High-throughput viscosity determinations

Citation for published version:

Digital Object Identifier (DOI):
10.1063/1.2976350

Link:
Link to publication record in Edinburgh Research Explorer

Document Version:
Publisher's PDF, also known as Version of record

Published In:
Review of Scientific Instruments

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Download date: 25. Oct. 2018
High-throughput viscosity determinations

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(Received 21 March 2008; accepted 3 August 2008; published online 11 September 2008)

A new high-throughput viscosimeter device was designed, built, and tested allowing measurement of the viscosity of 100 different solutions in a single experiment using the falling sphere approach. Using the corrected Stokes’ law, viscosities obtained by the HT device were compared to viscosities of the same solutions obtained on a conventional viscosimeter and showed excellent correlation. The theoretical set of data, correlated with Reynolds numbers, and the viscosities obtained for each sample showed very good correlation with the experimental data, demonstrating the robustness of the device.

I. INTRODUCTION

High-throughput and robotic methodologies have enabled the routine preparation and generation of large numbers of formulations in a highly efficient manner.1–3 This ability, however, then brings to the fore the requirement to rapidly characterize the formulations that are generated, with the need to determine a range of physical parameters, which includes viscosity and density.4 Viscosity is an especially important feature of fluids, relating to their resistance to deformation5–7 and plays an important role in not only understanding their behavior but also dictating the possible applications any specific fluid. Experimental viscosity measurements are thus very important in many industrial areas, for example, in printing or spraying process, which require materials that fall within specific viscosity regimes in order to have practical application; and thus with high-throughput formulation generation a need arises to be able to determine the viscosity of large numbers of samples in a parallel way.

Flow regimes are classified into laminar, transitional, and turbulent flow, with the specific flow conditions distinguished by so-called Reynolds numbers (Re) a dimensionless unit proportional to [(inertial force)/viscous force]. When Reynolds number is small, the fluid’s viscosity is dominant and, in this situation, fluid is described as being in laminar flow (and Stokes law applies). When the Reynolds number is large the fluid’s inertial effects rule and turbulent flow dominates (the flow conditions between laminar flow and turbulent flow are known as transitional flow).

There are four main types of viscometers designed to measure liquid viscosity:4–10 capillary viscometers, rotational viscosimeters, falling sphere, and vibrational viscosimeters, but the falling sphere approach has attracted attention due to simplicity of operation (the falling sphere approach allows fluid viscosity to be determined using gravitational effects and is described by Stokes’ law):

$$\mu = \frac{d^2 g (\rho_1 - \rho_2)}{18 V}.$$  \hspace{1cm} (1)

We, therefore, designed a novel device to characterize viscosity in an efficient high-throughput manner based on the parallelization of a falling sphere viscometer, using sphere shadow methodology as a detection approach as shown in Fig. 1. This allowed the viscosity of up to 100 Newtonian liquid compositions to be simultaneously determined by parallel measurements of the time required for a sphere to pass through the fluids.

II. EXPERIMENTAL

A. Materials

Polyvinyl chloride spheres with a diameter of 4.8 mm and a density of 1.41 g/cm³ were purchased from Engineering Laboratories inc. Glycerol, (+99.5%, spectrophotometric grade), density: 1.263 g/cm³ and ethylenediamine were purchased from Sigma-Aldrich.

B. Reference experiment for correction parameter determination

1 g of 4.8 mm diameter polyvinylchloride (PVC) spheres were added to 10 ml of an 80% solution of the ethylenediamine in distilled water and stirred at 80 °C for 1 h. The spheres were washed with water to remove excess ethylenediamine and dried at room temperature for 4 h.11

The PVC spheres were washed with glycerol and dried at room temperature for 5 min. Glycerol (5 ml) was added to a single 8 mm diameter tube and a PVC sphere was placed into a mesh hole plate, which upon withdrawing allowed the sphere to fall through the sample solution and the time for the sphere to fall from the 0 position to the 10.4 cm position was recorded.

C. High-throughput measurement of sample viscosities

The high-throughput viscosity device is depicted in Fig. 1. The system consisted of a light source, a mesh plate, an adapter mask plate, an array of holes, a base plate, and a web camera. The measuring tubes (8 mm diameter and PVC ma-
light material) were filled with 100 water-glycerol solutions library (shown in the Table I) and 5 ml of each sample were placed in each measurement tube of the high-throughput viscosity measurement device.

