Analysis of a Gelatin Capsule Using Controlled Humidity Dynamic Mechanical Analysis

ABSTRACT

This paper discusses the dynamic mechanical analysis of a gelatin capsule material under varying conditions of temperature and relative humidity.

INTRODUCTION

Gelatin is a naturally occurring material which is used in a wide array of applications, most typically in edible/foods, pharmaceutical, photographic and technical products. The use of gelatin in the manufacture of various pharmaceutical dosage forms dates back to the early 19th century and possibly earlier[1]. When stored in an ambient, low-humidity environment gelatin is remarkably stable. However, when combined with water, gelatin forms a semi-solid colloid gel and this can profoundly affect its mechanical properties. Particularly as it relates to storage and handling conditions, it is important to understand and quantify these effects *in situ*.

The TA Instruments DMA-RH Accessory allows the mechanical properties of a sample to be analyzed under constant and/or varying conditions of both relative humidity and temperature. It is designed for use with the Q800 Dynamic Mechanical Analyzer. The DMA-RH accessory is an integrated unit and contains the following components:

Figure 1: The TA Instruments Q800 Dynamic Mechanical Analyzer and DMA-RH Accessory

1. The sample chamber mounts to the DMA in place of the standard furnace and encloses the sample. Peltier elements in the chamber precisely control the temperature to within ±0.1°C. The sample chamber accommodates standard DMA clamps including tension,

cantilever, and 3-point bending, and can be easily removed for rapid conversion back to the standard DMA furnace.

2. The DMA-RH Accessory contains the humidifier and electronics which continuously monitor and control temperature and humidity of the sample chamber. The DMA Q800 and the DMA-RH Accessory are fully software-integrated.
3. A heated vapor transfer line is maintained above the dew point temperature of the humidified gas in order to avoid condensation and provide accurate results.

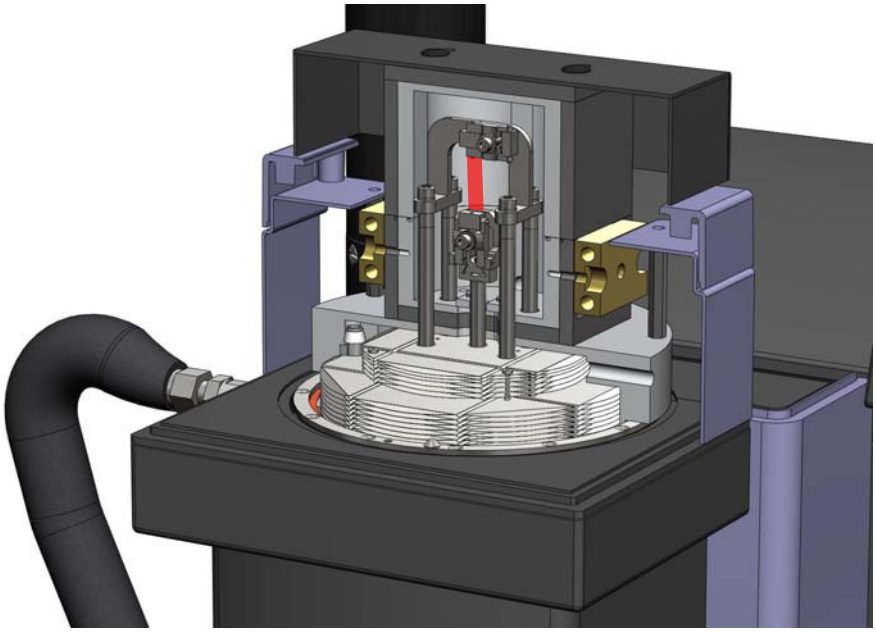

Figure 2: Sample Chamber of the DMA-RH Accessory

The DMA-RH accessory allows for the control of temperature over the range 5-120°C, and humidity over the range 5-95% RH. As such, it is well-suited to investigate gelatin and other polymeric materials which exhibit structural and mechanical instability within this temperature range.

RESULTS & DISCUSSION

A sample of dimension 9.2 mm x 3.2 mm x 0.12 mm was cut from the sidewall of a two-piece hard gelatin capsule. It is important to minimize the thickness of the sample to facilitate efficient water transfer throughout the polymer matrix. The sample was analyzed at a frequency of 1 Hz and amplitude of 20 µm (~0.2% strain) over the humidity range 5-95% RH at a rate[i] of 0.25% RH/min. The experiment was performed at both 25°C and 40°C. The resultant storage modulus and tan delta are shown in Figures 3 and 4 on the next page. The data demonstrate that the gelatin material undergoes a multi-step transition as the humidity is increased. At 25°C, an initial loss in modulus is seen around 25% RH, with a more dramatic loss of structure near 80% RH. At the elevated temperature of 40°C, the initial loss remains near 25% RH, with the subsequent transitions shifting to *ca*. 70% RH. This suggests that even at ambient temperature, the storage conditions for gelatin capsules should be maintained below 70% RH to ensure mechanical integrity of the material.

[i] The RH ramp was composed of a series of short isohume steps, 0.25%RH /1 min hold.

