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RHEOLOGICAL PROPERTIES OF POLYVINYLACETAL COLLOIDAL SYSTEMS

This study investigates the dynamic viscosity and thixotropic recovery of polyvinylacetal colloidal systems.

Rotational viscometry was employed to determine the dependence of dynamic viscosity (η) on polymer concentration and shear stress (τ). The rheological properties of the polymers examined exhibited a common pattern: viscosity increases with concentration as a result of intensified intermolecular interactions.

Detailed analysis of flow curves for polyvinyl butyral solutions revealed pseudoplastic behavior at low shear stresses. With increasing shear stress, a transition to Newtonian flow was observed, where the dynamic viscosity reached a minimum and stabilized. This effect is attributed to the breakdown of macromolecular structural conglomerates, which form in the resting state through hydrogen bonding with water molecules in the dispersion medium.

A key finding is the demonstrated ability of polyvinylacetal gels to rapidly restore their structure through thixotropic recovery. The degree of thixotropic recovery (α), calculated as the ratio of effective viscosities during reverse and forward

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measurement runs, increased substantially with polymer concentration, reaching 94.58%. This property ensures that, following mechanical disruption during application, the polymer layer quickly regains its structural integrity and adhesive performance, which is of practical significance.

The obtained results confirm the high efficiency of polyvinylacetals as adhesives and highlight the prospects for further research aimed at optimizing their compositions, exploring the influence of external factors, and elucidating adhesion mechanisms at the molecular level. Such efforts may lead to the development of novel, more advanced materials for the textile industry.

***Key words**: dynamic viscosity, shear stress, rotational viscometry, polyvinylacetal, polyvinyl butyral, polyvinyl ethylal, polyvinyl formal, rheological curve, thixotropic recovery.*

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РЕОЛОГІЧНІ ВЛАСТИВОСТІ КОЛОЇДНИХ СИСТЕМ ПОЛІВІНІЛАЦЕТАЛІВ

У цій праці автори досліджували динамічну в'язкість та тиксотропне відновлення колоїдних систем полівінілацеталів.

Для досягнення поставленої мети були застосовані методи ротаційної віскозиметрії, що дозволило визначити залежність динамічної в'язкості (η) від концентрації полімеру та дотичного напруження зсуву (τ). Встановлено, що реологічні властивості досліджуваних полімерів мають схожий характер: в'язкість зростає зі збільшенням концентрації, що є наслідком посилення міжмолекулярної взаємодії.

Детальне вивчення кривих течії для розчинів полівінілбутиралю продемонструвало їхню псевдопластичну поведінку при малих напруженнях зсуву. Зі зростанням напруження зсуву спостерігається перехід до ньютонів-

ської течії, коли динамічна в’язкість набуває мінімального і стабільного значення. Цей ефект пояснюється руйнуванням структурних конгломератів макромолекул, які утворюються у стані спокою завдяки водневим зв’язкам з молекулами дисперсійного середовища (води).

Ключовим результатом є виявлення здатності гелів полівінілацеталів до швидкого тиксотропного відновлення їхньої структури. Ступінь тиксотропного відновлення (α), розрахований як відношення сум ефективних в’язкостей при зворотному і прямому ходах вимірювання, значно зростає зі збільшенням концентрації полімеру, досягаючи 94,58%. Ця здатність гарантує, що після механічного руйнування структури в процесі нанесення, полімерний шар швидко відновлює свою міцність та адгезійні властивості, що є важливим.

Отримані результати підтверджують високу ефективність полівінілацеталів як адгезивів і вказують на перспективи подальших досліджень, спрямованих на оптимізацію їхніх композицій, вивчення впливу зовнішніх факторів та механізмів адгезії на молекулярному рівні для розробки нових, більш досконалих матеріалів для текстильної промисловості.

