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Plant-based Capsule Shell from Pectin and Glucomannan

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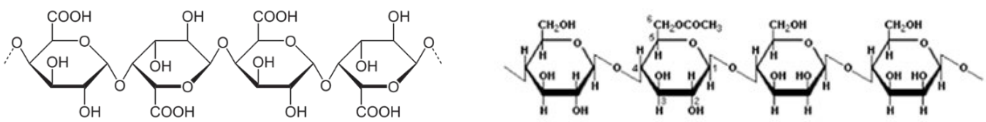
Capsules consist of a drug in a soluble hard or soft Shell. Capsule shells are generally made of gelatin which is made from pork skin and bones. This study aims to produce plant-based capsule shells using pectin and glucomannan. The composition used in the study was pectin to glucomannan ratio of 1:1, 1:2, 1:3, 1:4, 1:5. The results showed that the fastest disintegration time in the water was obtained at a composition ratio of 1:1 with a disintegration time of 24 min 18 s. The fastest dissolution time in acid was obtained at a composition ratio of 1:5 with a dissolution time of 2 min 52 s. The increase in glucomannan content affects the tensile strength of the material. Increasing glucomannan content increased the tensile strength so that the capsule shell becomes stronger. The increase in pectin content affects the modulus of elasticity. Increasing the pectin content would reduce the modulus elasticity and therefore the capsule shell becomes more elastic.

1. Introduction

Drug delivery system refers to the delivery of a pharmaceutical compound to its target site (Faridah and Susanti, 2018). Capsules are the most used solid drug dosage forms due to their convenience in covering the medicine's smell and taste (Rosmalasari, 2018). Generally, capsule shells are made of a material called gelatin, mostly based on pork products. (Faridah and Susanti, 2018) Over time, researchers began to study the potential plants alternative to replace gelatin, such as hydroxypropyl methylcellulose (HPMC) and other plant materials such as starch (Rosmalasari, 2018). One of the possible materials is pectin, pectin is a polymer of D-galacturonic acid linked by a β-1,4 glycosidic bond as shown in Figure 1 (a). Some of the carboxyl groups in the polymer undergo esterification with methyl to become methoxyl groups (Hanum et al., 2012). Pectin is a polymer compound that can form a gel, bind water, and thicken liquids. These properties cause pectin to be often used in the manufacture of jelly and various food industries (Sulihono et al., 2012). Pectin is a gelling agent that has good adhesive properties to form gels and make capsules (Amin and Alam, 2020). Pectin is present vastly in plants, including durian peels, banana peels, and green grass jelly (Nugroho, 2013). Each plant has different content of pectin (Sulihono, 2012). Moia (2018) research has successfully obtained high-quality pectin (with high purity, high content of oils, and high presence of piceatannol [0.05-1.3 μg/mL]) from passion fruit.



(a) (b)

Figure 1: Illustration of (a) structure of pectin compounds, and (b) structure of glucomannan compounds

Glucomannan is a polysaccharide of the hemicellulose type consisting of glucose, galactose, and mannose chains. The bond whose main chain is composed of linear bonds β-1,4-D-glucose, and D-mannose while the branch is galactose as shown in Figure 1 (b). Its D-mannose content is ± 67 % and D-glucose is ± 33 % (Anindita et al., 2016). Glucomannan has a unique property, which is soluble in water, can form gels and expand, films (transparent), sticky, can melt and precipitate (Rahmawati, 2018). Based on its distinctive properties, glucomannan solution can be used in the pharmaceutical industry as a binder in capsule manufacture

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(Kurniawan and Putri, 2016). The objective of this experiment was to utilize these unique properties of pectin and glucomannan which may be a possible capsule shell substitution made from plant materials that comply with the standards of Kapsulindo Nusantara, a pharmaceutical manufacturer in Indonesia, and Indonesian Pharmacopoeia.

