



The Characterization of Capsule Shell from Acid-Hydrolyzed Palm Oil Starch

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ABSTRACT. Acid hydrolysis of palm trunk starch can increase the amylose content in starch, providing a stronger and more stable film. This study aims to obtain the best-modified starch concentration ratio with hydroxypropyl methylcellulose (HPMC) and identify their characteristics. Modifying palm trunk starch was conducted with an acetic buffer using CH_3COONa and CH_3COOH , decolorizing starch with activated carbon. Manufacturing a capsule shell was performed with weight ratio variations of modified starch and HPMC 1:1 (F1), 2:1 (F2), and 3:1 (F3). All formulations produced firm and elastic capsule shells. The capsule products were consistent in an average weight of F1 (0.10 g), F2 (0.11 g), and F3 (0.14 g). The average disintegration test results were F1 (10 minutes, 27.57 seconds), F2 (6 minutes, 47.06 seconds), and F3 (4 minutes, 34.24 seconds). Tensile strength results were F1 (2.147 MPa), F2 (2.565 MPa), and F3 (2.159 MPa). Fourier Transform Infrared (FTIR) results showed a vibration at a wavenumber of 1560 cm^{-1} corresponding to the characteristic fingerprint of the C–O vibration stretching in the capsule shell made from starch modified by HPMC. The concentration of modified starch affects the capsule shell's characteristics, showing that capsule shell F2 (2:1) has the best formulation.

INTRODUCTION

Palm Oil (*Elaeis guineensis*), a major commodity in Indonesia, provides many benefits, including starch found in the trunk (Bakewell-Stone, 2023; Pratama and Widodo, 2020). A single extraction from a 10 m long and 50 cm diameter trunk can yield up to 67% starch content (Cahyaningtyas *et al.*, 2019). In pharmaceuticals, this starch is used to produce capsule shells, typically made from gelatin or other polymers, to create strong and elastic films (Watson and Cogan, 2020).

Research carried out by Azizah (2023) and Gracia (2023) shows the best capsule shell formulation using *E. guineensis* starch at 5% concentration with 5% of hydroxypropyl methylcellulose (HPMC) and 4% of glycerin. Azizah *et al.* (2023) utilized palm oil starch as a raw material for capsule shells with the best concentration ratio with a combination of palm oil stem starch and HPMC (1:1). The disintegration time reported was 24 minutes, but this reported duration did not appropriate the Indonesian Pharmacopoeia standard, which requires to be 15 minutes or less. Mutia *et al.* (2022) show that modifying the *E. guineensis* starch using acid hydrolysis at pH 7 improves the starch content and may enhance the quality of the capsules.

Acid hydrolysis modification uses an acetate buffer prepared from sodium acetate and acetic acid (Villar *et al.*, 2017). This buffer solution is the simplest and most widely used method in food and non-food industries (Cahyaningtyas *et al.*, 2019). The mechanism of action is to break the glycosidic bonds in the amylopectin branching and increase the amylose content (Marseno *et al.*, 2022; Sjöo and Nilsson, 2017). This modification can reduce molecular weight, increase starch crystallinity, reduce viscosity, and increase solubility in warm water (Bambardekar, 2020; Kweon and Han, 2023). The novelty of this research is the modification of acid-hydrolyzed palm oil starch to produce a capsule shell that has a disintegrating time appropriate to the Indonesian Pharmacopoeia standard. Considering these backgrounds, this study aimed to determine the best formulation and analysis of the effect of modified *E. starch* concentration on capsule shell properties.

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RESEARCH METHODS

The materials used in this study are oil palm (*Elaeis guineensis* Jacq.) trunks obtained from PT. Gawi Makmur, Sungai Danau, Satui, Tanah Bumbu, Kalimantan Selatan, sodium acetate (CH_3COONa) p.a. Merck Nitrakimia (Yogyakarta), CH_3COOH p.a. (Nurra Gemilang, Malang), HPMC K100 (Merck Nitra Kimia, Yogyakarta), glycerine (SmartLab), and active carbon Iodine 100 (Carbonex).

