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MORALITY BEFORE KNOWLEDGE

Early swelling kinetics of chitosan hydrogels under different acidity conditions for designing controlled-release fertilizers

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ABSTRACT

Background: Nutrient losses from conventional fertilization have increased the need for biodegradable hydrogel matrices that improve water retention and support controlled-release fertilizer (CRF) systems. This study investigated the effects of pH on the early-stage swelling kinetics of chitosan-based hydrogels, identified the most stable formulation, and examined the relationship between degree of deacetylation (DD) and swelling performance. **Methods**: A quantitative experimental design was applied to four formulations—ChGu, ChG, ChPu, and ChP—under acidic, neutral, and alkaline conditions. Swelling was measured for 120 minutes using replicate observations. The analysis included swelling ratio (SR), maximum swelling ratio (Max SR), area under the curve (AUC), net swelling change (ΔSR), means, standard deviations, statistical comparisons, and regression analysis. **Findings:** pH markedly influenced the swelling behavior of all formulations. Neutral conditions produced the highest average SR values, with ChP showing the best performance (86.4), followed by ChG (67.6), ChGu (63.8), and ChPu (30.4). ChP also recorded the highest Max SR (124) and AUC (11,100), while ChG reached a Max SR of 110 and an AUC of 9,150, indicating greater expansion capacity and more sustained swelling. In contrast, alkaline conditions caused deswelling in most samples, as shown by negative SR values. The relationship between DD and swelling performance was weak (R^2 = 0.14), indicating that hydrogel structure, formulation composition, environmental pH, and early kinetic transitions were more influential than DD alone. **Conclusion:** Neutral pH provided the most favorable swelling conditions, and ChP demonstrated the strongest overall performance. **Novelty/Originality of this article:** This study proposes Max SR, AUC, ΔSR, and dynamic transition intervals as integrated early-stage indicators for screening biodegradable chitosan hydrogels for CRF applications.

KEYWORDS: biodegradable; chitosan; hydrogel; pH; swelling.

1. Introduction

The increasing demand for food production has intensified fertilizer use, yet conventional fertilization often causes nutrient losses through leaching, runoff, denitrification, and volatilization. In Indonesia, nitrogen use efficiency remains relatively

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low, around 30–50%, meaning much of the applied nitrogen is not absorbed by plants (Flatian et al., 2020). Since the optimal soil pH for major crops generally ranges from 5.5 to 7.5, understanding material behavior within this range is important for fertilizer and water retention design (Indonesian Agency for Agricultural Research and Development, 2020; Flatian et al., 2020).

Controlled-release fertilizers (CRFs), especially hydrogel-based systems, are promising because they can absorb and retain water, support root-zone moisture, and regulate nutrient release (Govil et al., 2024; Jariwala et al., 2022; Chandran et al., 2021). However, classical diffusion-based release models may not fully capture the behavior of biopolymer systems due to heterogeneous networks, interactions with the medium, and time-dependent swelling (Katchali et al., 2025; Lawrencia et al., 2021; VanZanten et al., 2024; Lipin et al., 2023). According to Zhao et al. (2023) and Klein & Poverenov (2020), chitosan is widely studied as a CRF hydrogel material due to its biobased origin, biocompatibility, and modifiability. Its reactive amine groups enable three-dimensional network formation and pH-responsive swelling, making it suitable for adaptive nutrient-release systems (VanZanten et al., 2024; Hong et al., 2024; Liu et al., 2024). Biodegradable materials are also relevant to sustainable agriculture, with degradation influenced by material type, environmental conditions, and microbial activity (Strotmann et al., 2023; Eddarai et al., 2024; Firmanda et al., 2024).

Accordingly, based on the identified research gap, this study seeks to examine how different pH conditions influence the early-stage swelling kinetics of chitosan-based hydrogels, determine which hydrogel formulation demonstrates the most stable swelling performance, and assess the extent to which the degree of deacetylation (DD) is associated with hydrogel swelling behavior. Building on the studies of Klein & Poverenov (2020), Lawrencia et al. (2021), Chiaregato et al. (2022), and Govil et al. (2024), the novelty of this study lies in integrating early-stage swelling kinetics (0–120 min) with a multi-indicator evaluation comprising maximum swelling ratio (Max SR), area under the curve (AUC), changes in swelling ratio (ΔSR), and dynamic transition analysis. This integrated approach positions early-stage swelling kinetics as a preliminary predictive parameter and provides a framework for evaluating the performance of chitosan-based hydrogels in controlled-release fertilizer applications.

