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Diffusion and Relaxation Kinetics of Chitosan-graft-poly (Acrylic Acid) Hydrogels: A Theoretical and Experimental Study

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ABSTRACT

Hydrogels, as three-dimensional polymer networks, are extensively utilized due to their ability to absorb significant amounts of water while remaining insoluble. Chitosan-grafted poly(acrylic acid) hydrogels, in particular, combine high water absorption with improved mechanical properties, making them ideal for applications in agriculture and drug delivery. This study investigates the swelling kinetics of chitosan-graft-poly(acrylic acid) hydrogels synthesized with varying concentrations of the crosslinker N,N'-methylenebisacrylamide (MBA). The objective is to establish the relationship between crosslinker concentration and hydrogel performance by evaluating their swelling behavior using kinetic models. The hydrogels were prepared via free-radical polymerization, with MBA concentrations of 0.015 g (Hgel 1), 0.05 g (Hgel 2), and 0.1 g (Hgel 3). The research results indicate that swelling increases as the MBA concentration decreases. This was attributed to the lower crosslinking density, which reduces the entanglement of polymer chains. Hgel 1 displayed the greatest swelling ratio 171.71 g/g, while Hgel 2 150.21 g/g and Hgel 3 144.76 g/g. The research results indicate that the swelling behavior is best described by the diffusion kinetics model, as evidenced by the best fit between the experimental data and calculations, with R^2 values of 0.996, 0.996, and 0.984 for Hgel 1, Hgel 2, and Hgel 3, respectively. The applicability of pseudo-second order model, in good agreement with the one found for Hgel 2 (R^2 = 0.997). These results suggest that diffusion dominates the swelling behaviour and that hydrogel behaviour is sensitive to crosslinker concentration. This work highlights the potential use of chitosan-graft-poly(acrylic acid) hydrogels for sustainable applications in agriculture and biomedicine.

Keywords: *Hydrogel, swelling kinetics, diffusion model, relaxation model*

1. INTRODUCTION

Hydrogels are polymer networks with a three-dimensional structure created through cross-linking. Due to their high content of hydrophilic groups. they exhibit a strong affinity for water. allowing them to absorb significant amounts. often more than 20% of their weight. as well as other bodily fluids. Despite their ability to absorb water. they remain insoluble due to the physical or chemical bonds between polymer chains. These cross-linking bonds can be formed through chemical or physical processes. Chemically cross-linked hydrogels are typically synthesized using methods such as photopolymerization. thermal polymerization. or free-radical polymerization. whereas physically cross-linked hydrogels are formed via non-covalent interactions at the molecular level (Wagh et al., 2024; Kohar et al., 2024).

The development of synthetic hydrogel has raised concerns over non-biodegradability. Natural polymers are eco-friendly but have lower water retention. Hybrid hydrogel. combining synthetic absorption with natural biodegradability. address these issues. Semi-synthetic

hydrogel like cellulose-chitosan hydrogels enhance water retention and environmental safety (Czarnecka & Nowaczyk, 2020; Alam & Christopher, 2018). Using natural monomers and cross-linkers further improves biodegradability. offering sustainable. cost-effective alternatives (Yamuna & Kandhavadivu, 2023; Patiño-Masó et al., 2019). Biodegradable hydrogel decomposes over time. reducing plastic waste and supporting sustainability (Memon et al., 2022; Smagin et al., 2023).

Chitosan-grafted polyacrylic acid (PAA) superabsorbent hydrogels have been widely studied for their applications in agriculture and drug delivery due to their high-water absorption capacity and improved mechanical properties. These hydrogels. synthesized through methods like emulsion polymerization using cross-linkers such as N.N'-methylenebisacrylamide and gamma irradiation (Rekha Sahoo & Biswal, 2024; Jayanudin et al., 2023). They enhance soil moisture retention. especially in sandy soils. and act as carriers for slow-release fertilizers like urea. improving nutrient availability (Jayanudin et al., 2023; Jayanudin et al., 2022). In drug delivery. these hydrogels offer controlled release influenced by pH levels (Rekha Sahoo & Biswal, 2024). Despite their benefits. further research is needed to optimize their performance under diverse environmental conditions.

