Static measurement of yield stress using a cylindrical penetrometer

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Abstract

A novel and simple method using a cylindrical penetrometer is being developed for the measurement of yield stress. The principle of this technique is based on the measurement of the static equilibrium of a falling penetrometer in a yield stress fluid. The yield stress is simply determined by a balance of forces acting on the penetrometer. The yield stress of Carbopol gels and TiO₂ suspensions has been measured using this method. The results are in reasonable agreement with the values from conventional methods. The effects of the dimensions and weight of the penetrometer have been examined. The long-term behaviour was also observed. No measurable creep was seen and equilibrium was found to be very quickly established. The cylindrical penetrometer technique promises to be a simple, quick and reliable static method for the measurement of yield stress.

Keywords: yield stress, non-Newtorian fluid, penetrometer

1. Introduction

The yield stress of a material is defined as the shear stress that has to be overcome to cause significant flow of the material. It is an important characteristic in suspension rheology and is exploited in many industry applications. Although the existence of a true yield stress in fluids was questioned (Barnes and Walters, 1985), the concept remains in general use in rheological fluid investigations and applications. It has been extensively discussed by Barnes and his co-worker (1999a, b, 2001).

Numerous techniques have been developed to measure the yield stress based on dynamic or static principles, for instance, stress on an immersed plate (De Kee, 1980; 1985), vane torsion (Nguyen and Boger, 1985), falling and rolling ball techniques (Boardman and Whitmore, 1961; Hartnett and Hu, 1989; Schurz, 1990), inclined plate (Uhlherr, 1984; 1999), pendulum (Uhlherr, 1986; 1997). Recently, (Zhu et al., 2001) developed a technique using a slotted plate device for measuring static yield stress.

Penetration techniques have been used widely for testing the rheological properties of materials of high consistency. One of the earliest applications was to measure the consistency of building materials, such as cement and gypsum plaster pastes, using a simple constant-load penetrometer (Reiner, 1954). Penetrometers have also been used for testing the properties of bitumen, spreadability of butter, the The motion of a falling cylinder viscometer (needle rheometer) in Newtonian fluids and non-Newtonian viscoelastic fluids has been extensively studied (Ashare and Bird, 1965; Park et al., 1990; Cho et al., 1992; Zheng et al., 1994). The steady shear viscosity can be determined by measuring the terminal velocity of a thin cylinder in the fluids.

Park et al. (1988) extended the use of a falling needle viscometer to measure yield stress. However, as the density difference between the needle and the liquid can not normally be adjusted, it is difficult to reach a static balance between the driving force from the density difference and the resistance force from yield stress. Therefore, the needle is not stationary in the liquid. The static density difference can only be determined by extrapolating terminal velocity of the needle to zero. Thus, the falling needle technique provides an indirect method for measuring yield stress, similar in nature to the extrapolation of a flow curve.

The penetration technique introduced here provides a direct technique to measure yield stress of low-consistency fluids. As the weight of a hollow penetrometer can be easily adjusted, a static balance between the forces acting on the penetrometer can always be reached.

yield values of lubricating greases, even the tenderness of vegetables and fruits (Scott Blair, 1969). In such tests, "needle" penetrometers or cylinders with a conical tip were frequently used. Such geometries allowed materials of high consistency to be penetrated. A mechanical force normally needs to be applied to a penetrometer.

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2. Experimental apparatus and method

The penetrometer consists of a hollow brass or plastic cylinder with a hemispherical end. The size and weight of the penetrometers are chosen to allow for different magnitudes of the yield stress. The surface of the penetrometers was roughened to prevent slip in the liquid by coating with granular particles with a uniform size of 100 µm. The weight of the penetrometer can also be adjusted by adding dense powder into the hollow body, which will result in different depths of immersion. Tungsten powder was used in this study. A fine wire attached to one side of the cylinder at its top is used to ensure vertical immersion of the penetrometer by passing the wire through two small vertically aligned holes. However, some tests were carried out using the penetrometers without the wire, when the centre of gravity was near the closed end of the cylinder. The fluid samples were contained in a cylindrical jar or measuring cylinders with different diameters. A schematic diagram of the apparatus is shown in Fig. 1.

