Introduction

The TA Discovery HR-3 Rheometer is a premier research-grade rheometer with integrated linear Dynamic Mechanical Analysis. Suitable for a wide variety of materials from Newtonian and non-Newtonian fluids to soft solids like hydrogels and powders.

The HR3 provides high sensitivity enabling the measurement of lower viscosities and weaker liquid and soft-solid structures. Superior dynamic performance gives a higher level of accuracy in measurements of G' and G''. The HR3 also consumes less material by allowing for small sample amounts to be used.

Features of the TA Discovery HR-3 Rheometer

- Peltier-controlled sample plate from -40°C to 200°C.
- Geometries available:
  Flat Plate (Diameter: 20 mm SS & 40 mm Al)
  Cone (Diameter: 40 mm Al, Angle: 1°).
- Controlled stress, strain, strain rate, steady shear or oscillation; rotational, tensile, compressive or extensional deformation.

Torque (Oscillation)	0.3 - 200 nN.m
Torque (Steady Shear)	1 - 200 nN.m
Angular Velocity	0 - 300 rad/s
Max. Normal Force	50 N
Normal Force Sensitivity	0.005 N
Normal Force Resolution	0.005 N

Structural & Biophysical Core Facility

Greg Wasney

Manager, Structural & Biophysical Core Facility
Peter Gilgan Centre for Research & Learning
The Hospital for Sick Children
686 Bay Street, Rm. 21.9708
Toronto, ON. M5G 0A4

Email: *greg.wasney@sickkids.ca*

Office: 416.813.7209
Office Internal Ext. 307209
Lab: 416-813-7654 ext. 309442

http://lab.research.sickkids.ca/sbc-facility/

SickKids
THE HOSPITAL FOR SICK CHILDREN

Produced by Brandon Cordeiro

The Hospital for Sick Children's

Structural and Biophysical Core Facility

Discovery HR-3 Rheometer

- The study of stress - deformation relationships.
- Measure viscosity, rigidity, shear/ strain behaviour, and deformation behaviour of materials.
- Suitable for powders, hydrogels, Newtonian and non-Newtonian fluids, complex fluid such as emulsions, suspensions, paints, inks, coatings, slurries, and oils.

TA Discovery HR-3 Rheometer - Gelation Kinetics of Hydrogels

Theoretical Background

Rheology is the study of flow and deformation of materials under the influence of an external force or stress. The combination of stress, strain, and shear behavior forms the basis of rheology and are used to ensure successful material processing, optimize product performance, gain insights into complex microstructures, and develop novel materials.

When an external force is applied to a material, it will experience an amount of stress or strain relative to both that force and to the internal molecular interactions of that material. Measuring the resulting stress and strain behavior allows us to measure parameters such as viscosity and modulus, two physical properties important when engineering materials.

Many materials have viscoelastic properties, meaning they display some aspects of elastic solids and some aspects of viscous liquids. Polymers like hydrogels display a little of both properties. They have an elastic element, rooted in entanglement, that makes them resist deformation and return to their original shapes. They also have a viscous element, rooted in chain flow. That viscous element means that, when we distort polymeric materials, they might not return to exactly the same form as when they started out.

With a Rheometer, viscosity measurements extend beyond the limits of a traditional viscometer, characterizing non-Newtonian behaviours like shear thinning, and yield stress of complex fluids. Oscillatory rheology measures viscoelasticity (storage modulus, loss modulus, tan delta) of materials ranging from low-viscosity fluids to stiff solids.

Whitcomb, K., & Chen, T. T.. *Gelation kinetics from rheological experiments*. TA Instruments. https://www.tainstruments.com/pdf/literature/RH103.pdf

Determining the Gelation Point of a Hydrogel

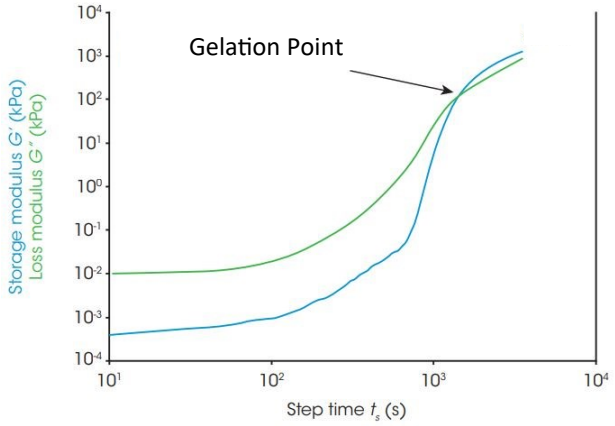
Sample: Hydrogel

Axial Force: 0 ± 0.1 N

Strain Rate: 0.05% (min torque 5 μNm)

Frequency: 1 Hz

In classical polymer rheology, the gelation point is defined as the point at which the storage modulus becomes larger than the loss modulus, indicating the fluid has transitioned from fluid flow like behaviour to solid elastic behaviour. The classical way of performing these experiments is by conducting multiple isothermal runs at various temperatures while keeping the frequency, strain, and axial force constant. The point at which the loss modulus crosses the storage modulus is the gel point and will be different at each temperature. The figure below depicts one such isothermal experiment conducted at 50 °C.

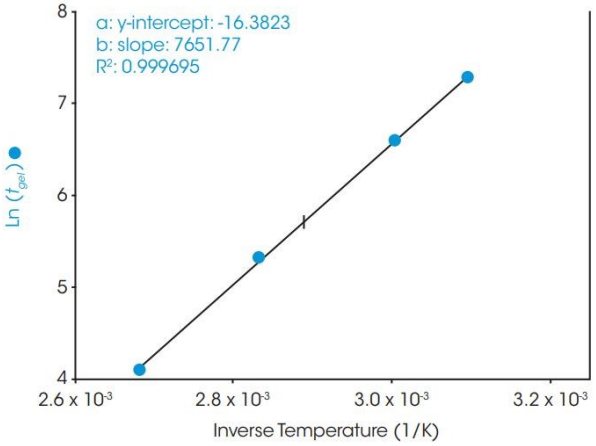


Using the Arrhenius equation (Eq.1) the rate of gelation can be estimated for each of the set temperatures. When the Arrhenius equation is linearized (Eq.2) and plotted, pre-exponential factor (A) and activation energy (Egel) can be estimated. This Information can be used to estimate the gelation time at any temperature.

$$t_{gel} = Ae^{\frac{E_{gel}}{RT}} \quad \textbf{(Eq. 1)}$$

$$\ln(t_{gel}) = \ln(A) + \frac{E_{gel}}{R}\left(\frac{1}{T}\right) \quad \textbf{(Eq. 2)}$$

The plotted linearized Arrhenius equation is displayed in the figure below. Where the



natural log of the gelation rate is plotted along the Y-axis and the inverse temperature in Kelvin is plotted along the X- axis. The slope of the line represents the ratio between the activation energy and the ideal gas constant. Now that the pre-exponential factor (A) is determined, as well as the activation energy (Egel), the temperature term (T) can be substituted to any temperature to calculate the gelation rate.

$$\ln(t_{gel}) = -16.38 + 7651\left(\frac{1}{303}\right)$$

$$t_{gel} = e^{-16.38+25.25} = 7115\ s$$