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Preparation and Characterization of Purple Sweet Potato Starch-Based Edible Film with Optimized Mixing Temperature

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Abstract – *In recent years the various types of edible starch films have been gaining commercial value. Purple sweet potato (PSP) contains more than 15 % starches in its root and displayed good potential to become the main raw material in producing edible film due to its functional antioxidant properties. By adding glycerol as plasticizer and kappa carrageenan as gelling agent to the starch helped strengthened and improved the formulation of the edible film. Film properties were found to be dependent on the mixing temperature processing conditions. Attributes like low water vapour permeability (WVP) of the film were obtained with a lower mixing temperature condition while the highest antioxidant capacity (75.84 %) was exhibited at 70 °C mixing temperature. In addition, scanning electron microscopy revealed the highest temperature (80 °C) used in the mixing process resulted in a film with smaller porosity compared to that produced with the lowest temperature (50 °C). Nevertheless, the optimum behaviour of PSP film was obtained by using defined formulations at mixing temperature of 70 °C to obtain high antioxidant capacity and low WVP (0.0214%).*

Keywords: Edible film, Mixing temperature, Purple sweet potato film

1.0 INTRODUCTION

Unlike ancient days when starch was mainly used as a basic food ingredients, food thickener, and food additives, nowadays starch is also used as food supplement and as edible food packaging. Various types of food packaging have included starch as their main ingredient such as medicine capsules, candy wrappers and film packaging. Film packaging made from starch has very good potential in the food industries as it retains its biodegradability and promotes eco-friendly packaging. Besides, starch, which consists of amylose and amylopectin elements, has swelling, water solubility and water binding capacities [1]. The formation of film can be improved after combining starch with water in a heat treatment, together with addition of some plasticizer and thickener-producing a stronger film.

Purple sweet potatoes (PSP), which are mainly harvested in tropical areas, possess high amounts of starch that can be used in producing starch film. Several researchers have studied the high anthocyanin content in the bright purple-coloured flesh of PSP which is believed to have antioxidant capacity [2][3][4]. However, a previous study has shown that the properties of the PSP tuber promotes easy oxidation and it tends to become unstable in humid conditions [5]. However, PSP film can be produced by using PSP starch added with plasticizer and gel

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strengthening element in heat treatment condition. Glycerol has been commonly used as a plasticizer to produce edible film since it is food grade, edible, safe for consumption and comes from natural sources [6]. Hence, addition of glycerol helps to plasticize together the starchy solutions into a film with plasticity effect. Meanwhile kappa carrageenan is another candidate for plasticizers since it has gel strengthening capacity with good film forming ability that helps to form a rigid film [7].

This study aims to prepare an optimum processing condition for producing PSP film, as well as determine the properties of the PSP film produced. The approach of this study involves the determination of the optimum mixing temperatures in producing PSP film and to evaluate the characteristics of the optimum PSP film.

2.0 MATERIALS AND METHODS

2.1 Film Formulation

The PSP film formulation was determined by adding PSP starch (3.14 g), glycerol (3.02 ml) and kappa carrageenan (1.84 g) in 100 ml solution, as suggested by Abdou and Sorour, (2014) [8]. The purple sweet potato (Chiran Murasaki) was purchased from a local store in Taman Universiti, Johor. Meanwhile, food-grade glycerol was purchased from Merck KGaA, Darnstadt, Germany, whereas Kappa-Carrageenan was purchased from a Malaysian agent for Sigma-Aldrich, Saint Louis, USA.

The mixed solution was heated on a hot plate (IKA C-MAG HS 7, China) until the solution gelatinized and began to solidify. An amount of 15 ml solution was then poured and casted on a 9 cm diameter petri dish.

2.2 Design of Experiment using Central Composite Design

The preliminary test showed that the heating temperature of the PSP solution, also influenced the characteristics of the PSP film. Therefore, various heat temperatures were used to determine the optimum mixing temperature based on the characteristic of the PSP film produced. The experiments were designed according to the 2-factor level central composite design (CCD) with a 10 factorial, as well as 2 central points, using Design Expert version 6.04. The independent variables, namely the; mixing temperature from 50 to 80 °C and mixing speed 2.00 to 4.00 Mot; where 1 Mot (stirring quantity maximum per stirring position) = 250 rpm (revolutions per minute) with respective ranges were used. The mixtures were constantly heated in a 500 ml beaker for 15 minutes. The optimization was carried out to determine the most suitable processing conditions that shows the best characteristics and properties in water vapour permeability, antioxidant activity and surface morphology.

