Development and Implementation of Biodegradable Hydrogels Based on Carboxymethyl Cellulose for Agricultural Sectors

Desarrollo e Implementación de Hidrogeles Biodegradables a Base de Carboximetilcelulosa para Sectores Agrícolas

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Abstract

This study aims to prepare superabsorbent hydrogels (PSA) from carboxymethyl cellulose (CMC), a water-soluble polymer, through crosslinking with $Al_2(SO_4)_3$, $CaCl_2$ and two comonomers. PSA evaluation included measuring gel content, swelling degree, and soluble fraction, comparing them with non-crosslinked CMC. Pitahaya growth tests in soils enriched with PSA demonstrated superior performance for CMC and $Al_2(SO_4)_3$-based PSA. Results showed a soluble fraction of 93.37%, gel fraction of 6.33%, and swelling degree of 1480.82, corresponding to water retention 15 times the weight of dehydrated PSA. The research highlights the effectiveness of CMC crosslinking for obtaining superabsorbent hydrogels with potential agricultural applications.

Hydrogels, Carboxymethyl Cellulose, Crosslinking

Resumen

En el presente trabajo se propone la preparación de hidrogeles superabsorbentes (PSA) a partir de carboximetilcelulosa (CMC), un polímero soluble en agua, mediante un proceso de entrecruzamiento con $Al_2(SO_4)_3$, $CaCl_2$ y dos comonómeros. La evaluación de los PSA se realizó mediante la medición del contenido de gel, grado de hinchamiento y fracción soluble, comparándolos con CMC no entrecruzada. Se llevaron a cabo pruebas de crecimiento de pitahayas en tierras enriquecidas con PSA, evidenciando un rendimiento superior del PSA a base de CMC y $Al_2(SO_4)_3$. Los resultados indicaron una fracción soluble del 93.37%, fracción de gel del 6.33%, y un grado de hinchamiento de 1480.82, equivalente a una retención de agua 15 veces el peso del PSA deshidratado. La investigación destaca la efectividad del entrecruzamiento de CMC para obtener hidrogeles superabsorbentes con potenciales aplicaciones agrícolas.

Hidrogeles, Carboximetilcelulosa, Entrecruzamiento

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1. Introduction

Hydrogels are three-dimensional matrices made up of polymeric networks. They have captured the interest of the scientific community since their discovery by Wichterle and Lím in 1960. Their unique ability to retain large volumes of water and their versatility position them as materials with great potential in various fields, such as food, biomaterials, agriculture, and water purification (C. Chang, L. Zhang, 2011). In this context, the present study focuses on obtaining superabsorbent hydrogels from carboxymethylcellulose (CMC) through a cross-linking process with divalent salts.

Carboxymethylcellulose, a water-soluble polymer, has proven to have significant applications in food additives, textiles, pharmaceuticals, and cosmetics. Although it has been extensively explored in various fields, its potential as a base material for obtaining superabsorbent hydrogels by cross-linking with divalent salts has been largely unexplored. The adoption of this technique offers substantial added value compared to other hydrogel formation methodologies (Bajpai et al., 2008). As noted in previous studies, CMC could respond to external stimuli, such as changes in solvent composition, temperature variations, pH fluctuations, among others (Sanna, 2013).

Unlike conventional hydrogels, superabsorbent hydrogels possess the exceptional ability to retain significantly larger volumes of water compared to their dehydrated weight. This study focuses on the preparation of CMC superabsorbent hydrogels through a cross-linking process with $Al_2(SO_4)_3$, $CaCl_2$ and two different comonomers. Evaluation of the performance of hydrogels is carried out by measuring the percentage of gel content, degree of swelling, and soluble fraction compared to non-intersected CMC. In addition, the viability of these hydrogels in agricultural applications is investigated, specifically in the growth of dragon fruit in soils enriched with superabsorbent hydrogels.

The interest in CMC-based superabsorbent hydrogels is based on their renewable origin and the potential ability to improve soil physical properties, reduce irrigation frequency, and promote healthier plant growth (Stahl et al., 2000; Weerawarna, 2009).

However, despite their advantages, the systematic exploration of the production and application of these hydrogels is still limited, which motivates the present investigation.

In this context, the aim is to optimize the production of CMC hydrogels by cross-linking with $Al_2(SO_4)_3$ and comonomers, thus exploring new perspectives in the use of these versatile materials.

2. Methodology

Materials and method

Sodium carboxymethyl cellulose (CMC) was purchased from Sigma Aldrich and was used without the need for any further purification process. All other chemical reagents used were of analytical grade. Distilled water was used throughout the experiment.

CMC Hydrogel Synthesis

The preparation of the CMC hydrogel was carried out following the methods published by (Che Nan, et al., 2019) and (Fidelia, N., et al., 2011). An 8% stock solution of CMC in distilled water was prepared (Figure 1).

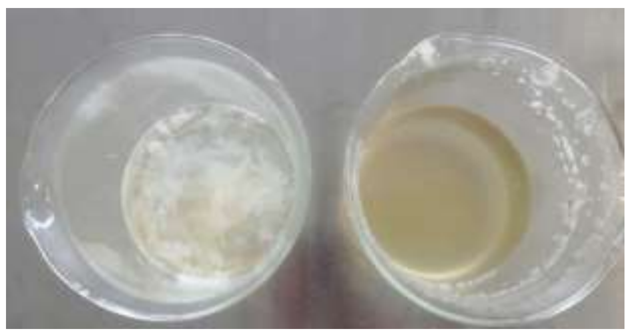

Figure 1 Preparation of 8% carboxymethylcellulose (CMC) solution in distilled water

From this solution, three different aqueous solutions were generated to investigate optimal cross-linking. These solutions contained 0.2% $Al_2(SO_4)_3$; In the second, 0.4% sodium alginate was added, and in the third, 0.2% acrylamide.

