

Stengthening of Edible Film From Corn Starch and Iota Carrageenan with Butterfly Pea Flower Extraction (*Clitoria ternate* L) Addition

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Abstract. One of the most enormous waste is plastic. Most plastic waste can damage the environment on land and in the oceans. Food packaging plastic is switched from conventional to natural materials. The research goal is an analysis of biodegradable, edible film plastic materials. A combination of corn starch (CS) (4, 3.5, 3, 2.5, 2% w/v), iota-carrageenan (IC) (0, 0.5, 1, 1.5, 2% w/v), and butterfly pea flower extraction (BPE) (0, 4, 8% v/v) can be developed as edible biodegradable films. The method used in this research is bench casting. These edible films are characterized based on physico-mechanical properties, water properties, and biodegradation. The results of water vapor permeability as water properties in CS/IC3 samples was 19.449 g.mm/m².h.kPa, and in the CS/IC/BPE 3-2 samples was 12.596 g.mm/m².h.kPa. Based on the results, the tensile strength as physical-mechanical properties of the samples increased as the iota-carrageenan and BPE content occurred in the CS/IC5 sample with a value of 3.32 MPa and the CS/IC/BPE 5-2 sample with a value of 4.93 MPa. The CS/IC/BPE sample extensively reduced cherry tomato weight loss and increased the duration of storage of the studied tomatoes by 10 days. The research shows that the natural material CS/IC/BPE produces a good edible film for packaging.

1 Introduction

The use of plastic is inseparable from human activities. The ever-increasing use of plastic due to its robust nature, good flexibility, and affordable price makes plastic an option for food packaging. Conventional plastics, which are not environmentally friendly, take hundreds of years to degrade by nature and produce high carbon emissions [1]. The plastic industry has made non-degraded plastic by nature. Edible film is a thin layer used as food packaging to extend the shelf life of food.

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Natural polymers such as polysaccharides, proteins and lipids can be used to make edible films[2]. Polysaccharides are natural polymers generated from starch, glycane, agarose, and cellulose. Starch is widely used as a raw material for making edible films. Starch comes from several plants, such as rice, potatoes, taro, corn, etc. Starch has primary polymer elements, namely amylose and amylopectin [3]. Corn starch is a natural polymer that has many sources. Corn starch offers renewable characteristics, biodegradability, edibility, cheap cost, excellent mechanical resistance, and water-holding ability, making it a good material for packaging and food preservation applications [4], [5]. Iota carrageenan is an extract from marine algae or red algae. Films mixed with iota carrageenan produce good mechanical properties and lower permeability than previous studies on arrowroot starch and iota carrageenan [2]. Carrageenan has three types, namely kappa, iota, and lambda. Iota carrageenan has good elasticity properties for food packaging manufacturing compared to kappa, which is stiff and brittle, and lambda, which does not have gel properties [6]. Butterfly pea flowers contain anthocyanins that are useful for natural food coloring. Anthocyanin extracts can be a mixture in film making because they have good mechanical, physical, and barrier properties [7]. The film can be used to monitor the freshness of packaged pork in combination with anthocyanins. Because of the eco-friendly nature of natural films, they can attract consumers, retailers, and manufacturers for food packaging [8].

The study hypothesized that incorporating anthocyanins in starch and carrageenan bases would help obtain the film's moisture-barrier and physico-mechanical properties. This study aimed to obtain a new biodegradable material from corn starch, iota carrageenan, and anthocyanins from butterfly pea flower extract.

2 Materials and methods

2.1 Material

Corn starch from PT Daesang Agung Indonesia, West Java, Indonesia. Butterfly pea flowers from a butterfly pea garden in Mojolaban, Sukoharjo, Central Java, Indonesia. Iota Carrageenan from Indonesia's handmade chemical shop, Surabaya, East Java, Indonesia. Glycerol was purchased from CV. Indrasari Semarang, Central Java, Indonesia.

2.2 Extraction of anthocyanin of butterfly pea flower (BPE)

Dried butterfly pea flowers are used for anthocyanin extraction raw materials. Aquadest is a solvent of anthocyanins, with a ratio of solid and liquid 1:20. The extraction is carried out at a temperature of 45°C for 60 minutes.