The principle of the penetrometer is simply based on the attainment of static equilibrium in a vield stress fluid. When the penetrometer is gently released in a fluid with yield stress, it quickly falls under gravity toward an equilibrium position. The resistance forces due to the yield stress on the surface of the penetrometer and buoyancy simultaneously apply to limit the motion. The penetrometer reaches static equilibrium when the force due to gravity is balanced by the resistance forces. A diagram of the forces acting on the penetrometer is shown in Fig. 2. It has been assumed that at the static equilibrium condition, the shear stress over the immersed surface is everywhere uniform. This is expected to be true for the cylindrical surface, but is only an approximation for the hemispherical end (Beris et al., 1985; Blackery and Mitsoulis, 1997; Beaulne and Mitsoulis, 1997).

A balance of forces acting on the penetrometer can be written as follows:

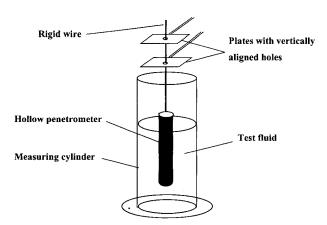


Fig. 1. Schematic diagram of cylindrical penetrometer.

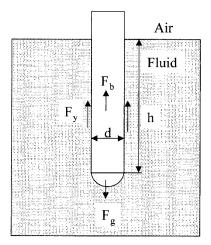


Fig. 2. Static equilibrium of cylindrical penetrometer in a yield stress fluid.

$$F_g = F_b + F_y \tag{1}$$

where F_g is the weight of the penetrometer, F_b is the resistance force due to buoyancy and F_y is the resistance due to yield stress. For a cylindrical penetrometer, equation (1) becomes:

$$mg = \tau_y \left(\pi dh + \frac{\pi^2 d^2}{8} \right) + \rho g \pi d^2 \left(\frac{h}{4} + \frac{d}{12} \right)$$
 (2)

where m is the total mass of the penetrometer, ρ is the fluid density, d and h are the diameter and immersed length of the cylindrical part only. Thus, the yield stress (τ_y) can be calculated using the following equation:

$$\tau_{y} = \frac{g \left[m - \rho \pi d^{2} \left(\frac{h}{4} + \frac{d}{12} \right) \right]}{\pi d \left[h + \frac{\pi d}{8} \right]}$$
(3)

Equation (3) indicates that the yield stress is a function of the weight and dimensions of the immersed penetrometer. Thus, the determination of the yield stress simply relies on the measurement of the equilibrium depth in the fluid for a penetrometer with known weight and diameter.

The penetrometer is initially positioned just in contact with the surface of the fluid. It is then released, and comes quickly to rest when the position corresponding to the yield stress is reached. In this way, the yield stress is approached from stress values above it. It should be noted that the penetrometer is only partially immersed for this technique, which is different from the complete immersion which occurs with the falling needle technique (Park *et al.*, 1988).

Several penetrometers with different dimensions were used in the current study. Details of these penetrometers are given in Table 1.

The solutions used include four Carbopol gels with yield

Table 1. Details of cylindrical penetrometers

No	Diameter mm	Total available length, mm	Surface	Material
1	3.54	130	rough	brass
2	6.97	140	rough	polystyrene
3	6.82	140	rough	brass
4	9.88	145	rough	polystyrene
5	16.45	150	rough	brass
6	9.41	145	smooth	polystyrene

stress ranging from about 6-60 Pa and two TiO₂ suspensions. Some of the Carbopol solutions were prepared for a previous study (Guo and Uhlherr, 1995). It was found that the rheological properties of these solutions had changed little despite some evaporation. A 0.1 wt% Carbopol (934) gel was freshly prepared. A 60 wt% TiO₂ suspension was prepared by mixing TiO₂ pigment (CR-826, Kerr-McGee Chemical) with 0.01 M KCl aqueous solution. This suspension was agitated for more than 24 hours, adjusted to pH 6.9-7.0 and allowed to age for 10 days. A 50 wt% TiO₂ suspension was produced by diluting 60 wt% suspension with 0.01 M KCl aqueous solution. All solutions were agitated for at least 24 hours prior to use.

