays images in a fraction of a second. For the

complex flow, we studied the flow of neutrally buoyant concentrated suspension past a step expansion and contraction in a cylindrical pipe. Interesting transition is observed at the expansion whereby the high fluids fraction outer layer before the expansion must spread to become the outer layer in the larger pipe. We discovered a spectacular effect of a high fluids fraction spike emanating from the center of the piston face as it approached the contraction. We have concluded that the effect does not depend on the presence of the contraction but is strictly an effect of the relative motion of the suspension with respect to the piston.

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Executive Summary

Nuclear magnetic resonance imaging (NMRI) techniques were used to study concentrated suspension flows. It was originally intended to develop sufficient technology to be able to measure the particle and fluid concentration and velocity distributions and profiles simultaneously in concentrated suspensions regardless of their optical opacity. Because the funding for the grant was cut during the second year of the project, not all of the targets were reached.

We have found that a combination of pharmaceutical beads in silicon oil is well suited for imaging both components. Because silicon oil is not inexpensive, the ideal flow geometry to study with this combination of materials would not be a pipe flow system but something along the lines of a couette.

The pipe flow experiment used 3 mm, neutrally buoyant, plastic particles, up to 40% by volume, in 80-90W transmission oil flowing in a 5 cm diameter pipe. A commercial mixer was used to homogenize the mixture at the beginning of the flow system. A series of distances ranging from 60 cm to 6m downstream from the mixer was studied. The flow is fully developed 6m downstream of the mixer and the velocity and concentration profiles agree with the earlier lower resolution experiments, i.e., blunted velocity profile and an elevated concentration of particles near the center.

The eddy current compensation scheme is needed to speed up the imaging sequences that require the magnetic field gradients to be switched. Time varying magnetic fields induce eddy currents in surrounding conducting surfaces which prevent rapid switching of the magnetic field gradients. Our hardware works well for

two channels, i.e., gradient components, and is being extended to eight channels including cross terms and the uniform field compensation term.

In addition, we have implemented a rapid reconstruction hardware that processes and displays images in a fraction of a second. Thus, in conjunction with a rapid imaging capability based on a good eddy current compensation scheme, it will be possible to view images nearly in real-time. At present, this rate is 1 Hz in our setup but we hope to push it to 10 Hz in the near future.

Finally, we studied the flow of neutrally buoyant concentrated suspension past a step expansion and contraction in a cylindrical pipe. Interesting transition is observed at the expansion whereby the high fluids fraction outer layer before the expansion must spread to become the outer layer in the larger pipe. Because of the large viscosity of the mixture, a piston rather than a pump was used to create the flow. We discovered a spectacular high fluids fraction spike ahead of the piston face as it approached the contraction. The effect does not depend on the presence of the contraction but is an effect of the relative motion of the suspension with respect to the piston. The high concentration of the fluids in the center of the pipe, caused by the spike, reverses within the smaller pipe after the contraction, as dictated by the normal migration of particles to the center of the pipe where the shear is a minimum.

Introduction

This is the final report for the above titled project which was initially funded for three years, approximately corresponding to Fiscal Years 95-97. It was fully funded for the first year, only partially funded for the second year, and no funds were received for the third year. Thus, the project was terminated part way through the second year.

The originally planned tasks were

1. Materials selection: To find suitable materials that can be imaged to yield velocity distribution in concentrated suspensions. [Months 1-12]
2. Horizontal cylinder flow: To perform suspension flow experiments in a horizontal rotating cylinder to establish the suitability of the particles selected in the first task. [Months 7-12]
3. Pipe flow: To obtain the velocity profile of particles in a flow of concentrated suspension in a pipe. [Months 12-24]
4. Fast imaging development: To develop a digital compensation scheme for attenuating the effects of eddy currents in order to do rapid imaging. [Months 1-18]
5. Flows in complex geometries: To measure the particulate behavior in steady flows in complex geometries. [Months 19-30]
6. Pulsatile flow: To study the concentration and velocity distribution of concentrated suspension undergoing pulsatile flow. [Months 25-36]

We performed all or parts of tasks 1, 3, 4, and 5.

