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Xanthan Gum Influence on Synthesis and Controlled Release from Sodium Acrylate Hydrogels

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Abstract

This study investigates the synthesis, swelling behavior, and ascorbic acid (AA) release kinetics of sodium acrylate (SA)-xanthan gum (XA) hydrogels. Hydrogels were synthesized with varying XA concentrations (7%, 5%, 2%, and 0%) using potassium persulfate (KPS) or 2,2-azobis(2-methylpropionamide) dihydrochloride (V50) as initiators. Swelling was evaluated in aqueous media at room temperature, and AA was used as a model bioactive compound for release studies. Results showed that XA concentration influences swelling and release. Hydrogels synthesized with KPS exhibited minimal swelling variation, whereas those with V50 displayed concentration-dependent swelling. AA diffusion coefficients decreased with increasing XA concentration in V50-based hydrogels, suggesting a more controlled release. Swelling was pH-dependent, reaching a maximum under neutral conditions due to carboxyl group ionization. AA release in KPS hydrogels followed Fickian diffusion. These findings highlight the potential of XA-based hydrogels for controlled release applications, allowing optimization through XA concentration and synthesis conditions.

Keywords

Ascorbic Acid, Controlled Release, Hydrogels, Sodium Acrylate, Xanthan Gum

1. Introduction

A hydrogel is a material composed of hydrophilic polymeric chains crosslinked in an aqueous medium, capable of forming three-dimensional networks that swell by absorbing and retaining water [1]. These properties have made hydrogels key materials for a wide range of applications, including controlled drug delivery, tissue engineering, optics, agriculture, and the food industry [2]-[5]. Smart hydrogels stand out for their ability to respond to specific stimuli, such as pH, temperature, or the presence of enzymes [6].

In biomedical applications, hydrogels offer significant advantages, including biocompatibility, biodegradability, and the ability to release drugs in a controlled manner at the required site and at the appropriate time. Additionally, their physical and chemical properties can be tailored to optimize performance according to therapeutic needs [7] [8]. These characteristics enable hydrogels to serve as platforms for cell growth, wound dressings, and advanced drug delivery systems [9].

The swelling capacity of hydrogels depends on factors such as the nature of the polymer, the degree of crosslinking, and their interaction with the solvent [10]. Previous studies have explored their response to stimuli such as pH, temperature, and ionic strength [11] [12]. In particular, ionic hydrogels can absorb aqueous solutions in large quantities, reaching up to hundreds of times their weight in water [13] [14]. This behavior, the degree of swelling (H), can be measured by the formula:

$$H = \frac{W_s - W}{W} \tag{1}$$

W_s *is* the weight of the swollen hydrogel, and W is the weight of the dry hydrogel [10]. One of the most relevant applications of hydrogels is the controlled release of bioactive compounds, including drugs and heavy metals [15]. The release kinetics can be described by the equation of Ritger-Peppas [16] for the calculation of the fraction of drug released (F):

$$F = \frac{M_t}{M_\infty} = kt^n \tag{2}$$

where M_t is the mass of the drug released at time t, M_∞ is the total mass of the drug, k is the kinetic constant, and n is the diffusional exponent. When $n = 0.5$, the release follows a Fickian diffusion model, whereas values of $n > 0.5$ indicate non-Fickian (anomalous) release behavior. This equation applies when the released fraction does not exceed 60%. Additionally, k can be determined as follows:

$$k = 4\left(\frac{D_i}{\pi h^2}\right)^{1/2} \tag{3}$$

where D_i is the diffusion coefficient of the released drug, and h is the thickness of the hydrogel. Despite recent advances in hydrogel design, several challenges remain, including precise control over release rates, stability of encapsulated compounds, and minimizing side effects. Moreover, further optimization is required

to enhance responsiveness to external stimuli and to improve the protection of sensitive drugs [17] [18].

In this context, the present study investigates the synthesis of a hydrogel composed of xanthan gum (XA) and sodium acrylate (SA) as a promising system for the controlled release of ascorbic acid (AA). The combination of XA's biocompatibility and biodegradability [19] with SA's structural robustness enables the optimized design of materials for applications requiring sustained and efficient release of bioactive compounds.

