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Development and Characterization of Chitosan-modified PVA/Borate Hydrogels for Controlled-release of Potassium Chloride

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Abstract

Controlled-release fertilizers (CRFs) are increasingly developed to enhance nutrient use efficiency (NUE) and reduce leaching losses in sustainable agriculture. The objective of this study was to developed poly (vinyl alcohol) (PVA)/borate/chitosan hydrogels as a controlled release carrier for potassium chloride (KCl) nutrient. The hydrogel composition was systematically optimized and the formulation with a PVA: borate: chitosan volume ratio of 8:1:5 (with 1 mL of 1 M KCl) exhibited the best structural homogeneity and mechanical integrity. Fourier-transform infrared (FTIR) spectroscopy revealed hydrogen bonding interactions between PVA and chitosan, as well as possible ionic interactions between K^+ ions and borate groups, contributing to network stabilization. Compared to PVA/borate-KCl CRF, the chitosan-modified system exhibited significantly higher swelling capacity (750.7% vs. 642.5%), prolonged water retention (234 h vs. 216 h) and a slower, more controlled nutrient release profile. Kinetics analysis showed that the release behaviour was fit described by second-order kinetics model, indicating that the rate-determining step of the release process is the hydration of the ions in the system. The improved performance of the CRF might be attributed to increased network compactness, enhanced intermolecular interactions and higher diffusion resistance within the hydrogel matrix. These findings demonstrated that the incorporation of chitosan effectively enhances both structural stability and release kinetics control of PVA/borate hydrogels, highlighting their potential as environmentally friendly matrices for sustainable potassium fertilizer delivery.

Keywords: Agrochemical, Chitosan, Controlled-release Fertilizer, Hydrogel, PVA/Borate/Chitosan-KCl.

Introduction

Fertilizers play an essential and highly significant role in supporting global food security by increasing agricultural productivity, improving crop yields and helping farmers meet the growing demand for food (1, 2). In addition, fertilizers contribute to the maintenance and enhancement of soil fertility by replenishing essential nutrients that are depleted through continuous cultivation, thereby promoting sustainable agricultural production over time. Among commonly used fertilizers, potassium chloride (KCl) is preferred due to its high potassium content and relatively low cost (3, 4). However, the high solubility of KCl makes it prone to leaching losses during rainfall or irrigation, resulting in poor nutrient use efficiency (5). Studies have shown that formulation improvements, such as granulated or coated fertilizers, can significantly reduce potassium leaching and improve nutrient retention (6). To address this issue, controlled-release fertilizers (CRFs) have been developed to minimize nutrient losses, extend nutrient supply duration and improve overall fertilizer efficiency (7, 8). Recent review studies have increasingly emphasized the importance of controlled-release fertilizers (CRFs) as innovative and highly effective tools for supporting sustainable and climate-smart agricultural systems. These fertilizers are designed to release nutrients gradually and in a more controlled manner, allowing plants to absorb essential nutrients according to their growth requirements over time (9-11). As a result, CRFs can significantly improve nutrient use efficiency, minimize nutrient losses through leaching, runoff and volatilization and reduce the frequency of fertilizer applications. Furthermore, their use contributes to lowering environmental pollution, mitigating the negative impacts of excessive

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fertilizer inputs on soil and water quality and promoting more environmentally responsible agricultural practices that are aligned with long-term sustainability and climate resilience goals. Hydrogels have been extensively studied and widely explored as promising matrix materials for controlled-release fertilizers (CRFs) due to their remarkable capacity to absorb and retain large amounts of water (12). Furthermore, their unique three-dimensional polymer network structure enables them to effectively regulate and control the release of nutrients over an extended period, thereby improving nutrient-use efficiency, reducing nutrient losses and supporting more sustainable agricultural practices. Poly (vinyl alcohol) (PVA) is a hydrophilic polymer often used in hydrogel preparation due to its biocompatibility and non-toxicity. However, pure PVA hydrogels are mechanically weak and highly soluble in water due to the abundance of hydroxyl groups (13). Crosslinking with borate improves hydrogel stability and swelling behavior (14), but PVA/borate hydrogels still suffer from poor mechanical strength (15).

