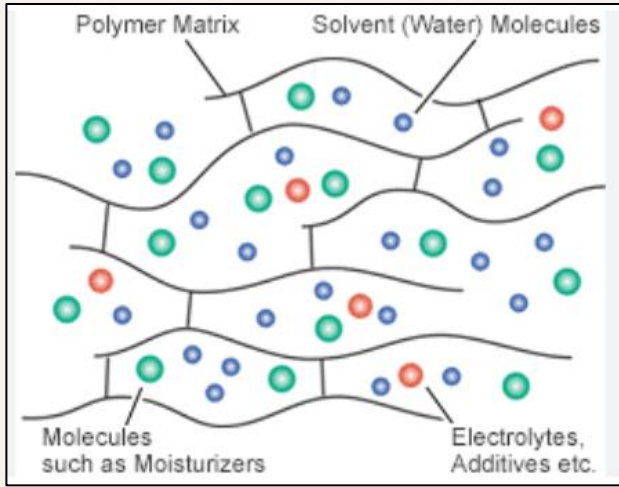


# Exploring Gelation in Biopolymer Solutions: A Study of Chemical Crosslinking Reaction and Determination of Gel Point

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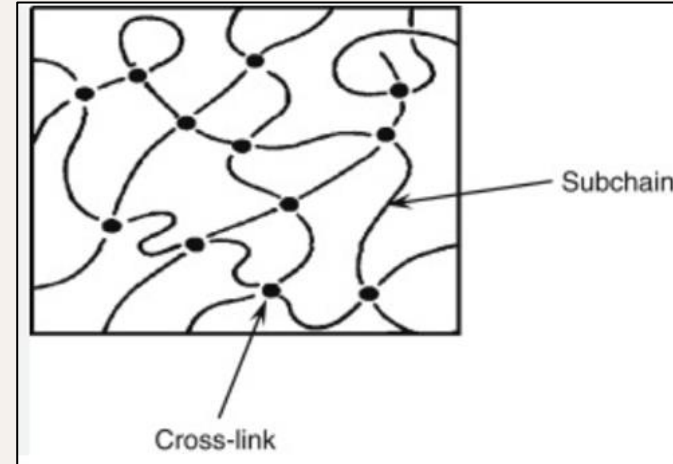


## What is a Gel?

- It is a type of material that exhibits a semi-solid or solid-like behavior, with properties similar to both liquids and solids.
- Gels typically have a three-dimensional network structure composed of particles or polymers dispersed in a liquid phase.
- This network structure is responsible for the gel's unique rheological properties.
- Gels are commonly found in jellies and custards, creams and lotions, and pharmaceuticals like topical gels and ointments

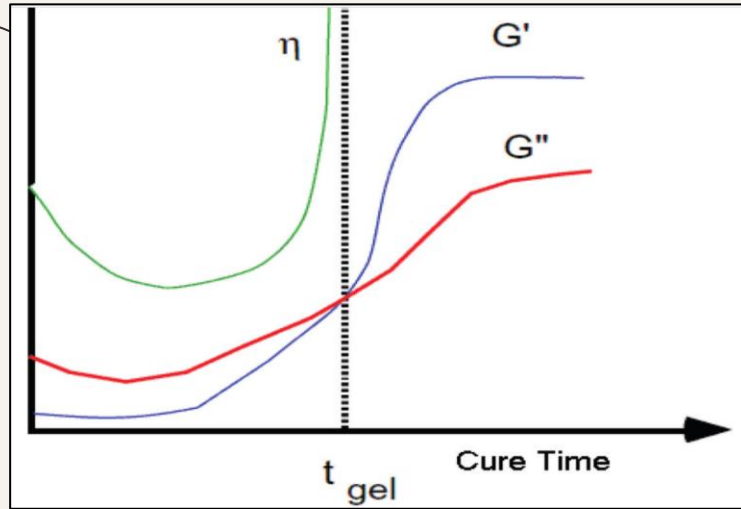
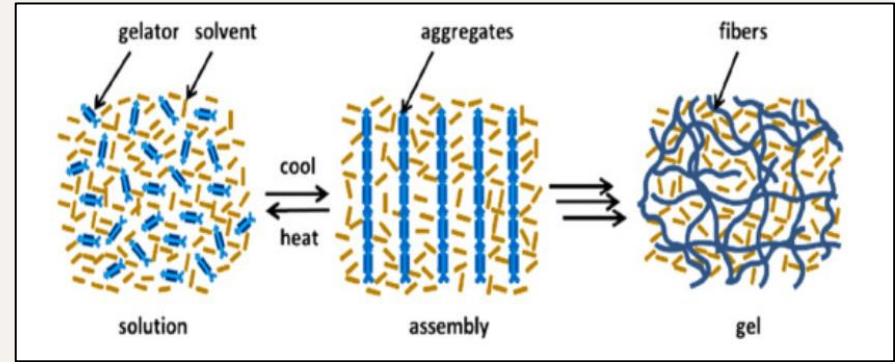
### Some unique properties of gels:

