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Research Article

High Pressure Falling Sinker Liquid Viscosity Determination without Supplementary Density Data: A New Approach

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Accurate measurement and determination of liquid viscosity data under high pressure conditions requires knowledge of liquid density data. In this study, a high pressure falling sinker-type viscometer was used to determine the viscosity of n-dodecane at elevated pressures up to 132 MPa without supplementary knowledge of liquid density. The viscometer, which involves the downward movement of a cylindrical sinker under the influence of gravity through the liquid contained within a closed tube, avoided the need for density data by repeating the sinker-timing experiments with two geometrically similar but different-sized sinkers thereby allowing the liquid density in the associated formulae to be eliminated. Furthermore, it was possible to subsequently derive liquid density. Both viscosity and density data were compared to published data for which good correlation was found for viscosity. To minimize errors, it is suggested that the two sinkers for such an approach should be of sufficiently differing densities.

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1. Introduction

The viscosity of liquids is an important property in fluid flow and the estimation of other physical properties such as in the estimation of diffusion in liquid systems [1]. Accurate data is therefore required for many applications in engineering design. The high pressure falling body viscometer is a simple and effective device for the accurate measurement of viscosity for liquids over a wide range of viscosities [2–12]. Consisting of a vertically positioned closed tube, a cylindrical sinker is allowed to descend at terminal velocity under the influence of gravity through a liquid under investigation. The viscosity is readily determined from the time for the sinker to descend a known distance and the physical details of the viscometer. However, under very high pressure conditions, the calculations are not so straightforward as liquids may not be assumed to be incompressible since density data is required to complete the viscosity calculations. Where experimental data may not be available, published experimental data may be available; predictive approaches based on critical point data or multiparameter corresponding states methods may be used for many pure fluids and some mixtures [13–16]. For many complex fluids and mixtures such as automotive fluids, liquid foods and mixtures such as biodiesels, density data may, however, not be available [17]. Although the critical point data can be predicted in some cases, this can lead to complication and mathematical uncertainty which can significantly influence the determined viscosity data. The resulting error in the data may be correspondingly very significant thus rendering it unreliable for good scientific and engineering purposes. To overcome the need for compressibility data for liquids at high pressure, we examined the use of two geometrically similar sinkers to determine liquid viscosity as a way of overcoming the need for supporting density data.

2. Falling Body Viscometer

The high-pressure viscometer used in this work was based on the falling sinker design. Designed and fabricated by the National Engineering Laboratory (NEL) in East Kilbride, UK, the viscometer consists of a vertical tube containing a test liquid through which the sinker falls freely under the influence of gravity (Figure 1). The rate of descent of the sinker which falls concentrically is related to the displacement of liquid and frictional effects in the annular
region between the tube and the sinker. As the sinker descends, the liquid is displaced upwards forcing it to pass through the annular gap between the walls of the tube and the sinker.

The sinker and the tube are fabricated from a solid bar of nonmagnetic stainless steel to minimise compressibility and thermal expansion effects. Designed to be self-centring, the sinker used has a cylindrical body with a hemispherical nose embedded with a ferrite core (Figure 2). Ratios of sinker to inner tube radii in excess of 0.95 are required for the sinker to descend concentrically within the tube. Sinkers which fall eccentrically are well known to cause significant errors [18, 19].

The viscosity is determined directly from the time taken for the sinker to descend a fixed distance. In this case, this is the distance between two detection coils wrapped around the outside of the tube with the location of the sinker being detected by the change in inductance in the coils. The coils are set a distance of 12 mm apart and consist of around 200 turns of lacquered copper wire, each with approximately equal electrical resistance, and both forming the active arm of a balanced bridge circuit. The out-of-balance signal from the bridge is amplified and passed through an AC/DC converter to give a DC signal which increases to a maximum when the ferrite core of the sinker is positioned at the centre of the coil (Figure 3). From the dimensions of the viscometer tube and sinker, and the time for the sinker to fall freely with terminal velocity between two points, the viscosity can be determined.