The swelling properties of hydrogels are essential for various applications. including agriculture. drug delivery. tissue engineering. and mechanical systems. These properties allow hydrogels to absorb significant amounts of water. enhancing their ability to mimic biological tissues. In agriculture. hydrogels aid in retaining soil moisture optimizing fertilizer efficiency and reducing environmental impact (Tripathy, 2023). In drug delivery, swelling enables controlled and targeted release through responsiveness to physiological stimuli (Kohar et al., 2024). For tissue engineering. swelling supports the transport of nutrients and improves scaffold effectiveness in tissue regeneration (Feng & Wang, 2023). Furthermore. swelling impacts mechanical strength and elasticity. making hydrogels suitable for flexible biomedical applications. while non-swelling hydrogels provide stability for specific purposes (Feng & Wang, 2023). Achieving a balance in these properties is crucial to maximizing hydrogel functionality (Wagh et al., 2024).

A theoretical model has been proposed to describe the swelling behavior of disk-shaped gels (Man & Doi, 2021). The overall swelling rate is influenced by the diffusion of water molecules into the hydrogel and/or the relaxation of the polymer chains (Ostrowska-Czubenko et al., 2015; Yavari & Azizian, 2022). Several kinetic models have been developed to explain swelling. with some focusing on diffusion mechanisms and others emphasizing polymer relaxation (Yavari & Azizian, 2022). Therefore. this study aims to determine the swelling kinetics of chitosan-graft-poly (acrylic acid)-based hydrogels using several models. followed by a comparison between experimental data and calculations.

2. MATERIALS AND METHODS

2.1 Materials

Acrylic acid (purity 99%) was supplied by Sigma-Aldrich. Chitosan. with a degree of deacetylation (DD) of 87.2%. was produced from PT. Biotech Surindo. Potassium hydroxide pellets (Merck) were used. N.N'-methylenebisacrylamide (MBA) was obtained from Sigma-Aldrich. while ammonium persulfate (APS) was sourced from Merck. Potassium sodium tartrate tetrahydrate was also purchased from Merck.

2.2 Preparation of Hydrogel from Chitosan-graft-poly(acrylic acid)

The synthesis began with dissolving 7.8 g of potassium hydroxide in 100 mL of distilled water. which was then used to neutralize 15 mL of acrylic acid. followed by stirring for 15 minutes. The neutralized acrylic acid solution was left to stand for 24 hours. Meanwhile. a 4% (w/v) chitosan solution was prepared using 2% (v/v) acetic acid as the solvent. To this chitosan solution. 0.05 g of ammonium persulfate (dissolved in 5 mL of distilled water) was added. and the mixture was combined with the neutralized acrylic acid solution. The resulting mixture was stirred at 500 rpm and heated to 70°C. After 5 minutes. N.N'-methylene bisacrylamide (MBA) in varying amounts (0.015 g. 0.05 g. and 0.1 g) was added sequentially. and stirring continued until the formation of a superabsorbent gel. Finally. the Chitosan-graft-poly(acrylic acid) hydrogel was dried at 65°C for 24 hours or until a constant weight was achieved.

Table 1. The symbols and definitions for the variables utilized in this study

No	Crosslinker Weight	Symbol
1.	0.015 g	Hgel 1
2.	0.05 g	Hgel 2
3.	0.1 g	Hgel 3

2.3 Swelling Measurement

A 0.5 g sample of the dry superabsorbent was placed in a tea bag and submerged in 200 mL of tap water at room temperature. The water absorption capacity was evaluated at specific intervals: 1. 3. 5. 12. and 24 hours. After each time point. the swelling capacity was calculated using Equation (1):

$$Swelling\ (g/g) = \frac{m_1 - m_0}{m_0} \qquad (1)$$

Where m_1 and m_0 were swollen and dry weight of sample

2.4 Swelling kinetics model

The swelling kinetics models used in this study are shown in Equations 2-5. The swelling kinetics models are derived from the research conducted by Chen et al., (2021); Zhao et al., (2019); B. Wang et al., (2018); Wang et al., (2009); Jayanudin et al., (2024); Yavari & Azizian, (2022).