Extraction of *E. guineensis* Starch

The *E. guineensis* used in this study were 20 – 25-year-old male trees, with 2 meters of sections taken from the top of the trunk. The pith of the trunk was crushed into sawdust, soaked in water at a 1:2 (%b/v) ratio, filtered using cloth, and settled for 24 hours. The wet starch formed at the bottom was washed with distilled water and dried in an oven at 50 °C for 3 hours. The dried starch was ground and sieved using a 50 mesh.

Modification of *E. guineensis* Starch by Acid Hydrolysis

The acid hydrolysis modification of *E. guineensis* starch was done by Cahyaningtyas *et al.* (2019) and Mutia *et al.* (2022) using acetate buffer at pH 7. The buffer solution was made by dissolving 44.52 g of sodium acetate in 50 mL of aquadest, added with glacial acetic acid to reach pH 7. Aquadest was added to the solution until the volume reached 1 L. *E. guineensis* starch was dissolved with the buffer solution at a 1:2 (%b/v) ratio, stirred, and heated on a hotplate at 40 °C until it thickened. The starch was dried at 50 °C using an oven, ground, and sieved using a 50-mesh sieve.

The Manufacture of Modified *E. guineensis* Starch Capsule Shell

The formulation used in this study was done by Azizah (2023) and Gracia (2023) and then developed by varying the concentration of modified *E. guineensis* starch with HPMC by 1:1, 2:1, 3:1, and 4:0, as presented in Table 1. HPMC was dissolved in one-third of the total formulation volume of aquadest at 70 °C then left for 30 minutes. The modified *E. guineensis* starch was dissolved in 10 mL of aquadest and decolorized by mixing it with activated carbon at a 1:0.1 (b/b) ratio for 10 seconds and filtered using filter paper. The filtered starch was added to the HPMC solution, and the excess of aquadest and glycerin was added. Stir the mixture using a magnetic hotplate stirrer at 40 °C and 200 – 400 rpm for 5 minutes or until the mixture thickens (Rizal *et al.*, 2023). Capsul molding was done using a cylindrical pin dipped 3 times, with a 3-second interval between dips. The capsule was left for 10 minutes at room temperature, then placed in an oven at 55 °C for 3 hours. The thoroughly dried capsule shells were carefully removed and stored in a desiccator (Rizal *et al.*, 2023).

Table 1. Formula of modified *E. guineensis* capsule shell.

Materials	Function	Amount of Materials Used			
		F1	F2	F3	Control
Modified <i>E. guineensis</i> starch	Film coating	1.25 g	2.50 g	3.75 g	5.00 g
HPMC	Gelling Agent	1.25 g	1.25 g	1.25 g	-
Glycerin	Plasticizer	1 mL	1 mL	1 mL	1 mL
Aquadest	Solvent	ad 25 mL	ad 25 mL	ad 25 mL	ad 25 mL

Characterization Test of Capsule Shell

Characterization tests included organoleptic test, capsule size (length, diameter, thickness, volume, and weight), disintegration test (Electrolab), Fourier Transformed Infrared (FTIR) (Bruker Alpha II), and Scanning Electron Microscopy (SEM) (Hitachi FlexSEM 1000).

RESULTS AND DISCUSSION

Modification of *E. guineensis* Starch by Acid Hydrolysis

The modified *E. guineensis* starch powder has a darker color compared to the starch powder before modification, as shown in Figure 1. This color difference occurs due to the presence of mono- and disaccharides in the starch undergoing heating. The modified *E. guineensis* starch in this study was heated twice, first at 40 °C when mixed with acetic acid using a hotplate and second at 50 °C during the drying process in the oven. The heating process results in the occurrence of caramelization reactions, including changes in the size of the monosaccharide ring, the breaking and re-forming of glycosidic bonds, dehydration, or the inclusion of double

bonds. These reactions lead to a darker color for the modified starch (Gullón *et al.*, 2016; Kocadağlı and Gökmen, 2019; Wardana and Yulia, 2018).



Figure 1. *E. guineensis* starch powder (a) before and (b) after acid-hydrolyzed modification.