Samples of n-dodecane oil were sealed into the tube using a shrinkable PTFE expansion sheath. It was essential that neither air nor particles remained entrapped with the sample which could influence the rate of sinker descent.
The tube and associated assembly was housed in a high-pressure vessel and maintained at a constant temperature by air circulation (Figure 4). The pressure is transmitted in the oil through the flexible PTFE sheath using a paraffin/Shell Tellus oil mixture as the hydraulic medium by pressure amplification to a maximum possible operating pressure of 500 MPa. This mixture offers good lubricating properties with a low freezing point. The entire high pressure vessel itself is mounted on bearings and rotated 180° to return the sinker to its original starting position between each measurement. A calibrated Kistler piezo-resistive pressure gauge, type 46180A and calibrated to 500 MPa, was used to measure the high pressure within the viscometer tube.

3. Analysis
The rate of descent of a sinker in a closed tube is based on a force balance of gravity, buoyancy and resistive shear forces with fully developed laminar flow and has been previously shown [20, 21] in which the viscosity can be expressed in the form

$$\mu = \frac{(1 - (\rho_f/\rho_o)) t}{A(1 + 2\alpha(T - T_o))(1 - 2/3\beta(P - P_o))},$$

where

$$A = \frac{2\pi L o L i O}{m g (\ln(r_2/r_1) - ((r_2^2 - r_1^2)/((r_2^2 + r_1^2)))}. \quad (2)$$

The thermal expansion coefficient and compressibility of the sinker are small ($7.6 \times 10^{-6}$ K$^{-1}$ and $3.075 \times 10^{-6}$ MPa$^{-1}$, resp.) and can thus be ignored. In contrast, small measurement errors in radii $r_1$ and $r_2$ result in significant errors; the effects of which have been studied by Wehbe et al. [22]. Ideally, the coefficient $A$ is based on the physical dimensions of the sinker as shown. In practice, however, there is often a deviation in viscosity determined experimentally due to wall and end effects [23–26]. The coefficient $A$ is therefore adjusted using a calibration liquid of known properties under high pressure [27]. To confirm the cause of these deviations 2D and 3D CFD studies have previously shown the presence of vortices shedding from the trailing edge of the sinker. The simulations have shown that fully developed laminar flow is not met within the annual gap [21, 28]. It has, however, been confirmed that coefficient $A$ tends towards the theoretical value for a modified Reynolds numbers below 60 [18], where

$$Re_m = 2r_1^2 \frac{\rho_f v}{(r_1 + r_2) \mu}. \quad (3)$$

For two geometrically similar sinkers tested in the same liquid at the same temperature and pressure, the density of the liquid can be deduced to be

$$\rho_f = \frac{\rho_1 \rho_2 A_2 t_1 - \rho_1 \rho_2 A_1 t_2}{\rho_2 A_2 t_1 - \rho_1 A_1 t_2}. \quad (4)$$

Likewise, for the two sinkers, the viscosity is obtained from

$$\mu = \frac{\rho_2 A_1 t_2 - \rho_1 t_1 t_2}{\rho_2 A_2 t_1 - \rho_1 A_1 t_2}. \quad (5)$$

To determine both the viscosity and density, this may be possible for two measurable and distinguishable values of $A$ and sinker descent times.

4. Materials and Methods
In this work, n-dodecane with a purity greater than 99% purchased from VWR International Ltd, UK was used as the test liquid. The properties of n-dodecane have been previously reported [29].

4.1. Density. The density data was based on using a Tait-type equation of the form

$$\rho_f = \rho_o \left(1 - C \log_{10} \left(\frac{B + P}{B + P_o}\right) \right)^{-1} \quad (6)$$

in which

$$\rho_o = \sum_{i=0}^{2} a_i T^i, \quad (7)$$

$$B = \sum_{i=0}^{2} b_i T^i. \quad (8)$$

4.2. Viscosity. The viscosity was found from

$$\mu = \mu^* \left(4.779 \times 10^{-9} V^{-2/3} M^{1/2} T^{1/2}\right) \quad (9)$$

for which the dimensionless viscosity was calculated from

$$\mu^* = \frac{1}{\sum_{i=0}^{3} d_i (V/V_o)^i} \quad (10)$$

where the core volume is dependent on the absolute temperature in the form

$$V_o = \sum_{i=0}^{3} e_i T^i. \quad (11)$$

The parameters are given in Table 1.

