IOP Conf. Series: Earth and Environmental Science 1038 (2022) 012066 doi:10.1088/1755-1315/1038/1/012066

Characterization of Edible Film based on Yam Bean Starch, Calcium Propionate and Agarwood *Bouya* Essential Oil

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Abstract. Fruit become defenceless against mechanical damage and water loss, causing loss of quality and quantity. To avoid these risks, the edible coating might be one solution. The advantage of starch creates a fence between oxygen, either moisture and the product. The additional antimicrobial can be used in the coatings us calcium propionate in limited quantities. In addition, agarwood *bouya* essential oil might also have potential as an antifungal agent for fruits. This study aimed to evaluate the effect of yam bean starch, calcium propionate, and essential oil concentrations on the relevant properties of edible films. The effect of treatments analyzed by analysis of variance and significant differences between experimental groups were examined by Tukey HSD. As a result, the viscosity and pH of the solution were found to be in the range of 28.34 to 35.97 cP and 4.79 to 4.97, respectively. As for the film properties, moisture content ranged about 32.27 to 49.80 %; *L**, *a**, and *b** values ranged from 90.22 to 91.29, 0.54 to 0.89 and 16.06 to 17.39 respectively; thickness ranged from 0.428 to 0.437 mm; WVTR ranged from 5.63 to 6.83 (g/day(m^2)), and the results of scanning electron microscopy show that it was found that the films still had the granule structure of starch.

1. Introduction

Either quality or quantity losses in postharvest are presently a significant issue in the horticulture industry. Mainly the losses caused by improper handling during the supply chain, the effects of the environment, physiological changes and the respiration from the horticultural products itself. One of the solutions to prevent horticultural products from those risks is applying an edible coating, which might stop microorganism contamination, extend shelf life, reduce water loss, avoid changes of color, and control gas exchange. That means the edible coating can also help relieve quality problems during storage [1].

Natural and safe material for edible coating is based starch. The advantage of the starch is creating a fence between oxygen, either moisture and the product. Starch-based coatings are most plasticized, with the characteristics of being relatively non-sweet, inexpensive, and safe [2]. A product tuber with high starch content is yam bean (*Pachyrhizus erosus*), which has a starch content of about 63.62 % [3,4]. Yam bean starch-based as an edible coating has been rarely in research studies.

Antimicrobial can also be applied to help the coating prevent microorganisms from expanding during storage. An additional antimicrobial that can be used in the coating is calcium propionate with a limited amount. Moreover, agarwood *bouya* essential oil (*Aetoxylon sympetalum*) might have potential as antimicrobial also antifungal on fruits. But despite this, not many studies have discovered combination between yam bean starch, agarwood *bouya* essential oil and calcium propionate as coating solutions and edible films the effect on films characterization. Some studies have used jicama starch as the main material for edible coating and films [5–7]. However, the role of agarwood *bouya* essential oil and calcium propionate as additives on coating solutions and films and heating of the solution at 65 °C had not been characterized yet.

In consequence, an appropriate formulation of edible coatings-based yam bean starch for fruits with calcium propionate and agarwood *bouya* essential oil needs to be discovered and developed to maintain the shelf life and identify the effect on the quality. The aims of this study were to evaluate concentrations of yam bean starch, calcium propionate, and agarwood *bouya* essential oil that might affect the properties of edible coating solutions and films.

2. Material and Method

Yam bean starch (ST) prepared for 1.5 % per treatment (w/w) dissolved the starch in distilled water and stirred with a magnetic stirrer for 60 min at 1,000 rpm. After that, the starch solution was heated at 65 °C and stirred with digital water bath stirrer for 60 min at 750 rpm. Then 0.5 % w/w of sodium alginate was gradually poured. When starch was heating, the solutions of essential oil (ES) was prepared by mixing agarwood *bouya* essential oil 0.5 % (w/v) with calcium propionate (CP) (0.01 % w/w) applied as an antimicrobial agent; tween 80 of 0.5 % (w/w); glycerol 1 % (w/w). Then ES solutions were mixed with ST solutions and homogenized at 15,000 rpm for 5 min using a rotor-stator homogenizer. To produce the films, each coating solution was poured 15 mL into ten plastic plates with a diameter of 85 mm. The films then placed in a program incubator for 12 hours at 25 °C. After the films had completely dried, they were removed from the plastic plate and stored in a desiccator at 25 °C in a stable environment.

