Rheology Of Aerospace Composites
(Picture frame and Squeeze flow testing of unidirectional polymer composites)

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Summary

This research examines the effect of the viscosity of a thermoplastic unidirectional polymer composite, Twintex, on its processing characteristics. Specifically trying to solve errors in comparisons of previous work, and to improve a model for textile composites developed at the University of Nottingham. Twintex is a thermoplastic polymer composite with a polypropylene matrix and 60% (by weight) glass fibre reinforcement. It is produced by Saint-Gobain in a multi-directional form only and so unidirectional samples were created specifically for testing. Testing was completed using a squeeze flow method in which the samples were compressed with the temperature and surface area being independently varied, and a picture frame method where the sample is extended with thickness of the sample varied. Both methods attempt to find a relationship between the two methods and remedy the problems of comparing the previous work by Shuler and Advani[2], and McGuiness and O’Braidaigh [3]. After having consulted the previous work, and tailored the experiments accordingly the results were analysed using a power law derivation for the squeeze flow data, and the analytical methods from the work done by McGuiness and O’Braidaigh [3] for the picture frame data.

The analysis showed that the transverse viscosity of the sample dropped with a higher temperature in the squeeze flow testing, which corroborates the micro-mechanical model by Christensen [10] and was expected, and also that with a greater surface area the transverse viscosity was lower, with the plate length being halved, the viscosity dropped by approximately a third. This was not as expected and goes against assumptions made by McGuiness and O’Braidaigh [3], and also the theory that the bulk viscosity is not affected by changing the length of the testing surface. There was not enough data from the picture frame testing to find a concrete relationship due to the lack of time available for testing, however, the data collected corroborates McGuiness and O’Braidaigh’s work and shows exactly the same disparity as the comparison of McGuiness and O’Braidaigh’s [3] work with Shuler & Advani’s [2] work. Heaters were designed for the picture frame apparatus due to edge effects affecting result accuracy in previous work, the use of these ensured a uniform sample temperature of the sample and their effect was proven using thermal imaging techniques, making the data more accurate.

It is clear that with a smaller surface area manufacturing speeds must be lower for high quality parts manufacture, and that the temperature of manufacturing should be specifically tailored to the material being used. The differences between the previous work done by Shuler and Advani[2], and McGuiness and O’Braidaigh [3] is suggested to be due to the analysis method proposed by McGuiness and O’Braidaigh [3], as the testing methods are valid and the squeeze flow data agrees with existing models, indicating a potential problem with the picture frame analysis. This requires further investigation.
Contents

Section                        Page

Nomenclature                   4
1. Introduction & Objectives   5
2. Experimental Set up         7
   • 2.1 Material Creation     7
   • 2.2 Squeeze Flow testing  9
     2.2a Calibration          9
     2.2b Testing             9
   • 2.3 Picture Frame testing 11
3. Results & Analysis          15
   • 3.1 Calibration Data      15
   • 3.2 Squeeze flow results  16
     3.2a Surface area testing 16
     3.2b Temperature testing  16
   • 3.3 Picture Frame Data    17
   • 3.4 Calibration Analysis  17
   • 3.5 Squeeze Flow Analysis 19
     3.5a Surface area         20
     3.5b Temperature          21
   • 3.6 Picture Frame Analysis 23
   • 3.7 Comparison Analysis  25
4. Discussion                  26
5. Conclusions                 27
6. References & Further Reading 27

Appendices                    29
   • Appendix A - Raw data     29
     A.1 Calibration data      29
     A.2 Squeeze flow data     29
     A.3 Picture frame data    29
   • Appendix B - Apparatus Designs 30
     B.1 Picture Frame General
     Arrangement Drawing
     B.2 Squeeze Flow Plates General
     Arrangement Drawing
   • Appendix C – Derivations  32
     C.1 Newtonian            32
     C.2 Power Law           35
     C.3 Calibration        37
   • Appendix D - Calibration verification 38
   • Appendix E - Work Plan  42
Nomenclature

Squeeze flow formulae

\( V_x \) - Velocity in the x direction
\( V_y \) - Velocity in the y direction
\( h \) - Gap between plates
\( h_0 \) - Original gap between plates
\( \frac{dh}{dt} \) – Closure rate of plates
\( L \) – Half of the plate length
\( w \) – Plate width
\( Q \) – Volumetric flow rate
\( \eta_0 \) - Transverse Viscosity
\( P \) – Pressure
\( F \) – Force
\( N \) – Power law model exponent
\( \dot{\gamma} \) - Shear rate
\( \frac{dp}{dx} \) – Pressure gradient

Picture Frame formulae

\( \dot{d} \) - Testing speed / rate of extension
\( \gamma^\prime \) - Rate of angular displacement
\( \gamma \) - Angular displacement
\( t \) – Thickness
\( L \) – Length of picture frame rig between hinges
\( m_T \) - Transverse viscosity
\( m_L \) - Longitudinal viscosity
\( d \) – Extension
\( n \) – Power law model exponent

Calibration formulae

\( t \) – Time
\( h \) - Gap between plates
\( F \) – Force
\( \frac{dh}{dt} \) – Closure rate of plates
\( L \) – Plate length
\( \eta \) - Viscosity
2. Introduction

Polymer composites are a relatively new material, with their own individual characteristics and production methods. Currently polymer composites are becoming increasingly popular, replacing more traditional materials such as steel in many applications due to their advantageous strength to weight ratios. As a result composites are playing a growing role in the commercial world. Not everything is understood of how polymer composites behave in manufacturing processes and so areas such as cost of production and part complexity suffer as a result. Use of composites used to be restricted due to the dominance of thermosetting polymers, but now thermoplastic composites in many forms are competing effectively against thermosets thanks to new manufacturing processes. This is due to thermoplastics offering clear advantages over thermosets, such as a relatively simple chemical structure that gives better resistance to solvents and impacts, as well as unlimited shelf life, ease of recycling, and the potential for high volume of production.

Polymer composites are no longer a specialist material with applications of thermoplastic polymer composites covering a wide range including high volume automotive parts, and sports and protective equipment. The real cutting edge use of thermoplastic composites is in high performance aerospace components, due to composites’ massive potential compared to traditional materials. With their advantages composites already almost make up the majority of components in modern jet fighter design. The uses of thermoplastic composites are being expanded continuously, an example is the unsuccessful Boeing prototype X-32 Joint Strike Fighter [1] whose undercarriage and wing section were proposed to be constructed solely from composite sheets. The complex shape of the nose section and lack of knowledge of the composite behaviour during forming resulted in voids and uneven thickness across the wing section, forcing Boeing to fall back on more traditional materials. This increased the weight of the prototype, which led to the loss of a multi-billion dollar contract. Research in this area and others, hopefully will solve problems such as this. In any case it is expected that growth in the use of thermoplastic polymer composites will continue.

