



## Original Research Article

# Investigation of the Physicochemical Behavior of a Thermosensitive Hydrogel Based on Carboxymethyl Cellulose (CMC) with Phosphate–Calcium Microfillers

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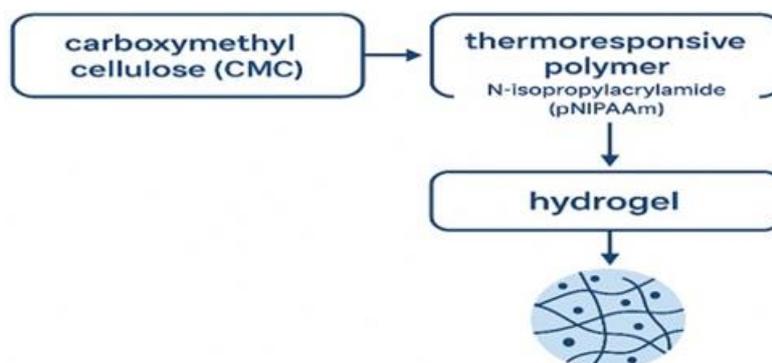
Poly(*N*-isopropylacrylamide)

Physicochemical behavior

## ABSTRACT

This study explores the physicochemical behavior of a heat-responsive hydrogel composite produced from carboxymethyl cellulose (CMC) and poly(*N*-isopropylacrylamide) (pNIPAAm), additionally reinforced with calcium phosphate microfillers. The purpose of the work was to design and evaluate a polymer material capable of rapidly absorbing water while providing controlled ion release. The hydrogel was synthesized through free-radical copolymerization under gentle heating, which supported the formation of a semi-interpenetrating network strengthened by ionic interactions between CMC functional groups and the mineral additives. Swelling tests have shown that the material absorbs a significant amount of water at temperatures below the lower critical solution temperature (LCST), and absorption clearly decreases after temperatures above approximately 30–34 °C, indicating temperature-related structural changes. Thermal analysis (TGA/DSC) showed a multi-stage decomposition pattern typical of cellulose-synthetic polymer systems, with the main decomposition occurring at temperatures between 250 and 450 °C. The porous polymer and mineral structure, which are well interconnected through microscopic studies, were determined by these studies. By adding calcium and phosphate to the composition, the mechanical integrity of the polymer was strengthened, increasing its ability to retain moisture and enhancing its applicability.

## GRAPHICAL ABSTRACT



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## Introduction

Water scarcity is one of the foremost constraints on crop productivity in arid and semi-arid regions. Superabsorbent polymers (SAPs) and hydrogels have been investigated for decades as soil amendments capable of absorbing and releasing large volumes of water, thereby increasing plant-available water and reducing irrigation frequency and volume [1,2]. However, traditional synthetic SAPs (*e.g.*, polyacrylamide derivatives) raise environmental concerns related to persistence and potential ecotoxicity; consequently, research emphasis has shifted toward biodegradable, bio-based hydrogels that combine high water capacity with controlled degradation and nutrient delivery functions [3,4]. Carboxymethyl cellulose (CMC), a cellulose derivative, is particularly attractive as a matrix polymer due to its biocompatibility, biodegradability, abundance, and capacity for chemical modification and graft copolymerization. Incorporation of thermoresponsive polymers such as poly(*N*-isopropylacrylamide) (pNIPAAm) introduces a temperature-sensitive swelling/deswelling response (phase transition near the lower critical solution temperature (LCST)) that can be exploited to release water or nutrients in response to diurnal or seasonal temperature changes [5,6].

