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Effect of Corn Starch and Glycerol Additions on the Characteristics of Capsule Shells from Seaweed Carrageenan

Aisha Ramadita, Hermawan Dwi Ariyanto*
Department of Industrial Technology, Vocational School, Universitas Diponegoro, Semarang, Indonesia
*Corresponding author: Hermawan Dwi Ariyanto | Email: hd.ariyanto@live.undip.ac.id

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Abstract: Capsule shells are generally made by gelatinee which from animal based. However, the capsule shell has issues related to the permissibility (halalness) and safety. Carrageenan has the potential as an essential ingredient of the capsule shell to replace gelatinee. The focus of this study was to determine the optimal formulation for the preparation of capsule shells. There were 9 different formulations to test the best quality by comparing it with commercial capsule shells. The samples were manually moulded with a dipping pen for 3 s at 45℃. The concentrations of corn starch were 1%, 2%, and 3% w/v and concentration glycerol were 2%, 3%, and 4% w/v. Weight uniformity, specifications, disintegration, and FTIR were analysed to determine the best quality of the capsule shell. The results showed that formula with concentration of 95% of distilled water, 2% w/v of carrageenan, 2% w/v of corn starch and 3% w/v of glycerol fit the standard of capsule shell.

Keywords: Capsule shell, carrageenan, corn starch, glycerol

1. INTRODUCTION

Availability of medicine is very important for the society because it can help in the process of curing a disease. Various drugs have been circulating in Indonesia in multiple forms, including tablets, capsules, ointments, creams, and syrups [1,2]. Capsules are considered simpler in production compared to the other forms [3]. Capsule shells are generally made from animal-based gelatinee. However, the capsule shell has issues related to the permissibility (halalness) and safety [4]. Therefore, one alternative to overcome this issue is to utilize plants found in Indonesia as the basic material for making capsule shells.

Several research related to capsule shells made from starch has previously been conducted by Suparman et al. (2019) [5] using cacao peel pectin with carrageenan. They reported three experiments were carried out with different formulas. The purpose of formula variation is to get a suitable capsule formula composition. In addition, there is also research by Lestari et al. (2021) [6], who made a capsule shell based on potato peel starch with honey as a plasticizer. They conducted five experiments with different formulations. The best formulation of potato peel starch capsule shell with the addition of plasticizer was formula with 2% wt. starch concentration.

Several research indicates the potential of various flora, including seaweed and corn, as essential components in capsule shell development. Seaweed with the type of *Euchemma cottoni* can produce polysaccharides in the form of carrageenan with demonstrated thickening, suspending, stabilizing, and emulsifying properties [7,8]. To obtain capsule shells with the optimal quality, starch plays a key role in stabilizing carrageenan for gel formation.

In manufacturing capsule shells, the used of raw materials is shifting away from animal-derived gelatine towards plant-based polysaccharides. These versatile biopolymers offer a sustainable and ethical alternative, with properties suitable for creating effective and safe capsules. Polysaccharides can be used to make capsule shells instead of gelatinee [9]. The types of polysaccharides are carrageenan, starch, alginate, and pectin [9]. Starch is composed of two polysaccharides, amylose (15-25%) and amylopectin (75-85%), which are stored in starch granules [10,11]. Corn starch and carrageenan, promising alternatives to gelatine, were explored for their suitability in capsule shell production.

Therefore, this study focused on characterizing their properties with glycerol and determining the optimal formulation of capsule shell.

2. MATERIALS AND METHODS

2.1. Materials

Carrageenan was obtained from Indoplant (Special Region of Yogyakarta), corn starch from Mitra Jaya Chemical (Bekasi, West Java), glycerol from CV. Indrasari (Semarang, Central Java), distilled water, HCl and vaseline. The materials used were a dipping pen, oven, magnetic stirrer, digital scale, stirrer glass, and beaker glass from Industrial Chemical Engineering Laboratory.