Potassium (K) is an essential macronutrient that underpins multiple physiological and biochemical processes in plants. It serves as a cofactor for enzymes involved in photosynthesis, protein synthesis and carbohydrate metabolism, thereby enhancing overall metabolic efficiency and biomass accumulation (16). Moreover, K plays a pivotal role in maintaining osmotic balance and turgor pressure, regulating stomatal aperture and optimizing water use efficiency (4). Its function in assimilate translocation, particularly the movement of sugars and amino acids from source to sink tissues, significantly influences yield formation, fruit quality and postharvest performance (17). In addition, adequate K nutrition enhances plant resilience to abiotic stresses - including drought, salinity and temperature extremes - through membrane stabilization, ion homeostasis and activation of antioxidant defense systems (18). Conversely, a deficiency of potassium (K) can significantly impair plant growth and development by disrupting various physiological and biochemical processes (19). Insufficient potassium availability reduces photosynthetic efficiency, limits the plant's ability to effectively utilize water and nutrients and weakens its tolerance to both biotic and abiotic stresses, such as drought, salinity and disease. These adverse effects highlight the crucial and irreplaceable role of potassium in maintaining plant health, optimizing crop performance and achieving sustainable agricultural productivity over the long term.

Chitosan, which is a naturally derived biopolymer obtained through the deacetylation of chitin, has been increasingly incorporated into poly (vinyl alcohol) (PVA)/borate hydrogel systems to improve their overall structural integrity, mechanical strength and long-term stability. The addition of chitosan also helps decrease the solubility of the hydrogel matrix in aqueous environments, thereby enhancing its durability and controlled-release performance. To date, the application of PVA/borate/chitosan hydrogels has been investigated predominantly in the biomedical sector, particularly for advanced drug delivery systems, tissue engineering, wound dressing materials and other healthcare-related technologies that require precise control over the release of active compounds (20-24). In the biomedical field, chitosan-based hydrogels are a highly sought-after material due to their various advantages, such as relatively low production costs, good biocompatibility and high biodegradability. These hydrogels can be synthesized through cross-linking of pure chitosan or chitosan combined with other polymers. They can be formed through chemical or physical cross-linking, each of which produces different material characteristics and properties (25). However, despite the potential benefits of chitosan incorporation, there is limited research specifically investigating PVA/borate/chitosan hydrogels as agricultural nutrient-delivery systems-particularly for potassium chloride (KCl) fertilizers, although chitosan-based hydrogels have been explored for controlled nutrient release and fertilizer coatings (26, 27).

Although chitosan-modified hydrogels have been widely explored in biomedical applications, their incorporation into borate-crosslinked PVA systems for potassium-based controlled-release fertilizers remains insufficiently investigated. In particular, systematic evaluation concerning its characteristics and performance as slow/controlled-release fertilizer in the presence of chitosan remains scarce. Therefore, this study aims to develop and characterize PVA/borate/chitosan hydrogels as matrices for KCl controlled release fertilizer and to elucidate the mechanisms governing their performance, such as swelling behavior, water retention and release kinetics. The results of this study will provide valuable new insights for the development of agrochemical materials with a slow/controlled release strategy.

Materials and Methods

Materials

The materials used in this study were potassium chloride and acetic acid (p.a. grade, Merck, Germany), fully hydrolysed poly (vinyl alcohol) (MW = 60000 g/mol), sodium borate and chitosan (technical grade). Aqua-dm was used throughout all experiments. The instruments employed in this study included a conductometer (Mettler toledo series LE703, United Kingdom), pH meter (Mettler toledo, United Kingdom), magnetic stirrer, digital screw micrometer and Fourier-transform infrared spectrophotometer (Shimadzu, Japan).

