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Effect of dangke whey/pectin ratio, stearic acid and cheese aroma concentration on characteristic properties of edible films made thereof

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Abstract. The successful fabrication of a flavored edible film is determined by the right selection of the materials used include polysaccharides, proteins, aroma and lipids or combination of these. This study investigate the effects of different ratio of dangke whey/pectin, stearic acid and cheese aroma concentration on moisture content, thickness, water solubility, and water vapor transmission rate, tensile strength and elongation of an edible film made thereof. Full factorial designs at two levels of ratio dangke whey/pectin (1:4 and 2:3), stearic acid concentration (0.125% and 0.825%) and aroma (without aroma and 2%) were used to analyze their's effects on the physical, mechanical and barrier properties of film. Of the ratio tested, ratio 1:4 found to be the lowest moisture content. Increase stearic acid concentration to 0.825% decreased the thickness of film. Incorporation of cheese aroma 2% to the polymer matrix increased both tensile strength and elongation. However, the ratio, stearic acid and aroma concentration did not improve the barrier properties of films. The results showed that film composition are the major factor influencing the film properties.

1. Introduction

Research on edible film has been extremely relevant due to it has functional properties such as barrier and carrier. Furthermore, edible film has the potential to extend the shelf life of food products. It was reported that cellulose-based film with added plasticizer significantly increased the permeabilities of CO₂, O₂ and water vapor but it was not found as lipid was added into hydroxypropyl cellulose-based films [1]. It was also indicated that edible films with added essential oils gained great interest as a carrier due to their ability to enhance the shelf life of food products [2].

Several studies have been conducted on how ingredients composition of edible film affected physical, mechanical and barrier its properties. Among others, some researchers Benerjee and Chen [3] explored the effects of whey protein concentrate and composite whey protein concentrate on water vapor barrier and mechanical properties such as water vapor permeability, tensile strengths, elongation and transmission electron microscopyfunctional properties of films. Results reported by the authors showed that whey protein concentrate films had good water vapor barrier and mechanical properties in comparing those of films derived from sodium caseinate, potassium caseinate, calcium caseinate, and whey protein isolate [3]. In addition, it was also studied the influence of stearic acid and pH in whey protein on mechanical properties, water vapor permeability and protein solubility. The level of stearic acid (0.0 to 1.0%) and pH (5.0, 6.0, 7.0 and 9.0) were used in this study [4]. Some researchers used whey protein concentrate incorporated with cinnamon essential oil [5]. The authors observed the



effects of cinnamon on microstructure, physical, mechanical and antimicrobial properties of the films. The authors found that incorporation of cinnamon essential oil exhibited good inhibitory effect on the studied fungi but it could decrease the tensile strength of the films.

In addition to whey protein, some authors also reported the use of pectin for fabrication of edible films. Pectin are group polysaccharides consisting almost of d-galacturonic acid and galacturonic acid methyl ester residues interspersed with a few (1→2)-linked l-rhamnose residues, which are linked to neutral sugar side-chains, such as l-arabinose, d-galactose, d-xylose, d-mannose, and d-glucose [6]. Some researchers investigated the behavior of pectin and thermally denatured whey proteins at both different whey protein /pectin ratios and different pH values. The results reported by the authors suggest the formation at pH 5.1 (complexation pH) of transglutaminase-catalyzed cross-links among soluble ionic whey protein/pectin complexes, which could be responsible for the observed increase of both tensile strength (2-fold) and elongation to break (10-fold) of films obtained in the presence of enzyme [7].

It was widely recognised that food processing and food matrix are attributable to the poor of flavor quality. Therefore, the aroma loss of products can be handled through the use of edible film. The aroma and flavour enhancers as well as nutritional substances could be integrated by using milk protein-based edible films [8].

This research was intended to enrich scientific information concerning to physical, mechanical and barrier properties of films composed with dangke whey/pectin ratio, concentration of stearic acid and cheese aroma. Unlike the previous studies, this research applied two level of dangke whey/pectin ratios (1:4 and 2:3), two levels of stearic acid concentration (0.125% and 0.825%) and two levels of cheese aroma concentration (no aroma and 2%)

2. Material and methods

2.1. Materials

Dangke whey was supplied from processing laboratory. Pectin purchased from Nura Jaya (Surabaya, Indonesia) were used as film-forming components of the hydrophilic continuous phase for emulsion-based films. Stearic acid purchased from local market was used as hydrophobic disperse phase, and glycerol purchased from local chemical store was added as a plasticizer. Cheese aroma used in this work was purchased from local market.