2.2. Methods

This study begins with preparing a capsule shell solution by heating distilled water in a beaker glass. Carrageenan and corn starch were added according to the variables. The solution was stirred until homogeneous, and glycerol was added, then stirred again until the mixture was perfectly mixed.

The samples were formed with a dipping pen that had been previously smeared with Vaseline so it would be easy to retrieve the results. Next, the dipping pen is dipped into the solution for 3 s at 45℃. Then, let the dipping pen set for 10 min before it is placed in the oven to heat up. The heating process uses a temperature of 50℃ and a time of 3 h [12]. The formed capsule shell is slowly released from the pin. After that, testing was carried out to determine its quality by comparing it with capsule shells generally made of gelatine [13].

Figure 1. Capsule shell molding process

2.3. Capsule Shell Formulation

This study is an experimental method in the laboratory with the formulation of capsule shells from carrageenan and corn starch. The concentrations of corn starch and glycerol were 1%, 2%, 3% w/v and 2%, 3%, and 4% w/v respectively. In addition, the formulation used in carrageenan, which is 2%, refers to previous research conducted by Lestari, Indah. etc. The formula of capsule shell was following in the Table 1.

Table 1. Formulation of capsule shell preparation

Formula	Distilled Water (mL)	Carrageenan (w/v)	Corn starch (w/v)	Glycerol (w/v)
1.	95	2%	1%	2%
2.	95	2%	1%	3%
3.	95	2%	1%	4%
4.	95	2%	2%	2%
5.	95	2%	2%	3%
6.	95	2%	2%	4%
7.	95	2%	3%	2%
8.	95	2%	3%	3%
9.	95	2%	3%	4%

2.3.1. Weight Uniformity Test

This test was conducted to determine the suitability of the weight of the capsules produced based on the requirements set by Fermakope Indonesia. Capsule weight uniformity is carried out by determining the difference in the weight of the contents of each capsule whose % deviation should not exceed the predetermined one [14].

2.3.2. Specifications Test

The specification tests observed are the length, diameter, thickness, weight, and volume. The specifications of length, diameter, and thickness were measured using a caliper. For the weight of the capsule shell, it was weighed using a digital balance. Volume measurement of the capsule shell was completed by filling the capsule shell with water.

2.3.3. Disintegration Test

The disintegration time test is required for the capsule to disintegrate in the specified medium. The disintegration time of each capsule must meet the requirements of the time specification as determined by a disintegrator tester. According to the Ministry of Health (2014) [15] in Pharmacopoeia, a suitable disintegration time on distilled water is 15 to 30 min and disintegration time on acid solution is less than 5 min [16,17].

2.3.4. Fourier Transform Infrared Test

One of the analyses used to determine the structure of the compound. Types of samples that can be analysed include solutions, pastes, powders, films, and so on [18,19]. FTIR is an infrared spectrum collection technique. The energy that has been absorbed by the sample at the frequency of infrared light will be recorded and then forwarded to the interferogram [20,21].

3. RESULTS AND DISCUSSION

3.1. Effect of corn starch and glycerol on the weight uniformity test of capsule shell

This test was conducted to determine the uniformity of the weight of the capsules produced by the requirements of Farmakope Indonesia. Capsule shells that have been made are then weighed on a digital balance sheet, and the weight of each capsule is recorded. From the results of these calculations can then be calculated % deviation. Figure 4 show the weight uniformity of capsule shell with the different concentration of corn starch and glycerol. The results showed that the capsule shells made from carrageenan and corn starch with the addition of glycerol had weights that deviated from the commercial standards set in formulas 1-4 and formula 6, which were more than 10%. This is caused by the thinness of the capsule shell formed so that it affects the capsule shell produced. While in formula 5, formula 7, formula 8, and formula 9 have complied with the set standards because the % deviation is still under the standard. The weight of the capsule shell is affected by the thickness of the capsule shell coating. It can be concluded that the thicker the capsule is, the more the weight the capsule increases.

