

Research Article

# Formulation and Characterization of Capsule Shells from Breadfruit Peel (*Artocarpus altilis* (Park) Fosberg)

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## Abstract

Capsule shells are commonly made of gelatin but can also be made from starch or other suitable materials. An alternative raw material to replace gelatin in capsule shell production is pectin. Natural sources containing pectin include breadfruit peel. This study aims to produce capsule shells from breadfruit peel and determine the characteristics of the resulting capsule shells. Pectin was obtained by extracting breadfruit peel with acidified water to pH 1.5 at 80 °C for 150 minutes. The capsule shells were formulated with pectin concentrations of 3% (F1), 6% (F2), and 9% (F3). The characteristics of the pectin obtained include an equivalent weight of 3168 mg, methoxyl content of 3.26%, and galacturonic acid content of 96.27%. The capsule shells produced from this pectin have the following characteristics: white color, not too hard texture, and odorless for F1; light brown color, slightly hard texture, and odorless for F2; dark brown color, hard texture, and odorless for F3. The size parameters (weight, total length, body diameter, and cap diameter) obtained were F1 (60 mg, 21.5 mm, 7.25 mm, 7.5 mm), F2 (70 mg, 22.07 mm, 7.125 mm, 7.5 mm), F3 (80 mg, 21.25 mm, 7.25 mm, 7.5 mm). The characteristics of the capsule shells that only meet Medisca standards are F3 (80 mg), and the total length, cap diameter, and body diameter of all three formulations do not meet Medisca standards. The disintegration time in all three formulations already meets the requirements set by the Indonesian Pharmacopoeia V edition, which is below 15 minutes.

## Keywords

Breadfruit Peel (*Artocarpus altilis* (Park) Fosberg), Pectin, Capsule Shell

## 1. Introduction

Capsules are solid preparations consisting of medication in a hard or soft shell that can dissolve. The shell is generally made from gelatin, but can also be made from starch or other suitable materials [1]. Capsule preparations are widely used as medicine because they are practical and able to mask the taste of the medicine [2].

Commercial capsule shells are generally made from gelatin. Gelatin is collagen produced from the process of partial hydrolysis of collagen tissue which can be extracted from skin,

bones, pork, cattle or fish [3]. The main animals that produce gelatin with good quality and suitable gel strength values for various preparations are cows and pigs. Gelatin from a mixture of pork skin can produce the best quality capsule shells compared to other formulas because the capsule shells produced are of high quality with a film layer that is strong, clear and not easily brittle. Data from Gelatin Manufacturers of Europe in 2018, almost 80% of the gelatin produced came from pig skin, 15% came from cow skin, while the remaining

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5% came from beef, fish and pork bones. Gelatin from a mixture of pork skin can produce the best quality capsule shells compared to other formulas because the capsule shells produced are of high quality with a film layer that is strong, clear and not easily brittle.

Gelatin can cause concern among people who adhere to certain religions who are prohibited from consuming ingredients from pork (Islam) and cows (Hinduism) because it is haram. Therefore, other alternative raw materials are needed that are abundant, cheap and halal and do not cause concern. One alternative to replace pork and beef gelatin in the raw material for making capsule shells is pectin. The structure of pectin in the form of a polymer can be used as gelatin substitute.

Pectin is a complex polysaccharide derivative compound containing  $\alpha$ -D-galacturonate residues with  $\alpha$ -1,4 bonds [4]. Pectin is a compound found in plant cell walls. Pectin is composed of polymers of D-Galacturonic acid linked by 1,4 Glycosidic bonds [5].

The level of galacturonic acid contained in pectin is one of the parameters that shows the level of purity of pectin. The higher the level of galacturonic acid, the higher the purity of the pectin obtained. According to IPPA (International Pectins Producers Association) the minimum level of galacturonic acid is set at 35%. Meanwhile, the methoxyl content contained in pectin plays a role in determining the functional properties of the pectin solution and can influence the structure and texture of the pectin gel. According to the IPPA, the methoxyl content is determined to be more than 7.12%, which can make the gel structure produced from pectin stronger [6].