2. Methods

The materials used were glucomannan flour (from a local chemical supplier), pectin flour (by CP Kelco), distilled water, aluminum foil, and Hydrochloric acid (HCl). The equipment used were ovens, stoves, capsule mold (dipping pens) size 000; 22.1 mm in length and 9.5 mm in diameter capsule body and 22.1 mm in length and 9.5 mm in diameter capsule cap, mechanical stirrers, thermometers, cutlery, glass boxes, and other glassware.

2.1 Preparation of mixed gel of pectin and glucomannan

The research method used in preparing the mixed gel in this study combined the methods used by Rosmalasari (2018) and Karimah (2016). A mixed gel of pectin and glucomannan was prepared by dissolving a mixture of pectin and glucomannan flour in various compositions (1:1, 1:2, 1:3, 1:4, and 1:5) with a total weight of 16 g of pectin flour mixture and glucomannan. The mixture of pectin and glucomannan flour was then dissolved in 384 mL of distilled water with continuous stirring for 2 h at 70 °C and a stirring speed of 700 rpm. The gel formed is molded into a capsule shell and a film layer.

2.2 Making capsule shells

The gel mixture of pectin and glucomannan formed was poured into a glass box. The capsule body mold with the dimensions of the capsule shell size 000 was dipped into the gel mixture, then flattened and heated in an oven at 50 °C. The exact process was carried out with a size 000 capsule cap mold. After all the molds were dipped and put in the oven, the gel mixture was reheated until it melted. The capsule shell that was heated in the oven for 20 min was dipped again in the gel mixture and then reheated at a temperature of 50 °C until dry. The dry capsule shells were removed from the mold for testing.

2.3 Making film layers

The gel mixture of pectin and the formed glucomannan was poured as much as 200 mL into an acrylic mold measuring 20 x 20 cm. The acrylic mold that has been poured with gel was then heated in an oven at 50 °C until it dries. The dried film layer was detached from the mold for testing.

2.4 Capsule shell specifications

The physical specifications of the capsule shells tested included the overall weight of the capsule, the length of each body and capsule cover, thickness, and the color and smell of the capsule shell. The overall weight of the capsule was weighed using an analytical balance. The thickness of the capsule shell was tested by Thickness Gauge Tester YG 1410. The length of the capsule body and cap was measured using a ruler. The color and odor of the capsule shells were tested organoleptically.

2.5 Disintegration test in water

The method used refers to the method used by Rosmalasari (2018). The capsule shells were put in 100mL water at 37 °C, then the water was stirred along with the capsule shells and the time for the capsule shells to dissolve was recorded.

2.6 Dissolution test in acid solution

The acidic solution was prepared from technical HCl to imitate the conditions in the stomach. In a method used by Rosmalasari (2018), 1 M HCl solution was prepared by adding 8.29 mL of 37 % technical HCl solution, and distilled water was added to 100 mL volumetric flask until the volume reached 100 mL, and stirred until homogeneous. The capsule shells, which had been filled with dye powder, were put into a container containing 1 M HCl solution. The time was observed for the coloring powder to come out of the capsule shell.

2.7 Mechanical properties test

The formed film layers were tested for their tensile strength and elongation. According to Maulana (2010), the tests were carried out by applying a load on two sides of the film with a certain cross-sectional area in opposite directions. The magnitude of the force before the film breaks was recorded to obtain the tensile strength value, and the increase in length after the test was recorded to obtain the elongation value. From the results of the tensile test and elongation test produced, it can be calculated to obtain the value of the modulus of elasticity with Eq (1), namely the value of tensile strength (σ) in Newton (N) divided by elongation (ε) in percentage (%).

$$E = \frac{\sigma}{\varepsilon} \quad (1)$$

3. Results and discussion

3.1 Composition of pectin and glucomannan

Glucomannan belongs to the group of polysaccharides which are polymers with many hydroxyl groups and generally interact strongly with water. All hydrocolloids that interact with water will reduce diffusion and stabilize their state (Rohmalasari, 2018). In the composition variation, the glucomannan flour was always more or equal to the pectin flour in weight. Siswanti (2008) research stated that the glucomannan concentration affects the glucomannan film thickness, thus increasing the glucomannan concentration tends to increase the film thickness thereby tensile strength. Therefore, in this experiment, higher glucomannan concentration was done to avoid thin film thickness which will be difficult to mold into capsule shells. According to Rahmawati (2018), the viscosity of glucomannan increase as the concentration increases, where the glucomannan flour gel solution was 1.5 to 21.5 times more viscous than the gelatin solution with the same concentration.

