



## The Characterisation of Casting Wax Rheology

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### Introduction

Investment casting waxes are injected or extruded into tooling cavities to form disposable patterns and related assemblies for subsequent processing in the precision investment casting (PIC) process. The flow characteristics, or rheology, of these waxy materials play a crucial part in determining dimensional and surface quality of the injected part.

Traditionally, wax flow has been characterised using a combination of simple viscosity measurement methods and injection trials. Such techniques do provide useful information but can be cumbersome and time consuming to run and tend to generate point data that typically fails to cover the full spectrum of temperatures and shear rates that waxes are subject to in the PIC process.

Today, the wax industry has access to powerful computer controlled rheometers that can quickly and accurately characterise the rheology of PIC waxes.

This paper examines the pros and cons of the traditional viscosity measurement methods mentioned above and goes on to demonstrate why the author believes a modern computer driven rheometer is the best option for the characterisation of wax rheology. Data is presented illustrating the many uses of such an instrument, including characterising the effect of filler morphology on wax flow, wax rheology at very high and low temperatures and the effect of shear rate on wax behaviour.

### Viscosity and Rheology

Before turning to the various methods used to characterise the flow properties of casting waxes, it is worth spending a little time to explain the differences between “viscosity” and “rheology”.

Viscosity is a measure of a liquid’s resistance to flow, i.e. how “thick” or “thin” the fluid is. Isaac Newton, the noted British physicist, derived the first mathematical definition of viscosity in the 1600’s:

$$\text{Shear stress} = \text{viscosity} \times \text{shear rate}$$

Simply put, the amount of deformation or flow exhibited by a fluid is the product of its viscosity and the shear rate or deforming force applied to it. For a given shear rate, a fluid with a low viscosity will flow more readily than a high viscosity liquid. It also follows that knowledge of the shear rate(s) involved is essential when measuring and reporting viscosity data.

Instruments that are limited to the measurement of viscous flow of liquids are described as “viscometers”. Much of the mathematics behind the measurement of viscosity assumes that the liquids under study behave in a linear, Newtonian fashion; the viscosity of a liquid is independent of shear rate, for instance.

In the real world, including various investment casting processes, the materials used exhibit more complex behaviours. Filled casting waxes, for instance, are non-Newtonian fluids. They respond to shearing forces with a mixture of elastic, viscous and, possibly, time related behaviours.

Rheology is the study of how these complex, real world materials react to stress and a “rheometer” is a device capable of characterising these visco-elastic properties.

A very thorough treatment of rheological theory can be found in *Reference 1*.

### **Wax Rheology is Important**

Why are the flow properties of casting waxes so important? Typically, waxes are supplied to the end user in solid form. Obviously, to be of any use at all in the PIC process, they must be melted, or at least softened, and forced into tooling cavities to produce the disposable patterns we are all familiar with. After the solid parts have been assembled and shelled, the pattern and sprue wax components must be eliminated from the shell before further processing steps can occur – the waxes are again liquefied (by autoclave or flash firing unit) and gravity is used to drain the molten wax from the shell cavities.

In both of these crucial steps, the flow characteristics of the waxes involved are vital. During pattern manufacture, the wax blend must flow into the tool cavity as efficiently as possible, yielding sound, dimensionally accurate, defect free parts requiring minimal hand finishing. Waxes that are too viscous can crack delicate ceramic cores, cause cold shuts, form flow and knit lines, etc. On the other hand, blends that are too fluid can deposit fillers in process equipment and may splash inside the tooling during injection, entraining air.

Dewax behaviour is also important. A viscous sprue wax can retard shell evacuation, leading, perhaps, to shell cracking. A low viscosity pattern wax may, as mentioned above, deposit filler leading to potential burn out issues.

At the very least, measuring wax viscosity will allow the manufacturer / user to make an assessment of lot-to-lot consistency. At best, rheology data can be used to tailor a blend to meet specific process requirements.

So, having established it's a good thing to characterise a wax's rheology, how do we go about it? Ideally, we require an instrument that facilitates rheological measurement across the temperature range that PIC waxes are exposed to during processing. In the next section, we look at a variety of techniques and instruments that are used to characterise casting wax viscosity / rheology in the industry today.

### **Measurement Techniques**

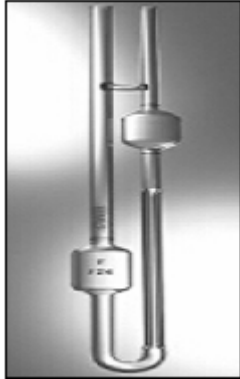
#### ***U-tube Viscometer***

A simple, relatively low cost method for measuring the kinematic viscosity of unfilled waxes, reclaimed waxes and waxy raw materials.