2. Methods

2.1 Research design

This study employed a quantitative experimental design to investigate the initial swelling kinetics of chitosan-based hydrogels and their implications for the development of controlled-release fertilizers (CRFs). Swelling kinetics plays a critical role in regulating water uptake and nutrient discharge, and hydrogel networks influence diffusion pathways and release mechanisms (Liu et al., 2024). Previous research demonstrates that early swelling responses are essential to understanding hydrogel performance, as they determine the extent of polymer network expansion and subsequent nutrient mobility (Tian et al., 2023; Lawrencia et al., 2021). The analysis focused on time-dependent swelling kinetics and the relationship between chitosan chemical characteristics and hydrogel responses, using initial swelling kinetics as an analytical framework to assess hydrogel stability under acidic, neutral, and alkaline conditions.

The dataset consisted of two main components: the degree of deacetylation (DD) of chitosan, which represents structural characteristics, and the swelling ratio (SR) of hydrogels, measured over time at different pH levels. The degree of deacetylation affects the availability of free amino groups, thereby altering electrostatic interactions, network hydrophilicity, and pH sensitivity in hydrogel matrices, as highlighted in recent chitosan hydrogel reviews (Tian et al., 2023; Hong et al., 2024). Four hydrogel formulations were examined: ChGu (chitosan-glucomannan-urea), ChG (chitosan-glucomannan), ChPu (chitosan-polyvinylpyrrolidone-urea), and ChP (chitosan-polyvinylpyrrolidone). Each sample was evaluated under acidic, neutral, and alkaline conditions to capture variations in swelling kinetics across both material types and chemical environments.

Chitosan was synthesized using 3% HCl for demineralization at 60 °C for 2 hours, followed by 3% NaOH treatment at 80 °C for 3 hours. Solid-to-liquid ratio was 1:20 (w/v). The material was washed until neutral pH (pH 7) and dried at 50 °C for 24 hours. The degree of deacetylation was determined using FTIR spectroscopy and calculated using the specified DD equation according to Hong et al. (2024) and Budianto & Amalia (2020). Swelling tests were conducted according to Yang et al. (2025) by drying the hydrogels to constant weight to obtain the initial dry mass (W0), immersing them in buffer solutions at designated pH levels, and measuring the swollen mass (Wt) at 0, 30, 60, 90, and 120 minutes. Relationships between DD and swelling performance were analyzed using Pearson and Spearman correlations, as well as simple linear regression. Differences in SR, Max SR, AUC, and ΔSR across pH conditions and formulations were analyzed using one-way ANOVA. One-way ANOVA was performed after normality (Shapiro–Wilk) and homogeneity (Levene's test) assumptions were satisfied. Post-hoc analysis was conducted using Tukey HSD at $p < 0.05$. All experimental procedures were performed under standardized laboratory conditions to ensure consistency and reproducibility of the measurements.

2.2 Data analysis

2.2.1 Descriptive analysis

Swelling data were measured in triplicate and plotted over time for each pH condition and hydrogel type (ChGu, ChG, ChPu, and ChP) to identify patterns in initial swelling kinetics. This analysis aimed to capture the rapid initial absorption phase, the approach to equilibrium, and any changes in the curve's slope that may indicate transitions in swelling dynamics. Mean values and standard deviations were calculated from replicate measurements at each pH condition and time point, with three independent replicates ($n = 3$) for each measurement.

2.2.2 Initial swelling kinetics analysis

The analysis of initial swelling kinetics was conducted to describe the time-dependent expansion behavior of chitosan-based hydrogels in response to interactions with a liquid medium (Nunziata et al., 2025; Yang et al., 2025). According to Márton et al. (2024), swelling is generally governed by two primary mechanisms: solvent diffusion into the polymer matrix and polymer chain relaxation, which lead to network expansion. Accordingly, this study adopted a descriptive-empirical kinetic approach because the limited number of observation points was better suited to identifying early swelling patterns than to fitting complex kinetic models. The swelling ratio (SR) was defined as a function of time as follows:

$$SR(t) = \frac{W_t - W_0}{W_0} \times 100\% \qquad \text{(Eq.1)}$$

Where W_t represents the hydrogel weight at time t and W_0 represents the initial dry weight. This parameter served as the primary indicator for evaluating material expansion at each observation interval. The analysis examined the relationship between time and SR across different samples and pH conditions. To strengthen interpretation, additional design indicators were used, including maximum swelling ratio (Max SR), area under the curve (AUC) over the 0–120 minute range, and net swelling change (ΔSR) between the initial (0 min) and final observation (120 min) to determine whether the system was dominated by swelling or deswelling kinetics. Additionally, an operational indicator of dynamic transition was introduced, defined as the time interval during which the largest change in swelling occurs. This indicator was not intended to precisely determine mechanistic transition points, but rather to provide an initial analytical reference for identifying phases in which the hydrogel response changes most significantly over the observation period. Given the limited number of time points, interpretation of this indicator was conducted cautiously and considered preliminary rather than definitive.