3.2 Making capsule shells

The capsule shells formed were homogeneous and in accordance with the capsule, the mold used, which was a 000 sized capsule mold with the dimensions of the capsule body being 22.1 mm in length and 9.5 mm in diameter, while the dimensions of the cap are 12.9 mm in length and 9.9 mm in diameter.

3.3 Physical specification test for capsule shells

The things observed in this specific test are weight thickness, body length, cap length, color, and odor. The test results obtained are shown in Table 1:

Table 1: Capsule shell specifications

Evaluation	Standard	Pectin and Glucomannan Ratio				
		1:1	1:2	1:3	1:4	1:5
Weight (mg)	170	136.7	163.3	226.7	166.7	186.7
Thickness (mm)	0.107	0.323	0.177	0.149	0.239	0.019
Capsule body length (mm)	22.1	22	22	22	22	22
Capsule cap length (mm)	12.9	13	13	13	13	13
Color		Dull transparent	Dull transparent	Dull transparent	Dull transparent	Dull transparent
Odor		Odorless	Odorless	Odorless	Odorless	Odorless

From the data generated, it was found that the capsule shell made from a gel mixture of pectin and glucomannan had a slightly transparent dull color and was odorless. Differences in composition variations do not show differences in color and odor specifications. Similar results were obtained from Rosmalasari (2018) experiment by conducting a fabrication of a halal hard capsule from a porang tube, where the capsule obtained was odorless and dull transparent in color. It was mentioned that the color obtained was the same color as the porang tube's and glucomannan gel's color. In the molding process, the capsule shells were made to have an excess length from the capsule's original size which will be then adjusted to the Kapsulindo Nusantara standard specifications, resulting in a uniform length of the capsule body and cap length. In an experiment done by Siswanti (2008), the increase in glucomannan composition will increase the thickness of the film. The thicker the capsule shell will add more weight to the capsule shell due to the increase in total dissolved solids in the capsule solution after the drying process. The inconsistent dipping in the molding process also affects the thickness of the capsule shell, resulting in uneven thickness (Suparman, 2019). In this experiment, the capsule shell weight and thickness from the test results show data inconsistencies from composition variations. This inconsistency was because when molding and drying the capsules take place, the mold was in an upright position, and the gel can still move positions since it is not dry yet; thus, there is inconsistency in the thickness and weight of the capsule shell. The capsule shell with a composition ratio of 1:4 pectin and glucomannan weighing 166.7 mg is closest to the commercial capsule shell size 000 weight from Kapsulindo Nusantara, which is 170 mg. The commercial capsule shell with a thickness of 0.107 mm is closest to the capsule shell with 1:3 pectin and glucomannan composition with a thickness of 0.149 mm.

3.4 Disintegration test in water

The disintegration test in water aims to ensure that the capsule is not damaged before it enters the mouth to avoid the bitter taste of the drug. The Pharmacopoeia provides a good disintegration time capsule in the water range of 15 to 30 min. The results of the capsule disintegration time in water are shown in Table 2:

Table 2: Disintegration time in water

Pectin and Glucomannan Ratio	Disintegration Time (min"s")			Standard Deviation
	Trial 1	Trial 2	Average	
1:1	120"48"	118"51"	119"49"	1.38
1:2	119"40"	108"11"	113"55"	8.12
1:3	114"27"	100"11"	107"19"	10.09
1:4	114"46"	106"32"	110"39"	5.83
1:5	117"25"	118"56"	118"0"	1.07