2.3 Water Vapour Permeability of PSP Film

Films were prepared and sealed over the circular opening of 8.042 x 10^{-2} m^2 of a glass jar. Calcium anhydride ($CaCl_2$ – 0 % RH) was filled in the glass jar and the jar was placed in a 25 °C desiccator containing sodium chloride solution (NaCl – 75 % RH), as depicted by Ghasemlou et al. [9]. Seven weight measurements were made at 24 hour intervals over 7 days. The changes found in the weight of the jar were measured to the nearest 0.0001 g by using Analytical Semi-Micro Balance (GR-200 Series, Tokyo) and were recorded and plotted as a function of time. The slope of each line was calculated by linear regression (IBM SPSS Statistic Ver.22), while the water vapour transmission rate (ΔWt) was calculated from the slope (g

H_2O/hr) divided by the opening area (m^2) of the jar. WVP value expressed as [g mm m^{-2} hr^{-1} Pa^{-1}], based on Equation 2.1 below:

$$WVP = \Delta Wt \times \frac{t}{A \times \Delta p} \quad (2.1)$$

where ΔWt is the weight gain of jar per day (slope, g/hr); t is the film thickness (mm); A is the permeation area (opening area, m^2); Δp is the water vapour pressure difference (Pa) across the film where the pressure remained at 1.333 x 10^2 Pa.

2.4 Antioxidant Capacity of PSP Film

The antioxidant capacity was determined based on the measurement of 2,2-diphenyl-1-picrylydrazyl (DPPH) in Equation 2.2,which reflected a radical scavenging activity with a modified method from Balakrishnan et al., [10]. DPPH (0.02 mM) solution was prepared by adding 3.94 mg of DPPH and it was marked up to 100 ml with absolute ethanol solution. The sample was diluted in absolute ethanol at concentration of 1 mg/ml. After that, 3 ml of the extracts was pipetted into a test tube together with 1 ml of 2,2-diphenyl-1-picrylydrazyl (DPPH) ethanol solution (0.02 mM). The sample was then left to stand for 30 minutes in a dark space to initiate reaction followed by measurement of the absorbance at 517 nm using spectrometer (Jenway 6305 UV-Vis, UK). In addition, Sanna et al., [11] stated that the calculation for antioxidant content by using UV-Vis measurements can be done as depicted in the Equation 2.2:

$$\text{DPPH radical scavenging activity: } [(A_0 - A_s) / (A_0 - A_i)] \times 100 \quad (2.2)$$

where A_0 is the absorbance at 517 nm of the DPPH solution without antioxidant source, A_s is the absorbance of the sample with DPPH solution while A_i is the absorbance of the solution when 100 % of DPPH was reduced. The test was conducted in triplicate for each batch. The unit of DPPH radical scavenging activity was stated as percentage of antioxidant capacity.

2.5 Surface Morphology Studies

The study was carried out on samples by using a Scanning Electron Microscope (Hitachi TM3000, Illinois) [12]. The sample was made to stand on double-sided adhesive tapes mounted on the studs. The studs were then placed on sample holder of SEM and after the studs were ready, they were placed on a sample holder of the Leica SEM to be scanned. The image was captured using 10 kV at 3000 magnifications and the images obtained were recorded.

3.0 RESULTS AND DISCUSSION

3.1 Film Evaluation

RSM was used to reduce the number of experimental trials required to a minimum by means of statistical predictions, which was also used to study the interaction effects between the multiple single factors [17]. The processing condition to produce PSP film was determined by the desired characteristics and the properties of the film. Table 1 shows the results obtained from the 10 experiments conducted by using response surface regression analysis of the processing conditions to produce PSP film; mixing temperature (°C) and mixing speed (Mot) that resulted in 3 responses; water vapour permeability of the film and antioxidant capacity. The lowest water vapour permeability [18] and the highest antioxidant capacity [19] were

identified as the most desirable factors in selecting the optimum processing condition to produce PSP film.