5. Sinker Selection
To determine the viscosity without supplementary density data, it is important to select sufficiently different sized but geometrically similar sinkers. There are certain other criteria that must also be fulfilled in this selection process. In particular, the sinker is required to be self-centering during descent. The time for descent for the two sinkers is also required to be appreciably different, for which the time for descent is

$$t = \frac{A \mu}{1 - (\rho_f/\rho_o)}. \quad (12)$$

It is worth noting that since the density of most liquids does not increase significantly with pressure then as the pressure increases large values of $A$ contributes to longer descent.
time. Conversely, similar values for $A$ imply the need for appreciably different sinker densities. The details of the two sinkers used in this study are given in Table 2.

For both sinkers used in this study, readings for the time for sinker descent were recorded over a pressure range of 0.1 MPa to 132 MPa. Above this applied pressure, the sinker was not found to descend. This may have been due to a pressure-induced phase change. This phenomenon is found in certain liquids including hydrocarbons and lipids. All readings were repeated in triplicate.

### 6. Results

The sinker fall times for both sinkers are shown in Figure 5. This shows a rising increase in time with increasing applied pressure up to 132 MPa. Beyond this pressure, the sinker failed to descend. The modified Reynolds number for both sinkers at these pressures is shown in Figure 6. For both sinkers, the experimental coefficients $A$ tend towards a constant value for which the average values were found to be $16122.5 \text{ Pa}^{-1}$ ($\sigma_{\text{n-1}} = 320.2 \text{ Pa}^{-1}$) for sinker 1 while for sinker 2, $A$ was $4062.6 \text{ Pa}^{-1}$ ($\sigma_{\text{n-1}} = 56.0 \text{ Pa}^{-1}$). For comparison, the values for $A$ calculated from the dimensions of sinker and tube are $16575.5 \text{ Pa}^{-1}$ and $3890.7 \text{ Pa}^{-1}$, respectively (Table 2), and are equivalent to a zero modified Reynolds number. While the two experimental and theoretical values are reasonably close, it should be noted that differences exist due to inaccuracies in measurement as well as end effects during sinker descent described earlier. Table 3 presents the sinker fall data and coefficients for the same applied pressures.

The deviation of evaluated viscosity and density data is presented in Table 4 and Figures 7 and 8.

Using this new approach, the determined viscosity data is comparable to that of Caudwell et al. [29]. The source of the errors obtained can be attributed to the variation of the coefficient $A$ for which a summary of the errors is presented in Table 5.

### 7. Error Transfer Analysis

Although the error transfer analysis can be used to identify parameters other than $A$ which are able to reduce the error caused by the coefficient in density determination, it is worth noting that the errors in both calibration coefficients may be of significance. In this analysis, the errors are assumed to be associated with both coefficients $A_1$ and $A_2$. In general, by assuming an error $\Delta R$ on the final parameter $R$ for which

$$R + \Delta R = f(u_1 + \Delta u_1, u_2 + \Delta u_2, \ldots, u_n + \Delta u_n)$$

then using Taylor’s series expansion with only first order terms, the error can be given in the form:

$$R + \Delta R = f(u_1, u_2, \ldots, u_n)$$

$$+ \left( \frac{\partial R}{\partial u_1} \Delta u_1 + \frac{\partial R}{\partial u_2} \Delta u_2 + \cdots + \frac{\partial R}{\partial u_n} \Delta u_n \right)$$

for which the relative error term is thus

$$\frac{\Delta R}{R} = \frac{\partial \ln R}{\partial u_1} \Delta u_1 + \frac{\partial \ln R}{\partial u_2} \Delta u_2 + \cdots + \frac{\partial \ln R}{\partial u_n} \Delta u_n.$$
Based on the above, the relative error in density evaluation can be written as

\[
\frac{\Delta \rho_f}{\rho_f} = \frac{\partial \ln f(A_1, A_2)}{\partial A_1} \Delta A_1 + \frac{\partial \ln f(A_1, A_2)}{\partial A_2} \Delta A_2
\]  

(15)

Using \( x \) to represent the relative error for the corresponding coefficient value of sinker, the error in the calculated density is, after simplification

\[
\frac{\Delta \rho_f}{\rho_f} = \frac{A_1 A_2 t_1 t_2 (\rho_{a1} - \rho_{a2})}{(A_1 t_2 \rho_{a1} - A_2 t_1 \rho_{a2})(A_1 t_2 - A_2 t_1)} (x_1 - x_2).
\]  

(17)

The relative error is therefore proportional to the difference of the two relative errors in \( A \). However, the magnitude of the errors shown in Table 6 do not conclude that the errors were attributed to the coefficients \( A_1 \) and \( A_2 \).