Materials selection:

Our quest for an suitable combination of imageable particles in a carrier liquid which will not dissolve the particles has led us to try pharmaceutical particles in

silicon oil. This combination does seem to last long enough for adequate NMR measurements. The measurement of NMR parameters has led to the following findings. There is a relative chemical shift of 212.5Hz at a Larmor frequency of 80.34MHz which is approximately 2.7 ppm. We have done chemical shift imaging of static samples in the past and succeeded in resolving resonances of oil from water in rocks. However, we have never done this with flowing fluids. Because this is perceived to be of marginal feasibility, we have also measured the T_1 relaxation times to see if that is a better parameter for distinguishing the two components.

The most convenient experiment to perform to distinguish the two images is an inversion recovery. All spins are assumed to recover exponentially from the inverted state to the upright state with time constants T_1 depending on the physical and chemical states of the atom containing the nucleus. Thus, each spin species obeys

$$M(t)=M(\infty)[1-2\exp(-t/T_1)] \quad (1)$$

which means the magnetization starts from $-M(\infty)$ at $t=0$, the inverted state, and recovers to $M(\infty)$ at $t=\infty$. The magnetization $M(t)$ goes through zero when $\exp(-t/T_1)=1/2$. In the usual inversion recovery experiment, as used in clinical medicine, all the spins are inverted at $t=0$ and the recovery is allowed to proceed until the difference between the two signals is a maximum. We found that the T_1 values are different by a factor of around 3 for the two materials, i.e., the silicon oil is more than 1 second while the pharmaceutical beads are less than 1 second. If the T_1 's are taken to be 0.5 and 1.5 seconds, the time of the largest signal divergence is at 0.83s and the difference in signal is 39% of maximum.

This is a difference that is large enough that we can do an easier experiment. After the inversion, we wait until one of the components, the faster one, passes through zero. We perform an NMR imaging experiment at that instant which yields an image only from the component whose magnetization is not, yet, zero. The experiment is then repeated with a suitably longer delay to obtain the image of the other, longer T_1 , component. Intensity corrections have to be made according to what the nonzero magnetizations are at the appropriate times.

Figure 1 shows T_1 -separated images of 4 mm diameter pharmaceutical beads in a film can filled with silicon oil. All protons were inverted at the beginning of the experiment and the upper left image was made after a suitable delay that nulled the signal from the beads. The upper right image was made with a longer delay to null the signal from the silicon oil. In order to check the cancellation, the two images were summed to yield the lower left image. Much to our surprise, there is a hole on the left side of the pile of beads. Upon careful examination, we discovered a single glass bead among the pharmaceutical beads. Because glass gives rise to no proton signals, the sum shows an absence of any signal from the glass bead. The other faint variations in the intensities where the beads are arise from the shells of the pharmaceutical beads which yield protons signals with too short a T_2 to be captured in this experiment.

Figure 1. A container filled with silicone oil and spherical, liquid-filled pharmaceutical pills was imaged with an inversion-recovery sequence which exploits the T_1 difference between the materials. The upper left image is the oil, obtained by waiting to make the image until the inverted signal from the particles went through a null, while the upper right image is that of the particles, obtained the same way. The lower image is of the sum of the two showing the glass bead that was accidentally mixed in with the pharmaceutical particles.

Thus, we have proved the feasibility of obtaining images from both components of a two-phase mixture. This is a major precondition for obtaining velocity images of both particles and liquids in a concentrated suspension flow.

Pipe flow:

Some years ago, we performed NMR measurements of velocity and concentration profiles of suspensions in a circular pipe. (See Altobelli, *et al.*, J Rheol 35(5) 721, 1991.) We used a 2.54 cm diameter pipe and flowed suspensions with 7-800 μm plastic particles up to 40% by volume. The liquid was 80-90W transmission oil and the particles were heavier than the oil. We planned to perform additional experiments with 1) neutrally buoyant particles, 2) larger pipe diameter, and 3) imageable particles. We have now performed an experiment with the first two of these new conditions.

The original experiments were performed with non-neutrally buoyant particles for expediency. Although the results were fascinating and new, we needed to do the

neutrally buoyant case to compare with theories. As for doing the experiment in a larger pipe, this gives us, effectively, double the spatial resolution that we had before, yielding more details of the flow in the boundary layer next to the pipe. It also increases the scale of the experiment, requiring much more space, material, and a stronger pump.