## 2. Materials and Methods

### 2.1. Material

#### 1) Apparatus

Knife, blender (PHilips®), sieve no. 40, filter cloth, analytical scale (Kern®), beaker (Iwaki®), hot plate (Velp Scientifica®), measuring cup (Pyrex®), Erlenmeyer (Pyrex®), mortar and stamper, Magnetic Stirrer, stirring rod, dropper pipette, oven (Mettler), pH meter, burette, parchment paper, capsule molding tool, compass shov, and disintegration tester, filter cloth, filter paper (Whatman) no. 41.

#### 2) Raw materials

Breadfruit peel (*Artocarpus altilis* (Park) Fosberg), 96% ethanol, 0.1 N HCl, 0.25 N HCl, 0.1 N NaOH, NaOH 0.25 N, acid ethanol, NaCl, phenolphthalein indicator, distilled water, sorbitol, Carrageenan.

### 2.2. Methods

#### 1) Simplicia Preparation and Extraction

##### 1. Sampling

The breadfruit chosen is old breadfruit, then it is peeled to obtain the skin of the breadfruit and washed with clean water

to remove any remaining dirt that is still attached. The skin of the breadfruit is cut into pieces and dried in the sun. The dried skin of the breadfruit was ground using a blender and sieved using a 40 mesh sieve.

#### 2. Pectin isolation and purification process

##### a) Addition of hydrochloric acid (HCl)

Weigh 150 grams of breadfruit peel powder, put it in an Erlenmeyer flask then add 1 L of water then add 0.1 N HCl solution and measure using a pH meter to pH 1.5. After that, insert a magnetic stirrer and heat it on a hotplate at a temperature of 80 °C for 150 minutes and stir at a speed of 600 rpm. The extraction results are then filtered while hot using a 200 mesh filter cloth while hot. The filtrate obtained was left to cool at room temperature (25 °C) [7].

##### b) Pectin deposition

This is done by adding acid ethanol to the filtrate with a ratio of 1: 1.5 (450 ml: 650 ml) acid ethanol and leaving it for 24 hours, then the pectin precipitate formed is filtered using Whatman filter paper no. 41 [7].

##### c) Pectin washing and pectin drying

The precipitate obtained from the previous process was washed using 96% ethanol until clear, then dried at 40 °C for 8 hours using an oven. The pectin has been ground and sifted using a no. 40 mesh sieve [7].

$$\text{Pectin yield (\%)} = \frac{\text{pectin weight (g)}}{\text{initial powder (g)}} \times 100\%$$

#### 2) Pectin Characteristics Test

##### 1. Determination of pectin equivalent weight

The equivalent weight was determined by weighing 0.5 g of the pectin obtained and then placing it in a 250 ml Erlenmeyer and moistening it with 5 ml of ethanol. As much as 1 gram of NaCl was added to it to sharpen the end point of the titration. 100 ml of aquadest and 6 drops of phenolphthalein indicator were added. The mixture is then stirred quickly to ensure that all the pectin substances have dissolved and there are no lumps attached to the walls of the Erlenmeyer. The titration was carried out slowly with 0.1 N NaOH titrant until the color of the mixture changed to pink and remained so for approximately 30 seconds. The solution is then neutralized in order to determine methoxyl levels [5].

$$\text{Equivalent weight} = \frac{\text{pectin weight}}{V_{\text{NaOH}} \times N_{\text{NaOH}}}$$

##### 2. Determination of methoxyl pectin content

The neutral solution from which the equivalent weight was determined was added to 25 mL of 0.25 N NaOH, then shaken and allowed to stand for 30 minutes at room temperature under cover. Next, 25 ml of HCl was added. 25 N and titrated with 0.1 N NaOH with 1% phenolphthalein indicator to the end point as in determining the equivalent weight of pectin [5].