Methods

Preparation of CRF hydrogels was carried out by mixing poly (vinyl alcohol) (PVA, 3% w/v), borate (4% w/v) and chitosan (0.3% w/v) solutions in different ratios, as shown in Table 1. To each mixture, 1 mL of 1 M KCl solution was added, followed by stirring with a magnetic stirrer and heating on a hot plate at 40-50°C until a homogeneous solution was obtained. The resulting solution was poured into 10 cm × 10 cm acrylic molds and dried at room temperature. The dried hydrogels were then cut into 2 cm × 2 cm flakes. For comparison, hydrogels of PVA/borate, PVA/borate-KCl and PVA/borate/chitosan were also prepared in selected compositions (based on optimization results) and used as reference samples for characterization and performance analysis.

In Table 1, the volume quantity of PVA and borate used in the first 6 compositions were 4:1. This was based on preliminary study where chitosan was not yet incorporated into the mixture. This composition was selected over others due to the excellent texture of the resulting PVA/borate hydrogel. We then introduced chitosan into the mixture, thus making the first 6 hydrogels having composition of 4:1:x, where x is the volume of chitosan that was introduced into the mixture. To anticipate the changes in the property of hydrogel due to the incorporation of chitosan into PVA/borate mixture, we also evaluate other compositions, namely the PVA: borate: chitosan ratio of 8:1:5 and 4:5:5, which were predicted based on the results from previous composition.

Table 1: Composition Variations of PVA/Borate/Chitosan Hydrogels

PVA 3% (mL)	Borate 4% (mL)	Chitosan 0.3% (mL)	KCl 1M (mL)
4	1	0.5	1
4	1	1	1
4	1	2	1
4	1	3	1
4	1	4	1
4	1	5	1
8	1	5	1
4	5	5	1

Fourier-transform infrared (FTIR) spectroscopy characterization was performed using a Shimadzu 8400 spectrophotometer. Hydrogel flakes were cut into small pieces, mixed with potassium bromide (KBr) at a ratio of 1:1000 and pressed into pellets. Spectra were recorded over the wavenumber range of 4000-500 cm^{-1}.

Hydrogel thickness was determined by first immersing hydrogel samples (4 cm × 4 cm) in aqua-dm for 30 seconds, then removed and immediately measured using a digital screw micrometer. Thickness measurements were recorded every 30 seconds over a 30-minute period to account for structural relaxation and water absorption effects. The reported values represent the average of three independent measurements.

Swelling ratio of hydrogels were determined by immersing hydrogel samples (2 cm × 2 cm) in 10 mL of aqua-dm for 3 minutes, removed, gently blotted to remove excess surface water and weighed. The procedure was repeated every 3 minutes until a constant mass was obtained (about 1 hour).

The swelling ratio (%SR*)* was calculated using the Equation [1]:

$$\%SR = \frac{W_t - W_0}{W_0} \times 100 \qquad [1]$$

Where, W_t is the swollen mass at time t and W_0 is the initial dry mass.

Water retention was determined as follows: After swelling equilibrium was reached (1 hour of immersion), hydrogel samples were weighed and then air-dried under ambient conditions. The mass was recorded twice a day (morning and afternoon) for over several days until a constant mass was reached.

Water retention (%WR) was calculated using the Equation [2]:

$$\%WR = \frac{(W_t - W_\infty)}{(W_o - W_\infty)} \times 100\% \qquad [2]$$

Where, W_t is the hydrogel mass at a given time, W_0 is the initial swollen mass and W_∞ is the final constant mass after drying.

Release behavior was determined by first immersing dried hydrogel samples in 500 mL of aqua-dm, continuously agitated at 200 rpm using an overhead stirrer. The conductivity of the medium was recorded at 30-second intervals until a constant value was obtained.

Results and Discussion

In terms of physical appearance, as shown in Figure 1, the optimum composition of CRF prepared from poly (vinyl alcohol), borate, chitosan and potassium chloride (abbreviated as PVA/borate/chitosan-KCl) was achieved when the volume ratio of PVA:borate: chitosan was 8:1:5 (case number 7 at Table 1), where the volume of KCl solution was 1 mL. The CRF produced with this composition formed a homogeneous, smooth and mechanically robust hydrogel. In contrast, the CRF prepared in other compositions were brittle, more irregular, exhibited rough surfaces and tended to be easily fractured during cutting, depending on the composition.

