

Effect of Edible Coating Material Composition Based on Chitosan-Gelatin-CaCl₂ and Cinnamon (*Cinnamomum verum*) Essential Oil on the Protection of Cocoa Beans (*Theobroma cacao* L.) During Storage

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Abstract

Chocolate, a highly demanded commodity, is derived from cocoa beans which undergo fermentation and drying to achieve a safe moisture content of up to 7.5% for storage. Storage conditions significantly impact cocoa bean quality. While edible coatings are commonly used to preserve food quality during storage, their application to cocoa beans using composite polymers and essential oils remains underexplored. This study aimed to evaluate the effectiveness of composite edible coatings, specifically chitosan-gelatin-CaCl₂ with cinnamon essential oil, in preserving cocoa bean quality over a 28-day storage period. Six variations of coating solutions were applied to peeled cocoa beans: K1= chitosan:gelatin 1:2 + CaCl₂ 1%, K2= chitosan:gelatin 1:2 + CaCl₂ 1% + 0.1% essential oil, K3= chitosan:gelatin 1:2 + CaCl₂ 1% + 0.3% essential oil, K4= chitosan:gelatin 2:1 + CaCl₂ 1%, K5= chitosan:gelatin 2:1 + CaCl₂ 1% + 0.1% essential oil, and K6= chitosan:gelatin 2:1 + CaCl₂ 1% + 0.3% essential oil, and KO serving as a control without treatment. Quality parameters such as water content, fat content, and pH were analysed using ANOVA ($\alpha = 0.05$). The results indicated that edible coatings significantly protected against changes in fat content but did not significantly affect water content or pH. The most effective treatment was K6, composed of chitosan:gelatin with the ratio of 2:1 + 1% CaCl₂ + 0.3% cinnamon essential oil, providing optimal protection during storage.

Keywords

Cocoa Beans, Edible Coating, Chitosan, Gelatin, Cinnamon Essential Oil

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1. INTRODUCTION

Chocolate and various chocolate products are products that are popular with various groups. In 2018-2019, it was recorded that the consumption of confectionery products was 7.7 million tons globally (Zhong et al., 2021). In addition to its rich taste, the consumption of chocolate products, especially dark chocolate, also has a number of health effects, including protection from diabetes, anti-inflammatory effects, maintaining good fat levels in the blood, and protection from oxidative stress (Samanta et al., 2022). Cocoa beans are the main food raw material in making chocolate which goes through a series of processing stages to produce chocolate that is ready to be consumed. The high polyphenol content in cocoa beans is around 10% of its dry weight, making cocoa beans a source of polyphenols that have health effects. Polyphenol compounds in cocoa beans include proanthocyanidins as much as 58%, catechins as much as 37%, and anthocyanidins as much as 4%. Cocoa bean processed products, namely dark chocolate, have a

relatively higher content of cocoa, theobromine, and flavonoid compared to other types of cocoa products, making cocoa beans and chocolate act as functional and nutritional foods (Montagna et al., 2019).

Cocoa beans are harvested from ripe cocoa pods that have been sorted. After harvesting, the cocoa pods are allowed to ripen for a period before being opened to extract the beans. Once the beans are collected, they undergo fermentation for 5 to 6 days. This fermentation process is essential for removing mucilage and preventing the beans from germinating. Additionally, it helps develop color and flavor precursors in the beans. After fermentation, the beans are dried to achieve a moisture content of 6-8%, or a maximum of 7.5% according to SNI standards. Drying can be done in direct sunlight or using artificial methods (Subroto et al., 2023).

Currently, cocoa beans are stored by packing dry beans in watertight sacks or plastic, aiming to maintain a storage environment with low humidity ($\leq 70\%$). Despite having been dried to the appropriate moisture level, the hygroscopic nature of

the beans means that water content can still change (Dumadi, 2011). To address the challenges of cocoa bean storage, one solution is the use of edible coatings. These coatings are thin layers made from biopolymers, which are eco-friendly and serve as a semipermeable barrier. They help regulate the exchange of gases like O₂, CO₂, and ethylene between the food products and their environment, as well as manage physiological changes caused by microbial activity or mechanical pressure (Saputra, 2019).

