

Influence of Plasticizer Type on Starch-Based Nanocomposite Films Reinforced with CMC and ZnO

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Abstract

The development of eco-friendly composite films from renewable resources represents a crucial strategy to reduce dependence on petroleum-based plastics. In this study, carboxymethyl cellulose (CMC) derived from oil palm empty fruit bunches (OPEFB) was utilized along with starch and zinc oxide nanoparticles (ZnO NPs) to fabricate biodegradable composite films. Two types of plasticizers, glycerol and sorbitol, were incorporated at varying concentrations of 20%, 30%, and 40% to evaluate their influence on the physical properties of the films, including density, thickness, moisture content, water absorption, and tensile strength. The results revealed that both plasticizers affected the structural and physical characteristics of the films. Sorbitol addition decreased density from 1.413 g/cm³ to 0.463 g/cm³ and increased moisture content and water absorption, whereas glycerol addition increased density from 1.330 g/cm³ to 1.559 g/cm³ and reduced moisture content and water absorption. The formulation containing 20% glycerol showed optimal results for higher density and water resistance, while 40% sorbitol provided superior performance with enhanced mechanical integrity and lower water sensitivity. Overall, the type and concentration of plasticizer play a critical role in determining the physical properties of OPEFB-derived CMC/starch/ZnO nanocomposite films, demonstrating their potential as environmentally friendly packaging materials to replace conventional plastics.

Keywords: Carboxymethyl Cellulose, Oil Palm Empty Fruit Bunches, Sorbitol, Glycerol, Composite Film.

Abstrak

Pengembangan film komposit ramah lingkungan berbasis sumber daya terbarukan merupakan strategi penting untuk mengurangi ketergantungan terhadap plastik berbasis minyak bumi. Dalam penelitian ini, karboksimetil selulosa (CMC) yang berasal dari tandan kosong kelapa sawit (TKKS) digunakan bersama pati dan nanopartikel seng oksida (ZnO) untuk memproduksi film komposit yang dapat terdegradasi

secara hayati. Dua jenis plasticizer, yaitu gliserol dan sorbitol, ditambahkan dengan variasi konsentrasi 20%, 30%, dan 40% untuk mengevaluasi pengaruhnya terhadap sifat fisik film, meliputi densitas, ketebalan, kadar air, daya serap air, dan kekuatan tarik. Hasil penelitian menunjukkan bahwa kedua jenis plasticizer memberikan pengaruh signifikan terhadap struktur dan sifat fisik film. Penambahan sorbitol menurunkan densitas dari 1,413 g/cm³ menjadi 0,463 g/cm³ serta meningkatkan kadar air dan daya serap air, sedangkan penambahan gliserol meningkatkan densitas dari 1,330 g/cm³ menjadi 1,559 g/cm³ serta menurunkan kadar air dan daya serap air. Formulasi dengan 20% gliserol menghasilkan film dengan densitas lebih tinggi dan ketahanan air yang lebih baik, sedangkan 40% sorbitol memberikan kinerja terbaik dengan integritas mekanik yang lebih baik dan sensitivitas air yang lebih rendah. Secara keseluruhan, jenis dan konsentrasi plasticizer berperan penting dalam menentukan sifat fisik film nanokomposit CMC/pati/ZnO berbasis TKKS, sehingga berpotensi digunakan sebagai bahan kemasan ramah lingkungan pengganti plastik konvensional.

Keywords: *Karboksimetil Selulosa, Tandan Kosong Kelapa Sawit, Sorbitol, Gliserol, Film Komposit.*

1. Introduction

The accumulation of non-biodegradable plastic waste has led to severe ecological consequences, including soil and water contamination, microplastic formation, and adverse effects on marine organisms and human health [1]. As a result, the development of biodegradable, renewable, and environmentally friendly alternatives to conventional plastics has gained significant attention in recent years. Among various candidates, polysaccharide-based biopolymers have emerged as promising materials for sustainable packaging applications due to their abundance, biodegradability, and film-forming properties [2].