- Relaxation

$$Q_t = Q_e(1 - e^{-k_1 t}) \quad (2)$$

$$Q_t = \frac{k_2 Q_e^2 t}{1 + k_2 Q_e t} \quad (3)$$

- Diffusion

$$Q_t = Q_e k t^n \quad (4)$$

$$Q_t = Q_e \left(1 - e^{-k_2 t^{1/2}}\right) \quad (5)$$

In this context. Q_e and Q_t denote the absorption capacities at equilibrium and at a specific time t. respectively. while $k_1(min^{-1})$ and $k_2(g.g^{-1}min^{-1})$ represent the kinetic constants corresponding to the two models. Where k represents the rate constant. and n is a parameter that reflects the swelling mechanism:

a) When n = 0.5. it corresponds to water molecule diffusion.
b) When n = 1. it indicates the relaxation of polymer chains.
c) For 0.5 < n <1. it describes a combination of diffusion and relaxation mechanisms.

3. RESULTS AND DISCUSSION

3.1. Swelling ratio

Swelling in hydrogels plays a crucial role in both drug delivery and agricultural applications. significantly influencing their effectiveness and functionality. The ability of hydrogels to swell allows them to retain water and nutrients. making them valuable in various fields. This overview will discuss the importance of swelling in hydrogels. focusing on their applications in agriculture and biomedicine. Fig. 1 illustrates the effect of the crosslinker N.N'-methylenebisacrylamide (MBA) weight on the swelling ratio of chitosan graft poly(acrylic acid)-based hydrogels.

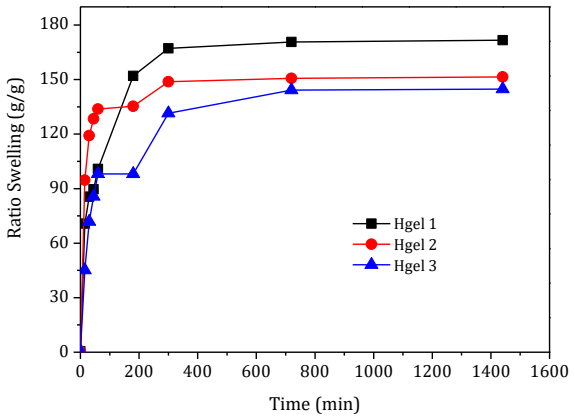

Fig. 1. The effect of crosslinker N.N'-methylenebisacrylamide (MBA) on the swelling ratio of chitosan graft poly (acrylic acid)-based hydrogels

The swelling ratio of chitosan graft poly (acrylic acid)-based hydrogels is significantly influenced by the concentration of the crosslinker. N.N'-methylenebisacrylamide (MBA). The data presented in Fig. 1 showed that reducing the weight of MBA leads to a noticeable increase in the swelling ratio of the hydrogel. This behavior is attributed to the reduction in crosslinking density. which allows the polymer chains to expand and absorb more water. Hgel 1. prepared with 0.015 g of MBA. exhibited the highest swelling ratio of 171.71 g/g. In contrast. Hgel 3. with the highest MBA concentration of 0.1 g. showed a swelling ratio of 144.76 g/g. The intermediate hydrogel. Hgel 2. synthesized with 0.05 g of MBA. demonstrated a moderate swelling ratio of 150.21 g/g. These results indicate that MBA concentration has an inverse relationship with the swelling ratio of the hydrogels. as higher crosslinker content forms a denser network structure. which restricts the diffusion and retention of water within the polymer matrix.