2.2. Methods

2.2.1 Film preparation. Eight films, varying in their formulations, were produced for this experimental according to a full factorial design based on three factors: the concentration of ratio dangke whey/pectin (WP) (4.5%) with two different ratio of WP (WP1=1:4, WP2=2:3), the stearic acid (SA) concentration (SA1=0.125%, SA2=0.825%), and cheese aroma concentration (A0= no added aroma, A1= 2%).

To produce edible films, the dangke whey was dissolved in distilled water using magnetic stirrer at 65°C for 15 min. The pectin was added successively into solution and stirred for 15 min. Then glycerol was added into solution under constant stirring for 15 min. Stearic acid and cheese aroma was finally added into film forming solution and stirred at 65°C for 15 min. The final film forming solution was cooled down to 40°C prior to casted in petri dishes (10 cm diameters). 20 ml of film-forming solutions were spread onto petri dishes and they were subsequently dried in an oven at 50°C for 18 h. Finally, dried film were peeled off from the petri dishes and kept in a dessicator at room temperature until evaluation.

2.2.2. Film characterization

Thickness. Film thickness was measured by using a digital caliper (KRISBOW KW06-422). The values showed represent the means of five measurements randomly taken during each evaluated samples.

Moisture content [9] The moisture content of samples was determined by calculating the loss of film weight after drying in an oven at 105°C for 24 h.

Tensile strength and elongation. A tensile strength and elongation specimen was created with dimensions those specified in the ASTM D638-02a-2002.

Water Vapour Transmisson Rate (WVTR) [10]. The WVTR was determined using gravimetric dessicant method that was modified. Small cups with lids were prepared and filled with silica gel. Then, the films were placed on the mouth of cup then sealed with wax. After sealed, the cups were weighed then placed in dessicator containing 27% of NaCl for interval time 0, 8, 24, 32, 48 h. Then, the weights of cup were recorded. Data obtained then made linear regression equation in order to obtain the slope of weight of cup. WVTR is expressed by the slope of weight of cup (g/h) divided by the area of the film tested (cm²). The mean value of three measurement replications was reported for each sample.

Water solubility [11]. The solubility of samples was expressed as the percentage of the film dry mass which is soluble after 24h immersion in distilled water. First, 3x2 cm of samples were dried in an oven at 105°C for 24 h and weighed (W1). After 24 h drying, each samples was immersed into a 50 ml tube containing 10 ml of distilled water. After 24 h immersion in distilled water, the solution was filtered using filter paper and finally the samples remained on the filter paper were oven dried at 105°C during 24 h after which the samples were weighed to determine the dried remnant insoluble mass (W2).

3. Statistical analysis

A full-factorial design was performed. Three replications were used to determine physical, mechanical and barrier properties. Analysis of variance was used and when the effects were significant ($p < 0.05$). Data analyses were performed using R software (R-3.3.1 release). The relationship between physical, mechanical and barrier properties was studied using explorative principal component analysis (PCA) function of the *FactoMineR* package.

4. Results and discussion

4.1. Moisture content

Films obtained with the various ratios, stearic acid and aroma concentration and their effect on the moisture content can be seen in table 1. The films moisture content ranges between 22.67-34.10% (table 1). For the film containing ratio 2:3 (WP2) with higher stearic acid (SA2) and aroma addition (A2), the highest moisture content was observed. The moisture content increased significantly ($p < 0.05$) as the dangke whey more close to the pectin (2:3) (table 3). In contrast, the use of stearic acid and aroma did not show this trend ($p > 0.05$). This may reflect the difficulty of the undissociated form of stearic acid to be integrated into the film structure.