The inherent high viscosity of thermoplastic resin requires high temperature and high pressure processing to achieve full consolidation of the component, making tooling costs and manufacturing costs high. Use of simulations and micro-mechanical models as well as the development of new materials and manufacturing processes have overcome this problem to a certain degree, but the best way to solve the problem is to understand how processes affect the viscosity of the preformed part, so that more accurate models can be made. As a result more efficient manufacturing methods can be created, thus further reducing the gap with thermosetting composites and other materials. Previous work by Shuler and Advani [2] looked at how the transverse viscosity of APC-2 affected its processing characteristics by squeezing samples between heated plates. McGuiness and O’Braidaigh [3] investigated the longitudinal and transverse viscosities of APC-2 using a picture frame test. Both were successful until the results were compared, as they differed by up to a factor of 1000. The testing methods have never been done simultaneously in one paper, this research has changed that and aimed to identify the source of the disparity. Though the key aim for this project is to expand upon the energy model for textile composites, which has been developed at the University of Nottingham. Through implementing the findings from this research it should result in a better understanding of both uniaxial and woven composites materials.

This project also aimed to show a clear relationship in how the viscosity is affected in a uniaxial long fibre thermoplastic composite, as well as corroborate previous work. The material used is Twintex, a thermoplastic composite made up of 60% glass fibres by weight and a polypropylene matrix. Twintex does not come in a preconsolodated uniaxial form but only in a woven form, as a result this meant creating a method to make uniaxial Twintex
samples from the fibre roving which has not been done before. With this done, the squeeze flow plates were then calibrated using a viscous Newtonian fluid, and verified by a rheometer, only then could testing begin. The material was tested by the squeeze flow method first, which varied first the temperature, and then secondly the surface area of the testing plates. Significant redesign of the apparatus was required to be able to vary the surface area in testing. The results were analysed using a derived power law expression based upon work by Shuler and Advani [2]. The picture frame testing was completed after this along similar lines to McGuiness and O’Braidaigh’s [3] work, and analysed using their formulae. Again, redesign of the rig was necessary to reduce edge heating effects in the sample and thus give more accurate results. Unfortunately the small amount of time available and demand upon the Hounsfield testing apparatus meant that an unsatisfactory amount of data for the picture frame testing was collected. There is enough however, to find a link between Shuler’s and O’Braidaigh’s work as they were the base upon which the testing was done, as a result any errors in either previous works will affect this research. This should clear up the differences in the data of the two, and give a clearer idea of how unidirectional thermoplastic polymer composites behave.

Modelling the tests computationally for comparison was also attempted based upon and expanding on the work done by Wheeler & Jones [4] and McEntee & O’Braidaigh [5], however the software available, FLUENT™, was not capable of modelling the requirements as it could not model satisfactorily the closure of two plates and the flow of the sample, it is better adjusted to model fluid flow, a different package POLYFLOW™, would be more appropriate but it was not available to use. This is unfortunate as mesh software has improved significantly since work done by Wheeler & Jones [4] and other work by McEntee & O’Braidaigh [5] was published offering a better potential for modelling such systems.

Starting this research with little knowledge of the subject meant that many previous papers had to be referenced in order to get the experience and knowledge necessary. For example, to get an idea of resin kinematics a paper by L.James Lee [6] was used. The list of background reading can be seen in the further reading section of the references page.

Objectives

• Read previous work on uniaxial polymer composite research to gain knowledge and experience.
• Create a process for making uniaxial Twintex samples.
• Redesign the squeeze flow apparatus so that samples of different surface area can be tested also.
• Redesign the picture frame apparatus so that heaters can be incorporated onto it, to eliminate edge heating effects.
• Calibrate the squeeze flow plates apparatus using a viscous Newtonian fluid, compare the results to data collected using a rheometer.
• Test the sample material using the squeeze flow apparatus to find the transverse viscosities, varying first the surface area of the sample tested, then the temperature of the sample, using Shuler and Advani’s [2] work as a guide.
• Test the sample material using the picture frame apparatus to find the longitudinal and transverse viscosities, varying the temperature, using McGuiness and O’Braidaigh’s [3] work as a guide.
• Analyse the results of the calibration data using the altered Goshawk, et al. [7] formula.
• Analyse the squeeze flow data using the derived power law expression.
• Analyse the picture frame data using the expressions in the McGuiness and O’Braidaigh [3] paper.
• Compare and analyse the data and find any possible relationship between the two testing methods and/or reason for the discrepancies in the past, and also how the viscosity of the sample is affected and can be manipulated in manufacturing.

2. Experimental Set up

2.1 Material Creation

In order for the results from testing to be accurate and reliable, the samples used must be as homogenous as possible. As a result, samples were pre-consolidated for ease of handling during testing as well as ease of storage. To do this Twintex roving was wrapped around a square aluminium plate, 220mm*220mm, three times for the desired sample thickness, with less windings voids appeared easily, with more windings it was difficult to remove the sheets from the template. The template is bigger than the maximum sample size needed, so as to allow for frayed edges, also all edges on the plates must be rounded otherwise the template will rip the vacuum bag during consolidation. A fully wound template can be seen in figure 2.1(1).

![figure 2.1(1) – Aluminium template with a three layer winding of Twintex roving before consolidation](image)

When winding the roving around the plate it is critical to ensure that the fibres do not twist or move too much, and are not wound at an angle as these both cause a slight weave of the sample making it a multidirectional composite and not valid for testing in this research. It was quite a challenge to ensure that the roving did not twist, as when it came off of the cheese it automatically wanted to twist, this caused the winding process to be painstaking, but not too time consuming as it was possible to wind a plate fully whilst another was being consolidated.

The process for consolidating the samples is as follows;
• Set oven to >180ºC to speed up heating, (set at 220ºC)
• Put a thermocouple into the material (connected to a digital thermometer). At least for the first time to get an idea of required time.
• Place pressure plates on top and below the template, and put into a vacuum bag.
• Put in oven and apply maximum vacuum immediately ($P_{\text{max}} = 1$ bar). This causes the vacuum to be applied whilst the material heats up, resulting in the lowest void content possible.

• Remove from oven when the temperature $>180^\circ \text{C}$ in the material.

• Quench in a water bath (whilst still in the vacuum bag).

• Remove sample sheets from plates and trim any frayed edges.

A sample ready to go into the oven in a vacuum bag is shown in figure 2.1(2), and a fully consolidated sample can be seen in figure 2.1(3) and figure 2.1(4).

The sheets of consolidated Twintex created were approximately 1mm thick. It is possible to predict the sheet thickness using a spreadsheet and material values such as the length of Twintex used, the density, etc.
2.2 Squeeze Flow testing

2.2a Calibration

In the squeeze flow testing the two variables looked at were surface area and temperature. However, before this was done a calibration of the apparatus was performed using a viscous Newtonian fluid, i.e. golden syrup. Using the 80mm*80mm plates at room temperature (19.5°C) the golden syrup was squeezed from a thickness of 2.5mm to a thickness of 0.5mm, at a rate of 3mm/min, and the results of the force recorded. Similar work done by Goshawk, et al. [7] meant that an expression for finding the viscosity, which was derived from original work done by Hays [8], could be manipulated to this case and the viscosity calculated using the expression from appendix C.3.