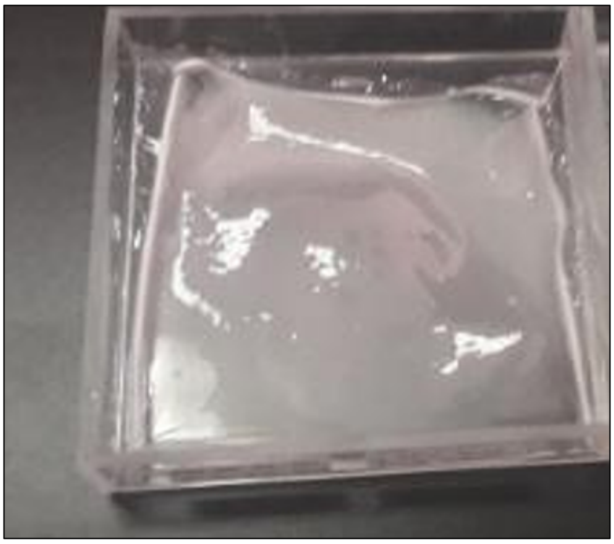

Figure 1: PVA/borate/chitosan-KCl CRF Prepared in Composition of PVA: Borate: Chitosan Ratio of 8:1:5 with 1 mL of KCl Solution

FTIR Characterization confirmed the successful formation of the crosslinked hydrogel network. The PVA/borate hydrogel exhibited characteristic bands at 3300–3500 cm^{-1} (O–H stretching), 2920 cm^{-1} (C–H stretching), 1635 cm^{-1} (H–O–H bending) and 1090 cm^{-1} (B–O–C stretching), indicating borate-mediated crosslinking, in Figure 2. Upon incorporation of KCl, a slight reduction in O–H stretching intensity was observed, suggesting possible interactions between K^+ ions and borate groups within the network (28). The addition of chitosan generated characteristic bands at ~1590 cm^{-1} and ~1410 cm^{-1}, which commonly indicate for the existence of N–H bending (29, 30) and C–

N stretching coipled with N-H (31), respectively. Furthermore, broadening and a slight shift of the O–H stretching band toward lower wavenumbers indicated enhanced hydrogen bonding between hydroxyl groups of PVA and amino groups of chitosan. In the PVA/borate/chitosan-KCl system, band broadening and minor peak shifts suggest the existence of cooperative hydrogen bonding and ionic interactions, which contribute to increased network compactness and structural stability.

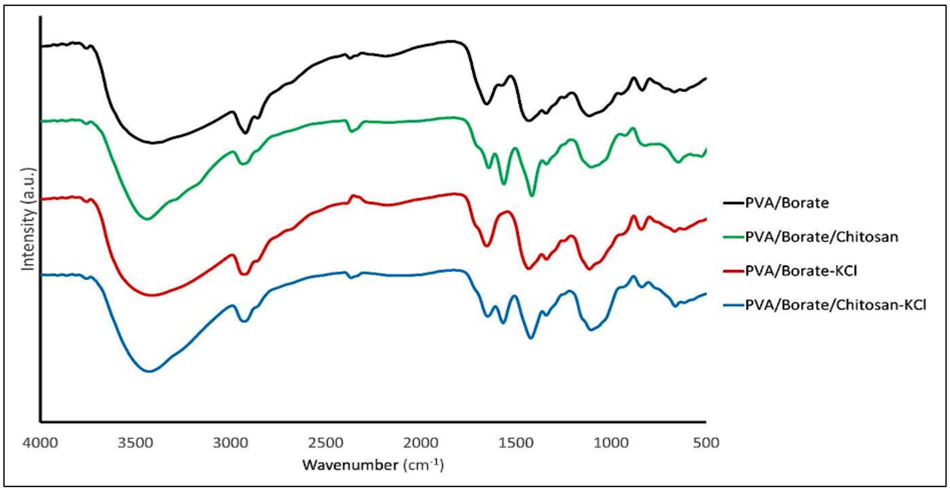

Figure 2: FTIR Spectrum of PVA/Borate, PVA/Borate-KCl, PVA/Borate/Chitosan and PVA/Borate/Chitosan-KCl