Edible coatings have been extensively used on a variety of food products, including fruits, vegetables, and meat. Chitosan, in particular, exhibits antibacterial properties against *Escherichia coli* and *Staphylococcus aureus* (Li and Zhuang, 2020), as well as antifungal effects against *Aspergillus fumigatus* and *Candida albicans* (Lopez-Moya et al., 2019). Polysaccharides like chitosan can help preserve the appearance and slow the quality decline of cherries (*Prunus avium*) for up to 14 days during cold storage (Pettriccione et al., 2015). Button mushrooms (*Agaricus bisporus*) treated with a combination of chitosan and corn zein protein have shown reduced weight loss and browning at a storage temperature of 4°C (Zhang et al., 2020). Coating Semarang water apple fruit (*Syzygium samarangense*) with sodium alginate and a crosslinking agent like CaCl₂ can extend its shelf life by up to 10 days while maintaining antioxidant activity and inhibiting lipid peroxidation (Duong et al., 2022). Using a chitosan and gelatin-based coating applied through dipping can effectively minimize lipid oxidation, weight loss, and color changes in beef stored for 5 days in retail settings (Cardoso et al., 2016). Cut Fuji apples (*Malus domestica*) are prone to enzymatic oxidation, but a study by Cofelice et al. found that applying an edible coating made from sodium alginate polysaccharide and citronella essential oil can inhibit browning for up to 7 days at a storage temperature of 4°C (Cofelice et al., 2019).

Essential oils are volatile compounds extracted from specific parts of aromatic plants through distillation. These oils are considered safe and are classified as GRAS (Generally Recognized as Safe). In addition to their use in the fragrance industry, essential oils serve as additives with biological properties, including antibacterial and antifungal activities, which can be incorporated into edible coatings (Jackson-Davis et al., 2023). One widely used essential oil known for its strong antimicrobial properties is cinnamon essential oil (*Cinnamomum verum*). This oil contains cinnamaldehyde, an aldehyde compound with various biological effects, such as antifungal, antibacterial, and antioxidant activities (Singh et al., 2021). Research by Kapetanakou et al. (2019) found that adding cinnamon essential oil to sodium edible coatings for apples and pears effectively inhibited the growth of *Aspergillus carbonarius* and significantly reduced the production of ochratoxin. Additionally, Munhuweyi et al. (2017) reported that an edible coating containing a blend of cinnamon (*Cinnamomum verum*), lemon (*Citrus limon*), and oregano (*Origanum vulgare*) essential oils successfully inhibited the growth of fungi such as *Botrytis sp.*, *Penicillium sp.*, and *Piliidiella granati* (Munhuweyi et al., 2017).

To the best of the author's knowledge, there has been no

research on using composite edible coatings with cross-linking agents and cinnamon essential oil for storing cocoa beans. Thus, this study aims to investigate how the composition of an edible coating made from chitosan and gelatin as biopolymer, CaCl₂ as crosslinking agent, and cinnamon essential oil (*Cinnamomum verum*) affects the quality of cocoa beans (*Theobroma cacao* L.), focusing on quality parameters such as moisture content, fat content, and pH. The research will be conducted in several stages, including formulating different variations of the edible coating solution, applying the coating to cocoa beans, testing various parameters, characterizing the coating solutions, and analyzing the data.

2. EXPERIMENTAL SECTION

2.1 Materials

Dried cocoa beans, distilled water, chitosan powder (with a deacetylation degree of at least 94% and a molecular weight of 134 kDa), bloom gelatin 150, CaCl₂, glacial acetic acid, cinnamon essential oil (*Cinnamomum verum*), glycerol, and tween 80, all meeting food-grade standards, as well as technical grade n-hexane. The equipment utilized in the study comprises glassware, a series of Soxhlet extractors, IKA RV-10 rotary evaporator setup, HORIBA LAQUA PH1100 pH meter, Heidolph magnetic stirrer, and FTIR instruments from Shimadzu IR Spirit.