Decolorization of Modified *E. guineensis* Starch

The decolorization results of the modified *E. guineensis* starch solution are shown in Figure 2. This process was solved with a better color—light yellow, clear, and odorless—when compared to the modified *E. guineensis* starch solution before decolorization, which had a cloudy, brownish color and the characteristic smell of starch. Activated carbon used in the process adsorbs dyes and impurities through the pores of the adsorbent, causing the final product to lose its color. This process is carried out to modify the appearance of the product and enhance its aesthetic value to increase consumer trust in the product (Hung *et al.*, 2022; Kan *et al.*, 2017).



Figure 2. *E. guineensis* starch (a) before and (b) after decolorization.

Characterization Test of Modified *E. guineensis* Capsule Shell

Capsule shells of modified *E. guineensis* starch and HPMC formula 1 (1:1), formula 2 (2:1), and formula 3 (3:1) were successfully obtained with good shape, firmness, and elasticity after passing the molding and drying process. There were slight differences in shape between the formulas. The F1 capsule has an uneven oval shape, the F2 has an even oval shape, and the F3 also has an even shape but will become mushy after storing at room temperature. The control formula produces a thin layer of capsule shell, making it difficult to separate from the molding medium without damaging it. Capsule shells in this study are shown in Figure 3.

The study examined the shape and properties of capsule shells manufactured by combining modified starch with HPMC. The control capsule shell, made only from modified starch, forms a thinner layer compared to the capsule shell made with the combination of modified starch and HPMC. It happens due to the ability of HPMC to increase the viscosity of the capsule shell through hydrogen bonding, which subsequently traps water and produces a more compact outcome (Joshi, 2011; Yang *et al.*, 2022). Further research is required to optimize the manufacturing procedures of this formula of capsule shells to adhere to industrial standards by paying attention to the dissolution procedure and appropriate drying time and temperature (Prakoso *et al.*, 2023).

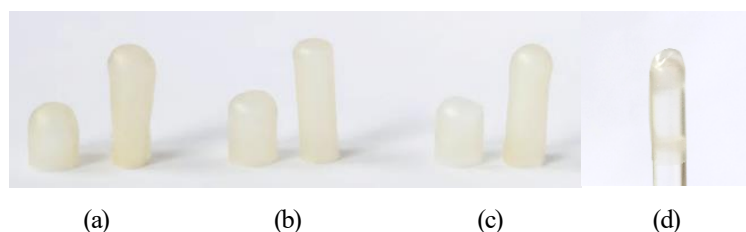






Figure 3. Modified *E. guineensis* capsule shells (a) F1, (b) F2, (c) F3, and (d) control.

Organoleptic Test

The organoleptic test is used to measure the shape, color, odor, and taste by relying on the five senses of the respondents to get accurate results (Dewi *et al.*, 2021). The results shown in Table 2 indicate that increasing the concentration of modified *E. guineensis* starch did not affect the odor and taste of the capsule shell, but affected the color, shape, and time required for the formula to thicken. Increasing the concentration of the modified *E. guineensis* starch causes the cream color of the capsule shell to fade and become more transparent. The clarity of the capsule color clarity is likely due to the presence of acetyl groups from the acid hydrolysis of modified *E. guineensis* starch, resulting in a higher clarity paste compared to unmodified *E. guineensis* starch (Sjöo and Nilsson, 2017; Teodoro *et al.*, 2015; Villar *et al.*, 2017).

The viscosity of capsule shell formulas (F1, F2, and F3) appeared visually identical during the molding process, but the duration for achieving varied. The time needed for the formulation to thicken decreased by increasing the concentration of modified *E. guineensis* starch at a constant temperature and stirring speed. The results of this study suggest that the modified starch contains more amylose than unmodified starch, leading to higher crystallinity and more intermolecular hydrogen bonds (Nisah, 2018). This contrasts with a previous study by Azizah (2023) on unmodified starch, where viscosity depended on the composition of HPMC used. Increasing starch content resulted in a less viscous solution and a softer capsule shell.