Ключові слова: *динамічна в’язкість, дотичне напруження зсуву, метод ротаційної віскозиметрії, полівінілацеталь, полівінілбутираль, полівінілетилаль, полівінілформаль, реологічна крива, тиксотропне відновлення.*

One important area of polymer solution application is their use as adhesives in the preparatory stages of weaving production, particularly for sizing warp yarns. The effectiveness of the applied polymer solution on the yarn determines the quality of all subsequent stages of textile manufacturing. Adhesives are predominantly based on aqueous solutions of natural or synthetic polymers and their various blends; among these, polyvinylacetal solutions have proven to be particularly effective. A key property of polymer solutions is their dynamic viscosity, which depends on temperature, concentration, and the molecular weight of the film-forming polymer. Consequently, the study of the rheological properties of polyvinylacetals is of high practical relevance.

The rheology of polymer solutions reflects the combined effects of intermolecular interactions between macromolecules. Due to their relatively large geometric dimensions, polymer macromolecules in solution undergo a variety of processes under flow conditions – either spontaneous or externally induced. In solution, macromolecular aggregates continuously form and disintegrate (Mezger, 2011).

When a highly diluted polymer solution is examined, its viscosity is nearly identical to that of the solvent. However, even at low concentrations, under shear stress the viscosity of the system increases, being directly proportional to the polymer’s volume fraction.

Upon extrapolating reduced viscosity to zero concentration, which corresponds to infinite dilution, the contribution of intermolecular interactions to the hydrodynamic behavior becomes negligible. In this regime, viscosity is determined solely by the intrinsic properties of the macromolecules and their interaction with the solvent (Sangroniz et al., 2023). Colloidal solutions at very low concentrations behave as Newtonian fluids, albeit with higher viscosity. At higher concentrations, however, mutual orientation, structuring and breakdown of macromolecules occur, accompanied by the transformation from laminar to turbulent flow. The properties of such highly structured dispersions depend on both the number of intermolecular interactions per unit volume and the strength of interparticle bonding.

The presence of water molecules adsorbed on polymer fragments is critical to the behavior of polymer solutions. The structure and stability of the resulting macromolecular conglomerates depend on the intrinsic molecular properties, the specifics of their interaction in aqueous medium, and the influence of external forces. These conglomerates determine the amount of water they retain; when they span the entire volume of the colloidal dispersion, only a small fraction of free water remains. Under such conditions, low shear stresses are insufficient to destabilize the system, and the viscosity of the polymer gel approaches its limiting value (Xu et al., 2024).

Polyvinylacetal macromolecules possess extended surfaces capable of retaining large volumes of water. As a result, the amount of free solvent decreases while the fraction of macromolecular conglomerates increases, forming a quasistatic structure. When subjected to sufficiently high shear stress to disrupt these conglomerates, flow begins. This point corresponds to the critical viscosity and shear stress. Beyond this threshold, the viscosity of the polymer gel decreases and gradually approaches a minimum, where it becomes independent of shear stress and the solution behaves as a Newtonian fluid. The decrease in dynamic viscosity is associated with the progressive release of water molecules during flow and the effective reduction of polymer concentration.

However, once shear stress is removed, the structure of macromolecular conglomerates gradually rebuilds over time, leading to an increase in viscosity through thixotropic recovery. This property is particularly important for the use of polyvinylacetals as adhesives in yarn sizing (Wang et al., 2024).

The aim of this study is to investigate the viscous-rheological properties of polyvinylacetal colloidal systems. The objectives include analyzing the structure and dynamic viscosity of aqueous polyvinylacetal solutions, constructing flow curves, and determining the degree of thixotropic recovery of polyvinylacetal gels. Rotational viscometry was employed for this purpose.

The dynamic viscosity η (Pa·s) of polyvinylacetal solutions with a low degree of acetalization – polyvinyl formal (PVF), polyvinyl ethylal (PVE), and polyvinyl butyral (PVB) – was calculated based on shear stress τ (Pa), measured using a Rheotest-2 rotational viscometer.

We established the dependence of dynamic viscosity for PVF, PVE, and PVB solutions on polymer concentration. Solutions were prepared as follows: water was poured into a beaker, polymer was added while stirring for 15...20 minutes, followed by heating to approximately 50°C with continuous agitation until complete dissolution was achieved.

