

# The Effects of tapioca starch-chitosan-palm olein composite edible film on the quality of milk candy during storage

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## The Effects of tapioca starch-chitosan-palm olein composite edible film on the quality of milk candy during storage

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**Abstract.** Edible film usage has been growing to replace the non-environmental friendly plastic packaging. It can be applied as primary packaging in confectionary/candy products. The current study objective was to evaluate the quality changes of milk candy product which was wrapped using tapioca starch (TS) – chitosan (CH) – palm olein (PO) composite edible film as its primary packaging. Milk candy was produced from the caramelization of UHT bovine milk, sugar and margarine mixture. Various TS-CH-PO composite film solutions were prepared with different CH concentrations i.e. 0.6%, 0.8%, 1.0% and 1.2% (w/v). The solutions were homogenized and dry casted on a teflon-coated pan, resulting in edible film. Milk candy wrapped in edible film and control were stored at ambient temperature and analyzed periodically for its quality changes. Data were collected from 3 repetitions and presented as a descriptive analysis. The resulted edible films had thickness of 0.036-0.053 mm and water vapor transmission rate (WVTR) of 0.038-0.042 g/hr. After 22 storage days, weight loss of control sample was 1.87% while the best treatment observed from 0.8% CH with 0.98% loss. The moisture content decrease for control and best treatment were 4.1% and 1.4%, respectively. Milk candy hardness showed an initial sharp increase followed by steady decrease pattern. The highest hardness value after 22 days storage amongst the treated samples was 3553 N from 0.8% CH concentration. The increase in total plate count of control was 2.25 logs CFU/g while the best treated sample showed an increase of 0.67 logs CFU/g after 8 days of storage. TS-CH-PO composite edible film has shown the ability to retard the negative quality changes of milk candy during storage.

### 1. Introduction

Food packaging waste, mainly produced from plastic, has been an environmental concern. Even though plastic is cheap and provides adequate protection toward food products, plastic has big disadvantage where it can be hardly degraded naturally. Consequently, plastic packaging has been the cause of pollution in water and soil. Edible film can be used to mitigate the problem as it is not only safe and biodegradable but also edible. The film is normally made using biopolymer such as polysaccharide, protein, fat or those in combination [1].

Tapioca starch is one of the ingredients that are often used to make edible film [2-4]. This research focused on the use of tapioca starch combined with other ingredients such as palm oil and chitosan in the preparation of edible film. Chitosan is known to have functional effects such as antioxidant, antimicrobial and emulsifier [5-7]. Meanwhile, palm oil has a hydrophobic property which increases the film polymer resistance to water vapour transfer [8]. Our previous study has shown a successful



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incorporation of palm olein in the tapioca starch-based edible film with the use of chitosan as emulsifying agent. The addition of palm olein and chitosan has improved the characteristic of the composite edible film [9].

In the current study, the TS-CH-PO composite edible film was used as a primary packaging for milk candy product. The use of edible films on milk candy has been done commercially. Therefore, the product is considered suitable to describe the performance of edible film made. Hence, this study aimed to observe changes in physical, chemical and microbiological properties of milk candy during storage as an effect of using TS-CH-PO composite edible film as the primary packaging.

## 2. Materials and Methods

### 2.1. Materials

Chitosan powder (92% diacetylation degree was purchased from Che-Mix, Yogyakarta). Analytical grade acetic acid was obtained from Merck, Germany. Commercial palm olein, tapioca flour, UHT milk, table sugar, margarine were purchased in Semarang. Glycerol was obtained from MKR Chemicals Ltd, Semarang.

### 2.2. Preparation of edible films

Edible films were prepared according to Pratama et al. [9] with little modification. Chitosan powder was dissolved according to the treatments (1.2%; 1.6%; 2.0% and 2.4% b/v) in 0.5% (v/v) acetic acid solution. Palm olein was later added to chitosan solution by 0.6% (v/v) and then homogenized (IKA T25 Ultra Turrax Homogenizer, Germany) for 10 minutes at the speed of 12000 rpm. Tapioca flour suspension with the concentration of 4% w/v was prepared with distilled water, then emulsions of palm oil chitosan was added with the ratio of 1:1 while stirred with a magnetic stirrer. Glycerol plasticizer was added with a concentration of 0.3 ml/gram of total tapioca flour and chitosan. The mixture was then heated while being stirred on the hot plate stirrer until temperature of 50°C was reached, and homogenized one more time (3 minutes, 5000 rpm). After the homogenization, heating, and stirring continued until the temperature of 75°C was reached and was held for 5 minutes to achieve complete gelatinization. A total solution of 60 ml was then poured on a casting plate covered with aluminium foil with an area of 320 cm<sup>2</sup> then dried in a dryer cabinet at 37°C for approximately 18 hours. Resulted edible film was peeled from aluminium foil and ready to be used as packaging. The treatment produced a final chitosan concentration of 0.6%, 0.8%, 1.0% and 1.2% w/v.

