Failure investigation – Processing difficulties in tube extrusion line

A company had been compounding a tubing resin for the medical device market for a number of years but recently had received a number of customer complaints that the resin was becoming difficult to control when drawn down during production.

The company carried out their normal quality control checks using their in-house laboratory but could not detect any differences between this batch and the retain samples[1].

PPRC solution:

Consulting with the company it became apparent that in drawing the tube extrusion process the dimensional control of the tube became unstable and that the drawdown caterpuller was struggling to correct this. The operators had found that a 10degC increase in the die temperature was helping to correct this but that the resin was running at a temperature were thermal degradation was a risk. This suggested that an underlying rheological issue was causing the change in the resins behaviour.

Capillary rheology is a technique which can be used to measure the viscosity of a resin over the broad range of shear rates and temperatures which are typically experienced during processing. Extensional viscosity can also be determined. This is the form of viscosity that controls the stretching and forming the resin post-extrusion or on this case, the forming of the tube.

Shear viscosity sweeps were carried out in accordance with ISO 11443 and resulting extensional viscosities were determined using the Cogswell method.

Testing methodology:

Shear viscosity was determined at the die temperature normally recorded at the tube extrusion company over the shear rate range 100 to 10,000s^{-1} using an RH7 Malvern Instruments Dual Capillary Rheometer fitted with a zero length die to allow the data to be Bagley corrected and to generate extensional viscosity data. This analysis was carried out on a number of retained resins and a sample of a "suspect" resin returned to the customer.

The shear viscosity profiles are shown in Figure 1 and it's clear that all resins are very similar in behaviour. This finding is consistent with the investigation in the in-house laboratory which included some basic rheological assessment of Melt Flow Rate.

However, looking at the extensional viscosity profile in Figure 2 it is clear that the suspect resin exhibits a higher extensional viscosity. Whilst shear viscosity is a measurement of the resistance to the polymer chains sliding across each other during processing, extensional viscosity measures the resistance to pulling the chains apart[2].

During the tube manufacturing process, once the melt exits the die it undergoes axial and radial stretching from the action of the haul off unit and calibrator. Extensional viscosity behaviour dominates here and has been shown to be very sensitive to the molecular characteristics of the resin such as molecular weight, polydispersity and chain branching to name a few.

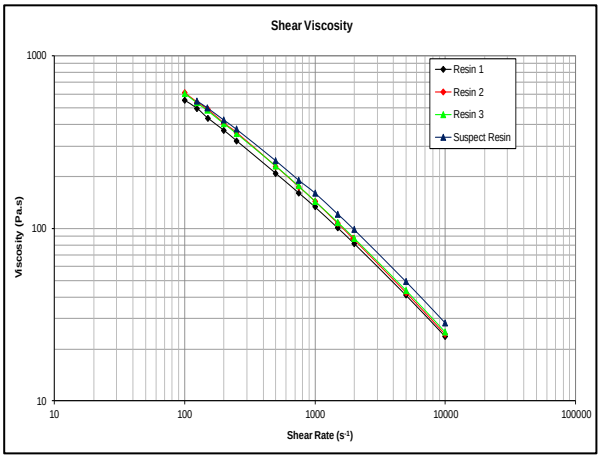

Figure 1: Shear Viscosity Profiles

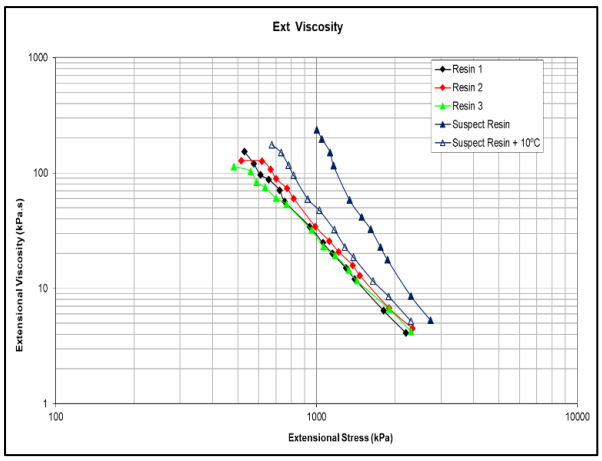

Figure 2: Extensional Viscosity Profiles

Conclusion:

Evidence of the suspect resin exhibiting increased extensional viscosity has been shown which results in the resin being more resistant to stretching in the downstream processing equipment. The small temperature increases being used by the operators would lower this extensional viscosity towards that exhibited by the retain samples, but results in thermal stability compromises.

As underlying molecular characteristics can greatly affect the extensional viscosity of a resin this was further investigated with the material supplier. It was found that the suspect resin had a slightly altered molecular weight and polydispersity than the previously supplied resins, albeit exhibiting the same melt flow rate, and that the effect on tube production had not been anticipated by the supplier. The supplier was able to correct this and the issue was resolved for the compounder.

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[1] Melt Flow Rate was the QC technique used to qualify incoming resin; a single shear stress viscosity measurement in accordance with ISO 1133.

[2] Imagine spreading honey across a piece of bread to make a sandwich and pulling the bread apart after, in the first action the honey molecules are being sheared and in the second they are experiencing extensional stresses.

If you have any questions relating to troubleshooting or characterisation in general, please get in touch with one of the team:

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