CMC is a water-soluble anionic cellulose ether frequently used to form hydrogels either by physical crosslinking or chemical grafting with synthetic monomers (*e.g.*, acrylic acid, acrylamide derivatives). Advantages include tunable charge density, hydrophilicity, and compatibility with mineral fillers and nutrients. Recent reviews emphasize CMC-based hydrogels' suitability for agricultural applications because they can be formulated to biodegrade under soil microbial activity while retaining good swelling capacity and mechanical integrity for months under field conditions [7,8].

pNIPAAm exhibits a well-known LCST near 32–34 °C in water: below this temperatures the polymer chains are hydrated and the network swells, while above the LCST hydrophobic interactions drive chain collapse and expulsion of water. Copolymerization or grafting pNIPAAm with hydrophilic biopolymers (like CMC) or incorporation of charged comonomers shifts LCST and modifies the amplitude and kinetics of swelling/deswelling. For agricultural use, tuning LCST close to soil surface temperatures (or to diurnal variation) allows engineered daytime water release (when evapotranspiration is high) and nocturnal re-uptake or retention [9,10].

Pure pNIPAAm is not inherently biodegradable; therefore, strategies to create degradable, thermoresponsive composites include (i) grafting pNIPAAm onto biodegradable backbones (CMC, polysaccharides), (ii) blending with biodegradable co-polymers or fillers that accelerate microbial attack, and (iii) designing labile crosslinks susceptible to hydrolysis or enzymatic cleavage. Reviews report biodegradable hydrogel formulations that achieve meaningful biodegradation (tens of percent) within months under soil conditions, while maintaining functional water retention for at least a crop season. Ensuring the degradation products are non-toxic and mineralize to CO<sub>2</sub>/water/biomass is essential for long-term environmental safety [11,12]

Thermosensitive hydrogels, which combine high water sorption capacity with temperature-responsive water release, present promising opportunities for adaptive plant hydration: during nights and cooler periods they retain water effectively, while at elevated temperatures, they release moisture in a controlled manner into the root zone. The use of biodegradable polymer matrices minimizes environmental risks associated with polymer accumulation in the soil profile [13,14].

Recent advances in the application of hydrogels reveal a broad spectrum of research directions

focusing on several key aspects. The first research line concerns the synthesis of superabsorbents—polymeric materials with extremely high water-retention capacity and resistance to multiple swelling–drying cycles. Such superabsorbents can absorb water up to 300–500 times their own weight, ensuring water availability to plants during periods of low precipitation. Particular attention is given to mechanical stability, as hydrogels must maintain their structure and functionality throughout the growing season under field conditions [15,16].

The second direction involves the modification of hydrogels for controlled water release through the development of stimuli-responsive polymer systems capable of altering their properties under specific environmental factors [17,18]. Among these, the most widely investigated are: (i) pH-sensitive hydrogels, which regulate water and nutrient release depending on soil acidity; (ii) salt-sensitive systems that respond to changes in irrigation water salinity and the ionic composition of the soil solution; and (iii) thermosensitive polymers exhibiting hydrophilic–hydrophobic transitions at the LCST. For instance, pNIPAAm possesses an LCST of approximately 32 °C—below this temperature it retains water, while above it becomes hydrophobic and releases moisture, synchronizing with the plant's physiological water demand during hot periods [19,20].

A third rapidly developing field is biodegradable hydrogels based on natural polymers such as cellulose, starch, alginates, and chitosan. These biopolymer-based matrices are safe for soil biota and degrade into non-toxic products, representing an environmentally preferable alternative to traditional polyacrylate hydrogels that may persist in soils and alter their physicochemical properties after prolonged use [21,22].

Field and laboratory experiments consistently demonstrate that hydrogel application contributes to reduced evaporative water losses,

increased water use efficiency (WUE), stabilization of the soil water balance [23,24]. However, the simultaneous integration of thermosensitivity and biodegradability within a single system remains insufficiently studied. Most existing studies address either purely synthetic thermosensitive hydrogels or biodegradable matrices lacking temperature-regulated functionality. This research gap is particularly relevant for Central Asian regions, including Kazakhstan, where climatic conditions are characterized by large diurnal temperature variations and acute water scarcity. In these environments, the potential of biodegradable thermosensitive hydrogels remains largely unexplored [25-27].

The main goal was to develop and characterize a multifunctional polymer system that can quickly retain water and control ion release. The hydrogel was obtained by free radical copolymerization under mild thermal conditions, which allowed the formation of a semi-penetrating polymer mesh enhanced by ionic coordination between CMC functional groups and inorganic filler particles.