2.2 Methods

2.2.1 Formulation of Edible Coating Solutions and Coating on Cocoa Beans

Chitosan solution was made by dissolving 1% (w/v) chitosan in 1% (v/v) acetic acid. Heating was carried out at a temperature of 60 °C on a hotplate magnetic stirrer while stirring at a speed of 800 rpm. Homogenization of the solution was carried out for approximately 1 hour and then cooled at room temperature. In another beaker glass, 1% (w/v) gelatin was dissolved in distilled water and stirred with a hotplate magnetic stirrer at a speed of 800 rpm until dissolved at a temperature of 55 °C. Chitosan and gelatin solutions with a ratio of chitosan: gelatin (CH: GEL, v/v) 1:2 and 2:1 were mixed in the same glass then added 1% (w/v) CaCl₂, 1% (w/v) glycerol as a plasticizer and 0.1% (w/v) Tween 80 as a surfactant. Variations of the solution with the addition of cinnamon essential oil (EO) were made by adding 0%, 0.1% and 0.3% (w/v) into the chitosan-gelatin-CaCl₂ mixture (Table 1) and mix homogenously (Zhang et al., 2021). Dried cocoa beans are initially peeled and sorted by weight for uniformity, with each treatment done in three replications. They are then immersed in the edible coating solution for roughly 1 minute, drained of excess solution, and air-dried. After drying, the beans are stored in a plastic container at room temperature for 28 days.

2.2.2 Characterization of Edible Coating Solutions

The freeze-dried edible coating film was analyzed using an FTIR spectrophotometer in the range of 4000-500 cm⁻¹ with a resolution of 4 cm⁻¹, and the measurements were conducted

3. RESULTS AND DISCUSSION

3.1 Characterization of Edible Coating Solutions

Characterization of the edible coating solution using FTIR is intended to identify the presence of IR-active functional groups within of the compound and to investigate potential interactions within the solution system. Specific chemical bonds absorb the appropriate energy from the infrared light, causing them to vibrate (Hashemi Gahruei et al., 2020; Khan et al., 2018). Figure 1 shows the FTIR spectrum of the edible coating solutions in comparison to the individual spectrum of chitosan and gelatin.

In the FTIR spectrum of the mixture of chitosan and gelatin (1:2), the K1 variation (Figure 1a), characteristic peaks for both chitosan and gelatin are observed, along with slight shifts at several wave numbers. The absorption intensity at 3250.84 cm^{-1} increased compared to the individual absorptions of chitosan and gelatin, indicating enhanced interaction and the formation of hydrogen bonds among the functional groups of chitosan, gelatin, essential oils, and other components in the solution. Comparing the FTIR spectrum of K1 to that of alginate cross-linked with Ca^{2+} , the peak for the asymmetric stretching vibration of the carboxylate group shifted from 1630.16 cm^{-1} to 1635.86 cm^{-1} , and the symmetric stretching vibration shifted from 1450.46 cm^{-1} to 1453.31 cm^{-1} , suggesting potential ionic cross-linking between the carboxylate group of gelatin and the divalent Ca^{2+} ion (Li et al., 2016; Pragya et al., 2021). A new absorption peak at 1035.43 cm^{-1} , which can be observed in edible coating solution spectrum on different variations (Figure 1b) emerged due to the addition of glycerol, indicating compatibility between the chitosan and gelatin mixture (Karydis-Messinis et al., 2023). The incorporated cinnamon essential oil within the solution showed only minor shift in peak positions (Roy et al., 2023).

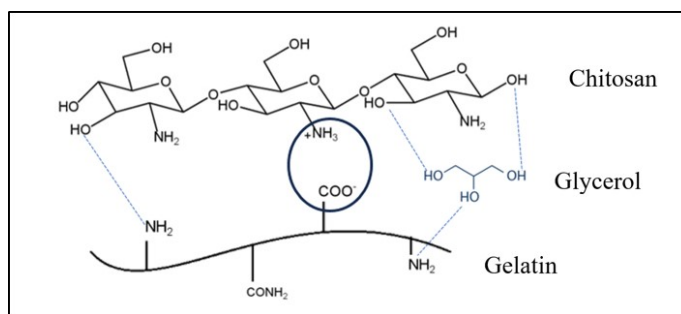


Figure 2. Interaction Between Chitosan, Gelatin, and Glycerol (Samimi Gharaie et al., 2018; Sethi et al., 2022)