These results were compared with viscosity data for the golden syrup from a parallel plate Rheometer, the rheometer and plates used can be seen in figure 2.2(1) & figure 2.2(2).

With this data any gap between the results can be accounted for in the testing results, giving more accurate results, variation in the viscosity values are due to the difficulty of controlling the temperature so closely.

2.2b Testing

The samples were tested using, as mentioned, two variables. Plates of different surface area were used to vary the length of the testing plates, whilst ensuring a condition of constant surface area during testing, they were mainly used because the differences in the comparison between the previous works was attributed to length effects by McGuiness & O’Braidaigh [3], so by varying the surface area, and as a result the plate length, this can be tested. The second variable, temperature, was chosen as varying temperature is analogous to varying the rate of testing. Also it would confirm whether the micro-mechanical model by Christensen [10] which approximates the bulk viscosity by taking account of the matrix viscosity and the fibre volume fraction, is correct.

In each test the material tested was four sheets thick (approx 4mm to 5mm) so as to ensure that boundary conditions did not effect the whole sample. The key assumptions of testing were taken from Shuler and Advani’s [2] work;

• The material used is incompressible.
• No body force acts on the material.
• The samples are tested at a relatively slow rate of motion.
• There is no flow in the fibre direction.
• The height of the sample is smaller than the width.
• There is a no slip boundary condition where the samples are in contact with the apparatus.
• Testing will be done using a constant rate of displacement.
• There is no flow parallel to the fibre direction.
• There is only the material between the plates is considered.
• The testing is done slowly enough to neglect inertia, and a ‘quasi-steady state’ can be assumed.

The settings used on the Hounsfield apparatus were as follows for all squeeze flow tests:

• Speed of 5mm/min
• Maximum force = 15kN
• 25kN Load cell

Testing involved compressing the sample until the force reached a value of 15kN, the data of force against extension was then taken from the computer. The surface areas at which the samples were tested were, 40mm*40mm, 60mm*60mm, 80mm*80mm, and 115mm*115mm, all done at a temperature of 180ºC. The temperature testing used the 80mm*80mm plates for testing at temperatures of 170ºC, 180ºC, 190ºC, and 200ºC. The data for the 180ºC is the taken from the surface area testing done on the 80mm*80mm plates. Each test was done three times to ensure repeatability. *figures 2.2(3) & 2.2(4)* illustrates the testing method and apparatus. Appendix B.2 shows the design of the testing plates.

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*Figure 2.2(3) – Illustration of squeeze flow testing process. Excess material is squeezed out of the side maintaining a constant surface area during testing. Outer fibres in contact with plate surface experience a no slip boundary condition.*
With the data collected it was analysed using the power law relationship derived from the Shuler & Advani [2] Newtonian relationship (appendix C.1). See appendix C.2 for the full power law derivation.

2.3 Picture Frame testing

The aim of this test was to test at many different temperatures so to be able to verify McGuiness & O’Braidaigh’s [3] work and compare to the squeeze flow data effectively. Unfortunately due to lack of time and the demand upon the Hounsfield apparatus by other projects the testing done was done at 170ºC, but with a variation in thickness to confirm McGuiness & O’Braidaigh’s [3] work. When testing one unidirectional sheet the sample failed during testing, as shown in figure 2.3(1).
So for any valid results to be found two or more sheets had to be used, with the fibres orientated at 90° to the previous sheet, creating better structural rigidity, although also creating a slight weave in the sample. The data gave at least a clear, if slightly inaccurate, idea overall as a result.

The picture frame rig is shown in figure 4.3(2), which has been taken from previous similar work done at the University of Nottingham by P. Harrison, et al. [11], and was modified to incorporate cartridge heaters from RS components [9], the design can be seen in appendix B.1.

![Figure 4.3(2) – picture frame diagram taken from an article by Dr. P. Harrison. Picture frame shear rig. Lpf is the side length measured from the centre of the hinges and the shear angle, θ, is defined as π/2 - 2Φ](image)

With the new heaters added it reduces the edge effects of heating the sample, i.e. the sample is cooler near the clamps as they take longer to heat, but with the heaters the picture frame is heated individually reducing the temperature difference and giving better testing accuracy. Before testing could be done the apparatus modifications had to be verified by loading a sample into the picture frame and putting it into the oven, and subsequently taking images of the set up using a thermal camera. The oven was set to 180°C and the heaters were set to 195°C. Images were taken with the heaters off and one with them on. Figure 2.3(3) is
the image with the heaters turned off, figure 2.3(4) is the image with the heaters on. Figure 2.3(5) shows the method of taking the thermal images.

![Thermal images](image1.png) ![Thermal images](image2.png)

**Figure 2.3(3) – thermal image of the picture frame in the oven with the heaters off**

**Figure 2.3(4) – thermal image of the picture frame in the oven with the heaters turned on**

**Key:** 19°C=purple, 65°C=violet, 112°C=dark orange, 150°C=light orange, 165°C=yellow, 205°C=white.

**Figure 2.3(5) – Thermal camera (foreground) taking images of the picture frame in the oven.**

*Figures 2.3(3) & 2.3(4) show the effectiveness of the picture frame heaters. Without the heaters the sample by and under the clamps is a darker orange than the centre of the sample, i.e. cooler. With the heaters turned on the sample temperature at the edges in this case is greater than the centre, shown by the lighter yellow. So if needs be the sample at the edge can be made to be hotter and not just the same temperature of the sample. For this research the heaters were set to give a uniform sample temperature.*

The tests were done by heating up the picture frame with no sample in the oven with the edge heaters on (oven set to 190°C, heaters set to 175°C). The oven and heaters were set slightly higher to speed heating and neglect losses. Once temperature was reached the frame
was removed and a sample was loaded in then replaced back into the oven with a thermocouple placed at the edge. It was then heated until the sample edge was slightly above 170ºC, which took approx. 8 minutes, as it was assumed that with the heaters the edges would heat up faster than the centre. This assumption was validated by placing a small piece of material at the same height in the oven as the centre of the sample had been and recording the temperature in the material over 8 minutes, after 8 minutes the temperature reached was 173ºC.

Once the sample was heated it was tested by extending the picture frame to a certain point. For the test with 2-ply’s the end point was 50mm of extension. For the 3-ply test the end point was 70mm of extension. For both tests the 1kN load cell was used as the values for force recorded were low and so the readings would be more accurate using this, both tests were done at a speed of 50mm/min. On each test sample white lines were marked perpendicular to the fibre direction to ensure that inter-ply friction was not a major effect, unfortunately these markings were only clear on the 2-ply test, as shown in figure 2.3(6) and figure 2.3(7).

With the data of force against extension from these tests, it was then analysed using the expressions for the longitudinal and transverse viscosities from the McGuiness & O’Braidaigh’s [3] paper as well as other constituent expressions from the P. Harrison, et al. [11] paper.
3. Results & Analysis

3.1 Calibration results

The data in figure 3.1(1) shows that the syrup used in calibration is clearly Newtonian as the curve can be assumed to be straight on the log plot. This is the data which was analysed to find the viscosity for calibration of the squeeze flow apparatus. Below is the data recorded from the Rheometer, figure 3.1(2) shows the syrup viscosity at 22.5ºC, which has an average of about 40Pas. Figure 3.1(3) shows the syrup viscosity at 26ºC, which is on average 25Pas.