Although SA and XA hydrogels have demonstrated interesting mechanical properties [20], significant knowledge gaps remain, particularly concerning the mechanisms of network formation and the role of the initiator in determining both the formation process and the resulting performance. While recent studies have focused mainly on enhancing mechanical properties [21], far less attention has been devoted to understanding how SA and XA chains interact to form the three-dimensional network.

Ascorbic acid (vitamin C) is a well-known bioactive compound with documented anti-inflammatory and antioxidant properties [22], which contribute to neutralizing reactive oxygen species and reducing oxidative stress in biological systems [23]. Beyond its classical role as an antioxidant, ascorbic acid has also demonstrated tumor-inhibiting activity [24] and the ability to selectively kill cancer cells at pharmacological concentrations [25], highlighting its potential as an adjuvant in cancer therapy. Given its bioactivity, favorable safety profile, and versatility, ascorbic acid serves as an excellent model compound for evaluating the performance of controlled-release delivery systems designed to maintain therapeutic concentrations, minimize degradation, and improve patient compliance.

The selection of the initiator during hydrogel synthesis can markedly affect the polymer network architecture, as radical generation mechanisms differ. In this study, two initiators with contrasting chemical properties were used: potassium persulfate (KPS) and 2,2-azobis(2-methylpropionamide) dihydrochloride (V50). KPS is an ionic initiator that produces highly reactive sulfate radicals, which tend to promote the formation of denser and more rigid crosslinked networks [26]. In contrast, V50 is a water-soluble azo initiator that generates neutral radicals with a more uniform spatial distribution, a feature associated with the development of more open and flexible network structures [27]. These mechanistic differences enable a direct assessment of how both xanthan gum concentration and initiator type influence network architecture and swelling behavior.

It is hypothesized that variations in XA concentration and the type of initiator employed during hydrogel synthesis will significantly affect both the swelling behavior and the release kinetics of ascorbic acid. Specifically, persulfate-based initiation (KPS) is expected to produce denser, more rigid networks via sulfate-derived radical formation, leading to swelling and release profiles that are less responsive to changes in XA concentration. In contrast, azo-based initiation (V50), which generates more uniformly distributed hydrophilic radicals, is anticipated to

yield more open and flexible network architectures. In such systems, increasing XA concentration is expected to decrease free volume and diffusion pathways, thereby enhancing control over swelling and release kinetics. This formulation-dependent tunability underscores the potential of AS/XA hydrogels for controlled-release applications.

2. Materials and Methods

2.1. Materials

Sodium acrylate (97%) and xanthan gum were purchased from Sigma-Aldrich. N, N'-methylenebisacrylamide (NMBA) from Sigma-Aldrich, potassium persulfate (KPS) from Productos Químicos Monterrey, and 2,2-azobis(2-methylpropionamide) dihydrochloride (V50) from Sigma-Aldrich were used for hydrogel synthesis. Ascorbic acid from Sigma-Aldrich was employed as the model drug for the release studies.

2.2. Hydrogel Preparation

Hydrogels were synthesized with an AS/XA proportion corresponding to 20% of total solids. The XA content was varied at concentrations of 7%, 5%, 2%, and 0% by weight. NMBA was used as the crosslinking agent at 1% of the total solids, while KPS and V50 were also used at 1% each of the total solids as initiators. The polymerization reaction was carried out in bidistilled water as the solvent at 50˚C for 24 hours.

For hydrogel synthesis, glass vials were filled with distilled water, and sodium acrylate was added with a homogenizer. Once dissolved, xanthan gum was added, and after its complete dissolution, the crosslinking agent was incorporated, followed by the initiator. The mixture was then placed in an oven at 50˚C for 24 hours. After this period, the hydrogel was obtained, cut into circular shapes, and dried to constant weight, yielding xerogels. The xerogels were polished to remove imperfections and then shaped into uniform disks with a thickness of 1.5 - 2 mm and a diameter of 10 - 12 mm.

2.3. Swelling Studies

Dried samples were weighed and immersed in swelling media at room temperature, maintaining a neutral pH (pH = 7). At specific time intervals, the samples were removed, surface water was gently blotted with filter paper, and the samples were reweighed. The swelling degree (H) at a given time (t) was calculated using Equation (1).