The positively charged protonated amine groups in chitosan interact electrostatically with the negatively charged carboxylate groups in gelatin, resulting in the formation of a complex with a three-dimensional structure as seen in Figure 2. In addition, polar functional groups like $-\text{OH}$, $-\text{NH}_2$, and $-\text{COOH}$ in chitosan, gelatin, and glycerol are capable of forming hydrogen bonds with each other indicating compatibility between

the compounds (Roy et al., 2023). The Ca^{2+} ions from dissolved CaCl_2 are likely to interact with the negatively charged carboxylate groups in gelatin, facilitating the formation of a network through electrostatic interactions. This process contributes to a more stable and mechanically strong cross-linked composite structure (Prasetyaningrum et al., 2021; Sethi et al., 2022). Furthermore, the essential oil is dispersed within the edible coating solution, leading to increased turbidity and promoting hydrophobic interactions between the polar molecules, polymers, and components of the essential oil (Sharma et al., 2021).

3.2 Visual Observation of Cacao Beans During Storage

The results of the visual observation of cocoa beans from both the control group and the group treated with an edible coating are presented in Figure 3. On day 1, when the beans were coated with the edible coating with different variations under the initial storage condition, their surfaces appeared shiny. Qualitative observations of the different treatment groups at this stage showed little variation. After 28 days of storage in plastic containers, white spots were observed on the surface of the cocoa beans treated with the edible coating when viewed under a stereo microscope, indicating changes during storage. In contrast, the control cocoa beans showed no significant qualitative differences after 28 days of storage. The white spots observed on the surface of the cocoa beans during storage could possibly be attributed to the growth of fungal mycelia (Rahayu et al., 2021). Among the various treatments, the K6 edible coating (chitosan:gelatin 2:1 with 1% CaCl_2 and 0.3% cinnamon essential oil) caused the least surface damage compared to the other treatments, though it still did not perform better than the control cocoa beans.

3.3 Effect of Edible Coating on Cocoa Beans Water Content

The water content in cocoa beans is a key factor influencing their quality. The acceptable threshold for water content during storage is 7.5%. This level is crucial as it relates to the potential for mold growth. The Figure 4 illustrates the water content of cocoa beans from six edible coating treatments and the control group over a 28-day observation period.

The average water content of beans receiving edible coating treatment on day 1 of storage showed an increase compared to the control beans without treatment. This increase is likely due to the water content present in the edible coating, which can raise the moisture levels of the cocoa beans initially. At the end of the storage period, the water content of control cocoa beans without treatment, as well as those with edible coating treatment, decreased compared to the initial condition. The change in water content for the control group was relatively lower than that of the beans treated with the edible coating. The percentage changes in water content for each group were as follows: 1.86% for K0 (control), 6.21% for K1, 6.56% for K2, 4.27% for K3, 5.42% for K4, 2.04% for K5, and 5.09% for K6. Duncan's test revealed that the change in water content for the control group (K0) was not significantly different from