## Experimental

### *Materials and methods*

#### *Synthesis of the hydrogel composite*

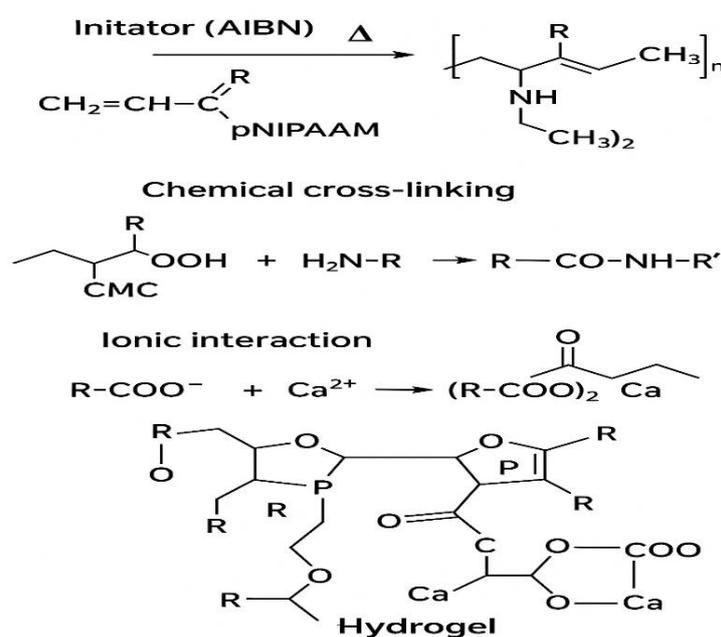
The composite hydrogel was synthesized via free-radical copolymerization of sodium CMC with *N*-isopropylacrylamide (NIPAAm) in the presence of a crosslinking agent (thermally cleavable diglycidyl ether) and an initiator (ammonium persulfate). To form a fertilized matrix, a fraction of reaction-precipitated mineral particles composed of sodium phosphate and calcium carbonate was incorporated into the reaction mixture prior to gelation in the form of microgranules. The resulting hydrogels were washed thoroughly, freeze-dried to obtain a granulated product, and subsequently ground to a

particle size of 1–3 mm suitable for soil application (Scheme 1).

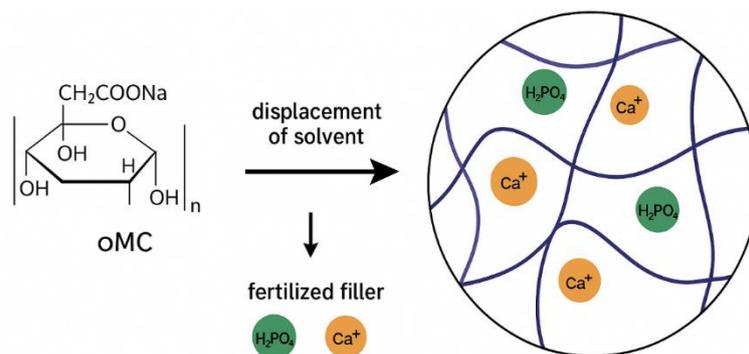
In this research work, the resulting hydrogel was synthesized by the interaction of two chemical compounds: CMC and the thermosensitive polymer pNIPAAm with the addition of calcium and phosphate compounds in the filler. During the polymerization process, this process occurs based on special physicochemical processes, which occur through chemical cross-contact. Calcium and phosphate compounds react with the carboxyl ( $-\text{COO}^-$ ) groups of CMC through ionic bonds with polymer compounds, forming additional bonding sites. This regulates the temperature fluctuations caused by the hydrogel. The combined structure therefore offers enhanced sorption capacity along with biodegradability, making the composite hydrogel a promising option for agricultural use,

particularly in regulating soil moisture and providing a more sustainable fertilization strategy (Figure 1).