2.4. Drug Release Kinetics

For ascorbic acid loading, the AS/XA xerogels were accurately weighed and immersed in a 5% (w/v) aqueous ascorbic acid solution at room temperature for 24 hours under gentle stirring to promote passive diffusion. A solution-to-xerogel ratio of 400 mL per gram of xerogel was used to ensure excess drug availability

and near-saturation conditions. After loading, the swollen hydrogels were gently blotted to remove surface solution and weighed. The amount of ascorbic acid loaded was then calculated from the weight difference between the hydrogel before and after swelling, along with the known concentration of the ascorbic acid solution.

Drug loading efficiency was expressed as the average ± standard deviation calculated from the different AS/XA compositions tested. Hydrogels synthesized using KPS showed a mean loading efficiency of 740.25 ± 35.87 mg AA/g dry hydrogel, while those synthesized with V50 exhibited a loading efficiency of 1030.70 ± 142.90 mg AA/g dry hydrogel.

For release experiments, the loaded hydrogels were transferred to 200 mL of distilled water at room temperature, with continuous stirring provided by an electric stirrer. At predefined time intervals, 5 mL aliquots were collected and replaced with fresh distilled water to maintain sink conditions. The concentration of released ascorbic acid was quantified using a UV-visible spectrophotometer (Genesys 10 UV-Visible) at 265 nm.

All experiments in Sections 2.3 and 2.4 were conducted in triplicate to ensure accuracy and reproducibility. The data reported are the averages obtained for each test.

2.5. Influence of pH of the Medium

The swelling media were modified to evaluate the effect of pH on hydrogel swelling behavior. Commercial Golden Bell buffer solutions were used at pH = 2 ± 0.01 (acidic medium), pH = 7 ± 0.01 (neutral medium), and pH = 10 ± 0.01 (alkaline medium). Phosphate buffers were employed, and their pH values were verified by direct measurement using an EXTECH ExStik pH meter. Hydrogels were immersed in buffer solutions at different pH values to assess the effect of pH on their swelling.

3. Results and Discussions

3.1. Swelling Behavior of Hydrogels

This section presents the experimental results characterizing the swelling behavior of the synthesized hydrogels and their relationships with composition and the swelling medium. Swelling kinetics and diffusion coefficients are analyzed, which are fundamental to understanding the dynamics of water absorption and their impact on drug-controlled release.

Figure 1 shows the swelling kinetics of xerogels synthesized using KPS as the initiator with different concentrations of XA. The data indicate that the swelling process progresses gradually over approximately 55 hours. During the initial 30 hours, samples with varying XA concentrations exhibit similar swelling profiles, with no significant differences observed.

However, after 55 hours, slight divergences between samples begin to appear, which may be attributed to material loss due to partial disintegration of the hy-

drogels. This phenomenon, associated with the structural degradation of the material, introduces a potential source of experimental error that should be considered when interpreting the results. Nevertheless, the observed changes in swelling are consistent with diffusion mechanisms previously reported in the literature for polymeric hydrogel systems with similar characteristics.

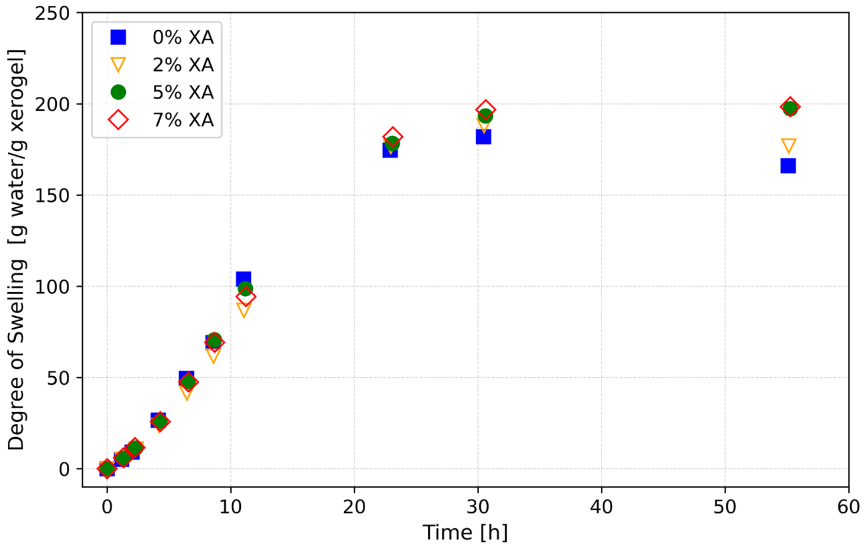


Figure 1. Equilibrium swelling of hydrogels synthesized with KPS.