Figure 3.1(1) – Data recorded from squeeze flow testing of golden syrup on Hounsfield rig (temp=19.5ºC). Lines show repeatability.

Figure 3.1(2) – Rheometer data of viscosity of golden syrup at 22.5ºC, visc.=40Pas.

Figure 3.1(3) – Rheometer data of viscosity of golden syrup at 26ºC, visc.=25Pas.
3.2 Squeeze Flow results

3.2a Surface area testing

Figure 3.2a(1) – raw data from testing done using square plates of varying length from 40mm to 115mm at 180°C

3.2b Temperature testing

Figure 3.2b(1) – raw data from testing done with the 80mm*80mm plates at varying temperatures, ranging from 170°C to 200°C
The bumps on both figure 3.2a(1) and 3.2b(1) are the points at which the samples re-consolidate, this is because there is inevitably air gaps between the sheets of pre-consolidated Twintex and this must be removed in the primary part of the squeeze flow testing for the subsequent results to be accurate.

3.3 Picture Frame testing

![Graph showing Log Force vs. Extension for 2-ply and 3-ply samples](image)

*Figure 5.3(1) – raw data from picture frame testing done with a thickness of 2-plys (approx. 1.875mm) and 3-plys (approx 2.725mm) at a temperature of 170°C*

The difference in the curves for the picture frame testing is due to the 3-ply sample being extended by 20mm more than the 2-ply. The height difference is due to the different thickness resulting in the 3-ply being stronger as expected.

3.4 Calibration Analysis

Having collected the raw data from the Hounsfield apparatus it was analysed using an expression taken from work done by Goshawk, et.al [7];

\[ h = \left( \frac{1}{h_0^2} + \frac{\pi^6 F_t}{384\eta L^2} \sum_{m=1}^{\infty} \sum_{n=1}^{\infty} \left( \frac{1}{m^2 n^2 (m^2 + n^2)} \right) \right)^{-1/2} \]  

(1)

This formula has been derived from previous early work by Hays [8] of squeezing of films between plates. Goshawk’s expression had to be further manipulated as it does not take into account that force is also a function of time in this case. Testing was done with a constant closure rate of 3mm/min for calibration, but Goshawks work was done with a constant force and so the subsequent altered expression derived was;
\[
\frac{dh}{dt} = -\frac{1}{2} \left( \frac{1}{h_0^2} + \frac{\pi^6 F_t}{384\eta L^3} \right)^{\frac{3}{2}} \cdot \frac{\pi^6 \frac{d(F_t)}{dt}}{384\eta L^4} / a \tag{2}
\]

where,

\[
a = \sum_{m=1, n=1}^{\infty} \left( \frac{1}{m^2 n^2 (m^2 + n^2)} \right) \tag{3}
\]

See appendix C.3 for the full derivation

The final formula was derived using a Matlab program by Dr. P. Harrison to find the force vs. time polynomial, checking this in a Microsoft Excel© spreadsheet, and then differentiating to get the final expression. Using an iterative method the values of the viscosity, \( \eta \), at certain times can be found, which can be seen below in table 3.4(1) & figure 3.4(2).

<table>
<thead>
<tr>
<th>T(s)</th>
<th>Viscosity(Pas)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>0.28</td>
</tr>
<tr>
<td>8</td>
<td>1.4</td>
</tr>
<tr>
<td>12</td>
<td>6</td>
</tr>
<tr>
<td>16</td>
<td>9</td>
</tr>
<tr>
<td>20</td>
<td>20</td>
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<tr>
<td>24</td>
<td>35</td>
</tr>
<tr>
<td>28</td>
<td>54</td>
</tr>
<tr>
<td>32</td>
<td>110</td>
</tr>
<tr>
<td>36</td>
<td>258</td>
</tr>
<tr>
<td>40</td>
<td>420</td>
</tr>
</tbody>
</table>

Table 3.4(1) – Calculated values of viscosity at periodic times during testing

\[\text{Figure 3.4(2) – Plot of calculated viscosity vs. time using the derived expression}\]
The shape of the curve is similar to the shape of the data curve taken directly from the Hounsfield apparatus shown previously, it differs however, to the curves taken from the Rheometer. This indicates a problem in calculation as the curve should be relatively flat, i.e. the characteristic curve for a Newtonian fluid. The original formula has been verified using Goshawk’s data in several ways, but the implementation of the expression refused to work. The verification of the formulas can be seen in appendix D. This does cause some doubt in the results and conclusions drawn from them, however, the raw data is available for verification in the appendices, and the previous use of the same apparatus shows that it is likely to give accurate results.

3.5 Squeeze Flow Analysis

Thanks to previous work done by Shuler and Advani [2] the analysis of the data from the squeeze flow testing was simple as it followed similar lines. Two methods for analysis were considered, a power law relationship and a Carreau-Yasuda relationship as the material can be assumed to be a Non-Newtonian fluid. The power law relationship was derived from the Newtonian expressions, to check the power law derivation you simply put a value of n=1 into the formulae and the Newtonian equivalent is produced. Both the Newtonian and power law derivations can be seen in appendices C.1 & C.2 respectively. There wasn’t enough time to fully develop a Carreau-Yasuda model also, however, this is not of too much consequence as the Carreau-Yasuda model is better to fit for lower rates, but behaves like a power law at higher rates. Due to the consolidation bump at the low rates the Carreau-Yasuda model analysis would have been dubious as the data before the re-consolidation of the samples cannot be seen to be accurate.

The key formula for analysing all the squeeze flow results is as follows;

\[
F = 2w \eta_0 \left( \frac{- \frac{dh}{dt}}{\left( \frac{2n}{1+2n} \right)} \right)^n \left[ \frac{3L_0^{n+2} - nL^{n+2}}{(n+1)(n+2)} \right] \tag{4}
\]

This is derived using the Navier-Stokes formula, with a power law model instead of a Newtonian model, as well as other fluid flow expressions. (See the nomenclature section at the beginning for symbol definitions.)

Through using this formula in a Microsoft Excel© spreadsheet all the data could be analysed, and values for the viscosity of the Twintex could be approximated, by fitting a power law curve to the data curves to find values for \( \eta_0 \) & \( n \). As illustrated in the figures over the page. The curve is fitted only to the data after the bump in the curves, giving the most accurate fitting.
3.5a Surface area

**Figure 3.5a(1)** – test data for 40mm*40mm plates with a power law fit of $\text{Visc}=9000\text{Pas}$ $n=0.5$

*figure 3.5a(1)* illustrates how the data was analysed, the red curve is the power law model, it was fitted by varying the values of $\eta_0$ & $n$ until a satisfactory fit was accomplished, this was done for all the surface area data and can be seen in *appendix A.2*. The fitted power law curves for all the surface area testing can be seen below in *figure 3.5a(2).*

**Figure 3.5a(2)** – comparison of power law fits for different testing surface areas, 115mm*115mm on top and 40mm*40mm on the bottom
The viscosities found for the surface area data are shown below in *table 3.5a(3)*.