All measurements were performed at controlled room temperature of 20°C. Immediately before use, the suspensions were stirred for 1 minute and then allowed to rest for 10 minutes before every test. The movement of the penetrometer in the fluid was accurately measured using a cathetometer.

The yield stress for the solutions was also characterised by other accepted techniques using a rate-controlled rheometer (Haake RV20) and a stress-controlled rheometer (DSR, Rheometric Scientific). The techniques used include the extrapolation of the steady shear flow curve, vane torsion, stress ramp and short term creep.

3. Yield stress characterisation using conventional techniques

3.1. Stress ramp

Stress ramp tests were carried out using a stress-controlled rheometer (DSR) equipped with a four-bladed vane. The dimensions of the vane are 25 mm diameter and 25 mm long. The leading surfaces of the vane were roughened by covering with sandpaper to prevent any slip, which was observed during tests at extremely small stress values.

In ramp tests, the stress was increased at a constant rate from zero to a level well above the yield stress. Several rates of increase were used. The resulting angular deformation was recorded and converted to shear strain in terms of the geometric constant of the vane in an infinite medium. Initially, the strain increases linearly with increase in stress, which is a typical solid-like behaviour. When the

stress reaches a certain level, continuous flow can be observed and the slope of the strain stress curve rapidly increases. The yield point of a material can be determined by extrapolating the two straight lines corresponding to solid-like and liquid-like behaviours to a point of intersection. The technique has been described by Zhu *el al.* (2001).

Figs. 3 and 4 show the results from the stress ramp tests for a 0.1 wt% Carbopol gel and for the 60 wt% TiO_2 suspension. It is evident that the ramp rate has a significant effect on the yielding process of the solutions: the lower the applied ramp rate, the smaller the critical stress observed. There appears to be a limiting ramp rate below which little change in yield stress is observed. Yield stress values obtained from this technique are shown in Table 4.

3.2. Creep

The yield stress for the test samples was also determined by creep measurement. Constant stress was applied, increasing in successive small steps. The variation of the strain with time was recorded. The critical stress for yielding can be determined by a significant increase in the creep rate. Figs. 5 and 6 show the creep profiles of 60 wt% TiO₂ suspension and 0.1 wt% Carbopol gel. Fig. 5 represents a method for determining the yield stress by observing the progress of creep for the material over a short period. This method has been used by others (Liddell and Boger, 1996; Zhu *el*

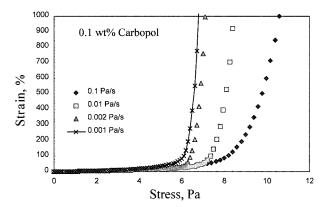


Fig. 3. Stress ramp results for 0.1 wt% Carbopol gel.

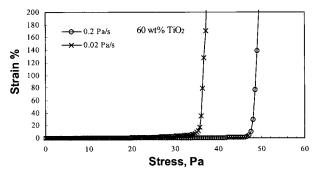


Fig. 4. Stress ramp results for 60 wt% TiO₂ suspension.

al., 2001). Fig. 6 shows the creep behaviour of Carbopol gel over a much longer period. It is obvious that the time frame is important in determining critical yield stress. A prolonged creep time can cause the failure of the material structure at lower stresses. For example, a value of 6 Pa could be determined for 0.1 wt% Carbopol gel if a period of 100 s is considered. However, the critical stress for yielding would drop to 5.0 Pa if creep time is increased to 10^4 s. This is consistent with the comments made by Cheng (1986). The results obtained using the arbitrarily chosen period of 100 s are included in Table 4.