- **Viscoelasticity:** They can flow like liquids under shear stress, but they also return to their original shape when the stress is removed.
- **High Viscosity**
- **3D Network Structure**
- **Shear-Thinning Behavior:** Their viscosity decreases when subjected to shear stress which makes them easy to apply or dispense when needed.



## What is Gelation?

- It is the process in which the macromolecular polymer chains are connected together by chemical or physical bonds which leads to gradually larger branched soluble polymers.
- It's a **constant conversion event**.
- Temperature, concentration, pH, and the presence of gelling agents (thickeners or gelators) influence gelation.



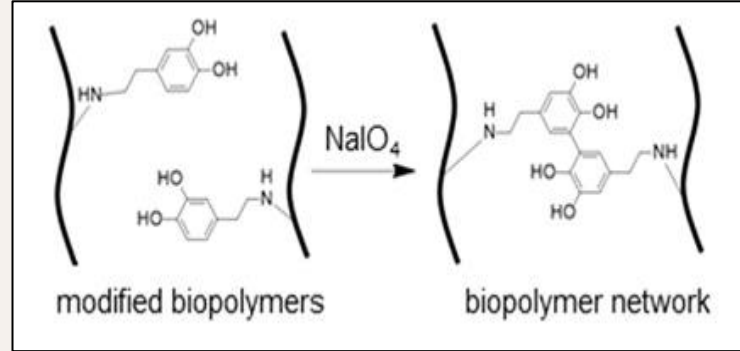
## What is gel point?

- At some point of polymer network formation, a single macroscopic molecule forms and this point is the gel point.
- At this point, the storage modulus of the fluid exceeds its loss modulus and the system loses its fluidity.
- It can be determined by observing a sudden rise in viscosity and transition to a more solid-like state.
- The  $t_{gel}$  shown in the given figure is the apparent gel point.

# Biopolymer Solutions & Cross Linking Reaction

## Biopolymers

- Polymers that are derived from natural sources, typically from living organisms.
- The biopolymer used in our experiment is gelatin.



## Cross-linkers

- Also known as gelators or gelling agents, these are molecules that contain two or more reactive ends capable of chemically attaching to specific functional groups on proteins or other molecules.

## Cross linking reaction

- It refers to the formation of covalent or ionic bonds between polymer chains or particles dispersed in a liquid, leading to the creation of a three-dimensional network structure.
- In our experiment, this occurs when the biopolymer (gelatin) is mixed with the cross-linker (EDC/NHS) and heated to 60° C.

# Methods to Measure gel point

## Viscosity Measurement:

- Measures changes in viscosity as gelation progresses.
- May not be as sensitive to small rheological changes.
- Provides limited information about the material's behavior at the gel point.

## Solubility Test:

- Qualitative assessment of solubility changes during gelation.
- Gelation process can be gradual and challenging to pinpoint.
- Lacks precision in identifying the gel point.

## Light Scattering and Spectroscopic Techniques:

- Suitable for characterizing molecular-level structural changes.
- Does not provide a direct measurement of the gel point.
- More focused on structural insights than rheological properties.



