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JOURNAL OF FOOD ENGINEERING

Journal of Food Engineering 78 (2007) 1117-1126

www.elsevier.com/locate/jfoodeng

Particle image velocimetry applied to suspensions of millimetric-size particles using a vane-in-a-large-baffled-cup rheometer

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Received 5 March 2005; accepted 19 December 2005 Available online 28 February 2006

Abstract

Millimetric particles suspended in four different fluids were sheared and video-recorded using a controlled-stress rheometer with a four-blade vane-in-a-large-cup. Experimental situations where the suspension next to the vane moves as a solid cylinder, i.e., where the Couette analogy can be applied, were determined. Velocity fields were measured by particle image velocimetry (PIV) in various suspensions: resin particles in oil (creamed particles), resin particles in water (settled particles), acetate particles in glycerin, and uniformly suspended gelatin particles in a commercial beverage. Data were expressed in terms of the Reynolds number calculated from experimental angular velocity measurements. The suspensions revealed flow patterns which include a considerable amount of vortices initially originating close to the millimetric-size particle bed and thereafter distributing and spreading within the large-baffled-cup. Flow characterization with a vane-in-a-large-cup was only possible for acetate particles in glycerin undergoing laminar flow, as verified by PIV and magnitude of the Reynolds number. Average shear stress and shear rate values were calculated and results for the suspension viscosity were in agreement to those predicted by the model of Happel (Viscosity of suspensions of uniform spheres. *Journal of Applied Physics*, 1957, *28*(11), 1288–1292).

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Keywords: Vane viscometer; Particle image velocimetry; Suspension rheometry

1. Introduction

The flow behavior of multiphase systems of complex fluids (foods, fermentation broths, suspensions) using mixer techniques (Cullen, O'Donnell, & Houska, 2003) has been given considerable attention. In some cases, the material in contact with the mixer impeller undergoes a rigid-solid motion, so that the flow between the hypothetical cylinders formed within the mixer possesses similarities to the simple-shear flow in a Couette geometry (Barnes & Nguyen, 2001). Using a vane-in-a-small-gap, Barnes and Carnali (1990) demonstrated that the torque on the vane was the same as the torque on a solid cylinder of the same diameter

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in highly shear-thinning fluids, confirming the similarity to simple shear flow.

Studies dealing with vanes-in-large-gaps are scarce in the reported literature (Baravian, Lalante, & Parker, 2002). Characteristics of some popular vane-in-a-cup systems are, among others, the following: vane heights, between 1 and 3 times the vane diameter (as recommended by Nguyen & Boger, 1983) and vane gaps, between 1.23 and 4.85. In general, most of the studies have been related to the yield stress determination of dispersed (suspensions, foams, gels, emulsions) or multiphase (fermentation broths) systems (Ainchinger et al., 2003; Baravian et al., 2002; Keentok, Milthorpe, & O'Donovan, 1985; Kovalenko & Briggs, 2002; Liddel & Boger, 1996; Nguyen & Boger, 1983, 1985; Servais, Ravji, Sansonnens, & Bauwens, 2003).

In a previous study the vane-in-a-large-cup was successfully tested for rheological characterization when laminar

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flow is maintained, and the absence of the baffled-cup effect was demonstrated. Moreover, this vane-in-a-large-cup device proved to be a successful alternative to characterize flow properties of suspensions containing coarse particles (mainly discs, as for example, in Mexican sauces, Martínez-Padilla & Rivera-Vargas, 2006).

When coarse particles are added to Newtonian fluids, a non-homogenous two-phase flow is observed, due to the interactions of millimetric-size particles. Since experimental data on flow in multiphase fluids is needed due to its complexity, particle image velocimetry (PIV), a laser-based technique, is useful to provide velocity data and hence flow patterns.

PIV is based on a laser beam which sweeps across the flow field at a known frequency. The fluid is seeded with micron-sized particles which are illuminated by successive sweeps of the laser beam. Images of the particles in motion are then recorded by a camera. The negatives of the flow field yield velocity vector fields, from which the stream function and vorticity contours can be calculated. Both small and large-scale structures in the flow field can be accurately identified. The use of PIV allows the acquisition of multiple frames closely resolved in space and time, relative to the principal scales of the unsteady motion (Shiang, Lin, Öztekin, & Rockwell, 1997). This optical technique was selected since it is non-invasive and can provide velocity measurements and multiple spatial points in planar (2-D) fields. In our work, the coarse millimetric-size particles were used as tracer particles and the laser beam was not required to video-record the particle flow.