Table 2. The result of the organoleptic test.

Formula	Color	Odor	Taste	Shape	Form of the mixture
 F1 (1:1)	Cream	Odorless	Tasteless	Oval, un-even shape	Thick
 F2 (2:1)	Cream	Odorless	Tasteless	Oval, even shape	Thick
 F3 (3:1)	Cream	Odorless	Tasteless	Oval, mushy at room temperature	Thick
 Control (4:0)	Cream	Odorless	Tasteless	Oval, thin, soft	Not thick

Capsule Size Test

As shown in Table 3, capsule dimensions were measured using a caliper to determine length, diameter, and thickness, and the capsule volume was estimated by filling the capsule to the upper meniscus limit with water using a measuring pipette. All the capsules yielded results similar to the standard sizes, as the capsule shells were adjustable during production.

Table 3. The result of capsule size.

Parameters	Formula (Modified <i>E. guineensis</i> starch: HPMC)									Standard Capsule Size
	F1 (1:1)			F2 (2:1)			F3 (3:1)			
	I	II	III	I	II	III	I	II	III	
Cap Length (mm)	10.7	10.7	10.7	10.7	10.7	10.7	10.7	10.7	10.7	10.72
Body Length (mm)	18.4	18.4	18.4	18.4	18.4	18.4	18.4	18.4	18.4	18.44
Cap Diameter (mm)	7.6	7.6	7.6	7.6	7.6	7.6	7.6	7.6	7.6	7.65
Body Diameter (mm)	7.3	7.3	7.3	7.3	7.3	7.3	7.3	7.3	7.3	7.34
Cap Thickness (mm)	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.107
Body Thickness (mm)	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.107
Volume (mL)	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.68

The weight of each capsule was measured on an analytical balance. The overall weight of the capsule shells for all three formulations was close to falling within the standard weight range for capsule shells, with a range of $0.096 \pm 10\%$ grams, as can be seen in Table 4 (Zilhada *et al.*, 2022). Capsule shells in F3 weighted more when compared to F1 and F2. These results are due to variations in the added concentration of modified *E. guineensis* starch, leading to differences in the amount of dissolved starch in each formulation (Mutia *et al.*, 2022). It is also possible that the increased weight of the F3 capsule shell is a result of the non-uniform distribution of the solution on the molding surface, caused by the shorter viscosity time of the F3 formula compared to the other formulas. This variation in solution distribution could also be due to the manual and individual manufacturing process of capsule shells.

Table 4. The result of capsule weight.

Parameters	Formula (Modified <i>E. guineensis</i> starch: HPMC)									Standard Capsule Weight
	F1 (1:1)			F2 (2:1)			F3 (3:1)			
	I	II	III	I	II	III	I	II	III	
Weight (g)	0.11	0.11	0.10	0.11	0.11	0.11	0.15	0.14	0.15	0.096 ± 10%
Mean ± SD	0.10 ± 0.005			0.11 ± 0			0.14 ± 0.005			

Disintegration Test

The disintegration test determines how quickly a capsule breaks apart in human body fluids (Dewi *et al.*, 2021). It shows how the mechanical breakdown of the capsule affects its surface area for a quick release of active ingredients (Floryanzia *et al.*, 2022). The test is performed using a disintegration tester with water as the media at a temperature of $37^\circ\text{C} \pm 1^\circ\text{C}$. The tester basket is filled with different formulas of capsules, and the vertical motion is applied until all the parts of the capsule disintegrate through the basket (Rizal *et al.*, 2023). The test should take no more than 15 minutes, according to the Farmakope Indonesia edition VI (Farmakope Indonesia, 2020).

The disintegration test for all formulations meets the requirements set by Farmakope Indonesia edition VI, where Formula 1 had the longest disintegration time of all formulas. Starch is often used as a binder or disintegrant in such contexts, and its concentration determines the balance between cohesiveness and the ability to break apart when exposed to moisture or mechanical stress. Starch may not provide sufficient binding or swelling action at lower concentrations, leading to weak structural integrity. Therefore, starch can act as an effective disintegrant at the optimal concentration by absorbing water, swelling, and exerting pressure on the material's structure. This helps break down the material efficiently and ensures timely and consistent disintegration. The result can be seen in Table 5.

