Submission ID #: 61468
Scriptwriter Name: Bridget Colvin
Project Page Link: https://www.jove.com/account/file-uploader?src=18747788

Title: Evaluation of the Curing of Adhesive Systems by Rheological and Thermal Testing

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Author Questionnaire

1. Microscopy: Does your protocol involve video microscopy, such as filming a complex dissection or microinjection technique? **N**

2. Software: Does the part of your protocol being filmed demonstrate software usage? **Y**

Videographer: All video files provided, do not film screen capture steps

3. Filming location: Will the filming need to take place in multiple locations (greater than walking distance)? **Y, 48 km apart**

Protocol Length
Number of Shots: **58**

Introduction

1. Introductory Interview Statements

REQUIRED:

1.1. **Ana Díaz**: Our methodology combines thermal analysis and rheology to characterize the curing process of an adhesive and to obtain useful information for industrial adhesive selection **[1]**.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

REQUIRED:

1.2. **Javier Tarrío**: This technique allows the creation of a standard guide for the curing process study of an adhesive system, making it easier to compare different adhesives **[1]**.

1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

OPTIONAL:

1.3. **Bárbara Sánchez**: This methodology can also be used as an acceptance criterion in the quality control of adhesive systems **[1]**.

1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

Protocol

2. Manufacturer Curing Condition Checking: Thermogravimetric Cured Sample Testing

2.1. To perform thermogravimetric test to determine the inorganic filler content and the temperature at which the material starts to degrade, open the **Procedure** tab **[1]** and click **Editor**. Drag the **Ramp** segment to the **Editor** screen and establish the ramp as 10 or 20 degrees/minute to 900 degrees Celsius **[2]**.

2.1.1. WIDE: Talent opening Procedure tab, with monitor visible in frame

2.1.2. SCREEN: screenshot_1: 00:00-00:13

2.2. Click **OK** and open the **Notes** tab. Select **Air** as the purge gas and set the flow rate to 100 milliliters per minute. Click **Apply [1]**.

2.2.1. SCREEN: screenshot_1: 00:14-00:22

2.3. Then close the furnace **[1]** and start the experiment **[2]**.

2.3.1. Talent closing the Furnace, with monitor visible in frame

2.3.2. SCREEN: screenshot_1: 00:23-00:30

3. Manufacturer Curing Condition Checking: Differential Scanning Calorimetry (DSC) of a Cured Sample

3.1. To perform a differential scanning calorimetry test of a cured sample, open the **Procedure** tab **[1]**, click **Test**, and select **Custom [2]**.

3.1.1. WIDE: Talent opening Procedure tab, with monitor visible in frame

3.1.2. SCREEN: screenshot_2: 00:00-00:04

3.2. Click **Editor** and drag an **Equilibrate** segment indicating the temperature at which to start the experiment **[1]**.

3.2.1. SCREEN: screenshot_2: 00:05-00:17

3.3. Drag a **Ramp** segment to the **Editor** screen and introduce a heating rate of 10 or 20 degrees/minute and the final temperature into the command editor window **[1-TXT]**.

3.3.1. SCREEN: screenshot_2: 00:18-00:27 **TEXT: Final temperature lower than degradation temperature**

3.4. Drag a **Ramp** segment to the **Editor** screen and introduce a 10 or 20 degrees/minute cooling rate to a temperature tentatively below the glass transition **[1]**.

3.4.1. SCREEN: screenshot_2: 00:28-00:39

3.5. Drag another **Ramp** segment to the **Editor** screen and introduce a 10 or 20 degrees Celsius/minute heating rate to a temperature slightly below the degradation temperature **[1]**.

3.5.1. SCREEN: screenshot_2: 00:40-00:47

3.6. Open the **Notes** tab and select **Nitrogen** as the flow gas. Set the flow rate to 50 milliliters/minute and click **Apply [1]**.

3.6.1. SCREEN: screenshot_2: 00:48-00:57

3.7. Then place a reference pan **[1]** and a pan with a sample inside the DSC cell **[2]** and launch the experiment **[3]**.

3.7.1. Talent placing reference pan into cell

3.7.2. Talent placing sample pan into cell

3.7.3. SCREEN: screenshot_2: 01:48-01:52

4. DSC Fresh Sample Analysis: Ramp Curing Test

4.1. To analyze the fresh sample through a heating-cooling-heating test, open the **Procedure** tab **[1]** and click **Test** and **Custom [2]**.

4.1.1. WIDE: Talent opening Procedure tab, with monitor visible in frame

4.1.2. SCREEN: screenshot_3: 00:00-00:04

4.2. Click **Editor** and drag an **Equilibrate at minus 80 degrees Celsius** segment to the **Editor** screen **[1]**.

4.2.1. SCREEN: screenshot_3: 00:05-00:13

4.3. Drag a **Ramp** segment and set the heating rate to 10 or 20 degrees Celsius/minute to a temperature slightly below the degradation temperature and insert another **Equilibrate at minus 80 degrees Celsius** segment **[1]**.