$$\text{Methoxyl levels (\%)} = \frac{V_{\text{NaOH}} \times N_{\text{NaOH}}}{\text{Pectin weight (mg)}} \times 100\%$$

### 3. Galacturonate levels

Galacturonate levels are calculated from mEq (milliequivalents) of NaOH obtained from BE and methoxyl content [5].

$$\text{Galacturonate levels (\%)} = \frac{\text{mEq (equivalent weight)} + \text{mEq methoxyl} \times 176}{\text{Pectin weight (mg)}} \times 100\%$$

### 3) Capsule Shell Formulation

**Table 1.** Formula for capsule shell preparations from breadfruit peel pectin.

Material	Function	Concentration % <sup>b/v</sup>		
		F1	F2	F3
Breadfruit Peel Pectin	Main Components	0,3	0,6	0,9
Carrageenan	Gelling Agent	4	4	4
Sorbitol	Plasticizer	0,1	0,1	0,1
Aquadest	Solvent	Ad 100	Ad 100	Ad 100

#### Making Capsule Shells

Breadfruit peel pectin is dissolved in distilled water to form a colloidal solution at a temperature of 60 °C, then the carrageenan is dissolved in distilled water until it forms a colloid at a temperature of 60 °C, then the pectin and carrageenan solutions are mixed and sorbitol is added, then heated at a temperature of 80 °C and stirred using a magnetic stirrer until the solution is homogeneous and thick so that a film sheet is formed, after that the capsule shell is molded at a temperature of 50 °C, then removed and let stand 5 minutes. Then dried in the oven at 55 °C for approximately 3 hours until the capsule shell is dry and released from the mold

#### 4) Capsule Shell Evaluation

##### 1. Organoleptic

Organoleptic evaluation is a physical test of the preparation which includes color, odor and texture [8].

##### 2. Test specific analysis

##### a) Test the uniformity of capsule shell weight

20 capsule shells were weighed, then weighed one by one and then calculated the average capsule shell weight, standard deviation and relative standard deviation  $\leq 2\%$  [9].

##### b) Capsule shell length and diameter test.

Length measurements were carried out on intact capsule shell using a caliper and diameter measurements were carried out on the cap and body using a caliper [10].

##### 3. Test disintegration time

A total of 6 capsules, put in each tube in the basket, under which there is a 10 mesh steel gauze. Air media with a temperature of 37 °C is used. Observe the capsule, all capsules must be destroyed, except for part of the capsule shell. If 1 or 2 capsules are not completely destroyed, the test is repeated with 12 other capsules, no less than 16 of the 18 capsules tested are completely destroyed. disintegration time under 15 minutes.

## 3. Results

From the isolation and purification of breadfruit (*Artocarpus altilis*) peel pectin, 10.18 grams of pectin weight was obtained and the yield was 6.78%. The dry pectin obtained is yellowish brown in color.

**Table 2.** Evaluation results of pectin characterization obtained.

	Concentration % <sup>b/v</sup>			Parameter
	Equivalent weight (mg)	Methoxyl levels (%)	Galacturonate levels (%)	
F1	3.168,5	3,26	96,27	600-800 mg (equivalent weight)
F2	3.168,5	3,26	96,27	>7,12% (methoxyl)
F3	3.168,5	3,26	96,27	Min 30% (Galacturonate)
Mean $\pm$ SD	3.168,5 $\pm$ 0	3,26 $\pm$ 0	96,27 $\pm$ 0	

**Table 3.** Results of organoleptic evaluation of capsule shells.

Formula	Color	Texture	Odor
F1	white	Not too hard, not too plastic	No odoris
F2	light brown	Slightly hard, plastic	No odoris
F3	sepia	Hard, plastic, a little stiff	No odoris