In terms of fall times \( t_1 \) and \( t_2 \), the approach to take the relative error as

\[
\frac{\Delta \rho_f}{\rho_f} = \frac{A_1 A_2 t_1 t_2 (\rho_{a1} - \rho_{a2})}{(A_1 t_2 \rho_{a1} - A_2 t_1 \rho_{a2})(A_1 t_2 - A_2 t_1)}
\]  

(18)

rewritten as

\[
\frac{\Delta \rho_f}{\rho_f} = \frac{(\rho_{a1} - \rho_{a2})}{(\rho_{a1} A_1 t_2 / A_2 t_1 - \rho_{a1} + (\rho_{a2} A_2 t_1 / A_1 t_2) - \rho_{a1})}
\]  

(19)

with further simplification in which

\[
\frac{A_1 t_2}{A_2 t_1} = \frac{1 - (\rho_f / \rho_{a1})}{1 - (\rho_f / \rho_{a2})}
\]  

(20)
In this case, to minimize the error in liquid density with respect to the liquid density in which
\[
\frac{d\left(\Delta \rho_f / \rho_f \right)}{\rho_f} = 0
\] (22)
gives \( \rho_f = \sqrt{\rho_1 \rho_2} \) suggesting that the liquid density should be the geometric mean of the two sinks. Care should be taken with this result since it implies that one density should be less than that of the liquid thereby violating the phenomenon of buoyancy.

### 7.2. Viscosity

Again, it is worth noting that the errors between average value of A and experimental data are of great importance. The errors for A have been shown previously (Table 6) and are not large. The error transfer analysis was carried out to show how the errors between the average values of the two calibration coefficients and the corresponding experimental data which can affect the final error in viscosity determination. The basic condition is that the errors between the average and experimental data of A are not big.

The error analysis for viscosity determination is based on
\[
\mu = f(A_1, A_2) = \frac{\rho_2 A_1 t_1 - \rho_1 A_2 t_2}{\rho_2 A_2 t_1 - \rho_1 A_1 t_2} \quad (23)
\]
in which the Taylor series expansion is
\[
\frac{\Delta \mu}{\mu} = \frac{\partial \ln f(A_1, A_2)}{\partial A_1} \Delta A_1 + \frac{\partial \ln f(A_1, A_2)}{\partial A_2} \Delta A_2, \quad (24)
\]
and
\[
\frac{\Delta \rho_f}{\rho_f} = \frac{(\rho_1 - \rho_f) (\rho_2 - \rho_f)}{(\rho_1 - \rho_2) \rho_f} (x_1 - x_2). \quad (21)
\]
Table 6: Relative errors in calibration coefficients.

| Pressure (MPa) | $x_1$ (%) | $x_2$ (%) | $x_1 - x_2$ (%) |
|---------------|----------|----------|----------------|
| 14.5          | 1.62     | 1.93     | -0.31          |
| 20.3          | 1.98     | 1.37     | 0.61           |
| 24.3          | 3.57     | 0.78     | 2.79           |
| 31.2          | 0.77     | 2.06     | -1.29          |
| 35.2          | 0.36     | 1.84     | -1.48          |
| 39.7          | -0.09    | 0.86     | -0.95          |
| 45.2          | 0.09     | 1.05     | -0.96          |
| 49.9          | 0.7      | 1.01     | -0.31          |
| 54.5          | 1.57     | -0.35    | 1.92           |
| 59.8          | 0.4      | -0.61    | 1.01           |
| 64.8          | 0.68     | -0.57    | 1.25           |
| 70.4          | 0.67     | -0.19    | 0.86           |
| 75            | -0.63    | -1.24    | 0.61           |
| 81            | 0.55     | -0.08    | 0.63           |
| 85.9          | -1.32    | -1.02    | -0.3           |
| 95.2          | -2.83    | -1.64    | -1.19          |
| 115.1         | -2.18    | -1.34    | -0.84          |
| 120.5         | -2.54    | -0.97    | -1.57          |