<table>
<thead>
<tr>
<th>Plate length</th>
<th>Viscosity((\gamma \text{s}^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>40mm</td>
<td>9000 Pas</td>
</tr>
<tr>
<td>60mm</td>
<td>7500 Pas</td>
</tr>
<tr>
<td>80mm</td>
<td>6800 Pas</td>
</tr>
<tr>
<td>115mm</td>
<td>5250 Pas</td>
</tr>
</tbody>
</table>

*Table 3.5a(3) – Viscosity values for different plate surface areas, found using a fitted power law model.*

### 3.5b Temperature

![Graph showing temperature data](image)

*Figure 3.5b(1) – data for 200°C test with a power law fit of Visc=5500Pas n=0.5*

The temperature data was analysed in the same way as the surface area data, as shown by *figure 3.5b(1)* above, *figure 3.5b(2)* over the page compares the fitted power law curves for all the temperature testing data. The individual fitting graphs can be seen in *appendix A.3*. 
The comparison between the transverse viscosity and the predicted transverse viscosity from Christensen’s [10] micro-mechanical model is shown below in table 3.5b(3).

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Viscosity(γ s⁻¹)</th>
<th>Christensen model</th>
</tr>
</thead>
<tbody>
<tr>
<td>170°C</td>
<td>7500 Pas</td>
<td>7000 Pas</td>
</tr>
<tr>
<td>180°C</td>
<td>6800 Pas</td>
<td>4375 Pas</td>
</tr>
<tr>
<td>190°C</td>
<td>6500 Pas</td>
<td>3500 Pas</td>
</tr>
<tr>
<td>200°C</td>
<td>5500 Pas</td>
<td>2450 Pas</td>
</tr>
</tbody>
</table>

All the data has been plotted logarithmically so that the relationships can be shown more clearly graphically. The material constants of viscosity and the power, n, affect the curves as you would expect, with changing the viscosity moving the curve up and down, and changing the value of n varies the curve gradient.

Using the results the Reynolds number was also calculated, using:

\[ Re = \frac{\rho [dh(t)/dt] h(t)}{\eta} \]  \hspace{1cm} (5)

This was to confirm that the assumed boundary condition for testing was correct, and for this to be true the Reynolds number has to be <1. The results can be seen below in Table 3.5b(4);

<table>
<thead>
<tr>
<th>Plate size</th>
<th>Viscosity</th>
<th>Mean R. No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>40*40</td>
<td>9000</td>
<td>4.00556E-11</td>
</tr>
<tr>
<td>60*60</td>
<td>7500</td>
<td>4.98236E-11</td>
</tr>
<tr>
<td>80*80</td>
<td>5850</td>
<td>5.5348E-11</td>
</tr>
<tr>
<td>115*115</td>
<td>5250</td>
<td>8.88412E-11</td>
</tr>
</tbody>
</table>

Table 3.5b(4) – Reynolds number for squeeze flow tests.

---

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As you can see the Reynolds numbers are all well below unity and so the boundary no
flow condition is correct.

3.6 Picture Frame Analysis

The picture frame experiments were done in the same way as those done by
McGuiness and O’Braidaigh [3] with few modifications, as a result the data can be analysed
using the same method as O’Braidaigh and McGuiness devised. The formulae are shown
below;

\[
\left( \frac{Fd}{\gamma^2 L^2 t (1 + \sin^2 \gamma)} \right) \left( \sqrt{\frac{1 + 3\sin^2 \gamma}{\cos^2 \gamma}} \right)^{1-n} = \left( m_i + m_i \left( \frac{2\sin^2 \gamma}{1 + \sin^2 \gamma} \right) \right)
\]

(6)

where,

\[
\gamma = \frac{4d^2}{(2L^2 - 2\sqrt{2}Ld - d^2)}
\]

(7)

and,

\[
\gamma = \frac{\pi}{2} - 2 \arccos \left[ \frac{1}{\sqrt{2}} + \frac{d}{2L} \right]
\]

(8)

By plotting a graph using this expression both viscosities can be found. As shown
below:

This gives results for both the transverse viscosity, \( m_T \), given by the gradient, \( \gamma \), and the
longitudinal viscosity, \( m_L \), given by the y-intercept. This allows for comparison with the
previous results as well as a clear picture of the viscosity in the other major plain. See the
results over the page;
Figure 3.6(1) shows the manipulated data curve for the 2-ply results with a trend line fitted to find the transverse and longitudinal viscosities. The trend line is fitted only to the first half of the curve, as the second half has a negative gradient which indicates that the sample has begun to disintegrate and so only the data with a positive gradient is accurate for analysis. The trend line shows a transverse viscosity of 6MPas, and a longitudinal viscosity of 3MPas.

Figure 3.6(1) – analysis plot of 2-ply thick test picture frame data. \( m_L = 3\text{MPas}, m_T = 6\text{MPas} \)

The data for the 3-ply test, shown in figure 3.6(2), can be seen to be inaccurate and unreliable for analysis. This can be seen from the negative gradient, which indicates that the sample began disintegrating almost immediately. This could be due to in a small part inter-

Figure 3.6(2) – analysis plot of 3-ply thick test picture frame data. \( m_L = 9\text{MPas}, m_T = -10\text{MPas} \)
ply friction, but more likely due to misalignment of the sample in the picture frame, and so the fibres being extended interfering in the testing of the transverse and longitudinal viscosities.

### 3.7 Comparison Analysis

For accurate result comparison the tests must be done at the same shear rate. To calculate the shear rate in the picture frame tests, for the longitudinal viscosity, $\eta_L$, it’s quite simple as it is the rate of angular displacement, $\dot{\theta}$ or $\dot{\gamma}$, as it is in the equation. The transverse shear rate is given by $2\dot{\theta}\sin\theta$ and so can be quite easily calculated.