This experiment was performed with Robert Hampton and Nick Tetlow, graduate students who are part of Alan Graham's group at Los Alamos National Laboratory. We studied neutrally buoyant suspensions of 3 mm Plexiglas spheres and a viscous, Newtonian, "three-component" suspending fluid. Flexible pipes with 5 cm ID were used in the most recent set of experiments. A flow loop which also incorporated a section of 5 cm ID rigid pipe was used in a preliminary experiment. A progressive cavity Moyno pump produced the flow which proceeded from the pump in series through a 9m flexible section, a 3m inline mixer, a 14m flexible section and an open reservoir that fed the pump input.

NMR measurements yielded axial velocity and fluid fraction profiles in slices at various positions of the pipe. The velocity and fluid fractions as functions of the radial coordinate were computed from the images by averaging the signal at each radius. The resulting plots, therefore, have S/N that decreases from the outside towards the center.

The flow loop was designed to allow measurements at a series of four distances from 0.6 to 6m from the outlet end of the inline mixer. At each distance, measurements were made at pump settings of 15 and 30, which correspond to average velocities of approximately 5 and 10 cm/s. This set of ten experiments was performed for five different concentrations plus the pure fluid; the overall solids fraction used was 0, 0.1, 0.2, 0.3, 0.4, and 0.45.

Figure 2a is the radial dependence of the axial velocity for the pure fluid and for the 45% concentration at 10 cm/s average velocity and the farthest location (6m) from the mixer. V_{ave} is the average velocity, ϕ is the local solids fraction, and R_o is the radius of the pipe. The figure shows that the peak axial velocity is twice the average velocity V_{ave} for the pure Newtonian fluid, as expected. Also, the concentrated suspension

Figure 2. Velocity (top) and fluid fraction (bottom) plots calculated from NMRI data. Radial dependence of the axial velocity for the pure fluid, normalized to the average velocity, and for the 45% concentration at 10 cm/s average velocity and the farthest location (6 m) from the mixer are shown on top. Fluid fraction $1-\phi$ as a function of the radius for the same two conditions below.

velocity profile is quite similar to the pure fluid near the wall but decreases significantly towards the center, as shown in our earlier work.

Figure 2b is the fluids fraction $1-\phi$ as a function of the radius for the same two conditions as in the upper figure. The measured fluids fraction is nearly flat for the

pure fluid while it is quite inhomogeneous, as expected, for the concentrated suspension. At this S/N, it is difficult to draw any conclusions about the profile close to the center of the pipe (see the discussion, above, about the S/N), but the solids fraction increases quite sharply towards the center, in good agreement with our earlier work, again.

Fast imaging development:

Gradient compensation

Despite its many advantages, the usual MRI is relatively slow, with most applications limited to stationary objects or to objects undergoing periodic motion imaged synchronously with the motion. Attempts to speed up imaging have to confront the eddy currents induced in conducting surfaces by the pulsed magnetic fields associated with the gradients. Such eddy currents recover slowly and adversely affect imaging speed and image quality.

A strategy in combating eddy current problems is to alter the shape of the driving waveforms going to the gradient coils so that the resulting “distorted” waveform is the desired one. This, in principle, is a standard procedure in electrical engineering and only requires the determination of a transfer function for the system from a known input and a measured output, followed by a modified input corrected for the newly determined transfer function. For eddy currents, the common compensation scheme consists of adding suitable amounts of RC filtered signal to the input, with the RC time constants matched to each eddy current decay times.

Unfortunately, the situation is much more complex in the real world because of the non-ideal geometries involved. Specifically, the conducting surfaces on which eddy currents are generated are neither homogeneous, symmetric, nor have the same conductivity and time-constants. As a consequence, the induced eddy currents are numerous and do not have the same symmetry as the original inducing field or even as each other. Thus, compensating the input waveform of a particular gradient component, even for all the time-constants, can only correct the induced gradient corresponding to that component. The other components of induced gradients must be corrected by separate hardware that are specific to those components as explained below.

