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VISCOELASTIC CHARACTERISATION OF AQUEOUS POLYACRYLAMIDE SOLUTIONS: COMPREHENSIVE RHEOLOGICAL INVESTIGATION

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This study investigates the viscoelastic properties of an aqueous solution of polyacrylamide using the Anton Paar MCR 92 Rheometer to assess its storage ($G'(\omega)$) and loss ($G''(\omega)$) moduli across various frequencies. Initial findings indicate that at lower frequencies, polyacrylamide exhibits viscous behaviour with minimal elasticity, attributed to the coiled state of the polymer chains. As frequency increases, the material transitions to displaying significant elastic characteristics, suggesting an uncoiling of the polymer chains. At higher frequencies, both $G'(\omega)$ and $G''(\omega)$ rise, indicating more solid-like behaviour due to molecular alignment, which reduces viscosity and enhances elasticity. The molecular mass, measured using an Ubbelohde capillary viscometer, aligned with literature values, affirming the reliability of the experimental data. Additionally, the activation energy (E_a=88.95kJ/mol) was determined using the Time Temperature Superposition (TTS) principle and the Williams–Landel–Ferry (WLF) model, emphasising polyacrylamide's high thermal and mechanical stability, ideal for applications requiring robust performance in dynamic environments.

Keywords: *Dynamic viscosity, loss modulus, MCR 92, modulus of elasticity, rheological properties, viscosity.*

1. INTRODUCTION

Polymer substances are widely used in various industries and technological fields. For example, they are used in various coatings, as matrices (stabilizers) for drugs in the pharmaceutical industry, as moisture retainers in agriculture, mining industry, and other fields [1], [2]. A polymer molecule is a macromolecular compound, and it is significantly larger than the molecules of other types of substances. Due to the unique size and structure of its molecules, studying their physical properties is currently very important [3], [4].

Rheometry is an experimental technique used to determine the rheological properties of materials, studying the interrelationships between force, deformation, and time through flow and deformation changes. The rheological properties of liquids provide a lot of information about the material composition, similar to solid materials [3], [2]. To understand the rheological properties of substances in liquid form, such as solutions and suspensions, it is necessary to determine the flow and viscosity characteristics of the substance [5]. The flow and viscosity characteristics of substances are typically determined using a special rheometer through rheological testing [3], [6].

Rheological investigations on polymer melts serve two main purposes. On the one hand, they are used to support the processing of polymers; on the other, from rheological properties, insights can be obtained into some features of molecular structure. These relations can contribute to tailoring polymer materials for distinct applications, but additionally, they can be employed to qualitatively follow up the change of molecular parameters under external parameters like heat and mechanical deformation that may occur during various processing operations [7].

There are two main types of flow: transverse flow and shear flow. In the transverse flow, layers of fluid slide past each other side by side. Transverse flow occurs when an external transverse force acts on the fluid, causing it to move [8]. In the transverse flow, the fluid layers move over each other, with the upper layer moving faster than the lower layer. The topmost layer has the highest velocity, while the bottom layer appears to be stationary [5], [9].

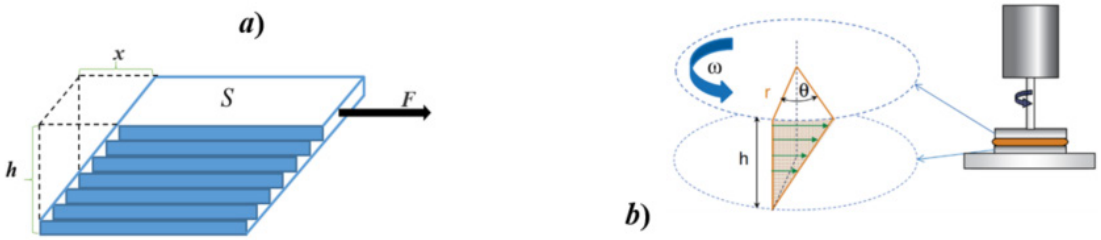

Fig. 1. a) 3D illustration showing the direction of force as fluid layers move over each other; b) schematic diagram of a controlled-stress rheometer in a parallel plate configuration [5], [8].

