Development and Characterization of Biodegradable Film Incorporated with Turmeric Oil

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Abstract — The study and experiment was conducted to evaluate the effect of different turmeric oil's volume used (20µl, 40µl, 60µl, 80µl and 100µl) and different temperature set during film forming solution (70°C, 75°C, 80°C, 85°C and 90°C) on film's solubility and their mechanical properties. This study also been performed to develop biodegradable film from natural resources such as gelatin and starch and incorporated them with essential turmeric oil. The materials and method that have been used in this study were gelatin from white mouse, tapioca starch, pure glycerol and film forming solution methodology. Analysis that have been done were solubility test and mechanical properties test. Basically for film forming solution, the ratio of gelatin to starch that have been used was 2:3. Based on the solubility test we can concluded that the larger the turmeric oil's volume been used in film preparation process, the larger values of solubility percentage or film's weight loss. Same trend can be seen if we increase the temperature during film preparation of film forming solution processes. Different batch was prepared under different temperatures and based on the result we can concluded that, higher temperature resulted in increases in film's solubility.

Keywords— Biodegradable, Different temperature, Different essential oil concentration, film, film properties.

I. INTRODUCTION

There are many problems rised when using chemicals materials as raw materials in order to develop synthetic biofilm product. Based on result that have been discovered by scientist, synthetic biofilm can severely affect on our health, maybe not in first or second years but it will for another six to five years if we continue to consume them in a long term. Not only it will negatively impact our health but it also gives an impact toward environment and accelerate the depletion of our non-sustainable resources. Hence, with concern about our limited resources, environmental impact and danger towards consumer health caused by usage of synthetic polymer there is a great interest in developing biodegradable film from natural resources.

Biodegradable films from natural resources represent an interesting alternative to conventional plastic materials, which is

why several natural resources have been used to develop ecofriendly biodegradable film. The most common materials for the formulation of edible and biodegradable films are polysaccharides, proteins and lipids, and the combination of these allows for producing blends of improved characteristics (Fabra, Jimenez, Atares, Talens, & Chiralt, 2009). However, for further study of biodegradable film characteristic, incorporation of turmeric essential oil has been proposed. Thus new method needed to be develop using natural resources as our main materials instead of chemical non-sustainable resources with addition of turmeric essential oil.

Turmeric essential oil extracted from turmeric plant exhibit antimicrobial and antioxidant properties (Viuda-Martoset al., 2010) and it also can affect other properties of biodegradable film which makes them interesting additives in food industry. However, their usage in food preservatives is often limited mainly due to their strong flavor. In order to avoid this problem, turmeric oil can be incorporated into edible films (Ruiz-Navajas et al., 2013). Thus, effect of different turmeric oil used in development of biodegradable film on film's characteristic need to be study. This to further expand our understanding in effect of turmeric oil on characterization of biodegradable film.

Studies performed by Stutchell and Krotcha, (1994) on effect of different temperature set during gelatinization process towards film's characteristic proved that temperature can affect the film's characteristic, thus further study in temperature effect is required to discover the optimum temperature setup in order to develop optimum biodegradable film.

II. METHODOLOGY

2.1 Film Forming Solution Preparation

Materials that required in preparation of biodegradable film incorporated with essential oil were gelatin (provided by Merck KGaA), tapioca starch (purchased from Giant Hypermarket), glycerol (provided by Merck KGaA), distilled water and turmeric oil (purchased from Bioessential Oil Corporation). To be specific, gelatin that has been use was gelatin from white mouse, starch from tapioca plant, and turmeric oil extracted from turmeric plant. Ratio of gelatin to starch was set at 2:3, basically amount of gelatin in one batch was 10g and for starch was 15g. Meanwhile for the amount of turmeric oil's volume used were varied (20µl, 40µl, 60µl, 80µl and 100µl) because this is our manipulate variable and glycerol required for each film was constant, 4ml each. 500 ml of distilled water was poured into the 600 ml beaker, then to increase the distilled water temperature the beaker was placed on hotplate (Corning PC-420D). The hotplate's temperature was set at 140°C, although the hotplate was set at 140°C the temperature of distilled water only rised till 70°C. This due to heat transfer coefficient of beaker, caused loss in heat. Then, distilled water was stirred using magnetic stirrer. The distilled water was stirred and heated up till reach 70°C. Thermometer has been used in order to detect the temperature rised in distilled water. For detection of temperature change, thermometer was set deep around half of the beaker. Gelatin and starch were measured while waiting for the temperature to rise. 10g of gelatin and 15g of starch needed to prepare one batch of film (5 films). The materials were measured and put into one container to be mixed. Spoon was used to mixed the gelatin and starch.