A noteworthy observation is the absence of significant differences in the swelling degree between hydrogels with varying XA concentrations when synthesized with KPS. This fact suggests that XA does not substantially influence the water absorption and retention capacity of these hydrogels. Although XA is a hydrophilic biopolymer with ionizable carboxyl groups, its interactions with the hydrogel matrix appear to be insufficient to alter the overall behavior of the material significantly.

Conversely, hydrogels synthesized with V50 as the initiator exhibit a different behavior. **Figure 2** shows the equilibrium swelling as a function of XA concentration. In this case, an inverse trend is observed: as XA concentration increases, swelling decreases. Although XA is a hydrophilic material expected to enhance water absorption, the opposite result suggests that XA occupies spaces within the polymer network, reducing the available volume for water absorption. This behavior has been previously reported in similar systems [10]. This effect can be attributed to XA's structural role within the polymer network. Owing to its high molecular weight and semi-rigid polysaccharide backbone, XA does not fully copolymerize as a linear chain but instead forms physical entanglements that locally restrict polymer mobility. These entanglement domains increase network tortuosity and reduce the free volume available for water penetration, thereby lowering swelling despite XA's inherently hydrophilic nature.

Figure 3 illustrates the swelling behavior of hydrogels in media with varying

pH values (acidic, neutral, and basic), with a focus on the formulation containing 7% XA. A significant difference in equilibrium swelling is observed depending on the pH of the medium, with maximum swelling of approximately 200% under neutral conditions. This behavior is related to the ionization of the hydrogel's functional groups, since ionization increases when the system's pH exceeds the pKa of these groups [28]. As pH rises, material ionization increases, leading to a greater number of fixed charges within the hydrogel structure. This increase in ionization enhances electrostatic repulsive interactions, boosting the hydrogel's hydrophilic properties. Given that XA contains carboxyl groups with relatively low pKa values, it is reasonable that swelling is more pronounced under neutral conditions compared to acidic media.

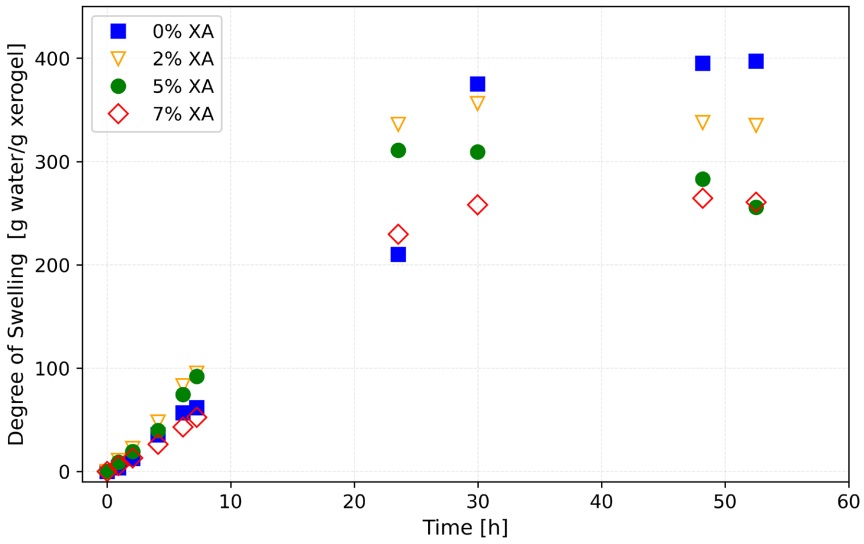


Figure 2. Equilibrium swelling of hydrogels synthesized with V50.