2.3 Production of biodegradable edible film

The edible film solution was made following the procedure from the previous study [2]. Corn starch (CS), iota carrageenan (IC), glycerol (Gly), and anthocyanins from butterfly pea flower extract (BPE) were prepared, as shown in Table 1. The film solution was prepared by dissolving corn starch into aqua dest heated to a temperature of 70°C on the hot plate stirrer with constant stirring at 200 rpm for 5 minutes. IC was added to the solution with further heating until well mixed, then anthocyanin extract was added with continuous stirring and finally added with glycerol at the same speed until the solution was mixed. The solution was transferred to a film mold and then dried in an oven at 40°C for 7 hours. The dried film was stored in a desiccator before further analysis.

Table 1. Edible film combination ratio

Film Code	CS (%, w/w)	IC (%, w/w)	Gly (% of dry weight)	BPE (v/v%)
CS1	4	0	30	0
CS/BPE1-1				4
CS/BPE1-2				8
CS/IC2	3,5	0,5	30	0
CS/IC/BPE2-1				4
CS/IC/BPE2-2				8
CS/IC3	3	1	30	0
CS/IC/BPE3-1				4
CS/IC/BPE3-2				8
CS/IC4	2,5	1,5	30	0
CS/IC/BPE4-1				4
CS/IC/BPE4-2				8
CS/IC5	2	2	30	0
CS/IC/BPE5-1				4
CS/IC/BPE5-2				8

2.4 Physico-mechanical properties

2.4.1 Film thickness

A digital micrometer was used to measure the film thickness in three different areas. Take a random area of the film at three points of the film and measure the film thickness using a digital micrometer.

2.4.2 Tensile strength and elongation

A universal testing machine (UTM) was used to determine the tensile strength. Samples were cut into 25 × 5 mm strips and measured at 1 mm/s. [2].

2.5 Barrier Properties of the film

2.5.1 Water vapour permeability

The gravimetric method from ASTM E96-95 was used to evaluate the water vapour permeability of film samples. [9]. In a test chamber, a glass beaker containing dry silica gel (10 g) and covered with a circular film sample was retained. The glass beaker was weighed every 24 hours for seven days. The water vapour permeability value can be calculated using below:

$$WVT \left(\frac{g}{m^2 h} \right) = \left[\frac{G}{t.A} \right] \quad (1)$$

$$WVP \left(\frac{g.mm}{m^2 h.kPa} \right) = \left[\frac{WVT}{S(RH_1 - RH_2)} \right] \quad (2)$$

Where:

WVT= water vapour transmission (g/m²h)

G = weight lost (g)

A = surface area of the beaker (m²)

WVP= water vapour permeability

RH₁ = relative humidity of dry silica

RH₂ = relative humidity of the test chamber

2.5.2 Swelling index

Film strips (20mm×20mm) soaked in 30ml deionized water for 5 hours at 25°C. The solution is filtered, and the film sample is wiped using soft paper and weighed. The swelling test was calculated using the equation below [2]:

$$Swelling (\%) = \left[\frac{w_1 - w_0}{w_0} \times 100 \right] \quad (3)$$

2.5.3 Moisture content

Film samples were cut to 2 cm × 2 cm, then weighed and dried in the oven at 105°C for 24 hours. Then, the dried sample was weighed again to calculate the moisture content using the equation below [10]:

$$moisture\ content (\%) = \frac{w_1 - w_2}{w_1} \times 100 \quad (4)$$

2.6 FTIR

Film samples were analyzed with an FTIR spectrometer. All analyzed samples FTIR spectra are presented as transmission (%) in the wavelength range 400 to 4000 cm⁻¹. [11].

2.7 Packaging tests

The packaging test was conducted using cherry tomatoes. Cherry tomatoes were weighed before the packaging test, and the weighted cherry tomatoes were put into a small glass beaker and covered with a film sample for ten days. After ten days, the cherry tomatoes were weighed again to determine their progress during the packaging test. The packaging test will calculate the mass of cherry tomatoes lost using the equation [2]:

$$\text{weight loss (\%)} = \left[\left(\frac{w_0 - w_1}{w_0} \right) \times 100 \right] \quad (5)$$

3 Results and discussion

3.1 Physico-mechanical properties

3.1.1 Film thickness

The highest thickness was obtained on the CS/BPE1-2 film, which was 0.13 mm, and the lowest was in the CS/IC5 film, which was 0.06 mm (Table 2). The decrease in film thickness in each fraction of corn starch and iota carrageenan is also influenced by the decrease in dry matter soluble in the film former. The decrease in starch content in the film matrix and the increased content in iota carrageenan can reduce film thickness, as found by Abdillah & Charles (2021) and Thakur et al. (2016) [2], [11]. The addition of anthocyanin extracts from the butterfly pea flower showed an increase in thickness compared to the control film. The increase in film thickness is due to changes in gelatin chain confirmation with the addition of butterfly pea flower extract, as found by Gasti et al. (2021)[7]. The increase in film thickness was due to an increase in the concentration of butterfly pea flowers and film matrix.