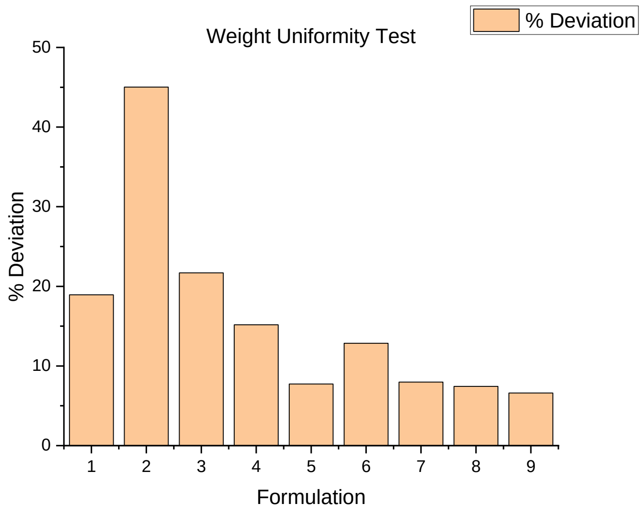

Figure 2. Weight Uniformity Test

3.2. Effect of corn starch and glycerol on the physical properties of capsule shell

The things observed in this specific test are length, diameter, thickness, weight, and volume are sown in Table 2.

Table 2. Specification test of capsule shell

Formula	Length (mm)		Diameter (mm)		Thickness (mm)	Weight	Volume
	Body	Cap	Body	Cap		(gr)	(mL)
1	16.1±0.100	7.21±0.006	7.91±0.010	7.65±0.006	0.11±0.010	0.045±0.005	0.43±0.026
2	16.24±0.069	8.5±0.010	7.7±0.006	7.74±0.006	0.13±0.006	0.02±0.002	0.41±0.000
3	16.18±0.010	9.82±0.015	7.71±0.006	7.62±0.015	0.21±0.010	0.04±0.002	0.38±0.015
4	16.41±0.010	9.67±0.058	7.42±0.029	7.7±0.006	0.1±0.010	0.056±0.004	0.49±0.012
5	18±0.006	10.22±0.025	7.52±0.029	7.71±0.012	0.11±0.010	0.103±0.006	0.58±0.104
6	17.6±0.090	10.57±0.064	7.5±0.000	7.81±0.006	0.21±0.010	0.065±0.048	0.53±0.021
7	17.54±0.036	10.07±0.058	7.53±0.026	7.8±0.000	0.31±0.015	0.1±0.002	0.53±0.036
8	17.67±0.113	10.52±0.040	7.61±0.017	7.71±0.012	0.33±0.006	0.107±0.003	0.28±0.072
9	17.67±0.029	10.52±0.035	7.7±0.006	7.52±0.029	0.31±0.010	0.118±0.002	0.19±0.040
SNI	18.87	11.23	7.37	7.7	0.11	0.099	0.65

3.2.1. Length test of capsule shell

The length of the capsule shell was recorded using a vernier caliper. The results of the length specification test can be seen in Table 2, which shows that Formula 1 has a body and cap length of 16.10 mm and 7.21 mm. Formula 2 is 16.24 mm and 8.50 mm. For formula 3 it is 16.18 mm and 9.82 mm. Formula 4 is 16.41 mm and 9.67 mm. Formula 5 is 18.00 mm and 10.22 mm. Formula 6 is 17.60 mm and 10.57 mm. Formula 7 is 17.54 mm and 10.07 mm. Formulas 8 and 9 amounted to 17.67 mm and 10.52 mm. In this research, the results of length measurements showed that formulation 5 was close to the length set by SNI.

3.2.2. Diameter test of capsule shell

Diameter size was also measured using a vernier caliper. The results of the diameter specification test can be seen in Table 2, which shows that Formula 1 has a body and cap diameter of 7.91 mm and 7.65 mm. Formula 2 is 7.70 mm and 7.74 mm. Formula 3 was 7.71 mm and 7.62 mm. Formula 4 to 7.42 mm and 7.70 mm. Formula 5 was 7.52 mm and 7.71 mm. Formula 6 was 7.50 mm and 7.81 mm. Formula 7 by 7.53 mm and 7.80 mm. Formula 8 by 7.61 mm and 7.71 mm. Formula 9 was 7.70 mm and 7.52 mm. Based on research, the results of length measurements showed that formulation 5 was close to the length set by SNI.

