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Research Article

Formulation and Swelling Evaluation of Starch–Glycerol-Based Biopolymer Films for Potential Wound Dressing Applications

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Abstract

This study investigates the structural behavior and swelling performance of biopolymer films formulated from starch, glycerol, carboxymethylcellulose (CMC), and polymeric β-cyclodextrin (PCD) for potential wound dressing applications. Binary starch–CMC and starch–PCD systems exhibited poor rheological properties, forming highly pasty mixtures in the absence of glycerol. Conversely, glycerol–CMC and glycerol–PCD systems remained excessively fluid, confirming the essential structural role of starch in film formation. Ternary starch–glycerol–CMC formulations enabled the production of stable and functional films, particularly 10A/6G/4CMC and 12A/4G/4CMC, which demonstrated substantial swelling capacities of 140% and 164% at 60 minutes, respectively. Swelling kinetics revealed a rapid initial water uptake within the first 60 minutes, followed by a slower approach toward equilibrium. The ability to incorporate CMC without compromising film integrity was strongly dependent on starch proportion, with instability observed when starch content fell below 10 parts. The introduction of PCD into optimized formulations (10A/6G/1CMC/3PCD and 12A/4G/1CMC/3PCD) resulted in moderate swelling capacities (123% and 136% at 60 minutes), lower than those observed for the corresponding binary and ternary systems. These findings suggest that PCD contributes primarily through its inclusion-complex forming capability rather than by enhancing swelling behavior. Overall, the results highlight the critical importance of the starch–glycerol ratio in achieving structurally stable films and confirm the reinforcing role of CMC, while identifying PCD as a promising component for future development of active wound dressings with controlled drug delivery potential.

Keywords: Wound dressings, Starch-based biopolymers, Glycerol, Carboxymethylcellulose (CMC), β-Cyclodextrin (PCD).

INTRODUCTION

The skin is a complex organ covering the entire body. With an average surface area between 1.5 and 2 m^2 in adults and representing approximately 15% of total body weight, it is the largest organ of the human body [1]. Its thickness ranges from 1 to 4 mm depending on the anatomical region and environmental conditions [2]. Throughout life, the skin undergoes numerous types of damage, varying in severity and depth, often resulting in wounds. These are mainly caused by burns, trauma, or other factors such as diabetes. Wound management is a routine medical practice due to the risks of endogenous and exogenous contamination and potential complications. In this context, protection of lesions through the use of dressings is fundamental. However, selecting the ideal dressing is not a simple process. Lesion type, wound depth, healing duration, and etiology help characterize the wound, predict its progression and possible complications, and allow the selection of the most appropriate treatment based on objective criteria [3].

Each wound encountered by a physician or surgeon is unique, given the wide variety of wound types and patient-specific clinical characteristics (origin of the wound, associated pathologies). In addition, the growing number of available wound dressings further complicates therapeutic decision-making.

The objective of this study was to design absorbent wound dressings based on starch and glycerol incorporating functional additives in order to improve the absorption capacity of wound exudates.

MATERIALS

The reagents used included starch extracted from plant cells, composed of two homopolymers of α-D-glucopyranose: amylose, a linear polymer formed by glucose units linked by α-(1→4) bonds, and amylopectin, a highly branched polymer consisting of glucose units linked by α-(1→6) bonds [4]. Glycerol (propane-1,2,3-triol), also known as glycerin, is a natural organic compound with the chemical formula $CH_2OH–CHOH–$

CH_2OH. It is a colorless, odorless, non-toxic, viscous, and highly hygroscopic liquid [5]. It acts as a humectant and plasticizing agent. Carboxymethylcellulose (CMC) is an absorbent material available as a granular or fibrous white powder, slightly hygroscopic, odorless, and tasteless. The polymer of β -cyclodextrin (PCD) consists of natural "host" molecules obtained by enzymatic degradation of starch. Cyclodextrins are cyclic glucose oligomers containing between 6 and 12 glucose units. The most abundant forms are α-cyclodextrin (hexamer), β-cyclodextrin (heptamer), and γ-cyclodextrin (octamer). These molecules have a truncated cone ("lampshade") structure. All polar hydroxyl (OH) groups are located on the external surface, delimiting a relatively hydrophobic internal cavity. This amphiphilic character allows cyclodextrins to include hydrophobic molecules within their cavity, forming water-soluble inclusion complexes [6].

METHODS

Preparation of dressings

The preparation method consisted of mixing the different components and completing with distilled water to a final volume of 100 mL, followed by heating until a viscous solution was obtained.

The viscous solution was then poured into metal plates with 5 cm diameter cavities and dried in an oven at 40°C for 24 hours.

Binary formulations

The following table presents the proportions used for binary formulations containing two components.