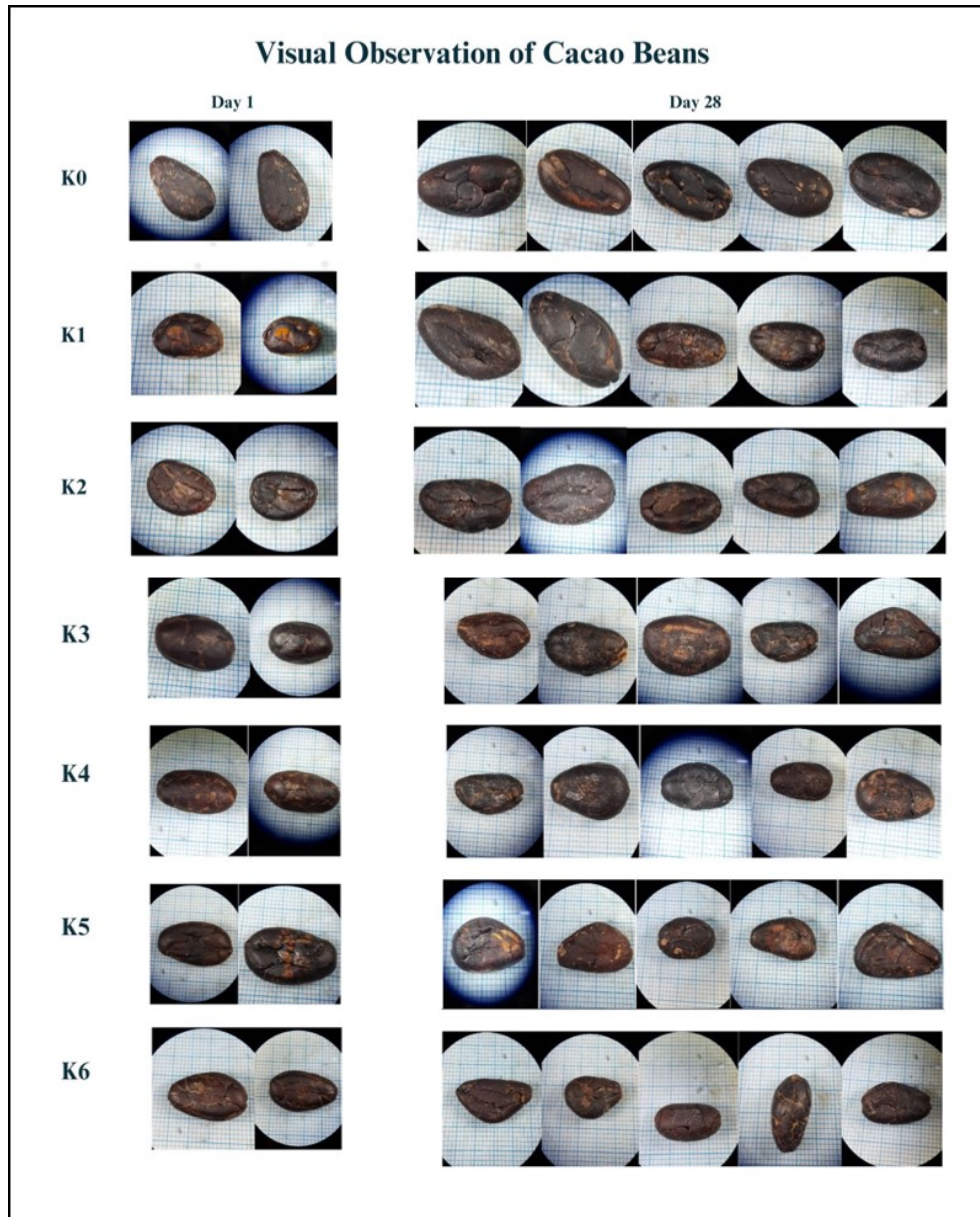


Figure 3. Visual Observation of Cacao Beans for Each Variation During Storage Obtained Under Stereo Microscope

that of the coated beans (K1, K2, K3, K4, K5, and K6). The results indicate that variations in the combination of chitosan, gelatin, and the concentration of cinnamon essential oil in the edible coating did not significantly affect changes in water content during storage, suggesting that the edible coating did not adversely impact water levels during storage.

Research by [Gupta et al. \(2022\)](#) reported that the water content of walnut kernels treated with a composite edible coating made from pea starch and soy protein isolate increased compared to untreated control kernels at the beginning of storage. This increase is believed to result from the high water content of the edible coating solution. However, the same treated walnut kernels showed a decrease in water content after being

stored for 30 days ([Gupta et al., 2022](#)). This finding aligns with the current study, which observed a reduction in water content of cacao beans after 28 days of storage compared to initial conditions. Such a decrease may be attributed to the evaporation of moisture from the edible coating solution and gas exchange with the environment during storage ([Moslehi et al., 2015](#)). The water content in the edible coating can influence the moisture levels of the coated sample. In contrast, the results from the edible coating treatment using a chitosan-gelatin- CaCl_2 composite polymer and the addition of cinnamon essential oil during the storage of cacao beans were less consistent with findings by [Farooq et al.](#), who studied almond kernels. Their research indicated that almond kernels treated with a gum mastic

edible coating at concentrations between 0.5% and 2.0% exhibited a lower increase in water content compared to untreated control kernels over one month of storage (Farooq et al., 2021).

