PolySciTech Division of
AKINA, INC.
3495 Kent Avenue
West Lafayette, IN 47906
http://www.polyscitech.com/
John Garner, Manager E-mail: jg@akinainc.com
Faye Jessmon, Laboratory Technician E-mail: fj@akinainc.com

WhitePaper: mPEG-PCL Thermal properties

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Introduction

The process of thermogelation is one by which the poly(ethylene glycol) (PEG) portion of a block copolymer hydrates at a decreased temperature and dehydrates at an increased temperature due to differences in the relative entropy/enthalpy of the PEG-water interaction. This process renders several polyester-PEG block copolymers thermosensitive when dissolved in water. The purpose of this research is to determine if thermosensitivity applies to select mPEG-(Polycaprolactone)PCL block copolymers.

Method

Rheology test

Each sample solution was dissolved at 20% w/v (1g of polymer plus 5ml of water) over 2 days with shaking at 4C followed by 15 minutes of sonication and storage at room temperature. After this the vials were observed to contain a milky-looking dispersion of the mPEG-PCL which was used for testing.

Rheology testing was performed on an AR550 (TA instruments) with 60mm 2degree cone on 2ml of polymer solution. The viscosity of solution at 0.1 (sec-1) and 5C was measured (1minute peak hold 5 second test intervals). Rheology thermal sweep was performed by oscillating at constant 6.283 rad/s, 0.1% strain, in increments of 2.5C ranging from 5-45C with 3 minutes of temperature equilibration at each point.

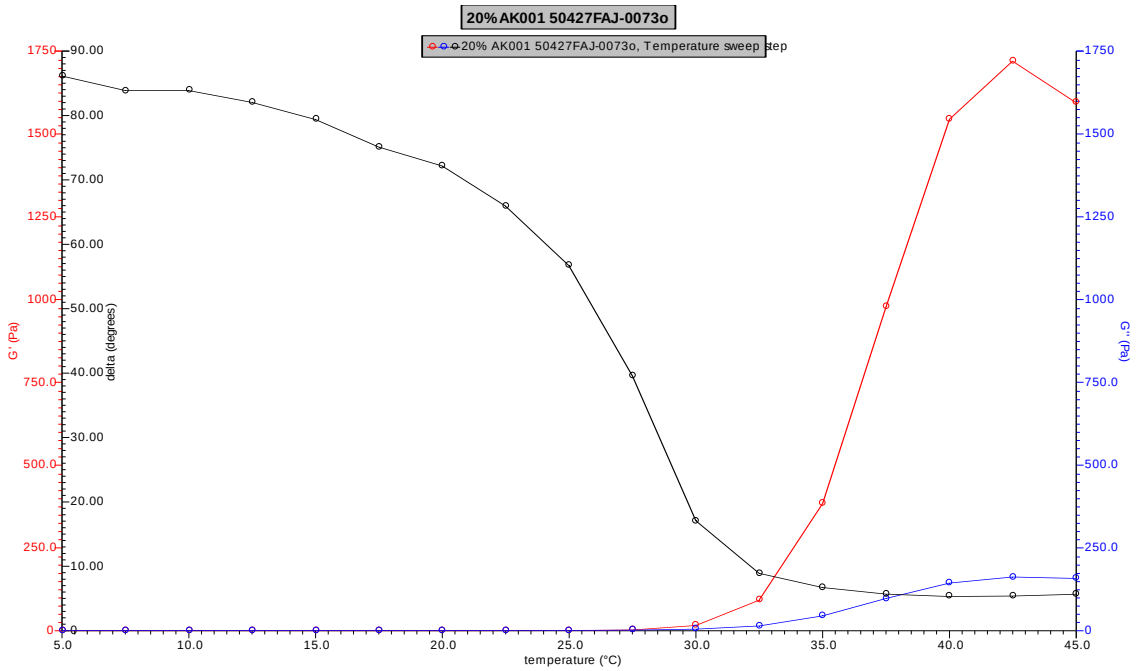
Tip Test

20% w/v solutions of each polymer were loaded into a 37°C orbital shaking incubator and equilibrated at least overnight at 37°C. Then each solution was flipped over and observed for gelation or flow.