To calculate the shear rate for the squeeze flow tests it is a more complex process, first you need to calculate $2D_{ij}$, the rate of strain tensor, as the shear rate is not uniform between the plates:

$$2D_{ij} = \left(\frac{dV_i}{dx_j} + \frac{dV_j}{dx_i}\right)$$

Having done that $II_{2D}$ needs to be found, as $\dot{\gamma} = \sqrt{-II_{2D}}$. This will be a function of time and position and so quite complex. It is easier to get a general idea of the maximum shear rate on the plates by finding the strain rate at the edge of the plates. This is done by using the power law derivation to find $\frac{dV_i}{dy}$ at $x=l$ and $y=h/2$ over time, as $\frac{dV_i}{dy} = \dot{\gamma}$. The formula is:

$$\frac{dV_i}{dy} = y^n \left[-x\frac{-2n}{(1+2n)}\left(\frac{h}{2}\right)^{(1+2n)}\right]^n$$

Unfortunately due to the extremely tight testing schedule it was not possible to calculate the shear rates before the picture frame tests and so the testing speed could not be accurately set, and thus the speed of 50mm/min was picked by referencing the previous work. The results of the shear rate comparison are below in Table 3.7(1);

<table>
<thead>
<tr>
<th>Test</th>
<th>Shear rate</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Squeeze flow</strong></td>
<td></td>
</tr>
<tr>
<td>40*40</td>
<td>1.48E-19</td>
</tr>
<tr>
<td>60*60</td>
<td>9.32E-20</td>
</tr>
<tr>
<td>80*80</td>
<td>3.33E-19</td>
</tr>
<tr>
<td>115*115</td>
<td>1.11E-18</td>
</tr>
<tr>
<td><strong>Picture frame</strong></td>
<td></td>
</tr>
<tr>
<td>Trans. shear</td>
<td>1.06E-04</td>
</tr>
<tr>
<td>Long. shear</td>
<td>9.47E-05</td>
</tr>
</tbody>
</table>

Table 3.7(1) – Shear rates for all tests performed.

As it can be clearly seen the shear rates are quite different by a factor of about $1 \times 10^{13}$ and so any comparison of the results will be inaccurate, but the results are still valid and can be compared.
4. Discussion

As it can be easily seen from the results from the squeeze flow testing that temperature does affect the viscosity in the expected way, with the higher temperature giving the lowest viscosity and vice versa, due to the weaker chemical bonds at higher temperatures. The Twintex also behaves in an expected way, exhibiting shear thinning behaviour, this is due to the polypropylene matrix being a shear thinning polymer. This corroborates Christensen’s [10] micro-mechanical model for approximating the bulk viscosity using the matrix viscosity and the fibre volume fraction. As the fibre volume fraction was constant the effect on varying the temperature was to vary the matrix viscosity and so the overall viscosity, this can be seen in the results. Christensen’s [10] results are slightly different but this can be attributed to variables that are unaccounted for in the micro-mechanical model and slight errors in testing.

The unexpected results are those from the surface area squeeze flow testing, its was predicted that the variation of temperature would give a greater variation of the viscosity than the variation of the surface area, the results show completely the contrary, with the larger surface areas having significantly lower viscosities than the smaller surface areas. This goes against the assumption made by McGuiness & O’Braidaigh [3], who assumed that the larger transverse viscosity from the picture frame testing was due to the sample having a longer length. With all the testing data and results showing a very good level of repeatability the relationships can be seen to be valid. However, the accuracy is dependant on the sample quality and homogenisation, as well as errors in testing. The viscosity values are within expected ranges, and the power law relationship fits satisfactorily. The reasoning for this relationship must be that with the smaller surface area the sample experiences a significantly higher pressure across it, and so with the higher pressure the polypropylene matrix compresses only slightly then much of the matrix is squeezed out first and so the fibre volume changes and the fibres take a higher proportion of the load. With the reduction in the matrix volume the friction between fibres increases as they are closer and so the viscosity of the material increases. This indicates that the speed of testing/manufacture is critical in affecting shear thinning thermoplastic polymer composites material behaviour. However, this must be checked by taking readings with each set of squeeze flow plates without a sample present, and removing this effect from the testing data to see accurately the effect on the sample, unfortunately there wasn’t sufficient time to do this.

The limited results of the picture frame testing means that the relationships taken from it cannot be seen to be reliable, but it does show some interesting differences. The 2-ply test did not have significant interply friction, and it can be seen to have been properly aligned, this is shown by testing results curve shape being very similar to those from McGuiness & O’Braidaigh’s [3] work, and the viscosities are very similar also. It can be seen to confirm their work and results, as well as build upon it with the apparatus modifications to prevent edge heating effects.

The disparity when comparing the results is the same as that found by McGuiness & O’Braidaigh [3] when comparing their results with Shuler & Advani’s [2], the difference between the transverse viscosities is again a factor of 1000, with the transverse viscosity for the squeeze flow being approximately 6 KPas, and the transverse viscosity from the picture frame testing being 6 MPas. The testing methods used would not account for this difference, even with the different shear rates, this indicate a problem in the methods for analysis.

Further work should be done with the picture frame apparatus to corroborate or discount the data found in this project. Also, investigating thermosets is a natural progression.
5. Conclusions

From this research clear conclusions can be taken;
• There is an obvious problem in the previous work done.
• Edge and length effects cannot be the cause of the disparity between the results.
• The testing methods are valid, this indicates that the disparity is in the methods for analysis.
• The agreement of the micro-mechanical model by Christensen [10] with the squeeze flow data suggests that the problem is with the analysis proposed by McGuiness & O’Bradaigh [3], this needs further investigation.
• For current micro-mechanical models, the squeeze flow method is the most reliable to predict the needed material constants for manufacture/simulation/modelling.
• There is clearly a potential decrease in viscosity with an increase in length of the testing/manufacturing apparatus.
• Increasing the temperature does decrease the viscosity as expected.

There is clearly much more work to do in this area, this project has shed light on many questions but has raised many more, which must be solved by further research.

6. References


Further Reading


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• Rogers, T.G. ‘Shear Characterisation and Inelastic Torsion of Fibre-Reinforced Materials’. Inelastic deformation of Composite Materials edited by D.J. Dvorak 1990.
Appendices

Appendix A - Raw Data

A.1 Calibration data

(See CD for testing results, individual calculations, and rheometer data)

A.2 Squeeze flow data

(See CD for testing data and power law calculations & analysis)

A.3 Picture Frame data

(See CD for testing data and analysis)

Appendix B - Apparatus design

See over the page
Appendix C - Derivations

C.1 - Newtonian Derivation – Re-derived by Dr. P. Harrison from Schuler & Advani Paper [2]

\[ \nabla V = \frac{dV}{dy} + \frac{dV_y}{dy} = 0 \]

Find \( V_y \),
\( V_y = 0 \) when \( y = 0 \)
\[ V_y = -\frac{1}{2} \frac{dh}{dt} \quad \text{when} \quad y = \frac{h}{2} \]
\[ V_y = \frac{1}{2} \frac{dh}{dt} \quad \text{when} \quad y = -\frac{h}{2} \]

\( V_y = -\frac{y}{h} \frac{dh}{dt} \) satisfies above conditions.

\[ \frac{dV_x}{dy} = -\frac{dV_y}{dy} = -\frac{1}{h} \frac{dh}{dt} \]

\[ V_x = \int -\frac{1}{h} \frac{dh}{dt} \, dx \]

\[ V_x = \left[ -\frac{x}{h} \frac{dh}{dt} + C \right] \]

when \( x = 0 \) \( V_x = 0 \) thus \( C = 0 \) (assumes (0,0) is at the centre)

\[ V_x = -\frac{x}{h} \frac{dh}{dt} \]

\[ Q = \int_{-h/2}^{h/2} V_x \, dy = \left[ -\frac{x}{h} \frac{dh}{dt} y + C \right]_{-h/2}^{h/2} \]

find \( Q = -x \frac{dh(t)}{dt} \)

\[ \frac{dp}{dx} = \eta_0 \frac{d^2V_x}{dy^2} \]

\[ \frac{dV_x}{dy} = \frac{1}{\eta_0} \frac{dp}{dx} \cdot y + C \]

when \( y = 0 \) \( dV_x = 0 \), thus \( C = 0 \)

\[ \frac{dV_x}{dy} = \frac{1}{\eta_0} \frac{dp}{dx} \cdot y \]