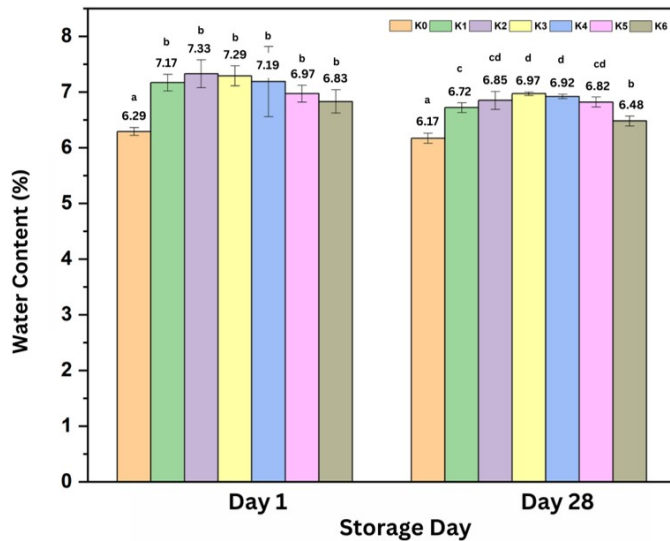


Figure 4. Water Content of Cocoa Beans from Different Treatments at The Start and End of Storage. Identical Letter Symbol Denotes no Significant Difference

The water content in food can fluctuate during storage. Applying an edible coating is intended to regulate the exchange of gases and humidity between the food and its environment. However, the effectiveness of edible coatings in maintaining water content is influenced by the properties of the polymers used. In this study, a mixture of chitosan and gelatin was employed, both of which are known for their ability to form effective films and provide better barriers to water vapor and air than single polymers (Luo et al., 2022). The addition of the cross-linking agent CaCl_2 , as noted in research by (Li et al., 2022), was reported to enhance the strength of the polymer's three-dimensional structure. However, the findings from this study suggest that the chitosan-gelatin- CaCl_2 edible coating was still insufficient to prevent a reduction in water content during storage, and may have even contributed to an increase in water content (Li et al., 2022). This could be attributed to several factors, including the high water content of the edible coating itself and potential incompatibility between the compounds in the coating and the surface of the cocoa beans (Priya et al., 2023).

3.4 Effect of Edible Coating on Cocoa Beans pH

The pH measurements for cocoa bean samples in both the control group and the edible coating treatment group are presented in Figure 5. On day 1 of storage, the control cocoa beans, which received no treatment, had an average pH of 5.72. This aligns with findings from Mulono et al. (2016) which reported that dry cocoa beans typically have a pH ranging from 5 to 5.7. The edible coating treatment, involving varying ratios of chitosan,

gelatin, and different concentrations of cinnamon essential oil (K1 to K6), showed no significant differences in pH values at the start of storage. By the 28th day, the measured pH of the cocoa beans remained consistent within the initial pH range.

Observation data indicated that cocoa beans did not undergo significant changes after being stored for 28 days, whether from the control group or those treated with edible coatings (variations K1 to K6). The average pH change between the control and treatment groups over the 28-day period was not significantly different, as demonstrated by ANOVA results, which yielded a significance value greater than 0.05. This suggests that the edible coating made from chitosan and gelatin, along with essential oils, does not affect the pH of cocoa beans during storage. These findings are consistent with those reported by Perdones et al., who observed that applying a chitosan edible coating to strawberries resulted in an initial pH of 3.68, which decreased to 3.54 with the addition of lemon essential oil; however, this reduction was not significant (Perdones et al., 2012).