# Methods to Measure gel point

## RHEOMETRY

Rheometry is a powerful method for measuring the gel point and assessing the rheological properties of materials undergoing gelation. It offers several advantages over some other methods, but it may not be suitable for all situations.

- Provides quantitative data on mechanical properties ( $G'$ ,  $G''$ ).
- Real-time monitoring of the gelation process.
- High sensitivity for capturing subtle changes at the gel point.
- Allows for controlled testing conditions (e.g., temperature, shear rate).
- Versatile and adaptable to various materials and rheological tests.



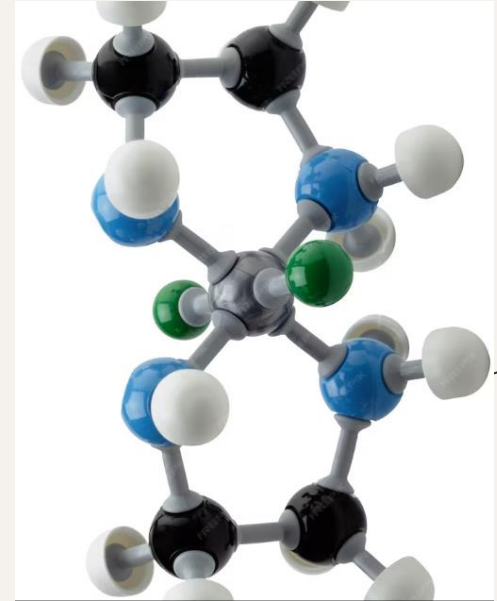
# Brief Overview of the key components used in the experiment -

## Gelatin Protein:

- Gelatin protein : Primary biopolymer in this experiment
- 10% protein solution : 1 gram of gelatin are dissolved in 10 ml of solution.

## EDC/NHS Crosslinker:

- Chemical crosslinker used in this experiment : Combination of EDC (1-Ethyl-3-(3-dimethylaminopropyl)carbodiimide) and NHS (N-Hydroxysuccinimide)
- EDC/NHS ratio : 60:30 millimolar
- Amount of EDC (molecular weight = 125) used :18.6 milligrams
- Amount of NHS (molecular weight = 115) used: 6.9 milligrams.
- Volume of crosslinking solution = 2 ml