Under the influence of an external force, transverse stress σ is generated, and this quantity is defined as the force *F* acting on a unit area *S*, as shown in (Fig. 1a) [5], [8], [4].

$$\sigma = \frac{F}{S}. \quad (1)$$

When an external force is applied, the uppermost layer moves a certain distance *x*, while the lower layer remains stationary. Under these conditions, the shear gradient or shear deformation along the sample can be calculated using the equation $\gamma = x/h$ [8]. The rate of deformation change over time is governed by the differential equation $\dot{\gamma}=d\gamma/dt$ [8], [10], [11].

Friction between the fluid layers results in a decrease in both the fluid velocity and its kinetic energy. This loss is primarily attributed to the internal or dynamic viscosity, η of the fluid [8], [12].

The relationship between transverse stress and shear deformation rate is mediated by the viscosity of the fluid, expressed quantitatively as $\eta=\sigma/\dot{\gamma}$. Notably, the viscosity of the fluid is pressure- and temperature-dependent, generally increasing with rising pressure and decreasing with falling temperature [8], [2].

In experimental setups where a solution or suspension is placed between two horizontal plates with the upper plate rotating at a cyclic frequency ω, it is possible to ascertain the coefficient of internal friction by observing the movement of the fluid layers as depicted in Fig. 1b. However, when solutions contain particles of various sizes, such as nanoparticles, the parallel plate method may yield inaccurate results. In such instances, alternative rheometer configurations, such as cylindrical or other specialised types, should be employed due to their efficacy in handling high-frequency particle orientations [10], [12].

Various models have been proposed by scientists to elucidate the stress-strain relationships in materials subjected to external forces, significantly contributing to our understanding of material viscosity [10], [13]. For solid bodies, such as springs, the deformation under applied stress is described by Hooke's law, represented $\sigma = G\gamma$, where $\gamma=(l_0-l)/l_0$ denotes the relative elongation, with $l0$ being the initial length and l – the length after load application. Conversely, for fluids under pressure, the stress relationship adheres to Newton's law, expressed as $\sigma = \eta\dot{\gamma}$ [8], [10].

Maxwell introduced a model that describes the viscoelastic response of materials in a sequentially connected state, given by $\sigma = \sigma_0 e^{-\frac{t}{\theta}}$, where θ is the relaxation time of the system. Complementarily, the Kelvin–Voigt model, applicable to systems in parallel configurations, uses the equation $\gamma = \gamma_0\left(1-e^{-\frac{t}{\theta}}\right)$ (Fig. 2). While both the Maxwell and Kelvin–Voigt models provide similar mathematical forms for calculating relaxation times, they differ in their physical interpretations and implications. These models, however, present limitations in fully capturing the complex viscoelastic behaviour of polymers and other complex fluids. To address these shortcomings, Clarence Zener proposed the Standard Linear Solid model, which effectively integrates the principles of the Maxwell and Kelvin–Voigt models. This generalized Zener model offers a more comprehensive framework for understanding fluid behaviours under various stress conditions, significantly enhancing the predictive accuracy for the viscoelastic properties of complex materials [5], [8].

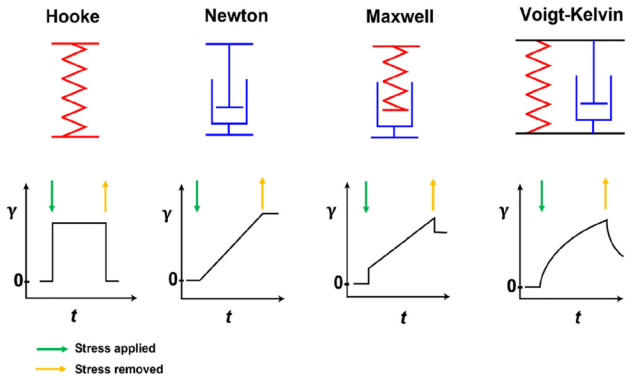

Fig. 2. Hooke, Newton, Maxwell and Voigt–Kelvin elements with the corresponding creep behaviour at constant stress. In a Maxwell element, the fluid can completely 'flow away', while in a Voigt–Kelvin element this is limited to the maximal extension of the spring [8].