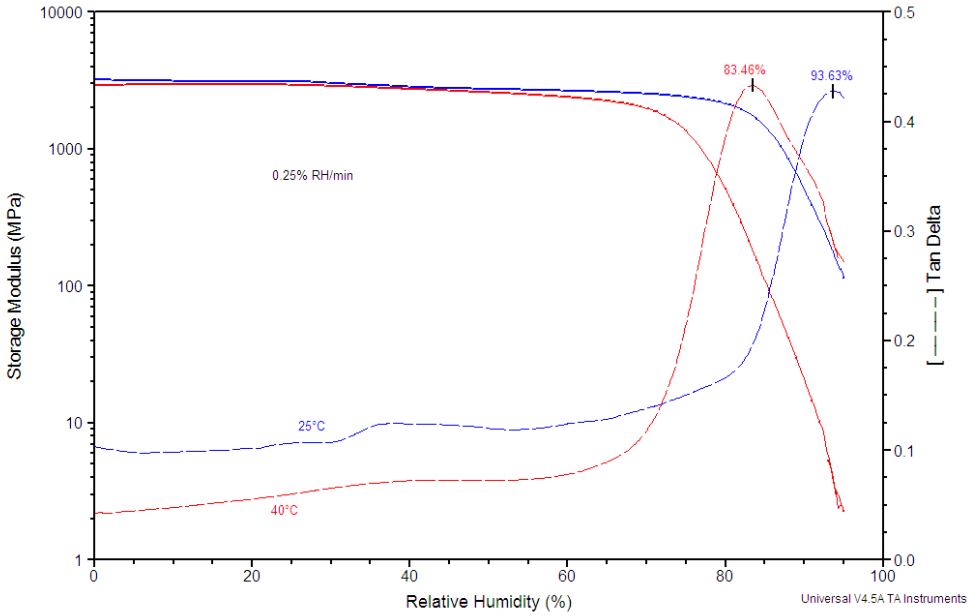

Figure 3: DMA-RH Analysis of Gelatin

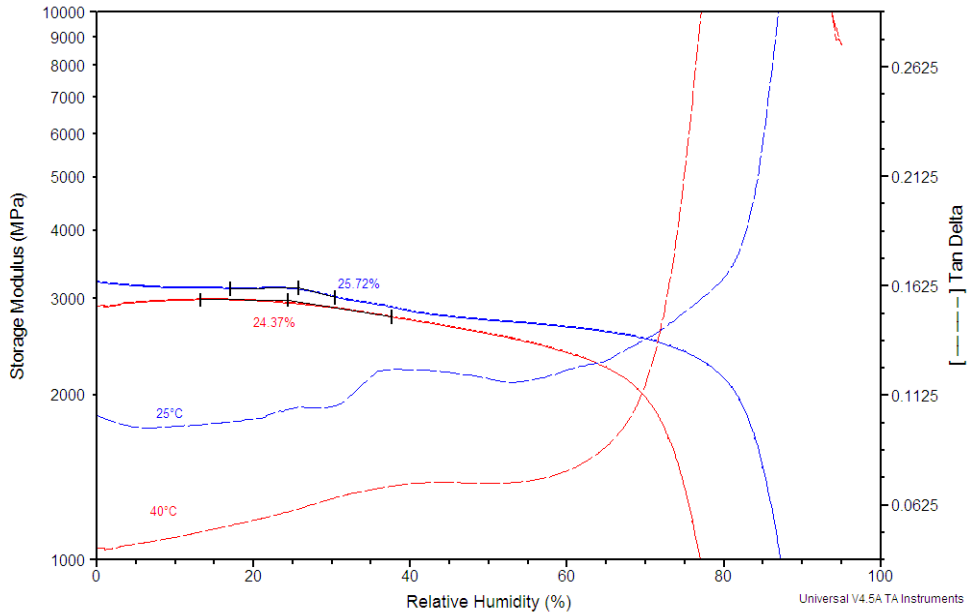

Figure 4: DMA-RH Analysis of Gelatin (Expanded Scale)

CONCLUSIONS

The data presented illustrate the dramatic effect of water on the mechanical properties of gelatin. The TA Instruments Q800 DMA and DMA-RH Accessory provide the ideal platform for this investigation.

REFERENCES

1. http://www.gelatin-gmia.com/html/rawmaterials_app.html (Gelatin Manufacturer's Institute of America)

TA INSTRUMENTS

United States
109 Lukens Drive, New Castle, DE 19720 • Phone: 1-302-427-4000 • E-mail: info@tainstruments.com

Canada
Phone: 1-905-309-5387 • E-mail: shunt@tainstruments.com.

Mexico
Phone: 52-55-5200-1860 • E-mail: mdominguez@tainstruments.com

Spain
Phone: 34-93-600-9300 • E-mail: spain@tainstruments.com

United Kingdom
Phone: 44-1-293-658-900 • E-mail: uk@tainstruments.com

Belgium/Luxembourg
Phone: 32-2-706-0080 • E-mail: belgium@tainstruments.com

Netherlands
Phone: 31-76-508-7270 • E-mail: netherlands@tainstruments.com

Germany
Phone: 49-6196-400-7060 • E-mail: germany@tainstruments.com

France
Phone: 33-1-304-89460 • E-mail: france@tainstruments.com

Italy
Phone: 39-02-2742-11 • E-mail: italia@tainstruments.com

Sweden/Norway
Phone: 46-8-555-11-521 • E-mail: sweden@tainstruments.com

Japan
Phone: 813-5479-8418 • E-mail: j-marketing@tainstruments.com

Australia
Phone: 613-9553-0813 • E-mail: sshamis@tainstruments.com

India
Phone: 91-80-2839-8963 • E-mail: india@tainstrument.com

China
Phone: 8610-8586-8899 • E-mail: info@tainstruments.com.cn

Taiwan
Phone: 886-2-2563-8880 • E-mail: skuo@tainstruments.com

Korea
Phone: 82.2.3415.1500 • E-mail: dhrhee@tainstruments.com

To contact your local TA Instruments representative visit our website at www.tainstruments.com