Here, a glass u-tube, formed to a particular design (usually Ubbelohde or Cannon Fenske) is suspended in an oil bath set to the desired test temperature (typically 210°F / 99°C). A set volume of the liquid under test falls under gravity through a calibrated capillary in one arm of the tube. The time taken is multiplied by a calibration derived constant to yield a value for kinematic viscosity, usually expressed in mm<sup>2</sup>/s or centi Stokes (cSt).

Kinematic viscosity  $\nu$  is related to dynamic viscosity  $\eta$  by the density  $\rho$  of the material;

As mentioned, this technique is cost effective and generates accurate results. There are a number of drawbacks, however;



- The method is not suitable for filled waxes
- Not suitable for temperatures close to or below the material's congealing point
- Tubes have to be kept scrupulously clean
- Several tubes are required to cover the range of viscosities encountered in the industry
- The tubes are fragile

*Figure 1: U-tube viscometer*

### **Rotational Viscometers (Brookfield type)**

Robust, moderately priced instruments that measure dynamic viscosities across a very wide range of values. A spindle is rotated in the test fluid - the unit measures the drag on the spindle and calculates a viscosity value. They can be used to test filled and unfilled waxes and have an extensive range of accessories available, including computer control software and small volume handling units. The units are relatively easy to use and are suitable for use in routine QC applications, especially for filled wax viscosity.

Again, there are some drawbacks;



- Not suitable for measurements close to or below the congealing point of the test material
- A relatively limited range of shear rates are available
- A stirring arrangement to prevent filler drop-out is required when testing filled materials
- Measurement at high temperatures is awkward without the use of specialised sample holders.

*Figure 2: Brookfield viscometer*

### **Vibrating Sphere Viscometers (Nemetre type)**

This type of unit uses a metal sphere suspended in the test liquid to measure viscosity. The sphere is electrically vibrated, the test liquid damps the vibration and the amount of power required to maintain the vibration is measured and related to dynamic viscosity. These units are very robust and can make measurements over a wide range of temperatures and viscosities. Drawbacks include;



- Expensive to purchase
- Laboratory units no longer available
- Only one shear rate available
- Measurements through a temperature range take time
- No direct display of viscosity – collected data requires processing

Figure 3: Nametre Viscoliner viscometer

### **Other Measurement Systems**

The paper will shortly discuss an instrument type which the author believes offers the best compromise in terms of routine and experimental rheological measurement of investment casting waxes. There are, however, several other types of rheometer that should be mentioned.

Capillary rheometers are frequently used in the polymer processing industry to characterise molten plastic resins used in injection moulding, film blowing, etc. In these devices, the material under test is extruded through a heated capillary and output from in-line pressure, flow and temperature sensors is used to generate rheological profiles of the melt.

As wax injection is somewhat analogous to plastic injection, this type of unit seems an obvious choice for studying wax behaviour. To the author's knowledge some preliminary work has been done but believes that this type of unit is not routinely used in the PIC wax industry.

Another type of unit, which may lend itself to wax characterisation, is the torque rheometer. Again, this type of device is used in the plastics processing sector. They are essentially small screw extruders – the torque required to melt and process a thermoplastic material is measured and related to its rheological properties. Given the recent industry interest in extruders for wax injection the author wonders if this type of unit might find future application in our sector.

### **Dynamic Rotational Viscometers**



This type of rheometer, the author feels, offers the best solution for characterising the rheological properties of PIC waxes. Several manufacturers offer suitable units. This paper concentrates on an instrument in the author's Muskegon, MI laboratory – a HAAKE RheoStress 600 model, manufactured and supplied by the Thermo Electron Corporation – See *Figure 4*.

This type of instrument is classified as an absolute rheometer. This means that measurements made on the unit can be directly linked to absolute units of physics – force, time, sensor dimensions, etc. Data collected on an absolute rheometer should be independent of the actual model or unit used and can be directly compared with data from other absolute units.

Figure 4: HAAKE RS600 rheometer

The principles of rheological measurement utilised by the RS 600 are fairly simple to describe but the underlying technology and mathematics are complex.

The material under test is sandwiched between two parallel plates. The bottom plate is fixed in rotational terms but can be moved in the vertical axis to facilitate sample loading, cleaning, etc. It also contains a Peltier device so the temperature of the sample can be accurately controlled.