The PVC spheres were washed with the corresponding sample solutions, dried at room temperature for 10 min, placed in the mesh holes plate, and released into the solutions upon slowly withdrawing the mask plate, with the web camera triggered upon release and recording the events. When the spheres reached the holes, the holes were filled and became “dark” as is shown in Fig. 2 with a digital camera recording the images. Data processing was achieved using the methods described in elsewhere 2,3 by Image ProPlus.

D. Characterization of density and standard viscosity

The density of each sample solution was measured by a liquid density transmitter L-DENS. 1 The standard viscosity of each sample solution was measured using an automated microviscometer1 useful for a viscosity range from 80 to 2500 mPa s and was based on the falling ball principle. This consisted of a glass capillary (4 mm diameter \( \times \) 15.8 cm length with a stainless steel ball (3 mm diameter and a density of 8.75 g/ml). The measurement of dynamic viscosity was based on rolling of the ball inside the capillary at different inclination angles (from 20° to 70°). In these experiments the measurement of the viscosity was calculated taking the capillary through an inclination angle of 50° to get data comparable to the HT viscometer.

III. RESULTS AND DISCUSSION

A. Experimental, wall, and end corrections

As mentioned above Stokes’ law has limitations as it is valid only for laminar flow. In reality, the true terminal velocity of the falling ball must be corrected with respect to the determined experimental velocity value. This correction for terminal velocity is due to the fact that Stokes’ law works well only in fluid media with no inertial forces (when the ball moves slowly with no acceleration), no end effects (the ball falls steadily) and no wall effect (infinite Newtonian medium). In practical experiment, wall, inertial, and end effects need to be corrected for and \( K \) factors must be calculated to compensate for deviations in Stokes’ law caused by experimental procedures. 12–14 In this case corrections for wall and end effects were carried out, but no corrections were necessary for the inertial effect due to the slow motion of the ball inside the measurement tube.

A reference experiment was first performed using a single tube and pure glycerol. The time needed \( (t=790 \text{ s}) \) for a PVC sphere to reach the bottom of a single tube (10.4 cm long) in pure glycerol was measured and used in the corrected Stokes’ Eq. (2) to calculate the experimental correction factor \( K_0=0.0787 \).

\[
K = 18 \mu V/d^2 g(r_1 - r_2) = 18 \mu V/(d^2 g(r_1 - r_2) t). 
\]  (2)

The second correction considered the wall effect, 15–17 being the ratio between experimental and corrected terminal velocities calculated considering the ratio \( d/D \), using three equations:

(a) Francis equation,

\[
V/V_c = K_1 = [(1 - (d/D))/[1 - 0.475(d/D)]^4]. 
\]  (3)

(b) Ladenburg equation,

\[
V/V_c = K_2 = 1/[1 + 2.1(d/D)]. 
\]  (4)

(c) Faxen equation,

\[
V/V_c = K_3 = (1 - 2.104(d/D))^3 + 2.09(d/D)^5 - 0.95(d/D)^5. 
\]  (5)

The values of the \( K \) factors considering the wall effect correction are as follows: \( K_1=0.098, K_2=0.442, \) and \( K_3=0.115 \).

The third correction considered the end effect. 18 This was calculated considering various distances \( (h) \) of the ball with respect to the bottom of the tube, to ascertain the constant velocity nature of the ball falling into the tube. Values of \( h \) close to zero were studied: 0.0002, 0.00025, 0.0003, and 0.0004 m (the tube was 0.104 m). The equation for the end effect is as follows: 15,18

\[
V/V_c = [1 + (9/8)(d/2h)]^{-1} = K. 
\]  (6)

Four values of \( K \) were determined as a function of \( h \):

\( K_4=0.0689 \) \( (h=0.0002) \); \( K_5=0.0847 \) \( (h=0.00025) \); \( K_6=0.1 \) \( (h=0.0003) \); \( K_7=0.129 \) \( (h=0.0004) \).