When periodic external stress is applied to a solution, the physical parameters of the sample undergo dynamic changes. These changes in the physical properties are dictated by the frequency of the applied external stress and the inherent relaxation time of the system. Under conditions of periodic external loading, the storage modulus G' and loss modulus G'' of the material can be mathematically expressed as follows:

Storage module

$$G'(\omega) = G\frac{(\omega\theta)^2}{1+(\omega\theta)^{-1}}. \qquad (2)$$

Loss module

$$G''(\omega) = G\frac{\omega\theta}{1+(\omega\theta)^2}. \qquad (3)$$

These equations illustrate the frequency-dependent viscoelastic behaviour of the sample, where ω represents the angular frequency and θ denotes the relaxation time. Furthermore, when external stress is periodic, there is a phase lag between the stress and deformation responses. This phase difference is quantified by the ratio of the storage modulus to the loss modulus. The critical point at which the values of storage and loss moduli are equal ($\tan\delta$=1) is known as the gel point. At this juncture, occurring within the range $0<\omega<\infty$, the polymeric molecules within the material network interconnect extensively, culminating in the formation of a structure with an infinitely large molecular mass, indicative of a gel state (Fig. 3) [8].

By utilising the data obtained from the storage modulus (G') and the loss modulus (G''), one can determine the activation energy E_α and evaluate the stability of the sample. Analysing the frequency dependence of these moduli allows for the estimation of activation energy through the examination of viscoelastic properties as they vary with temperature and frequency [8], [11].

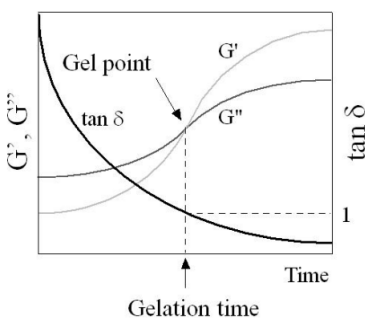

Fig. 3. Schematic representation of dynamic viscoelastic properties of gelation system as a function of reaction time [8].

An important rheological quantity derived from dynamic mechanical analysis is the plateau modulus G^0_N, which is defined as the frequency-independent storage modulus. Employing the theory of rubber elasticity, the entanglement molar mass M_e corresponding to the molar mass of polymer strands between the crosslinks in rubbery materials can be calculated as follows [7], [10]:

$$G_N^0 = \frac{4}{5}\frac{\rho RT}{M_e}, \quad (4)$$

where ρ represents the density, T – the absolute temperature, and R – the universal gas constant. While alternative prefactors are occasionally cited in the literature, for thermoplastic materials exhibiting a narrowly distributed polymer with polydispersity indices M_ω/M_n close to 1, a distinct plateau is typically observed [7]. The frequency dependency of $G'(\omega)$ holds particular analytical relevance for particle-filled polymer melts, as it provides insights into the conditions under which network formation may occur [7], [10].

2. EXPERIMENTAL PART

2.1. Materials

Polyacrylamide (PAA), a constituent of the polyelectrolyte complex, is produced at the Navoi Chemical Plant as per TSh 6.1-00203849-64:1997. This polymer is synthesized through the polymerization of acrylamide, incorporating nitrogen into its molecular structure, resulting in a linearly branched framework as illustrated in Fig. 4 [1], [14].

Polyacrylamide is a colourless, amorphous solid at room temperature (295–297 K) with a density of approximately 1.302 g/cm³. It exhibits excellent thermal stability with a decomposition temperature near 463 K. As a polyelectrolyte possessing hygroscopic and nontoxic properties, polyacrylamide forms a soft gel upon dissolution in distilled water at neutral pH (Fig. 4). This property is particularly valuable for various industrial applications due to its benign nature and effective solubility [15], [16].