Table I: Variation of component proportions

Component 1	12.5%	25%	37.5%	50%	62.5%	75%	87.5%
Component 2	87.5%	75%	62.5%	50%	37.5%	25%	12.5%

- *Binary starch/glycerol formulation*

In this formulation, proportional quantities of starch and glycerol were mixed in a beaker. The proportions used are presented in Table II.

Table II: Variation of starch and glycerol quantities (grams)

STARCH (g)	**2**	**4**	**6**	**8**	**10**	**12**	**14**
GLYCEROL (g)	14	12	10	8	6	4	2

- *Binary starch/CMC formulation*

In this formulation, proportional quantities of starch and CMC were mixed in a beaker.

Table III: Proportions of Starch and CMC

STARCH	0%	25%	50%	75%	100%
CMC	100%	75%	50%	25%	0%

- *Binary starch/PCD formulation*

Proportional quantities of starch and PCD were mixed in a beaker. The mixture remained highly pasty in all combinations due to the absence of glycerol, which is responsible for plasticity and fluidity.

- *Binary glycerol/CMC formulation*

Proportional quantities of glycerol and CMC were mixed.

Table IV: Binary glycerol/CMC proportions

GLYCEROL	0%	25%	50%	75%	100%
CMC	100%	75%	50%	25%	0%

Glycerol quantities ranged from 0 to 16 g, while CMC ranged from 16 to 0 g. After mixing, the solution remained liquid for all combinations and no stable film was obtained.

- *Binary glycerol/PCD formulation*

Proportional quantities of glycerol and PCD were mixed. The mixture remained liquid for all combinations, and no film formation was achieved.

Ternary formulations (starch/glycerol/carboxymethylcellulose)

Variable quantities of starch, glycerol, CMC, and PCD were mixed. The proportions are presented below.

Table V: Ternary formulation quantities

STARCH	6	8	10	12
GLYCEROL	10	8	6	4
CMC	4	4	4	4

- *Glycerol/CMC/PCD formulation*

Variable proportions were used as shown below.

Table VI: Glycerol/CMC/PCD proportions

GLYCEROL	**12.5%**	**25%**	**50%**	**62.5%**
CMC	62.5%	50%	25%	12.5%
PCD	25%	25%	25%	25%

The mixture remained liquid for all combinations.

- Starch/Glycerol/CMC/PCD formulation

Proportional quantities were used as follows:

Table VII: Quaternary formulation proportions

STARCH (g)	6	8	12
GLYCEROL (g)	10	8	4
CMC (g)	1	1	1
PCD	3	3	3

Determination of absorption capacity

Absorption tests were performed in 0.9% NaCl solution to simulate physiological wound exudate conditions. Unlike distilled water, wound exudate contains electrolytes such as sodium (Na^+) and chloride (Cl^-) ions, which influence swelling behavior of hydrophilic polymer networks. Ionic strength modifies electrostatic interactions within polymer matrices, particularly for ionizable materials such as CMC. Therefore, saline medium provides a more clinically relevant evaluation of dressing absorption capacity. After drying, films were weighed to obtain the initial weight (P0), then immersed in 0.9% NaCl solution for 15 and 60 minutes, and reweighed (Pt).

The absorption rate (AR) was calculated using:

$$AR = \frac{(P_t - P_0)}{P_0} \times 100$$

Where:

P0 = initial dry weight

Pt = weight after immersion

RESULTS

Appearance and flow properties

Direct mixing of starch and CMC did not produce satisfactory results. A highly pasty mixture was obtained, highlighting the essential role of glycerol in providing plasticity to the formulation.

Figure 1: Proportional mixture of starch and CMC

Starch/PCD formulation

The mixture remained highly pasty for all tested combinations. This behavior is attributed to the absence

of glycerol, which plays a key role in providing plasticity and improving the fluidity of the system.

Glycerol/CMC or Glycerol/PCD formulation

After mixing, the solution remained liquid for all combinations, and no stable film could be obtained. The absence of starch explains this behavior, as gelatinized starch forms a cohesive gel matrix necessary for film formation.

Ternary formulations

- Starch/glycerol/carboxymethylcellulose

Following the incorporation of CMC into the initial starch–glycerol formulations, the aim was to identify dressings that remained stable without degradation while maintaining high water absorption capacity. The formulations retained for further evaluation were:

8A/8G/4C

10A/6G/4C

12A/4G/4C

These systems exhibited satisfactory structural stability and absorption behavior.

Figure 2: Starch, glycerol, and CMC mixture cast onto perforated metal plate

- Glycerol/CMC/PCD formulation

The proportional mixture of glycerol, CMC, and PCD resulted in a liquid system for all tested combinations. No stable film formation was observed under these conditions.