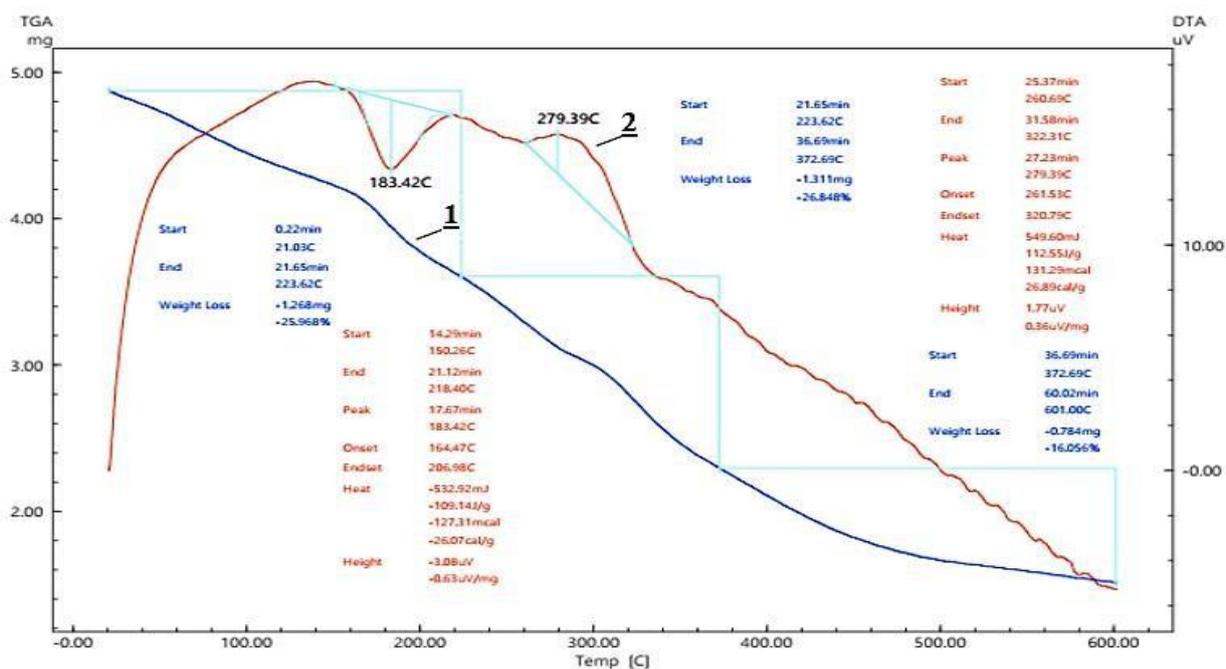
The swelling behavior was determined in both distilled water and 0.01 M KCl solution at 20 and 35 °C under static and kinetic conditions. The LCST was measured by differential scanning calorimetry (DSC) and visually confirmed by observing the loss of optical transparency. Mechanical strength was assessed using a universal testing machine by compressing the samples up to 50% deformation. Biodegradation was evaluated by incubating the hydrogel samples in agricultural soil at 25 °C and recording mass loss after 30, 90, and 180 days. The nutrient release profile was examined under model conditions by determining phosphorus and calcium concentrations in aqueous extracts over time.



**Scheme 1.** Synthesis of the hydrogel composite via free-radical polymerization, chemical cross-linking, and ionic interactions involving  $\text{Ca}^{2+}$  ions



**Figure 1.** Schematic representation of the synthesis and structural configuration of the biodegradable thermosensitive hydrogel composite based on CMC and pNIPAAm with a phosphate–calcium fertilized filler. The network formation involves the polymerization and partial cross-linking of CMC and pNIPAAm chains, while phosphate and calcium ions participate in coordination and ionic bonding within the matrix, enhancing both water retention and nutrient-controlled release properties.