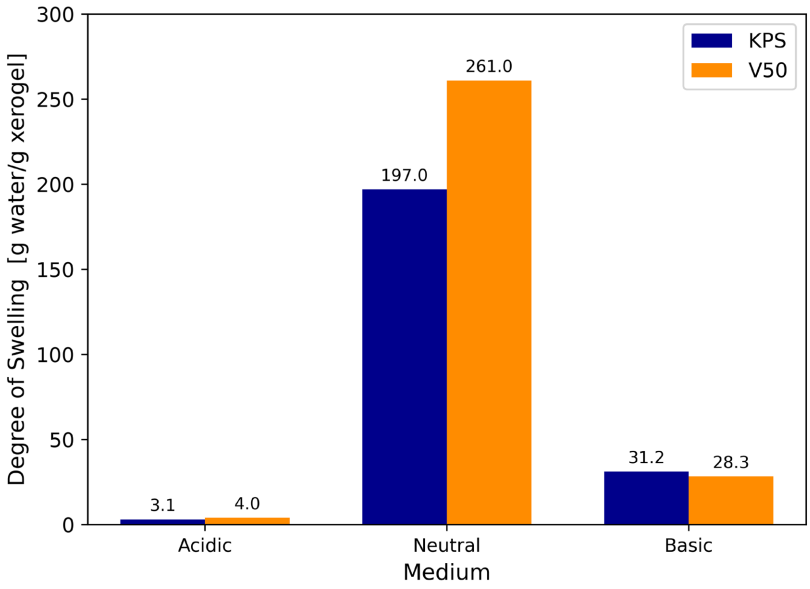


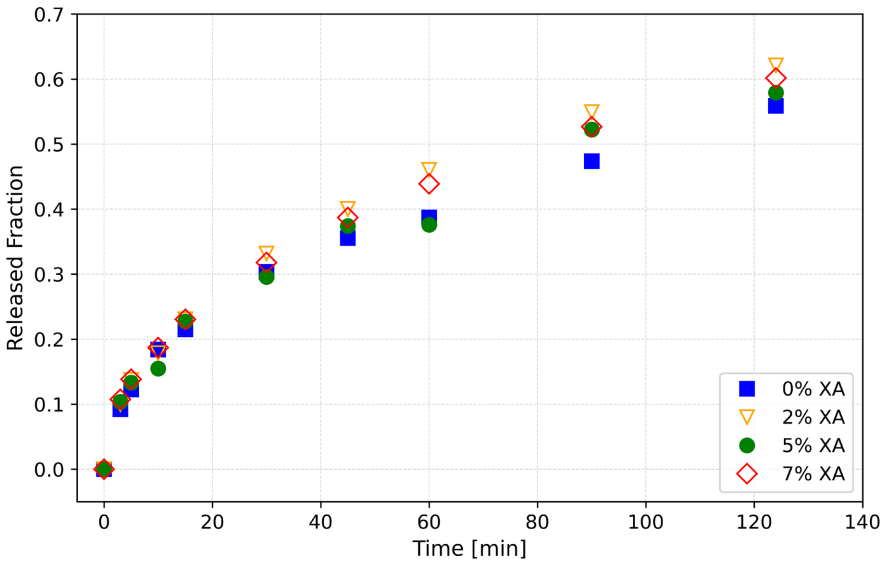
Figure 3. Swelling degree of hydrogels in different pH conditions (7% XA).

Finally, a drastic decrease in equilibrium swelling is observed under basic conditions. This response is consistent with the findings reported by Tushar *et al.*, who observed similar results in sodium polyacrylate and xanthan hydrogels [29]. According to Donnan's theory, the hydrogel's swelling capacity decreases as interactions with counterions in the medium intensify, limiting the expansion of the polymer network [30].