After distilled water reached 70°C, in order to make sure it stay at 70°C and there is no temperature fluctuation additional 2 minutes waiting time was considered. Mixture of gelatin (10g) and starch (15g) was poured into 70°C distilled water. Magnetic stirrer of hotplate was turn on and set to stirred at 700 rpm. Then, for plasricizer effect 4 ml of glycerol was poured into the mixture. The mixture was left stirred at 70°C for 30 minutes.

After 30 minutes passed, the hotplate was turned off and 5 smaller beaker with volume of 75 ml each were prepared. 500 ml of film forming solution was poured into these 5 small beakers with volume of 75 ml each. Then before proceed to addition of turmeric oil, the solution was let to cool till reached room temperature. Then, turmeric oil was added. There were 5 small beakers containing 75ml of film forming solution each, thus different volume of turmeric oil was added into each beaker.

The first beaker was poured with 20μ l of turmeric oil, second beaker with 40μ l, third beaker with 60μ l, fourth beaker with 80μ l and last beaker with 100μ l. Pipette was used to measured the amount of turmeric oil volume. Then using rod glasses, the mixture was stirred for 5 minutes. 5 petri dishes were prepared, then the mixture was poured into each petri dish. First petri dish for first solution with volume turmeric oil of 20μ l, second petri dish for solution with turmeric oil's volume of 40μ l and so on. Then all these petri dishes containing film forming solution with different turmeric oil volume were placed inside 35° C oven for 24 hours.

The petri dishes were took out and film is ready to be peeled after 24 hours inside the oven. For testing process the films were peeled. Using differet temperature set, these steps were repeated with different temperature set. First temperature set was 70°C, then the processes were repeated for 75°C, 80°C, 85°C and 90°C.

2.2 Determination of Film Properties

2.2.1 Film Thickness

Film thickness (mm) was measured using micrometer with precicision of 0.001 mm. Three random location were measured to get the average measurement of film's thickness.

2.2.2 Mechanical Properties

Mechanical properties was measured using Universal Testing Machine (LLOYD instrument, Hampshire, England). This method was adopted from Phakawat Tongnuanchan, Soottawat Benjakul and Thummanoon Prodpran. (2014). Before the testing, films were cut with length of 60mm and width of 20 mm. Three samples with same length and width were obtained from one film. The film was cut using scissor. These three films were tested to get the average value of mechanical properties. There were three mechanical properties that were considered and they are , tensile strength, Young Modulus and elongation at break. Load that have been choosen was 25 N. Cut film (60mm x 20mm) was placed between the UTM grip for mechanical properties analysis. Distance between upper grip and lower grip was set at 2 cm and speed of the holders movement was set at 20 mm/min. This setup was constant for all batches. The gripper pull the film untill it break, then it showed the mechanical properties result. Tensile strength was calculated using equation below:

Tensile Strength
$$\left(\frac{N}{m^2}\right) = \frac{Maximum \text{ Load to Break (N)}}{Cross Sectional Area of The Film (m2)}$$

Elongation at break was calculated by using equation below:

Elongation at Break (mm) = $\frac{\text{Film Elongation Till Break}}{\text{Initial Gauge Length}} \times 100$

Young modulus was calculated using equation below:

Young Modulus =
$$\frac{\text{Tensile Strength}}{\text{Strain}}$$

Strain = $\frac{\text{Elongation}}{\text{Original Film Length}}$

2.2.3 Film Solubility

First step in determination of film solubility was to cut the small portion of film. This method was adopted from S Yanwong and P Threepopnatkul., (2015). Then the films were placed in the glass petri dishes, different film inside different glass petri dishes. This to separate the film based on turmeric oil concentration and temperature set during film forming solution processes. The petri dishes then were placed inside 100°C oven and left for 24 hours. After 24 hours, the films were peeled and weighted using weight analyzer. Initial mass were recorded.