The rheological properties of PVF, PVE, and PVB gels (Fig. 1) demonstrated a common trend: viscosity increases with increasing polymer concentration. The higher the concentration, the stronger the intermolecular interactions (Kalyanasundaram et al., 2000).

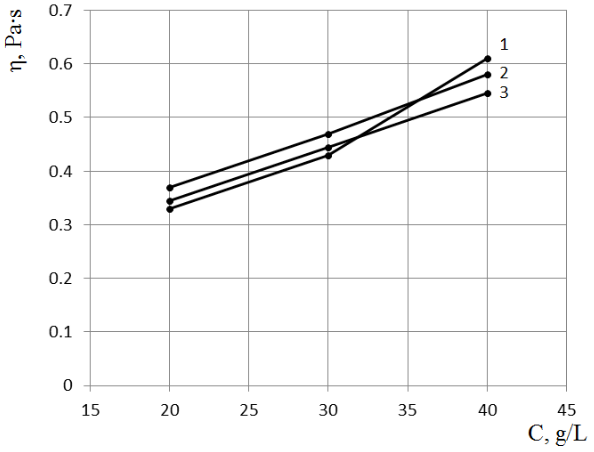

Fig. 1. Dependence of dynamic viscosity on the concentration of aqueous solutions: 1 – PVF; 2 – PVB; 3 – PVE

Subsequently, PVB solutions were investigated at concentrations (g/L): 1) 20, 2) 25, 3) 30, 4) 35, and 5) 40. In the previous experiment, PVB exhibited similar behavior; however, this polymer is the most readily soluble in water. Figures 2–5 present the plots of the following dependencies: $\eta = f(\tau)$, $\ln \eta = f(\ln \tau)$, $\gamma = f(\eta)$, and $\gamma = f(\tau)$. The viscosity values increase systematically with polymer concentration in the gel. The flow behavior indicates that at low shear stresses the system exhibits pseudoplastic characteristics, subsequently transitioning into Newtonian flow (Sangroniz et al., 2023). However, concentrated polymer solutions may behave as Bingham – Shvedov bodies. The corresponding equation describes this type of flow, where the strength of the undisturbed gel structure can be quantitatively recorded.

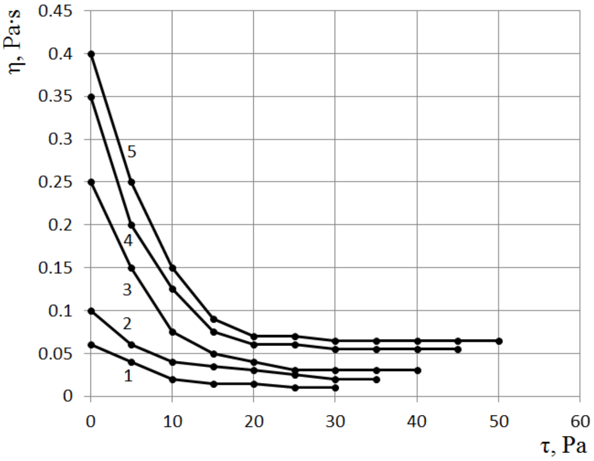

Fig. 2. Dependence of the dynamic viscosity of aqueous PVB solutions on shear stress

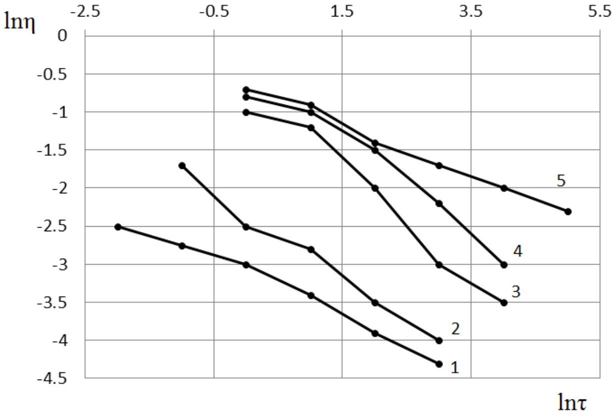

Fig. 3. Logarithmic dependence of the dynamic viscosity of aqueous PVB solutions on shear stress

Since we have identified the flow behavior as pseudoplastic, the Ostwald – de Waele equation describing it can be expressed as:

$$\tau = K\gamma^n \qquad (1)$$

where γ is the shear rate under steady-state flow, [s^{-1}];

K is the consistency coefficient;

n is the flow behavior index.