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\[ V_x = \frac{1}{\eta_0} \frac{dp}{dx} \left( \frac{y^2}{2} \right) + D \]

when \( y = \frac{h}{2}, V_x = 0 \)

\[ D = -\frac{1}{2\eta_0} \frac{dp}{dx} \left( \frac{h}{2} \right)^2 \]

\[ V_x = \frac{1}{2\eta_0} \frac{dp}{dx} \left( y^2 - \frac{h^2}{4} \right) \]

\[ \mu = \eta_0 \]

\[ \int_0^{h/2} V_x \, dy = \int_0^{h/2} \frac{1}{2\mu} \frac{dp}{dx} \left( y^2 - \frac{h^2}{4} \right) \, dy = \left[ \frac{1}{6\mu} \frac{dp}{dx} y^3 - \frac{1}{8\mu} \frac{dp}{dx} h^2 y \right]_0^{h/2} \]

\[ = \frac{1}{48\mu} \frac{dp}{dx} h^3 - \frac{3}{48\mu} \frac{dp}{dx} h^3 \]

\[ = -\frac{1}{24\mu} \frac{dp}{dx} h^3 = \frac{Q}{2} = -\frac{x}{2} \frac{dh}{dt} \]

\[ = \frac{1}{12\mu} \frac{dp}{dx} h^3 = -x \frac{dh}{dt} \]

\[ \frac{dp}{dx} = 12\mu \frac{x}{h^3} \frac{dh}{dt} \]

\[ \therefore P = 6 \frac{x^2}{h^3} \mu \frac{dh}{dt} + c \]

when \( x = L \) \( P = P_0 \) \( C = \frac{6L^2}{h^3} \mu \frac{dh}{dt} \)

\[ P = \frac{6\mu}{h^3} \frac{dh}{dt} (x^2 - L^2) \]
\[ F = 2 \int_0^L P \, dx = \left[ \frac{6\mu}{h^3} \frac{dh}{dt} \frac{x^3}{3} - \frac{6\mu}{h^3} \frac{dh}{dt} L^2 x \right]_0^L \]

\[ = 2w \left[ -\frac{4\mu}{h^3} \frac{dh}{dt} L^3 \right] \]

\[ F = -\frac{8w\mu}{h^3} \frac{dh}{dt} L^3 \]
C.2 - Power Law Derivation

If a value of \( n=1 \) is put into any stage of the derivation below the Newtonian equivalent will be given, this can be checked against the Newtonian derivation in appendix 10.3a.

\[ \tau = A(\dot{\gamma}) \]

but,

\[ A(\dot{\gamma}) = \eta_o \dot{\gamma}^{n-1} \]

\[ \tau = (\eta_o \dot{\gamma}^{n-1}) \dot{\gamma} \]

\[ \tau = \eta_o (\dot{\gamma})^n \]

\[
\frac{dp}{dx} = \frac{dT_{xy}}{dy}
\]

\[
\therefore \frac{dp}{dx} = \frac{d\eta_o(\dot{\gamma})^n}{dy}
\]

\[
\int \frac{1}{\eta_o} \frac{dp}{dx} dy = \int \frac{d(\dot{\gamma})^n}{dy} dy
\]

\[
\frac{1}{\eta_o} \frac{dp}{dx} \cdot y + c = \dot{\gamma}^n
\]

when \( y = 0 \), \( c = 0 \)

and \( \dot{\gamma} = \frac{dV_x}{dy} \)

\[
\frac{dV_x}{dy} = \frac{1}{\eta_o} \frac{dp}{dx} \cdot y
\]

\[
\int \frac{dV_x}{dy} \cdot dy = \int \frac{1}{\eta_o} \frac{dp}{dx} \cdot y dy
\]

\[
V_x = \left( \frac{1}{\eta_o} \frac{dp}{dx} \right)^\frac{1}{n} \int y^n dy
\]

\[
V_x = \left( \frac{1}{\eta_o} \frac{dp}{dx} \right)^\frac{1}{n} \cdot \frac{n}{(1+n)} \cdot y^{(n+1)} + D
\]

when \( y = \frac{h}{2}, V_x = 0 \)
\[ D = \left( \frac{1}{\eta_0} \cdot \frac{dp}{dx} \right)^{\frac{1}{n}} \cdot \frac{n}{(1+n)} \left( \frac{h}{2} \right)^{(1+n)} \]

\[ V_x = \left( \frac{1}{\eta_0} \cdot \frac{dp}{dx} \right)^{\frac{1}{n}} \cdot \frac{n}{(1+n)} \left[ y - \left( \frac{h}{2} \right)^{(1+n)} \right] \]

\[ \int_{0}^{h} V_x dy = \int_{0}^{h} \left( \frac{1}{\eta_0} \cdot \frac{dp}{dx} \right)^{\frac{1}{n}} \cdot \frac{n}{(1+n)} \left( \frac{h}{2} \right)^{(1+n)} y - \left( \frac{1}{\eta_0} \cdot \frac{dp}{dx} \right)^{\frac{1}{n}} \cdot \frac{n}{(1+n)} \left( \frac{h}{2} \right)^{(1+n)} \]

\[ = \left( \frac{1}{\eta_0} \cdot \frac{dp}{dx} \right)^{\frac{1}{n}} \cdot \frac{n^2}{(1+n)(1+2n)} \left( \frac{h}{2} \right)^{(1+2n)} - \left( \frac{1}{\eta_0} \cdot \frac{dp}{dx} \right)^{\frac{1}{n}} \cdot \frac{n}{(1+n)} \left( \frac{h}{2} \right)^{(1+n)} \]

as \( Q = \int_{-h/2}^{h/2} dy = -x \cdot \frac{dh(t)}{dt} \)

then,

\[ \frac{dp}{dx} = \left[ \frac{-x}{2 \left[ \frac{n^2}{(1+n)(1+2n)} - \frac{n}{(1+n)} \left( \frac{h}{2} \right)^{(1+2n)} \right]} \frac{dh}{dt} \right] \eta_0 \]

\[ = \int \frac{dp}{dx} dx = \int \left[ \frac{-x}{2 \left[ \frac{-n}{(1+2n)} \left( \frac{h}{2} \right)^{(1+2n)} \right]} \frac{dh}{dt} \right] \eta_0 dx \]

\[ a = \left( \frac{dh}{dt} \right) \left( \frac{-2n}{(1+2n)} \left( \frac{h}{2} \right)^{(1+2n)} \right) \]