- Starch/glycerol/CMC/PCD formulation

By varying the quantities of CMC and PCD in the previously optimized starch–glycerol dressings, it was possible to identify the formulation exhibiting the most suitable performance profile. This approach allowed optimization of structural stability while maintaining adequate absorption capacity.

Figure 3: Mixture of starch, glycerol, CMC, and PCD

Absorption capacity of binary formulations

The results of the water absorption measurements for the different binary formulations are presented in Table VIII. The table summarizes the proportions of starch and glycerol used, along with the initial dry weight (P_0), the weight after immersion (P_t), and the calculated mean absorption percentage for each formulation.

Table VIII: Mean absorption capacity of starch–glycerol films (60 min)

Formulation	P_0 (g)	P_t (g)	Absorption (%)
10A/6G	0.21	0.61	160%
6A/10G	0.19	0.48	152%
12A/4G	0.25	0.70	195%
8A/8G	0.15	0.36	140%

These results indicate that the 10A/6G and 12A/4G formulations exhibited the highest absorption profiles, as illustrated in Figure 4.

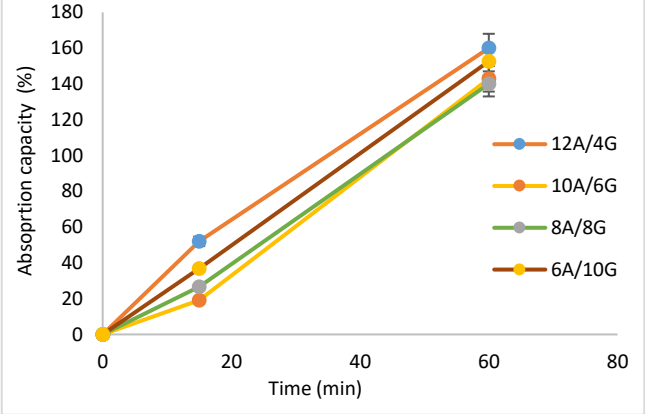

Figure 4: Absorption capacity of starch–glycerol films as a function of time

A significant increase in absorption capacity was observed during the first 60 minutes of immersion. Beyond this initial phase, absorption continued to increase gradually over time. Among the tested formulations, 12A/4G exhibited the highest absorption profile.

Absorption capacity of ternary starch–glycerol–CMC formulations

Table IX presents the results of the selected ternary formulations evaluated in this study. The absorption capacity of starch–glycerol films incorporating CMC was determined by measuring the initial dry weight (P_0) and the weight after immersion in 0.9% NaCl solution (P_t). The mean absorption percentage for each formulation was subsequently calculated.

Table IX: Mean absorption capacity of starch–glycerol–CMC films (60 min)

Formulation	P_0 (g)	P_t (g)	Absorption (%)
10A/6G/4CMC	0.31	0.75	140%
12A/4G/4CMC	0.36	0.95	164%

The results indicate that incorporation of CMC maintained good structural stability while providing substantial absorption capacity, although values remained lower than those observed for the corresponding binary starch–glycerol systems.

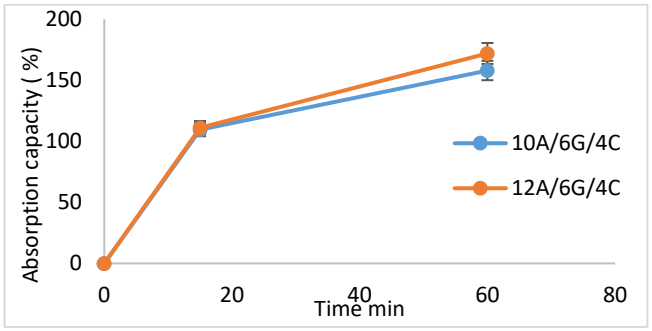

Figure 5 : Variation curve of the absorption capacity of the starch–glycerol–CMC system as a function of time

As shown in Figure 5, absorption capacity increased markedly during the first 15 minutes of immersion. After this initial phase, the increase became less pronounced over time, indicating indicating that the swelling process approached equilibrium after the initial rapid uptake phase.

Absorption capacity of starch–Glycerol–CMC–PCD formulations

Table X presents the results obtained from the evaluation of absorption capacity for starch–glycerol systems supplemented with CMC and PCD. The best-performing binary formulations were selected and further modified by incorporating CMC and PCD. The initial dry weights (P_0), weights after immersion in 0.9% NaCl solution (P_t), and calculated mean absorption percentages are reported below.

Table X: Mean absorption capacity of 10A/6G/1CMC/3PCD and 12A/4G/1CMC/3PCD films (60 min)

Formulation	P_0 (g)	P_t (g)	Absorption (%)
10A/6G/1CMC/3PCD	0.45	1.002	123%
12A/4G/1CMC/3PCD	0.55	1.302	136%

The incorporation of PCD resulted in moderate absorption values, which were lower than those observed for the corresponding binary and ternary systems.

