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Analysis of suspension sedimentation in fluids with rheological shear-thinning properties and thixotropic effects

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ABSTRACT

Suspension sedimentation in fluids with shear-thinning properties is of interest in several industrial operations, such as oil and gas exploration. This paper offers new information about the behavior of a group of particles settling in shear-thinning fluids. The tests carried out in this work differ from those reported in earlier works, because they involved a quantitative analysis of the settling behavior of particulate materials in regions with high concentrations of solids (where sediment is formed). To this end, the gamma-ray attenuation technique was used to monitor the concentration of solids in different regions of the tube over time. The results revealed significant differences in the behavior of particles settling in Newtonian and non-Newtonian fluids. Sediment was formed faster in the shear-thinning fluid when compared to the Newtonian fluid of similar apparent viscosity. However, final sediment compaction took longer in the non-Newtonian fluid. Overall, this study provides relevant information about sedimentation in non-Newtonian fluids, and addresses a subject about which several aspects should be further explored and better understood.

1. Introduction

Operational stops are frequent during oil-drilling processes. This leads to the interruption of drilling mud pumping, causing some particles that were moving up to the surface to begin settling through the annular space. Sedimentation of a large amount of particulate matter is an undesirable phenomenon in drilling operations, since it may give rise to several operational problems, such as stuck pipe.

Notwithstanding the importance of the theme, most of the experimental studies pertaining to gravitational sedimentation that have been conducted involve Newtonian fluids [1–5]. Studies using fluids with similar rheological properties to those commonly used in drilling (shear-thinning fluids) have limited their scope to the behavior of isolated particles [6–7] or a few particles [8–13].

Information about the settling of suspensions in shear-thinning fluids is even scantier in the literature. The main works in this area demonstrate that particles cluster together, forming columns throughout. This phenomenon has been observed in both strongly elastic shear-thinning fluids [14–18] and weakly elastic, thixotropic, shear-thinning fluids [19,20].

Particle agglomeration can have a great impact on the settling of solids. Unlike the phenomenon observed in Newtonian fluids, particle settling rates may rise as local solids concentrations increase under such circumstances [19,21].

Despite the relevant information reported in the literature to date, further research is required for a more in-depth understanding of this phenomenon. Moreover, studies that provide quantitative information about settling in shear-thinning suspensions have not examined the region near the bottom of the tube, where sediment is formed. Therefore, empirical knowledge about particle behavior in this region is still incomplete. The difficulty in carrying out quantitative studies on sedimentation in regions with high concentrations of solids (such as those where sediment is formed) stems from the infeasibility of using quota sampling methods (for they interfere in and disturb the system) and from the awkwardness of taking indirect measurements of local solids concentrations [22–25].

Indirect measurements require the use of high-energy electromagnetic waves, such as gamma-rays, when applied to solid-liquid systems with high concentrations of solids. This type of wave has a high power of penetration into physical media, and enables measurements to be taken when it reaches the detection system [22–28].

In this context, this paper describes an investigation into the settling behavior of suspensions in non-Newtonian fluids with shear-thinning properties. The gamma-ray attenuation technique was used to monitor the variation in solids concentration in different regions over time. The tests assessed the effects of both fluid rheology and particulate matter concentration on particle dynamics. Newtonian fluids (glycerin solutions) and shear-thinning non-Newtonian fluids with memory effects

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(xanthan gum solutions) were used to analyze the different settling behaviors of particulate matter.

2. Material and methods

2.1. Characterization of solid materials

Samples of calcium carbonate and sand were used as suspended particles in this research. Sand was used to obtain photographic images of the particle behavior during sedimentation in the non-Newtonian fluid. The photographic images of settling calcium carbonate particles were not clear enough for a qualitative analysis because of the poor contrast between the fluid and the carbonate particles.

The densities of the samples of sand and calcium carbonate used in this study were, respectively, $\rho_{\text{sand}} = 2.72 \text{g/cm}^3$ and $\rho_{\text{carb}} = 2.82 \text{g/cm}^3$ (values obtained from helium-gas pycnometry). The samples were obtained by sieving: $-d_{\#} = 212$ μm and $+d_{\#} = 150$μm. (os símbolos $-d_{\#}$ e $+d_{\#}$ indicam respectivamente o diâmetro de abertura da peneira no qual o sólido foi passante e retido).

The particle size of the material was analyzed using a Malvern Mastersizer Microplus MAF 5001 particle size analyzer. Table 1 describes the volume diameters of the particles at 10% ($D_{0.1}$), 50% ($D_{0.5}$) and 90% ($D_{0.9}$) in the cumulative distribution and the Sauter mean diameter ($D_{3.2}$).

2.2. Characterization of the fluids

The settling tests were performed using glycerin solutions at 92% v/v (GL) and xanthan gum solutions at 0.2% w/w (GX) as fluid phase (v/v: volume/volume, w/w: weight/weight).