where

$$\frac{\partial \ln f(A_1, A_2)}{\partial A_1} = \frac{\rho_{11} t_2}{(\rho_{22} A_2 t_1 - \rho_{11} A_1 t_2)},$$

$$\frac{\partial \ln f(A_1, A_2)}{\partial A_1} = \frac{\rho_{22} t_2}{(\rho_{22} A_2 t_1 - \rho_{11} A_1 t_2)}. \quad (25)$$

The largest possible relative error in the calculated density is, after simplification

$$\frac{\Delta \mu}{\mu} = \frac{x_1 - (\rho_{22} A_2 t_1 / \rho_{11} A_1)}{(\rho_{22} A_2 t_1 / \rho_{11} A_1) - 1}. \quad (26)$$

Writing in the form of fluid density gives

$$\frac{\Delta \mu}{\mu} = \frac{(\rho_{22} - \rho_f) x_2}{\rho_{11} - \rho_f} - \frac{(\rho_{11} - \rho_f)x_1}{\rho_{11} - \rho_{22}}. \quad (27)$$

To minimize the error in which

$$\frac{d(\Delta \mu/\mu f)}{\rho f} = 0 \quad (28)$$

gives $1/(\rho_{11} - \rho_{22}) \rightarrow 0$. This suggests that the widest possible difference in density is required.

8. Conclusions

It is possible to use the high pressure falling body sinker to determine the viscosity of liquid without the need for supplementary knowledge of liquid density data. Good agreement is found with determining the viscosity data although less so for the derived density data. There are inherent errors in this new approach which have been addressed. However, in order to ensure that the errors remain as small as possible, it is recommended that sinkers should be used, which exhibit good self centering properties. The radius ratio of the sinker to inner tube should be greater than 0.95. Additionally, it is necessary to use two sinkers with a large difference in value of A to avoid inaccuracies. The density of the sinkers should be obviously greater than that of the liquid tested so that it is capable of descend although the bulk densities of the two sinkers should be sufficiently dissimilar.

Nomenclature

$a$–$e$: Constant

$A$: Calibration coefficient (Pa$^{-1}$)

$B$: Constant (Pa)

$C$: Constant (–)

$g$: Gravitational acceleration (m$\cdot$s$^{-2}$)

$L_i$: Length of sinker (m)

$L_{d}$: Distance between the centre of two detecting coils (m)

$L_{0}$: Original length of the sinker (m)

$L_{0d}$: Distance between two detecting coils (m)

$m_{s}$: Mass of sinker (kg)

$M$: Molecular weight (kg$\cdot$mol$^{-1}$)

$P$: Pressure (Pa)

$P_{0}$: Reference pressure (Pa)

$r_{2}$: Inner radius of tube (m)

$r_{1}$: Radius of sinker (m)

$R$: Parameter

$Re_{m}$: Modified Reynolds number (–)

$t$: Falling duration of the sinker (s)

$T$: Temperature (K)

$T_{0}$: Reference temperature (K)

$u$: Parameter

$v$: Terminal velocity (m$\cdot$s$^{-1}$)

$V$: Molar volume

$V_{0}$: Molar core volume

$x$: Relative error (%).

Greek

$\alpha$: Thermal expansion coefficient (K$^{-1}$)

$\beta$: Compressibility of the sinker (MPa$^{-1}$)

$\mu$: Viscosity of fluid (Pa$\cdot$s)

$\mu^{*}$: Dimensionless viscosity (–)

$\rho_{f}$: Density of fluid (kg$\cdot$m$^{-3}$)

$\rho_{2}$: Reference density (kg$\cdot$m$^{-3}$)

$\rho_{s}$: Density of sinker (kg$\cdot$m$^{-3}$).

Subscript

1: Sinker 1

2: Sinker 2.
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