B. High-throughput measurement of viscosities

The traveling time of a PVC sphere going through the sample solution was recorded by a web camera with each “hole” representing a sample solution. From Fig. 2 it can be seen, at the beginning, that all the holes were empty (the holes were bright), 100 s later, some holes were filled with spheres and became dark, and the number of dark holes increased with time. At 808 s all the holes were filled with spheres and became dark as shown in Fig. 2. Using this approach the time of the fall in each well was obtained and based on the corrected Stokes’ law equation as follows:
\[
\mu = K \rho d^2 \frac{g (\rho_1 - \rho_2)}{18 V_c}.
\]  

(7)

In order to study the performance of the high-throughput viscosity measurement device the viscosity of 100 samples were determined using both the HT device and a conventional automated viscosimeter. Corrections of terminal velocity \(V_c\) (considering experimental, wall, and end effects) were

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<th>Water (g)</th>
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FIG. 2. Sphere shadow pictures. (a) \(t=0 \) s, (b) \(t=100 \) s, (c) \(t=300 \) s, (d) \(t=650 \) s position, (e) \(t=750 \) s, and (f) \(t=808 \) s.
Reynolds numbers were taken in the range from 0.01 to 1.55 and were introduced into the Eqs. (10) and (11) to determine theoretical viscosity and theoretical terminal velocity.

\[
\mu = \left[\frac{4r^3g(p_1 - p_2)}{(9 \text{Re})}\right]^{1/2}, \tag{10}
\]

\[
V = (\text{Re} \mu)/(dp_2). \tag{11}
\]

The corrected experimental velocities (with different \(K_n\) values) were compared to the theoretical terminal velocities as shown in the Fig. 4.

Figure 4 shows that the terminal velocities of the samples determined with the HT viscometer increased as the time of the fall of the ball increases.

It can be seen that long fall times (100–1000 s) at all the terminal velocities were very close, suggesting high sample viscosity and low Reynolds number and low deviation from Stokes’ law. Theoretical velocity values estimated with low values of Reynolds numbers were very close to the corrected terminal velocities corresponding to the wall effect corrections \((K_1)\), end effect \((K_6)\) and the experimental correction factor \((K_0)\).

Reynolds numbers for the HT viscometer were also calculated using

\[
\text{Re} = \frac{2\rho_2 r V}{\mu} = \frac{4r^3(p_1 - p_2)g/9\mu^2}. \tag{12}
\]

Figure 5 shows that there is a dramatic deviation of the Reynolds numbers of the samples from both devices; however, the viscosity values are very similar in both cases due to the corresponding correction constants. It can be said that for low Reynolds number values in the range from 0.1 to 0.01, the majority of the samples in both devices coincides, because the viscosity values in both of them are higher and thus Stokes’ law complies very well. On the other hand there are deviations in both devices when the Reynolds numbers are in the range from 0.1 to 0.5, due to the low viscosity of the samples as well as the physical parameters of each device such as spheres (size and density), the lengths and diameters of the capillary (conventional viscometer) and tubes (HT).

\[
V_c = V \exp/K_n. \tag{9}
\]
C. Statistical treatment

Statistical analysis based on both descriptive analysis and viscosity values comparisons were carried out. Descriptive analysis allowed determination of the main statistical descriptors for each viscosity data set determined for each correction factor ($K_0$ and $K_5$) and for the standard viscometer. Test analyses were carried out by analysis of the correction factors determined from the high-throughput viscometer and those determined using the standard viscometer. Despite the many correction factors determined, those with the best fit with the viscosity values of the standard viscometer were chosen, with two correction factors $K_0$ (experimental) and $K_5$ (end effect for $z=0.00025$) selected.

1. Descriptive analysis

This was carried out by considering an analysis of the distribution of each population of viscosity values, determined individually, for each correction factor with the standard viscometer. This analysis was based on the study of cumulative frequency, which gives the values of the viscosity population for a given value of percentage, considering 100% of the population. Analysis of the data distribution was carried out as shown in the Table II.

Table II shows that the data distribution was not symmetric with a skew to the right. It was found that when comparing, both the mean and the standard error of the standard viscometer with the HT viscometer ($K_0$), their values were nearly identical, however, there was more variation in the case of the standard viscometer when compared to the HT viscometer, with a $K_5$ value of 6.32% for the mean and 8.85% for the standard error.

For lower values of the percentile (10% to 25%), the data distribution showed viscosity values were virtually identical for the standard and the HT viscometer ($K_0$). The data distributions were indeed similar for the case of the standard viscometer and the HT viscometer, for percentile values equal or greater than the median values.

Confidence intervals from the HT and the standard viscometers were determined as shown in Table III.