The CMC hydrogel obtained from each sample was transferred to glass containers and allowed to react at room temperature for 2 hours. Subsequently, the solution was spread on steel trays and dried at 65°C until a thin film of the dried hydrogel was formed. The film was crushed and then pulverized with a mortar.

This was repeated for each type of agent investigated, which were evaluated by determining the gel content and the degree of swelling (Figure 2).

Figure 2 Examples of the various types of hydrogels manufactured and extracted from the original solution

On the other hand, the preparation of the hydrogel in calcium chloride solution was carried out according to the method of (Chia et al., 2015), using variable concentrations of CMC and calcium. The resulting solution was allowed to react for 24 hours at room temperature, and the optimization was determined by the percentage of gel content and the degree of swelling.

The optimization of CMC hydrogels was determined by the percentage of gel content and the degree of swelling.

Determination of Soluble Fraction and Gel Fraction

The soluble fraction and gel fraction were calculated using the following formulas:

$$\text{Soluble fraction (\%)} = \frac{(Wi-Wo)}{Wi} * 100 \quad (1)$$

Gel fraction (%) = 100 – Soluble fraction
Where *Wi* is the initial weight of the sample and *Wo* is the weight of the sample after the drying process.

Determination of the Degree of Swelling

The degree of swelling was determined with the equation:

$$\text{Degree of swelling} = \frac{(Wi-Wo)}{Wo} * 100 \quad (2)$$

Where *Wi* is the initial weight of the sample and *Wo* is the weight of the sample after the drying process.

Flowerpot Experiment

Petri dishes were used to grow seeds, incorporating different polymers into the soil mixture. Pitahayas (*Selenicereus undatus*) were planted, and water was applied according to the established schedule (Table 1).

Number of days after planting	Amount of water per day (ml)
0	2
7	1
14	0.5
21	0
28	0

Table 1 Seed irrigation program

3. Results

Soluble Fraction, Gel Fraction, and Degree of Swelling

The swelling capacity of the hydrogel shows an inversely proportional relationship with the gel fraction, which means that a high percentage of gel results in a low degree of swelling due to the limited availability of spaces for the entry of free water into the hydrogel network. hydrogel (Table 2 and 3). The hydrophilicity of the carboxylic group in CMC hydrogels influences this swelling behavior (Jamingan et al., 2015). The lowest degree of swelling is observed when the gel content is highest, due to the limited space available for water. The swelling process is divided into three stages: diffusion of water molecules, relaxation of polymer chains and expansion of the polymer network (Che Nan, et al., 2019). This behavior was manifested when the CMC hydrogel was immersed in deionized water, generating osmotic pressure, and leading to the swelling of the hydrogel matrix (Abd El-salam Deghiedy, 2004).

Flowerpot Experiment

During the first two weeks, the seeds were optimally watered. Subsequently, the irrigation frequency was reduced to evaluate the response of the plants to drought conditions.

Seeds in soil without PSA enrichment ceased growth after two weeks and showed obvious signs of dehydration. In contrast, seeds in PSA-enriched soil continued to grow beyond the first two weeks, appearing noticeably healthier (Figure 3).

Figure 3 Petri dishes containing hydrogels that promoted seed development despite drought conditions

4. Annexes

SAP	$Al_2(SO_4)_3$ (%)	Alginate (gr)	Acrylamide (gr)	Soluble fraction (%)	Gel fraction (%)	Swelling degree
0	0	0	0	84.69	15.31	553.11
1	0.4	0	0	93.67	6.33	1480.82
2	0.4	0.5	0	79.19	20.81	380.48
3	0.4	1	0	83.35	16.65	500.43
4	0.4	0	0.5	92.90	7.10	1308.55
5	0.4	0	1	80.34	19.66	408.72

Table 2 Detail of the reagent concentrations for each PSA generated with $Al_2(SO_4)_3$, along with the results of the gel fraction and swelling index. All tests were carried out with a CMC concentration of 8%

SAP	$CaCl_2$ (%)	Alginate (gr)	Acrylamide (gr)	Soluble fraction (%)	Gel fraction (%)	Swelling degree
0	0	0	0	78.20	21.80	475.67
1	0.4	0	0	87.80	12.20	1190.41
2	0.4	0.5	0	73.70	26.30	300.19
3	0.4	1	0	78.01	21.99	425.37
4	0.4	0	0.5	87.75	12.25	954.26
5	0.4	0	1	75.24	24.76	324.36

Table 3 Summary of reagent concentrations in each PSA produced with $CaCl_2$, complemented by gel fraction, and swelling coefficient data. All tests were carried out with a CMC concentration of 8%

5. Conclusions

The results obtained in this study support the feasibility and promising potential of superabsorbent polymers derived from carboxymethyl cellulose (CMC) cross-linked with $Al_2(SO_4)_3$ as an ecological soil amendment. Optimization of the CMC hydrogel was achieved with a concentration of 8% CMC, 0.4% Al_2 $(SO_4)_3$, and a reaction time of 2 hours at room temperature. Application of this superabsorbent polymer to soil revealed significant improvements in water retention during periods of water stress.

The findings indicate that soil amended with this superabsorbent polymer supports healthy plant growth, especially compared to unamended soil.

This phenomenon translates into greater resistance of plants to water stress, evidencing the importance of water retention in the soil during irrigation. Furthermore, the results suggest that this approach could represent a sustainable alternative to petroleum-based superabsorbent polymers, highlighting its potential to improve water efficiency in agriculture.

For future research, it is recommended to further explore application conditions, cross-linking agents and evaluate the long-term impact of incorporating these superabsorbent polymers in various soil types and crops.

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