To discover the effect of each treatment, a number of objective observations for the solutions and the films include viscosity; pH; thickness; moisture content; color; microstructure; water vapor transmission rate (WVTR), in this study WVTR was using 25 °C as the temperature and 25 % of relative humidity per hour for total 12 hours. Each objective observation was using ten films per treatment as replication. Data of research were evaluated using IBM SPSS Statistics Version 25. Analysis of Variance (ANOVA) was used to examine the effects of treatment, and Tukey Honestly Significant Difference (HSD) tests were used to examine the significant differences between experimental groups. Furthermore, if the means of the data differ by $P < 0.05$, the difference is considered statistically significant.

3. Result and Discussion

3.1. Viscosity and pH

Means from the five of viscosity for each treatment were 28.34 to 35.97 cP, as showed in Table 1. The viscosity mostly decreased in line with calcium propionate added into the solutions. Besides that, on the second treatment, viscosity increased due to addition of the essential oil. The significant differences ($P < 0.05$) in viscosity were found on samples ST; ST-ES and ST-CP; ST-ES-CP.

Table 1. Characteristic of the solutions

Characteristic	ST	ST-ES	ST-CP	ST-ES-CP
Viscosity (cP)	34.94 ± 0.46 [a]	35.97 ± 1.1 [a]	29.30 ± 0.51 [b]	28.34 ± 0.43 [b]
pH	4.79 ± 0.37 [b]	4.81 ± 0.01 [b]	4.97 ± 0.04 [a]	4.96 ± 0.03 [a]

Tukey HSD ($P < 0.05$) indicates that data followed by different letters are statistically different.

Significant differences in pH measurement between samples were also found in the ST; ST-ES and ST-CP; ST-ES-CP. Meanwhile, pH characteristic was found increased along with others material added into starch solutions.

3.2. Thickness

As shown in Table 2, the range of thickness values shown are 0.428 to 0.437 µm. Also, the differences between the values by statistical analysis explained in the Table 2. The thickness of films in this study were not uniformly, and other areas were too thick or too thin.

Table 2. Physical and chemical properties of the films

Characteristic	ST	ST-ES	ST-CP	ST-ES-CP
Thickness (µm)	0.428 ± 0.00[a]	0.431 ± 0.00[a]	0.432 ± 0.01[a]	0.437 ± 0.01[a]
Moisture content (%)	33.40 ± 4.27[b]	49.80 ± 3.18[a]	32.27 ± 2.61[b]	33.03 ± 2.36[b]

Tukey HSD ($P < 0.05$) indicates that data followed by different letters are statistically different.

The difference in edible coating thickness is influenced by the physical properties of the solution (density, viscosity, surface tension, drying time), as well as the coating formation technique [8]. If a thick film is applied to fruits, it can restrict the exchange of respiratory gases, causing the fruits to accumulate a lot of ethanol and increase off flavors [9].

3.3. Moisture Content

The moisture content of films ranged between 32.27 % to 49.80 % shown in Table 2. Films are made from yam bean starch and essential oil has the highest moisture content that was 49.80 %, while those made from yam bean starch, and calcium propionate has the lowest moisture content was 32.27 %.
Moisture content in agricultural products affects their stability, appearance, and texture, as well as controlling microorganism growth. Films with low moisture content that can be used for edible coating will be better able to reduce damage and extend the shelf life of agricultural products.
The moisture content contributed by the components included in each formulation, the relative humidity of the surrounding environment, and the hydrophilic/lipophilic balance of the emulsifier all affect this characteristic, which is directly related to the usable life of the films [10]. In the Table 2 also showed that films with calcium propionate were lower than others, a previous study found that calcium propionate greatly improved the water-absorbing capacity [11].

3.4. Color

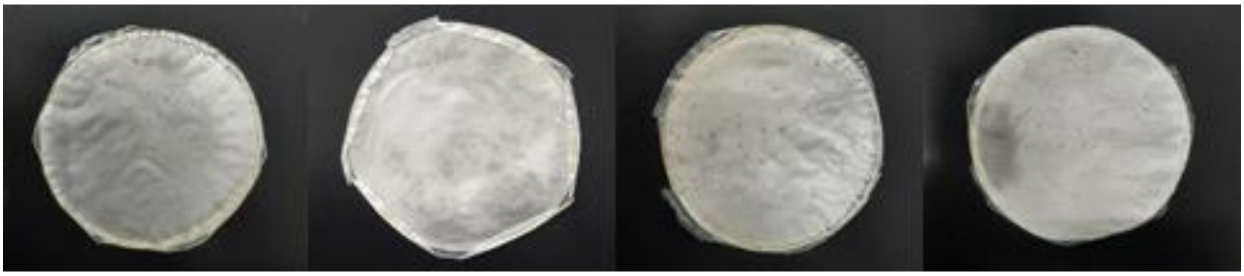
The effect of the added essential oils and calcium propionate on starch to the index L^* (lightness), a^* (redness/greenness) and b^* (yellowness/blueness) of the edible films are tabulate in Table 3.