3.3. Extrapolation of flow curve in steady shear

Extrapolation of the flow curve in steady shear is a very popular indirect method to determine the yield stress. The measurements were carried out using a rate-controlled rhe-

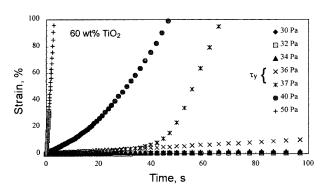


Fig. 5. Creep profile of 60 wt% TiO₂ suspension.

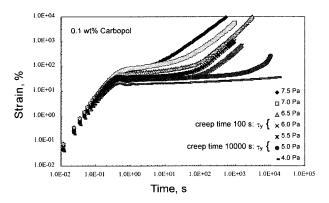


Fig. 6. Creep profile of 0.1 wt% Carbopol gel.

ometer with profiled bob and cup system. The shear rate was corrected for non-Newtonian behaviour in the gap space by the Krieger method (1954). Data (8 points) at the lowest shear rates available (0.15-6.3 1/s) were fitted by a 4th order polynomial equation, and either the Herschel-Bulkley model or the Casson model. The yield stress was then determined by extrapolation of the fitted flow curve to zero shear rate. These results are included in Table 4.

3.4. Vane torsion

The vane torsion technique is a well-recognised method developed by Nguyan and Boger (1985) and has become increasingly popular over the last 20 years for the measurement of flow properties of non-Newtonian liquids (Barnes and Nguyen, 2001). With this measurement, each sample was tested with two vanes with different sizes and at different rotational speeds. A simple average value and its standard deviation are included in Table 4.

4. Yield stress measurement using the penetration technique

Yield stress measurements using penetrometers have been carried out on four Carbopol gels and two ${\rm TiO_2}$ suspensions with yield stress values from about 6 Pa to 60 Pa. For each solution, systematic measurements were conducted using penetrometers with different dimensions and masses. A mean value of yield stress for each sample was obtained. Results are summarised in Table 2.

In general, the results present remarkably consistent standard deviations for the different solutions. Larger relative deviation occurs in the measurements for the solutions with low yield stress. No systematic effect can be identified for the variation of any particular factor. The magnitude of observed deviation appears reasonable in view of similar variability for conventional techniques of yield stress measurement, specifically the vane technique.

Equation 3 is based on a simple force balance and the assumption of uniform shear stress acting on the whole surface of the penetrometer. The validity of the equation can be examined by using the independent measurements of yield stress. Equation 3 can be rearranged as follows:

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Table 2. Summary	or results	for beneuration	measurement

Solution	Penetrometer	No of Measurements	Yield Stress, Pa (Mean Value)	Standard Deviation σ	
0.1% Carbopol	2, 4, 5	16	6.77	1.38	
Carbopol A	2, 4	4	17.1	1.14	
Carbopol B	1, 2, 3, 4	8	41.7	1.60	
Carbopol C	1, 2, 3, 4	9	61.4	1.80	
50 wt% TiO ₂	2, 4	3	10.7	1.03	
60 wt% TiO ₂	4	1	60.9	-	

$$h = m \left[\frac{g}{\pi d\tau_{y} + \rho \frac{\pi d^{2}}{4}} \right] - \left[\frac{\frac{\pi^{2} d^{2}}{8} \tau_{y} + \rho \frac{\pi d^{3}}{12}}{\pi d\tau_{y} + \rho \frac{\pi d^{2}}{4}} \right]$$
(4)

It is expected that the immersed depth should be directly proportional to the mass for a penetrometer with a given diameter in a fluid with a given yield stress and density. This relationship is examined in Fig. 7 which shows a comparison between the calculation based on equation (4) and experimental measurements for 0.1 wt% Carbopol gel. The value of yield stress was independently determined by vane torsion. The agreement is excellent, which supports the validity of the equation. The small negative intercept seen in Fig. 7 is due to the second term on the right hand side of Equation (4). This term contains the stress distribution over the hemispherical end of the penetrometers. Clearly, replacing the actual distribution by a uniform value has little effect on the overall accuracy of the technique.