4.3.1. SCREEN: screenshot_3: 00:14-00:26

4.4. Then drag a **Ramp** segment and set the heating rate to 10 or 20 degrees Celsius/minute to the same temperature as before. Click **Ok [1]**.

4.4.1. SCREEN: screenshot_3: 00:27-00:37

4.5. Then place a reference pan **[1]** and a pan with a sample inside the DSC cell **[2]** and click **Start** to launch the experiment **[3]**.

4.5.1. Talent placing reference pan into cell

4.5.2. Talent placing sample pan into cell

4.5.3. SCREEN: screenshot_3: 01:23-01:30

5. DSC Fresh Sample Analysis: Isothermal Curing Test

5.1. To perform an isothermal curing test, open the **Procedure** tab **[1]**, click **Test**, and select **Custom [2]**.

5.1.1. WIDE: Talent opening tab, with monitor visible in frame

5.1.2. SCREEN: screenshot_3: 00:00-00:04

5.2. Click **Editor** and drag a **Ramp** segment to the Editor screen. Introduce a 20 degrees Celsius/minute to the chosen isothermal temperature **[1]**.

5.2.1. SCREEN: screenshot_4: 00:02-00:18 *Video Editor: can speed up*

5.3. Then introduce an Isothermal segment with enough time to complete the cure **[1]**.

5.3.1. SCREEN: screenshot_4: 00:19-00:23

5.4. To check the degree of curing reached, introduce an **Equilibrate at 0 degrees Celsius** segment, add a **Ramp** segment, and set the heating rate between 2 and 20 degrees Celsius/minute to the maximum temperature **[1]**.

5.4.1. SCREEN: screenshot_4: 00:24-00:35

5.5. Drag the **Mark end of cycle** segment to the editor window and insert another **Equilibrate** segment with a temperature of minus 80 degrees Celsius **[1]**.

5.5.1. SCREEN: screenshot_4: 00:36-00:48

5.6. To obtain the final glass transition, add a **Ramp** segment with a heating rate between 2 and 20 degrees Celsius/minute to the same temperature as indicated before and click **OK [1]**.

5.6.1. SCREEN: screenshot_4: 00:49-00:55

5.7. In the **Tool** tab, select **Instrument Preferences** and **DSC** and set a temperature lower than the isotherm temperature of the experiment **[1]**.

5.7.1. SCREEN: screenshot_4: 00:56-01:04

5.8. Click **Apply** and open the **Control** tab to select **Go to Standby Temperature [1]**.

5.8.1. SCREEN: screenshot_4: 01:05-01:10

5.9. Then place a reference pan **[1]** and a pan with a sample inside the DSC cell **[2]** and click **Start [3]**.

5.9.1. Talent placing reference pan

5.9.2. Talent placing sample pan

5.9.3. SCREEN: screenshot_4: 02:38-02:41

6. **Logarithmic Strain Sweep Test**

6.1. To perform a logarithmic strain sweep test, open the **Procedure** tab **[1]** and select **Oscillation Amplitude [2]**.

6.1.1. WIDE: Talent opening Procedure tab, with monitor visible in frame

6.1.2. SCREEN: screenshot_5: 00:00-00:09

6.2. Set the experimental temperature to room temperature, the frequency to 1 hertz, and the logarithmic sweep from 1 x 10^{-3} to 100% of strain **[1]**.

6.2.1. SCREEN: screenshot_5: 00:10-00:30 *Video Editor: please speed up*

6.3. Place the sample on the bottom plate with the upper plate separated about 40 millimeters from the lower plate **[1]** and lower the upper plate until a gap of about 2 millimeters is observed between both plates **[2]**.

6.3.1. Talent placing sample onto bottom plate

6.3.2. Upper plate being lowered

6.4. Then trim the excess adhesive **[1]** and start the experiment **[2]**.

6.4.1. Adhesive being trimmed

6.4.2. SCREEN: screenshot_5: 00:31-00:33

7. **Isothermal Multifrequency Curing Test**

7.1. To monitor the curing of the adhesive, click the **Procedure** tab **[1]** and select **Conditioning Options [2]**.

7.1.1. WIDE: Talent clicking Procedure tab, with monitor visible in frame

7.1.2. SCREEN: screenshot_6: 00:00-00:12

7.2. Set the **Mode** to Compression, the **Axial Force** to 0 Newtons, and the **Sensitivity** to 0.1 Newtons **[1]**.

7.2.1. SCREEN: screenshot_6: 00:13-00:19

7.3. Click **Advance** and set the Gap change limit to 2000 microns in both the up and down directions **[1]**.

7.3.1. SCREEN: screenshot_6: 00:20-00:36 *Video Editor: please speed up*

7.4. Insert a new oscillatory time sweep step and set the experimental temperature to room temperature, the test duration as a function of the estimated curing time based on the Data Sheet of the adhesive, and the strain percentage acquired from the previous logarithmic strain sweep test **[1]**.