3.2.3. Thickness test of capsule shell

This test also used a vernier caliper to measure the capsule shell's thickness. The results of the thickness specification test can be seen in Table 2. The results showed that the formulas by SNI were formulas 1 and 5, which amounted to 0.11 mm. Formula 2 has a thickness of 0.13 mm, formula 3 of 0.21 mm, formula 4 of 0.10 mm, formula 6 of 0.21mm, formula 7 of 0.31 mm, formula 8 of 0.33 mm, and formula 9 of 0.31 mm. The thickness of the capsule shell will increase as the concentration of starch used increases. The variety of results on this thickness is because the research still uses a manual dipping system, so the results obtained are unequal. However, in contrast to the capsule shells on the market, the product is produced using a molding machine so that the thickness and weight can be uniform.

3.2.4. Weight test of capsule shell

The weight of capsule shells was measured using a digital balance and evaluated based on SNI. Table 2 show the results of this study. Formulas 1, 2, 3, 4, and 5 show values that are still below the predetermined standards. Which are 0.045 gr, 0.020 gr, 0.040 gr, and 0.056 gr. In the other formulations, the values are obtained almost close to the standard: formulas 5, 7, 8, and 9, with each weight of 0.103 gr, 0.100 gr, 0.107 gr, and 0.118 gr. It can be concluded that the more starch concentration is used, the weight produced will increase.

3.2.5. Volume test of capsule shell

The results of the volume specification test on the capsule shell can be seen in Table 2 from the different formulations; the volume results obtained were closest to the standard in Formula 5, which is 0.58 mL. Other formulations obtained values still below the predetermined standards, including formula 1 of 0.43 mL. Formula 2 amounted to 0.41 mL. Formula 3 amounted to 0.38 mL. Formula 4 amounted to 0.49 mL. Formulas 6 and 7 amounted to 0.53 mL. Formula 9 amounted to 0.19 mL. The volume produced in the study has a variety of values. This is because the concentration of starch used is different. The more starch concentration used will increase the thickness of the capsule shell, which causes less volume-produced.

3.3. Effect of corn starch and glycerol on the disintegration time test of capsule shell

A suitable disintegration time, according to the Indonesian Ministry of Health (1995) [22], ranges from 15-30 min in distilled water solution and 5 min in acidic solution (HCl). In this research, 9 formulations were made with different variables. The results of the measurement of the disintegration of the capsule shell are in Table 3.

Table 3. Disintegration Test

Formulation	Distilled Water (min)	HCl (min)
1	20.04±0.075	4.00±0.035
2	23.06±0.167	4.16±0.060
3	23.27±0.261	4.07±0.064
4	24.18±0.156	4.53±0.289
5	21.34±0.053	4.73±0.058
6	25.18±0.137	5.69±0.081
7	35.89±0.248	5.78±0.127
8	36.48±0.072	5.12±0.240
9	37.26±0.047	5.92±0.067
SNI	30.00	5.00

3.3.1. Disintegration test in distilled water

Based on the results of this study, the disintegration time required in Formula 1 was 20.04 min, Formula 2 was 23.06 min, Formula 3 was 23.27 min, Formula 4 was 24.18 min, Formula 5 was 21.34 min, Formula 6 was 25.18 min, Formula 7 was 35.89 min, Formula 8 was 36.48 min, and Formula 9 was 37.26 min. The disintegration of a capsule shell is influenced by the thickness of the capsule shell itself. The thicker the capsule shell, the longer the disintegration time. The value of disintegration time in samples 1 to 6 is still in accordance with the standards set by Farmakope, which is 15-30 min.

