#25 Investigating the Effect Of Successive Heat and Cool Cycles on a Thermoplastic Material (EVA, Hot Melt Glue)

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I. INTRODUCTION

Description
In this investigation, a mini glue gun is used as both an injection molding simulator and a melt index viscometer. Hot melt glue is squeezed into rubber tubing sections and allowed to cool. A slit in the side of the rubber tubing allows the cooled hot melt glue sections to be removed and remelted in the hot melt glue gun; hence samples of successive heated and cooled thermoplastic can be produced. The samples are then evaluated by weighing the glue extruded over a constant time period.

Student Audience
This investigation is appropriate for high school, general or polymer chemistry, and chemical technology students.

Goals for the Experiment
By performing this investigation, students will
• recognize properties of thermoplastics and understand that successive heat and cool cycles may affect the viscosity of thermoplastics,
• use a balance to determine the weight of melted thermoplastic extruded in a set time period, and
• experience various aspects of experimental design [as demonstrated by the use of an ordinary household tool (a mini hot melt glue gun) as both a polymer manufacturing item (an injection mold press) and a polymer testing instrument (a melt index viscometer)], and
• gain insight into the inherent limitations of the experiment and its accuracy and precision.

Recommended Place in the Curriculum
This investigation could be used during a discussion of
• thermoplastics,
• physical properties, and/or
• viscosity.
II. STUDENT HANDOUT

Investigating the Effect Of Successive Heat and Cool Cycles on a Thermoplastic Material (EVA, Hot Melt Glue)

Scenario
An injection molding company has just contacted your company, a supplier of thermoplastic resin, complaining that they suspect a shipment of “out of specification” thermoplastic resin. Their story is that a problem with an automatic dispensing machine resulted in the presses running with many incompletely filled molds. The incomplete molded articles were subsequently ground. Upon reprocessing, the ground thermoplastic generated many “burned” articles. You have been asked to check if the viscosity of the polymer changes by more than 5% with three heat and cool cycles.

Introduction
Thermoplastics soften and flow as viscous liquids when heated. The molten material solidifies when cooled. Ideally, the heating and cooling cycles can be repeated many times with little change in the properties of the polymer. In practice, heating and cooling cycles may shorten or lengthen polymer chains. Polymer additives (anti-oxidants, UV stabilizers, etc.) or other additives such as mold release agents may influence the properties of the polymer with repeated heating and cooling. Viscosity is dependent upon polymer length. A melt index viscometer melts a polymer and measures the polymer flow for a set time under variables set by the viscometer. In this experiment a mini hot melt glue gun is used to melt a polymer. The amount extruded during a set time period will be determined by weighing the extruded polymer.

Safety, Handling, and Disposal
• Safety goggles are required in the laboratory.
• If tubing molds are to be prepared by the students, use of an exacto or craft knife also requires caution.
• The mini hot melt glue gun and melted glue are hot! Be careful handling both the hot melt glue gun and the hot melt glue samples. Additional safety suggestions concerning use of the hot melt glue and gun are included in the procedure.

Materials
• goggles
• close fitting cotton or garden type gloves to protect hand against cuts and/or thermal burns
• 12 cheap, thin mini glue sticks
• 12 weighing papers or boats to extrude hot melt glue samples onto
• a balance that weights to 0.001 grams
• 9 plastic tubing sections, each 1.5 inches long and slit lengthwise (The inside diameter of the plastic tubing must match the diameter of the glue stick.)

If the plastic tubing molds are prepared by students:
• plastic tubing (The inside diameter of the tubing must match the diameter of the glue stick.)
• sharp scissors
• exacto or craft knife
Procedure

To Prepare Plastic Tubing Molds
1. Cut the plastic tubing into nine 1.5 inch sections with scissors.
2. Using a craft knife, carefully slit the tubing section lengthwise. (This will allow the tubing section to be opened and the cooled glue plug to be removed.)

Use of the Hot Melt Glue Gun for Injection Molding
1. Load a mini hot melt glue gun with a glue stick and allow it to warm up.
2. Extrude the hot melt glue into a mold by filling it halfway on one end, starting at the center and filling to the top. Turn the tubing mold over and fill the other half, again starting at the middle and filling to the top. (See Figure 1.)
   CAUTION: Hold the plastic tubing molds in the middle of the tubes with the first finger and thumb. The plastic housing of the tubing insulates the fingers again thermal burns. Do not attempt to remove the glue from the tubing mold until it is cloudy and cool.

   Figure 1: Hold the tube between the thumb and first finger when filling it with glue.

3. Allow the filled tubing molds to cool. While the tubing molds are cooling, the melt index viscometer part of the experiment may be done with the commercial (“virgin”) glue stick.
4. After the tube is cool, the sides of the tube can be pulled apart at the slit, releasing the molded glue stick. This molded glue stick has been through one heat and cool cycle.
5. Repeat steps 2–4 until you have nine molded glue sticks. Be sure to put the glue sticks from each batch in a labeled tray, cup or beaker.
6. Set aside three of the molded glue sticks (one heat and cool cycle) for the viscosity testing. Repeat steps 2–4 for a second heat and cool cycle using the remaining 6 molded glue sticks.
7. Set aside three of the molded glue sticks (two heat and cool cycles) for the viscosity testing. Repeat steps 2–4 for a third heat and cool cycle using the remaining 3 molded glue sticks.

Use of the Hot Melt Glue Gun As a Melt Index Viscometer
1. Label and weigh a weighing paper or boat.
2. Allow the hot melt glue gun to warm for at least one or two minutes after its last extrusion. This will insure that the temperature of the gun is the same (its maximum) for each trial.
3. Using virgin glue sticks, extrude hot glue onto the labeled weighing paper or boat for a set time period. The time should be from 1 to 5 seconds, timed as accurately as possible and constant for all determinations in this part of the experiment. (Experience suggests that one steady squeeze of the trigger in 1 second, may be more reproducible than a 4 or 5 second extrusion. The smaller extrusion time also allows for more samples to be extruded giving more data with the same amount of thermoplastic. The barrel of the gun can be emptied in under 10 seconds so the rate of reheating of the glue stick introduces that error into times of 10 seconds or longer.)

4. Weigh the labeled paper or boat and glue extrusion. Determine the weight of glue extruded.

5. Repeat the procedure for a total of at least 5 determinations.

6. Analyze and, if appropriate, average the data from the 5 or more determinations.

7. Repeat the simulated melt index determination (steps 1-6 above) for the glue sections that have been through one, two, and three heat and cool cycles.

Questions
1. Does the viscosity of hot melt glue, EVA (ethylene vinyl acetate copolymer), change with repeated heat and cool cycles?

2. Does the viscosity of thermoplastic resin change more than 5% with three heat and cool cycles, suggesting “out of specification” material?

3. What are some potential sources of error in this investigation and how would each affect the results?
III. INSTRUCTOR NOTES

Investigating the Effect Of Successive Heat and Cool Cycles on a Thermoplastic Material (EVA, Hot Melt Glue)

Purpose
The purpose of this experiment is to mold a thermoplastic, EVA, ethylene vinyl acetate copolymer (hot melt glue), into mini glue sticks and determine how the number of heat and cool cycles affects the viscosity of the thermoplastic.

Time Required
This can be accomplished in a one to two hour session depending upon the number of samples of each heat and cool cycle students determine. Use of 4 to 5 second extrusion time in the melt index viscometer part of the experiment will lengthen the time the experiment takes and the amount of hot melt glue required.

Suggested Group Size
Groups of two students are suggested. Results may be more constant if one student does all the squeezing for the melt index viscometer part of the experiment. The other student can monitor the time of the extrusions and do the molding in the injection molding part of the experiment. Groups of more than two students may result in too much observation only by some of the students.

Materials
- mini hot melt glue guns
- hot melt glue sticks (12 per pair of students)
- weighing paper or boats
- balances that weigh to 0.001 grams
- plastic tubing, the inside diameter of with matches the outside diameter of the glue stick
- scissors
- exacto or craft knife

Safety
- Safety goggles are required in the laboratory.
- If tubing molds are to be prepared by the students, use of an exacto or craft knife also requires caution.
- Thermal burns resulting from hot glue are the greatest hazard in this experiment. Gloves might help protect against these. For safety reasons, a mini hot melt glue gun is used, this limits the amount of hot melt glue to be extruded in one time to about 1.5 grams, less than 2 mL. This limits the accuracy of the timing possible in the melt index viscometer part of the experiment but the safety benefit outweighs any accuracy which might be gained from a larger capacity gun. It is important to remember that the hot melt glue gun is only simulating specialized polymer processing and testing equipment.

Points to Cover in Pre-Laboratory
- The properties of thermoplastics and polymer processing could be discussed. In this case the testing method, that of a melt index viscometer, is very similar to some of the conditions of the polymer processing, extrusion, and molding.
- The limitations of this experiment can be discussed, the small amount of plastic heated at any one time, molded glue sticks with an air bubble or two inside, molded sticks that bulge at bit at
the end, etc.
• Accuracy and precision, and the process of developing a reproducible testing method might also be considered.
• Students should be warned against thermal burns with the hot melt glue and glue guns. (The glue becomes opaque in the tubing molds when cool.)

**Procedural Tips and Suggestions**

• Since this is a home-made version of specialized polymer processing and testing equipment, students should be challenged and encouraged to recognize the variables they may not be able to control (Does each mini glue gun’s barrel heat the glue to the exact same temperature? Does each experimenter pull on the trigger of the glue gun with the same pressure? How accurately is the time of the extrusions measured?) How do these variables affect the accuracy and precision of this crude simulation?

• In testing the activity, the use of a glitter hot melt glue was also explored. The glitter hot melt glue works in the experiment, but it is formulated with far more tactifier such that removing the glue sections from the plastic tubing molds was far more difficult. The use of a cheap hot melt glue is suggested, it may be formulated with less added tactifier and thus easier to remove from the plastic tubing molds. Different hot melt glues might be compared. Glues with more tactifier can be used if the tubing molds are only used once, and the mold release on the plastic tubing is still available.

**Sample Results**

The quality of the data may, as shown here, be less than optimum. The students can still draw conclusions based upon their data and compare their conclusions with those of other groups. If their conclusions vary, the importance of reproducibility can be introduced and discussed.

The information provides is, in sequence: mean (cycle data) (number of samples, range of samples, small sample standard deviation).

**15 Second Extrusion Time**

1.46 grams (virgin cheap hot melt glue) (2 samples; 1.36–1.56 g; 0.14 g)
1.50 grams (one heat and cool cycle) (2 samples; 1.40–1.60 g; 0.15 g)
1.52 grams (two heat and cool cycles) (2 samples; 1.50-1.54 g; 0.03 g)

**10 Second Extrusion Time**

1.46 grams (virgin cheap hot melt glue) (7 samples; 1.37–1.54 g; 0.070 g)
1.35 grams (one heat and cool cycle) (7 samples; 1.12–1.61 g; 0.18g)
1.50 grams (two heat and cool cycles) (5 samples; 1.39–1.57 g; 0.068 g)
1.37 grams (three heat and cool cycles) (4 samples; 1.11–1.69 g; 0.24 g)

The similar extrusions for 15 and 10 seconds both indicates, the barrel of the glue gun is emptied in under 10 seconds.

**5 Second Extrusion Time**

0.88 grams (virgin cheap hot melt glue) (9 samples; 0.72–1.03 g; 0.12 g)
0.84 grams (one heat and cool cycle) (5 samples; 0.78–0.87; 0.04 g)
0.91 grams (two heat and cool cycles) (5 samples; 0.75–1.02 g; 0.13 g)
1.06 grams (three heat and cool cycles) (3 samples; 0.93–1.23 g; 0.15 g)
0.77 grams (virgin glitter glue) (6 samples; 0.57–0.99; 0.16 g)
0.56 grams (one heat and cool cycle) (13 samples; 0.38–0.79 g; 0.098 g)
0.57 grams (two heat and cool cycles) (3 samples; 0.53–0.62 g; 0.04 g)
0.63 grams (three heat and cool cycles) (3 samples; 0.59–0.68 g; 0.06 g)

1 Second 1 Steady Squeeze of the Glue Gun Trigger
0.20 grams (virgin cheap hot melt glue) (12 samples; 0.15–0.23 g; 0.022 g)
0.31 grams (one heat and cool cycle) (12 samples; 0.28–0.39; 0.030 g)
0.32 grams (two heat and cool cycles) (12 samples; 0.25–0.39 g; 0.051 g)
0.30 grams (three heat and cool cycles) (15 samples; 0.22–0.42 g; 0.047 g)

Plausible Answers to Questions
1. Does the viscosity of hot melt glue, EVA (ethylene vinyl acetate copolymer), change with repeated heat and cool cycles?
A: Answers will vary but should agree with the data gathered. Using the “1 Second 1 Steady Squeeze…” data from the Sample Results, the viscosity changes significantly from virgin to heated and cooled. The number of heat and cool cycles does not appear to have much impact on the viscosity.

2. Does the viscosity of thermoplastic resin change more than 5% with three heat and cool cycles, suggesting “out of specification” material?
A: Yes, both the five second and one second extrusion times in the Sample Results show changes greater than 5% after three heat and cool cycles.

3. What are some potential sources of error in this investigation and how would each affect the results?
A: Answers will vary but should include, for example, such things as not letting the glue gun regain its maximum temperature before using it as a viscosity tester. In this case, the glue would not be as viscous and thus the amount extruded would be too low.

Variation and Extensions
• Have students develop a method for testing the following scenario.

Additional Industrial Scenario
A customer, a supplier of thermoplastic resins, has just contacted your company, inquiring whether use of a different mold release agent will change the recyclability of your thermoplastic. You have been asked to find out if the viscosity of the thermoplastic changes by more than 5% with 3 or 4 heat and cool cycles and use of the new mold release agent.

Reference
Gates, J., Henkel Corporation, private communication.