The top plate consists of a circular metal plate fitted to a shaft “floated” on a sophisticated air bearing which reduces friction to a very low level. A very sensitive electric motor is fitted to the other end of the shaft together with extremely sensitive torque and optical rotation sensors.

To make a measurement, a sample of liquid wax is carefully placed on the preheated base plate – See *Figure 5*. The base plate is then raised until a pre-determined gap between it and the top plate is reached, sandwiching the wax between the two plates – See *Figure 6*.



*Figure 5*



*Figure 6*

After a short time to allow the sample to reach thermal equilibrium, the top plate is then rotated by the motor according to pre-set experimental parameters. The electronics in the unit monitor rotational displacement, motor torque, etc. This data, along with sensor dimensions and sample temperature are used to calculate a variety of rheological parameters.

All rheometer functions are controlled by a software package running on a PC based workstation. The software also allows collected data to be manipulated and displayed as required.

### **Operational Notes**

The RS 600 unit is a research grade device and is capable of a variety of rheological measurements. The device is new to the author's lab so his initial approach has been to keep things simple. This means that, routinely, wax blends are characterised by running a viscosity-temperature analysis using a constant shear rate. The following list summarises the key settings and operational steps used.

- *Sensor dimensions*  
Two parallel plate sensors are used – a 35mm diameter plate for filled samples and a 60mm plate for unfilled blends. The larger diameter plate gives greater sensitivity with low viscosity materials. Both test plates are machined from titanium – for durability and, more importantly, for lower rotational inertia. This allows for more accurate rotational control of the sensors.

- *Shear rate*  
The shear rate used to test a material's rheological properties is one of the fundamental factors in such analysis. After a little experimentation, the author settled on a shear rate of  $50\text{s}^{-1}$  for routine testing, i.e. the rheometer runs in CR (constant rate) mode at this shear rate. The effect of shear rate on wax behaviour is discussed further in the Results section.
- *Test Gap*  
The gap between the lower and upper test plates must be at least 0.3mm and no more than 3.0mm for optimum measurement accuracy. Additionally, for filled waxes, the gap should be at least three times greater than the largest sized particle present in the test material. The author routinely uses a gap setting of 1.000mm for both filled and unfilled product. Typically, filler particle size tops out around 250 microns (0.25mm).
- *Test Temperatures*  
Obviously, temperature has a significant effect on the rheological properties of PIC waxes. The author's routine testing regime uses a temperature range of  $100^{\circ}\text{C}$  to  $45^{\circ}\text{C}$  with a ramp of  $-2.5^{\circ}\text{C}/\text{minute}$ . The Peltier heating element in the rheometer base plate will run up to  $185^{\circ}\text{C}$ , so it is possible to test waxes at temperatures seen inside a steam dewaxing autoclave unit. An example of high temperature measurement is presented in the Results section.
- *Sample Presentation*  
It is very important that test samples are presented to the rheometer in a controlled and reproducible fashion. It is possible to melt wax directly on the base plate but the author prefers to load liquid samples. The test material is melted in an oven set a little warmer than the initial load temperature, quickly but thoroughly mixed and loaded onto the base plate using a disposable plastic pipette – See *Figure 5*. The pipettes are cut down for filled products and viscous materials like soluble blends.

Using pipettes allows the operator to load the correct amount of sample every time. This is crucial because the test gap must be filled just so. Too much sample and the material will ooze out from between the plates. Too little and the gap will not be totally filled. Both instances lead to measurement errors. The author has found that it is better to load slightly too much sample then carefully wipe away the excess to give the correct fill level.

With a little training, the RS600 is a very straightforward instrument to use and samples can be analysed very quickly. In fact, the author's lab now tests every production batch of PIC wax for viscosity-temperature response using the procedure outlined above.

## Results

The following section presents some results from the rheometer and illustrate what a flexible tool it is in investigating PIC wax behaviours.

*Figure 7* is a chart displaying viscosity-temperature data gathered from samples of pattern and sprue wax used by a commercial PIC foundry. The test parameters used show how the wax's viscosity response vary as temperature drops from  $100^{\circ}\text{C}$  through  $45^{\circ}\text{C}$ . It's immediately obvious that the sprue wax is significantly lower in viscosity than the pattern material across the temperature range – a desirable separation with respect to minimising dewax shell cracking. The RS600 is capable of generating meaningful data well below the congealing points of the two waxes. This is one of the major benefits of such an instrument – it works in the 'paste' region where other rheometers and viscometers often struggle. Obviously, at extremely low temperatures the test sample breaks up and the plate sensor is no longer effectively coupled with the sample, so data from this region should be treated with caution.