The objective of this study is to obtain the flow patterns of suspended coarse particles in different fluids using the PIV technique, when these are subjected to flow in a vane-in-a-large-baffled-cup rheometer. Particular attention is given to cases in which the suspension in the vane moves as a rigid cylinder, i.e., systems where the Couette analogy can be applied. Millimetric-size particles were suspended in four liquids: glycerol and oil (moderately viscous fluids), water (low-viscous fluid) and a commercial beverage (visco-elastic fluid). The proposed large cup accounts for the fact that the gap between cylinders must be sufficiently large and even much larger than the size of the coarser particles for an adequate characterization of the flow behavior.

2. Materials and methods

Four suspension systems (500 mL) were prepared with three Newtonian fluids, glycerol (Fermont, México), oil (SAE 15W-40, Akron, México), distilled water and a viscoelastic fluid (obtained from a commercial beverage, Atomos by Bioprocess, México). Particles were semi-spherical resin particles (Dupont, México), semi-spherical gelatin particles (from the same commercial beverage), and spherical acetate particles (Engineering Laboratories, USA). Three situations were observed: (a) particles were settled (resin particles–water, 6.5% w/w), (b) particles were creamed (resin particles–oil, 8% w/w), and (c) particles were uniformly suspended (acetate particles–glycerin, 20% w/w; and gelatin particles–commercial beverage, 8.5% w/w).

2.1. Physical properties

The relative fluid density, using the water density as the reference at a given temperature, was measured with a DMA 38 densimeter (Paar, USA). The particle density was measured by picnometry and the particle equivalent diameter and particle sphericity were measured with a digital micrometer (Mitutoyo, Japan, Koichi, Keishi, & Ko, 1991).

Shear viscosity of the Newtonian fluids was measured in a controlled stress rheometer (Haake Rotovisco RT 20, Germany) at 25 or 30 ± 0.1 °C using the Couette geometry with conical bottom cylinders (Z20), bob-and-cup radius of 10 mm and 10.85 mm (DIN 53019), respectively. Temperature was controlled using a water circulating bath (F12 Julabo, Germany). Viscoelastic and flow properties of the commercial beverage were measured with a controlled low-stress rheometer (Low stress LS100, Paar Physica, USA), with a double gap concentric cylinder (DG10) fixture (48 mm and 50 mm of internal and external diameter, respectively; 36 mm in length and a radii ratio of 1.0417). The same protocols previously reported were followed (Martínez-Padilla, López-Araiza, & Tecante, 2004).

2.2. Particle image velocimetry

A Hi-8 digital video-camera (Sony, Argentina) was used to video-record (30 frames/s) a lateral view of the large cup containing the suspension under study. The cup has a vertical scale that was included in the recorded images. Only the flow region where the particles are observed was recorded. Illumination was provided by a light beam. Video-recordings were digitalized with XCAP Imaging software (Epix, USA) to obtain the frame images. A Flow-Map PIV software (Dantec Measurement Technology A/S, Denmark) was used to analyze each frame image to calculate the average movement of each particle between correlated frame images to obtain average-motion vector maps. These maps, related to the elapsed time, result in average-velocity vector maps that can be readily transformed into flow patterns.

2.3. Vane in-a-large-cup rheometer

All tests were carried out in a controlled stress rheometer (Rotovisco RT20, Haake, Germany). A schematic representation of the vane-in-a-cup system used in the present study is shown in Fig. 1. A four-blade-vane geometry (FL40), 40 mm diameter and 55 mm height, was used. The blades were 0.9 mm thick and the hub had a radius of 12 mm. A baffled-cylindrical container (100 mm internal diameter, 100 mm length, with eight enlarged cubic baffles, $10 \times 10 \times 100$ mm placed symmetrically, with an effective cup diameter of 80 mm) was used as a large cup. To avoid



Fig. 1. Schematic representation of the vane-in-a-large baffled cup.

end-effects, the vane top was placed at the fluid surface, thus leaving 35 mm between the vane's end and the base of the cup.

Two shear programs were used; the first one for settled or creamed particles and another one for suspended particles. In program A for settled or creamed particles, samples were pre-sheared in two steps to guarantee a homogenous initial structure before video-recording.