### 2.3. Preparation of milk candy and experimental design

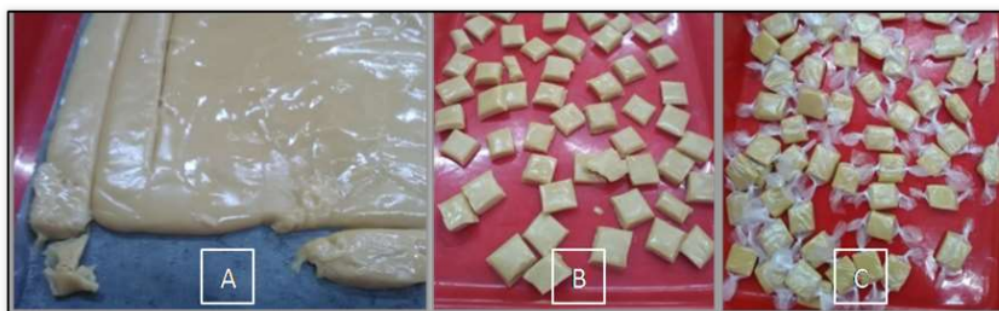
Milk candy was prepared based on Faradillah [10] where mixture of UHT milk, table sugar and margarine with ratio of 50:10:1 was by stirring under mild heating until sugar complete dispersed and the mixture caramelized. The caramel was cut into dimension of 2x2x0.5 cm and was left to harden and cool for 10-15 minutes and then wrapped in edible film that had been prepared. A Descriptive Analysis was obtained from 4 treatments and 1 control (unwrapped candy) with 3 repetitions. Each treatment encountered 3 times of repetition. The packaged candy is then stored in a sealed plastic box (but not airtight) and analyzed on the 1<sup>st</sup>, 8<sup>th</sup>, 15<sup>th</sup> and 22<sup>nd</sup> days of storage.

### 2.4. Observed parameters

Several parameters observed include film thickness with micrometre, water vapor transmission rate (WVTR) with ASTM Method E96-95 [11], weight loss, water content with oven method, hardness (with texture analyzer, Brookfield-Germany), and total plate count (TPC) by using nutrient agar. All data were processed using Microsoft Excel to obtain suitable mean, standard deviation or graph.

### 3. Results and discussions

The mixture of milk, sugar and other materials caramelized and thickened while the colour changed to brown. After pouring it into the baking pan, this mixture was immediately cut to the desired size before it hardens. The resulting candies were brownish yellow and had glossy surface. Edible films were made at the same time of milk candies preparation. Milk candies were cut into pieces and then wrapped with edible film. Texture of the milk candy hardens as it cools down, therefore the cutting must be done when it was still warm and thus elastic [10]. Figure 1 showed the milk candy mixture before being cut, after being cut and after being wrapped with edible film.



**Figure 1.** (A) Caramelized milk candy mixture; (B) Milk candy before wrapping; (C) Milk candy wrapped with edible film

#### 3.1. Thickness and WVTR edible film

As a packaging medium, edible films can be categorized as flexible packaging that can be applied as packaging for food products such as milk candy. One of the factors which determine the flexibility of this film is the thickness of the film. The films that were too thick will be rigid and easily cracked/torn when used to wrap. As shown in Table 1, the resulting thickness of the film is very thin, which is in the range of 0.036-0.053 mm. The resulted thickness which was very thin was affected by the casting method. The casting method was performed using a baking sheet covered with aluminium foil. The adhesion force between the edible film mixture and aluminium foil was quite high, resulting the ability of the mixture to spreads well. In the preliminary study, non-sticky teflon pan was also used as the base of the cast. However, the mixture did not spread well so it requires more volume of mixture to coat all the surface of the casting base which resulted in thicker films.

The treatment of different chitosan concentrations did not show a specific pattern on the thickness of the film produced. This may be due to the ratio of chitosan to the overall mixture which is not too high, where the largest part of the solid used is derived from tapioca starch so the effect is not too visible.