The thickness of the synthesized CRFs, measured dynamically after immersion (Methods), is presented in Figure 3. Among the samples, the thickness of PVA/borate-KCl CRF exhibited a higher value (2.63 mm) compared with that of PVA/borate/chitosan-KCl (1.66 mm). The high value in thickness of the PVA/borate-KCl CRF could be attributed to the rapid KCl diffusion during gelation, which created larger internal voids. In contrast, the thinner structure of PVA/borate/chitosan-KCl CRF demonstrates a more compact network, which is most likely due to stabilizing hydrogen bonding between chitosan amino groups and the PVA/borate matrix.

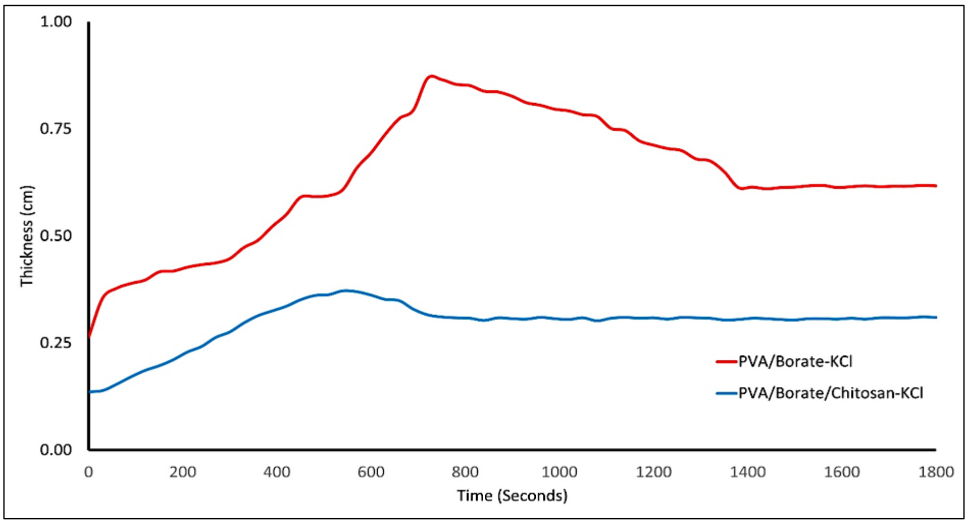

Figure 3: Thickness of PVA/Borate-KCl and PVA/Borate/Chitosan-KCl CRFs Over Time of Immersion

The swelling rate of PVA/borate-KCl CRF was faster than that of PVA/borate/chitosan-KCl CRF, as illustrated in Figure 4, at least up to approximately the 20th minute or during the initial immersion period. Beyond this point, the overall volume of PVA/borate-KCl CRF no longer increased and instead tended to decrease over time. In contrast, the volume of PVA/borate/chitosan-KCl CRF continued to increase until 32.5 minutes and subsequently reached a plateau. The higher equilibrium swelling ratio observed in PVA/borate/chitosan-KCl

(750.7%) compared to PVA/borate-KCl (642.5%) indicates that chitosan incorporation modifies network architecture. Although chitosan enhances intermolecular interactions, it may also increase network hydrophilicity (32) and osmotic driving force (33, 34), facilitating greater water uptake. Additionally, improved structural integrity prevents surface erosion during prolonged immersion, allowing the hydrogel to maintain its three-dimensional network and sustain water absorption over time (35, 36). This balance between crosslink density and hydrophilicity is critical in optimizing hydrogel-based CRFs.