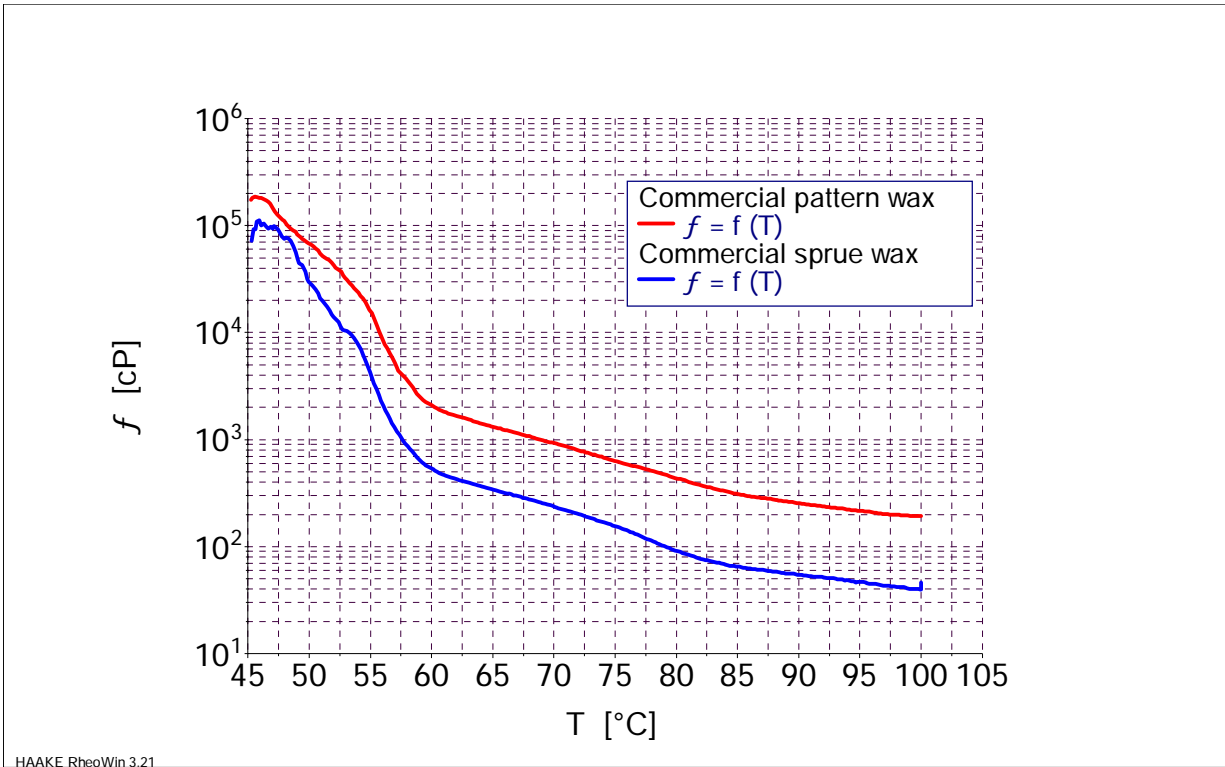


Figure 7

**Repeatability**

Figure 8 is a chart overlaying viscosity-temperature curves from three separate runs on pellets taken from a lot of unfilled sprue wax.