**Figure 2.** Derivatogram of the hydrogel based on CMC pNIPAAm (1 — Dynamic thermogravimetric analysis (DTGA) curve; 2 — DSC curve)

## Results and Discussion

The hydrogel exhibited a static swelling ratio of  $310 \pm 25 \text{ g}\cdot\text{g}^{-1}$  in distilled water at  $20^\circ\text{C}$ . When the temperature exceeded the LCST, the equilibrium swelling decreased to  $255 \pm 20 \text{ g}\cdot\text{g}^{-1}$  at  $35^\circ\text{C}$ , confirming temperature-sensitive moisture release behavior. The swelling kinetics indicated

that 90% of the equilibrium value was reached within 6–8 hours. DSC and visual observation determined the LCST to be in the range of  $30\text{--}34^\circ\text{C}$ , during which the gel structure became denser and partial expulsion of bound water occurred.

The thermogravimetric (TGA) and DSC data demonstrate a multi-stage thermal degradation

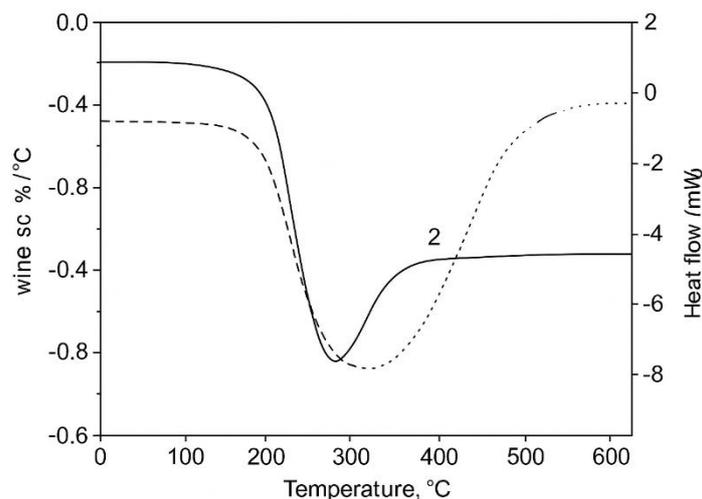
process of the CMC/pNIPAAm-based hydrogel (Figure 2). In the low temperature zone, up to about 100 °C, a slight decrease in mass is observed in the sample, which is mainly due to the release of moisture, which is poorly preserved in the hydrogel structure. As the temperature rises to the range of 100-300 °C, the material begins to lose weight significantly; this period indicates the decomposition of CMC and the rupture of several side groups that form the polymer network. The most intense changes occur at temperatures between 300 and 450 °C, at which the pNIPAAm spine undergoes thermal decomposition, and its functional groups are dehydrated. After about 450 °C, the mass curve smooths out, indicating that the waste has transformed into a stable carbonized residue. The DSC profile also contains endothermic and exothermic thermal phenomena corresponding to polymer decomposition reactions and the destruction of certain horizontal bonds, which aligns with the behavior usually

observed in biodegradable polymer—cellulose composite materials. Table 1 shows how the CMC–pNIPAAm hydrogel responds to heating from 50 to 600 °C based on changes in mass, decomposition rate, and energy exposure. Mass losses at the beginning of the heating process (50-150 °C) are relatively small, from 0.98% to 14.65%, which mainly indicates water removal and primary regulation of polymer chains. After the temperature exceeds 200 °C, a significant shift occurs: the mass loss increases to 27.15%, marking the beginning of significant decomposition of the polymer trunk. The rate of decomposition is uneven. Instead, it shows several peaks between 250 and 400 °C (2.18-3.19 mg/min) that may correspond to the main degradation phases of CMC and pNIPAAm.

Figure 3 presents the thermal analysis of the biodegradable thermosensitive hydrogel synthesized from CMC and pNIPAAm, showing both the DTGA, Curve 1, and DSC, Curve 2 results.

