Tg and Melting Point of a Series of Polyethylene Glycols Using the Material Pocket

Summary

Polyethylene Glycol was examined using the Material Pocket in the PerkinElmer® DMA 8000. The glass transition temperature for a series of molecular weight samples was determined by analysis of the tan δ against temperature plot. In addition, the melting point was also observed.

Introduction

Dynamic Mechanical Analysis (DMA) is one of the most appropriate methods to investigate relaxation events. Polyethylene Glycol (PEG) is a material with multiple industrial uses. Specifically in the pharmaceutical and cosmetic industries it is used as an excipient for a variety of formulations. PEG can be produced with a variety of molecular weights and the properties vary as a result. Of particular importance is the glass transition temperature as this can effect the application that the material is used for.

DMA works by applying an oscillating force to the material and the resultant displacement of the sample is measured. From this, the stiffness can be determined and the modulus and tan δ can be calculated. Tan δ is the ratio of the loss modulus to the storage modulus. By measuring the phase lag in the displacement compared to the applied force it is possible to determine the damping properties of the material. Tan δ is plotted against temperature and glass transition is normally observed as a peak since the material will absorb energy as it passes through the glass transition.

PEG can be a relatively soft material at room temperature, especially low molecular weight samples. For this reason, the samples in this application note were investigated in the Material Pocket. The Material Pocket is used to support the material in the DMA 8000. The other purpose for using the Material Pocket is that it is possible to heat the samples through melting without destroying the sample geometry or contaminating the instrument.

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Experimental

Temperature scan of PEG.

The sample was added to the Material Pocket and crimped closed. The Material Pocket was mounted in the Single Cantilever Bending clamps and run from -120 °C to 75 °C. The three PEG samples were run in the same way.

Equipment	Experimental Conditions	
DMA 8000 1L Dewar	Sample:	PEG4000, 6000 and 20000
	Geometry:	Single Cantilever Bending
	Support:	Material Pocket
	Temperature:	-120 °C to 75 °C at 3 °C/min^{-1}
	Frequency:	1.0 and 10.0 Hz

Results and conclusion

Figure 1 shows the tan δ response for PEG4000 as an example of the multi-frequency experiments. The glass transition is observed in the tan δ curves and is shown to be frequency dependant confirming it as a relaxation event. The modulus signal is less useful as there is a strong contribution from the Material Pocket to the modulus value, although not to the transitions. The melting point is also shown as the higher temperature peak and shows no frequency dependence.

The tan δ response for the three PEG samples investigated is shown in Figure 2. All data shown was analyzed at 1 Hz frequency. The glass transition is shown as a strong event in all samples and there is a clear trend of increasing Tg with decreasing molecular weight. The samples of PEG4000 and PEG6000 are shown to be almost overlapping which is an indication that these materials are similar in properties due to their close molecular weights. The melting point is seen to increase with increasing molecular weight. There are larger differences seen in the melting points due to the larger differences in lattice energy between the three samples.

This application note has demonstrated that a series of different molecular weight polymers can be investigated by DMA using the Material Pocket. Both the melting point and the glass transition temperature can be obtained with ease. It is worth noting that these materials, and consequently their transitions, will be very dependent on water content. A study under defined relative humidity conditions using the fluid bath and a Controlled Humidity Generator would be a useful exercise.
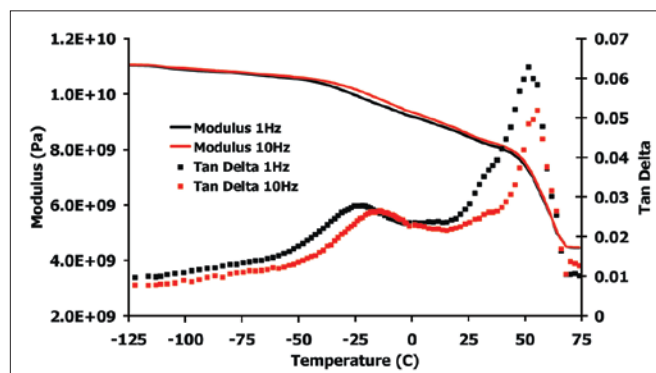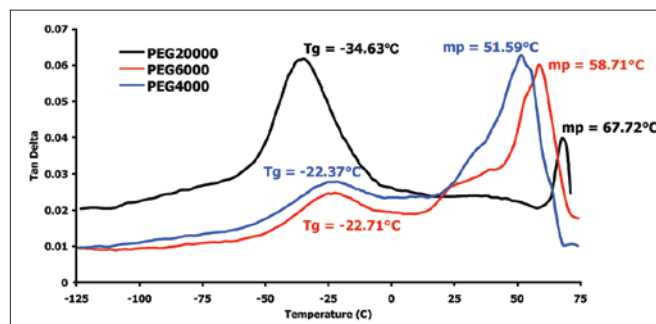

Figure 1. Tan δ response for PEG4000.

Figure 2. Tan δ response for PEG4000, 6000, and 20000.

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