In order to understand the proposed compensation, consider a y-gradient $G_y(t)$. After the gradient $G_y(t)$ is turned off, we model the eddy current generated magnetic field by

$$B_y(t) = b_{oy}(t) + xg_{xy}(t) + yg_{yy}(t) + zg_{zy}(t) + \text{higher order terms in } x, y, z, \quad (2)$$

where $b_{oy}(t)$ is a spatially uniform magnetic field along B_0 induced by $G_y(t)$, $g_{xy}(t)$ is a linear magnetic field gradient in x-direction induced by $G_y(t)$, and $g_{yy}(t)$ and $g_{zy}(t)$ are y and z gradients induced also by $G_y(t)$. In this work, we will ignore higher order terms in x, y, z. An equation like the above exists for each driving gradient. The general interaction can, thus, be represented by a matrix with up to 12 elements relating the input and output gradients. Usually, some of these elements will be zero but each

nonzero term requires a hardware compensation circuit, each with an array of time constants and weights.

Fig 3A. Uncompensated System

Fig. 3B Compensated System

Figure 3. Block diagrams of the model used in eddy current compensation are shown.

In the following paragraphs, we illustrate the eddy current correction for one channel, y , with the cross terms neglected. Neglecting the cross terms in Eq.(2) will leave only two contributions, a uniform field $b_{oy}(t)$ and a y -gradient $g_{yy}(t)$:

$$B_y(t) = b_{oy}(t) + y g_{yy}(t) . \quad (3)$$

This model before compensation is shown in Fig. 3. The transfer functions $G_{yy}(s)$ and $G_{oy}(s)$ model the magnetic field (including the eddy-current induced magnetic fields) generated by current $i_y(t)$. Ideally $G_{yy}(s)$ should be constant and $G_{oy}(s) = 0$.

The output of $H_{yy}(s)$ goes to the y-gradient coil and through the compensator $H_{oy}(s)$ to the B_o coil. The magnetic field $B_y(t)$ is now given by

$$B_y(t) = b_{oy}(t) + yg_{yy}(t) + b_{oo}(t) + yg_{yo}(t). \quad (4)$$

Our goal is to design the compensators $H_{yy}(s)$ and $H_{oy}(s)$ so that $B_y(t) = 0$ after $i_y(t)$ is shut off. $H_{yy}(s)$ is chosen to be

$$H_{yy}(s) = a_0 + \sum_{i=1}^{12} a_i \frac{s}{s + p_i}, \quad (5)$$

where the gain constants a_i , $i = 1, 12$ are chosen to cancel the eddy current effects. These values are controlled by digital-to-analog converters. The value of a_0 is part of the gradient channel gain and can be set to unity. The time constants $1/p_i$ are fixed values, chosen to be equal to 2^{i-1} ms, $i = 1$ to 12 , a geometric time series. The different time constants are realized by RC circuits. $H_{oy}(s)$ is chosen with a similar structure and with the same time constants.

$$H_{oy}(s) = \sum_{i=1}^{12} b_i \frac{s}{s + p_i}, \quad (6)$$

$H_{yy}(s)$ and $H_{oy}(s)$ are determined by iteration. $H_{oy}(s)$ is determined first. The sample probe is placed at the electrical center of the y-gradient coil ($y=0$). Then

$$B_y(t) = b_{oy}(t) + b_{oo}(t). \quad (7)$$

$H_{oy}(s)$ is determined to make $B_y(t)$ equal to zero by suitably generating $b_{oo}(t)$ from the B_o coil to cancel $b_{oy}(t)$. Let $B_{yu}(t)$ be the uncompensated eddy current response and $B_{yi}(t)$ be the eddy current response with $b_i = 1$, for $i = j$ and zero for other i values and $a_0 = 0$. Then the basis function corresponding to the time constant p_i is given by

$$h_{oy}(i, t) = B_{yi}(t), \quad (8)$$

for $i = 1, 12$. The values of b_i , $i = 1, 12$ are then determined by minimizing the least squares error

$$E_o = \int (B_{yu}(t) + \sum_{i=1}^{12} b_i h_{oy}(i, t))^2 dt. \quad (9)$$

This is a linear least squares problem and the coefficients b_i can be determined by matrix inversion. Note that we do not use the functions $\exp(-p_i t)$ corresponding to the time constants $1/p_i$, but use the experimentally determined basis functions $h_{oy}(i, t)$. By doing so, we have accounted for the gradient coil transfer functions $G_{oy}(s)$ and $G_{oo}(s)$.

