The Use of Low Shear-Stress Rheological Data in Settling of Particles in Paints

Sujan E. Bid Wadud
TA Instruments, 109 Lukens Drive, New Castle DE 19720, USA

ABSTRACT

The solid particles in paints and inks may undergo settling under the force of gravity. Rheology measured stress-dependent viscosity profiles predict widely different settling characteristics.

INTRODUCTION

In solid-liquid suspensions, such as paints and inks, the particles may settle under the force of gravity if the suspending medium viscosity is not high. However, if the viscosity of the suspending medium is increased, the particles are unable to move under gravity. The yield stress in the system aids this liquid medium to keep the solid particles suspended.

Stress-controlled rheometry is the preferred tool for studying yield stress in paints and inks. By applying very small stresses directly to the specimen and monitoring the displacement, its yielding behavior is observed. If the applied stress is below the yield stress, then the sample undergoes no motion, and the (rate of) displacement measured by the instrument is very small. This makes the apparent viscosity extremely large. When the stresses applied to the sample exceeds the yield stress, the sample undergoes greater shear-rate and the apparent viscosity shows a precipitous drop when plotted versus stress.

Two paint samples with known settling properties are studied. By performing steady-state stress sweeps (automatic creep analysis) the differences between the two samples is observed and their settling characteristics explained.

EXPERIMENTAL

Two water-based paints identified as A and sample B are examined. The particles in paint A are known to settle but those of B didn’t. An AR1000 rheometer with a 40 mm parallel plate rotor and Peltier plate is used in these experiments. Because of the size of the particles, cone geometries are not used. The solvent trap cover is utilized to prevent water evaporation from the paint. Stress sweeps are performed at 25 °C. At each stress, the steady-state shear-rate is automatically determined based on the following settings:
Sampling time : 10 s  
Tolerance : 10 %  
Consecutive within : 3  
Maximum point time : 1:20 min.

RESULTS AND DISCUSSION

Figure 1 displays the logarithm of shear rate on the Y-axis versus logarithm of the shear stress on the X-axis for samples A and B. The shear rates induced by the low stresses in sample B are significantly lower than those in sample A. As the shear stress is increased further, the shear rate in B shows a significant step increase. This sort of rheological behavior is indicative of the presence of yield stress in B. The onset of the increase in shear rate, or a change in slope, can be used as the indicator of yield stress and is estimated here at about 0.65 Pa.

When the same data is plotted with the apparent viscosity on the Y-axis in Figure 2, the viscosity of sample B appears significantly higher than A at lower shear stresses. As the stress is increased and the yield stress is exceeded, the material starts to flow, and the apparent viscosity undergoes a sharp drop in value. The yield stress can also be calculated as the onset point of the drop in viscosity. This is more easily observed than the slope change in Figure 1 as the drop in viscosity is very sharp. The onset of this drop in viscosity is about 0.61 Pa. Sample A does not show this kind of behavior. It has a traditional zero-shear-viscosity, followed by a shear-thinning segment.

Figure 1 - Shear Rate Versus Shear Stress at 25 °C.

The settling behavior is explained by these viscosity profiles. The particles are under a very small stress due to gravity. In paint A, the viscosity of the fluid around each particle is low, such that it cannot prevent the particles from moving under the stress of
gravity. But in paint B, the viscosity is so high, that the particles cannot move under that stress.

![Figure 2 - Viscosity Versus Shear Stress at 25 °C.](image)

**SUMMARY**

The stress-controlled AR1000 rheometer is an excellent tool in determining the yield stress of structured materials such as paints. In a single experiment, the instrument is able to analyze the flow characteristics over a wide range of stresses and shear-rates. This enables one to distinguish between paint samples whose particles settle over time and those whose particles do not.

**KEYWORDS**

paints and coatings, rheology, viscosity
TA Instruments

United States, 109 Lukens Drive, New Castle, DE 19720
Phone: 1-302-427-4000 • Fax: 1-302-427-4001 • E-mail: info@tainst.com

United Kingdom • Phone: 44-1-372-360363 • Fax: 44-1-372-360135 • E-mail: uk@tainst.com

Spain • Phone: 34-93-600-9300 • Fax: 34-93-325-9896 • E-mail: spain@tainst.com

Belgium/Luxembourg • Phone: 32-2-706-0080 • Fax: 32-2-706-0081 • E-mail: Belgium@tainst.com

Netherlands • Phone: 31-76-508-7270 • Fax: 31-76-508-7280 • E-mail: Netherlands@tainst.com

Germany • Phone: 49-6023-9647-0 • Fax: 49-6023-96477-7 • E-mail: germany@tainst.com

France • Phone: 33-1-304-89460 • Fax: 33-1-304-89451 • E-mail: france@tainst.com

Italy • Phone: 39-02-27421-283 • Fax: 39-02-2501-827 • E-mail: italia@tainst.com

Sweden/Norway • Phone: 46-8-594-69-200 • Fax: 46-8-594-69-209 • E-mail: Sweden@tainst.com

Japan • Phone: 813 5479 8418 • Fax: 813 5479 7488 • E-mail: nurayama@taij.po-jp.com

Australia • Phone: 613 9553 0813 • Fax: 613 9553 0813 • E-mail: steve_shamis@waters.com

To contact your local TA Instruments representative visit our website at www.tainst.com