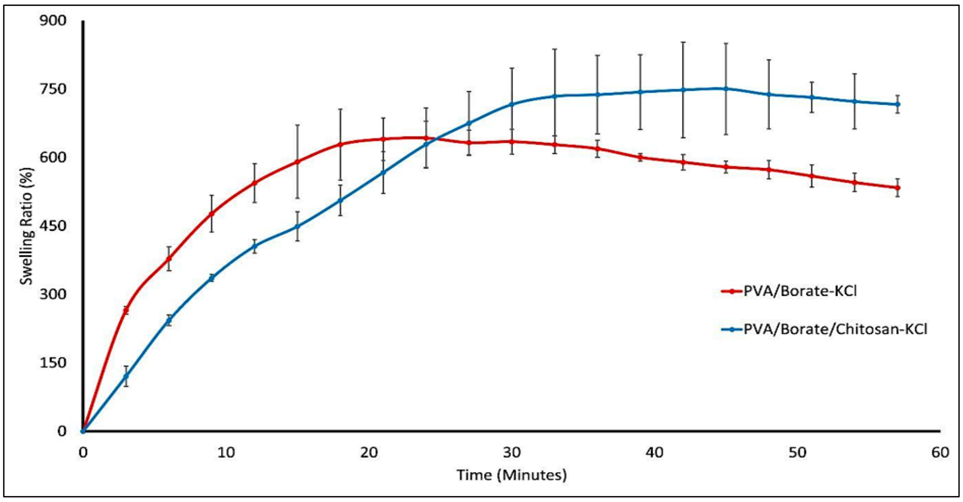

Figure 4: Swelling Ratio of PVA/Borate-KCl and PVA/Borate/Chitosan-KCl CRFs Over Time of Immersion

Water retention (WR) profiles are displayed in Figure 5. The PVA/borate/chitosan-KCl hydrogel retained water for up to 234 hours, significantly longer than PVA/borate-KCl (216 hours). This extended WR is consistent with the denser, more stable hydrogel network formed when chitosan was combined with PVA/borate and KCl. These findings demonstrate that the addition of chitosan not only improves hydrogel homogeneity and mechanical stability but also enhances swelling capacity and prolongs water retention—key features for controlled release fertilizer applications (27, 37).

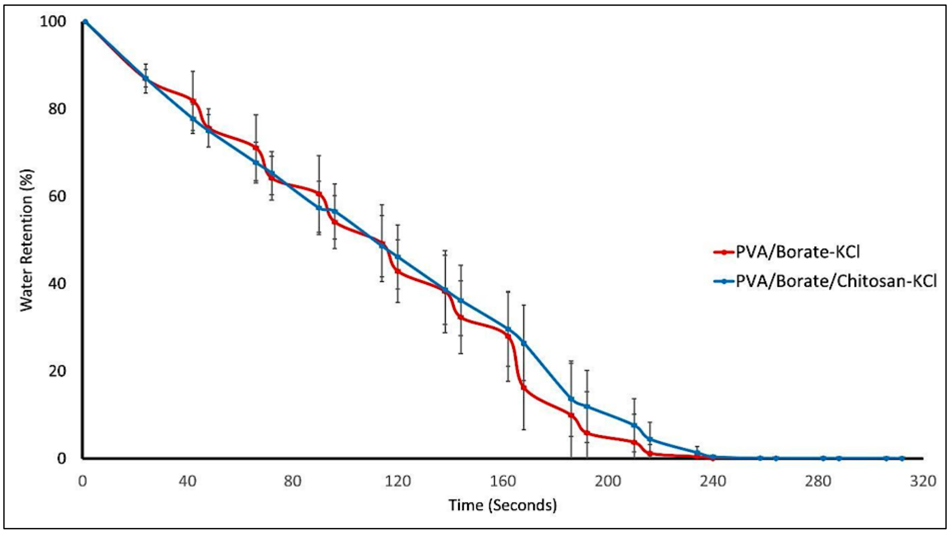

Figure 5: Water Retention of PVA/Borate-KCl and PVA/Borate/Chitosan-KCl Hydrogels Over Time of Immersion

Release Behavior profiles of the CRFs, evaluated through conductivity measurements, were as shown in Figure 6. The release profiles reveal a biphasic pattern characterized by an initial rapid release followed by a slower, sustained phase. The initial burst effect is attributed to surface-associated KCl and diffusion through water-filled pores. In the PVA/borate system, the release rate decreased rapidly due to structural weakening and partial dissolution in aqueous media.

