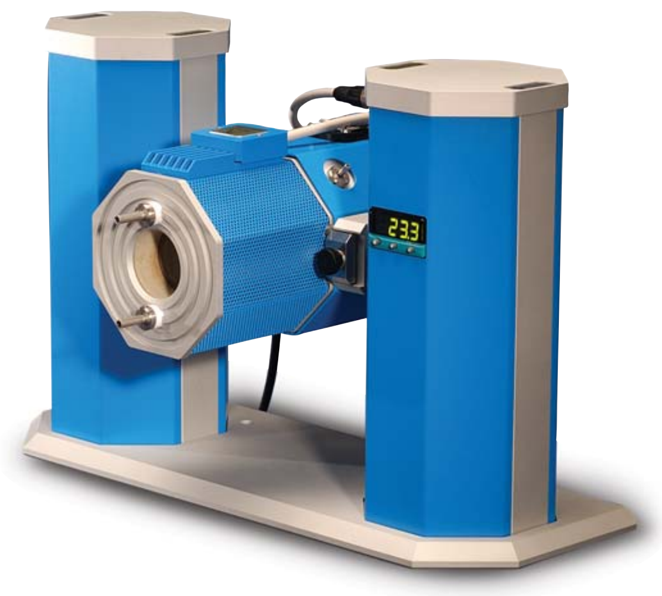
Dissolution of Gelatin Monitored by DMA



Summary

This application note describes the dissolution process of gelatin obtained from an empty pharmaceutical gelcap using a PerkinElmer® DMA 8000. The experiment was performed by cutting a piece of gelatin from the gelcap and mounting it in the DMA 8000. The sample was immersed in water and the mechanical properties monitored as a function of time. It will be shown how the temperature greatly influenced dissolution rate. The modulus gives a good indication of the softening of the material over time and the tan   gives an indication of the material becoming more viscous over time.

Introduction

Gelatin is commonly used in both foods and pharmaceuticals. Pharmaceutically, a gelcap is used to encapsulate an active ingredient or therapeutic formulation. The composition of the gelatin can be formulated to give the best dissolution profile with respect to pH, temperature etc. so that the contents are released at the appropriate time after swallowing. The thickness, overall size, shape and composition of the gelcap can all influence the dissolution properties.

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 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.

This application note will describe some experiments where a sample of gelatin is immersed in water at different temperatures. One advantage of the DMA 8000 is the ability to immerse samples in any geometry. The mechanical properties of the sample as dissolution commences will be discussed and comparison of the different temperature data will be discussed.

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Experimental

Isothermal immersion study of gelatin.
An empty gelcap was cut up to provide small strips of gelatin which were mounted in the DMA 8000. The samples were immersed in water and the tan δ and modulus were monitored as a function of time. The experiment was repeated at a second temperature.

Equipment	Experimental Conditions	
DMA 8000 Fluid Bath Circulator	Sample:	Empty Gelcap
	Geometry:	Single Cantilever Bending
	Dimensions:	3.2 (l) x 5.0 (w) x 0.7 (t) mm
	Temperature:	25 °C and 38 °C isothermally
	Frequency:	1.0 Hz

Results and conclusion

Figure 1 shows the response from the DMA as a function of time. The time data is adjusted so the point of immersion is shown as 30 seconds after the start of the experiment.

In both samples, the modulus decreases with time after immersion reflecting the sample getting less stiff as it dissolves. Eventually, the sample disintegrates so much that data is meaningless and this is the point where the data collection was ceased. The sharp decrease in modulus indicates this point. It is worth noting that the rate of softening and the time taken to destroy the sample were both faster at 38 °C than 25 °C. Also, the initial ingress of water into the gelatin to start the dissolution process was much faster at 38 °C as shown by the short time between immersion and modulus decrease starting.

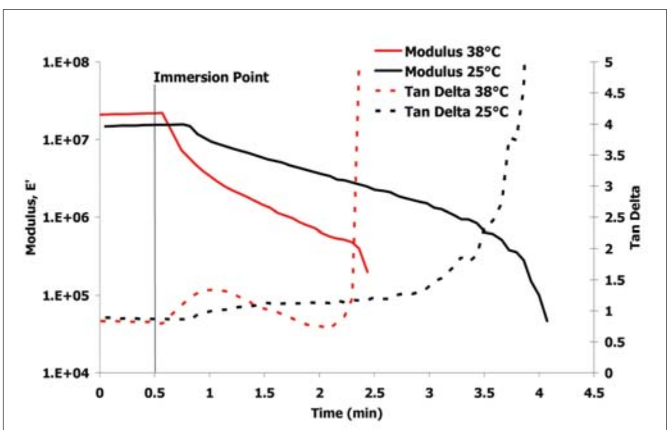

Figure 1. DMA data from immersed gelcap.

The tan δ data is often referred to as the damping factor and can indicate the sample becoming less elastic and more viscous if tan δ increases. The end point of both experiments show this behavior as expected. The sample is no longer a self supporting solid but rather a viscous semi-solid which would display more viscous characteristics. The 38 °C data, and to a lesser extent the 25 °C data, shows a broad peak which might indicate a swelling of the material as a prelude to dissolution.

It has been demonstrated how the DMA 8000 can investigate dissolution and swelling behavior of materials by utilizing the immersion function of the fluid bath. Valuable mechanical information was generated from gelatin using this approach. Testing in solution will often give information not available from running samples in air.

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