First, samples were sheared at 2.212×10^{-2} N m for 60 s where flow was observed before 10 s (step 1). Second, an oscillatory pre-shear at constant frequency (6.3 rad s⁻¹) and a constant small torque of 2.212×10^{-3} N m during 400 s was applied (step 2). These conditions were determined in previous tests through dynamic shearing until reproducible and constant storage (G') and lost (G'') modulus were obtained (constant tan $\delta = G''/G'$). At the end of the pre-shearing stage, a particle bed was observed (top or bottom). During video-recording, a constant torque as a function of time was applied to the sample, 1.106×10^{-3} N m or 4.423×10^{-3} N m.

In shear program B for homogenous suspensions, a linear increase-decrease of the angular velocity was applied (from 0.02 to 10 s^{-1} ; from 0.02 to 20 s^{-1} , and from 0.02 to 30 s^{-1}) during a total time of 120 s. Finally, a steady flow curve was obtained within the same torque range. Video-recording was carried out during this stage. Three angular velocity ranges were studied depending on the limits imposed by the onset for turbulence.

To verify the amplitude of the laminar flow region, the vane Reynolds number $(Re = \rho \Omega (r_1)^2 / \eta$, where ρ is the fluid density, η is the fluid shear viscosity, r_1 vane radius) was estimated. The average shear rate and average shear stress for this vane-in-a-large-cup were calculated at the given angular velocity (Ω) and the measured torque (M) (Couette analogy), when possible, using Eqs. (1) and (2), respectively (Steffe, 1996)

$$\bar{\sigma} = \left(\frac{(1+\alpha^2)}{4\pi r_2^2 h}\right) M = F_{\sigma} M \tag{1}$$

$$\bar{\tilde{\gamma}} = \left(\frac{(\alpha^2 + 1)}{(\alpha^2 - 1)}\right)\Omega = F_{\gamma}\Omega$$
⁽²⁾

where h is the vane height, α is the r_2/r_1 is the effective cup radius/vane radius. Shear geometric factors F_{σ} and F_{γ} were modified in the software.

3. Results and discussion

3.1. Physical properties

Physical properties of fluids and particles are summarized in Table 1. Sphericity of gelatin particles is close to 1. The equivalent circle diameter of gelatin and resin particles is 4.1 mm while that of acetate particles is 1 mm. Viscosity of the three Newtonian fluids is widely different: a fluid with very low viscosity (water), a low-viscosity fluid (oil) and moderately viscous fluid (glycerin). The mechanical spectra of the commercial beverage render the viscoelastic properties (Fig. 2). Characteristic features of a weak gel, as described by Lapasin and Pricl (1995), reveals that G'and G'' are independent of frequency and G' is one to two orders of magnitude larger than G''. Nevertheless, in this case G'' has larger frequency dependence than that of G'. The elastic modulus is larger than the viscous modulus in the range of working frequencies, revealing clear viscoelastic properties. These properties stabilize the suspension of coarse gelatin particles in the commercial beverage. When the suspension was submitted to a shear stress ramp, no flow is observed up to a yield stress of 0.14 Pa. Thereafter, the viscosity of the system rapidly decreases down to a very low value ($\sim 8 \text{ mPa s}$). This behavior is similar to that observed in xanthan-gellan binary fluid gels at a total concentration of 0.1 wt% (Martínez-Padilla et al., 2004).

Table 1Physical properties of fluids and particles

	Relative density	Temperature (°C)	Viscosity (Pa s)	Sphericity	Equivalent circle diameter (mm)
Water	1	25	0.0009		
Oil	0.882 ± 0.0005	25	0.199 ± 0.013		
Glycerin	1.234 ± 0.047	25	0.75 ± 0.14		
		30	0.56 ± 0.035		
Commercial beverage	1.033 ± 0.000075	25	а		
Semispherical resin particles	0.948 ± 0.0084			0.83	4.1 ± 0.14
Semispherical gelatin particles	1.028 ± 0.078			~ 1	4.1 ± 0.31
Spherical acetate particles	1.28			1	1 ± 0.002

^a Viscoelastic fluid.



Fig. 2. Mechanical spectra of the commercial beverage.