4.2. Thickness

Table 1 shows the thickness of edible films including ratio dangke whey/pectin 1:4 or 2:3, stearic acid concentration 0.125 or 0.825%, cheese aroma concentration 0% and 2%. For the films containing ratio 1:4 (WP1) with higher stearic acid (SA1) and aroma addition (A1), the highest thickness was obtained (table 1). The results of ANOVA indicated that WP ratio, concentration of stearic acid and aroma affected film thickness ($P < 0.05$). Inreasing pectin to the dangke whey (and the corresponding decrease of dangke whey) (1:4) provided more dissolved pectin into solution leading in thicker films. The increased concentration of the materials used will lead to an increase in film thickness [12]. In

addition, the addition of stearic acid in the fabrication edible film resulted in thicker films. This is due to the addition of stearic acid in solution provides more hydrogen bonding with polymer formed.

Table 1. Physical, mechanical and barrier properties of edible films

WP ratio	Stearic Acid (%)	Cheese aroma (%)	MC (%)	Thickness (mm)	Water solubility (%)	WVTR x 10 ⁻³ (g/h.cm ²)	TS (MPa)	Elongation (%)
1:4 (WP1)	0.125	0 (A0)	26.38±0.05	0.15±0.00	83.20±3.43	1.3±0.00	4.73±0.04	20.83±1.48
	(SA1)	2 (A1)	25.34±0.34	0.22±0.00	20.08±0.51	1.7±0.00	16.63±1.79	59.67±5.34
	0.825	0 (A0)	26.39±1.29	0.20±0.00	59.50±7.19	1.6±0.00	4.66±0.03	18.93±1.34
	(SA2)	2 (A1)	22.67±0.45	0.22±0.02	25.38±0.19	1.6±0.00	15.95±3.29	67.00±3.12
2:3 (WP2)	0.125	0 (A0)	31.40±0.01	0.16±0.00	80.39±19.36	1.6±0.00	8.72±0.23	54.23±5.13
	(SA1)	2 (A1)	25.22±0.18	0.18±0.01	21.18±0.25	1.3±0.00	11.95±0.48	65.93±3.07
	0.825	0 (A0)	28.58±0.21	0.20±0.00	59.31±7.71	1.5±0.00	8.80±0.28	46.67±6.67
	(SA2)	2 (A1)	34.10±0.90	0.18±0.01	31.36±0.1	1.5±0.00	9.56±1.86	54.10±10.13

Table 2. ANOVA on the physical and barrier properties of films

		MC (%)	Thickness (mm)	Water solubility (%)	WVTR (g/h.cm ²)	TS (MPa)	Elongation (%)
WP ratio	F	119.22	8.39	0.03	0.31	0.49	13.19
	p-value	<.0001	0.001	0.85	0.58	0.49	0.001
Stearic acid	F	3.98	17.80	1.73	0.31	0.52	0.87
	p-value	0.06	<.0001	0.21	0.58	0.47	0.36
Aroma	F	10.23	16.41	68.25	0.08	41.35	49.94
	p-value	0.001	<.0001	<.0001	0.78	<.0001	<.0001
WP ratio : Stearic acid	F	26.46	0.13	0.11	0.17	0.14	2.74
	p-value	<.0001	0.72	0.74	0.68	0.71	0.12
WP ratio: Aroma	F	5.88	15.96	0.20	2.34	20.64	20.40
	p-value	0.03	0.001	0.66	0.14	<.0001	<.0001
Stearic acid:Aroma	F	28.20	13.81	7.28	0.02	0.53	0.11
	p-value	<.0001	0.001	0.01	0.89	0.48	0.74
WP ratio : Stearic acid : Aroma	F	71.63	1.19	0.01	2.78	0.19	0.81
	p-value	<.0001	0.29	0.92	0.11	0.66	0.38

4.3. Water solubility

One approach to explore the ability of film to enhance product integrity and water resistance is water solubility. Once the films were exposed to the solution, they dissolved and it found that film composed with a lower dange whey to the pectin (and corresponding to increase pectin) (1:4), lower stearic acid and no aroma showed the highest water solubility, which was 83.20%. In contrast, the use same ratio and stearic acid but with aroma 2% showed the lowest water solubility, which was 20.08% (table 1). The result of ANOVA in table 2 indicated that concentration of aroma and interaction between stearic acid and aroma affected the film solubility ($P < 0.05$). Increased concentration of aroma was followed by decrease the film solubility (table 3). This caused by the hydrophilic of cheese aroma leading to increasing number of bonds between molecules in film forming solution. This result is in line with the results reported in the fabrication of edible film from mung bean [13].