The average molecular weight of polyacrylamide has been determined to be approximately $3.2 \cdot 10^6$ *g/mol* using an Ubbelohde viscometer in an aqueous medium at 25 °C. Furthermore, the dynamic viscosity was quantitatively analysed as a function of concentration employing the Huggins equation, as shown in Fig. 5 [14], [17], [9].

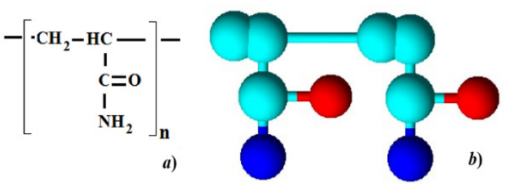

Fig. 4. Chemical formula (*a*) and model structure of PAA (*b*) [15], [4].

2.2. Sample Preparation

The research sample of polyacrylamide was prepared under controlled conditions at a room temperature (25 °C). Due to the slow dissolution rate of polyacrylamide in water and to ensure thorough mixing, 8 % concentration of the sample was fully dissolved using a specialised dispenser, facilitating uniform consistency.

2.3. Research Equipment

The investigations were conducted with the Anton Paar MCR 92 (Modular Compact Rheometer), one of the most advanced and sensitive rheometers available. The MCR 92 is engineered for high sensitivity, supporting continuous operation for periods less than 24 *hours*. It generates rotational and oscillatory torque ranging from 1 to 125 mN·m, with angular velocity spanning from 10^{-4} to 157 *rad/s*, and operating speeds (CSS/CSR) from 0.001 to 1500 *rpm*. This instrument is designed to function over a broad temperature spectrum, from -50 °C to +400 °C, accommodating various experimental conditions.

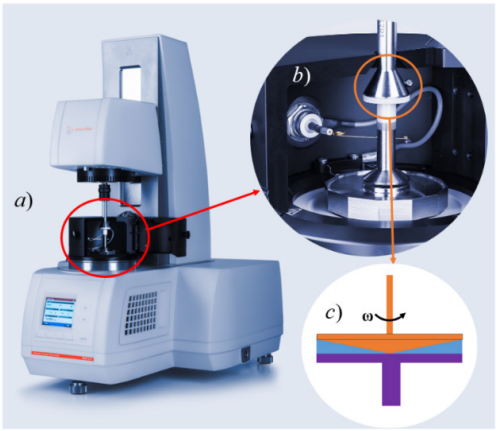

Fig. 6. Anton Paar MCR 92 (Modular Compact Rheometer): a) Full view of the device: This panel offers a complete visual representation of the Anton Paar MCR 92, showcasing its external structure and layout, b) Sample placement area: Detailed view of the area where samples are loaded into the rheometer, highlighting specific components that facilitate sample testing, c) Schematic view of the internal sample placement area: This diagram provides a detailed schematic of the internal mechanics and structure of the sample placement section, offering insight into the operational framework of the device.

For rheological assessments, the sample was placed between relatively flat, slightly conical plates, ensuring minimal deformation and optimal contact. Throughout the rheological evaluations, the temperature of the sample was meticulously controlled and maintained at 25 ± 0.5 °C (see Fig. 6).

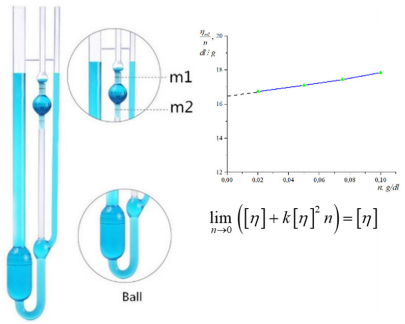

Fig. 5. Determination of the molecular weight of polyacrylamide via an ubbelohde viscometer.