In the suspension systems, a density difference of +6.9% was measured for the resin particles in oil (creamed particles), while a difference of -5.5% was measured for the

resin particles in water (settled particles). In the system constituted by acetate particles and glycerin, a relatively small density difference of +3.6% was measured, while for gelatin particles in the commercial beverage, a very small difference of -0.5% was found. In the two latter cases, particles were uniformly suspended due in part to the high viscosity of glycerin and the yield stress of the commercial beverage.

3.2. Flow patterns

3.2.1. Resin particles–water or oil—particle image velocimetry

Resin particles contained in the vane-in-a-large-cup rheometer at rest were fully creamed. When a constant torque of 1.106×10^{-3} N m or 4.423×10^{-3} N m is applied, particles start to move towards the bottom of the cup. Fig. 3 shows the particle front as a function of time. Particles reach the bottom of the cup in less than 1 s and this time diminishes as the torque increases. Thereafter, particles are distributed homogenously within the cup.

Resin particles in water, or in the low-viscous oil, show similar flow patterns with opposite directions. Creamed in



Fig. 3. Resin particle front in water as a function of time.

water, particles move towards the bottom of the cup, meanwhile settled in oil, particles move towards the top. Fig. 4 shows the flow patterns for the resin particles in water as a function of time. It reveals vortex formation at the beginning of the test, mainly near the particle bed (Fig. 4a). Finally, these vortices reach asymmetric distributions along the vane axis (Fig. 4c).

For the resin particles in oil, vortices appear slightly later than in the latter system, maybe due to the oil viscosity effect. However, the particles do not appear near the particle bed because they settle at the cup bottom avoiding contact with the vane (Fig. 5). The presence of vortices in both systems confirms the onset for turbulent flow.

3.2.2. Resin particles-water or oil-vane in-a-large-cup

Vane velocity of the resin suspension in water or in the low-viscosity oil suspension was recorded as a function of time following two pre-shearing steps contained in Program A. Fig. 6 shows the vane velocity evolution as two values of the torque are applied. At 1.06×10^{-3} N m, resin particles show more numerous velocity fluctuations in water than in oil, due to the viscosity effect. Usually,



Fig. 4. Flow patterns for resin particles in water when a constant torque $(1.106 \times 10^{-3} \text{ N m})$ is applied at different times.

Fig. 5. Flow patterns for resin particles in oil when a constant torque $(1.106 \times 10^{-3} \text{ N m})$ is applied at different times.

when a constant torque is applied to the vane-in-a-largecup rheometer, a constant velocity is rapidly attained, confirming the results obtained with PIV.

To determine the flow regime, Reynolds numbers (Re) were calculated considering the fluid viscosity at the precedent conditions. Suspensions viscosity was calculated using the mathematical model proposed by Happel (1957). It is generally accepted that laminar flow exists for Re less than 63 and turbulent flow is assured for Re larger than 63,000. In some cases, turbulent flow may be present with an impeller Re number as low as 1900 (Steffe, 1996). Approximate *Re* numbers for particles in water lie between 25,000 and 47,000, while Reynolds numbers for pure water lie between 10,000 and 30,000. As expected, *Re* numbers are lower for particles in oil (between 92 and 234) within the transitional flow region.

3.3. Acetate particles–glycerin—particle image velocimetry

Acetate particles are fully suspended in glycerin, and their flow patterns at various times are presented in Fig. 7. Similar results are obtained when the suspension

Fig. 6. Vane angular velocity as a function of time for the two applied torques for resin particles in water and in oil.

is sheared from 0.02 to 10; from 0.02 to 20, and from 0.02 to 30 s^{-1} . Flow patterns are established within 1 s, and some small vortices are observed at the cup bottom probably due to particle resistance to flow. After 25 s, horizontal parallel lines are detected, confirming the presence of laminar flow. This behavior is maintained up to the end of the first range $(0.02-10 \text{ s}^{-1})$. From 0.02 to 20 s^{-1} , and from 0.02 to 30 s^{-1} , comparable results are obtained but vortices clearly manifest themselves at the cup center. Nevertheless, the flow stabilizes before 15 s in the second range (from 0.02 to 20 s^{-1}).

3.3.1. Acetate particles-glycerin-vane-in-a-large-cup

Pre-shearing was not necessary for these homogenous suspensions. Torque as a function of the vane angular velocity was plotted in Fig. 8 (Program B). When a linear increase-decrease of the angular velocity was applied at different suggested ranges, a slight difference between the increasing and decreasing curves is observed, which diminishes in the largest range. This may be caused by the increase in rate since all tests were carried out in a total time of 120 s. In the same figure, data of the steady curve in the third range overlap, implying that the representative flow curve for this suspension has been found.