Starch is widely used in biodegradable film production, while cellulose can be converted into Carboxymethyl Cellulose (CMC), a water-soluble derivative with good film-forming properties [3] [4]. Indonesia's oil palm industry produces large quantities of Oil Palm Empty Fruit Bunches (OPEFB), a cellulose-rich byproduct that represents an abundant and low-cost raw material for CMC production[5]. Utilizing OPEFB for CMC synthesis not only reduces agricultural waste but also supports circular bioeconomy principles through waste valorization. However, films made solely from CMC are often brittle and hydrophilic, which limit their mechanical strength and moisture resistance [6]. To address these limitations, CMC is often combined with starch and reinforced with inorganic nanoparticles such as Zinc Oxide (ZnO), which enhance mechanical strength, thermal stability, and antimicrobial properties [7].

Plasticizers are commonly introduced to overcome this brittleness by disrupting hydrogen bonding and increasing polymer chain mobility. Glycerol and sorbitol are two natural polyol-based plasticizers frequently used in polysaccharide films [8]. The type and concentration of

plasticizers critically determine film characteristics such as tensile strength, elongation at break, water absorption, and thickness [8]. Despite numerous studies on starch- or cellulose-based films, comparative analyses on how different plasticizers influence the performance of nanocomposite films containing both CMC and ZnO remain limited.

Therefore, this study aims to investigate the influence of plasticizer type specifically glycerol and sorbitol on the physical and mechanical properties of starch-based nanocomposite films reinforced with CMC derived from OPEFB and ZnO nanoparticles. The investigation focuses on evaluating how the variation in plasticizer type and concentration affects the structural integrity, moisture sensitivity, and overall performance of the films

2. Method

2.1 Materials

OPEFB were sourced from a palm oil processing plant located in Jambi Province, Indonesia. All chemicals utilized in this study were of analytical grade, including sodium hydroxide (NaOH), hydrogen peroxide (H₂O₂), glacial acetic acid (CH₃COOH), trichloroacetic acid (TCA), starch (amylose), ZnO nanoparticles with an average particle size of 57 nm, a molecular weight of 81.38 g/mol, and a purity of $\geq 99\%$, methanol, glycerol and sorbitol were obtained from Merck (analytical grade), distilled water (aquadest). All reagents were used as received without any additional purification.

2.2 Preparation of Composite Films

2.2.1 Isolation of Cellulose

Cellulose isolation was carried out through delignification and bleaching processes. Delignification was performed on OPEFB fibers using 4% NaOH at 80°C for 2 h, followed by washing to neutral pH and drying. The two-stage bleaching involved (i) treatment with 10% H₂O₂ and 20% CH₃COOH at 80°C for 1.5 h, and (ii) treatment with 10% NaOH and 10% H₂O₂ at 80°C for 1.5 h. The bleached fibers were then washed to neutrality and oven-dried at 80°C for 4 h.

2.2.2 Synthesis of Carboxymethyl Cellulose (CMC)

CMC synthesis was carried out in two stages: alkalization and carboxymethylation. In the first step, cellulose was treated with 30% NaOH at 60 °C for 1 h. Then, trichloroacetic acid was added to perform carboxymethylation at 50 °C for 3 h. The resulting CMC was filtered, neutralized, immersed in methanol for 24 h to remove residual chemicals, and dried at 60 °C.

2.2.3 Preparation of Composite Films

Composite films were prepared by the solvent casting method. CMC and starch (3.5 gr each) were dissolved separately in distilled water at 75 °C for 5 min, then combined and stirred until homogeneous. ZnO nanoparticles (5 wt% of CMC), pre-dispersed in 20 mL distilled water, were added gradually under stirring. Two types of plasticizers were used: sorbitol and glycerol (20–40 wt% based on the total weight of CMC and starch), which were added to the mixture under stirring. The final mixture was cast into silicone molds (1:2 mL cm⁻²) and dried at 65 °C for 12 h to obtain uniform films.

2.3 Characterization of Composite Films

2.3.1 Film Thickness

Film thickness was measured using a Mitutoyo digital micrometer (accuracy ± 0.0001 mm) at five random points on each sample. The average thickness (t) was calculated as the mean of the five readings.

2.3.2 Mechanical Properties

Tensile strength (TS) and elongation at break (EB) were determined following ASTM D882–02 using a Tinius Olsen H5KS testing machine. Samples (2×8 cm) were tested at a crosshead speed of 10 mm min^{-1} with an initial grip distance of 50 mm. TS and EB were calculated using:

$$TS = \frac{P}{A_0}, EB(\%) = \frac{L-L_0}{L_0} \times 100 \quad (1)$$

where P is the maximum load, A_0 the initial cross-sectional area, L_0 the initial length, and L the length at break.

2.3.3 Water Absorption

Water absorption was tested by immersing film samples in 25 mL of distilled water for 24 h at room temperature. The percentage of absorption was calculated as:

$$\text{Water Absorption (\%)} = \frac{W_t - W_0}{W_0} \times 100 \quad (2)$$

where W_0 and W_t are the initial and final film weights, respectively.

2.3.4 Film Density

Film density (ρ_s) was determined from the film mass and thickness to evaluate compactness and nanoparticle distribution uniformity:

$$\rho_s = \frac{m}{A \times \delta} \quad (3)$$

where m is the mass (g), A the area (cm^2), and δ the mean thickness (cm).

2.3.5 Moisture Content

Moisture content (MC) was determined by drying film samples at 105°C for 24 h. The percentage was calculated using:

$$MC(\%) = \frac{W_1 - W_2}{W_1} \times 100 \quad (4)$$

where W_1 and W_2 are the sample weights before and after drying, respectively.

3. Result and Discussion

3.1 Film Thickness and Density

The starch/CMC/ZnO nanocomposite films derived from OPEFB were successfully fabricated through the solvent casting method using glycerol and sorbitol as plasticizers at concentrations of 20%, 30%, and 40% (w/w). The resulting films exhibited a transparent

appearance with a relatively smooth and homogeneous surface. This transparency indicates the formation of a uniform polymer matrix, in which the starch, carboxymethyl cellulose (CMC), and ZnO nanoparticles were well dispersed without noticeable phase separation.

Prior to film preparation, the successful synthesis of CMC was confirmed by FTIR analysis. The FTIR spectrum exhibited characteristic absorption bands at 1642.66 cm^{-1} and 1315.28 cm^{-1} , corresponding to the asymmetric and symmetric stretching vibrations of carboxylate groups ($-\text{COO}^-$), indicating successful carboxymethylation of cellulose. The broad absorption band at 3332.29 cm^{-1} was attributed to O–H stretching vibrations, while the band at 2899.13 cm^{-1} corresponded to C–H stretching. In addition, the prominent band at 1028.94 cm^{-1} associated with C–O–C ether linkages further verified the formation of CMC [9]. The cellulose obtained from OPEFB exhibited a cellulose content of 75.17%, indicating effective purification prior to CMC synthesis.

Table 1. Effect of Plasticizer Type and Concentration on The Thickness and Density of Starch/CMC/ZnO Nanocomposite Films

Plasticizer	Thickness (mm)	Density (g/cm^3)
Glycerol		
20% (w/w)	0.141 ± 0.004	1.330 ± 0.124
30% (w/w)	0.177 ± 0.004	1.457 ± 0.265
40% (w/w)	0.186 ± 0.001	1.559 ± 0.381
Sorbitol		
20% (w/w)	0.126 ± 0.003	1.413 ± 0.028
30% (w/w)	0.144 ± 0.001	1.127 ± 0.069
40% (w/w)	0.209 ± 0.004	0.463 ± 0.006

The results presented in Table 1 show differences in the thickness and density of the nanocomposite films as a function of plasticizer type and concentration. For glycerol-plasticized films, the thickness increased slightly from $0.141 \pm 0.004\text{ mm}$ at 20% to $0.186 \pm 0.001\text{ mm}$ at 40%. A similar trend was observed in sorbitol-plasticized films, with values ranging from $0.126 \pm 0.003\text{ mm}$ to $0.209 \pm 0.004\text{ mm}$ as the sorbitol concentration increased. Variations in the concentration of the film-forming materials may increase the number of dissolved solids in the solution, which consequently raises its viscosity and produces a thicker film matrix after drying [10]. A similar observation was also reported by Mohammed et al. (2023), who found that increasing the plasticizer content in wheat starch-based films led to a progressive increase in film thickness, as the plasticizers permeated and expanded the polymer network, resulting in a more flexible and thicker film structure [11]. Our findings align with those of Akachat [12], who examined the effect of varying plasticizer concentrations on pectin-based films. They reported that increasing the plasticizer content resulted in thicker films, a trend attributed to the hygroscopic and swelling characteristics of plasticizers. As the amount of plasticizer increases, its capacity to absorb moisture and expand within the polymer matrix enhances, leading to the formation of films with greater thickness [12].

Although both glycerol and sorbitol produced thicker films with increasing concentration, the magnitude of change differed between the two. Sorbitol-based films showed a more pronounced increase in thickness, particularly at 40% (0.209 mm), compared to glycerol-based ones (0.186 mm). This difference is likely due to the higher molecular weight and lower hygroscopicity of sorbitol [13].

Interestingly, the trend in film density varied inversely between the two types of plasticizers. In the case of glycerol, density values increased consistently from 1.330 ± 0.124 g/cm³ to 1.559 ± 0.381 g/cm³ as the concentration rose from 20% to 40%. The higher density likely results from improved packing and stronger intermolecular interactions facilitated by glycerol's smaller molecular size and higher polarity. In contrast, sorbitol-based films showed a substantial reduction in density with increasing concentration, from 1.413 ± 0.028 g/cm³ at 20% to 0.463 ± 0.006 g/cm³ at 40%. This decrease may be associated with the formation of a more open polymer network structure due to the bulkier sorbitol molecules, which create free volume and reduce overall compactness within the matrix.

In addition to the effect of plasticizers, ZnO nanoparticles may also influence the structural characteristics of the films. The high surface area of ZnO nanoparticles facilitates interactions with the hydroxyl groups present in starch and CMC, which can improve compatibility between the filler and the polymer matrix. As a result, stronger intermolecular interactions may be established, contributing to a more cohesive and stable film structure [14]. Similar findings have been reported in previous studies, where the incorporation of ZnO nanoparticles enhanced filler matrix adhesion and improved the structural integrity of biopolymer-based films [15].

3.2 Moisture Content and Water Absorption

The moisture content and water absorption of the starch/CMC/ZnO nanocomposite films were influenced by the type and concentration of plasticizer used, as shown in Table 2. These parameters are critical indicators of the hydrophilic nature, water resistance, and overall stability of biodegradable films when exposed to humid environments.

For films plasticized with glycerol, both moisture content and water absorption increased progressively with higher plasticizer concentrations. Moisture content rose from $28.18 \pm 0.002\%$ at 20% glycerol to $34.49 \pm 0.021\%$ at 40%, while water absorption increased markedly from $30.26 \pm 0.004\%$ to $59.00 \pm 0.004\%$. This trend agrees with previous reports, which demonstrated that glycerol, due to its hydrophilic nature and multiple hydroxyl groups, readily forms hydrogen bonds with water molecules and hydroxyl groups in the polymer matrix. As glycerol concentration increases, its capacity to retain water within the film network also rises, leading to greater moisture retention and swelling of the polymer structure. Furthermore, the incorporation of glycerol enhances molecular mobility and free volume by disrupting intermolecular hydrogen bonding between polymer chains. This structural relaxation facilitates the diffusion and entrapment of water molecules, resulting in higher equilibrium moisture content and water absorption at elevated glycerol levels, consistent with the findings of and the observed increase in film thickness [12].

Table 2. Effect of Plasticizer Type and Concentration on The Moisture Content and Water Absorption of Starch/CMC/ZnO Nanocomposite Films

Plasticizer	Moisture Content (%)	Water Absorption (%)
Glycerol		
20% (w/w)	28.18 ± 0.002	30.26 ± 0.004
30% (w/w)	30.43 ± 0.009	40.42 ± 0.031
40% (w/w)	34.49 ± 0.021	59.00 ± 0.004
Sorbitol		
20% (w/w)	26.65 ± 0.026	28.67 ± 0.006
30% (w/w)	19.11 ± 0.015	21.89 ± 0.012
40% (w/w)	18.63 ± 0.035	19.46 ± 0.008

In contrast, sorbitol-plasticized films displayed an opposite behavior, with both moisture content and water absorption decreasing significantly as sorbitol concentration increased from 20% to 40%. Specifically, moisture content dropped from $26.65 \pm 0.026\%$ to $18.63 \pm 0.035\%$, while water absorption decreased from $28.67 \pm 0.006\%$ to $19.46 \pm 0.008\%$. This reduction is attributed to the lower hygroscopicity of sorbitol compared to glycerol. Sorbitol molecules exhibit stronger intermolecular interactions with polymer chains due to their structural similarity to glucose units, which limits their ability to interact with water molecules. Consequently, sorbitol-plasticized films retain less moisture and display improved water resistance at higher concentrations [16].