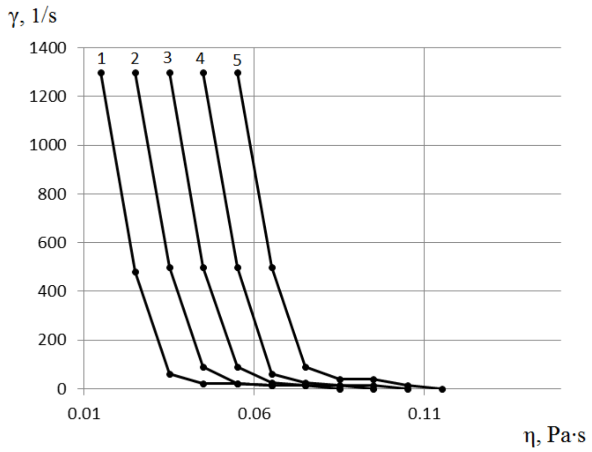

Fig. 4. Dependence of the shear rate on the dynamic viscosity of PVB solutions at various concentrations

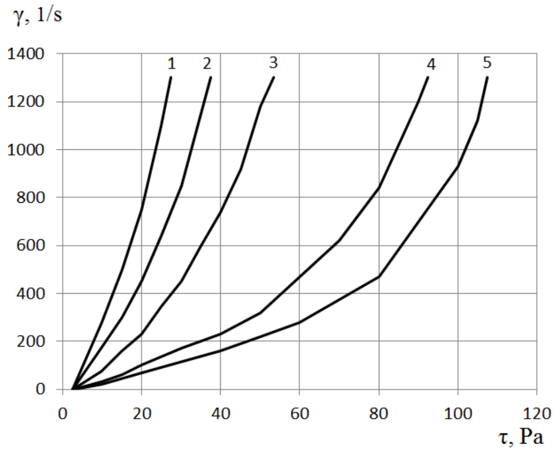

Fig. 5. Dependence of the shear rate on the shear stress of PVB solutions at various concentrations

At all concentrations, the flow behavior of PVB gels is similar; however, in every case, viscosity depends on polymer concentration. The power-law expression (1) describes pseudoplastic gels, where $\eta = f(\tau)$. Consequently, the obtained graphical dependencies $\ln \eta = f(\ln \tau)$ appear as lines approximating straight lines. As can be seen from all curves in Fig. 2, at certain shear stresses the dynamic viscosity of PVB gels reaches a minimal constant value and no longer depends on the applied force. In other words, at these levels of shear stress, and across all studied concentrations, PVB gels behave as Newtonian fluids (Farmer et al., 1990). At lower stresses, however, they exhibit pseudoplastic behavior.

Polymer solutions, which exist as gels in a resting state, form structural three-dimensional conglomerates that involve not only polymer macromolecules but also molecules of the dispersion medium, namely water. Within the internal structure of polymer macromolecules, water molecules establish hydrogen bonds in such a way that the resulting spatial conglomerate is highly stable at rest (Li-Ping et al., 2021). When shear forces are applied to such systems, this structure can be partially or completely disrupted. Nevertheless, colloidal spatial networks gradually rebuild their structure over time, undergoing thixotropic recovery.

PVB gels demonstrated a pronounced ability to restore their structure via thixotropy. The molecular conglomerates of PVB reorganize, with macromolecules aligning along the direction of shear stress during flow. The thixotropic recovery process occurs rapidly, indicating a high degree of structural stability of the PVB gel. Attempts to fix the strength of the system at the point of complete structural breakdown were unsuccessful, as the polymeric conglomerate began its recovery almost immediately.