**Table 1.** Thickness and WVTR of TS-CH-PO composite edible film with different chitosan concentration

Chitosan (%)	Thickness (mm)	WVTR (g/h)
0.6	0.053 ± 0.014	0.0387 ± 0.0006
0.8	0.041 ± 0.008	0.0380 ± 0.0004
1.0	0.036 ± 0.004	0.0420 ± 0.0010
1.2	0.048 ± 0.008	0.0400 ± 0.0003

WVTR showed how much water mass (water vapour) can pass through the film. High WVTR showed that the packaging function as a barrier was bad, which results in loss of mass of the product due to evaporation or increase in mass in the case of hygroscopic products. This change may affect the



quality characteristics of food products inside the packaging. As could be seen in Table 1, treatment with 0.8% chitosan had the lowest WVTR and thus the best moisture barrier properties. Film thickness was one of the determinants of WVTR. In general, the increase in thickness will increase the barrier properties of the packaging so that the value of WVTR was lower. However, films that were too thick will become inflexible which can lead to film disintegration and resulting in the loss of the film's barrier properties. The film composite matrix also determines the nature of film retention. A proportion that was not ideal will produce an unfavourable matrix and it can also reduce the WVTR value.

### 3.2. Weight loss and moisture content of milk candy during storage

Figure 2 showed changes in moisture content and weight of milk candy during storage. In general, candy milk products experience an increase in weight loss (decrease in weight) and decrease in water content during storage. This showed that the loss of water due to evaporation was the cause of the weight decrease of milk candy. After 22 days of storage, the control treatment that did not use edible film packaging had a weight loss of 1.87%. While the lowest weight loss was shown by 0.8% chitosan treatment which was 0.98% on the 22<sup>nd</sup> storage day. This value showed a decrease in weight loss up to 50% lower than the control treatment.

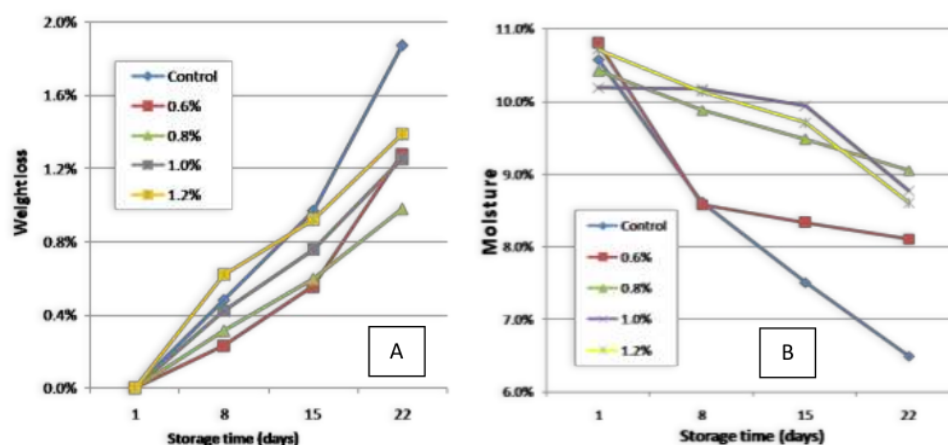


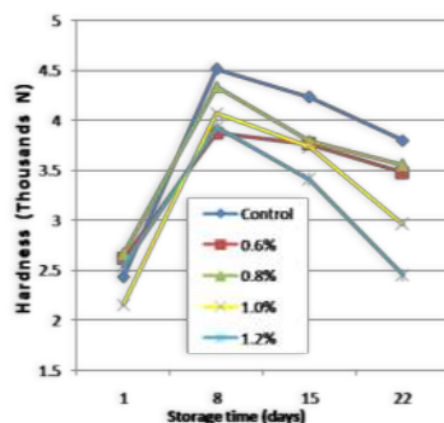
Figure 2. Weight loss [A] and moisture content [B] of milk candy during storage

Initial water content of milk candy ranged from 10 – 11% and decreased during storage. This variation is due to the cooking process that is done manually so that the evaporation process experienced by each treatment is not exactly the same. Control treatment without edible film packaging with an initial moisture content of 10.6% had decreased the water content up to 6.5% (a decrease of 4.1%). Meanwhile, the best treatment was seen in chitosan 0.8% which had an initial water content of 10.4% and the final water content after 22 days of storage was 9.0% (a decrease of 1.4%). The rate of decrease in water content is 66% lower than the control or only one third. The addition of lipid fraction (palm olein) in the film composite increased the hydrophobicity of the film hence improving its barrier properties toward moisture loss [8].