In contrast, the PVA/borate/chitosan hydrogel exhibited prolonged release, likely governed by diffusion through a denser polymer network and restricted ion mobility resulting from hydrogen bonding and possible ionic complexation with borate groups. The incorporation of chitosan thus increases diffusion resistance and enhances structural integrity, resulting in improved nutrient release control (27).

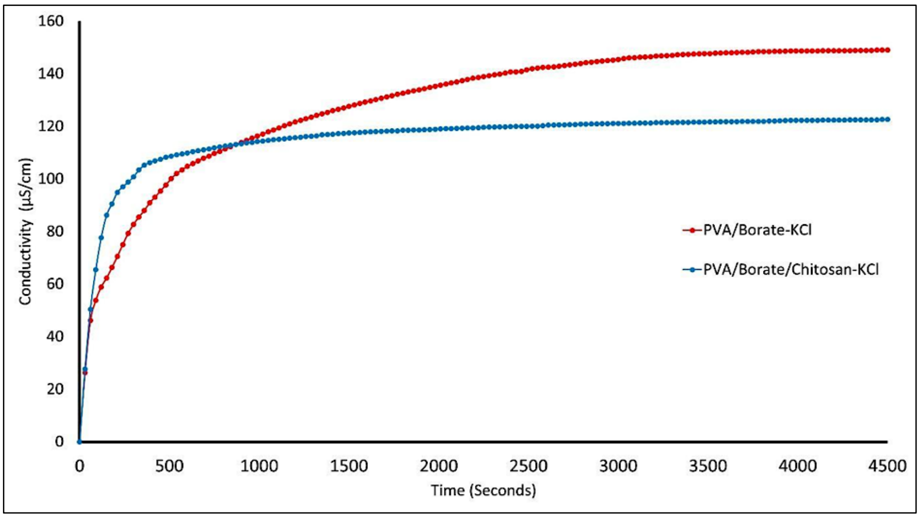

Figure 6: Release Behavior of PVA/Borate-KCl and PVA/Borate/Chitosan-KCl CRFs

The data of KCl release over time was then fitted against several kinetics models, namely first-order (38), second-order (39), Korsmeyer-Peppas (38, 39) and Weibull (38) – which the results are given in Table 2. The equation for first and second-order kinetics were derived from mass conservation and were then subtracted from initial value resulting in the equation in Table 2.

Table 2: Comparison of Fitting Results Using Several Release Kinetics Equations

Model	Equation	Hydrogel	Parameter	R^2	References
First-order	$\lambda = \lambda_0(1 - e^{-kt})$	PVA/Borate	λ_0 = 142.46 k = 2.20×10^{-3}	0.9022	(38)
		PVA/Borate/Chitosan	λ_0 = 118.91 k = 7.39×10^{-3}	0.9455	
Second-order	$\lambda = \frac{\lambda_0^2 kt}{\lambda_0 kt + 1}$	PVA/Borate	λ_0 = 157.57 k = 2.14×10^{-5}	0.9790	(39)
		PVA/Borate/Chitosan	λ_0 = 123.95 k = 1.08×10^{-4}	0.9944	
Korsmeyer-Peppas	$\lambda = kt^n$	PVA/Borate	k = 25.19 n = 0.22	0.9647	(38, 39)
		PVA/Borate/Chitosan	k = 54.88 n = 0.10	0.8395	
Weibull	$\lambda = \lambda_0\left(1 - e^{-kt^\beta}\right)$	PVA/Borate	λ_0 = 159.87 k = 3.96×10^{-2} β = 0.51	0.9981	(38)
		PVA/Borate/Chitosan	λ_0 = 120.73 k = 5.62×10^{-2} β = 0.59	0.9808	

Note: R^2 is coefficient of determination.