Then, centrifuge test tube filled with 50 ml of distilled water were prepared. One test tube for one film only, thus there are 25 samples and 25 test tube. After completed, all 25 test tubes containing films were placed inside the centrifugal machine and left for another 24 hours. The centrifugation system was set at 500 rpm.

After 24 hours the films been centrifugated, the films were removed and filter using filter paper. These films were placed back inside glass petri dishes and once again placed inside the 100°C oven for drying process. The films were left inside the oven for another 24 hours. Then, the films were measured using weight analyzer for second time to determine their final weight. By obtaining the final weight, film's solubility was calculated using equation below:

 $Solubility = \frac{Initial mass of film - Final mass of the film}{Initial mass of the film}$

The result was tabulated in table 1.

III. RESULTS AND DISCUSSION

3.1Film Solubility

Effect of diferent turmeric oil volume used with different temperature on water solubility is shown in table 1 and figure 1. There were two manipulate variables, first was volume of turmeric oil used during film forming solution process and the second one is temperature set also during the film forming solution processes. Different volume of turmeric oil were used starting with 20µl, 40µl, 60µl, 80µl and 100µl. Meanwhile, for second manipulate variable the starting temperature that has been set was 70°C, then proceeded with 75°C, 80°C, 85°C and 90°C. First, we wil discuss about the effect of different turmeric oil volume used on film's solubility. Based on the results, it clearly shown that relationship between turmeric oil's volume used with film's solubility is directly proportional. Basically the larger the volume of turmeric oil used during the film forming solution process resulted in higher film's solubility. Examples of them are at 80°C batch where 20µ1 resulted with solubility of 55.56%, then at 40µl resulted in 57.14% of solubility. The solubility keep increasing when volume of turmeric oil being use incease. Based on film solubility calculation, the higher the percantage value of solubility mean more loss in term of film's mass. Hence, it can be concluded that turmeric oil will increase the solubility and weight loss of the film. Main reason this happened because presence of turmeric oil cause reduction in interaction between gelatin chains, which lead to the weakening of the interaction between gelatin films and allowed increasing of its leeching out (S.Yanwong and P Threenotpapkul., 2015). Second manipulate variable is temperature set, different temperature was set for different batch. Based on result that have been produced we can clearly see that relationship between temperature set during film forming solution with film's solubility is also directly proportional. Meaning, the higher the temperature set during the film forming solution process the higher the film's solubility. This indicated that rise in temperature also cause increase in film's mass loss. Reason for this is increase in heat treatment during film forming solution process caused increased in exposure of monomer and peptides of low molecular weight and non-protein materials, leading to increases in solubility of these films (Stutchell and Krotcha, 1994).



Fig 1: Effect of different volume of turmeric oil and temperature on film's solubility

| Volume of Turmeric Oil | Solubility | | | | | |
|------------------------|---------------------|----------|------------|--|--|--|
| (µl) | Initial | Final | Solubility | | | |
| | mass (g) | mass (g) | (%) | | | |
| At | At temperature 70°C | | | | | |
| 20 | 0.074 | 0.028 | 62.16 | | | |
| 40 | 0.029 | 0.011 | 62.07 | | | |
| 60 | 0.033 | 0.011 | 66.67 | | | |
| 80 | 0.032 | 0.010 | 68.75 | | | |
| 100 | 0.042 | 0.013 | 69.05 | | | |
| At temperature 75°C | | | | | | |
| 20 | 0.034 | 0.014 | 58.82 | | | |
| 40 | 0.048 | 0.019 | 60.42 | | | |
| 60 | 0.024 | 0.009 | 62.5 | | | |
| 80 | 0.068 | 0.025 | 63.24 | | | |
| 100 | 0.053 | 0.018 | 66.04 | | | |
| At | At temperature 80°C | | | | | |
| 20 | 0.027 | 0.012 | 55.56 | | | |
| 40 | 0.056 | 0.024 | 57.14 | | | |
| 60 | 0.056 | 0.023 | 58.93 | | | |
| 80 | 0.033 | 0.013 | 60.61 | | | |
| 100 | 0.036 | 0.014 | 61.11 | | | |
| At | At temperature 85°C | | | | | |
| 20 | 0.032 | 0.015 | 53.13 | | | |
| 40 | 0.028 | 0.013 | 53.57 | | | |
| 60 | 0.168 | 0.076 | 54.76 | | | |
| 80 | 0.054 | 0.023 | 57.41 | | | |
| 100 | 0.043 | 0.018 | 58.14 | | | |
| At temperature 90°C | | | | | | |
| 20 | 0.040 | 0.020 | 50.00 | | | |
| 40 | 0.083 | 0.038 | 54.22 | | | |
| 60 | 0.025 | 0.011 | 56.00 | | | |
| 80 | 0.043 | 0.018 | 58.14 | | | |
| 100 | 0.037 | 0.015 | 59.46 | | | |