2.3 Methodological limitations

This study focuses on the initial swelling kinetics of chitosan-based hydrogels under varying pH conditions to provide a basis for understanding mechanistic aspects of material design. It does not include direct nutrient release testing, soil biodegradability assessment, or agronomic performance evaluation in plant systems. Furthermore, the analysis of the relationship between DD and swelling kinetics is exploratory and reflects associations between base chitosan characteristics and hydrogel formulation response rather than direct causal relationships within each final matrix. Due to the limited number of replicates and data points, correlation and regression analyses are intended as preliminary exploratory interpretations rather than strong inferential conclusions. Thus, the findings provide an initial mechanistic basis for future research toward developing biopolymer-based CRF applications.

3. Results and Discussion

3.1 Degree of deacetylation characteristics and structural characterization of chitosan

The degree of deacetylation (DD) is the proportion of free amine groups ($-NH_2$) in the chitosan structure and chemically determines the potential charge density of the polymer matrix. These $-NH_2$ groups can undergo protonation to $-NH_3^+$ under acidic conditions and deprotonation under neutral to alkaline conditions. As a result, DD functions not only as a characterization parameter but also as a structural variable that influences pH responsiveness, electrostatic interactions, and the network expansion behavior of hydrogels. To capture the evolution of chemical structure during the stepwise treatment process, changes in DD values at each stage of synthesis were quantitatively analyzed. This visualization is essential for evaluating the consistency of the deacetylation process while also identifying potential non-monotonic patterns across treatment stages. The measured DD values at each stage are presented in Figure 1.

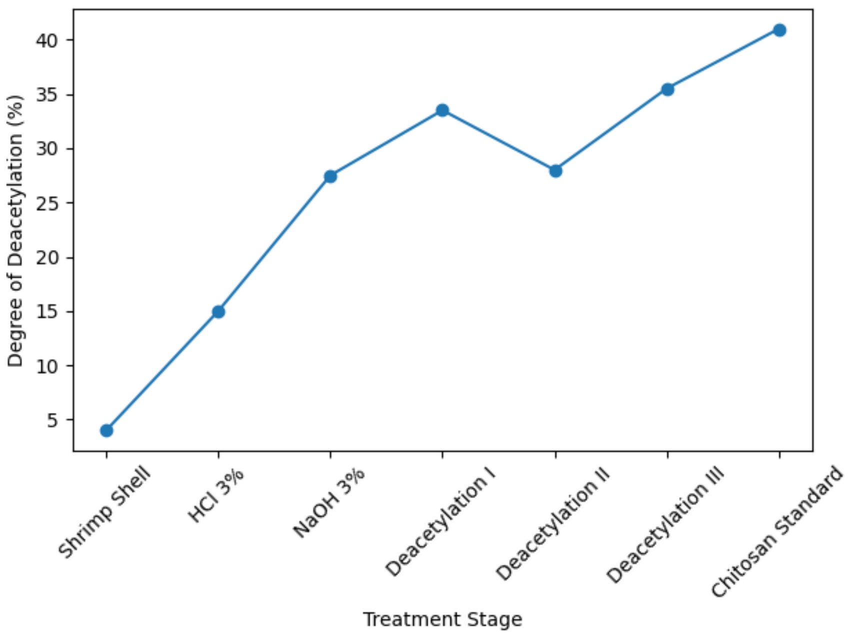

Fig. 1. Changes in the degree of deacetylation at each treatment stage

The degree of deacetylation (DD) values in Figure 1 exhibit a gradual increase; however, the pattern is not entirely linear. At the initial stage, the very low DD value of 4.24% indicates that the material is still dominated by acetylated units and does not yet have sufficient free amine groups to exhibit chitosan's responsive characteristics. The limited number of $–NH_2$ groups results in low protonation under acidic conditions, leading to minimal electrostatic repulsion between polymer chains and thereby maintaining a relatively compact network structure. Consequently, the network remains dense, and the swelling capacity is not yet optimal. This observation is consistent with previous studies emphasizing that the formation of free amino groups during deacetylation underlies the emergence of reactivity, cationic charge, and pH-responsive behavior in chitosan (Hong et al., 2024; Budianto & Amalia, 2020).

A more pronounced increase in DD is observed following treatment with 3% HCl and 3% NaOH, and this trend continues through subsequent deacetylation stages. The DD increases from 14.98% after 3% HCl treatment to 27.63% after 3% NaOH treatment, and further to 33.33% after deacetylation I. This increase reflects a growing density of free amine groups that can be protonated under acidic conditions (Klein & Poverenov, 2020). Theoretically, this relationship is widely recognized as the basis of pH responsiveness in chitosan hydrogels, where a higher number of ionizable amino groups enhances the potential for swelling changes in response to environmental pH variations (Rodríguez-Rodríguez et al., 2025; Kumar et al., 2023). Therefore, the increasing DD trend observed in this study is consistent with the general understanding that deacetylation enhances the material's responsiveness, although it does not directly quantify the magnitude of swelling without functional measurements.

However, the results also indicate that the increase in DD is not strictly monotonic. At the deacetylation II stage, the DD decreases to 28.12%, then increases to 35.48% at deacetylation III, and ultimately reaches 40.93% in the chitosan standard. This temporary decline suggests heterogeneous reactions, non-uniform alkali distribution within the matrix, partial over-treatment, or structural reorganization leading to localized degradation (Hellmann et al., 2024; Kaya et al., 2017). These findings indicate that the deacetylation process does not necessarily follow a simple linear pattern but is influenced by the complexity of reaction kinetics and base diffusion within the polymer network. Furthermore, DD variation is governed not only by the intensity of chemical treatment but also by the material's intrinsic properties during processing.

At higher DD levels, approximately 35–41%, as observed in deacetylation III and the chitosan standard, the density of $–NH_2$ groups increases, thereby conceptually enhancing the material’s sensitivity to pH changes. Under acidic conditions, the higher number of protonated groups is expected to strengthen electrostatic repulsion and promote structural expansion (Hellmann et al., 2024; Klein & Poverenov, 2020). Nevertheless, this structural potential does not automatically guarantee improved functional performance, as its expression depends on the environmental conditions under which the hydrogel operates (Hellmann et al., 2024; Márton et al., 2024). As a result, the relationship between DD and swelling response in this study is more appropriately interpreted as an initial structural tendency rather than a directly measurable mechanistic relationship based solely on the available data.

Compared with findings from other studies, these results are noteworthy because they represent a DD trajectory in the low-to-moderate range, whereas many contemporary chitosan applications use materials with significantly higher DD values. Namli et al. (2023) reported that chitosan with DD values ranging from approximately 65.0% to 93.1% demonstrates that variations in DD, together with molecular weight, significantly influence aerogel structure, porosity, and water absorption capacity. They also found that higher DD at lower molecular weight tends to correlate with increased water uptake. In this context, the data from the present study represent an early transitional phase in which the material has not yet reached the high-DD category. As a result, the primary value of these findings lies not in achieving maximum swelling performance but in illustrating the gradual development of material responsiveness during the deacetylation process.

Compared with the study by Márton et al. (2024), swelling in native chitosan coatings increases significantly as pH decreases, from approximately 250% to 500%, due to increased protonation of amino groups. In contrast, more acetylated coatings exhibit significantly lower swelling and minimal sensitivity to pH changes. These findings support the assumption that low DD values in the early stages of this study are associated with weak swelling responses, while higher DD values at later stages may enhance sensitivity in acidic media. However, other studies emphasize that the swelling performance of chitosan hydrogels is determined not only by DD but also by the type of crosslinking, molecular weight, network density, ionic composition of the medium, temperature, and degradation stability (Yadav et al., 2023; Yildirim et al., 2025). Thus, in this study, DD is more appropriately interpreted as an initial structural parameter reflecting the potential for pH responsiveness, rather than as a single factor that directly determines the magnitude of swelling. Overall, the results presented in Figure 1 indicate that DD is an important indicator of the evolution of chitosan's chemical structure toward increasingly responsive materials, although functional performance must still be verified through swelling tests across various pH conditions.

3.2 Early kinetic pattern of swelling under pH variations

To obtain a comparative overview of the dominance of pH as a factor influencing hydrogel responsiveness, the mean swelling ratio (SR) values along with their standard deviations are summarized in Figure 2. This visualization compares the degree of expansion across environmental conditions and illustrates the magnitude of variability in structural response for each treatment. One-way ANOVA showed significant differences in swelling kinetics among the tested pH conditions, $F(2.33) = 18.42$, $p = 0.001$, $\eta^2 = 0.62$.

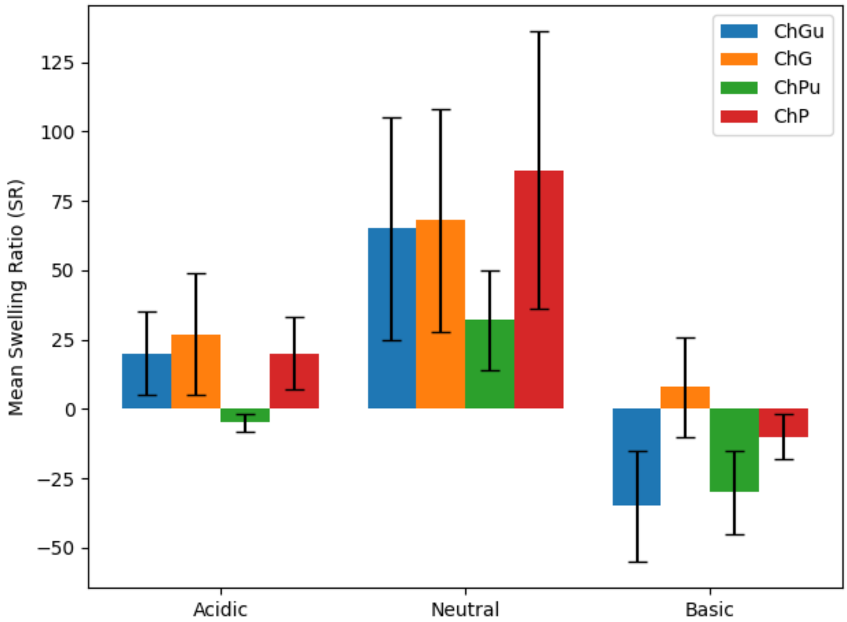

Fig. 2. Mean SR at each pH condition along with standard deviation

Descriptively, variation in swelling kinetics across pH conditions was more pronounced than variation among formulations in the observed dataset. Unlike previous studies focusing on equilibrium swelling or diffusion-only models, this study introduces early-stage swelling kinetics over 0–120 min as a predictive framework integrating multiple indicators, including Max SR, AUC, ΔSR, and transition time, for controlled-release fertilizer design. This approach provides a simplified but application-oriented framework for evaluating hydrogel performance in agricultural systems (Rodríguez-Rodríguez et al., 2025; Kumar et al., 2023). Under acidic conditions, ChG and ChP exhibited greater expansion than

ChGu and ChPu, although the overall swelling response remained limited. In acidic media, protonation of chitosan amino groups to $-NH_3^+$ may increase electrostatic repulsion between polymer chains, thereby promoting network expansion (Dou et al., 2023; Li et al., 2022). However, the magnitude of swelling also depends on the internal network structure, crosslinking density, and composition of the hydrogel matrix.

Under neutral conditions, a marked increase in mean swelling ratio was observed across all formulations. ChP recorded the highest mean SR value (86.4), followed by ChG (67.6), ChGu (63.8), and ChPu (30.4). Thus, neutral pH produced the most favorable swelling response among the tested conditions. The relatively large standard deviations under neutral conditions indicate considerable variability among replicates, suggesting that the structural responses were not entirely homogeneous. This variability may be associated with microstructural heterogeneity, pore distribution, differences in crosslinking density, and polymer-chain relaxation dynamics (Namli et al., 2023; Roas-Escalona et al., 2024). Because specific post-hoc comparisons among formulations are not reported, differences among the formulations are interpreted descriptively.

Under alkaline conditions, all samples exhibit a drastic decrease, with values even negative, particularly for ChGu (−34.1), ChPu (−32), and ChP (−10.7). These negative values indicate deswelling, which may occur because deprotonation of amino groups reduces positive charge density, weakens electrostatic repulsion, promotes chain contraction, and decreases water retention within the hydrogel network. Under such conditions, the reduction of positive charges within the matrix decreases electrostatic repulsion between polymer chains, leading to network collapse. As a result, Figure 2 in this study clearly demonstrates that pH is the most dominant factor controlling the swelling response in the tested hydrogel system. These descriptive findings are further supported by early swelling kinetic design indicators, which capture not only the magnitude of expansion but also cumulative stability and the final direction of structural change. Accordingly, these indicators are visualized quantitatively in Figure 3 using Max SR, AUC (0–120 minutes), and ΔSR.

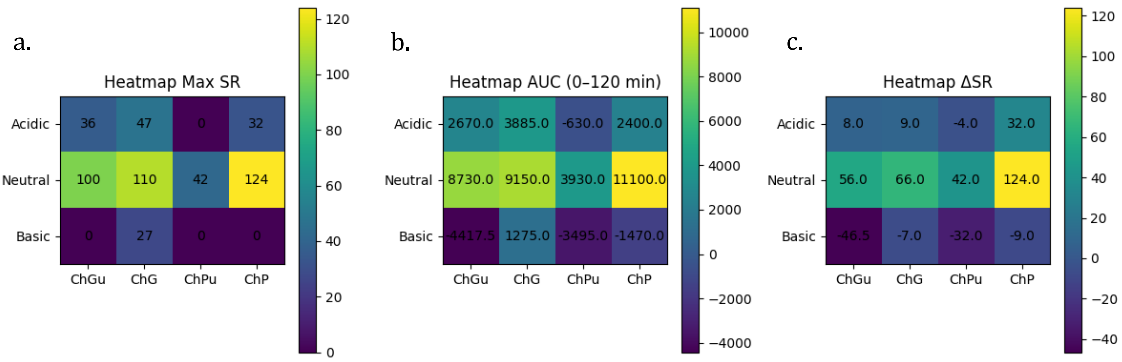

Fig. 3. Early swelling kinetic design indicators: (a) Max SR, (b) AUC 0–120 minutes, and (c) ΔSR

Statistical analysis confirmed significant differences across pH conditions. Figure 3a shows that the highest Max SR values are consistently observed under neutral conditions for all samples, particularly ChP (124) and ChG (110). This confirms that the hydrogel's maximum expansion capacity is achieved at neutral pH. Under acidic conditions, Max SR values are moderate, whereas under alkaline conditions, they decrease drastically to zero in some samples, indicating the network's inability to maintain expansion in an alkaline environment. A similar pattern is observed in Figure 3b, where the highest AUC values again occur under neutral conditions, reaching 11,100 for ChP and 9,150 for ChG. High AUC values indicate sustained expansion during the observation period, which may support prolonged water retention and provide a more stable hydrated matrix for future nutrient-release applications. Conversely, negative AUC values under alkaline conditions, particularly for ChGu (−4417.5) and ChPu (−3495), indicate that contraction dominates over expansion during the observation period.

Figure 3c presents ΔSR values, which describe the final direction of swelling dynamics. Under neutral conditions, large positive ΔSR values indicate that the hydrogel maintains its expansion throughout the observation period, particularly for ChP (124), ChG (66), ChGu (56), and ChPu (42). In contrast, negative values under alkaline conditions, such as −46.5 for ChGu and −32 for ChPu, confirm progressive shrinkage. Under acidic conditions, ΔSR values are relatively small and variable, indicating that expansion is more limited and less stable than under neutral conditions.

Overall, the patterns observed in Figures 2 and 3 confirm that pH is the primary controlling factor governing hydrogel swelling dynamics in this system. The observed swelling patterns are consistent with the combined contributions of solvent diffusion and polymer-chain relaxation, although dedicated kinetic modeling is required to confirm the underlying transport mechanism. In the context of chitosan-based controlled-release fertilizer (CRF) material design, these findings are consistent with the literature describing chitosan hydrogels as environmentally responsive carriers, where release and retention are influenced by swelling kinetics, network structure, and medium conditions (Eddarai et al., 2024; Liu et al., 2024).

3.3 Relationship between degree of deacetylation (DD) and swelling

To understand the relationship between the material's chemical characteristics and its physical response in the hydrogel system, the association between chitosan's DD and swelling performance was analyzed quantitatively. Theoretically, DD represents the proportion of free amine groups within the polymer structure that contribute to hydration capacity, ionic interactions, and sensitivity to pH changes. This suggests that analyzing the relationship between DD and swelling indicators is essential for evaluating the extent to which changes in chitosan's chemical structure contribute to hydrogel expansion behavior (Žigrayová et al., 2024; Hong et al., 2024).

The relationship between DD and swelling indicators was examined using an exploratory approach due to the limited number of data points; thus, the results do not demonstrate strong statistical significance. Accordingly, the findings in this subsection should be interpreted as preliminary indications of structure–function relationships rather than conclusive inferential evidence. The results of the correlation and regression analyses are presented in Table 1.

Table 1. Relationship between DD and Swelling Indicators

Analysis	Parameter	Value	*p-value*
Pearson (DD and Max SR)	r	0.21	0.65
Spearman (DD and Max SR)	ρ	0.25	0.60
Regression (DD and ΔSR)	β	3.87	0.47
Coefficient of Determination	R^2	0.14	–

The Pearson correlation coefficient (r = 0.21, p = 0.65) and the Spearman correlation coefficient (ρ = 0.25, p = 0.60) suggest that there is no significant linear or monotonic relationship between DD and maximum swelling values. Linear regression analysis yielded a coefficient β = 3.87, R^2 = 0.14, and p = 0.47, indicating that only approximately 14% of the variation in swelling is explained by changes in DD. This suggests that DD alone is insufficient to predict swelling kinetics without considering the network architecture and environmental pH. Theoretically, increasing DD should enhance the number of free amine groups ($-NH_2$), which participate in protonation–deprotonation mechanisms, thereby potentially strengthening electrostatic interactions and increasing hydration capacity (Hong et al., 2024; Wang et al., 2019). However, the empirical results demonstrate that increases in DD are not consistently followed by increases in swelling.

In relation to the findings, variations in pH produce much more significant changes in the swelling response than variations in DD. This observation aligns with the literature indicating that chitosan hydrogels are pH-responsive materials, as changes in pH directly affect the degree of ionization of amino groups, ultimately altering swelling and deswelling kinetics (Rodríguez-Rodríguez et al., 2025; Yadav et al., 2023). Furthermore, neutral conditions in this study consistently result in maximum expansion, whereas alkaline conditions induce deswelling in nearly all samples. This pattern indicates that pH acts as the primary controlling factor through amino group ionization mechanisms, while DD is more appropriately interpreted as a structural parameter that provides the capacity for responsiveness but does not fully determine how that response is expressed under specific environmental conditions (Roas-Escalona et al., 2024; Rodríguez-Rodríguez et al., 2025).