7.4.1. SCREEN: screenshot_6: 00:44-01:16 *Video Editor: please speed up*

7.5. Select **Discrete** and set the frequencies 1, 3 and 10 hertz for all of the samples **[1]**.

7.5.1. SCREEN: screenshot_6: 01:17-01:38 *Video Editor: please speed up*

7.6. Then load a new sample **[1]** and start the experiment **[2]**.

7.6.1. Sample pan being placed

7.6.2. SCREEN: screenshot_6: 01:41-01:48

8. Torque sweep and Temperature Scan Testing

8.1. To perform a torque sweep test, open the **Procedure** tab **[1]** and select **Oscillation Amplitude [2]**.

8.1.1. WIDE: Talent opening Procedure tab, with monitor visible in frame

8.1.2. SCREEN: screenshot_7: 00:00-00:05

8.2. Then set the temperature to room temperature, the frequency to 1 hertz, and the logarithmic sweep from 10 to 10000 micronewton-meters of torque and start the experiment **[1]**.

8.2.1. SCREEN: screenshot_7: 00:06-00:36 *Video Editor: please speed up*

8.3. From the torque sweep test, choose a torque amplitude within the linear viscoelastic region to use in the Temperature Ramp test **[1]**. Then, select **Temperature Ramp** and set up the experiment at room temperature with a ramp rate of 1 degree Celsius/minute to ensure a uniform distribution of the temperature into the sample, a frequency of 1 hertz, and the torque amplitude determined from the torque sweep test **[2]**. NOTE to VO: Long one, feel free to break up into 1 and 2.

8.3.1. Choosing the torque amplitude within the linear viscoelastic region

8.3.2. SCREEN: screenshot_8: 00:00-00:45

8.4. Close the furnace of the rheometer **[1]** and open the air stopcock of the furnace **[2]**.

8.4.1. Talent closing the furnace

8.4.2. Talent opening the air stopcock

8.5. Then start the experiment **[1]**.

8.5.1. SCREEN: screenshot_8: 00:52-00:54

Protocol Script Questions

A. Which steps from the protocol are the most important for viewers to see?
n/a

B. What is the single most difficult aspect of this procedure and what do you do to ensure success?

n/a

Results

9. Results: Representative Cured Adhesive System Rheological and Thermal Testing

9.1. These thermogravimetric results show different degradation temperatures **[1]** and different inorganic fillers for each studied adhesive **[2]**. The mass loss observed between 600-800 degrees Celsius suggests the presence of calcium carbonate as filler **[3]**.

9.1.1. LAB MEDIA: Figure 1A *Video Editor: please emphasize data lines between 185 and 240 °C (x-axis) and 110-80% (y-axis)*
9.1.2. LAB MEDIA: Figure 1A *Video Editor: please emphasize data lines at 900 °C*
9.1.3. LAB MEDIA: Figure 1A *Video Editor: please emphasize blue and dotted green lines between 600-800 °C.*

9.2. For this two-component adhesive, in the heat flow curve, there is no evidence of residual cure **[1-TXT]** and the small deviation **[2]** cannot be assigned with certainty to the glass transition reported by the manufacturer **[3]**.

9.2.1. LAB MEDIA: Figure 2A **TEXT: tetrahydrofurfuryl methacrylate + benzoyl peroxide**
9.2.2. LAB MEDIA: Figure 2A *Video Editor: please emphasize green data line at 60 °C*
9.2.3. LAB MEDIA: Figure 2A

9.3. In this Table, the degree of curing **[1]** of a two-component system at different temperatures **[2]** was calculated by comparing the curing enthalpy acquired at each temperature to that obtained in a heating ramp **[3]**.

9.3.1. LAB MEDIA: Table 1 *Video Editor: please emphasize Degree of curing column*
9.3.2. LAB MEDIA: Table 1 *Video Editor: please emphasize Temperature column*
9.3.3. LAB MEDIA: Table 1 *Video Editor: please emphasize Curing enthalpy column*

9.4. Through a rheological multifrequency test of a fresh two-component adhesive sample **[1]**, the gelation time of the material can be observed as the point at which the phase angle becomes frequency independent **[2]**.

9.4.1. LAB MEDIA: Figure 5
9.4.2. LAB MEDIA: Figure 5 *Video Editor: please emphasize green data lines*

9.5. In these isothermal multifrequency tests using the one- and two-component silane polymer adhesive systems **[1]**, no sign of gelation is observed **[2]** and a comparison of the slopes of the moduli of both adhesives reveals that the two-component silane polymer adhesive cures faster **[3]**.

9.5.1. LAB MEDIA: Figures 6 and 7
9.5.2. LAB MEDIA: Figures 6 and 7 *Video Editor: please emphasize green data lines in both graphs*
9.5.3. LAB MEDIA: Figures 6 and 7 *Video Editor: please emphasize blue data lines in Figure 7*

9.6. In this rheological temperature scan test of a two-component adhesive sample cured for one hour **[1]**, the glass transition can be clearly observed **[2]**.

9.6.1. LAB MEDIA: Figure 8
9.6.2. LAB MEDIA: Figure 8 *Video Editor: please emphasize gray data line at 60.2 °C*

Conclusion

10. Conclusion Interview Statements

10.1. **Silvia Gómez**: Do not delay starting the test when using a fresh sample and be sure to prepare a thoroughly mixed solution when using a two-component system **[1]**.

10.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera