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Lab-scale Reproduction of Siloxane Foam Synthesis

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[CLSE207] | **Team members:** Albert DeAngelis, Benjamin LeDoux, Conrad Roos | **Faculty adviser:** Dr. Frank Gupton, Dr. Ben Ward | **Sponsor:** Evonik Industries | **Sponsor adviser:** Dr. Anthony Beauglehole, Lyndon McClure, Afton Trent

The Problem

Evonik currently has a plant operation to produce Siloxane foam for their customers. When the same production was attempted on the lab-scale for the purposes of experimentation and further innovation, the foam produced did not show the same qualities as the plant's product.

The Plan

- Mimic the heating and agitation profile of the plant-scale reactor
- Developing a method utilizing spectroscopy (Ramen, IR, or UV-Vis) to determine the quality of the product prior to the foaming process
- Use a 1 liter glass jacketed reactor with an agitator, recirculating heating bath, and thermocouple

Agitation Profile

We used the equivalent rates of mass transfer scale ratio for agitation to determine the revolution's per minute needed on the lab-scale to best mimic the plant-scale agitation.

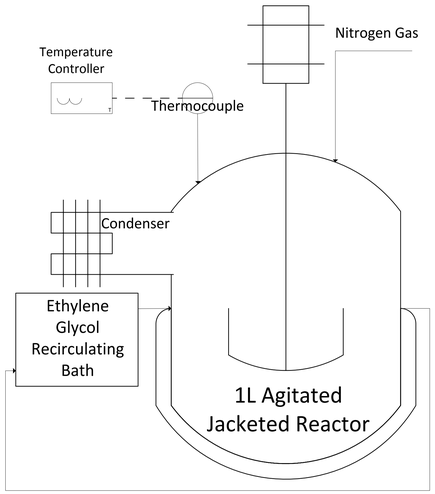
$$N_2 = N_1 \times \left(\frac{D_1}{D_2}\right)^{2/3}$$

$N_2 = rpm\ of\ agitator\ for\ lab-scale\ reactor$
$N_1 = rpm\ of\ agitator\ for\ plant-scale\ reactor$
$D_1 = diameter\ of\ plant-scale\ agitator$
$D_2 = diameter\ of\ lab-scale\ reactor$

Constraints

- 1 liter vessel
- Fit in a bench top fume hood

Lab-scale Reactor



Results

What was found was that the off-specification foams exhibited an absorption peak at 300 to 320 nm on UV-Vis Spectroscopy. On the other hand, React IR and Ramen Spectroscopy were inconclusive, because they cannot determine the arrangement of the molecules.

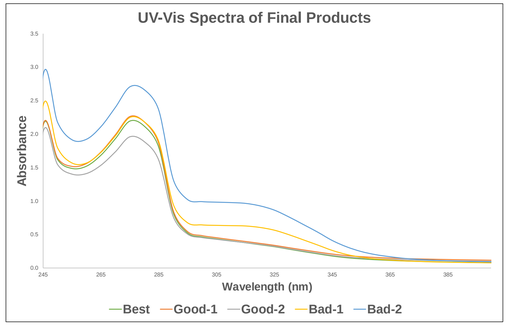


Figure 1. UV-Vis spectra for products of various quality.

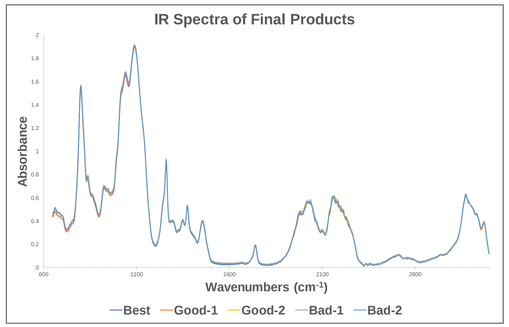


Figure 2. IR spectra for products of various quality. All products returned the same spectra and overlap on the graph, showing that IR would **not** be a viable test to differentiate products of varying quality.

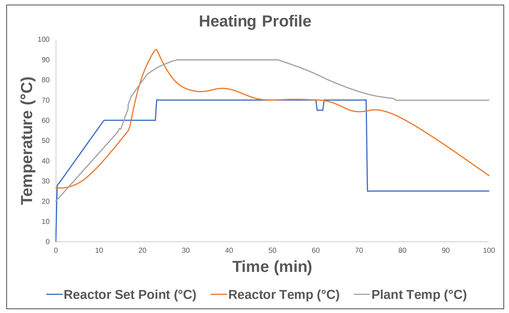


Figure 3. Heating profile for polyether in lab-scale reactor

What Was Delivered

- A comparable heating profile to the plant reactor was created using a programmable recirculation bath.
- The mass transfer scale ratio for agitation was found for the plant scale and applied to the lab process to create similar agitation.
- A possible new method to determine the product quality before foaming was found through the use of UV-Vis spectroscopy.

Future Work

A Design of Experiment (DOE) surface can be created using 11 carefully controlled experiments at various temperatures and agitation speeds to allow for the identification of pertinent variables as well as optimum settings for those variables to produce an acceptable quality product.

Acknowledgements

Dr. Jeremie Zoueu, Rudy Krack

Sources

1. Coker, A. K. (2001). *Modeling of chemical kinetics and reactor design*. Boston, MA: Gulf Professional Pub.