In Rosmalasari (2018) experiment, the thickness of the capsule shell affected the disintegration time in water, where the thicker the capsule shell resulted in a longer disintegration time in the water. The results of the disintegration time test in this experiment showed an irregular relation between the disintegration time and the composition ratio of pectin and glucomannan. This was due to differences in the surface thickness of the capsule shells produced during the molding process (Dirjen POM, Ministry of Health of the Republic of Indonesia, Indonesian Pharmacopoeia, 1979). The fastest disintegration time occurred in the capsule shell with a ratio of pectin and glucomannan of 1:3, which used 4 g of pectin and 12 g of glucomannan in 384 mL of distilled water composition, with a time of 107 min 19 s and a relatively high standard deviation value of 10.09. The most stable test results were in the capsule shell with a ratio of pectin and glucomannan 1:5, namely using 2.67 g of pectin and 13.35 g of glucomannan in 384 mL of distilled water which resulted in an average disintegration time of 118 min 10 s with a standard deviation value of only 1.07.

3.5 Dissolution test in acid solution

The time taken for the pectin and glucomannan capsule shell to dissolve in an acid solution is shown in Table 3 as follows:

Table 3: Dissolution time in acid solution

Pectin and Glucomannan Ratio	Dissolution Time (min"s")			Standard Deviation
	Trial 1	Trial 2	Average	
1:1	03"13"	03"33"	03"23"	0.23
1:2	04"06"	03"36"	03"51"	0.35
1:3	03"52"	04"12"	04"02"	0.11
1:4	03"17"	03"42"	03"30"	0.30
1:5	03"01"	02"42"	02"52"	0.22

The Ministry of Health of the Republic of Indonesia (1995) stipulates that the capsule shell must be able to distribute the drugs into the stomach in less than 5 min (Rahmawati, 2018). The research results show that the capsule shells tested for dissolution in the acid solution were able to release the dye powder into the environment in less than 5 min. The dissolution time indicates that the capsule shells made from a mixture of pectin and glucomannan pass the dissolution test in acidic solutions according to the 1995 regulations of the Indonesian Ministry of Health. The viscosity of the gel mixture of pectin and glucomannan significantly affects the dissolution of the capsule shells in acid solutions (Rosmalisa, 2018). In addition to viscosity, the capsule shell wall's thickness also affects the dissolving time of the capsule shell in the acid solution. Capsule shells that are thicker and have a flat surface will be more difficult to dissolve than capsule shells with uneven surfaces. Thus, some parts are thinner than others, and the permeability of one surface is large and causes drug solids to pass into the environment easily (Rahmawati, 2018). The results showed that the ratio of variations in the gel's composition of pectin and glucomannan did not significantly affect the dissolving time of the capsule shells in an acid solution.

3.6 Mechanical properties test

Mechanical tests carried out on mixed pectin and glucomannan films were tensile strength tests and elongation tests. The Modulus Young was calculated using Eq (1). The results are shown in Table 4:

Table 4: Tensile strength and elongation values

Pectin and Glucomannan ratio	Tensile Strength Value (N)	Elongation Value (%)	Modulus Young (Pa)
1:1	9.81	15.77	0.62
1:2	11.77	17.00	0.69
1:3	20.11	4.97	4.05
1:4	28.45	20.27	1.40
1:5	29.43	12.00	2.45