3.3 Tensile Strength and Elongation at Break

Mechanical strength represents one of the most critical parameters in evaluating the performance of biopolymer-based films for potential use in packaging applications. The tensile strength (TS) and elongation at break (EAB) of starch/CMC/ZnO nanocomposite films were influenced by both the type and concentration of plasticizer, as presented in Figure 1a and 1b.

Films plasticized with glycerol exhibited higher tensile strength values compared to those plasticized with sorbitol at equivalent concentrations. The tensile strength of glycerol-plasticized films decreased progressively from 1.533 MPa at 20% (w/w) to 0.383 MPa at 40% (w/w), indicating that higher glycerol content increased the mobility of polymer chains, thereby weakening intermolecular interactions. Plasticizers weaken the internal hydrogen bonds between polymer molecules and diminish the intermolecular attraction forces among adjacent polymer chains, thereby decreasing the tensile strength of the film [17]. This phenomenon is commonly attributed to the small molecular size and high hygroscopic nature of glycerol, which facilitates the penetration of water molecules into the polymer matrix, reducing hydrogen bonding between starch and CMC chains [16].

Besides the influence of plasticizers, ZnO nanoparticles are also likely to contribute to the mechanical properties of the starch/CMC films. ZnO nanoparticles may enhance the mechanical performance of starch/CMC films through improved filler–matrix interactions and stress transfer [15]. However, nanoparticle aggregation can create weak regions within the film structure. Nevertheless, when present in excessive amounts, ZnO nanoparticles tend to aggregate, which may create weak points within the film structure and reduce its ability to

stretch[18]. In the present study, the mechanical properties of the films are likely governed by the combined effects of ZnO reinforcement and the plasticizing action of glycerol and sorbitol.

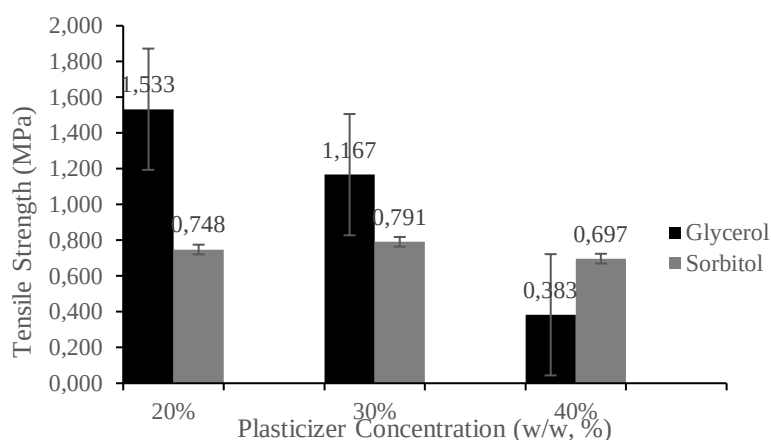


Figure 1a. Effect of Plasticizer Type and Concentration on Tensile Strength of Starch/CMC/ZnO Films