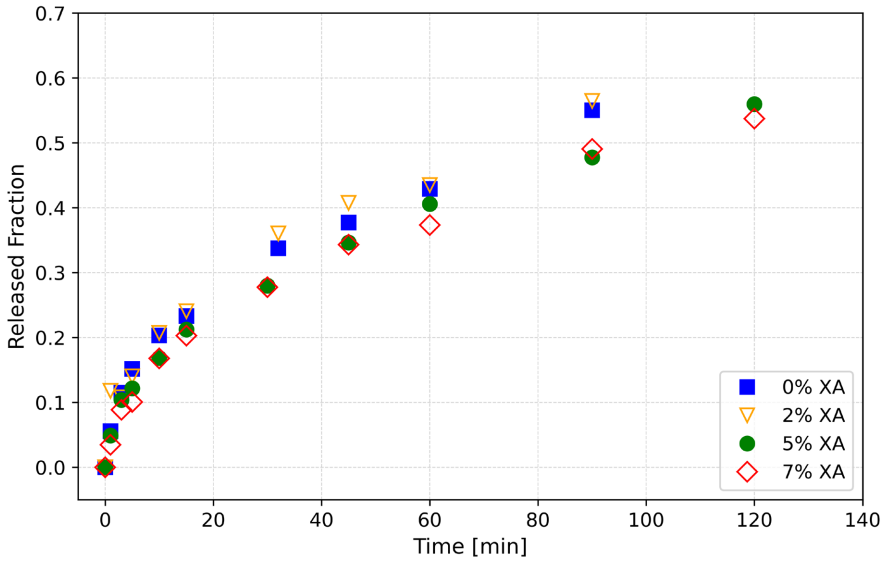
3.2. Drug Release Behavior

A significant difference in ascorbic acid loading efficiency was observed between hydrogels synthesized with KPS (740.25 ± 35.87 mg/g) and those synthesized with V50 (1030.70 ± 142.90 mg/g). This variation arises from the distinct network architectures promoted by each initiator. As previously discussed, KPS generates sulfate-derived radicals that tend to form more compact, highly cross-linked networks, thereby reducing the free volume available for solute penetration during loading. In contrast, V50 produces neutral radicals that promote the formation of more open and flexible network structures with larger pore domains and greater water-uptake capacity, thereby facilitating higher ascorbic acid incorporation. From a practical perspective, this suggests that V50-based hydrogels may be better suited to applications requiring high drug-loading capacity and sustained release. In contrast, KPS-based hydrogels may be preferable for applications requiring lower loading and more stable release profiles.

Figure 4(a) and **Figure 4(b)** depict the release kinetics of ascorbic acid from AS/XA hydrogels synthesized using KPS and V50 as initiators, respectively, in an aqueous medium. **Figure 4(b)** highlights the correlation between ascorbic acid release and the XA concentration in the hydrogel. An inverse relationship is observed: as the XA concentration increases within the polymer network, the amount of drug released decreases.



(a)



(b)

Figure 4. Ascorbic acid release kinetics from hydrogels synthesized with KPS (a) and V50 (b).

Conversely, **Figure 4(a)** shows no clear trend between the amount of drug released and the XA concentration. This behavior is attributed to the swelling kinetics being closely linked to the void spaces within the polymer network [31]. Incorporating XA into the network reduces these void spaces, thereby decreasing overall porosity and resulting in lower equilibrium swelling. This effect is evident in **Figure 4(b)**, where higher XA concentrations result in reduced drug release.

In contrast, hydrogels synthesized using KPS do not exhibit this trend. This behavior can be attributed to changes in polymer network porosity that are not significant enough to yield a clear correlation across different XA concentrations. The equilibrium swelling results further support this, where the differences among these hydrogels are not substantial (**Figure 1**).

The Ritger-Peppas equation (Equation (2)) was used to determine the nature of the diffusion system, employing water as the solvent. The exponent n and the kinetic constant k were obtained from the slope and intercept of the $\ln(F)$ vs. $\ln(t)$ plot, respectively. Equation (3) was used to calculate the diffusion coefficient, taking into account its limitations: specifically, a maximum released fraction of 0.6 and the requirement that the sample thickness be sufficiently small compared to its diameter, allowing the hydrogel to be considered as a thin film (the diameter-to-thickness ratio should be between 5 and 8) [10]. It is worth noting that the hydrogel disks used in this study satisfied the required diameter-to-thickness ratio (5 - 8), confirming the suitability of the Ritger–Peppas model and supporting the reliability of the calculated diffusion coefficients. The diffusion parameters for hydrogels synthesized with both KPS and V50 are summarized in **Table 1**.

Table 1 shows that for hydrogels synthesized using KPS, the diffusion coefficient remains relatively constant across different XA concentrations (**Figure 5**

provides a more precise visualization of this trend). The calculated exponents are approximately 0.5, suggesting the drug follows a Fickian diffusion mechanism [16].

Table 1. Diffusion parameters of drug release in AS/XA hydrogels synthesized with KPS and V50.