The trends observed in this study are consistent with previous literature. For example. Safaraliyeva et al., (2024) demonstrated that as the concentration of MBA increased. the swelling ratio of chitosan graft poly(acrylamide) hydrogels decreased significantly due to the formation of a more compact and rigid crosslinked network. Similarly. Che Ani et al., (2017) reported that a reduction in MBA weight from 2 wt.% to 0.5 wt.% led to an increase in swelling capacity from 5.1 g/g to 20.6 g/g in polyacrylamide-based hydrogels. In another study. Jozaghkar et al., (2022) observed a comparable trend. noting that chitosan graft poly (acrylic acid) hydrogels achieved maximum swelling ratios at lower MBA concentrations due to the enhanced flexibility of the polymer chains. These findings highlight that optimizing crosslinker concentration is crucial for tailoring hydrogel properties to specific applications.

In addition to crosslinker concentration. the swelling behavior of hydrogels is also influenced by environmental factors such as pH. temperature. and ionic strength. Studies had shown that maximum swelling occurs in alkaline environments. where ionization of functional groups within the hydrogel matrix is maximized. For instance. Gupta & Purwar, (2021) noted that hydrogels prepared with MBA follow a second-order kinetic model for swelling behavior across various pH levels. Jozaghkar et al., (2022) confirmed that swelling in acidic conditions is typically lower due to the reduced ionization of carboxyl groups. which limits water uptake. Safaraliyeva et al., (2024) further emphasized the role of ionic strength in modulating swelling ratios. as increased ionic strength leads to a shielding effect that reduces the osmotic pressure difference and. consequently. the swelling ratio. These findings suggest that environmental conditions should be carefully controlled or considered when designing hydrogels for specific applications.

3.2. Swelling kinetics

In this study, the kinetic model used was based on diffusion and relaxation. Fig. 2 shows the calculation

results using diffusion and relaxation kinetics and compared with experimental data.