Table 1 The Mixing Temperature and The Mixing Speed Processing Condition of PSP film responses to water vapour permeability (WVP) and antioxidant capacity.

Run	Mixing temperature (°C)	Mixing speed (1 Mot = 250 rpm)	WVP , X_1 (g mm m^{-2} hr^{-1} Pa^{-1})	Antioxidant capacity, X_2 (%)
1	80.0 (+1)	2.0 (-1)	0.02245	74.51
2	43.8 (-1.68)	3.0 (0)	0.02732	75.00
3	50.0 (-1)	2.0 (-1)	0.02524	76.60
4	80.0 (+1)	4.0 (+1)	0.02180	74.83
5	50.0 (-1)	4.0 (+1)	0.02648	76.51
6	65.0 (0)	4.4 (+1.68)	0.02373	76.14
7	86.2 (+1.68)	3.0 (0)	0.02342	74.02
8	65.0 (0)	1.6 (-1.68)	0.02483	76.60
9[c]	65.0 (0)	3.0 (0)	0.02367	76.51
10[c]	65.0 (0)	3.0 (0)	0.02389	76.44

[c] Central point

A sequential sum of squares and model summary statistics tests were carried out on the experimental data to decide on the adequacy of the quadratic models and the results are listed in Table 2. The value in Table 2 indicate a high level of conformance with quadratic polynomial estimates (P value < 0.05 and $R^2 = 0.85$), where water vapour permeability (X_1) and antioxidant capacity (X_2) have significant differences (P value < 0.05), which meant that in a batch experiment, these three factors played an important role in affecting the PSP film.

Table 2 Analysis of variance for the application of both processing condition to produce PSP film.

Factor	DF	Squares	Mean square	F-value	P-value[*]	Regression, R^2
X_1	5	2.39×10^{-5}	4.78×10^{-6}	7.76	0.0347	0.9066
X_2	5	8.29	1.66	7.63	0.0443	0.9051

[*] $p < 0.05$

A surface response of the quadratic polynomial model was also generated by varying 2 of the independent variables within the experimental range. Therefore, Fig. 1 are the response surface plots that portray the effects on PSP films produced using processing conditions.

Based on the 3D surface and the trace interaction response model given by the software on the water vapour permeability response different mixing temperatures have been found to influence the results of water vapour permeability (WVP), which displayed a decreasing trend. The WVP given by all the runs is between (0.0218 - 0.0273 $g.mm.hr^{-1}.m^{-2}.Pa^{-1}$) which produced results within the 0.025 ± 0.005 range. According to Al-Hassan and Norziah (2012) [16], the average WVP for starch film incorporated with glycerol to form film resulted between 1.8×10^{-4} and 2.5×10^{-4} $g.mm.hr^{-1}.m^{-2}.Pa^{-1}$ lowering the WVP. A low water vapour permeability is a preferred physical property since low WVP refers to a lower moisture transfer between the food and the surrounding atmosphere [21][22]. Also with approximately similar thicknesses (0.12 - 0.16 mm) for all film conditions, the mixing temperatures have affected the WVP of

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the film. From the 3D diagram illustrated in Fig. 1 (a), it can be seen that at the highest mixing temperature (80 °C), the WVP is at its lowest value while at the lowest mixing temperature (50 °C) resulted in higher WVP. However, at lower speed, the increasing temperature does not significantly influence the changes of WVP but at higher speed, the increment of temperature shows a higher influence as the WVP is reduced. Akhtar et al. (2012), [23] asserted that an edible film, which contained antioxidant from flower extract, have to be produced at 65 °C to avoid heat oxidation. Therefore, the mixing temperature (50 - 80 °C) have been used for this experiment to avoid heat oxidation, yet the optimum temperature is required to produce low WVP in the edible film.