2. Viscosity value comparison: Test analysis

The comparison was carried out comparing the results of the standard viscometer (reference) and the HT viscometer, using two factor corrections: $K_0$ (experimental) and $K_5$ (end effect for $z=0.00025$) as follows.

Partial least square analysis. In this case correlation analysis was carried out to check the fit of both correction factors with the standards, with analysis carried out using linear regression, using

$$y = a + bx.$$  \hspace{1cm} (12')

The partial least-squares analysis gave the main parameters as shown in Table IV.

Table IV shows that there was good correlation between the two correction factors (slope $b$ were very similar, with only approximately 7% of variation), while their intercepts ($a$), had only 5.7% variation. However the standard error of the estimate ($S_{yx}$) of the regression was higher in the case of the comparison with the correction factor ($K_5$) than the comparison of the correction factor ($K_0$) (approximately 8.6% of variation).

Fisher distribution-test. A Fisher distribution-test ($F$-test) was used with the aim at comparing the variance of two populations using a one-tailed test for $p=0.05$. This comparison was carried out by determining the $F$ ratio of the two populations as shown in expression (13) and comparing the ratio with a critical value of the Fisher distribution ($F_c =1.394$) for a distribution with 95% and 99% degrees of

### Table II. Statistical descriptors of the viscosity data distribution.

<table>
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<tr>
<th>Statistical descriptor</th>
<th>Standard viscometer (mPa s)</th>
<th>HT viscometer (correction factor: $K_0$) (mPa s)</th>
<th>HT viscometer (correction factor: $K_5$) (mPa s)</th>
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<td>465</td>
</tr>
</tbody>
</table>

### Table III. Confidence intervals for the HT and the standard viscometers.

<table>
<thead>
<tr>
<th>Data distribution (%)</th>
<th>HT viscometer (correction factor: $K_0$) (mPa s)</th>
<th>HT viscometer (correction factor: $K_5$) (mPa s)</th>
<th>Standard viscometer (mPa s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>95</td>
<td>485 ± 56</td>
<td>522 ± 60</td>
<td>480 ± 55</td>
</tr>
<tr>
<td>99</td>
<td>485 ± 73</td>
<td>522 ± 79</td>
<td>489 ± 72</td>
</tr>
</tbody>
</table>

### Table IV. Main parameters of the PLS analysis.

<table>
<thead>
<tr>
<th>Viscosity comparison between HT correction factors and Standard</th>
<th>$a$</th>
<th>$b$</th>
<th>$r^2$</th>
<th>$S_{yx}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Correction factor ($K_0$) vs standard</td>
<td>−12.69</td>
<td>1.0191</td>
<td>0.9975</td>
<td>14.0</td>
</tr>
<tr>
<td>Correction factor ($K_5$) vs standard</td>
<td>−13.45</td>
<td>1.0964</td>
<td>0.9975</td>
<td>15.31</td>
</tr>
</tbody>
</table>
the correction factors and the standard viscosimeter was not
therefore it can be said that for sample mean difference
differences between the sample means were significant.
standard viscometers the higher the mean
viscosity values for the standard viscometer:
Viscosity comparison between the
and the viscosity values of the HT method for each correction factor
and a one tail distribution.
F
F
freedom (v).
If the variance of the population \( 1[\sigma^2(1)] \) is similar to
the population \( 2[\sigma^2(2)] \) and \( F < F_c \), then the two populations
have the same variance.
\[
F = \frac{\sigma^2(1)^2}{\sigma^2(2)^2} \frac{v_2}{v_1}.
\] (13)
In this case, comparison of the viscosity data set for the two
factor corrections (\( K_0 \) and \( K_5 \)) with the viscosity values of the
standard viscometer was carried out looking at the \( F \) ratio
of variance, considering a 5% confidence level (\( p=0.05 \)),
and a one tail distribution.
Table V shows the values of \( F \) determined for the viscosity
values of the HT method for each correction factor
and the viscosity values for the standard viscometer:
Table V shows that \( F < F_c \) for both correction factors.