The greater the PVB content in the studied gel, the higher the degree of thixotropic recovery. At such levels of structural self-restoration, the recovery of disrupted polymer conglomerates proceeds even at low shear rates. As a result, the dynamic viscosity of the gel increases, and this effect becomes more pronounced with higher polymer concentrations. The degree of thixotropic recovery of PVB gels was calculated as the ratio of the sum of effective viscosity values recorded during the reverse measurement run to those obtained during the forward run, as shown in Table 1 and Fig. 6.

The rheological properties of polyvinyl acetal (PVAce) gels, as vinyl polymers, exhibit characteristics analogous to polyvinyl alcohol, particularly their ability for thixotropic recovery (Bassan et al., 2023). During the dissolution of PVAce, the spatial configuration and molecular structure remain essentially unchanged. This assumption is supported by the fact that the gel is capable of structural recovery under small shear deformations, while simultaneously orienting its flow in the direction of applied stress. With increasing thixotropic recovery capacity, the quantitative and qualitative performance of the polymer as a film-forming agent improves, which is critical in preparatory stages of textile manufacturing.

Table 1

Dependence of the thixotropic recovery degree of PVB gel on its concentration

PVB Content, g/L	Degree of the Thixotropic Recovery, α, %
20	80.46
25	86.19
30	92.47
35	93.56
40	94.58

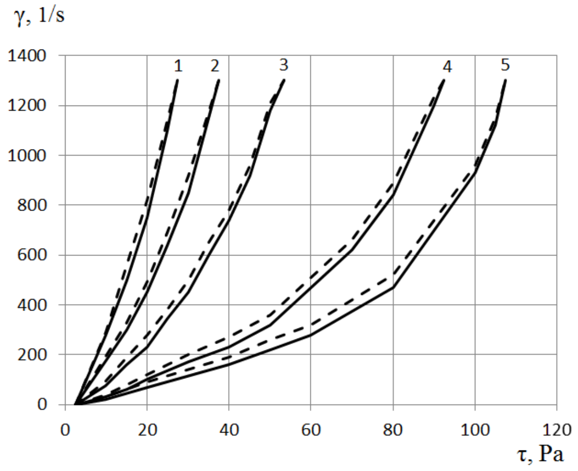

Fig. 6. Flow rheograms of PVB solutions at various concentrations. Solid line – forward run of the instrument, dashed line – reverse run

Experimental investigations of aqueous PVAce solutions confirmed their suitability as adhesives for textile applications. The dynamic viscosity of the solutions was found to correlate directly with polymer concentration. At low shear stresses, the solutions exhibit pseudoplastic behavior, while at higher stresses they transition toward Newtonian flow. This behavior can be attributed to the formation and disruption of supramolecular aggregates of macromolecules, incorporating water molecules bound through hydrogen interactions.

A key conclusion is the pronounced ability of polyvinyl acetal gels to undergo rapid thixotropic recovery, which is particularly advantageous in sizing processes. The degree of thixotropic recovery increases with polymer concentration, indicating the high stability of structural assemblies. This property ensures that, following mechanical impact (e.g., structural disruption during application), the polymeric film rapidly regains its strength and adhesive capacity, thus providing effective protection for warp yarns. Structural recovery occurs under relatively low shear deformation rates, positively influencing the quality and uniformity of the film-forming layer.

Considering the results obtained, further research may focus on optimizing the formulation of composite adhesives based on polyvinyl acetals, particularly through the incorporation of plasticizers or other modifiers. Promising directions include examining the effects of temperature and pH on rheological behavior and recovery kinetics, as well as investigating the adhesion mechanisms of these polymers to different textile fibers at the molecular level. Identifying optimal conditions for maximizing film strength would enable the development of novel, more efficient, and environmentally safe adhesives for textile industry applications (Tkachuk et al., 2025).

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