**Table 1.** Analysis of the hydrogel based on CMC pNIPAAm

No.	Temperature (°C)	Mass loss (%)	Decomposition rate of hydrogel (mg/min)	Energy consumption (μV·s/mg)
1	50	0,984	0,145	1,20
2	100	8,985	0,487	2,72
3	150	14,651	0,562	2,22
4	200	27,145	0,178	3,75
5	250	35,85	2,178	1,32
6	300	40,49	2,455	4,25
7	350	51,22	3,199	2,49
8	400	66,15	3,125	4,52
9	450	74,10	3,265	2,66
10	500	88,14	1,452	1,42
11	600	91,40	1,568	1,67



**Figure 3.** Diagram of the hydrogel based on CMC and poly(*N*-isopropylacrylamide)

The DTGA curve (1) reveals a multi-step mass loss pattern typical for hydrophilic polymer networks. These studies have shown that the initial stage below 150 °C corresponds to the evaporation of physically bound and absorbed water molecules. The loss of the main mass, which occurs at temperatures from 250 to 400 °C, is caused by thermal destruction of the polymer base, including the breaking of amide bonds in the C-O-C and CMC–pNIPAAm matrix, which becomes unstable. The DSC (2) curve shows an endothermic transition of about 100 °C due to the separation of water, followed by a unique endothermic exothermic complex in the 280-360 °C region, corresponding to the structural transformation and decomposition of polymer chains. The temperature interval of 32-34 °C corresponds to the LCST of the solution; this phenomenon confirms the heat-sensitive nature of the hydrogel and its phase transition. Overall, these results demonstrate that the CMC–pNIPAAm hydrogel maintains structural stability up to approximately 250 °C, exhibits a clear LCST transition within the expected temperature range, and possesses suitable thermal resistance for agricultural and environmental applications where moderate heating and water retention are critical (Figure 3).

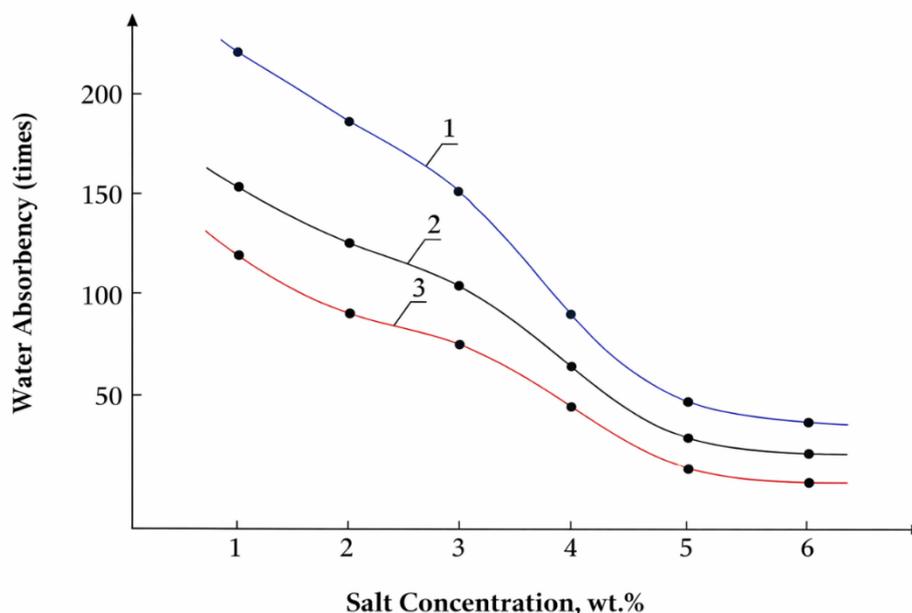
In the early stages of formation, CMC acts as the main hydrophilic support, giving the hydrogel the ability to bind large amounts of water and ensuring harmony with the natural environment. When pNIPAAm is introduced, it becomes a temperature-responsive network, which causes the material to expand or shrink depending on the thermal conditions of the environment. After the polymerization and crosslinking reactions are completed, both components are combined into a single three-dimensional structure, shown in the lower part of the scheme. This frame looks like a continuous, interconnected mesh that can retain significant moisture while being sensitive to changes in external conditions.

## Discussion

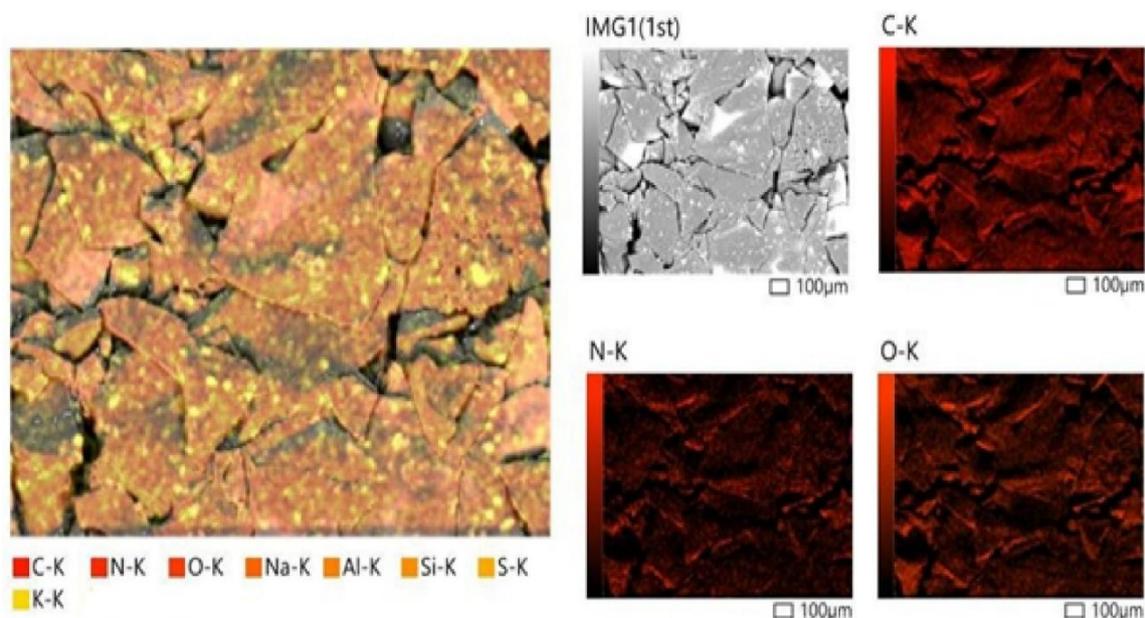
The swelling behavior of the composite, along with its LCST-related transitions, indicates that the hydrogel effectively retains water at normal temperature and gradually releases some of this moisture as the temperature rises. Such a reaction is best suited to the natural rhythm of plant water demand: on cool nights and mornings, the material retains water, and during hot periods, it releases additional moisture to compensate for the increased transpiration. This temperature-dependent regulation distinguishes the material

from traditional SAPs, which usually passively release water without responding to daily changes in the environment. The ability to modulate the availability of moisture during the day is especially effective for climates where the contrast of day and night temperatures is clearly noticeable. The study of biodegradability also shows that a significant part of the hydrogel decomposes in about six months. This prevents long-term accumulation in the soil and reduces possible destruction by soil microorganisms, which makes the composition more environmentally compatible than many synthetic water-retaining materials. The incorporation of CMC as a biopolymer backbone, combined with the use of mild crosslinking agents, ensured an optimal balance between structural durability during the growing season and subsequent biodegradability under soil conditions.

Figure 4 illustrates the influence of increasing salt concentration on the swelling capacity of the hydrogel samples. As the concentration of NaCl increases from 1 to 6 wt.%, a gradual decline in water absorbency is observed for all compositions (curves 1–3). This decrease can be attributed to the charge-screening effect of ions, which reduces the electrostatic repulsion between the negatively charged  $-\text{COO}^-$  groups of CMC and consequently limits the polymer network expansion. Curve 1 represents the hydrogel without mineral filler, curve 2 corresponds to the composite with a moderate phosphate–calcium additive, and curve 3 depicts the formulation with the highest filler content. The data demonstrate that the introduction of mineral filler slightly decreases the overall swelling capacity due to partial occupation of hydrophilic sites; however, the material retains significant water-holding ability even under saline conditions, indicating its potential for use in arid and semi-arid soils.