When a periodic stress is applied to a sample, a phase difference between stress and deformation emerges. This phase lag in the polymer solution is quantitatively determined by the ratio *tan*δ, which is the quotient of the loss modulus G″ and the storage modulus G′ [5], [8]:

$$\tan\delta = \frac{G''}{G'}. \tag{5}$$

By analysing the phase difference between deformation and stress in the polymer solution, the molecular orientation within the solution and the material's response to external stimuli can be elucidated. This analysis assists in understanding how the molecules in the solution react to external influences and contributes to evaluating the stability of the material's deformation under such conditions.

The activation energy E_α is critical for understanding the temperature and frequency dependence of material behaviour. To compute E_α, the Williams–Landel–Ferry (WLF) method and the Arrhenius equation for viscosity were employed. The shift factor a_T, indicative of changes in viscosity with temperature, was plotted as a function of the inverse of temperature ($1/T$), and calculated using the equation [18], [19]:

$$a_T = \exp\left[-\frac{C_1\left(T-T_0\right)}{C_2+\left(T-T_0\right)}\right], \tag{6}$$

where C_1 *and* C_2 are empirical constants derived from experimental data, R denotes the universal gas constant, and T_0 is the reference temperature. These calculations allow for the derivation of the activation energy E_α, essential for predicting the material's performance under varied environmental conditions [18], [19].

3. RESULTS AND DISCUSSION

The rheological properties of an 8 % aqueous solution of polyacrylamide were systematically evaluated using the Anton Paar MCR 92 rheometer, which assessed the frequency-dependent behaviour of the storage modulus $G'(\omega)$ and loss modulus $G''(\omega)$. The analysis covered a broad frequency spectrum, providing insights into the viscoelastic characteristics of the polymer.

Low-Frequency Behaviour. At lower frequencies, ranging from 0.1 to 1 rad/s, the storage modulus $G'(\omega)$ is observed to be relatively low, suggesting that the material behaves more like a viscous fluid in this range. Conversely, the loss modulus $G''(\omega)$, which indicates energy dissipation within the system, is significantly higher, reflecting the dominant viscous characteristics of the polyacrylamide solution.

Intermediate-Frequency Behaviour. As the frequency increases to an intermediate range of 1 to 10 rad/s, there is a notable transition in the rheological behaviour. The storage modulus $G'(\omega)$ rises sharply, indicating an increase in the elastic properties of the solution. During this range, $G'(\omega)$ begins to converge with $G''(\omega)$, suggesting a balanced viscoelastic state where the elastic and viscous components contribute equally to the material's response.

High-Frequency Behaviour. At higher frequencies, between 10 and 100 rad/s, both moduli, $G'(\omega)$ and $G''(\omega)$, increase steadily. This trend illustrates that the polyacrylamide solution starts to exhibit more solid-like characteristics, with both the storage and loss moduli reflecting a strong elastic

response. This behaviour is indicative of the polymer chains' alignment and entanglement under rapid deformation conditions.

These detailed findings, illustrated in Fig. 7, underscore the complex frequency-dependent viscoelastic behaviour of polyacrylamide solutions and highlight the utility of the Anton Paar MCR 92 rheometer in capturing these nuances.