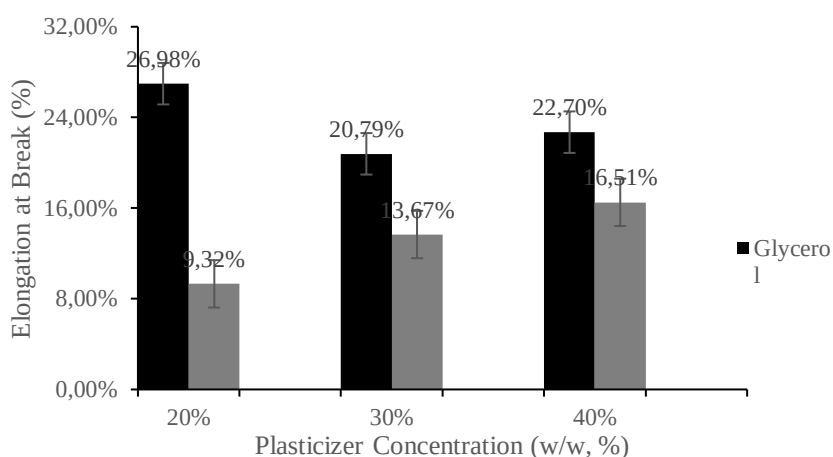


Figure 1b. Effect of Plasticizer Type and Concentration on Elongation at Break of Starch/CMC/ZnO Films

Figure 1b shows that increasing the plasticizer concentration resulted in higher elongation values, which can be attributed to the reduction of intermolecular forces and enhanced chain mobility within the polymer matrix [19]. The addition of a higher amount of plasticizer weakens the cohesive interactions between polymer chains, making the film softer and more flexible and consequently more prone to break under tensile stress. This finding is consistent with previous studies reporting that the increase in elongation percentage with increasing plasticizer concentration is due to the greater incorporation of plasticizer molecules within the polymer matrix and their occupation of free spaces through hydrogen bonding. These interactions disrupt the regular structure of the polymer, resulting in a more flexible and disordered structure with higher chain mobility [13].

According to Figure 1b, the elongation at break (EAB) of glycerol-plasticized films was considerably higher, ranging from 26.98% to 22.70%, compared to sorbitol-plasticized films, which exhibited lower EAB values between 9.32% and 16.51%. The highest elongation was obtained from glycerol-plasticized films, consistent with previous observations reported for starch-based films [13]. This trend can be attributed to the smaller molecular size and higher hydrophilicity of glycerol, which allow it to penetrate more effectively between polymer chains, increase free volume, and impart greater flexibility to the film matrix.

The tensile strength of the starch/CMC/ZnO nanocomposite films ranged from 0.383 to 1.533 MPa. Although these values were higher than the minimum tensile strength requirement of 0.3 MPa specified in JIS Z 1707:2019 for food packaging films, they were still considerably lower than the range reported in SNI 7188-7:2022 (24.7–302 MPa) and those of many starch- and biocellulose-based films described in previous studies [20]. This relatively low tensile strength is likely related to the presence of glycerol and sorbitol, which act as plasticizers by increasing polymer chain mobility and weakening intermolecular interactions within the film matrix [19]. A similar trend was observed for elongation at break, which ranged from 9.32% to 26.98%. These values were below the minimum elongation requirement of 70% reported in JIS Z 1707:2019, suggesting that the films still have limited flexibility [20]. Nevertheless, the developed films demonstrate potential as biodegradable packaging materials, particularly for applications that do not require high mechanical strength. Further optimization of the film formulation, including adjustment of the plasticizer content and reinforcement strategy, is needed to enhance the mechanical properties and broaden their potential packaging applications.

4. Conclusion

This study successfully evaluated the effect of plasticizer type and concentration specifically glycerol and sorbitol on the physical and mechanical properties of starch-based nanocomposite films reinforced with CMC derived from OPEFB and ZnO nanoparticles. The results reveal that both plasticizer type and concentration affect the structural and moisture-related behavior of the films. Increasing plasticizer concentration led to thicker films, with glycerol-plasticized films exhibiting higher density and moisture sensitivity due to glycerol's small molecular size and high hygroscopicity. In contrast, sorbitol produced thicker but less dense films with lower moisture uptake, reflecting its lower hygroscopicity and stronger interaction with the polymer matrix. Glycerol provided higher tensile strength at low concentrations but caused significant strength reduction and water absorption at higher levels. Meanwhile, sorbitol maintained better dimensional stability and water resistance with moderate flexibility. Overall, the results indicate that sorbitol at around 30–40% (w/w) provides the best balance between mechanical performance, flexibility, and moisture resistance, making it a promising plasticizer for developing biodegradable starch/CMC/ZnO nanocomposite films suitable for eco-friendly packaging applications.

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