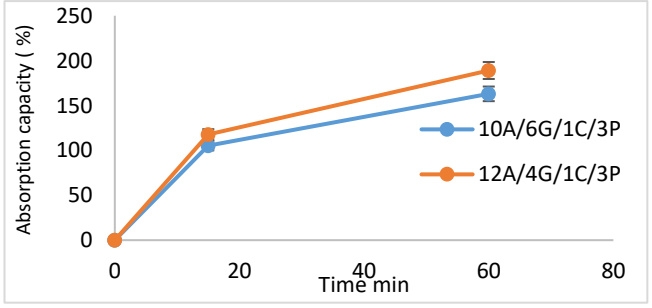

Figure 6: Absorption capacity of the starch–Glycerol–CMC–PCD system as a function of time

Even in the presence of both CMC and PCD, absorption capacity remained significantly higher during the first 15 minutes of immersion. Between 15 and 60 minutes, only minor variations in absorption were observed, indicating that the swelling process reached a near-equilibrium state after the initial rapid uptake phase.

DISCUSSION

Absorption capacity represents one of the most critical properties of wound dressings, as it directly reflects their ability to manage wound exudates. This parameter was therefore the primary focus of the present study. Film-forming systems were prepared using varying proportions of starch and glycerol, followed by the incorporation of carboxymethylcellulose (CMC) and polymeric β -cyclodextrin (PCD) in order to evaluate their influence on swelling behavior.

Among the starch–glycerol systems, the highest absorption values were obtained for 12A/4G (195%) and 10A/6G (160%), while 8A/8G and 6A/10G exhibited moderate absorption capacities (140% and 150%, respectively). Formulations with extreme ratios (14A/2G) were brittle, whereas those containing excessive glycerol (4A/12G and 2A/14G) were pasty and failed to form stable films, preventing absorption measurement.

The absorption behavior of the films can primarily be attributed to starch, which exists in the form of granules composed of amylose and amylopectin. The swelling capacity of starch has been associated with amylopectin content, while amylose is considered to act as a diluent and swelling inhibitor [7,8]. Initially, starch granule swelling is reversible, with volume increases up to approximately 30% [9]. As water uptake and temperature increase, hydrogen bonds progressively weaken, allowing water penetration and partial solubilization of starch. This process results in irreversible swelling and a substantial increase in granule size. Above the gelatinization temperature, the crystalline regions lose structural order and undergo irreversible expansion [10], highlighting the importance of precise temperature control during film preparation.

The incorporation of CMC influenced both structural stability and absorption behavior. For the 12A/4G formulation, the addition of 4CMC maintained high absorption (195%), while 3CMC reduced absorption to 146%. Lower CMC contents (1CMC and 2CMC) led to unstable, pasty systems. A similar trend was observed for 10A/6G, where 4CMC and 3CMC resulted in absorption values of 160% and 135%, respectively. However, in systems containing lower starch proportions (e.g., 8A/8G), high CMC content compromised structural integrity, limiting film formation. These findings suggest that sufficient starch content (≥10 parts) is necessary to maintain network cohesion in the presence of CMC.

Although CMC is known to absorb nearly twice its weight in water, its isolated contribution within the composite matrix is difficult to quantify without advanced analytical techniques. Nevertheless, comparison of absorption values before and after CMC incorporation revealed an approximate 15% increase for both 12A/4G and 10A/6G

systems, indicating a reinforcing effect of CMC within an adequately structured starch–glycerol network.

The subsequent incorporation of PCD did not enhance absorption capacity under physiological saline conditions (0.9% NaCl). Absorption values at 15 minutes (123% and 136%) were lower than those observed for binary and ternary systems. This behavior may be explained by structural reorganization of the polymeric network, resulting in a more compact matrix. Additionally, the ionic strength of the saline medium may have limited polymer expansion by screening electrostatic interactions. Therefore, the primary interest of PCD in this system appears to lie in its inclusion-complex forming ability rather than in improving swelling capacity.

CONCLUSION

This study evaluated the absorption properties of starch–glycerol-based biopolymer films for potential wound dressing applications. Binary starch–glycerol systems demonstrated the highest absorption performance, reaching 190% and 195% at 15 minutes for 10A/6G and 12A/4G formulations, respectively. The incorporation of carboxymethylcellulose improved film stability while maintaining substantial absorption capacity (140–164%). In contrast, the addition of polymeric β-cyclodextrin did not significantly enhance swelling under physiological conditions, although it offers promising potential for drug encapsulation and controlled release applications.

Overall, the results highlight the critical role of the starch/glycerol ratio in achieving functional film formation and confirm the potential of these natural polymer matrices for the future development of improved absorbent wound dressings.

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