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Table 1: Effect of different volume of turmeric oil and temperature on film's solubility

3.2 Mechanical Properties

3.2.1 Tensile strength

Effect of different turmeric oil volume used and gelatinazation temperature set during film preparation on mechanical properties was studied. Result can be seen in table 2 and figure 2.1, 2.2 and 2.3. First, effect of different temperature and turmeric oil volume used on tensile strength was discussed. As we can see from figure 2.1 it clearly showing that batch with temperature set of 85°C during film preparation process possesed highest average tensile strength range from 25000 to 35000 N/m². We can also see that thermal gelatinazation temperature lead to increases in tensile strength, however above 85°C the decreases in tensile properties has been recorded. This due to protein denaturation took place and consequently bury free –SH groups in the hydrophobic pockets such that they remain unavailable for disulfide bond formation (Gracial and Sorbal., 2005). Thus, this give negative effect towards film bond and automatically resulted in decrease of tensile strength.

Then, from figure 2.1 it indicated that volume of turmeric oil used clearly affect the tensile strength. Peak of tensile strength for each batch can be seen range from 40 μ l to 60 μ l hence we can conclude that optimal turmeric oil volume range between these two values. However after 60 μ l, te decreases trend was observed. It possibly due to interaction between gelatin chains occurred, thus diminishing the tensile strength of the films (S Yanwong and P Threepopnatkul., 2015). Benefits of good tensile strength in film's characteristic is it can prolong the shelf life of the film and make it become more durable.



Fig 2.1: : Effect of different volume of turmeric oil and temperature on film's tensile strength

3.2.2 Elongation at Break of the film

Next, an ability of film to elongate before brake was tested. Higher elongation at break is better, because with this characteristic the film is more durable compared to commercialize one. The trend of this ability was it is directly proportional to turmeric oil volume used. Basically more turmeric that been used during the film preparation the higher the ability to elongate (Atarés, L., & Chiralt, A. (2016). This probably due to plasticizing effect, where turmeric oil enhance the ability to elongate. However, different batch show different ability to elongate this probably due to different temperature setup. For an example, we can see that film with temperature setup of 80°C immadietely decrease it ability to elongate when more concentrated the turmeric oil been used, the turmeric oils can led to more interaction reduction between gelatin chains and caused easy rupture of the film. Moreover, the presence of excess essential oils in gelatin matrix could interfere in the interactions between polymer chains and reduce the flexibility of the gelatin matrix (S Yanwong and P Threepopnatkul., 2015).



Fig 2.2: Effect of different volume of turmeric oil and temperature on film's ability to elongate before break

Effect of different temperature setup on film's ability to elongate also has been studied. However there were no trend was observed.

3.3.3 Young Modulus

Last but not least, young modulus properties also has been studied. Young modulus is a measure of the ability of a material to withstand changes in length when under lengthwise tension or compression. Basically it is an ability of film to maintain it original shape under any external forces. The result show that the trend of increasing in young modulus properties for all batches is when range of $20\mu l$ to $40\mu l$ was used. This probably due to presence of turmeric oil cause significant increase in young modulus properties.

This mainly due to polymer chains could be able to move, making the film flexible according to (Zinoviadou et al). These could be explained by the presence of β -component in turmeric oils. Such component could interact with proteins which promoted their cross-linking in gelatin film, resulting in the rigidity behavior in gelatin film. This led to an increase in young modulus properties of the films.