The next step is to determine $H_{yy}(s)$. The sample probe can be moved a sufficient distance off-center to have sensitivity to the eddy current response while keeping the measurements within the linear range of the technique. The method for calculating $H_{yy}(s)$ is similar to that used for calculating $H_{oy}(s)$. We first experimentally measure the uncompensated eddy current response $B_{yu}(t)$ (with $a_i = 0$, $i = 1, 12$) and the basis functions $h_{yy}(i, t)$, $i = 1, 12$ with the previously calculated $H_{oy}(s)$. Then, as before, a_i are determined from the linear least squares problem by minimizing

$$E_1 = \int (B_{yu}(t) + \sum_{i=1}^{12} a_i h_{yy}(i, t))^2 dt. \quad (10)$$

The above calculation for $H_{oy}(s)$ and $H_{yy}(s)$ can be iterated for better values, namely, go back to the electrical center and calculate the improvement to $H_{oy}(s)$, measure a new set of basis functions around the operating point of the previous compensation, and follow this, as before, by linear least squares minimization. The same is repeated for $H_{yy}(s)$.

Figure 4. Block diagrams of NMR imaging systems before and after adding the new compensation system.

The linear least squares fitting and loading the appropriate gain constants to the 12 serial MDAC's (multiplying digital to analog converters) will be done through a parallel port of an IBM PC compatible computer. The use of MDACs with serial inputs greatly simplifies the interfacing, especially when we consider that 12 bits are required to specify the amplitude of each of twelve time constants. The proposed scheme not only speeds up the minimization of the eddy current responses but its precision and reproducibility allow for the tailoring of the eddy current corrections to different experimental conditions such as different samples, rf coils, gradient coils, and magnets. Figure 4 shows block diagrams of an unmodified imager/spectrometer and the proposed add-on hardware to both measure and compensate the eddy currents.

Rapid image reconstruction

In addition to the ability to obtain NMR data in the shortest possible time, it is desirable to be able to rapidly construct images from the data so that it can be seen in real-time as flows are taking place. With the assistance of Prof. K. Kose of Tsukuba University, we have designed and constructed a real-time image reconstruction system out of a Pentium PC running Windows 95. The reconstruction and display required for a 128x128 image was 350 ms.

Figure 5. Successive images (a) through (f) show an air bubble emerging , detaching and rising to the top of a liquid soap filled bottle. The interval between images is 1.12 s.

At the moment, the fastest image sequence we can use is FLASH with acquisition time of approximately 770 ms. This would allow image acquisition, processing, and display with a repetition time of 1.1 seconds. Figure 5 shows six successive 89 mm x 89 mm FLASH images of an air bubble in a Dial soap solution taken and displayed 1.12 seconds apart. The bubble forms at the end of a plastic tube in frame (b) and rises on the left side in (c). The bubble moves out of the 6.5 mm thick slice which is being imaged in (d) and also shows some susceptibility artifacts above and below it. It reaches the top of the bottle in (f) where it comes back into the slice. The average velocity of the bubble between (c) and (d) was 2.27 cm/s but decreases to 0.65 cm/s between frames (e) and (f). The real-time reconstruction was useful in order to adjust the flow rate to make bubbles of appropriate size.

In this scenario, the majority of the time is spent in acquiring data. Therefore, we will continue in our quest to shorten the acquisition time which has been our goal for a long time. Our target is to get it down to less than 100 ms which would allow full acquisition and display at least four times per second.

Flows in complex geometries:

Shear-induced migration of suspended particles can cause large particle concentration gradients in what initially was a well-mixed suspension (the "demixing" of the suspension). Since the effective viscosity of a suspension depends strongly on particle concentration, this demixing can dominate the rheology of the system, and complicate quality assurance issues when a well dispersed system is desirable. Direct measurements of both velocity and particle distribution in concentrated suspensions are made difficult by the large number of particle-fluid interfaces (which scatter light, sound waves, and particle beams). We, at Lovelace, have pioneered the use of nuclear magnetic resonance (NMR) imaging to make noninvasive, accurate, and reproducible measurements of particle and velocity distributions in highly filled suspensions. Despite this increase in the quantity of flow data available, the behavior of suspensions in complex flows remains largely unknown.