4.4. Water vapor transmission rate

The WVTR values obtained can be seen in table 1. The films water vapor transmission ranges between 1.3×10^{-3} - 1.7×10^{-3} (table 1). The lowest WVTR value was observed in edible film composed with lower concentration of stearic acid whatever WP ratio and concentration of aroma (table 1). In general, no significant effect of WP ratio, stearic acid, aroma addition, and their interaction on the water vapor transmission rate ($P>0.05$) have been observed (table. 2). Because the amount of stearic acid incorporated is very low ($<1\%$), it seems stearic acid has no ability to increase the tortuosity factor for transfer of water molecules.

4.5. Tensile strength (TS)

Tensile strength is the mechanical property of an edible film which reflects the maximum stress that the film sustains before it eventually breaks. The films tensile strength ranges between 4.66-16.63 MPa (table 1) depending on the WP ratio, concentration of stearic acid and aroma. The result of ANOVA in table 2 indicated that the treatment of aroma addition and interaction between WP ratio and aroma addition significantly affected the TS, while WP ratio and stearic acid were not significant ($P>0.05$). TS increased with the addition of aroma (table 3). This is due to the hydrophilic nature of cheese aroma used. As aroma was added, the hydrogen bonds were formed between aroma and dangeke whey leading compact structure of films. In addition, cheese aroma interacts with material such as dangeke whey or pectin plays a stabilizing role in the interface leading to increase tensile strength. According to Baldwin et al., 2012 incorporating volatile active compound affects the structural organization of the film-forming substance network and consequently its physical and mechanical properties. It is also important to note that the study by researchers found that the TS value of film composed with protein and starch increase with the increase in the base material concentration used [14, 15].

4.6. Elongation

The results of ANOVA in table 2 showed that WP ratio, concentration of aroma and their interaction significantly affected the elongation ($p<0.05$) of film, whereas the concentration of stearic acid was not significant ($p>0.05$). The highest elongation of films was observed on the films composed with WP ratio 1:4, concentration stearic acid 0.825% and aroma addition 2%, which was 67.00% (table 1). The results of ANOVA in table 2 showed that WP ratio and aroma significantly affected the elongation of films, whereas the concentration of stearic acid were not significant ($P>0.05$). The elongation increased with increasing ratio dangeke whey to pectin (and corresponding to decrease pectin) and increasing the concentration of aroma (table 3). The influence of the component ratios on the mechanical properties of films made from polysaccharide and protein blends was also observed [16].

Table 3. Main factor analysis using Bonferonni test

	MC (%)	Thickness (mm)	Water solubility (%)	WVTR $\times 10^{-3}$ (g/h.cm ²)	TS (MPa)	Elongation (%)
WP ratio						
1:4	25.19 \pm 0.55 ^a	0.20 \pm 0.01 ^a	47.04 \pm 7.96 ^a	1.50 \pm 0.00 ^a	10.49 \pm 1.92 ^a	41.60 \pm 6.74 ^a
2:3	29.82 \pm 1.01 ^b	0.18 \pm 0.01 ^b	48.06 \pm 8.31 ^a	1.49 \pm 0.00 ^a	9.76 \pm 0.57 ^a	55.23 \pm 3.55 ^b
Stearic acid						
0.125	27.09 \pm 0.77 ^b	0.18 \pm 0.01 ^a	51.22 \pm 10.13 ^a	1.47 \pm 0.00 ^a	10.51 \pm 1.37 ^a	50.17 \pm 5.54 ^b
0.825	27.93 \pm 1.30 ^b	0.20 \pm 0.01 ^b	43.88 \pm 5.23 ^a	1.54 \pm 0.00 ^a	9.74 \pm 1.46 ^a	46.67 \pm 5.94 ^b
Cheese aroma						
0	28.18 \pm 0.68 ^b	0.18 \pm 0.01 ^a	20.23 \pm 5.84 ^a	1.50 \pm 0.00 ^a	6.73 \pm 0.62 ^a	35.17 \pm 5.03 ^a
2	26.83 \pm 1.33 ^b	0.20 \pm 0.01 ^b	4.64 \pm 1.34 ^b	1.52 \pm 0.00 ^a	13.50 \pm 1.25 ^b	61.67 \pm 3.05 ^b