**Table 4.** Evaluation results of capsule shell disintegration time.

Formula	Disintegration	Disintegration Time Requirements
F1	11 minutes 43 seconds	Not >15 minutes (Pharmacopoeia Indonesian edition V)
F2	12 minutes 53 seconds	
F3	14 minutes 21 seconds	

**Table 5.** Evaluation results of capsule shell specifications.

Parameter	Aparatus	F1	F2	F3	Standard Medisca (mm)
Total capsule length (mm)	Vernier calipers	21,5 mm	22,07 mm	21,25 mm	19,4 ± 0,3
Body diameter (mm)	Vernier calipers	7,25 mm	7,125 mm	7,25 mm	6,59 ± 0,261
Lid diameter (mm)	Vernier calipers	7,5 mm	7,5 mm	7,5 mm	6,91 ± 0,272

**Table 6.** Evaluation results of capsule shell weight.

No.	F1	F2	F3	Parameter Standard Medisca
1	70	70	80	79 mg ± 5
2	60	70	80	
3	70	70	80	
4	60	70	80	
5	70	70	80	
6	60	70	80	
7	60	70	80	
8	70	70	80	
9	70	70	80	
10	70	70	80	
Mean ± SD	66 ± 4,9	70 ± 0	80 ± 0	
%RSD	7,4%	0%	0%	

## 4. Discussion

Capsule shells are made with pectin obtained from the skin of ripe breadfruit because ripe fruit contains protopectin which breaks down into pectin and the pectin content is high [11].

In the pectin isolation process, it is carried out under acidic conditions using HCl solvent so that changes in the compound can occur, namely from protopectin to pectin due to heating [12]. From the isolation and purification of breadfruit (*Artocarpus altilis*) peel pectin, 10.18 grams of pectin weight was obtained and the yield was 6.78%.

The dry pectin obtained is yellowish brown in color. Based on the Indonesian Pharmacopoeia V edition (2014), pectin is described as coarse or fine powder, yellowish white in color, almost odorless and has a mucilage taste. According to the Food Chemical Codex (1996), the description of pectin is in the form of coarse to fine powder; yellowish white, gray or brownish in color.

The equivalent weight is the content of free galacturonic acid groups that are not esterified in the pectin molecular chain, the equivalent weight is based on the saponification reaction of the carboxyl group by NaOH where the equivalent weight is inversely proportional to the volume of NaOH used to react with the carboxyl group [13].

The equivalent weight obtained was 3,168.5 mg (Table 2). Based on IPPA standards (2003), the equivalent weight of pectin is between 600-800 mg, so the results obtained do not meet existing standards. However, in previous research, testing the characteristics of breadfruit pectin carried out by Anwar, et al (2022) resulted in an equivalent weight of 3,123.34 mg. This is because the molecular weight of pectin depends on the growing area, weather, soil elements, soil pests, type of plant, quality of raw materials, extraction method and treatment in the extraction process. It is very likely that the thing that influences the equivalent weight value is the nature of the pectin extraction itself, as well as the process. extraction carried out [14].

In the identification test, methoxyl levels were obtained amounted to 3.26%, (Table 2, Figure 1) the results obtained included low methoxyl levels. Based on IPPA (2003), low methoxyl levels range from 2.12-7.12%. The low methoxyl levels are thought to be caused by an increase in non-pectate compounds in the cell walls which are also dissolved during the extraction process. The low methoxyl content obtained is more profitable because pectin can be used as an adsorbent so it is produced without a demethylation process.

Pectin with low methoxyl content in gel formation is called gelation with ions, where the ion that is mostly used is  $\text{Ca}^{2+}$  [15]. The meeting zone in LMP (low methoxyl pectin) is formed due to calcium cross-linking between the free carboxyl groups. The carboxyl groups can be aggregated to form a hydrogel that is stiff and resistant to acidic pH and will swell and dissolve at neutral pH [16].