The accuracy of the result is greatly dependent on the measurement of immersed depth of the penetrometer at equilibrium. Creep may occur under the action of a small applied stress for any yield stress fluid, and this normally complicates the measurement of yield stress using a static method. In order to establish the importance of creep in the penetrometer measurement, the movement of the penetrometer was subjected to a series of long-term measurements. Fig. 8 shows a plot of the change in the immersed depth with time of observation. It was found that the penetrometer quickly approached its equilibrium position when it was released in a yield stress fluid and the immersed depth remained effectively constant after a measuring time of about 10 minutes. The resistance forces due to the buoyancy and shear stress both increase with increasing immersion, while the driving force due to gravity remains constant. This speeds up the equilibration process and appears to effectively minimise creep. This is an advantage of this technique over other static methods investigated by the authors. Thus, in the pendulum technique, equilibration takes several hours and creep is clearly

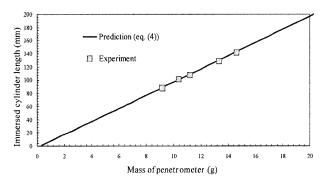


Fig. 7. Comparison of theoretical calculation and experimental measurements.

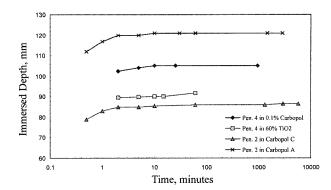


Fig. 8. Dependence of immersed depth of penetrometer on time.

discernible (Guo and Uhlherr, 1996).

In order to minimise sample volume, it is desirable to use a tall, small-diameter container. However, this could conceivably introduce wall effects into the measurements. The effect of the container wall on the penetrometer measurement has been investigated by changing the diameter ratio. Fig. 9 shows the effects of D/d (the ratio of container diameter to penetrometer diameter) on the immersed depth at equilibrium. It is evident that a significant wall effect is present when D/d < 4. The added drag force from the wall results in a higher value of yield stress. This result is consistent with earlier studies (Nguyen and Boger, 1986; Atapattu *et al.*, 1990, 1995). Therefore, the ratio of D/d must be greater than 4 to eliminate any wall effect.

Wall "slip" is a common problem for the measurement of rheological properties of suspensions. Slip effects can potentially be present in all viscometers, but are especially serious for low shear rate measurements (Barnes, 1995; Roberts and Barnes, 2001). To prevent possible slip, the surface of the penetrometers was roughened by coating with uniformly sized angular particles. Several comparative tests were carried out to estimate the effect of slip (see Table 3). It was found that a penetrometer with a smooth surface tends to underestimate the value of yield stress by up to 40%. The size of the roughness elements (particle size) has not been varied. It is conceivable that a roughness size of

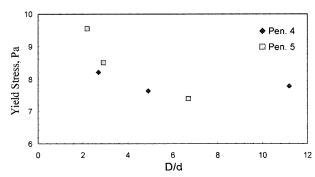


Fig. 9. Effect of ratio of container diameter to penetrometer diameter.

about $100\,\mu m$ is not sufficient to eliminate slip in all instances. The unexplained underestimate of τ_y for the 50 wt% TiO_2 suspension may be due to this cause. More systematic measurements are required for this effect.

The accuracy of the results is limited by a number of experimental constraints. Firstly, due to an unevenness in the particle coating, there is an error in penetrometer diameter of about 0.1 mm. Since the same particle size was used for the coating of all penetrometers, this effect is obviously greater for the penetrometers of smaller diameter. Secondly, the free surface of a yield stress fluid need not be flat and this, together with a small depression of the surface at the point of entry of the penetrometer, gives rise to some uncertainty in the immersed depth. This is particularly noticeable for particulate suspensions. In this case, the uncertainty may be as large as 4 mm. In the case of Carbopol gels, the uncertainty is rather less. Thirdly, the distribution of shear stress over the hemispherical end of the penetrometer at equilibrium is taken to be the yield stress. This disregards the actual distribution, as predicted by Beris et al. (1985); Blackery and Mitsoulis (1997); Beaulne and Mitsoulis (1997). This effect would be significant for penetrometers with large diameter and small immersed depth. In the current study, the ratio of immersed depth to diameter of penetrometer was carefully kept greater than 7. The effect of the approximation for the hemispherical end should therefore be negligible. This can be seen in Fig. 7. Other errors introduced by experimental technique, such as lack of verticality and disturbance of fluid surface by introduction of the penetrometer, can be easily minimised and