In addition, the weak relationship between DD and swelling suggests the influence of other factors within the system, such as the spatial distribution of amine groups within the matrix, microstructural heterogeneity, degree of crosslinking, and polymer chain relaxation dynamics. Recent literature indicates that the performance of chitosan hydrogels is governed not only by DD but also by crosslinking strategies, network density, porosity, and medium characteristics, which collectively regulate hydration capacity, permeability, and swelling stability (Budianto & Amalia, 2020; Carpa et al., 2022). Variations in these factors may lead to cases where two samples with different DD values exhibit non-parallel swelling kinetics. This suggests that the structure–function relationship in chitosan hydrogels in this study does not follow a simple deterministic pattern. DD may be considered a necessary factor for enabling responsiveness to pH changes, but it is not sufficient to guarantee improved swelling performance.

These findings indicate that swelling kinetics is more strongly influenced by environmental conditions that control the degree of amino-group ionization than by intrinsic structural capacity alone. In practice, this implies that the design of chitosan-based hydrogels should not focus solely on optimizing chemical parameters but should also consider compatibility with the intended environmental conditions. This is consistent with recent studies on chitosan-based hydrogel fertilizer systems, which demonstrate that water retention and nutrient release behavior are strongly influenced by the material's response to medium pH. Because the relationship between DD and swelling in this dataset does not exhibit strong statistical patterns, subsequent analysis is better focused on interpreting swelling dynamics over time using early kinetic indicators that emphasize response patterns across time and pH conditions. This approach is methodologically more cautious, as it avoids extending inferential claims beyond the support of the available data.

3.4 Indications of dynamic transition as a basis for preliminary interpretation

The analysis of dynamic transition indicators shows that hydrogel swelling does not occur uniformly but instead exhibits different response rates across pH conditions. In general, neutral conditions tend to exhibit a more stable expansion phase, whereas alkaline conditions tend to show faster transitions leading to deswelling. In this study, indications of dynamic transition were operationally defined as the time interval during which swelling changed most. The results should not be interpreted as precise mechanistic transition points but rather as preliminary markers for identifying dominant response phases during the observation period. The results of the dynamic transition point indication ($t*$), determined based on the interval with the highest swelling rate change, are presented in Table 2. Based on these results, most samples exhibit their primary changes during the early observation phase, specifically within the 0–30 minute interval. This finding indicates that the hydrogel's response to the medium is strongest immediately after initial contact. Literature on responsive hydrogels also suggests that the early swelling phase is typically dominated by rapid solvent uptake, followed by a slower relaxation phase toward a more stable state (Protsak et al., 2025; VanZanten et al., 2024). In this study, the midpoint of each interval was used as an operational approximation to facilitate pattern interpretation; therefore, t = 15 minutes should be understood as a descriptive representation of the dominant response interval rather than a precise measurement at a single time point.

Table 2. Indication of dynamic transition points (t^*) based on intervals with the highest swelling rate changes under different pH conditions

pH Condition	Sample	Interval of Greatest Change	t* (min)
Acidic	ChG	0–30	15
	ChGu	0–30	15
	ChP	0–30	15
	ChPu	0–30	15
Neutral	ChG	0–30	15
	ChGu	0–30	15
	ChP	0–30	15
	ChPu	0–30	15
Basic	ChG	60–90	75
	ChGu	0–30	15
	ChP	0–30	15
	ChPu	0–30	15

However, for the ChG sample under alkaline conditions, the main transition point is identified at a later interval, namely 60–90 minutes (equivalent to t = 75 minutes). This pattern suggests that the system undergoes a delayed transition phase compared to other samples. Conceptually, this delay is consistent with studies indicating that early swelling kinetics can be slowed by internal network constraints, variations in medium properties, and differences in crosslinking density that influence relaxation time and mass transfer rates (VanZanten et al., 2024; Piroonpan et al., 2024). Thus, the exception observed in ChG under alkaline conditions indicates that not all samples respond identically, even at the same pH.

With this approach, swelling analysis is not limited to the magnitude of expansion but also accounts for the temporal evolution of the response. In this study, dynamic transition indicators are proposed as a preliminary interpretative tool for assessing the temporal stability of hydrogels. Materials that maintain expansion for a longer duration may be considered more promising as water-retention matrices, whereas those that rapidly transition to deswelling indicate limited stability under certain conditions. The relevance of this temporal approach is supported by studies on chitosan hydrogels for controlled-release and water-retention applications, which demonstrate that material performance is determined not only by maximum swelling capacity but also by the stability of its response over time and across different media. In the context, this indicator should be positioned as a preliminary analytical basis that requires further validation in future studies with higher temporal resolution.

3.5 Implications for the design of biodegradable CRF hydrogels

The results of this study indicate that early swelling kinetics can serve as an initial mechanistic indicator for evaluating hydrogel performance in controlled-release fertilizer (CRF) applications. Although direct nutrient-release testing was not conducted, the observed swelling patterns provide a preliminary basis for estimating how hydration behavior may influence nutrient diffusion pathways in future CRF formulations. In this context, an early swelling–kinetics–based approach provides a framework for linking hydration phases to nutrient-release dynamics and serves as a preliminary mechanistic basis for data-driven development of biopolymer-based CRF materials.

