

Green Synthesis and Characterization of Glycerol-Plasticized and Citric Acid-crosslinked PVA/CMC Bioplastics Reinforced with Coconut Fiber-Derived Cellulose

Yusmaniar^{1*}, Rias Gesang Kinanti², Kartika Ratna Pertiwi³, Wulan Kharisma Dera¹,
Setia Budi¹, Yussi Pratiwi¹ and Neng Tresna Umi Culsum¹

¹Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Negeri Jakarta,
Jl. Rawamangun Muka, Jakarta Timur, 13220, Indonesia

²Lab Farmakologi, S1 Kedokteran, Fakultas Kedokteran, Universitas Negeri Malang Jln. Semarang No. 5,
Kota Malang, Indonesia

³Faculty of Medicine, Universitas Negeri Yogyakarta, Colombo Street No. 1, Yogyakarta 55281, Indonesia

*Corresponding author (e-mail: yusmaniar@unj.ac.id)

Currently, the development of bioplastics is an effort to overcome environmental problems caused using conventional plastics which are difficult to decompose. In this research, polyvinyl alcohol (PVA)-based bioplastics were synthesized with the addition of carboxymethyl cellulose (CMC) from coconut fibers with variations in the weight percent of CMC using glycerol as a plasticizer and citric acid as a cross linking *agent* using the solution casting method. The results of functional group analysis using FTIR show that CMC was successfully synthesized with the presence of characteristic wave groups of 1593 cm⁻¹ (*Stretching* C=O) and 1412 cm⁻¹ (*bending* -CH₂). In addition, functional group analysis samples on bioplastic films show that there is a wave number of 1731 cm⁻¹ (*Stretching* -C=O) in typical citric acid and 1543 cm⁻¹ (*Stretching* -C=O) of carboxyl groups, which do not appear in PVA. Increasing variations in CMC, glycerol and citric acid in the PC7GA sample resulted in the highest air resistance value (59.9%). However, on the contrary, there was a decrease in the value of the mechanical properties (5.02 MPa). The best sample variation in the biodegradation test was PC1G with a weight loss value of 50.47%. In the shelf-life test, the best sample variation was obtained, namely PC3GA with a chili weight loss value of 27%. Based on research results, adding additives to PVA-based bioplastic films can increase air resistance, biodegradation and shelf life without significant improvement in mechanical properties.

Keywords: Polyvinyl alcohol (PVA), carboxymethyl cellulose (CMC), biopolymer composites

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Currently, the use of petroleum-based plastic packaging is increasing and has been widely adopted [1]. This is because plastics are versatile materials that are inexpensive, flexible, transparent, lightweight, non-corrosive, and easy to manufacture. Such plastics are commonly derived from synthetic polymers such as polyethylene (PE), polystyrene (PS), polypropylene (PP), and others, which pose significant environmental challenges due to their finite and non-renewable nature, as well as their resistance to microbial degradation [2]. It is estimated that plastics require approximately 20–500 years to fully degrade, depending on the type of plastic and environmental conditions. Indonesia ranks as the second-largest contributor of plastic waste in the world, after China, with an estimated production of 3.2 million tons [3, 4].

Therefore, solutions are needed to overcome these problems. One of the approaches that has been developed is biodegradable plastics derived from renewable natural resources that are environmentally

friendly, such as cellulose, starch, proteins, chitosan, and other natural compounds, which can be degraded by microorganisms. These bioplastics can decompose into environmentally harmless compounds such as carbon dioxide, water, and humic substances within a maximum of 6 to 9 months [5].

Based on this capability, biodegradable plastics in the form of composites have become an attractive material for further development. Several studies have focused on composites of polyvinyl alcohol (PVA) and carboxymethyl cellulose (CMC) due to their ease of synthesis and flexibility as eco-friendly food packaging materials [6, 7]. Polyvinyl alcohol (PVA) is utilized because of its unique characteristics. PVA is a hydrophilic, non-toxic synthetic polymer with excellent film-forming ability. However, it still exhibits low water resistance and limited biodegradability. When applied in composites, PVA is generally used as the matrix, which requires additional reinforcing additives to improve the functionality of bioplastic films [8].

Carboxymethyl cellulose (CMC) is a modified form of cellulose that is non-toxic, water-soluble, biocompatible, and naturally biodegradable, making it a potential additive for bioplastics [9]. CMC can be synthesized from agricultural waste rich in cellulose, as cellulose is the most abundant biopolymer on Earth. One such source is coconut fiber, which contains approximately 43.44% cellulose [10]. Indonesia is the world's largest producer of coconut fiber, with an annual production of about 17,500 MT. The high production volume of coconut fiber in Indonesia leads to an increase in coconut fiber waste generation. Therefore, coconut fiber has been utilized as a raw material for producing CMC [11].

Despite its advantages, PVA/CMC-based bioplastics also have limitations, particularly their low elasticity. This drawback can be addressed by the addition of plasticizers. Glycerol is commonly used as a plasticizer because of its hydrophilic nature, which enhances the flexibility of the films [12]. The combination of these polymers is highly hydrophilic, making them easily soluble in water. Therefore, several previous studies have incorporated PVA/CMC with citric acid as a crosslinking agent. Citric acid is a natural compound containing three carboxyl functional groups, enabling it to form ester bonds with the hydroxyl groups of polysaccharides. Citric acid is selected as a natural crosslinking agent because it can enhance the water resistance of films, particularly for applications in food packaging [13, 14].

The present study aims to develop PVA-based bioplastics with the addition of CMC, glycerol (as a plasticizer), and citric acid (as a crosslinking agent) to address environmental issues. The objective is to obtain a bioplastic composition with optimal mechanical properties, water resistance, biodegradability, and shelf life. Accordingly, the novelty of this study is the incorporation of cellulose derived from coconut fiber waste into a glycerol-plasticized and citric acid-crosslinked PVA/CMC bioplastic system, followed by a systematic evaluation of its effects on the properties of the resulting bioplastic films.

EXPERIMENTAL

Material

The materials used in this study were polyvinyl alcohol (Sigma Aldrich, 99%), sodium hydroxide (Sigma Aldrich, 98%), sodium hypochlorite (Sigma Aldrich), glacial acetic acid (Sigma Aldrich, 99%), isopropanol (Sigma Aldrich, 99%), monochloroacetic acid (Sigma Aldrich), methanol (Sigma Aldrich, 99%), ethanol (Sigma Aldrich, 99%), glycerol (Sigma Aldrich, 99%), Coconut fiber received from Indonesia.

Method

Isolation of CMC from Coconut Fiber

The preparation of cellulose and CMC from coconut fiber was conducted according to the method described by Ndruru et al. [15], with slight modifications. The initial preparation (pre-treatment) of coconut fiber began by separating the husk and shell, followed by cutting and washing with distilled water until clean, and then drying under sunlight to reduce the moisture content of the fibers. The dried samples were sieved using a 40-mesh sieve and then blended with distilled water for 10 minutes. Afterward, the coconut fiber was heated in distilled water at 50 °C for 2 hours and filtered. This process was repeated once to remove residual impurities. The next step was delignification. The resulting residue was heated in 500 mL of 2% sodium hydroxide (NaOH) solution and stirred at 80 °C for 2 hours. The residue was then filtered and rinsed with distilled water until a neutral pH was reached. This process was repeated once. The delignified coconut fiber was subsequently dried in an oven at 70 °C for 8 hours.

The delignified coconut fiber was then subjected to a bleaching process by heating it in a 1.7% sodium hypochlorite (NaOCl) solution with the addition of glacial acetic acid, while stirring for 1 hour. The residue was then filtered and rinsed with distilled water until neutral pH was achieved. This process was repeated four times until the fibers turned white. The bleached coconut fiber was subsequently dried in an oven at 70 °C for 8 hours. The cellulose obtained was then characterized using FTIR.

The next step was the isolation of carboxymethyl cellulose (CMC). Five grams of the bleached cellulose were mixed with 100 mL of isopropanol and 20 mL of 20% sodium hydroxide (NaOH), followed by stirring at room temperature for 1 hour. Subsequently, 6 grams of monochloroacetic acid (MCA) were added, and the mixture was stirred at 60 °C for 3 hours, then filtered. The resulting residue was neutralized with glacial acetic acid and soaked in 100 mL of methanol overnight. The residue was filtered again, washed with ethanol, and dried in an oven at 70 °C for 8 hours.

Preparation of Bioplastic Film

Bioplastic films were prepared using the solution casting method according to previously reported procedures, with slight modifications [14, 16]. A total of 5 g of PVA was dissolved in 100 mL of distilled water at 90 °C for 2 h. In a separate beaker, different concentrations of CMC (0%, 1%, 3%, and 7%) were dissolved in 50 mL of distilled water, along with the addition of 0.15 mL of glycerol in variations 2, 3, 4, and 5. Furthermore, 360 mg of citric acid was added in variations 3, 4, and 5. The two solutions were then

combined and stirred at room temperature for 2 hours. The resulting mixture was cast onto a glass plate and dried at room temperature. Finally, the film was carefully peeled off.

Characterization

Carboxymethyl cellulose (CMC) and the bioplastic films were characterized using FTIR (Shimadzu IR Prestige-21) to analyze their functional groups [14]. The mechanical properties of the bioplastic films were evaluated using a Toyoseiki Stograph VGS-5E following the ASTM D882 method, with three repetitions. Film thickness was measured using a micrometer screw gauge [17]. Water resistance was tested by immersing the samples in water for 24 hours [18]. Biodegradability was assessed by burying the samples in soil for 10 days [19]. Finally, shelf-life testing was conducted by wrapping samples with the bioplastic films and storing them in a refrigerator for 6 days [20]. The chemical composition of coconut fiber (α -cellulose, hemicellulose, lignin, and moisture content) can be analyzed using standard ASTM methods (ASTM D1103-55T, ASTM D1104-56, ASTM D1106-56, and ASTM D4442-92, respectively). However, in the present study, these analyses were not experimentally performed.

RESULTS AND DISCUSSION

Characterization of Functional Groups of Cellulose and Carboxymethyl Cellulose (CMC)

Four types of samples were subjected to functional group analysis: coconut fiber after pre-treatment, after

delignification, after bleaching, and carboxymethyl cellulose (CMC) are presented in Figure 1.

According to Figure 1, the absorption band at 3344 cm^{-1} corresponds to O–H vibration, indicating the presence of hydroxyl groups in cellulose structure, while the band at 2908 cm^{-1} indicates C–H stretching of aliphatic groups. In the IR spectrum of raw coconut fiber, a band at 1731 cm^{-1} was observed, corresponding to C=O vibration in hemicellulose, which disappeared after the delignification and bleaching stages. This peak disappeared after delignification and bleaching treatments, indicating effective removal of hemicellulosic components. The disappearance of the 1731 cm^{-1} band has been widely reported as an indicator of hemicellulose removal during alkaline and bleaching treatments of natural fibers [18]. In the delignified coconut fiber, the band at 1592 cm^{-1} indicated the aromatic framework of lignin, which also disappeared after bleaching [14]. After bleaching, the disappearance of the bands at 1731 cm^{-1} and 1592 cm^{-1} confirmed the removal of hemicellulose and lignin.

Moreover, the bands at 1593 cm^{-1} (COOH stretching) and 1412 cm^{-1} ($-\text{CH}_2$ bending) indicate the successful synthesis of carboxymethyl cellulose (CMC). Additionally, the absorptions at 1160 cm^{-1} and 1031 cm^{-1} correspond to C–O–C groups and C–O stretching in cellulose. Finally, the peak at 895 cm^{-1} represents the glucose ring stretching vibration in cellulose. These functional group changes confirm successful conversion of cellulose into CMC, in agreement with reported FTIR analysis of carboxymethyl cellulose derived from agricultural biomass.

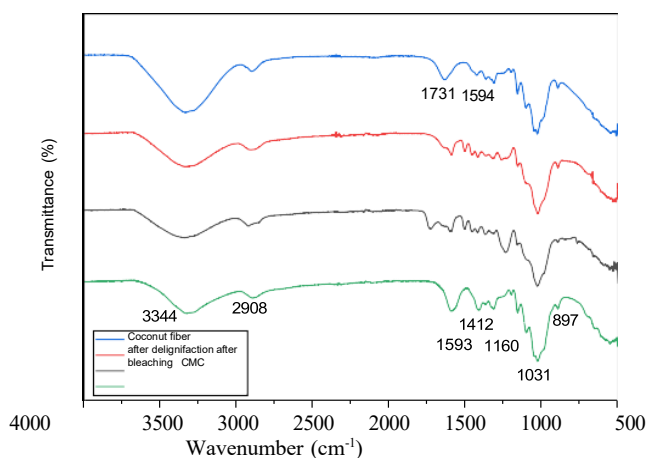


Figure 1. FTIR spectra of (a) raw coconut fiber, (b) after delignification, (c) after bleaching, and (d) CMC.

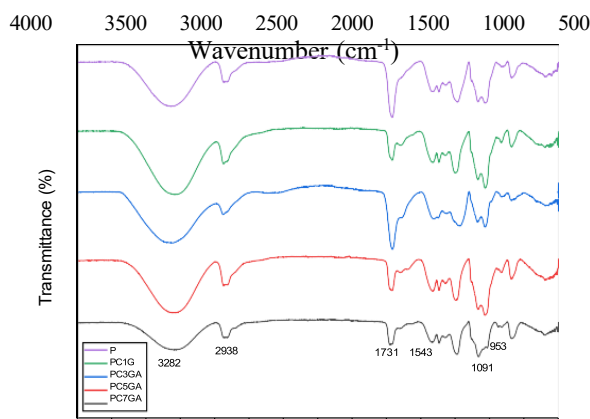


Figure 2. FTIR spectra of bioplastic.

Characterization of Functional Groups of Bioplastic

Different formulations of PVA/CMC composite films with varying compositions of CMC and crosslinking agent were subjected to functional group analysis to evaluate the effect of composition on molecular interactions within the biopolymer matrix. Figure 2 presents the FTIR spectra of the PVA/CMC composite films.

Figure 2 presents the FTIR spectra of PVA/CMC composite films. The broad band at 3282 cm^{-1} corresponds to O–H stretching vibrations, indicating hydrogen bonding interactions between PVA and CMC. The appearance of a band at 1731 cm^{-1} is associated with ester carbonyl (C=O) groups, suggesting successful crosslinking between PVA and citric acid, which is consistent with previous reports on citric acid-crosslinked polymer films. Citric acid crosslinking in PVA-based films typically introduces ester C=O peaks around $1720\text{--}1740\text{ cm}^{-1}$ as reported in PVA/CMC composite systems [14–16]. Meanwhile, the band at 1543 cm^{-1} is attributed to carboxyl group vibrations, further confirming the interaction between polymer chains. The absence or reduction of certain characteristic peaks of pure PVA indicates strong intermolecular interactions

and structural modification in the composite film system.

Thickness Measurement of Bioplastic Films

The thickness of the bioplastic films ranged from 0.0875 to 0.14 mm (Table 1), which complies with the Japanese Industrial Standard (JIS) requirement of $< 0.25\text{ mm}$. These values are also consistent with previous findings for PVA/CMC films with citric acid variations, reported at 0.07–0.11 mm [6].

Thickness Measurement of Bioplastic Films

The mechanical properties of the bioplastic films were evaluated in terms of tensile strength, and the results are presented in Figure 3.

The tensile strength values are presented in Figure 3. The film containing only PVA exhibited a tensile strength of 21.93 MPa. This result is higher than that reported in a previous study, which was 10.37 MPa [14]. The difference can be attributed to variations in formulation; in this study, 5% PVA was dissolved in 100 mL of solution, whereas the previous study used 5% PVA in 150 mL. The pure PVA sample also demonstrated the highest tensile strength compared to the other variations.

Table 1. Measurement of Bioplastic Film Thickness.

Sample name	Point (mm)					Average
	1	2	3	4	5	
P	0.07	0.1	0.1	0.08	0.07	0.0875
PC1G	0.07	0.08	0.1	0.1	0.08	0.09
PC3GA	0.10	0.12	0.12	0.12	0.13	0.1225
PC5GA	0.09	0.11	0.11	0.12	0.09	0.1075
PC7GA	0.11	0.14	0.14	0.12	0.16	0.14

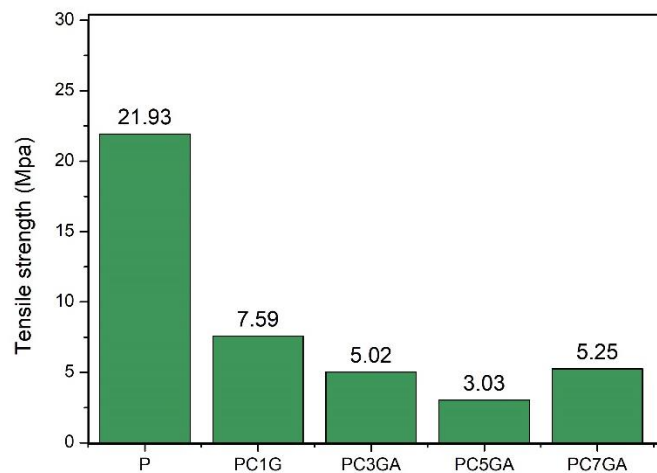


Figure 3. Effect of bioplastic film variations on tensile strength.

In the second sample variation (PVA/CMC 1%/glycerol), the tensile strength decreased to 7.59 MPa. This result contradicts previous findings, which indicated that the addition of additives such as CMC and glycerol should increase the tensile strength of the film. Moreover, when compared with earlier studies, this tensile strength value remains below 12 MPa [14].

In the third, fourth, and fifth sample variations, which included higher concentrations of CMC (3%, 5%, and 7%) along with citric acid (360 g), the tensile strength values were 5.02 MPa, 3.03 MPa, and 5.25 MPa, respectively. These results also differ from

previous studies, which suggested that the addition of citric acid as a crosslinker should increase tensile strength. The discrepancy may be due to the fact that, in addition to functioning as a crosslinker, citric acid might also act as a plasticizer, producing highly elastic films that consequently reduce tensile strength. This interpretation is supported by the high elongation at break value observed in Figure 4, which reached 304.4%. Nevertheless, the tensile strength values obtained in this study are still higher than those reported in an earlier study, which showed that PVA/CMC/citric acid films had a maximum tensile strength of 3.54 MPa [13].

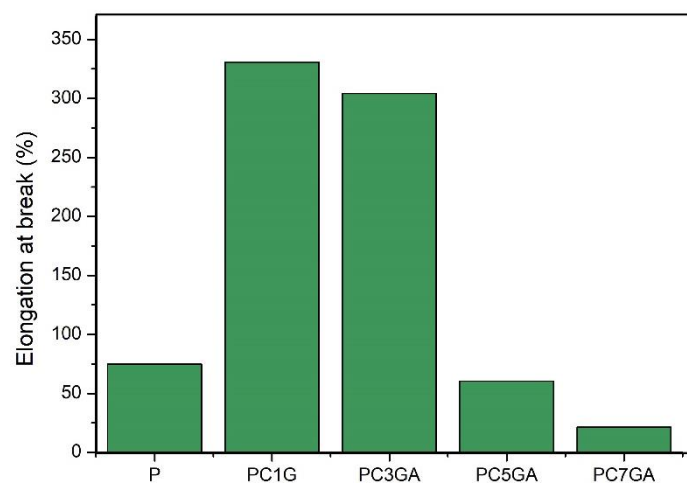


Figure 4. Effect of bioplastic film variations on elongation at break.

Thickness Test of Bioplastic Film

The swelling behavior of the bioplastic films was evaluated to determine their water absorption characteristics. Figure 5 presents the swelling behavior of the films. Pure PVA exhibited a swelling value of 0% due to its high-water solubility, attributed to the presence of hydroxyl groups and the absence of additives. With increasing concentrations of CMC, citric acid, and glycerol, the polymer network became more rigid, restricting water diffusion and consequently reducing the swelling values across the film variations. The water resistance of the bioplastic films was evaluated to determine their stability against water and the results are presented in Figure 6.

The PC1G sample, with the addition of 1% CMC, exhibited improved water resistance compared to pure PVA due to hydrogen bonding between PVA, CMC, and glycerol. However, increasing CMC content led to higher swelling values owing to its hydrophilic nature. In contrast, the incorporation of citric acid in the PC3GA, PC5GA, and PC7GA samples enhanced water resistance through ester bond formation, which reduced hydroxyl groups and increased structural density. The water resistance values obtained were 0%, 50%, 51.2%, 55.2%, and 59.9%, with the highest performance observed in the PC7GA sample containing 7% CMC.

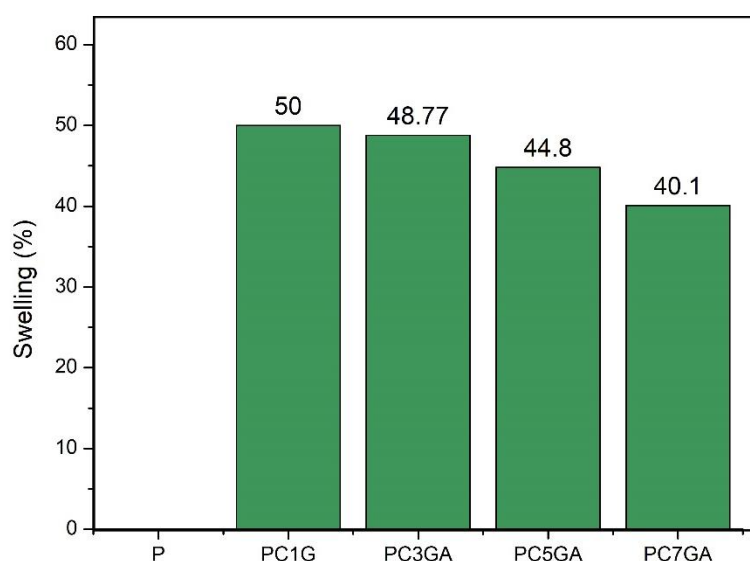


Figure 5. Effect of bioplastic film variations on swelling test.

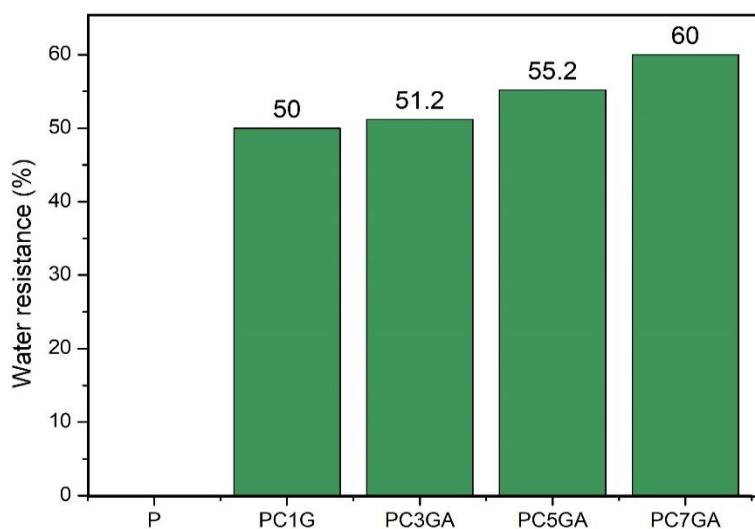


Figure 6. Effect of bioplastic film variations on water resistance.

Biodegradation Analysis

The biodegradation performance of the bioplastic films was evaluated by soil burial testing, and the results are presented in Figure 7. The highest biodegradation performance was observed in sample PC1G, with a weight loss of 50.74%. This result is attributed to the presence of PVA, 1% CMC, and glycerol, all of which are hydrophilic and thus more susceptible to microbial degradation. In contrast, samples containing higher CMC concentrations (3%,

5%, and 7%) with citric acid exhibited progressively greater weight loss, reaching 56.79%, 71.00%, and 83.84%, respectively.

Shelf-Life Evaluation

The shelf-life performance of the bioplastic films was evaluated by assessing their effectiveness as packaging materials for fresh produce, and the results are presented in Figure 8.

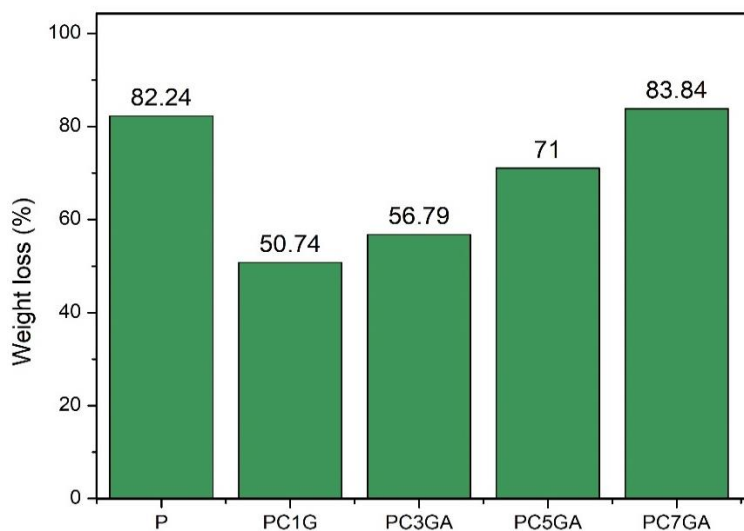


Figure 7. Effect of bioplastic film variations on weight loss.

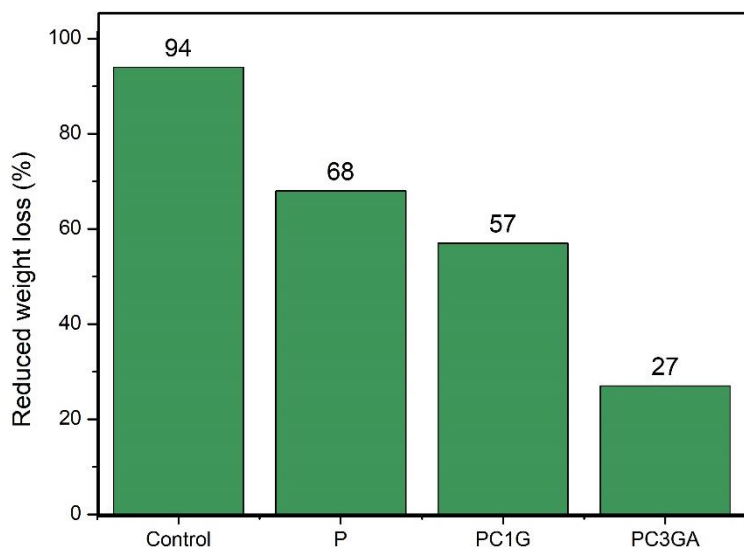


Figure 8. Effect of bioplastic film variations on the shelf life of packaged chili peppers.

Figure 8 demonstrates that the incorporation of bioplastic films as packaging significantly reduced weight loss in chili peppers. The unpackaged control exhibited the highest weight loss (94%), whereas samples packaged with bioplastic films (P, PC1G, and PC3) showed lower losses of 68%, 57%, and 27%, respectively. These results suggest that bioplastic films effectively minimize moisture loss and thereby extend the shelf life of chili peppers. Moreover, as reported by Kanatt and Makwana [14], mechanical strength and water resistance in PVA/CMC-based films are influenced by hydrogen bonding interactions and citric acid crosslinking. In addition, Diop et al. [16] reported that crosslinking contributes to improved structural stability in biopolymer films. Furthermore, Pires et al. [5] highlighted that biodegradation behavior of bio-based polymers is generally affected by polymer hydrophilicity and network structure. This agreement with previous studies provides scientific support for the observed trends in this work.

CONCLUSION

Cellulose was successfully isolated from coconut fiber through pre-treatment, delignification, and bleaching, yielding purified cellulose. Moreover, carboxymethyl cellulose (CMC) was synthesized via alkalization and carboxymethylation, confirmed by FTIR through the appearance of characteristic COOH and CH₂ groups. The incorporation of CMC, glycerol, and citric acid influenced the film thickness and mechanical properties, where increasing additive content enhanced flexibility but reduced tensile strength. Swelling and water resistance behavior were governed by the balance between hydrophilic functional groups and crosslinked network structure, resulting in reduced swelling and improved water resistance at higher crosslinking levels. Biodegradation analysis confirmed that all films were biodegradable, with weight loss ranging from 50.74% to 83.84%. Shelf-life evaluation demonstrated that the bioplastic films effectively reduced weight loss in chili peppers compared to the control sample, indicating improved packaging performance. The developed bioplastic films exhibited a synergistic combination of mechanical flexibility, barrier properties, biodegradability, and shelf-life performance, supporting their potential application as sustainable food packaging materials. Future studies may explore the scalability of the fabrication process and evaluate the performance of these bioplastic films for broader food packaging applications under real storage conditions.

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