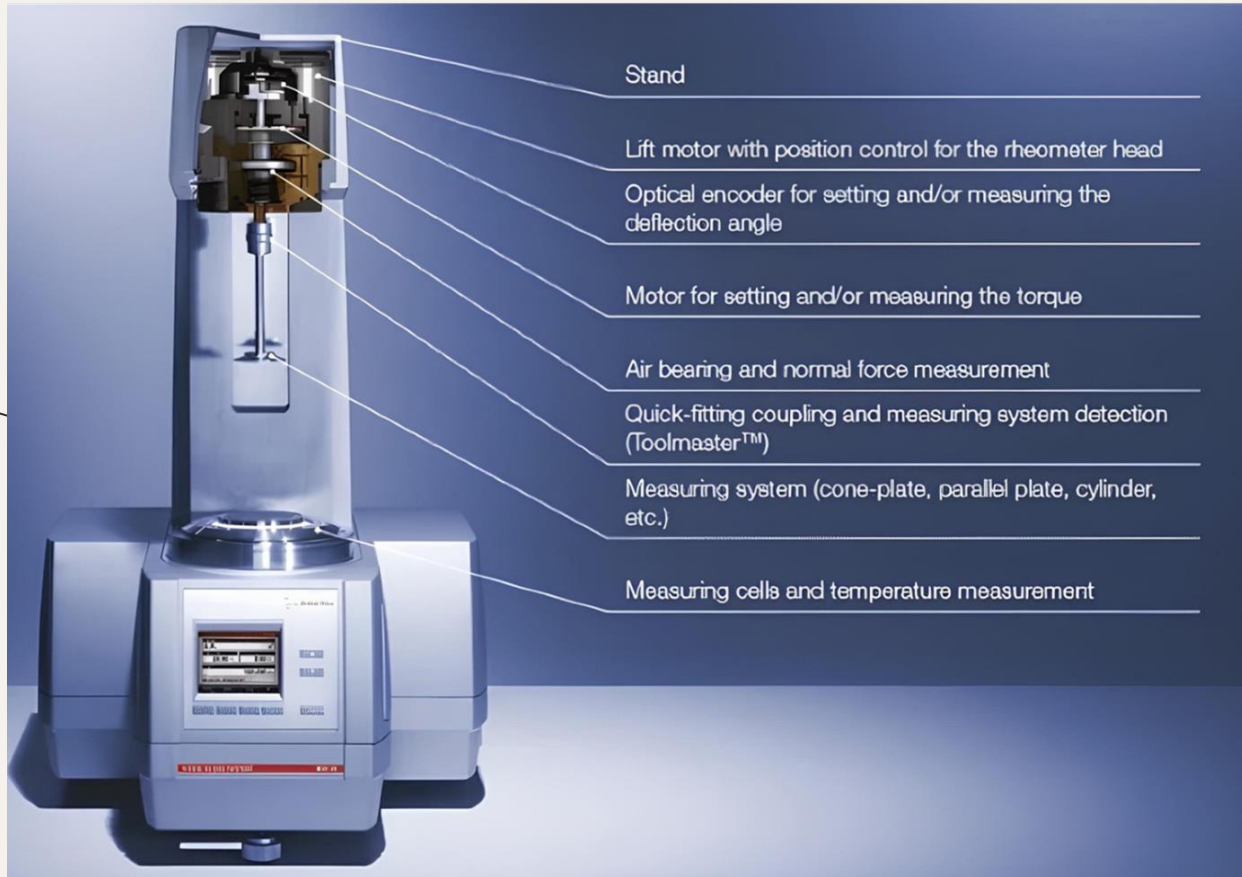
# Cyclic Frequency Sweep

## Why Rheology to measure gel point rather than traditional viscosity measurement method?

- Viscosity measurements are highly dependent on the applied frequency, making it challenging to obtain a frequency-independent gel point.
- The apparent gel point is introduced due to the frequency dependence of viscosity measurements.
- Traditional viscosity measures may yield different points as the "gel point" at different frequencies, making it apparent rather than an absolute measure.

## Identifying gel point using Rheometry

- Rheometry provides a robust method to measure the gel point in a **frequency-independent** manner.
- By examining the storage and loss moduli and their dependence on frequency, the apparent gel point can be identified.
- Gel point determination involves finding the frequency at which the storage and loss moduli exhibit identical frequency dependence, albeit with different constants ( $G' = Aw^n$ ,  $G'' = Bw^n$  where  $n$  is any exponent and  $A$  and  $B$  are constants).
- At this point, the loss factor becomes independent of frequency, signifying the gel point.
- At the true gel point, the loss factor becomes independent of frequency, reflecting the cessation of frequency dependence.