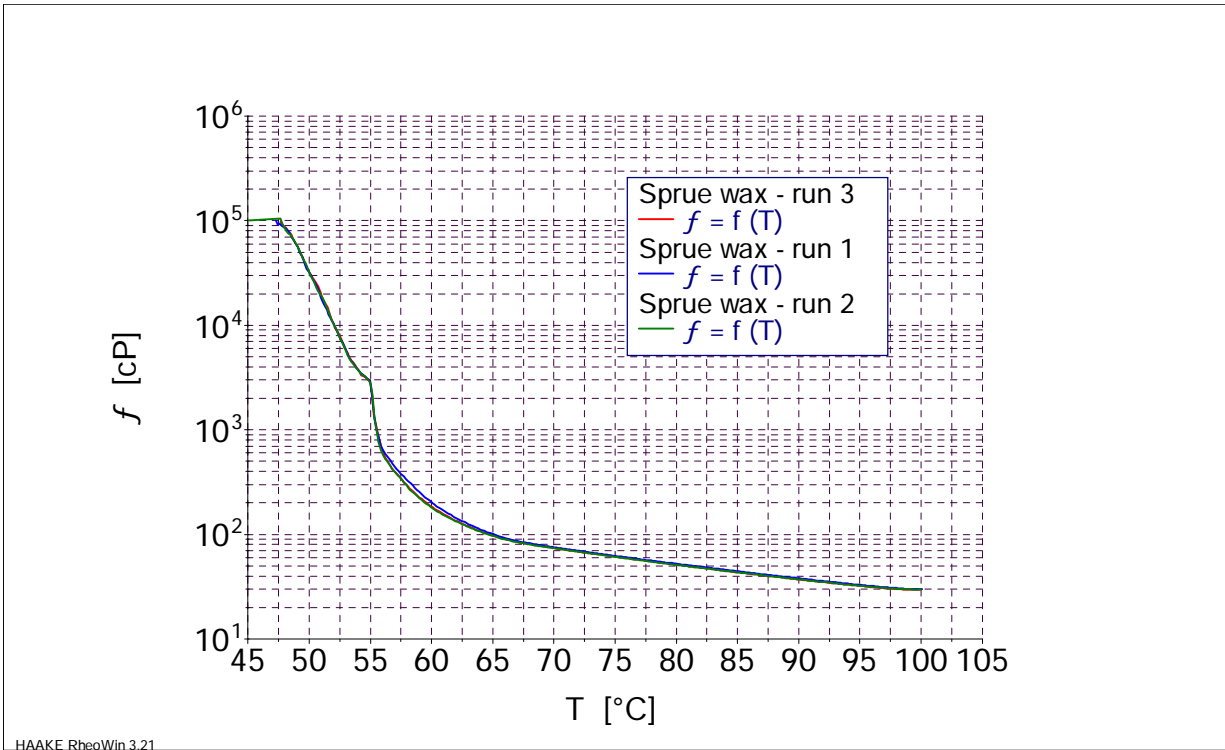


Figure 8

Clearly, the device is capable of generating very reproducible results. As mentioned earlier, a seasoned operator with good technique is required to get the best results.

## The Effect of Shear Rate

Figure 9 shows four viscosity-temperature curves from a filled pattern wax using shear rates from  $1\text{s}^{-1}$  up to  $1000\text{s}^{-1}$ .