Results

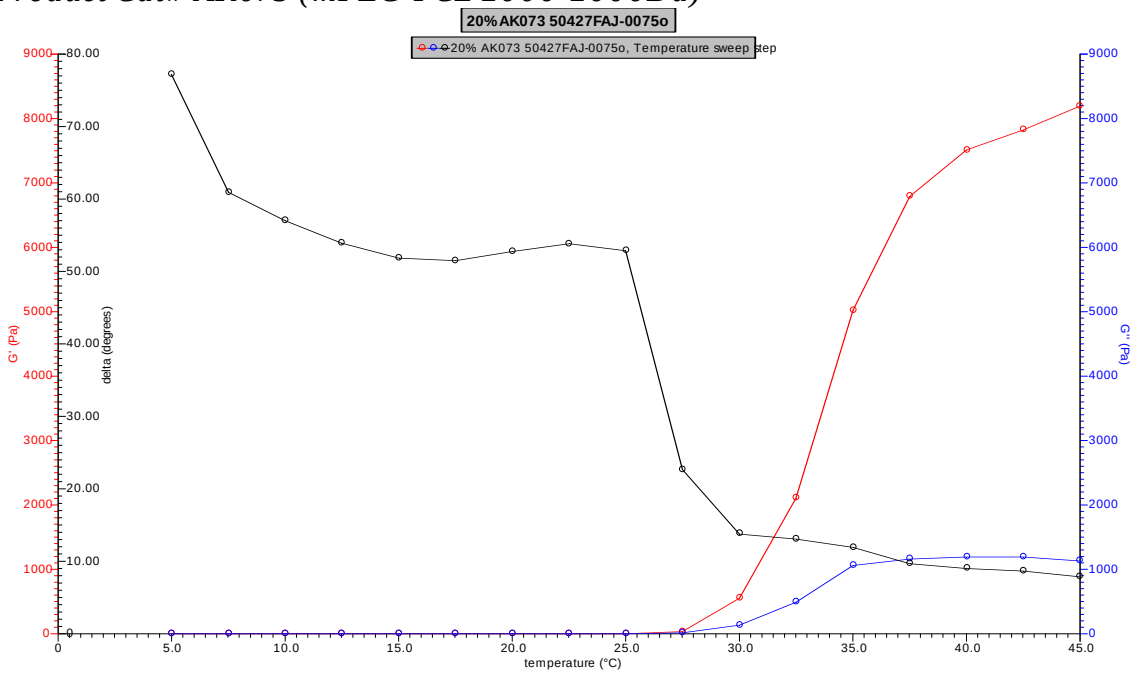
The resultant rheological data was obtained for each solution

Product Cat# AK001 mPEG-PCL (Mw 2000-5200 Da)



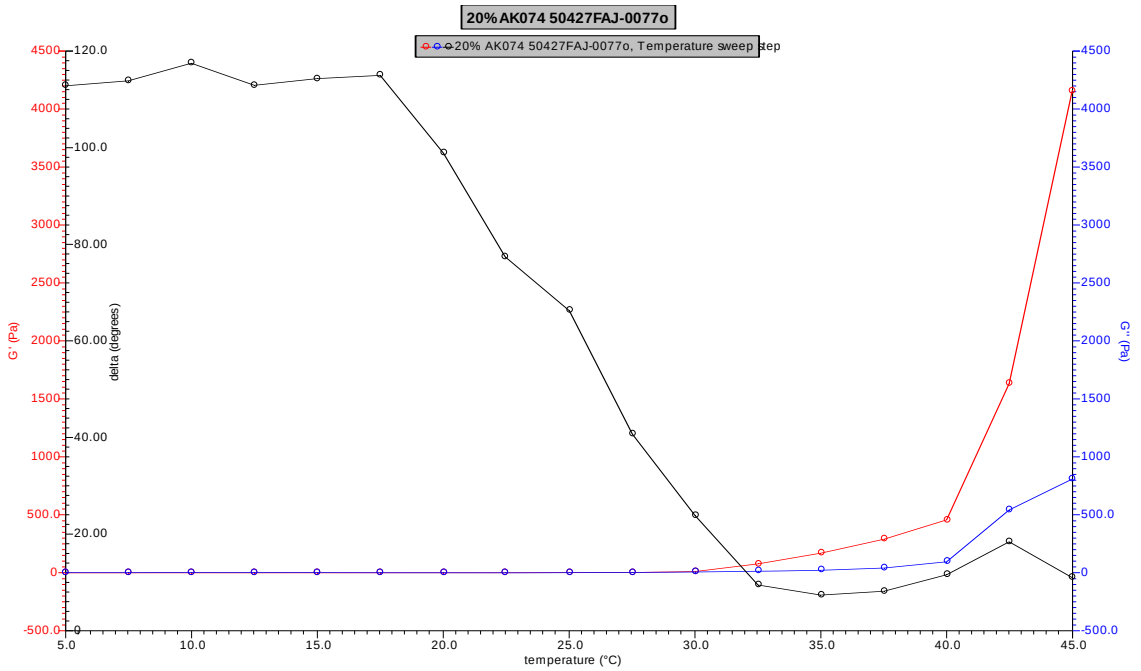
Viscosity 20% w/v solution at 5C	0.2465 Pa.s