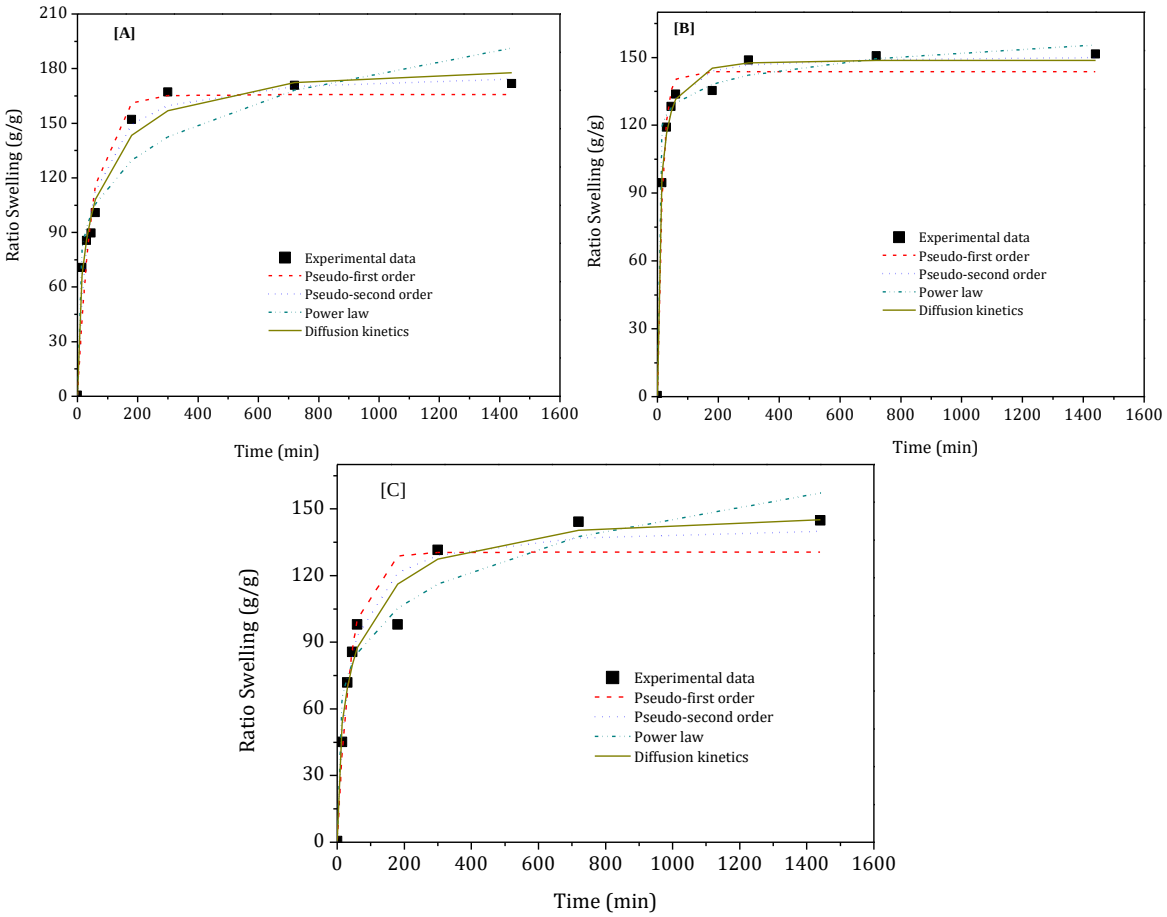

Fig. 2 Comparison of the results of diffusion and relaxation kinetics calculations with experimental data based on changes in the weight of the crosslinker N.N'-methylenebisacrylamide (MBA) [A] 0.015 g, [B] 0.05 g, and [C] 0.1 g

The swelling kinetics of hydrogels were analyzed using four kinetic models: pseudo-first order. pseudo-second order. power law. and diffusion kinetics. to evaluate the mechanisms governing the swelling process. The fitting results showed that the diffusion kinetics model achieved the highest R^2 values for all hydrogels (Hgel 1: 0.986; Hgel 2: 0.982; Hgel 3: 0.977). indicating that the swelling process is primarily controlled by diffusion through the hydrogel matrix. The pseudo-second order model also exhibited good compatibility. particularly during the transition phase of swelling. where the process shifts from rapid kinetics to saturation (Hgel 1: 0.977; Hgel 2: 0.980; Hgel 3: 0.971). In contrast. the pseudo-first order model had lower R^2 values. reflecting that the swelling rate is not fully dependent on the concentration of unabsorbed areas. The power law model effectively described the swelling kinetics in the initial phase but tended to overestimate values during the saturation phase.

The swelling ratio graphs revealed consistent patterns across the three hydrogels. The swelling ratio increased significantly during the initial phase (0–300 minutes) due to the high diffusion rate. then slowed down and approached saturation after 720–1440 minutes. Hgel 1 exhibited the highest swelling ratio compared to Hgel 2 and Hgel 3. which could be attributed to its structural properties. such as higher porosity or greater water retention capacity. Hgel 2 demonstrated a lower swelling ratio than Hgel 1. while Hgel 3 showed the lowest values among the three. reflecting differences in the physicochemical characteristics of each hydrogel. This analysis confirmed that the diffusion kinetics model provides the most accurate description of the overall swelling process. as it captures the dominant role of diffusion as the primary mechanism. The pseudo-second order model serves as a viable alternative for describing more complex adsorption mechanisms during the transition phase.