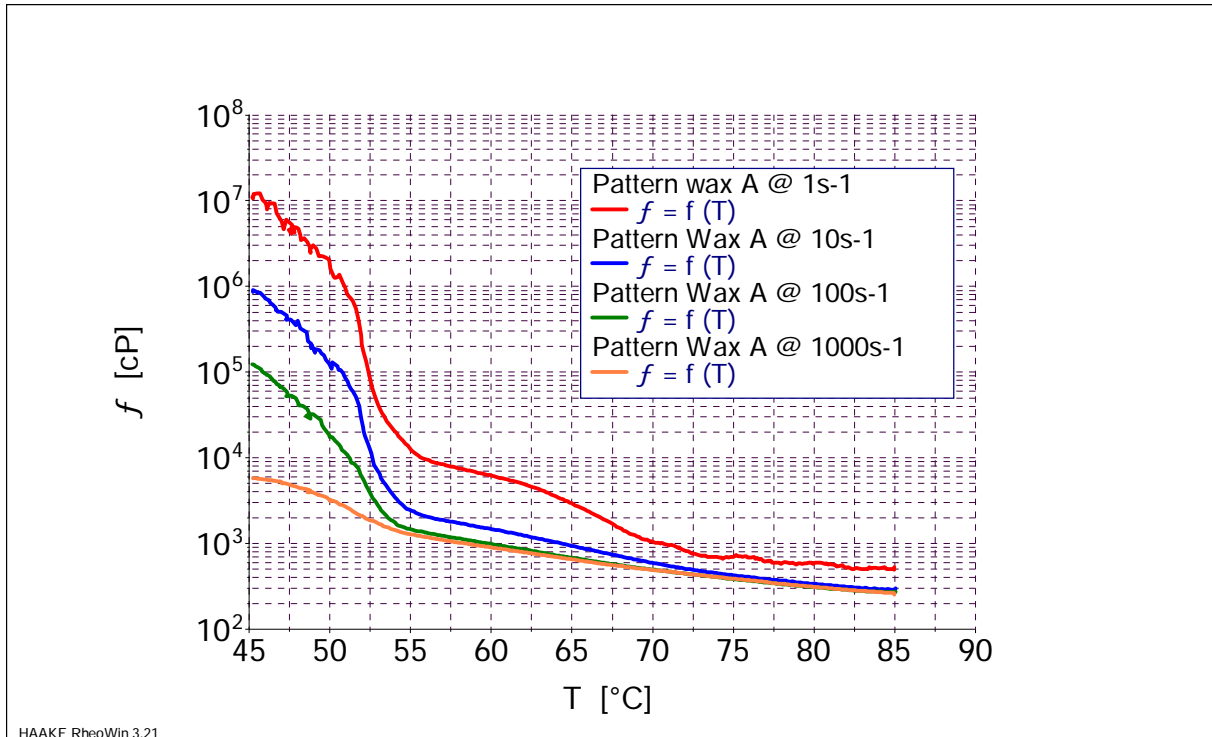


Figure 9

As mentioned, shear rate is an important parameter in rheological measurement. In this instance, the wax blend under test is clearly sensitive to shear rate – it's not a Newtonian liquid and exhibits visco-elastic properties. As shear rate increases the apparent viscosity of the material drops quite significantly.

This data leads to an important question with respect to characterising PIC wax properties – what shear rate (or range of shear rates) do the materials experience during the various stages of the process? The author settled on a shear rate of  $50\text{s}^{-1}$  for routine analysis because it produces good-looking, reproducible curves. There is no reason to believe, however, that this is the best shear rate to use in characterising PIC waxes. In one sense, it doesn't matter if the data generated is used for purely comparative purposes. On the other hand, it's good to generate data that is directly applicable to the real world processes we work with.

Rheological literature (Reference1) indicates that shear rates involved in the injection moulding of polymers fall into the range of  $10^1$  to  $10^4 \text{s}^{-1}$ . The author could not find any explicit reference to shear rates involved in PIC wax injection but thinks that the numbers quoted for polymers may well apply. It is worth noting that wax experiences a variety of shear rates during its trip from the press reservoir into the tooling.

Shear rates for the dewaxing process are probably lower than those related to injection because the forces applied to the wax inside the shell stem from gravitational and hydrostatic sources.

Part of the appeal of a rheometer such as the RS600 is the fact that shear rates can be dialed in with ease. Other viscometers and rheometers traditionally used in PIC wax work do not offer this kind of flexibility.

## High Temperature Analysis

The routine temperature sweep described above looks at wax rheology from typical meltdown temperatures down into the pasty stage (85 - 45°C). But what about the dewax process where, in a steam autoclave, wax temperatures may eventually reach 190 – 200°C?

As mentioned, the RS600 can, when fitted with a Peltier device, record data at temperatures up to 185°C. *Figure 10* is a composite chart for a filled pattern wax used in the commercial sector – one section shows data from a regular cooling run, the other is a heating ramp from 85°C through 180°C.