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\[
\int \frac{dp}{dx} dx = \int (-ax)^n \eta_0 dx
\]

\[
= \eta_0 (-a)^n \int x^n dx
\]

\[
= \eta_0 (-a)^n \cdot \frac{x^{n+1}}{n+1} + K
\]

when \( x = 1 \), \( P = P_0 \)
\[
\therefore K = -\eta_0 (-a)^n \cdot \frac{L^{n+1}}{n+1}
\]

\[
P = \eta_0 (-a)^n \cdot \frac{(x-L)^{n+1}}{(n+1)}
\]

\[
F = 2w \int_0^L P dx = 2w \eta_0 (-a)^n \int_0^L \frac{x^{n+1}}{(n+1)} - \frac{L^{n+1}}{(n+1)} dx
\]

\[
= 2w \eta_0 (-a)^n \left[ \frac{L^{n+2}}{(n+1)(n+2)} - \frac{L^{n+2}}{(n+1)} \right]
\]

\[
\therefore F = 2w \eta_0 \left( \frac{\frac{dh}{dt}}{(1+2n) \left( \frac{h}{2} \right)^n} \right)^n \left[ \frac{3L^{n+2} - nL^{n+2}}{(n+1)(n+2)} \right]
\]

Where in this case \( \eta_0 \) is \( m_T \) the transverse viscosity.

**C.3 - Calibration Derivation**

Original formula taken from Goshawk paper [7].

\[
h = \left( \frac{1}{h_0^2} + \frac{\pi^6 Ft}{384 \eta L^4} / \sum_{m=1}^{\infty} \sum_{n=1}^{\infty} \left( \frac{1}{m^2 n^2 (m^2 + n^2)^2} \right) \right)^{-1/2}
\]

\[
a = \sum_{m=1}^{\infty} \sum_{n=1}^{\infty} \left( \frac{1}{m^2 n^2 (m^2 + n^2)^2} \right)
\]

\[
\frac{dh}{dt} = -\frac{1}{2} \left( \frac{1}{h_0^2} + \frac{\pi^6 Ft}{384 \eta L^4} / a \right)^{-3/2} \cdot \frac{\pi^6 \frac{d(Ft)}{dt}}{384 \eta L^4} / a
\]
Appendix D - Calibration Verification

As the viscosity calculated for the calibration data was giving out inaccurate data and not corresponding to the expected values, such as the data taken from the rheometer, the original expression had to be checked through.

First to ensure that the original equation taken from Goshawk, et al. [7] was true, the data was taken from their paper was re-plotted to ensure that a similar curve was found and that the data didn’t contain erroneous results which would throw the calculations. The curve can be seen in figure D(1).

The method was then checked by trying to find similar values of the viscosity which found they in their research, by putting in the constraints and data that they recorded, into the original equation (below).

\[
F = -1.0457 \times 10^{-11} t^{10} + 2.3162 \times 10^{-9} t^9 - 2.1663 \times 10^{-7} t^8 + 1.1177 \times 10^{-5} t^7 - 3.4856 \times 10^{-3} t^6 \\
+ 6.7752 \times 10^{-3} t^5 - 0.081572 t^4 + 0.58612 t^3 - 2.2916 t^2 + 4.5909 t + 3.5709
\]

\[
\frac{d(Ft)}{dt} = -1.15027 \times 10^{-10} t^{10} + 2.3162 \times 10^{-8} t^9 - 1.94967 \times 10^{-6} t^8 + 8.9416 \times 10^{-5} t^7 - 2.4399 \times 10^{-3} t^6 \\
+ 0.0406512 t^5 - 0.40786 t^4 + 2.34448 t^3 - 6.8748 t^2 + 9.1818 t + 3.5709
\]
The average viscosity was found to be 70 Pas for a constant force of 200g. The viscosity calculated can be seen in figure D (2).

As it can be clearly seen the average viscosity found was approximately 35 Pas, i.e. half the expected. This indicates an error in calculation or the values used, and so does not validate or invalidate the original expressions. However, all values used were those taken from the paper, and so compounded values errors are the most likely cause.

As a result a further step was taken, the original expression was differentiated, still with force as a constant and the rate of closure was calculated. The differentiated expression is below:

\[
h = \left( \frac{1}{h_0^2} + \frac{\pi^6 F t}{384\eta L^4} \right)^{-\frac{3}{2}} \sum_{m=1}^{\infty} \sum_{n=1}^{\infty} \left( \frac{1}{m^2 n^2 (m^2 + n^2)} \right)
\]

\[
\frac{dh}{dt} = -\left( \frac{1}{h_0^2} + \frac{\pi^6 F t}{384\eta L^4} \right)^{-\frac{3}{2}} \left( \frac{\pi^6 F}{384\eta L^4} \right) a
\]

\[
a = \sum_{m=1}^{\infty} \sum_{n=odd}^{\infty} \left( \frac{1}{m^2 n^2 (m^2 + n^2)} \right)
\]

At the same time a tenth order polynomial was fitted to the data curve from Goshawk, et al.’s work and this was differentiated to give the actual closure rate, and the two curves were compared. The two curves are shown in figure D(3).
The closure rate comparison shows that the original equation is valid, the reason that the Goshawk rate line moves above the x-axis, which it shouldn’t as the closure rate is always negative, is that the fitted polynomial oscillates at later data values, this is unfortunate, but when taken into account the lines can be assumed to be analogous.

With the original expression validated, the manipulated expression for this case was checked again. The polynomial for the varying force was checked against the plotted data to ensure that it was correct, and that it was also correctly implemented in the differentiation. The polynomial comparison is shown in figure D(4) below.

---

**Figure D(3)** – Comparison between calculated rate for Goshawk data, and actual rate.

**Figure D(4)** – Comparison between of the fitted polynomial for force against time to the recorded data.
As figure D(4) shows the curves are almost indistinguishable from one another, and so the polynomial is a very good fit. The derivation of the final expression (appendix C.3) was also checked and checked again and is correct.

Thus with the whole expression shown to be valid, it is a curiosity that it does not work when implemented, this could be down to Microsoft Excel© having problems implementing it, but many different methods of implementation were attempted, all without success. The ideal solution would be to formulate an expression from first principles, unfortunately there was not sufficient time to do this.
Appendix E - Work plan

<table>
<thead>
<tr>
<th>Task</th>
<th>Week</th>
</tr>
</thead>
<tbody>
<tr>
<td>Literature review</td>
<td>1-5</td>
</tr>
<tr>
<td>Testing method discussion</td>
<td>6-8</td>
</tr>
<tr>
<td>Apparatus redesign</td>
<td>9-11</td>
</tr>
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<td>Sample method creation</td>
<td>12-16</td>
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<tr>
<td>Sample Making</td>
<td></td>
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<tr>
<td>Derivations</td>
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<td>Squeeze flow testing</td>
<td>21-24</td>
</tr>
<tr>
<td>Picture Frame testing</td>
<td>25-27</td>
</tr>
<tr>
<td>Analysis of results</td>
<td>28-30</td>
</tr>
<tr>
<td>Write up</td>
<td>31-32</td>
</tr>
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