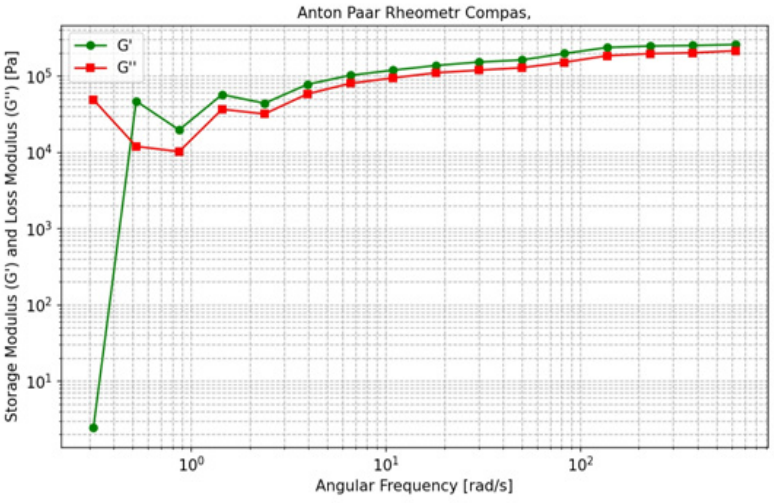

Fig. 7. Frequency response of storage and loss moduli for polyacrylamide: This graph presents the frequency-dependent behaviour of the storage modulus $G'(\omega)$ and the loss modulus $G''(\omega)$ for an 8 % aqueous solution of polyacrylamide, illustrating significant transitions across the spectrum from viscous to elastic properties.

For the 8 % polyacrylamide solution at 25 °C, the plateau modulus, G^0_N, was found to be approximately 28 MPa. This relatively moderate value suggests that the polymer chains are only lightly branched and exhibit minimal elastic properties under the tested conditions. Such characteristics indicate that while the polymer solution can stretch, it lacks extensive cross-linking that typically contributes to higher elasticity.

When subjected to periodic external stress, the deformation and stress responses of the polyacrylamide solution were found to oscillate in distinct phases. The analysis involved calculating the phase difference between stress and deformation as a function of angular velocity. Results from this investigation, as depicted in Fig. 8, show that the storage modulus $G'(\omega)$ and the loss modulus $G''(\omega)$ approach each other at higher frequencies. However, no point was reached where the moduli values converged ($\tan\delta$=1), indicating the absence of a definitive gel point under the conditions tested. This lack of a gel point suggests that at the frequencies analysed, the polyacrylamide molecules do not achieve a state of critical entanglement or cross-linking required for gel formation, typically characterised by the equilibration of energy storage and dissipation.

This nuanced understanding of the polyacrylamide behaviour under dynamic conditions helps clarify the material's viscoelastic properties and its potential applications where elasticity and deformation response are crucial factors.

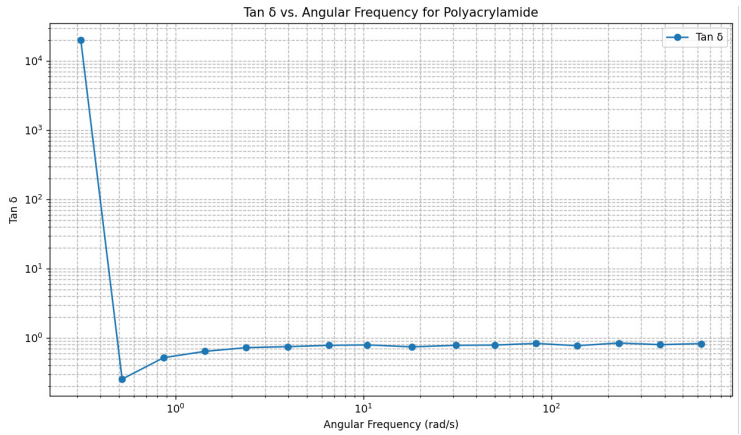

Fig. 8. Relationship between *tan*δ and angular frequency for polyacrylamide.

The analysis depicted in the graph clearly demonstrates that at lower angular velocities, the tanδ value, a ratio indicative of the viscoelastic nature of the material, is considerably high. This suggests a predominant viscous behaviour in the polyacrylamide solution. As the angular velocity increases, the *tan*δ value decreases sharply, reaching a minimum at around 8 *rad*/*s*, indicative of a transition in the material's behaviour from predominantly viscous to more elastic. Beyond this point, the *tan*δ value slightly increases and then stabilises, indicating that the material maintains consistent elastic properties at higher frequencies.