The fluids were characterized using two techniques: (1) graphs of viscosity as a function of shear rate; and (2) stepwise changes in the shear rate. The viscosity graphs correspond to the time in which the shear rate applied to the fluid was high enough for the shear stress (or the apparent viscosity) to remain constant over time. The tests were performed at a temperature of 25 °C, using an Anton Paar MCR 302 rheometer. Fig. 2.1 illustrates the results obtained for both fluids (XG, GL) in log-log scale.

Fig. 2.1 shows that variation in shear rate did not change the viscosity of the GL solution, indicating that these solutions had a Newtonian behavior. The proportionality constant μ estimated from the results obtained in the tests was 0.30 Pa·s.

Fig. 2.1 also shows that the increase in the XG fluid shear rate reduced its apparent viscosity, indicating that the XG solutions had a shear-thinning behavior.

The Power-law model (Eq. 2.1) was chosen to fit the experimental points in Fig. 2.1.

$$\eta = m\gamma^{n-1} \tag{2.1}$$

where m is the consistency coefficient and n the behavior index. Table 2 lists the estimated parameters of the power law equation (m and n) and the linear correlation coefficient (r).

Mewis and Wagner [29] explained that the viscoelasticity in thixotropic fluids can be evaluated through a stepwise test in the shear rate. Hence, shear stress can be evaluated as a function of time response while the shear rate is sharply reduced from γ_1 to γ_2. This study used a step-down change in shear rate from 100 s^{-1} to 1 s^{-1}. The results are shown in Fig. 2.2.

Table 1
Volume diameter of the particles used as suspended material.

Solid	$D_{0.1}$ (μm)	$D_{0.5}$ (μm)	$D_{0.9}$ (μm)	$D_{3.2}$ (μm)
Calcium carbonate	138.65	193.21	293.49	188.55
Sand	152.65	184.90	234.99	184.75

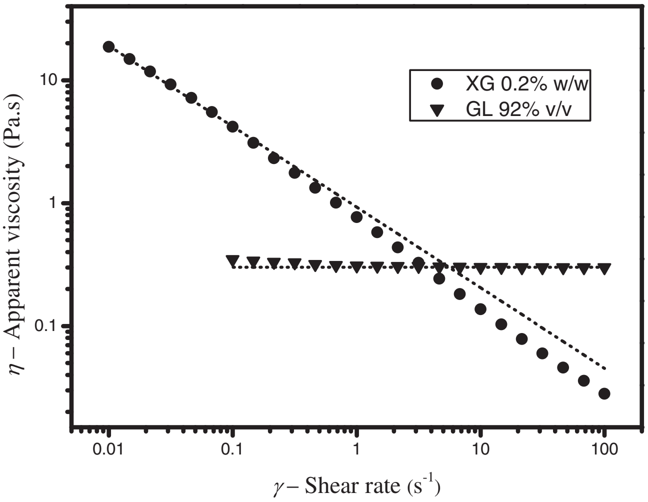

Fig. 2.1. Apparent viscosity of the GL and XG solutions as a function of shear rate in the steady-state condition.

For the GL solution, Fig. 2.2 indicates a sharp decrease in the shear stress followed by a constant value over time.According to Mewis and Wagner [29], this type of response indicates a fluid without thixotropic or viscoelastic properties, confirming that the glycerin solution can be considered a Newtonian fluid.

The XG solution showed a sharp decrease in shear stress, followed by a gradual increase in stress over time. According to Mewis and Wagner [29], this type of response is typical of inelastic fluids with thixotropic properties.

Based on the aforementioned analyses, it was concluded that the GL solutions had a Newtonian behavior and the XG solutions had shear-thinning properties and thixotropic effects.

2.3. Experimental apparatus for the application of radioisotopes

In this study, a test device for the application of radioisotopes was used to monitor the local solids concentration. The test device consisted of a radiation source (^{241}Am) sealed within a lead overlay, a glass test tube and a radiation detection system containing a gamma photon-absorbing scintillation crystal.The radiation source and the detector were placed on a platform that can be moved vertically to record measurements from the bottom of the tube ($z = 0$) to the top of the suspension column (Fig. 2.3).

To carry out the experiments, first the fluid (GL or XG) and the particulate matter were placed in the tube, after which they were homogenized with a perforated agitator in ascending and descending movements. The next step consisted in quantifying the photons that reached the scintillation detector. The temperature was kept at 25 °C throughout the tests.

The initial solids concentrations of the suspensions (ε_{s0}) used in this study were 4% and 9%. After homogenization, the height of the suspensions in the columns was 28.4 cm for $\varepsilon_{s0} = 4\%$ and 30.1 cm for $\varepsilon_{s0} = 9\%$.

2.3.1. Gamma-ray attenuation technique

The Lambert equation determines the variation in intensity of a collimated monoenergetic beam of gamma-rays crossing a physical medium. When the beam crosses a solid-liquid suspension and the reference

Table 2
Power law parameters for the XG solutions obtained by nonlinear estimation.

m	n	r
0.93	0.34	0.99