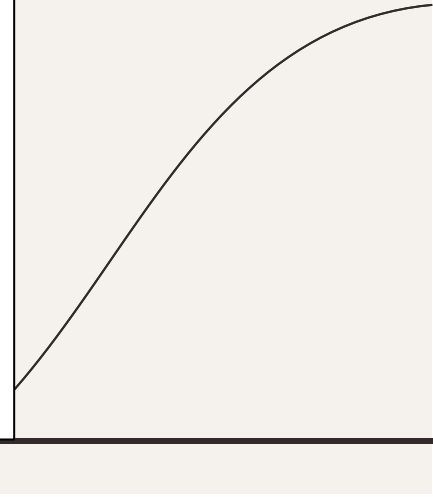
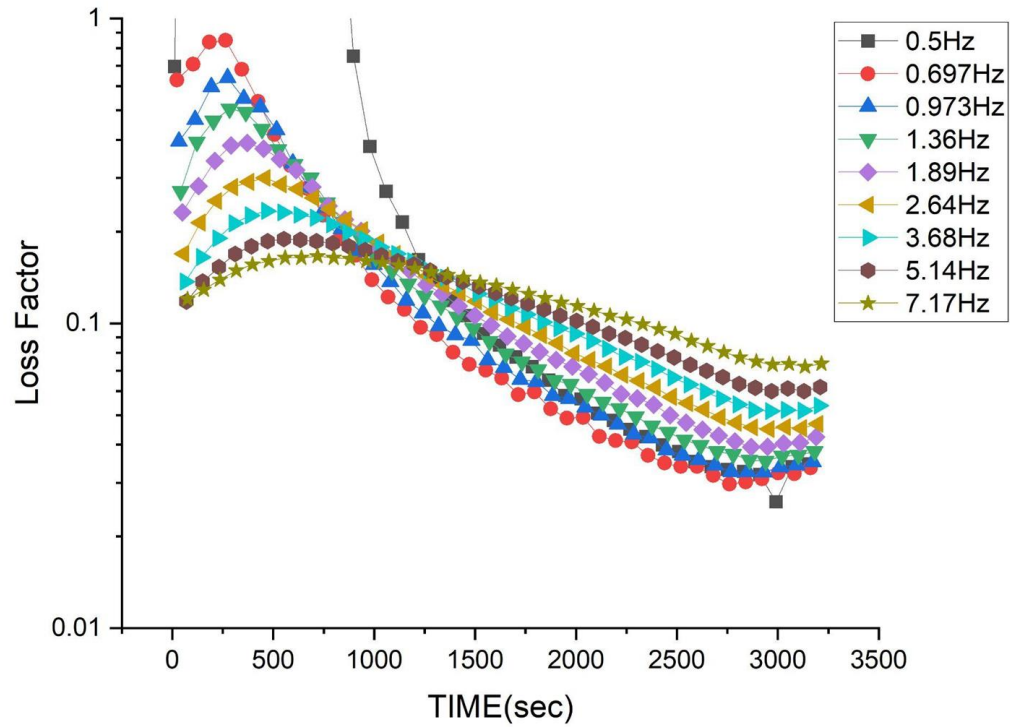
## Experimental Setup

- Parallel Plate Rheometer
- Temperature:  
maintained at 25°C
- Diameter of the Upper Plate:  
25mm
- Frequency Range:  
varying from 0.5 Hz to 10 Hz.
- Strain:  
constant strain of 0.5%
- Gap between the Plates:  
1mm