Table 3. Color of films

Color	ST	ST-ES	ST-CP	ST-ES-CP
L^*	91.24 ± 0.73[ab]	90.22 ± 1.37[c]	91.29 ± 0.85[a]	90.76 ± 0.63[b]
a^*	0.59 ± 0.21[b]	0.54 ± 0.16[b]	0.89 ± 0.20[a]	0.62 ± 0.16[b]
b^*	17.32 ± 1.37[a]	16.06 ± 0.1.09[b]	17.39 ± 1.02[a]	16.78 ± 1.68[a]

Tukey HSD ($P < 0.05$) indicates that data followed by different letters are statistically different.

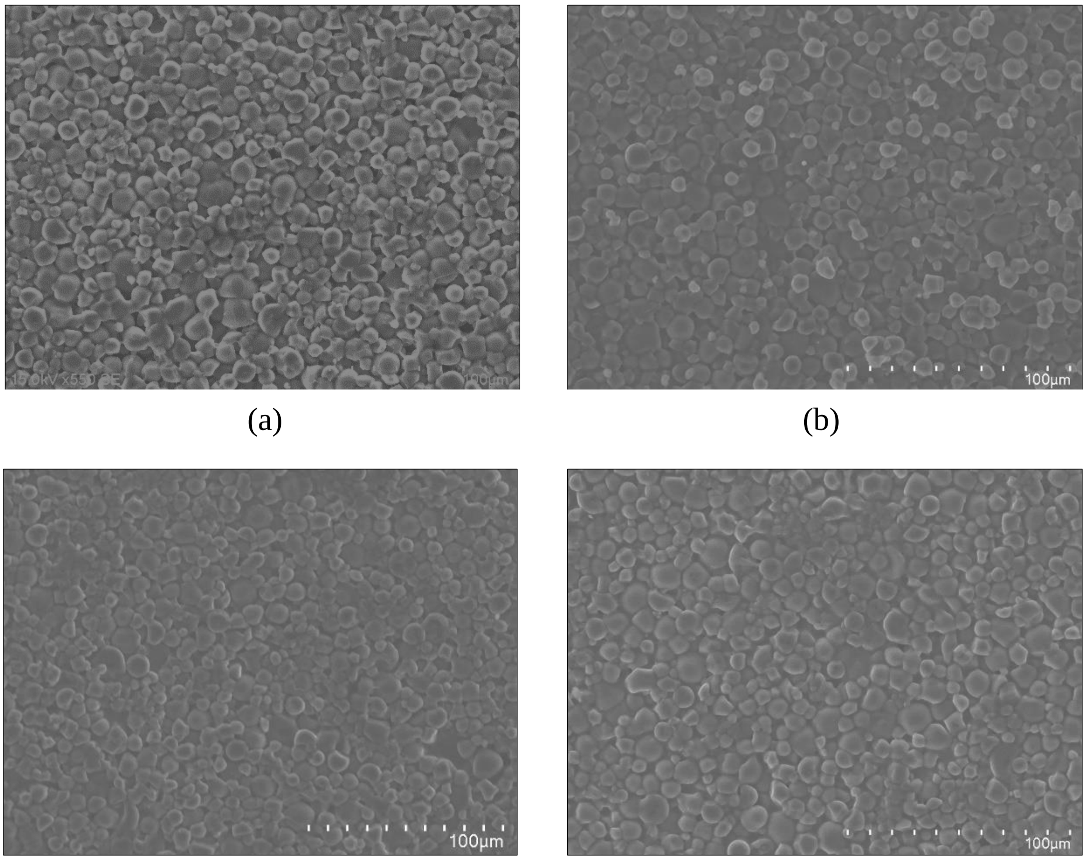
The film made from starch and calcium propionate (ST-CP) showed the highest lightness in all chromaticity indexes. Meanwhile, starch and essential oil (ST-ES) film was the lowest one. The film that without essential oil has higher chromaticity values nevertheless L^*, a^* or b^*. While films that added essential oil has lower chromaticity, although the essential oil was used has a strong yellow appearance. These are inverse with some studies that have been reported to affect color, especially on yellowness [12]. The films' appearance in this study showed in Figure 1.