The vane Reynolds numbers were calculated with glycerin without particles and varied between 0.04 and 62, corresponding to laminar flow. Rheological data (torque and angular velocity) for these three systems and for glycerin were transformed to shear stress and shear rate using Eqs. (1) and (2), considering the Couette analogy and negligible end-effects. Only torque values recorded at velocities higher than 1 s⁻¹ were considered for regression analysis, since lower velocities lie outside the rheometer accuracy range and non-linearity could be observed. The viscosity of glycerin measured in the vane-in-a-large-cup was 0.53 ± 0.013 Pa s, while the shear viscosity measured in the concentric cylinders fixture (Z20) was $0.56 \pm$ 0.035 Pa s, resulting in a 5.4% difference. This agreement validates the suggested approach using this experimental set-up. The suspension viscosity value was 1.58 ± 0.185 Pa s and corresponds to a relative viscosity of 2.68. When comparing this value to that predicted using the model of Happel (1957) for semi-concentrated suspensions of spherical particles in Newtonian fluids (2.83 Pa s, for a volume fraction of 0.2), a relative error of 5.3% was calculated. This result is similar to that obtained with a helical ribbon in suspensions containing coarse discs (Martínez-Padilla, Cornejo-Romero, Cruz-Cruz, Jáquez-Huacuja, & Barbosa-Cánovas, 1999). This agreement further validates the use of the vane-in-a-large-cup in the flow characterization of suspensions containing coarse spherical particles.

3.4. Gelatin particle-commercial beverage—particle image velocimetry

Gelatin particles are fully suspended in the commercial beverage. When this suspension was sheared from 0.02 to 10, from 0.02 to 20, and from 0.02 to 30 s^{-1} , strong differences with the flow patterns of the acetate particles in glycerin are found and they are presented in Fig. 9. During the first seconds the flow pattern is not completely established, since vortices can be observed close to the particles, probably due to their resistance to flow (or yield stress). Past

Fig. 7. Flow patterns for acetate particles in glycerin for different times when sheared from 0.02 to 10 s^{-1} .

61 s, particles move along the fluid streamlines but the flow patterns are distorted, and they look like vortices in a turbulent flow rather than streamlines in laminar flow. This behavior can be explained by the low viscosity of the commercial beverage (~ 8 mPa s). For longer times, flow patterns are more organized with a tendency to arrange themselves along parallel streamlines, and yet in the presence of vortices which are distributed asymmetrically along the vane axis (Fig. 9c). Evidently, as in the case of particles in water, these results confirm that the flow of this suspension lies in the turbulent regime.

3.4.1. Gelatin particles-commercial beverage-vane-in-alarge-cup

Pre-shearing was not applied for these homogenous suspensions. Torque as a function of the vane angular velocity is plotted in Fig. 10 (Program B). When the angular velocity is increased following a linear mode, the torque presents a parabolic response comparable to that shown in the water suspensions patterns. This response is quite similar along the three suggested velocity ranges. A slight difference between the increasing and decreasing curves is also observed.

Fig. 8. Torque as a function of vane angular velocity for acetate particles in glycerin for three applied ranges.

Fig. 10. Torque for gelatin particles in commercial beverage as a function of vane angular velocity for three applied ranges. Water (+) is overlapped for comparison purposes.

Fig. 9. Flow patterns for gelatin particles in commercial beverage for different times when sheared from 0.02 to 10 s^{-1} .

4. Conclusions

Validation of vane measurements is a crucial problem especially in the case of flow characterization. Particle image velocimetry allows the measurement of suspension flow patterns containing millimetric-size particles. Most systems studied here present flow patterns where the presence of vortices is evident. At short times, these vortices originate near the particle bed, while at longer times, they uniformly distribute within the large baffled cup.

Flow characterization with the vane-in-a-large-cup is only possible for acetate particles in glycerin undergoing laminar flow. The laminar flow regime was confirmed according to the value of the Reynolds number and particle image velocimetry.

Acknowledgements

We thank Dr. Roberto Zenit for his technical support on the use of the PIV technique. We also acknowledge the support from Consejo Nacional de Ciencia y Tecnología (CONACYT, México) through the grant awarded to M. Ramirez-Gilly.

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