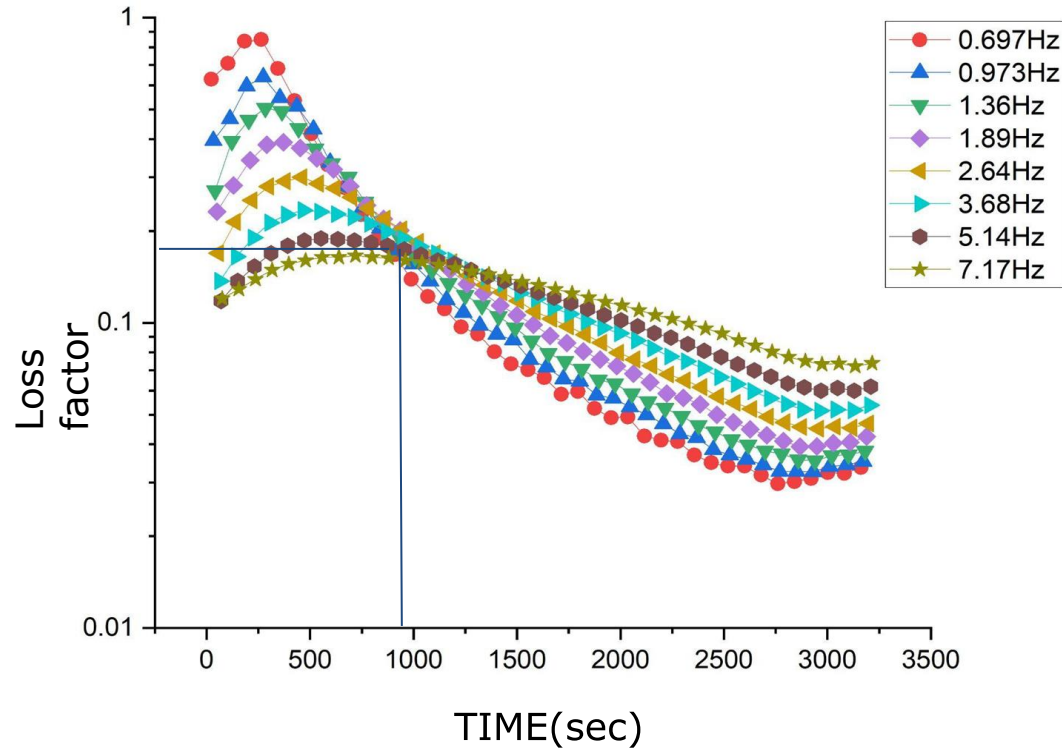
## Collected Data

Interval and data points:	1	10		
Interval data:	Time	Frequency	Loss Factor	Shear Strain
	[s]	[Hz]	[1]	[%]
	12	0.5	0.696	0.493
	22.89	0.697	0.628	0.499
	32.77	0.973	0.396	0.5
	41.73	1.36	0.271	0.5
	49.86	1.89	0.231	0.5
	57.24	2.64	0.169	0.5
	63.93	3.68	0.137	0.5
	70.01	5.14	0.118	0.5
	75.52	7.17	0.12	0.499
	80.52	10	0.0873	0.497

## Plot



## Observations -



As per the graph, we obtained the gel point approximately after 900 seconds and the corresponding loss factor is approx. 0.19

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# Real Life Examples

## **1. Food Products:**

- **Jellies and Jams:** Fruit juices or purees can form gels when combined with gelling agents like pectin. The gelation process gives these products their characteristic texture and allows them to hold their shape.
- **Custards and Puddings:** Ingredients like eggs, milk, and starch can undergo gelation when heated, resulting in the formation of a semi-solid texture.

## **2. Pharmaceutical and Medical Applications:**

- **Gel Capsules:** Gelatin-based capsules used for drug delivery are designed to dissolve slowly in the body, releasing the medication gradually.
- **Topical Gels:** Many pharmaceutical and medical products, such as wound gels and ointments, use gelation to create a semi-solid consistency for easy application and prolonged contact with the skin.

## **3. Cosmetics and Personal Care Products:**

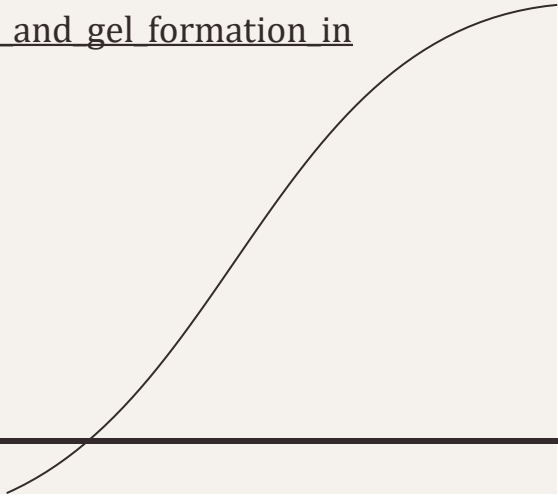
- **Hair Gel:** Styling products like hair gel contain gelling agents that help maintain the desired hair shape.
  - **Toothpaste:** Gels are used in toothpaste to provide a consistent texture and ensure the paste adheres to the toothbrush and effectively cleans teeth.
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# Summary



- In summary, our experiment aimed to determine the gel point in a protein using gelation as a key technique.
- At the lower frequency graph starts from top, shows more liquid-like character, but with increase in time, the value of loss factor decreases shows increase in the solid-like character, This transition point, known as the gel point, marks the critical concentration at which the protein undergoes a phase change from a sol to a gel.
- Understanding the gel point in proteins has important implications in various fields, such as food science, pharmaceuticals, and biotechnology, as discussed before.

## References

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  - <https://link.springer.com/article/10.1007/BF00701120>
  - <https://www.researchgate.net/publication/225859527> Aggregation and gel formation in biopolymer solutions
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THANK

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