(a) (b) (c) (d)

Figure 1. The films' appearance for each treatment respectively (a) ST, (b) ST-ES, (c) ST-CP, (d) ST-ES-CP

Scanning electron microscopy for each film surface showed in Figure 2. All of the results clearly showed that films still have granule form. This might cause by the improper temperature that used for the heating of the solution. Despite the others component (i.e., emulsifier, plasticizer) might also influence the thermal (gelatinization) and post-thermal (retrogradation) on starch-based edible coating solutions, this occasion has been thriftily studied.



(a) (b)

(c) (d)

Figure 2. SEM images of films (a) ST; (b) ST-ES; (c) ST-CP; (d) ST-ES-CP were taken at a voltage of 15.0 kV and magnifications of 100×

Gelatinization occurs when starch granules undergo a shift from a semicrystalline to an amorphous state when exposed to high temperatures and abundant water. The loss of crystallinity takes place in two stages, the first of which involves the expansion of the starch molecule at 60–70 °C [13]. During this process, birefringence is lost, indicating that crystallite dissociation has begun without a considerable rise in viscosity [14]. Above 90 °C, the second stage occurs, resulting in severe granule swelling and solubilization, as well as full structural integrity loss [15]. This study used 65°C, this decision was based on a heating temperature recommendation to reach the gelatinization stage for yam ben starch that should be between the range 53-63 °C [3]. Using 65 °C involved microstructure of the films was insoluble, and structure of starch was still rigid.

The amylose-amylopectin ratio, water content, and dispersion temperature all influence the changeover process. The chance of starch recrystallization during retrogradation is determined by a combination of these factors [16,17]. When all feasible chain interactions are performed, the mass of the gel is reduced by continuing water evaporation until most of the free water is eliminated. When the amylose content is higher, the gelatinization temperature is essentially linear with the amylose concentration. This case is due to the linearity of the amylose molecule and the fact that it has more extensive hydrogen bonding, it takes more energy to break these bonds and gelatinize the starch [18].

3.5. Water Vapor Transmission Rate (WVTR)

The water vapor transmission rate (WVTR) is the rate of water vapor permeating through the film. The range of the WVTR were between 5.639 to 6.838 (g/day)/m^2 (Table 4). The highest rate was on film made from starch and calcium propionate (ST-CP), and then the lowest was the film made from starch, essential oil and calcium propionate (ST-ES-CP).

Table 4. WVTR and water vapor permeability of the films

Film	WVTR ((g/day)/m^2)	WVP (g.mm/ (m^2.day.mmHg))
ST	6.018 ± 0.21^b	1.590 ± 0.05^b
ST-ES	5.907 ± 0.19^b	1.338 ± 0.04^c
ST-CP	6.838 ± 0.52^a	1.806 ± 0.13^a
ST-ES-CP	5.639 ± 0.14^b	1.489 ± 0.03^b

Tukey HSD ($P < 0.05$) indicates that data followed by different letters are statistically different.

The effect of water activity on the surroundings on both sides of the film influences the vapor pressure difference across the film, resulting in the mass transfer of water vapor from the high vapor pressure to the low vapor pressure surroundings. Because a rise in temperature causes an increase in the WVTR, the water vapor permeability also increases. In terms of the barrier, when the water molecule content rises, the water vapor barrier qualities decrease [19]. The thickness of the films can also be one of the factors that affect the WVTR values.

4. Conclusions

In this part of study, edible films based on yam bean starch, agarwood *bouya* essential oil and calcium propionate were successfully created. This study was not specifically determined which film has the best result. However, one of the most important parameters that should be considered in an edible film is the WVTR. If there is an increasing in WVTR then water vapor and gas are also easier to absorb into the product resulting in weight loss, flavor loss. The best result of WVTR were films that has the lowest level of water vapor permeability, then in this study the lowest WVTR was films that based on starch and essential oil (ST-ES). Viscosity of ST-ES films was the highest at 35.95 cP, while pH placed as low acidity. For the thickness of ST-ES film was 0.431 as the second lowest, has the highest moisture content, and has the lowest color indexes. In this study since the coating solution has not yet been applied, the recommendation of future work is direct application on fruits and vegetables is required in order to ensure the effectiveness of the films. Moreover, the heating temperature of solutions (65 °C) was found ineffective to produce appropriate solutions, for future study using a higher temperature is highly recommended.

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