However, after 40μ l the value start to flactuate due to possible error in handling the preparation process. No clear trend can be seen.

Effect of temperature setup on young modulus properties of the film also has been studied. Based on the trend, it can be concluded that the relation between young modulus properties with temperature setup is directly proportional. The higher the temperature setup result in higher young modulus average value. This probably due to increases in temperature resulted in formation of film network with high rigidity (Meritaine da Rocha a, Márcia Regina Loiko b, Gabrielle Victória Gautério a, Eduardo César Tondo b, and Carlos Prentice., 2013)



Fig 2.3: Effect of different volume of turmeric oil and temperature on film's Young Modulus.

| Volume of | Menchanical Properties | | | | | |
|----------------------|--------------------------------|------------|--------------------------------|--|--|--|
| Turmeric Oil (µl) | Tensile | Elongation | Young | | | |
| (μι) | Strength | at break | Modulus | | | |
| | (N/ ^{m²}) | (mm) | (N/ ^{m²}) | | | |
| At temperature 70°C | | | | | | |
| 20 | 3895.833 | 7.34 | 31846.04905 | | | |
| 40 | 8812.5 | 9.4 | 56250 | | | |
| 60 | 13541.67 | 17.58 | 46217.29238 | | | |
| 80 | 4375 | 10.59 | 24787.53541 | | | |
| 100 | 17229.17 | 13.08 | 79032.87462 | | | |
| | At temperature 75°C | | | | | |
| 20 | 12291.66667 | 19.58 | 37665.9857 | | | |
| 40 | 14937.5 | 16.78 | 53411.79976 | | | |
| 60 | 10687.5 | 15.48 | 41424.4186 | | | |
| 80 | 8520.833333 | 14.68 | 34826.29428 | | | |

| 100 | 13812.5 | 19.78 | 41898.3822 | | |
|---------------------|-------------|--------|-------------|--|--|
| At temperature 80°C | | | | | |
| 20 | 8395.833333 | 9.9625 | 50564.61731 | | |
| 40 | 20562.5 | 9.66 | 127717.3913 | | |
| 60 | 14437.5 | 8.46 | 102393.617 | | |
| 80 | 16395.83333 | 8.15 | 120705.5215 | | |
| 100 | 10895.83333 | 7.688 | 85035.11967 | | |
| At temperature 85°C | | | | | |
| 20 | 26333.33333 | 12.285 | 128612.1286 | | |
| 40 | 33458.33333 | 19.78 | 101491.4055 | | |
| 60 | 31866.66667 | 18.58 | 102906.3509 | | |
| 80 | 31083.33333 | 18.22 | 102360.0439 | | |
| 100 | 31675 | 18.55 | 102452.8302 | | |
| At temperature 90°C | | | | | |
| 20 | 11104.16667 | 12.585 | 52940.00795 | | |
| 40 | 20208.33333 | 15.58 | 77824.1335 | | |
| 60 | 27633.33333 | 11.79 | 140627.6506 | | |
| 80 | 18062.5 | 7.88 | 137531.7259 | | |
| 100 | 16041.66667 | 6.135 | 156886.7156 | | |

Table 2: Effect of different volume of turmeric oil and temperature on film's mechanical properties

IV. CONCLUSION

As the conclusion different usage of turmeric oil volume and temperature during the development does affect the film's characteristic. Based on this study, it has been proven that with increasing usage of turmeric volume can increase the film's solubility. Same trend can be seen with increasing in temperature set during the film preparation processes.

Not only solubility of the film, but mechanical properties of the film also been affected. When increases in temperature been used, mechanical show significant increases untill it optimum point. After this values, it started to deteroriated probably due to denaturation. Similar trend can be seen when increases in turmeric oil volume. The mechanical properties show increases then started to decrease after certain value probably due to bond disruption. However, the result show fluctuation and this due to errors occurred. Hence, for future research project it is highly recommend to use wider range of temperature and tumeric oil concetration. Its also recommend to study different effect that can be brought upon if different essential oil been used, with concentration and temperature set as their manipulative variable.

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