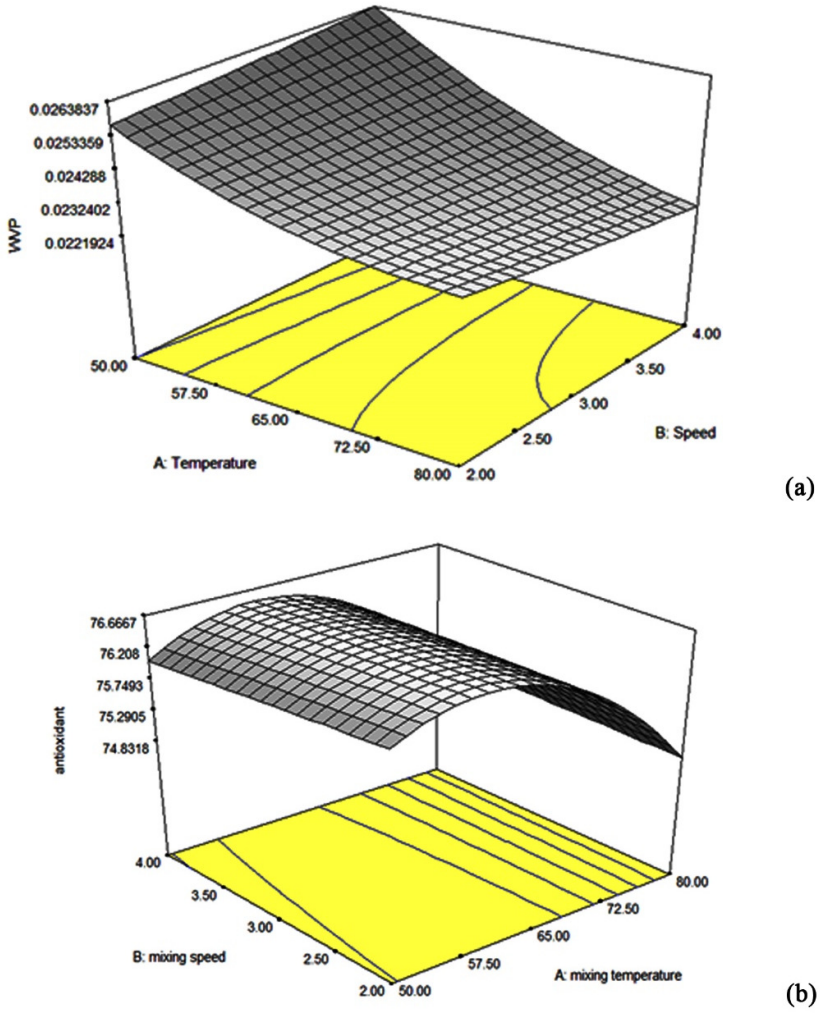

Fig. 1 Response surface plots and trace interaction graph displaying the effects of different mixing processing conditions on (a) water vapour permeability and (b) antioxidant capacity.

It is important to note that natural antioxidant properties in edible film can provide a sustained release of antioxidants in food during storage [24]. Antioxidants incorporated in food packaging can also minimize lipid oxidation, maintain the nutritional quality and prolong their shelf life [25]. The results depicted in Fig. 1 (b) showed that the antioxidant (%) are in the range of 75.0 ± 3.0 % and that variation in mixing temperature did not significantly influence

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the antioxidant value. This is parallel to a statement claimed by Moreno et al. [26]. Despite the small changes of antioxidant, a higher heat treatment caused a reduction in DPPH scavenging activity as studied by Lou et al. [27]. In addition, Okutsu et al. [28] also investigated heat treatment (45 - 75 °C) on *koji*, where they found that at higher heat treatment, the antioxidant capacity of the sample was reduced due to the probability of being catalysed by Maillard reaction [28].