The viscoelastic response of polyacrylamide was further analysed to determine the activation energy E_α using the Williams–Landel–Ferry (WLF) method, which is well-suited for polymers. The Time Temperature Superposition (TTS) principle was employed to plot the logarithm of the shift factor (a_T) as a function of the inverse of temperature ($1/T$). This plot, which adheres to the Arrhenius-type behaviour, illustrates the temperature dependence of the material's dynamics, facilitating the calculation of E_α from the viscosity data.

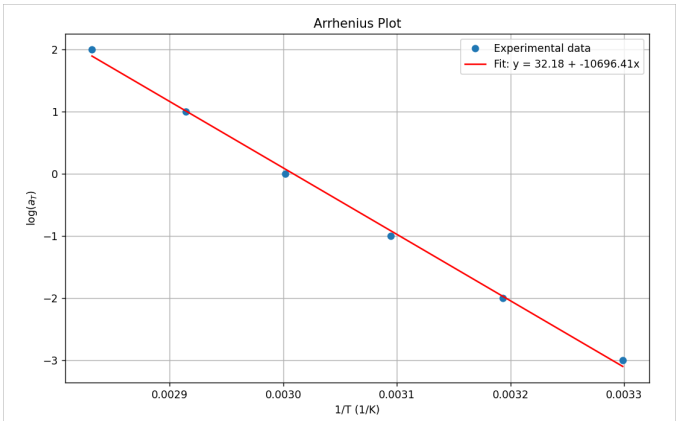

Fig. 9. Linear relationship of the logarithm of the shift factor with inverse temperature for polyacrylamide: This plot displays the linear correlation between the logarithmic shift factor log(a_T) and the inverse of temperature ($1/T$), adhering to the Arrhenius model, which is instrumental in calculating the activation energy of the polyacrylamide solution.

$$a_T = \exp\left[\frac{E_\alpha}{R}\left(\frac{1}{T}-\frac{1}{T_0}\right)\right]. \quad (6)$$

The calculated activation energy of E_α=88.95 *kJ*/*mol* reflects the material's resistance to flow under applied stress and suggests a strong temperature dependence of the molecular mobility. High activation energy signifies that considerable energy is required to mobilize the polymer chains, thus indicating robustness against external physical influences and potential for applications requiring high performance under dynamic conditions.

This comprehensive analysis not only confirms the strong viscoelastic properties of polyacrylamide but also enhances our understanding of its behaviour under varying thermal and mechanical conditions, as shown in Fig. 9. This understanding is crucial for optimising industrial processes where polyacrylamide is used under diverse environmental conditions.

4. CONCLUSIONS

In this comprehensive study, the rheological properties of an 8 % aqueous solution of polyacrylamide have been explored across a spectrum of frequencies to understand the viscoelastic behavior of the polymer. Measurements of the storage modulus ($G'(\omega)$) and the loss modulus ($G''(\omega)$) have revealed significant variations in response to changing frequencies, reflecting the complex interplay between the material's structural characteristics and its mechanical responses.

Low Frequency Dynamics: At lower frequencies, the solution predominantly exhibited viscous behaviour with minimal elastic properties. This is likely due to the polymer chains remaining in a highly coiled state, which restricts their ability to store elastic energy. The higher loss modulus compared to the storage modulus at these frequencies underscores the material's tendency to dissipate energy rather than store it.

Transition to Viscoelasticity: As the frequency increased to intermediate levels, a notable transition occurred where the material demonstrated enhanced elasticity. This shift is attributed to the extension and possible partial uncoiling of the polymer chains, allowing the material to store more energy and behave more like an elastic body. The equivalence of the storage and loss moduli in this range signifies a balanced viscoelastic state where the material exhibits both solid and liquid characteristics.

High Frequency Solid-like Behaviour: At higher frequencies, polyacrylamide displayed characteristics akin to solid substances, evidenced by an increase in both moduli. This behaviour suggests that the molecular chains align more rigidly under rapid oscillatory stress, resulting in decreased viscosity and enhanced elasticity. The alignment leads to more pronounced solid-like behaviour, with the material effectively responding to external stresses with significant elastic recovery.

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