<table>
<thead>
<tr>
<th>Correction factor (( K_0 )) vs standard</th>
<th>( F )</th>
<th>( p )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Correction factor (( K_5 )) vs standard</td>
<td>1.041</td>
<td>0.420</td>
</tr>
<tr>
<td></td>
<td>1.205</td>
<td>0.177</td>
</tr>
</tbody>
</table>

The values of these sample mean differences (\( X_{HT-X_5} \)) were
higher than the values of the difference of the population mean
(\( \mu_{HT-X_5} \)), being \(-3.5 \) mPa s for the case of the correction
factor (\( K_0 \)) and \(33.7 \) mPa s for the case of the correction
factor (\( K_5 \)), respectively. For each correction factor (\( K_0 \) and
\( K_5 \)) of the HT viscometer and its comparison with the standard
viscometer the following results (Table VI) were generated.
Table VI shows that the higher the sample mean difference
between the correction factors of the HT and the standard
viscometers the higher the \( z \) values were and so the differences between the sample means were significant.
Therefore it can be said that for sample mean difference
\( \pm 150 \) mPa s, the difference between the viscosity values of the
correction factors and the standard viscosimeter was not
significant and \( z < z_c \), complying with the null hypothesis.

\[
H_0: (X_{HT-X_5}) \leq 150 \quad (\text{sample mean difference no significant at } p = 0.05),
\]

\[
H_1: (X_{HT-X_5}) > 150 \quad (\text{sample mean difference significant at } p = 0.05).
\] (15) (16)

On the other hand for the correction factors with viscosity
values of 250 mPa s more than the viscosity values of the
standard viscometer, the difference was significant because
the probability (\( p \) values) was very low and <0.05.

IV. CONCLUSIONS

A high-throughput viscosity measurement device was fabricated based on the principle of a falling sphere viscometer
and assisted sphere shadow methodology. A series of 100
glycerol/water fluids was used to test and validate the high-
throughput viscosity measurement device in comparison to a
traditional viscometer. The results demonstrated that the
high-throughput viscosity measurement device worked
exceptionally well under low Reynolds number flow conditions,
and that this device could be considered as an easy to
use and low cost novel measurement device. The one limitation
was that because light must pass through the fluids, the
device is limited to physically similar transparent solutions.

Statistical treatment showed a slightly skewed trend, but
the data distribution was similar to the standard viscometer
with similar standard errors. Partial least-squares (PLS)
analysis gave good correlation coefficients, while \( F \) and \( z \)
tests showed precision and accuracy in the comparison of the
correction factors (\( K_0 \) and \( K_5 \)) from the HT and standard
viscometers.
V. Supplemental material available

Supplemental tables with the determination of density, viscosity, Reynolds numbers of the 100 samples, and the velocity of the ball inside the device, for both the HT and the standard viscosimeter devices are available, as well as the data for the theoretical values of Reynolds number, viscosity, and velocity.

ACKNOWLEDGMENTS

The authors thank the EPSRC-GB for funding.

NOMENCLATURE

- $d =$ ball diameter (m)
- $D =$ tube diameter (m)
- $L =$ tube length (m)
- $t =$ falling time (s)
- $V_{\text{exp}} =$ experimental velocity (m/s)
- $V_c =$ corrected velocity (m/s)
- $V =$ theoretical velocity (m/s)
- $\mu =$ viscosity (mPa s)
- $\rho_1 =$ ball density (kg/m$^3$)
- $\rho_2 =$ sample density (kg/m$^3$)
- $g =$ gravity acceleration (9.8 m/s$^2$)
- $R_e =$ Reynolds number
- $r =$ ball radius (m)
- $h =$ distance of the ball from the bottom (m)
- $K_n =$ correction constant for $n=0-7$
- $a =$ intercept
- $b =$ slope
- $r^2 =$ correlation coefficient
- $S_{\text{HT}} =$ standard error of estimate
- $X_{\text{HT}} =$ viscosity mean value of the chosen ten samples from the HT viscometer
- $X_S =$ viscosity mean values of the chosen ten samples from the standard viscometer
- $\mu_{\text{HT}} =$ viscosity mean value of the population of the HT viscometer

$\mu_s =$ viscosity mean value of the population of the standard viscometer

$p =$ level of significance

$v_1 =$ freedom degree of population 1

$v_2 =$ freedom degree of population 2

$\sigma^2_{\text{HT}} =$ variance of the viscosity values of the HT viscometer

$\sigma^2_S =$ variance of the viscosity values of the standard viscometer

$N_{\text{HT}} =$ number of observations of the population belong to the HT viscometer

$N_s =$ number of observations of the population belong to the standard viscometer