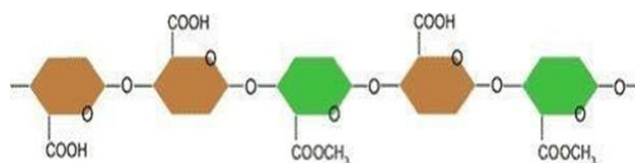


Figure 1. Pectin molecules with low methoxyl content.

The lower the DE of LMP, the easier the ability to form gel. The characteristics of the gel formed are very sensitive to the presence of  $\text{Ca}^{2+}$  ions, the lower the  $\text{Ca}^{2+}$  ions used, the gel formed will have lower elasticity and gel strength than using  $\text{Ca}^{2+}$  ions at the optimum concentration [15].

In the identification test, the galacturonate level was found to be 96.27% (Table 2). Based on IPPA (2003), the galacturonate content is at least 30%, so the galacturonate content still meets the specified pectin quality requirements. Galacturonate levels play an important role in determining the functional properties of pectin solutions and influence the structure and texture of the pectin gel formed [17]. The higher the galacturonate, the higher the pectin quality.

Capsule shells are made by dissolving breadfruit peel pectin with distilled water at a temperature of 60 °C to form a gel solution. Pectin is used as a capsule shell formula because pectin functions to form polymers, the mechanical properties of capsule shells and can form gel [18]. Gel formation occurs through hydrogen bonds between free carboxyl groups and hydroxyl groups [19]. Carrageenan is also dissolved in distilled water at a temperature of 60 °C until a colloidal solution is formed. Carrageenan functions as a source of edible film material to form capsule shells [20], as a strong thickener and gel former and at the same time increases the viscosity of the solution which is widely used in making capsule shells. Next, the pectin and carrageenan are mixed and sorbitol is added and then heated at a temperature of 80 °C. This heating aims to re-dissolve the carrageenan, pectin and sorbitol until the material is homogeneous and forms a colloidal solution. The sorbitol in this formula functions as a plasticizer to make hard capsules that are not too stiff and can be taken from the mold because sorbitol has higher tensile strength and eloquence values [9].

Then printing is carried out by dipping the printing tool into the mixture while the mold is rotated, dipped for 4 cm, printing is carried out at a temperature of 50 °C so that the film sheet formed does not harden quickly, then the mold is lifted and placed in an inverted position, after a few minutes the capsule is pulled up about 3 cm The purpose of pulling is to make it easier for the capsule to be removed from the mold when dry, then dried in the oven at 55 °C for 3 hours, to remove the water content in the capsule shell.

The pectin concentration used in this study was for F1 (0.3%), F2 (0.6%), and F3 (0.9%) with the addition of 4 grams of carrageenan concentration in each formula, 0.1 grams of sorbitol. and distilled water ad 100 ml. Carrageenan is used to increase the strength of the gel and add weight, sorbitol is used as a plasticizer so that the gel is not stiff and easy to mold [9].





Figure 2. F1; F3; F2.

In the formula 1 experiment, the layer was thin and the shell was not too hard and the color was clear white, while the capsule shell in formula 2 produced a thick film layer and the capsule shell was slightly hard and the color was light brown, plastic and formula 3 produced a thick film layer. Capsules are hard and dark brown in color. The pectin concentration in each formula affects the coating and gel of the capsule shell mixture. The color of each capsule shell formulation is different because the pectin concentration in each formula is different and the texture of each formula is influenced by the added carrageenan and sorbitol base [21].

In the organoleptic test, the results obtained (Table 3) showed that the capsule shell was white to dark brown because it was influenced by different pectin concentrations. The texture is influenced by carrageenan and sorbitol, the use of larger carrageenan can cause the total amount of dissolved solids to increase so that the film thickness increases [10], while in the disintegration time test the results were obtained in Table 4. There are differences in the disintegration time of each formula, this is because it is influenced by different pectin concentrations. According to Suptijah (2012) who stated that the capsule disintegration time will be longer along with the thickness of the capsule shell, the thicker the capsule shell, the longer it will take to disintegrate. Apart from that, it turns out that additional substances also influence the disintegration time, such as the addition of carrageenan as This additional substance is caused by the chemical structure of carrageenan itself.