We used NMR imaging to measure the concentration and velocity profiles of two suspensions flowing through a straight pipe into a sudden 4:1, axisymmetric contraction, and out of a sudden 4:1 expansion. Both suspensions are 50% by volume of roughly monodisperse, spherical particles (100 or 675 nm in diameter) neutrally buoyant in a viscous Newtonian liquid. In this "creeping" flow, inertial, Brownian, and surface forces are negligibly small. We expected and found that shear induced migration and the attendant non-uniformities of viscosity would result in a flow field that is significantly modified from that of a single-phase Newtonian liquid.

The extruder, shown schematically in Figure 6, consists of three 38 cm long sections joined end-to-end. Flow proceeds from an upstream reservoir pipe (Region 1) through a smaller diameter pipe (Region 2) into another larger catch pipe (Region 3). The larger-diameter pipes are 5.08 cm ID polymethyl methacrylate (PMMA) tubing with 0.635 cm-thick walls. The smaller pipe is made from 6.35 cm-OD PMMA rod with a 1.27 cm-diameter hole bored through the center.

Figure 6. The extruder assembly is constructed from three 38 cm long sections press fit together. The inner diameters of the sections were in the ratio 4:1:4. The suspension was loaded into the extruder between two moveable pistons. The entire assembly was inserted into the horizontal bore magnet so that the region of interest was centered in the NMR sensitive region.

As shown in the figure, the contraction and expansion are abrupt. Two identical pistons, sealed with O-rings, slide in the larger pipes. A long push rod connects a motor-driven screw located across the room (to avoid the magnet). Initially, the reservoir tubing and smaller pipe are filled with suspension, and the free piston on the expansion end of the pipe is near, but not necessarily adjoining, the expansion joint. The entire test section is placed coaxially in the magnet and braced at the end opposite the motor. The screw pushes the piston at a steady rate in the reservoir tubing, forcing the suspension to flow through it and into the contraction, through the smaller tube, and into the expansion. The plunger moves at a constant velocity of 0.0625 cm/s, resulting in a mean velocity of 1.0 cm/s in the smaller pipe.

The suspensions are 50%, by volume, of PMMA spherical particles suspended in a viscous liquid with the same density as the particles (1.18 g/cm^3). Two sets of particles were used, both with fairly broad, but unimodal, distributions of diameters. One, Diakon MG102, has a mean diameter of 675 nm while the other, Lucite 4F, has a mean diameter of approximately 100 nm. The suspending liquid is a solution of practical grade 1,1,2,2 tetrabromoethane (14.07% by weight); UCON oil (H-90,000), a polyalkylene glycol (35.66% by weight); and Triton X-100, an alkylaryl polyether alcohol (50.27% by weight). This liquid exhibits Newtonian rheology with a viscosity of 4.95 Pa s at 23.15 °C.

The basic NMRI techniques of velocity measurement used in this work were similar to those used earlier in our granular flow experiments [M. Nakagawa, S. A. Altobelli, A. Caprihan, E. Fukushima, and E.-K. Jeong, "Non-invasive Measurements of Granular Flows by Magnetic Resonance Imaging," *Experiments in Fluids*, 1993, 16:54-60] but, hardware improvements allowed us to create images in shorter times (static images were performed in 4 minutes and each velocity component was measured in two minutes). Image slice thickness was 0.5 cm, and in-plane spatial resolution was approximately 0.2 mm yielding a voxel size of $0.5 \times 0.5 \times 5 \text{ mm}^3$. As with previous work, the time-averaged location and flow of the liquid phase of suspensions is measured with a NALORAC Quest 4400 imager interfaced with a 1.9 T, 31 cm horizontal bore magnet

We imaged various slices of the suspension-filled device within the magnet, including three cross-sections along the length of Region 2, single cross sections in Regions 1 and 3, and slices containing the axis of symmetry in Regions 1 and 3, (Region numbers refer to Figure 6). Velocity images were obtained with conventional pulse gradient spin-echo imaging sequences in which a single, selectable, velocity component is encoded in the phase of the image; static reference images were used to remove extraneous contributions to the phase. The velocity images represent a time average whereas the concentration images show the conditions obtained after moving the piston a known distance. Static images confirmed that the suspension in the upstream reservoir was initially well-mixed.