Observation data on weight loss and moisture content were in line with the results of WVTR analysis. The lowest WVTR value, which was at 0.8% treatment, resulted in lowest weight loss and lowest decrease in moisture content as well. This was understandable given the low WVTR resulting in a low rate of evaporation from the product, so that the quality of the product can be maintained longer.

### 3.3. Milk candy hardness during storage

Changes in candy milk hardness during storage can be seen in Figure 3. In general, hardness of the candy milk increased in the first week of storage and decreased later. The increase of initial hardness may be caused by the stabilization of the crystal after the heating and caramelization process. As soon as the cooking/heating process was stopped, the milk candy mixture which was initially elastic and tends to be sticky started to harden. This process was caused by sugar which initially dissolved in milk, crystallizes when the water level decreases to a certain level due to heating. Sugar crystallization was expected to occur for several days until the most stable crystal structure was formed, which resulted in a hardened candy texture. Therefore, the hardness on the second observation day (day 8) was much higher than the first observation on day 1 of storage.



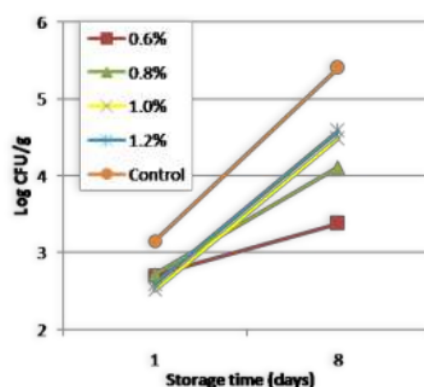
**Figure 3.** Milk candy hardness during storage

After reaching the peak, the hardness value decreased during storage. The decrease was expected to be caused by the decreasing binding force among sugar crystal particles due to the decrease of moisture. Water functions as a sugar bridge which stabilizes the matrix between sugar crystals, so that even though the sugar crystals become more stable, the bonds between the particles become more fragile. From the four treatments using edible film packaging, the highest hardness at the end of the storage period was shown by 0.8% chitosan treatment with the value of 3553 Newton. This can be attributed to the lowest rate of decrease in water content compared to other treatments. However, this hypothesis was not fully supported by the results of observations because the control treatment that experienced the highest decrease in water content turned out to show the lowest hardness decline. The author speculated that this was caused by the formation of crust or hard surface in control treatment due to a decrease in water content that was too fast [12].

### 3.4. Total plate count (TPC)

Microbiological parameter was one of the important parameters in determining the shelf life of food products. Indonesian Standard for hard confectionery (SNI 3547.1: 2008) [13] stated that the TPC maximum limit for the product is  $5 \times 10^2$  CFU/g or equivalent to 2.7 log CFU/g. Figure 4 showed the number of the microbes from milk candy during storage. Observation on day 1 showed that samples wrapped in edible film composite had microbial count at border limit already (above 2 log CFU/g) while the control has already exceeded the standard. At 8<sup>th</sup> storage day, all treatments have exceeded the maximum limit of microbial standard but still better than control. The lowest TPC increase was at 0.6% chitosan. A high initial microbial amount might be caused by the process of milk candy production milk

that was less hygienic. Therefore, improvements are needed so that product quality will be able to meet SNI requirements. Moreover, the expected antimicrobial effect with the presence of chitosan on edible film was not particularly pronounced, most probably due to the small concentration of chitosan and high initial load of microbes. Microbial load in milk candy may be due to handling (manual cutting and wrapping) as well as the contact with air [10].



**Figure 4.** Total plate count of milk candy during storage

#### 4. Conclusions

Edible film packaging has been successfully made from tapioca starch-chitosan-palm olein composite. Tapioca starch-chitosan-palm olein composite edible film can retard the negative quality changes of milk candy during storage. Products that were packed with edible films have a lower rate of weight loss and a lower water content reduction than control products which did not use edible film. The best packaging treatment was at the concentration of 0.8% chitosan which had a weight reduction of 50% lower and the decrease in water content of 66% lower than the control. This was in line with WVTR film analysis which shows the lowest value on the treatment of 0.8% chitosan. On the other hand, edible films were expected to have no direct influence on the texture and TPC of the product. However, changes in texture and TPC were affected by changes in the moisture content of the product. Improvement in the method of making candy milk is needed so that the product will be able to meet the quality requirements according to SNI standards. The benefits of this research were the innovations of primary packaging that were edible and biodegradable, so that they were eco-friendly and can be used as an alternative to extend the shelf life of food products.

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