3.2 Optimization of PSP Film Processing Condition

Optimization formulation had been derived from the results of the optimum conditions is required to produce the best PSP starch-based edible film. The desired water vapour permeability had been the best at the lowest WVP (%) [30], while the ranges of antioxidant capacity were the best at maximum percentage [27]. Mixing temperature of 69.20 °C and 4.0 Mot (1000 rpm) speed showed the highest desirability value (100 %). According to Al-Hassan and Norziah [16] who studied mixing temperature from a range of 40 °C to 90 °C, the gelatinization temperature for mixture of starch to produce edible film was observed at 67.13 °C. Thus the authors proved that the optimum mixing temperature was in the range of 67 to 70 °C. Meanwhile, a study carried out by Wittaya (2012) [31], also showed that when the mixing temperature increased, the hydrophilic interactions increased, but the hydrogen bonds and the electrostatic interaction decreased. At higher mixing speed; 4.0 Mot (1000 rpm), an increasing temperature of 70 °C influenced the reduction of WVP and high antioxidant capacity were obtained. On the other hand, with lower mixing speed; 2.0 Mot (500 rpm), a slight increase in WVP and decreased the antioxidant capacity were observed. The desired value of mixing temperature of 69.20 °C and mixing speed of 4.0 Mot were examined in triplicate to ensure that they tallied with the response results. In addition, the mixing temperature in this study was altered to 70 °C, while the mixing speed was retained at 4.0 Mot. The results showed that water vapour permeability exhibited a slight increment from the expected results given by the DX6 software. It can be concluded that the actual measurement of independent variables have shown significant results and can be used for further tests and analysis, and be used as the optimum condition to produce PSP starch-based edible film.

3.3 Morphological Characteristics

Different samples were observed at 3000 magnification, in order to identify the surface and the overall morphology of the cross-sectional area of the film. The surface morphology of PSP film for all the processing conditions portrayed different sizes of porosity and condition of the surface area [32]. Fig. 2 (c) shows that at the highest mixing temperature and the highest speed shows a smoother cross sectional surface image that shows less porosity was observed while the lowest temperature with the lowest speed, as illustrated in Fig.2 (a), resulted in a more porous film and rougher surface. The results obtained in this study are in line with those of Mali et al. [33], where increasing temperature of gelatinization resulted in a smoother surface and compact structure. Despite being least porous at the highest mixing temperature and speed, it failed to exhibit good water vapour permeability, which was probably caused by lesser film thickness and water vapour penetrated easily throughout the film. Run 2 with the lowest temperature and the lowest speed also showed a low water vapour permeability based on WVP results. Therefore, the cross-sectional PSP film did not correlate with the changes of WVP in PSP film.

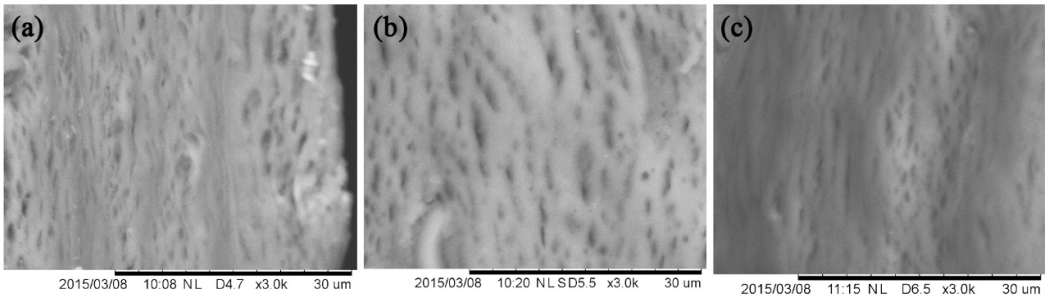

Fig. 2 SEM images of PSP film produced at different processing condition; (a) Run 2 (50ºC, 2.0 Mot), (b) Run 4 (65ºC, 3.0 Mot) and (c) Run 9 (80ºC, 4.0 Mot).

4.0 CONCLUSION

Various mixing temperatures displayed different effects on film formation. The optimized processing conditions to produce PSP film were identified as follows: 70 °C mixing temperature and 4.0 Mot (1000 rpm) mixing speed. This formulation exhibited the ability to obtain low (0.0241 g mm m^{-2} hr^{-1} Pa^{-1}) water vapour permeability, which enables the film to be used for food packaging. Besides, by achieving high antioxidant capacity of 76.19 % and adequate transparency value (2.443) on the film, the PSP film would become a favourable functional edible film from starch-based materials. The porosity of the film was also influenced by different mixing temperatures. Thus the results from this study reflect a positive prospect in commercializing starch food packaging in the food industry.

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