The models were then compared based on the average coefficient of determination (R^2) for both hydrogels. Based on the value of R^2, it can be seen that Korsmeyer-Peppas model performed the worst compared to other models. This might be due to its application mainly for the beginning of the release process, that is still far from equilibrium. The first-order model performed better with average R^2 of 0.9239, despite being a simple release model. The best-performing model was the Weibull model, which is quite similar to the first-order kinetic, with additional shape parameter on as the exponent of time. However, the addition of an extra parameter into the model increases the possibility of overfitting of the model. Because of this, we are therefore justified in concluding that the release kinetics of the reaction actually follows second-order kinetics, as its R^2 only slightly lower than Weibull model, but with less parameter. The second-order kinetics of the release might be explained as the release occurring in 3 steps, which are water absorption into the hydrogel, KCl hydration and KCl-water removal into the bulk (5, 40, 41). Compared to the PVA/borate-KCl CRF shown in Figure 4, the PVA/borate/chitosan-KCl CRF exhibited a slower solvent diffusion rate, but significantly improved solvent accommodation capacity. However, Figure 6 showed that the KCl release rate from the hydrogel showed the opposite tendency, i.e., PVA/borate/chitosan-KCl CRF desorps KCl slower than PVA/borate-KCl CRF. Both phenomena indicate that solvent diffusion into the hydrogel is not the overall determining factor. Consequently, in this 3-step process, the KCl-hydration/solution step is likely the rate-controlling step of the release process, making the overall release process to follow second-order kinetics release model**.** Nevertheless, considering that this model only involves water (aquadest) as the testing environment; to be able to apply it to a real agricultural environment, it still needs to be tested in an environmental model that more closely resembles field conditions.

Conclusion

This study demonstrates that chitosan incorporation significantly enhances the structural and functional performance of PVA/borate hydrogels for controlled potassium chloride delivery. The optimized 8:1:5 formulation exhibited improved swelling capacity, extended water retention and slower nutrient release compared to the PVA/borate system. FTIR analysis suggests that hydrogen bonding and possible ionic interactions contribute to increased network compactness and diffusion resistance. The PVA/borate-KCl CRF exhibited a higher value of thickness (2.63 mm) compared with that of PVA/borate/chitosan-KCl (1.66 mm). The fitting of release data against several kinetics models revealed that the release behaviour seems to follow the second-order kinetics model, indicating that the rate-determining step of the release process is the hydration of the ions in the system. These results confirm that chitosan incorporation not only improves hydrogel stability but also enhances control over nutrient release kinetics. PVA/borate/chitosan hydrogels represent a promising, low-cost and environmentally compatible platform for improving potassium fertilizer efficiency. However, this study is still limited to laboratory-scale experiments conducted in an aqueous model environment so it is possible that there are variables that have not been taken into account when it will be applied to other environmental conditions. Future work should include soil incubation studies and mechanical characterization to further validate field applicability.

Abbreviations

CRF: controlled-release fertilizer, CRFs: controlled-release fertilizers, FTIR: Fourier-transform infrared, KBr: potassium bromide, KCl: potassium chloride, PVA: poly (vinyl alcohol), SR: swelling ratio, WR: water retention.

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Author Contributions

Zakiah Dzulummah: Writing – Original Draft, Investigation, Methodology, Formal Analysis, Hendrawan Hendrawan: Writing – Review and Editing, Validation, Supervision, Data Curation, Funding Acquisition, Conceptualization, Hafiz Aji Aziz: Writing – Review and Editing, Data Curation, Visualization, Fitri Khoerunnisa: Writing – Review and Editing, Formal Analysis.

Conflict of Interest

The authors confirmed that there is no conflict of interest in the publication of this research.

Data Availability

Upon reasonable request, the corresponding author can provide the data supporting the findings of this research work.

Declaration of Artificial Intelligence (AI) Assistance

No AI system was involved in data analysis, interpretation of results, forming scientific conclusions, or Figures preparation. The authors take full responsibility for the content's originality, interpretation and accuracy.

Ethics Approval

Not Applicable.

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