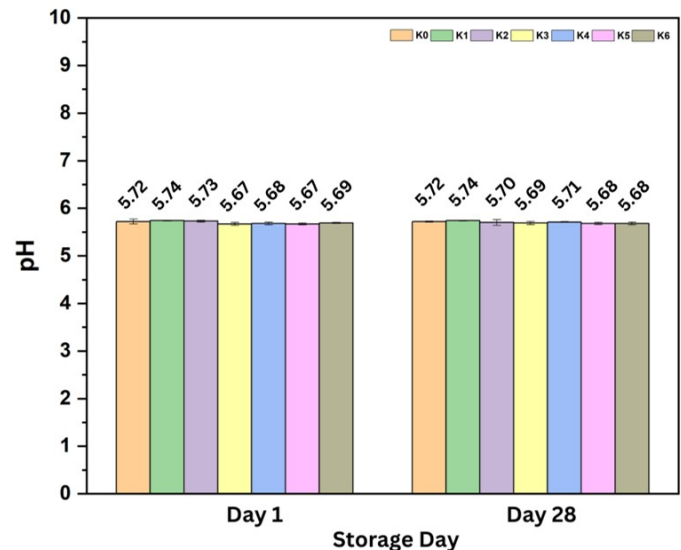


Figure 5. Comparison Graph Showing The pH Levels of Cocoa Beans from Different Treatments at The Start and End of Storage. Identical Letter Symbol Denotes no Significant Difference

pH is a crucial quality parameter for fermented dry cocoa beans, as it influences the resulting chocolate flavor. The acidity level in cocoa beans is linked to acetic acid content, which increases during fermentation. This rise in acetic acid contributes to the formation of aromatic compounds such as alcohols, esters, and acids (Fang et al., 2020). Furthermore, the pH level of the beans plays a significant role in the formation of aroma compounds during the roasting process. Research by Biehl et al., cited in Assa et al. (2019) indicates that cocoa beans with a pH between 5.0 and 5.5 are more likely to produce superior flavors compared to beans with either excessively acidic or nearly neutral pH levels. A pH that is too acidic, around 4.0 to 4.5,

results from excessive acetic acid during fermentation, leading to a more acidic and astringent flavor profile. Conversely, a pH exceeding 5.5 or near neutral can create conditions conducive to mold growth on the beans (Assa et al., 2019; Subroto et al., 2023).

3.5 Effect of Edible Coating on Cocoa Beans Fat Content

The application of an edible coating aims to reduce the decrease in fat content in cocoa beans by creating a barrier that limits oxygen exposure to the samples. The fat content of both control cocoa beans and those treated with the edible coating is illustrated in Figure 6.

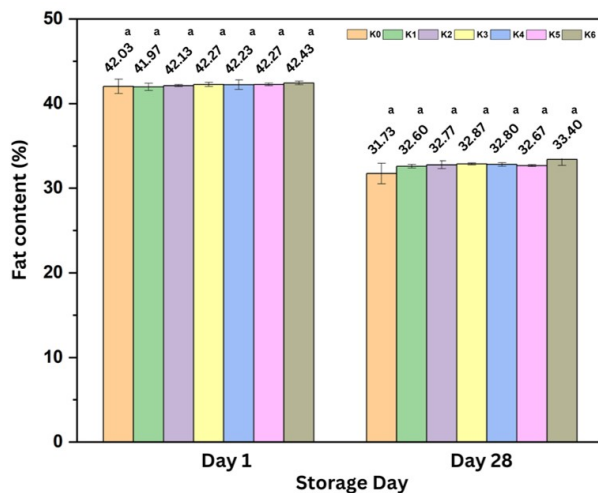


Figure 6. Fat Content of Cocoa Beans Across Various Treatments at The Beginning and End of Storage. Identical Letter Symbol Indicates No Significant Difference

At the start of the storage period, the fat content of the control cocoa beans (without treatment) was 42.03%. The application of edible coatings with varying polymer compositions and essential oil concentrations did not result in significant differences in fat content at the beginning of storage. By the end of the storage period on day 28, all cocoa beans experienced a decrease in fat content. ANOVA statistical analysis indicated significant differences in fat content changes during storage ($p < 0.05$) between the control and treatment groups. However, Duncan's test revealed that while the change in fat content for the control group (K0) was significantly different from all treatment groups, there were no significant differences among the treatment groups themselves. Among the six treatments, K6 comprising a chitosan:gelatin with the ratio of 2:1 coating with 1% CaCl₂ and 0.3% cinnamon essential oil exhibited the lowest change in fat content, at 21.13%, compared to 24.53% in the control group.