% XA	KPS			V50		
	k	*n*	*D* (cm²/s)	*k*	*n*	*D* (cm²/s)
0	0.0584	0.47	5.43E−06	0.0636	0.48	9.85E−06
2	0.0599	0.49	5.43E−06	0.0659	0.48	8.77E−06
5	0.0604	0.47	5.68E−06	0.0546	0.49	7.94E−06
7	0.0650	0.47	7.07E−06	0.0497	0.50	4.14E−06

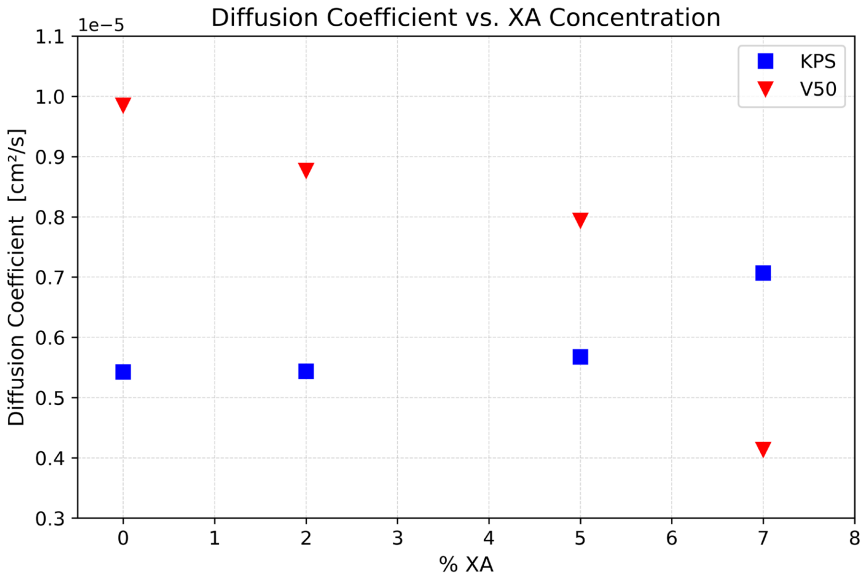


Figure 5. Effect of XA concentration on the diffusion coefficient of hydrogels.

For hydrogels synthesized with V50, the diffusion coefficients decrease with increasing XA concentration. Similar to the samples synthesized with KPS, this trend suggests that higher XA concentrations decrease the free volume within the polymer network, thereby restricting drug diffusion. This reduction in diffusivity is directly linked to the decrease in swelling capacity, as shown in **Figure 2**.

4. Conclusions

The hydrogels synthesized from xanthan gum (XA) and sodium acrylate (AS) exhibit a complex behavior regarding their swelling capacity and controlled drug release, which is significantly influenced by the XA concentration and the surrounding medium conditions. Although XA, as a hydrophilic biopolymer, can enhance swelling and water uptake in hydrogels, its effect on hydrogel performance varied significantly between initiators. KPS produced denser networks with min-

imal changes in swelling and release behavior as XA concentration increased, whereas V50 generated more open structures, in which increasing XA concentration progressively reduced free volume and diffusion, enabling finer control over the release process.

The observed pH-dependent behavior underscores the importance of carboxyl group ionization in XA, which affects both swelling and drug release kinetics. These findings emphasize the need to consider functional group ionization and molecular interactions when designing hydrogels for specific applications.

The variability in the ascorbic acid release as a function of XA concentration suggests that a precise adjustment of this biopolymer is crucial for optimizing hydrogel properties in pharmaceutical applications. These results highlight the importance of a deeper understanding of the relationship between the polymer network structure and its interactions with the surrounding environment, which could lead to the development of more efficient controlled drug-release systems.

Furthermore, this study emphasizes the importance of optimizing synthesis methods and selecting suitable polymerization initiators to enhance the properties and performance of hydrogels in various applications. The findings suggest that it is possible to design hydrogels with tailored characteristics suited for specific therapeutic needs by modulating composition and synthesis conditions.

Future research should focus on practical applications, such as evaluating these hydrogels in combination with other bioactive compounds to assess their versatility in drug delivery. Conducting in vitro assays will be essential to confirm biocompatibility and release profiles under physiological conditions. Additionally, adapting these hydrogel systems for transdermal or oral delivery routes could expand their therapeutic potential and facilitate their integration into clinical practice.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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