3.3.2. Disintegration test in acid solution

The disintegration time in HCl can be seen in Figure 5. Formula 1 was 4.00 min, Formula 2 was 4.16 min, Formula 3 was 4.07 min, Formula 4 was 4.53 min, Formula 5 was 5.22 min, Formula 6 was 5.69 min, Formula 7 was 5.78 min, Formula 8 was 5.12 min, and Formula 9 was 5.92 min. The value of the destruction time in samples 1 to 4 is still by the standards set by Farmakope, which is around 5 min. The research results show that the capsule shells tested for disintegration in the acid solution were able to release the dye powder into the environment in less than 5 min [23]. This is also influenced by the thickness of a sample, where the thicker the capsule shell, the longer the destruction time required. In addition, what affects the length of the destruction time is carrageenan. Carrageenan material will affect the destruction time of a capsule to be longer because the chemical structure of carrageenan has hydrophobic properties [24].

3.4. Fourier Transform Infrared Test of Capsule Shell

To identify the molecular functional groups contained in a sample, a tool such as Fourier Transform Infrared Spectroscopy (FTIR) can be used. From the results of the FTIR specta in this study (Figure 3), it can be seen that there is an O-H hydroxyl absorption at 3292.97 cm-1. There is also a C-H functional group at 2940.65 cm-1 absorption. The C≡C functional group is shown in the 2110.94 absorption spectrum. The absorption peak of 1640.25 cm-1 shows the presence of C=C functional groups. Then, there is also an O-H functional group at 1420.68 cm-1. Sulfate ester bond S=O indicates all types of carrageenan content in the sample, namely at 1367 cm-1 and 1147.75 cm-1. The analysis done on samples made from carrageenan and corn starch shows that the carrageenan obtained is kappa carrageenan. The model has already fit the standard so that it is suitable for use as a raw material for capsule shells.

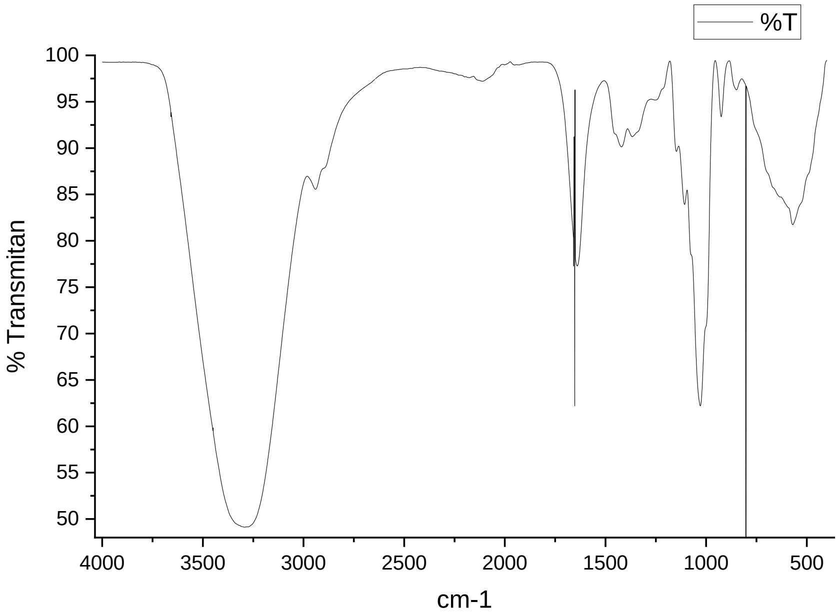

Figure 3. Fourier Transform Infrared Test

4. CONCLUSION

This study successfully fabricated capsule shells utilizing carrageenan and corn starch as primary components. Characterization and optimization of formulations containing carrageenan, corn starch, and glycerol resulted in formula 5 as the most suitable composition for capsule shell preparation, with 2% w/v of carrageenan, 2% w/v of corn starch, 3% w/v of glycerol, 95% wt. of distilled water.

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