**Figure 4.** Effect of salt concentration on the water absorbency of the hydrogel (1 — Hydrogel based on CMC/pNIPAAm (without filler); 2 — CMC/pNIPAAm hydrogel with phosphate additive; and 3 — CMC/pNIPAAm hydrogel with calcium–phosphate composite)



**Figure 5.** Microphotographs of the CMC-based hydrogel containing a thermoresponsive polymer component, pNIPAAm, together with a phosphorus–calcium fertilizer filler. The images were taken using a scanning electron microscope (SEC Alpha SEM) at a magnification corresponding to 100  $\mu\text{m}$

In **Figure 5**, microphotographs of the CMC-based hydrogel containing a thermoresponsive polymer component show that the synthesized hydrogel demonstrates a dense, heterogeneous microstructural property due to the fact that the resulting polymer compounds contain dispersed inorganic compounds. The detection of calcium and phosphorus elements in the EDX spectra (not shown) corresponds to the embedded phosphate–calcium fraction, which is responsible for the hydrogel’s fertilizing functionality and potential ion-exchange capacity. The surface morphology exhibits micro-porosity favorable for water retention and gradual nutrient release, suggesting that the composite material can act as a multifunctional soil conditioner for controlled moisture regulation and nutrient delivery.

The results of the study indicate that the hydrogel synthesized from CMC and pNIPAAm combines several important properties: sensitivity to temperature, strong water retention ability, mechanical stability and the capacity to gradually release nutrients. These features collectively make the material a viable candidate

for improved soil moisture management in dry agriculture. According to the swelling tests, the hydrogel can absorb more than 300 g of water per gram of material at normal temperature; this performance mainly depends on the hydrophilicity of CMC and the semiconductor structure formed by pNIPAAm. When the temperature exceeds the LCST point (approximately 30-34  $^{\circ}\text{C}$ ), the swelling begins to decrease, confirming that the system can independently respond to heat and release moisture when the demand from crops is high. This temperature-regulated behavior parallels the natural mechanisms found in plants, giving the composition a clear advantage over standard hydrogels, which do not adjust their emission patterns to environmental conditions. Thermal tests provide additional insight into the strength of the composite and how it breaks down. The gradual loss of mass observed in TGA curves—especially the main decay from 250 to 450  $^{\circ}\text{C}$ —indicates the sequential degradation of the CMC and pNIPAAm components. The porous microstructure observed in SEM images reveals

favorable pathways for water uptake and diffusion, contributing to improved soil aeration and moisture distribution when applied in field conditions.

## Conclusion

Based on a comprehensive physicochemical assessment, the synthesized CMC/pNIPAAm hydrogel composite demonstrates a set of structural and functional properties that confirm its effectiveness as a heat-sensitive, moisture-retaining and nutrient-bearing polymer system. Copolymerization of CMC with *N*-isopropylacrylamide under conditions of mild radical action resulted in the formation of a semi-penetrating grid with a well-defined swelling-delamination dynamics controlled by a lower critical temperature of the solution. The inclusion of phosphate-calcium microgranules in the polymer matrix led to the appearance of additional ion crosslinking centers, which increased mechanical stability and created regions of controlled release of nutrient ions. Thermal analysis confirmed that the composite undergoes a multi-step decomposition typical of polysaccharide and synthetic polymer hybrids. The primary decomposition intervals correspond to the destruction of the main chain obtained from cellulose, the splitting of side chains, and the subsequent destruction of amide-containing segments. Thermal analysis showed that the material decomposes in a stable, controlled manner: no sharp exothermic peaks were detected and the mass gradually decreased as the temperature increased. The presence of interconnected pores in this hybrid structure may be responsible for the increase in sorption and diffusion properties observed in the composition. Overall, the results show that the developed hydrogel composite combines heat sensitivity, biodegradability, structural strength, and the ability to release ions within a single material platform. Such a multifunctional polymer system

has the potential for use in environments that require controlled moisture retention, gradual availability of nutrients, and adaptive physicochemical behavior.

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## Disclosure Statement

No potential conflict of interest was reported by the authors.

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