Figure 7 shows static concentration images of the suspension of larger particles (675 μm) flowing through the extruder. There are two slice orientations: circular cross-sections (a, g) and horizontal slices containing the center line (b-f, h-j). Because our NMR imager has an effective region for imaging that is a 7-8 cm diameter sphere, the extruder extends beyond this region. Thus, the horizontal slices have image intensity that drops off on both sides of each image that are artifacts of the magnet homogeneity, coil dimension, filter, etc.

For these large particles, we find that the contraction affects the flow upstream very little while the expansion leads to an interesting concentration and flow pattern. Images a-f were taken in Region 1, the contraction end of the model. Images a and b show that the suspension is initially well mixed and images c and d, obtained after the piston moved 2 and 4 large diameters, respectively, show that the distribution of solids remains virtually unchanged. Even images e and f, taken after 6 and 6.25 diameters of piston travel, do not show any concentration inhomogeneity associated with the contraction.

Much to our surprise, a sharp high fluid-fraction spike (light colored streaks, corresponding to fluid fraction of 0.55-0.6) appeared along the model centerline when the piston face approached the field of view, i.e., to within a diameter of the contraction (note: the piston can be seen in image f). We believe that this feature is caused by the interaction of the suspension with the piston face and has nothing to do with the presence of the contraction.

The expansion region, Region 3, shows a more dramatic effect of particle migration than Region 1. Figure 7(g), a concentration cross-section 0.5 cm downstream of the expansion, shows a light colored ring of high fluid content,

surrounding a core having a higher solid fraction. As the suspension travels from Region 2 into the larger pipe of Region 3, the high liquid-rich regions along the wall of the small pipe expand outward towards the wall of the large pipe, as shown in Fig. 7(h-j). In Fig. 7(i), the high fluid fraction lines may hint of the existence of recirculation zones immediately downstream of the expansion. After the plunger has traveled 6 large diameters (30 cm) the particles in the "corners" appear to have been largely swept away, as shown in Figure 7(j), leaving large, somewhat stagnant, regions with 10-15% higher liquid content.

Figure 7. Static NMR images of the contraction (a-f) and the expansion (h-j) regions for a suspension that is 50% by volume of 675-mm-diameter particles. A and g are transverse images 0.5 cm and 1.5 cm away from the contraction and the expansion, respectively. The remainder are horizontal slides containing the centerline. Images a and b show the initial condition. Images c, d, e, and f show the fluid fraction after the piston has moved 2, 4, 6, and 6.25 large section diameters, respectively. Images h, i, and j show the expansion region after 2, 4, and 6 diameters of piston travel.

Figure 8 shows profiles of fluid fractions at normalized locations Z/L of 10%, 50%, and 95% along the small tube (Region 2 of Fig. 6), where $L=38$ cm, for these 675 mm spheres. The particles migrate toward the center, as expected, even starting from an opposite distribution caused by the previously mentioned spike of high fluid fraction in the center.

Figure 8. Fluid fraction as a function of the radial coordinate for three stations along the small tube for the suspension of 675-mm spheres. Z is the distance along the small tube and L is the length of the small tube. At the upstream station the fluid rich region along the axis is seen. In the central and downstream profiles solids progressively accumulated near $r=0$.

When a suspension of 50% by volume of 100 mm-diameter particles was pushed through the contraction and expansion, the concentration inhomogeneities created by the migration of particles were much less than for the suspension of 675 mm particles described above. Figure 9 shows static images of the 100 mm suspension after a 6 large section diameter push. Both effects seen with the larger particles, namely, the liquid-rich spike in front of the piston face in Region 1 and the divergence of the fluid-rich layer at the expansion, are discernible albeit faintly. The top image shows the light-colored feature that has formed in front of the piston face while the lower image shows streaks of enhanced fluid fraction emanating from the expansion at a much sharper angle than they did for the 675 mm suspension.

Figure 9. Images of liquid fraction in the contraction region (top) and the expansion region (bottom) after 6 diameters of piston travel in the 100 mm suspension show small areas of slightly enhanced fluid fraction at the driven piston face (top) and at the expansion. The nominal concentration of solids in this suspension is 50% by volume.