Product Cat# AK073 (mPEG-PCL 2000-2000Da)



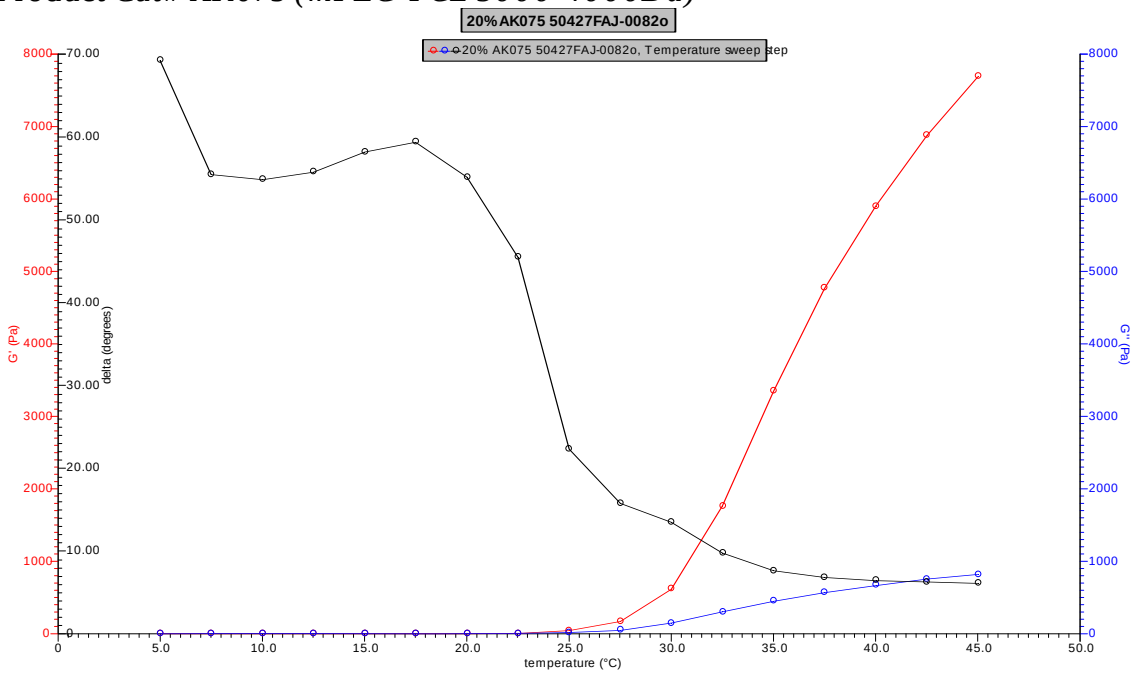
Viscosity 20% w/v solution at 5C	1.982 Pa.s

Product Cat# AK074 *(mPEG-PCL 2000-4000Da)*



Viscosity 20% w/v solution at 5C	0.1675 Pa.s

Product Cat# AK075 *(mPEG-PCL 5000-4000Da)*



Viscosity 20% w/v solution at 5C	3.109 Pa.s

Tip Test

When the vials were tipped, the solution from each flowed easily down the sides of the vial with little to now resistance. The only polymer which resisted this was the AK074 (mPEG-PCL 2000-4000) product which had separated into an

aqueous and a solid layer. The bulk of the aqueous layer left the side of the jar and went down the side while a thin layer of white solid was left indicating polymer precipitation.

Conclusion

The rheological properties indicate formation of a gel by a sharp G' increase. However, the lack of a substantial G" increase indicates that this transition is a transitory effect. Taken together, these tests indicate that, when processed in this manner, there is a thermal transition for these mPEG-PCL polymers, but it is unsuitable to form a full gel at 37°C. This transition is rather more of a precipitation than a gelation effect.