Maximum swelling, typical of most agricultural soils (pH 5.5–7), occurs under near-neutral conditions. For acidic soils, liming or pH-buffering adjustments can enhance swelling performance, whereas for alkaline soils, crosslinking optimization, stabilizing additives, or blending with other biopolymers can mitigate deswelling and improve field applicability. These results indicate that near-neutral pH produced the most favorable swelling response among the tested conditions, but do not imply that hydrogel applicability is restricted to such soils. In practical applications, site-specific soil management and formulation engineering can extend the effective use of chitosan-based hydrogel CRF matrices across heterogeneous soil conditions.

Chitosan hydrogels may function as water-retention matrices by maintaining a hydrated network in the root zone. This potential application requires confirmation through nutrient-release, soil-column, and field-performance testing (Eddarai et al., 2024; VanZanten et al., 2024). Conversely, deswelling observed under alkaline conditions reflects limited structural stability due to reduced amino-group protonation and polymer network contraction, emphasizing pH responsiveness as a key factor in hydrogel performance and field applicability (Mikhailidi et al., 2024; Behera et al., 2020).

Dynamic transition indicators (t*) provide reference points for when hydrogel swelling shifts from rapid absorption to stabilization, informing practical application timing and stability assessment across various soil environments. These indicators help estimate the onset of significant changes in swelling rate, guiding future studies on the relationship between swelling dynamics and nutrient release. Together with early kinetic markers, this approach allows CRF design to move beyond empirical methods toward a mechanistically informed framework.

The hydrogel's pH-responsive behavior regulates functional-group ionization, thereby affecting swelling capacity and network stability. Soil pH also influences nutrient solubility, ion mobility, and microbial activity (Mikhailidi et al., 2024; Thirupathi et al., 2022), highlighting the importance of evaluating early swelling kinetics under different pH conditions to support agroecosystem adaptability. Although nutrient-release and soil biodegradability testing were not included, these findings provide a mechanistic foundation for further development. Overall, chitosan-based hydrogel performance arises from interactions among structural characteristics, environmental conditions, and early kinetic dynamics rather than a single factor, supporting precise, data-driven development of CRF systems aligned with sustainable agricultural practices.

4. Conclusions

This study demonstrates that the swelling performance of chitosan-based hydrogels is influenced by interactions between structural characteristics and environmental pH, with pH exhibiting the most pronounced descriptive effect in the observed dataset. The degree of DD increases during the synthesis stages, though not strictly linearly, and its relationship with swelling performance remains weak, suggesting that increases in DD do not necessarily improve swelling behavior. In contrast, pH variation exerts a more pronounced influence: neutral conditions result in maximum expansion and greater swelling stability, whereas alkaline conditions induce deswelling and reveal limitations in the hydrogel's structural stability. Based on the observed swelling kinetics, chitosan hydrogels show potential as biopolymer-based controlled-release fertilizer (CRF) matrices. Neutral pH produced the most favorable swelling response among the tested conditions; in acidic environments, liming or formulation adjustments may improve performance, whereas in alkaline conditions, crosslinking optimization or stabilizing additives may help maintain swelling stability and broaden environmental applicability. Future studies should include nutrient-release experiments, soil biodegradability assessment, higher temporal resolution, and field-scale performance evaluation under different soil pH conditions.

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Conceptualization, S.R.P.M.; Methodology, S.R.P.M.; Software, S.R.P.M.; Validation, S.R.P.M., T.S.B., and A.D.M.; Formal Analysis, S.R.P.M. and K.A.; Investigation, S.R.P.M., A.D.M., and F.K.; Resources, T.S.B. and K.A.; Data Curation, S.R.P.M. and A.D.M.; Writing – Original Draft Preparation, S.R.P.M.; Writing – Review & Editing, T.S.B., K.A., and A.N.R.; Visualization, S.R.P.M.; Supervision, T.S.B. and A.N.R.; Project Administration, S.R.P.M.; Funding Acquisition, T.S.B. and K.A.

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Ethical Review Board Statement

Ethical approval was not required for this study, as it does not involve human or animal participation and is unrelated to public health or safety issues.

Informed Consent Statement

Not available. This study does not involve human participants or identifiable personal data.

Data Availability Statement

The data generated and analyzed during this study are available from the corresponding author upon reasonable request. No publicly archived dataset is currently available.

Conflicts of Interest

The authors declare no conflict of interest.

Declaration of Generative AI Use

During the preparation of this work, the authors used ChatGPT to assist in drafting, paraphrasing, and improving the academic language of the manuscript, and Grammarly to enhance grammar, clarity, and readability. After using these tools, the authors reviewed and edited the content as needed and took full responsibility for the content of the publication.

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