Measurements halfway along the small tube of Region 2 showed a blunt velocity profile but no detectable particle migration, except for a thin fluid-rich layer along the wall, as shown in Figure 10. The suspension, exhibits a blunt profile that can be represented by

$$V_r(r) = \bar{V} \left(1 + \frac{2}{2.8} \right) \left(1 - \left(\frac{r}{R_s} \right)^{2.8} \right), \quad (11)$$

where R_s is the radius of the small tube. All of the velocity profiles observed in the small tube section were similar.

Figure 10. Fluid fraction (asterisks) as a function of radius at the central site in the small tube for the 100 mm suspension is nearly constant. The velocity profile (pluses) fits a power law profile (solid curve).

Orthogonal velocity components V_z along the axis of the model and V_x transverse to it in the horizontal plane were measured using the horizontal slice imaging sequence. The top images of Fig. 11 show the longitudinal velocity component V_z , scaled between 0 and 2 cm/s. The suspension of 100 mm spheres are shown on the left, and the 675 mm suspension are shown on the right. The lower two images of Fig. 7 show the transverse velocity component V_x whose absolute value is the radial velocity component. These velocity measurements confirm the much steeper angle of divergence of the expansion flow in the suspension of smaller spheres than in the suspension of larger spheres as it was observed in the concentration images as mentioned earlier.

When the driven piston face entered the imaging region, a filamentous fluid-enriched feature was always observed along the model axis, starting from the piston face. This effect was much greater for the suspension of larger particles and is a spectacular, straight and thin, fluid-rich spike. To our knowledge, this effect has never been seen before. We have now confirmed in a separate experiment that the spike forms in front of the piston and travels with it, without regard for any downstream contraction.

Figure 11. Images of the axial (top) and transverse (bottom) velocity components for the 100 mm (left) and 675 mm (right) suspensions are shown. Dark represents fast axial velocity while light represents slow axial velocity. For the transverse velocity, dark represents velocities towards the top of the image and light towards the bottom. Velocity units are in cm/s.

Flow into the abrupt contraction by itself did not produce observable particle migration, but flow in the small tube eventually resulted in migration of the large particles toward the axis of the model and concomitant migration of the fluid toward the wall. Migration in the small tube is noticeable compared to the large pipe of Region 1

because the shear rate in the small tube is much higher than in the large tube. Velocity profiles were blunt, even for the smaller particles, compared to fully developed Newtonian fluid profiles, and did not appear to change along the length of the small tube. These observations are at least qualitatively in agreement with predictions based on shear induced particle migration as described by Leighton and Acrivos [Leighton, D. and Acrivos, A., The Shear-Induced Migration of Particles in Concentrated Suspensions. J. Fluid Mech. 1987, 275, 155-199.]

The inhomogeneities created in the small tube of Region 2 were convected into the larger diameter expansion in Region 3. Static images of fluid fraction and two-dimensional velocity measurements indicate that the flow field in the expansion region depends more strongly on the particle size than does the flow in the other sections of the model. In the small-sphere suspension, the flow diverges more sharply at the expansion than the flow in the larger sphere case, resembling a pure Newtonian fluid. Our two suspensions were made to have the same bulk effective properties but they had vastly different size particles. We conclude that interparticle interactions affect multiphase flows and this effect is made large or small depending on the particle size. Thus, we cannot predict suspension behavior based only on bulk, or "effective", properties.

In conclusion, we can induce complex spatial variations in particle concentration by flowing a suspension through sudden contractions and expansions. The practical ramifications are that it may be difficult to maintain a well-mixed suspension while filling a vessel from an inlet tube having a cross-section smaller than the vessel. The demixing effects are much larger in suspensions of coarse particles than in suspensions of finer particles.

Conclusions:

All in all, NMRI yields unparalleled data for these concentrated suspension flows, both in terms of overcoming optical opaqueness and being able to measure both velocity and concentration in the same experiment.

Publications emanating from this grant

Kose, T. Haishi, A. Caprihan, E. Fukushima, "Real-Time NMR Imaging Systems Using Personal Computers," J. Magn. Reson. 124, 35-41 (1997).

E. Hampton, A. A. Mammoli, A. L. Graham, N. Tetlow, and S. A. Altobelli, "Migration of particles undergoing pressure driven flow in a circular conduit," in press, J. Rheol., 97.

S. Altobelli, E Fukushima and L. Mondy, "Note: NMR Imaging of Particle Migration in Suspensions Undergoing Extrusion," submitted to J. Rheol., 97.

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