The pseudo-first order model evaluates the theoretical swelling capacity and the rate of swelling in hydrogels. The parameter Qe , which represents the equilibrium swelling capacity, was highest for Hgel 1 (165.665 g/g), indicating its superior swelling capacity compared to Hgel 2 (143.776 g/g) and Hgel 3 (130.525 g/g). The rate constant k_1 was highest for Hgel 2 (0.063), suggesting that Hgel 2 swells at a faster rate than Hgel 1 and Hgel 3. In terms of model accuracy, R^2 for Hgel 2 was the highest (R^2 = 0.99), showing good agreement with experimental data. However, the R^2 for Hgel 3 (R^2 = 0.96) indicates that this model is less suitable for describing the swelling kinetics of Hgel 3. The pseudo-second order model

assumes that the swelling rate depends on the available adsorbed area. Hgel 1 had the highest Qe value (178.403 g/g), confirming its large swelling capacity. Hgel 2 and Hgel 3 also showed high Qe values, indicating realistic predictions of swelling behavior. However, the rate constant k_2 was extremely small (approaching zero) for all hydrogels, indicating a slow swelling rate under the given conditions. The R^2 value for Hgel 2 was perfect (R^2 = 0.997), demonstrating that this model is highly accurate for describing the swelling mechanism of Hgel 2

Table 2. Kinetic constants of the diffusion and relaxation models

Symbol	Pseudo-first order			Pseudo-second order		
	Qe	k_1	R^2	Qe	k_2	R^2
Hgel 1	165.665	0.020	0.98	178.403	0.00016	0.989
Hgel 2	143.776	0.063	0.99	150.780	0.00078	0.997
Hgel 3	130.525	0.024	0.96	143.105	0.00022	0.98
Symbol	**Power law**				**Diffusion kinetics**		
	Qe	**k**	**n**	**R^2**	**Qe**	**$k_{2'}$**	**R^2**
Hgel 1	99.117	0.496	0.187	0.97	180.6	0.172674	0.996
Hgel 2	143.430	0.717	0.057	0.98	148.7559	0.278918	0.996
Hgel 3	87.956	0.440	0.193	0.97	146.8142	0.116904	0.984

The power law model provides insight into the diffusion mechanism during swelling. The Qe values for this model were generally lower than those predicted by the pseudo-first and pseudo-second order models, with Hgel 2 having the highest Qe (143.43 g/g). The diffusion exponent n, which was less than 0.2 for all hydrogels, indicates that the swelling process is governed by non-Fickian diffusion mechanisms. The R^2 values ranged from 0.97 to 0.98, suggesting that while the model is adequate for describing initial swelling phases, it lacks precision during the saturation phase. The diffusion kinetics model is most effective for describing the swelling process, as evidenced by the highest Qe values, particularly for Hgel 1 (180.6 g/g), which closely matches experimental results. The diffusion constant $k_{2'}$ was highest for Hgel 2 (0.279), indicating a faster diffusion process compared to the other hydrogels. Additionally, the R^2 values were the highest among all models (R^2=0.996 for Hgel 1 and Hgel 2, R^2 = 0.98 for Hgel 3), confirming that diffusion is the dominant mechanism controlling the swelling process.

The diffusion kinetics model is the most suitable for describing the swelling kinetics of all hydrogels, given its highest R^2 values and Qe values that closely match experimental data. The pseudo-second order model also provides a relevant alternative, especially for Hgel 2, with its perfect R^2 = 0.997, indicating the influence of adsorbed surface area on swelling. Among the hydrogels, Hgel 1 demonstrated the highest swelling capacity, whereas Hgel 3 exhibited the lowest capacity and swelling rate. These differences reflect variations in internal structure, such as porosity and water retention ability, which are critical for tailoring hydrogel properties for specific applications

4. CONCLUSION

This study analyzed the swelling kinetics of chitosan-graft-poly (acrylic acid) hydrogels with varying crosslinker concentrations (MBA: 0.015 g, 0.05 g, 0.1 g). Results showed that lower crosslinker concentrations enhance swelling capacity due to reduced crosslinking density, allowing greater water absorption. Among the kinetic models evaluated, the diffusion kinetics model demonstrated the highest accuracy ($R^2 \approx 0.996$), indicating that diffusion is the dominant mechanism in swelling. The pseudo-second order model also provided strong compatibility, particularly for Hgel 2 (R^2 = 0.997). These findings emphasize the importance of optimizing crosslinker concentration to tailor hydrogels for applications in agriculture and drug delivery

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