Previous research by Zibaei-Rad et al. (2024) on pistachio fruit coated with polysaccharides derived from *Lallemania royleana* demonstrated significant protection against changes in fat content after 35 days of storage. This protection is attributed to the coating's ability to limit oxygen entry into the

fruit matrix, thereby minimizing oxidation and fat decomposition (Zibaei-Rad et al., 2024). Similarly, Azimzadeh and Jahadi (2018) found that cashew nuts coated with a chitosan-based edible coating, enhanced with antioxidants from *Laurus nobilis* extract, exhibited a lower increase in peroxide value compared to untreated nuts, with a significant difference observed (Azimzadeh and Jahadi, 2018). The findings of the current study are consistent with these earlier studies, though the effectiveness of the protective measures differs.

The chemical composition of cacao beans includes macronutrients such as 10% protein, 25% carbohydrates, and fat (Penido et al., 2021). Fat makes up a significant portion of cocoa beans, representing 47-58% of their dry weight and holding substantial economic value. The fat in cocoa beans is composed of 57-64% saturated fatty acids and 36-43% unsaturated fatty acids (Sari et al., 2023). During storage, the unsaturated fatty acids with double bonds are particularly vulnerable to oxidation. This oxidation leads to the formation of hydroperoxide compounds as primary products, which can decompose further into secondary products, including aldehydes such as hexanal. As a result, the oxidation process contributes to a reduction in fat content during storage, as saturated fatty acids can convert into volatile aldehyde compounds (Gama et al., 2018; Geng et al., 2023). Even with the application of an edible coating, the observed decrease in fat levels may occur due to differences in the characteristics of the edible coating solution and the properties of the cocoa bean surface, resulting in an uneven protective layer.

High fat content in dried coco beans resulting in hydrophobic characteristic associated with high surface tension against water or polar compounds and indicates low surface energy (Lapcik et al., 2017; Law, 2015). Low surface energy suggests that interactions between the cocoa bean surface and any applied solution or compound will primarily occur through dispersion forces, limiting polar interactions between the cocoa bean surface and the edible coating solution. To achieve a uniform layer across the entire surface of the sample, it is essential to optimize the balance between the cohesion properties of the particles in the solution and the adhesion of that solution to the sample surface. Generally, edible coating solutions exhibit high hydrophilicity due to their content of polar compounds and the relatively large amounts of solvents used.

Edible coatings with high hydrophilicity tend to have low adhesion when applied to surfaces with high hydrophobic characteristics (Priya et al., 2023). The incorporation of surfactants, such as Tween 80 and cinnamon essential oil, is intended to lower the surface tension of the coating solution and enhance its hydrophobic properties, allowing for better adherence to the surface of cocoa beans (Sapper et al., 2019). The balance between the adhesion force of the edible coating solution and the intermolecular cohesion forces within the coating is crucial for maintaining the quality of cocoa beans. The best protection against fat content loss during storage was achieved with the treatment variation K6, which included a chitosan and gelatin with ratio of 2:1, CaCl₂, and 0.3% cinnamon essential

oil. These findings align with research by Yusof et al. (2019), which indicated that composite edible coatings made from chitosan and starch, combined with glycerol, showed that increasing chitosan concentration reduces solution hydrophilicity, while adding cinnamon essential oil enhances the hydrophobic properties of the coating, thereby increasing adhesion (Yusof et al., 2019). Increasing the content of chitosan in the matrix of chitosan-cassava starch bioplastic also demonstrates higher contact angle, leading to higher hydrophobic characteristic of the bioplastic or film surface (Kusumawati et al., 2025). Further analysis of water content, pH, and fat content for each variation is necessary to assess the quality of edible-coated cacao beans stored for over a month

4. CONCLUSIONS

The study demonstrates that the edible coating composed of chitosan, gelatin, and CaCl_2 effectively protects cocoa beans from fat content changes during storage compared to the control group, although variations among treatment groups were not significant. While the coating successfully minimizes fat loss, it does not significantly impact water content or pH levels over time. Additionally, incorporating cinnamon essential oil into the coating does not enhance its protective effects on fat, water, or pH. The most effective edible coating formula, consisting of chitosan and gelatin in a 2:1 ratio with 1% CaCl_2 and 0.3% cinnamon essential oil, offers the best protection against changes in fat content, reducing it to 21.13% compared to 24.53% in the control group. This highlights the potential of this edible coating in preserving the quality of cocoa beans during storage.

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