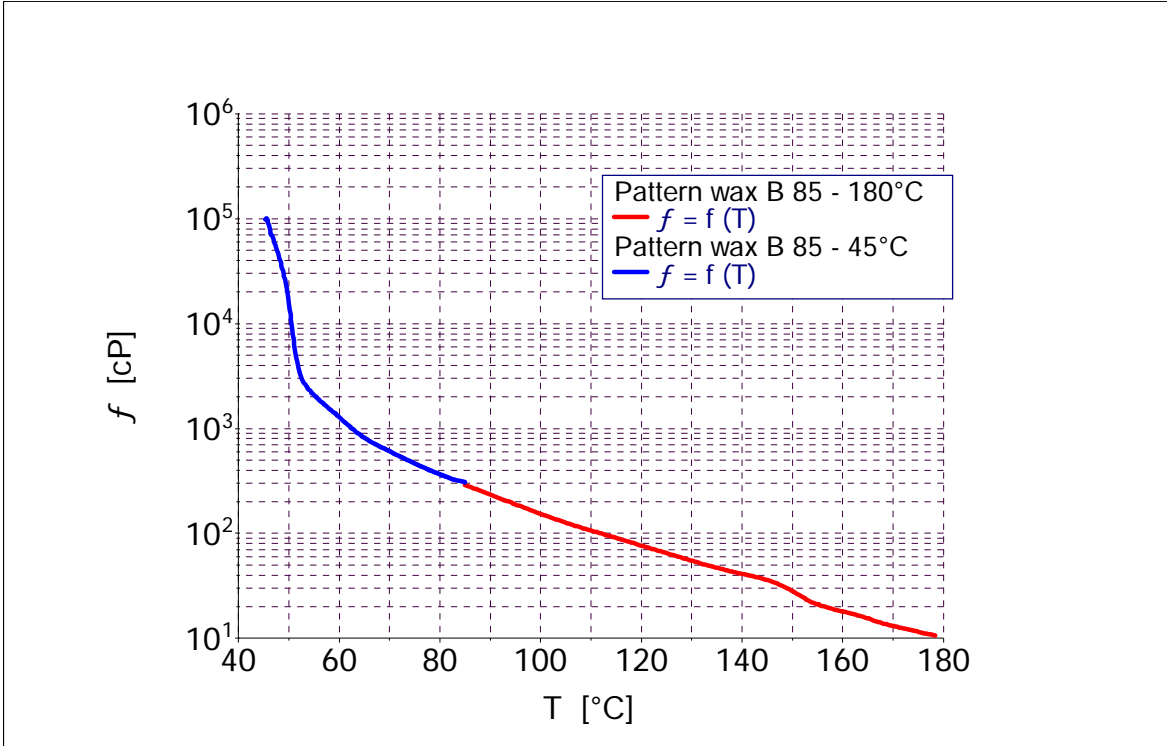


Figure 10

The chart effectively details the viscosity-temperature response of the blend over the complete temperatures range it's exposed to during processing. There is a slight disconnect at 85°C, where one curve ends and the other starts (due to initial instability), but the high temperature curve is an obvious continuation of the regular run.

The high temperature curve exhibits a sudden dip around 150°C. The blend in question is partially filled with bisphenol A, which melts at 153°C. The author believes the rheometer is detecting the melting of the bisphenol A – another interesting perspective that wouldn't have been picked up with more routine devices.

## Filler Morphology

Filled waxes play a vital role in today's PIC industry. A modern rheometer can give the wax formulator a valuable insight into the impact that fillers have on PIC wax properties. *Figure 11* shows viscosity-temperature curves for two wax samples prepared from a common base wax. One sample is filled with spherical beads of cross-linked polystyrene, the other with ground cross-linked polystyrene particles.

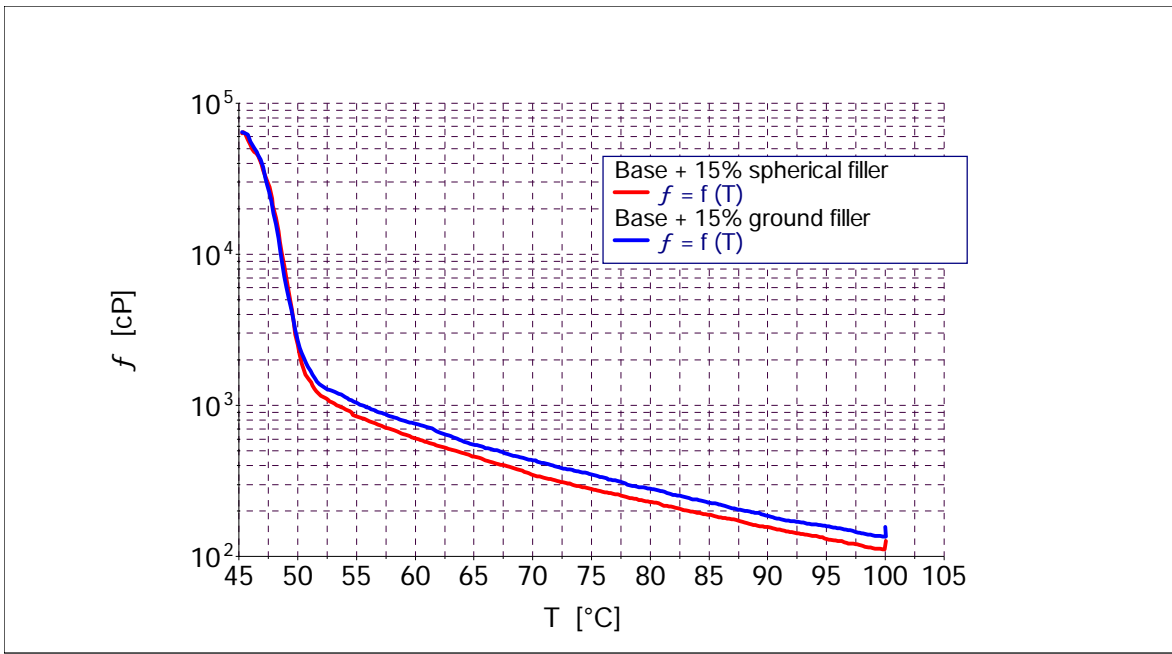


Figure 11

The blend filled with the spherical material has a slightly lower viscosity than the one with ground material. The difference is not great but the rheometer readily distinguishes between the two blends.