4.7. Principal component analysis

A PCA was carried out to determine the dominant physical, mechanical and barrier properties among the different films and to classify the films according to the similarity of their properties. The process of data analysis by PCA results in the formation of a score matrix and a loading matrix. Fig.1 presents the PCA of the first two components of films and show how the ratio of WP, different concentration stearic acid and aroma affected the physical, mechanical and barrier properties of films. The first two components (PC1 and PC2) together explained 75.19% of the variance, with PC1 and PC2 explaining 57.15% and 18.04% of the variance, respectively. PCA (fig. 1) shows that WVTR and thickness were well correlated since they are plotted close to each other. This is in line to the previous results where films composed with WP ratio 1:4, concentration of stearic acid 0.125% and concentration of aroma 2% presented the highest thickness and WVTR (table 1). Whereas TS versus MC and solubility, plotted on opposites sides of the PC, were negatively correlated. MC and solubility loadings which were placed against WVTR and thickness do not correlate.

PC1 reflects the effects of film WVTR and thickness properties and showed mainly a positive correlation with WVTR and thickness and a negative correlation with MC and solubility. PC2 is positively correlated with solubility, MC, WVTR and thickness and negatively correlated with tensile strength.

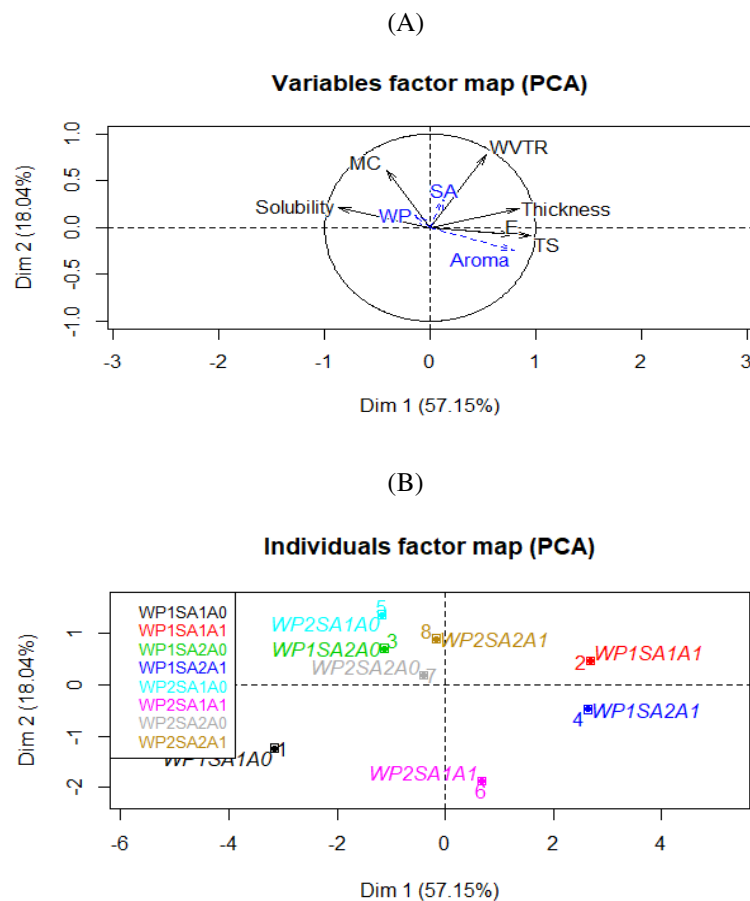


Figure 1. A Biplot representation based on the two first dimensions of a PCA performed on the physical, mechanical and barrier properties data obtained for the 8 films (as a mean of samples). The individuals were the 8 film samples and the variables the 6 parameters monitored. The films were coded according to their WP ratio (WP1 = 1:4, WP2 = 2:3), concentration of stearic acid (SA1 = 0.125%, SA2 = 0.825%) and concentration of cheese aroma (A0 = no aroma, A1 = 2%).

5. Conclusion

The results provided useful information on physical, mechanical and barrier properties of edible films composed with different ratio of dangke whey/pectin, concentration of stearic acid and cheese aroma. WP ratio, concentration stearic acid and aroma modify physical and mechanical properties. However, the ratio of WP, concentration stearic acid and aroma did not improve the barrier properties of edible films.

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