3.1.2 Tensile strength and elongation

The results of Table 2 show that the highest tensile strength shown in CS/IC/BPE5-2 film is 4.93 mPa and decreases in samples with reduced iota Carrageenan concentration and increased corn starch content in CS1 samples as well research conducted on arrowroot starch and iota carrageenan [2]. Increasing anthocyanin content also increases the tensile strength value of corn starch film and iota Carrageenan edible films [7].

Increasing corn starch on the film can increase the film value of corn starch and iota carrageenan. Increasing dry matter can increase elasticity in corn starch films and iota carrageenan, as in the research of Abdillah & Charles (2021)[2]. Increasing the anthocyanin content in corn starch films can reduce the elongation value caused by the formation of crosslinking interactions between phenolic groups and striped flower extracts and -NH groups₂ and -OH from the film matrix, which decreases the flexibility of the film, also reducing the consequences of plasticizers [7]. In research, Yong et al. (2022)[12] found that adding anthocyanins to the film matrix did not change the TS value significantly due to the small addition of anthocyanins.

3.2 Barrier properties of the film

3.2.1 Water vapor permeability

The addition of iota carrageenan can reduce the WVP value compared to the CS1 films that do not contain iota carrageenan, namely 18,335 g.mm/m².kPa.h, which goes down to the CS/IC2 film with iota carrageenan 0.5% w/v to 13.852 g.mm/m².kPa.h, as well as research conducted by Abdillah & Charles (2021)[2] and Thakur et al. (2016)[11]. CS/IC/BPE3-1 films decreased the WVP value from CS/IC3 films without anthocyanins. The addition of anthocyanin to the film matrix had no significant impact on the CS/IC film's WVP value. [12], [13].

3.2.2 Swelling index

Because of the physical hydrogel characteristic of iota carrageenan as a water absorber, decreased corn starch content can enhance the swelling index. As in CS/IC3 film, which is 140.483% to 178.497% (CS/IC4 films) due to the increased iota carrageenan content [2], [11]. The addition of anthocyanin extracts from the butterfly pea flower also increased the swelling index from 222.135% to 269.658% [8].

3.2.3 Moisture content

Increased concentrations of iota carrageenan and the addition of anthocyanin extracts can reduce the moisture content, which shows the limited ability of water absorption when releasing anthocyanins. The addition of 0.5% w/v iota carrageenan increased the moisture content by 25.95% (CS/IC2) from CS/BPE1-2 samples, which do not contain iota carrageenan but contain 8% v/v anthocyanin extract by 40.86% and decrease the moisture content as iota carrageenan increases in the film matrix. The results of this study are similar to those of Thakur et al. (2016)[11]. Adding anthocyanin extracts can reduce the film matrix's moisture content, as in Koshy et al.'s research (2021)[8].

Table 2. physicomechanical properties and barrier properties of edible film

Film	Thickness	TS	Elongation	MC	PWV	Swelling
Code	(mm)	(Mpa)	(%)	(%)	(g.mm/m ² .kPa.h)	(%)
CS1	0,10	1,15	42,32	18,22	18,335	154,255
CS/BPE1-1	0,11	2,37	29,79	36,67	14,197	155,799
CS/BPE1-2	0,13	3,19	20,10	40,86	13,551	188,563
CS/IC2	0,08	1,96	36,14	25,95	13,852	75,294
CS/IC/BPE2-1	0,08	2,50	25,81	55,00	16,161	102,387
CS/IC/BPE2-2	0,10	3,71	17,29	33,61	18,825	96,120
CS/IC3	0,07	2,57	34,49	50,00	19,449	140,483

CS/IC/BPE3-1	0,08	3,35	21,68	58,89	13,103	341,378
CS/IC/BPE3-2	0,09	4,15	18,92	49,77	12,596	214,728
CS/IC4	0,07	2,84	29,41	36,35	13,823	178,497
CS/IC/BPE4-1	0,07	3,76	19,08	24,39	17,872	311,429
CS/IC/BPE4-2	0,08	4,47	24,99	45,38	19,851	227,037
CS/IC5	0,06	3,26	24,99	29,19	13,601	59,415
CS/IC/BPE5-1	0,07	4,28	17,24	53,57	18,331	222,135
CS/IC/BPE5-2	0,07	4,93	11,51	47,05	19,051	269,658

3.3 FTIR

All films displayed the same FITR spectra pattern. In all samples, the OH group is seen at 3267 – 3306 cm⁻¹, while the C-H group is observed at 2931 – 2942 cm⁻¹, signifying glycerol, starch, and glucose units. With the addition of Iota Carrageenan to composites, the CS/IC2 film -OH value increased to 3291 cm⁻¹ in this investigation. This phenomenon is interpreted as mixing corn starch, iota carrageenan, and glycerol, increasing hydrogen bond interactions and tensile strength [2]. The bands shifted from 3291, 2926, 1646, and 998 cm⁻¹, respectively, to 3293, 2927, 1644, and 999 cm⁻¹. The interaction of the matrix functional groups of corn starch/iota carrageenan film with butterfly pea flower extract causes this band shift. FTIR results show that anthocyanins have high compatibility with the corn starch/iota carrageenan film matrix and produce the uniform film through intermolecular interactions between the -OH group of anthocyanin extract and the -N-H, O-H, and O-H groups of corn starch and iota carrageenan without modification in molecular structure, this study is following the research report from Gasti et al. (2021)[7].

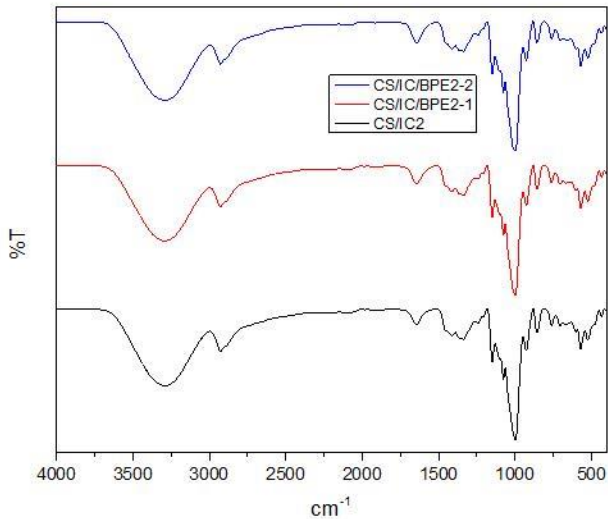


Fig 1. FT-IR edible film of corn starch and iota carrageenan with anthocyanins

3.4 Packaging tests

Tomato cherry control not coated with edible film experienced a weight loss of 14.739% smaller than tomatoes covered with edible film CS1, CS/IC2, and CS/IC/BPE3-1, which reached more than 30% and experienced faster decay. In contrast to tomatoes covered with other edible films, tomatoes experience shrinkage. Tomatoes that experienced the smallest weight loss were in sample CS/IC/BPE4-1 containing 2.5% corn starch, 1.5% iota carrageenan, and 4%v/v anthocyanin extract. Tomato weight loss and softening are connected with moisture loss, which leads in fresh fruit crispness loss [14].

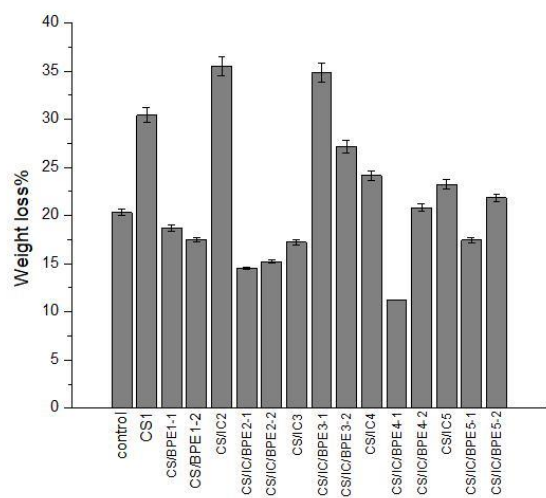


Fig 2. Weight loss tomatoes coated edible film corn starch and iota carrageenan with anthocyanins

4 Conclusion

The biodegradable film of corn starch (CS), iota carrageenan (IC), and butterfly pea flower extract (BPE) blends has physicomechanical and barrier properties that support their characterization as biodegradable films and prove effective biopolymers for producing films with potential food packaging applications. The thickness and flexibility combined with the corn starch are potential food packing materials. The IC fraction was responsible for increased tensile strength, a larger swelling index, and a decrease in WVP. FTIR results indicated an interaction between the film matrix and butterfly pea flower extract anthocyanins. CS/IC/BPE samples shown important advantages for increasing shelf life and food quality, suggesting their prospective applications in the food the sector, which strives to minimize its reliance on synthetic polymers.

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