Table 5. The result of the disintegration test.

Replication	Duration Time (Minutes : Seconds : Milliseconds)		
	F1 (1:1)	F2 (2:1)	F3 (3:1)
1	10 : 20 : 23	06 : 41 : 17	04 : 15 : 25
2	10 : 27 : 20	06 : 48 : 24	04 : 57 : 41
3	10 : 36 : 07	06 : 51 : 38	04 : 30 : 05
Mean \pm SD	$10 : 27 : 57 \pm 0.005$	$06 : 47 : 06 \pm 0.003$	$04 : 34 : 24 \pm 0.014$

The disintegration time confirms the effectiveness of acid hydrolysis with an acetate buffer. Hydroxonium ions (H_3O^+) weaken the glycosidic bonds by attacking the high-energy oxygen atoms in the starch molecules, allowing water to bind and form new bonds. This destabilizes the hydroxyl groups, leading to the breakdown of amylopectin molecules into amylose molecules. As the degradation progresses, the amorphous part decreases, forming a new crystalline part. This fragmentation also increases the number of hydroxyl groups, making it easier for starch to dissolve in warm water. These combinations give the product firm physique while still dissolving easily in warm water (Bambardekar, 2020; Sjöö and Nilsson, 2017).

Tensile Strength Test

The tensile strength determines the ability of a sample to withstand a certain amount of applied force by maintaining the strength of the sample shortly before breaking (Tafa *et al.*, 2023). The test was performed using a set of equipment and applied load to both ends of the sample until it was cut in half (Kweon and Han, 2023). The

results of all capsule shell formulations shown in Table 6 meet the minimum standard tensile strength value for packaging preparations in the form of edible films based on the Japanese Industrial Standard (1997) of 0.392 MPa (Santoso and Atma, 2020). The tensile strength of capsule shells made of unmodified *E. guineensis* starch and HPMC with a ratio of 1:1 done by Gracia (2023) resulted in an average tensile strength of 0.976 MPa. The tensile strength results of capsule shells made of modified *E. guineensis* starch show a stronger and more elastic outcome due to the successful modification in starch (Mutia *et al.*, 2022).

These results show that increasing the concentration of modified *E. guineensis* starch in the capsule shell formulations can increase its tensile strength. This happens due to stronger hydrogen bonding between hydroxyl groups of modified starch and HPMC with increased concentrations. This leads to greater strength and elasticity. However, at specific concentrations, this interaction weakens, resulting in a decrease in tensile strength, as observed in the case of the F3 capsule shell. This may be attributed to the optimal ratio of modified starch and HPMC being reached at a concentration of 2:1, beyond which additional modified starch has no further impact on the structure and ingredient compatibility. Modified starch in F3 capsule shell formulation weakened the interaction between the molecular matrices, resulting in reduced tensile strength and heightened water absorbability (Frangopoulos *et al.*, 2023; Mutia *et al.*, 2022).

Table 6. The result of the tensile strength test.

Tensile Strength	Formula (Modified <i>E. guineensis</i> starch : HPMC)		
	F1 (1:1)	F2 (2:1)	F3 (3:1)
kgf/cm ²	21.898	26.161	22.025
MPa	2.147	2.565	2.159

Fourier Transformed Infrared (FTIR)

Fourier Transformed Infra-Red (FTIR) was used to identify the functional groups present in molecules by analyzing their interactions within specific fingerprint regions. The FTIR results of modified *E. guineensis* starch are shown in Figure 4.

