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. 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.

![Image: Sample Chamber of the DMA-RH Accessory](image)

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\(^1\) 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.

\(^1\) The RH ramp was composed of a series of short isohume steps, 0.25%RH /1 min hold.

TA367 2
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

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