Characterization and testing of capsule shells can use 3 capsule shells and this is based on the Indonesian Pharmacopoeia edition VI (Ministry of Health of the Republic of Indonesia, 2020). In the capsule shell specification test, the results obtained can be seen in Table 5, including total length, body diameter, diameter of the capsule shell lid which were measured using a caliper. The capsule shell weight test aims to determine the thickness of the capsule shell, the thicker the capsule shell, the greater the weight [8]. If we look at the research results according to the Medisca reference, the capsule shell requirements for total length, body diameter and cap diameter do not meet the requirements, but they do if we refer to previous research in Suparman's research (2019) which uses the PT Kapsulindo Standard with specification require-

ments for capsule shells. The total length of the capsule is 22.05 mm, the body diameter is 7.23 mm, and the cap diameter is 7.65 mm. All of this data meets the requirements of PT Kapsulindo.

In terms of capsule shell weight, the results obtained can be seen in Table 6. From these results, capsule shells that have good weight diversity are found in F2 and F3 due to the relative standard deviation (RSD%)  $\leq 2\%$ , but in F1 the weight diversity is not good due to the results. The measurement of the weight of the capsule shells is very different between the capsule shells so that the resulting RSD value is above 2%. The uniformity of capsule shell weight is influenced by the weight of all empty capsule shells being weighed. Different capsule shell weights can be influenced by pectin and the drying process because pectin functions to form polymer films which can increase the mechanical properties of the capsule shell [19].

In this study, methoxyl levels were produced, including low levels of methoxyl, which affected the properties of pectin gel formation and affected the weight of the capsule shell, then during the drying process the weight of the water contained was lost in the capsule shell, so it also affected the weight of the capsule shell.

The thickness of the capsule shell is influenced by the dipping process and irregular mold rotation which can produce uneven capsule shell thickness, as well as the formation of the resulting film sheet and the manual manufacturing process can also produce different thicknesses [10]. The length of the capsule shell is different due to manual cutting of the capsule shell.

Pectin can be used as a substitute for gelatin in capsule shell formulations because pectin meets the characteristics and when formulated forms polymerization with carrageenan, as well as the presence of sorbitol as a plasticizer. Based on the characteristics of the weight of the resulting capsule shell, it meets the Medisca standards, however, the capsule shell requirements such as the organoleptic test in F3 are fulfilled and the specification test does not meet the Medisca requirements and the disintegration time meets the requirements according to the Indonesian Pharmacopoeia Edition V.

Capsule preparations are very effective because they can mask the taste and odor of the drug, are easier to swallow, are quite stable in storage, and can be filled with single ingredients or a combination of other medicinal ingredients.

## 5. Conclusions

Pectin from breadfruit peels (*Artocarpus altilis* (Park) Fosberg) can be used as raw material for making capsule shells. Capsule shell characteristics from breadfruit (*Artocarpus altilis* (Park) Fosberg) rind pectin produced by the weight of the capsule shell meet the Medisca characteristics in F3 but for length, body diameter and lid diameter in F1, F2 F3 do not meet the Medisca standards and disintegration time already meets the requirements of the Indonesian Pharma-

copoeia edition V.

## Abbreviations

LMP	Low Methoxyl Pectin
DE	Degree of Esterification
IPPA	International Pectins Producers Association
F1	Formula 1
F2	Formula 2
F3	Formula 3

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## Author Contributions

**Rosiana Rizal:** Conceptualization, Methodology, Resources, Writing – review & editing

**Nofrizal Nofrizal:** Data curation, Methodology, Visualization

**Leadly Permanda Bakri:** Formal Analysis, Investigation, Validation, Writing – original draft, Writing – review & editing

## Conflicts of Interest

The authors declare no conflicts of interest.

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