Table 3. Effects of surface condition of penetrometer

Solution	Penetrometer		Yield stress, Pa	
0.1 // Corbonal	4	Roughened	7.6	
0.1 % Carbopol	4	Smooth	6.4	
Combonal D	4	Roughened	39.0	
Carbopol B		Smooth	22.4	
Combonal C	2	Roughened	58.9	
Carbopol C	2	Smooth	45.4	

are likely to be far less significant than the relatively uncontrolled history of the fluid sample for example.

5. Comparison of results using different techniques

Table 4 shows a comparison of the results from all the different techniques employed in this study. The results from the penetrometer measurement are generally in good agreement with the data from other methods, in particular, those obtained by vane torsion. It can be seen that the results for TiO₂ suspension are variable and relatively discordant. This may be due to uncontrolled processes occurring in the suspensions. The results from the extrapolation of the steady shear flow curve are markedly lower. In fact, the accuracy of this method is very much dependent on the available range of low shear rate data. There is usually a significant difference when the results are obtained by extrapolating from shear rates greater or less than 1 1/s. Sedimentation of the 50 wt% TiO₂ suspension can also not be ruled out, although there was no visual evidence of it. Surface slip is a further possible cause for the relatively erratic behaviour of this suspension, the low value of τ_v from the penetrometer being consistent with the presence of slip. Shear history may also be more important in the case of TiO₂ suspensions as these materials are extremely time-dependent compared with Carbopol gels.

6. Conclusions

A simple static method using a penetrometer for measuring yield stress of a viscoplastic fluid has been systematically studied. The results from testing different Carbopol gels with a wide range of yield stress show reasonable agreement with values obtained using a rate-controlled rheometer and a stress-controlled rheometer. In the case of TiO_2 suspensions, there is a large variability in τ_y between methods. There is no reason to believe that the penetrometer results are less reliable than those from the other methods. No simple explanation can be offered for

Table 4. Comparison of results from different techniques

Solution	Penetration τ_y , Pa/ σ^*	Vane torsion τ_y , Pa/ σ^*	Extrapolation of flow curve τ _y , Pa Polynomial Casson/Herschel-Bulkley		Stress ramp τ _y , Pa	Creep** τ _y , Pa
0.1% Carbopol	6.8/1.38	7.3/0.15	6.9	-/ 5.6	6.2	6.0
Carbopol A	17.1/1.14	16.9/1.0				
Carbopol B	41.7/1.60	45.7/4.1		•		
Carbopol C	61.4/1.80	66.3/4.5				
50% TiO2	10.7/1.03	18.4/2.8	6.9	4.1/-	10.0	10.0
60% TiO2	60.9/-	56.6/4.1	21.0	8.8/-	36.0	37.0

^{*}σ is the standard deviation about the mean values tabulated

^{**}Yield stress was determined from a creep profile for a period of 100 s.

the discrepancies observed. Thus, this technique provides a relatively quick and low cost method of obtaining a realistic value of yield stress without the need for costly stress-controlled or speed-controlled rheometers.

The influence of creep on the measurement is negligible, provided the penetrometer has a well-roughened surface. The measuring time required for the penetrometer method could be as short as two minutes, although 5 to 10 minutes is recommended on the basis of the results presented here. The dimensions of the penetrometer have no systematic effect on the measurement. Wall effects can be neglected provided the diameter of the container is more than 4 times the diameter of the penetrometer.

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Nomenclature

- F_b Resistance force due to buoyancy.
- F_g Weight of the penetrometer.
- F_y Resistance due to yield stress.
- m Total mass of the penetrometer.
- d Diameter of penetrometer.
- h Immersed length of the cylindrical part.
- ρ Fluid density.
- $\tau_{\rm v}$ Yield stress.

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