### Raw Material Analysis

The use of the rheometer is not limited to studying finished products. The unit lends itself to raw material development issues too - Figure 12 illustrates one such situation. It shows viscosity-temperature data from samples of two hydrocarbon resins blended 50/50 with paraffin wax.

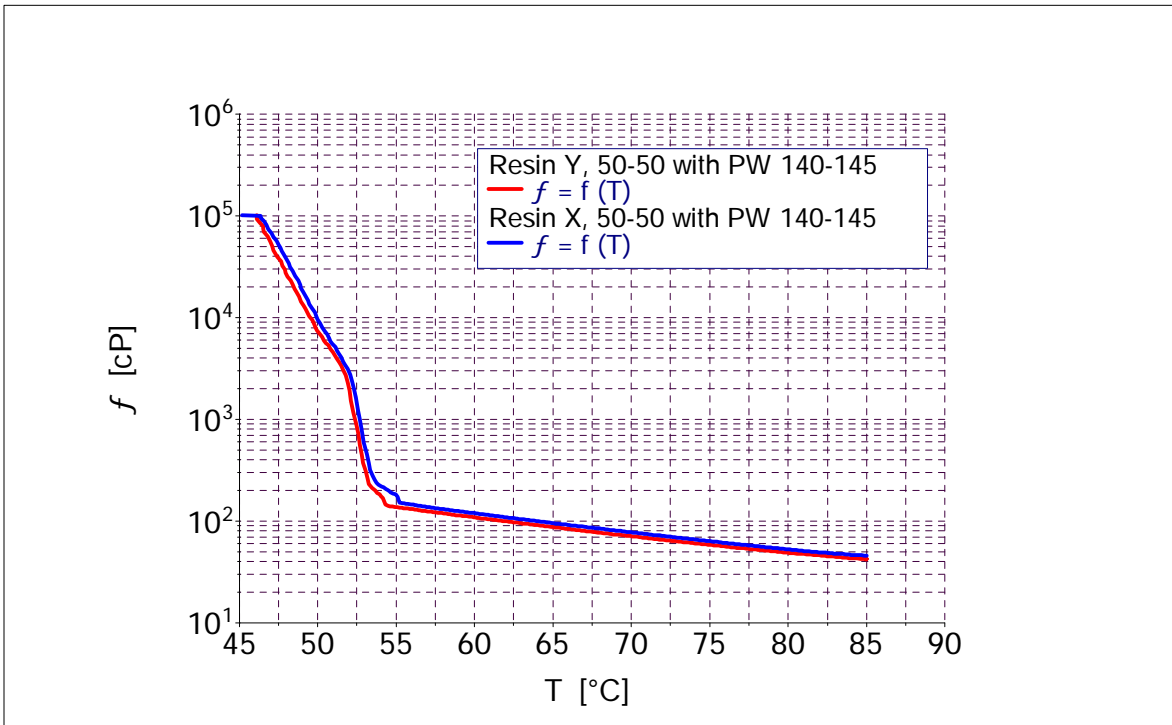


Figure 12

Resin X is from a supplier offering an alternative product to an established material, resin Y. The data from the rheometer indicates that the resins are close cousins – they have very similar chemistries, molecular weight distributions, etc. Obviously, the wax formulator cannot rely entirely on this type of data when making a decision about potential new materials but the information gives a great insight into likely behaviours relative to proven materials.

### **Conclusions & Further Work**

A modern dynamic rotational rheometer is a fantastic tool for characterising several important and fundamental properties of PIC wax blends - the examples presented here clearly demonstrates this. Such devices are expensive but they do offer the user great flexibility, sensitivity and analytical power in one robust, easy-to-use package.

As is often the case when preparing a paper of this nature, avenues of further exploration open up. The author freely admits that the work done so far on the rheometer is merely scratching the surface of its capabilities.

For example, the unit is capable of running in oscillatory mode. Here, instead of subjecting the sample to continuous deformation in one direction, a back and forth oscillation of very small amplitude is applied. Such an approach allows the user to probe the visco-elastic nature of materials without destroying the properties they are trying to measure. This type of approach may offer the wax scientist a window on wax behaviour during dewax conditions, where the material sits in the shell, subject to relatively small forces of a gravitational and hydrostatic nature.

Ultimately, the uses of such rheometers are limited only by the user's imagination. If you can visualise a rheologically related experiment, the rheometer and associated software will generally make the work a viable proposition.

### **References**

1. A Practical Approach to Rheology and Rheometry, 2<sup>nd</sup> edition: Gebhard Schramm, Thermo Haake GmbH, Karlsruhe, Germany.