The results of the tensile strength test showed that the tensile strength values were uniform for each composition variation. The tensile strength value indicates that the greater the Glucomannan composition in the film material mixture, the stronger the tensile strength value of the film (Maulana, 2010). As the glucomannan concentration increases, the thickness of the film will thicken resulting in stronger tensile strength due to the stronger interactions between the glucomannan polymers (Siswanti, 2008). The highest tensile strength value was obtained in the composition ratio of pectin and glucomannan 1:5, which was 29.43 N. The lowest tensile strength value was obtained in the composition ratio of pectin and glucomannan 1:1, valued at 9.81 N. These results were also obtained from Maulana (2010) experiment, where the highest tensile strength produced by the biopolymer film with 0% tapioca content, with 100% glucomannan content, was 963.92 kPa, the lowest value was at the point of 50% tapioca content, with 50% glucomannan content of 320.51 KPa. The elongation value obtained from the test was irregular. The highest elongation value was obtained in the composition with a ratio of pectin and glucomannan 1:4, which was 20.267 %, while the lowest elongation value was obtained in the composition with a ratio of pectin and glucomannan 1:5, which was 12 %. The modulus young is the degree of stiffness of an elastic material, the greater the modulus young, the stiffer the material is, and vice versa (Maulana, 2010). The elastic modulus value obtained indicates an increase in the elastic modulus value for increasing glucomannan composition except for the pectin and glucomannan ratio of 1:3 compositions, which experienced an increase in the elastic modulus. Figure 2 shows an overall increase in modulus young with a decrease in pectin composition which is in accordance with Maulana (2010) experiment.

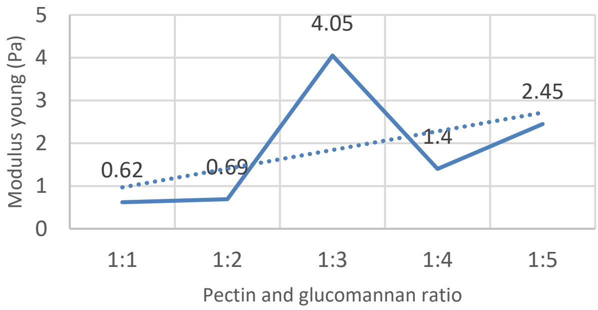

Figure 2: Modulus Young curve with various pectin and glucomannan compositions

The highest value of the elastic modulus was found in the composition ratio of pectin and glucomannan 1:3, which was 4.05 Pa, and the lowest value of the modulus of elasticity was found in the composition ratio of pectin and glucomannan 1:1, which was 0.62 Pa. The film becomes stiffer as the value of modulus young increases.

5. Conclusions

Based on the research on plant-based capsule shells made from a mixture of pectin and glucomannan, a mixture of pectin and glucomannan can become the primary material for making plant-based capsule shells to replace capsule shells made of gelatin which is widely available in the market. The capsule shells are 000 in size with body length dimensions of 22.1 and 12.9 mm. With a weight ranging from 130 to 190 mg, which is close to the theoretical weight of 170 mg. The resulting capsule shell is clear and odorless. The results of the dissolution test of the capsule shell in an acid solution, to resemble conditions in the stomach, showed results that met the pharmacopeia requirements, which said that the capsule shell must be able to release the drug it carries in less than 5 min. The results showed that capsule Shells with variations in composition ratio 1:5 gave the fastest test results with an average time of 2 min 52 s with a standard deviation of 0.22. The test results show an inconsistent dissolution time over the increase in glucomannan composition, thus it cannot be concluded that the ratio of glucomannan and pectin affects the dissolving time of capsule shells in an acid solution. However, in the disintegration time test in water, the capsule shells made from a mixture of pectin and glucomannan did not meet the disintegration time requirement of 15 to 30 min. Capsule shells made from a mixture of pectin and glucomannan took more than 90 min to be delivered in water. Based on the mechanical test, it can be concluded that the more glucomannan composition in the material mixture, the greater the tensile strength value of the material so that the material is robust. On the other hand, the more pectin composition in the material mixture, the smaller the elastic modulus value of the material, improving the material's elasticity. This study found that a mixture of pectin and glucomannan could be an alternative material for making hard capsule shells because the capsule shells made in this study approached the requirements of pharmacopeia capsule shells and the properties of commercial capsule shells. For further research, a precise experiment is needed in terms of molding which will largely affect the overall characteristics analysis of the capsule shells.

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