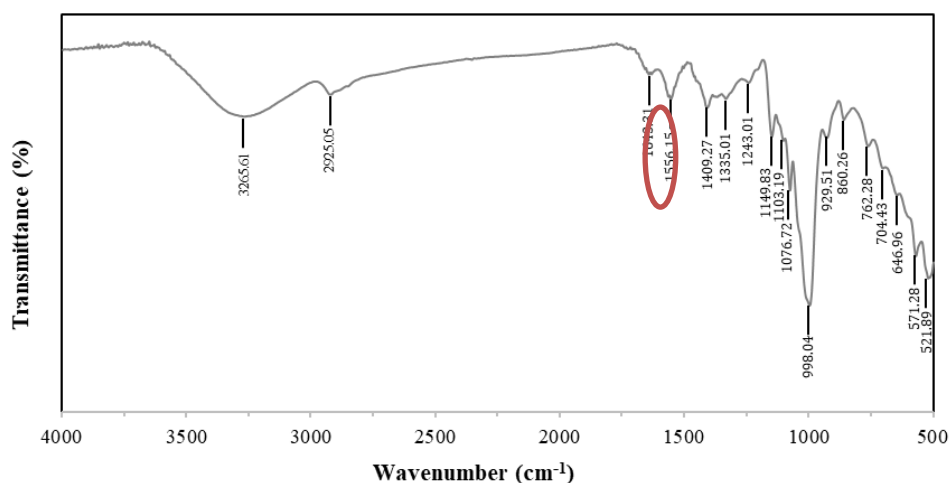


Figure 4. FTIR spectra of modified *E. guineensis* starch.

A study conducted by Gracia (2023) found similarities in the shape and functional groups of modified to unmodified *E. guineensis* starch. However, a significant difference was observed in the absorption peak at 1556.15 cm⁻¹, indicating the presence of R-COO groups in the modified starch. This finding aligns with the previous research by Mutia *et al.* (2022) and Sakeer *et al.* (2017), which also identified peaks with wavenumber 1560 and 1556 cm⁻¹ respectively (Gracia, 2023; Mutia *et al.*, 2022; Sakeer *et al.*, 2017). These peaks indicate the presence of acetate groups with weak and sharp spectra. The literature suggests that the acetate group appears in the wavenumber range of 1550-1620 cm⁻¹ for the asymmetric COO stretching group and 1300 – 1420 cm⁻¹ for the symmetric COO stretching group, while the carboxylic acid group appears in the 1700 – 1725 cm⁻¹ range for the C=O stretching group (Ibrahim *et al.*, 2005; Nandiyanto *et al.*, 2022).

The FTIR results of modified *E. guineensis* capsule shells are shown in Figure 5. The spectra obtained are similar in shape and functional groups to the unmodified starch capsule shell by Gracia (2023). Significant differences were also noticed in the peaks formed as a marker of the presence of acetate groups (R-COO) asymmetric stretching as found at wavenumber 1565.93 cm^{-1} , 1564.80 cm^{-1} , and 1564.49 cm^{-1} for F1, F2, and F3 respectively (Mutia *et al.*, 2022).

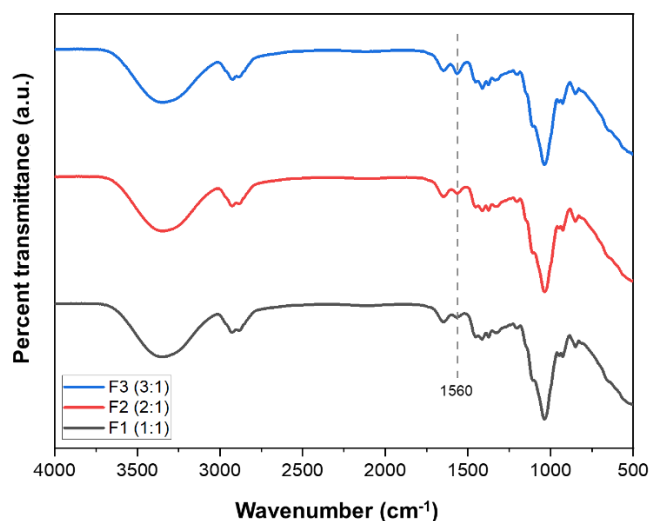


Figure 5. FTIR spectra of modified *E. guineensis* capsule shells.

Acetate groups are present in the modified starch after the successful acid hydrolysis using an acetate buffer solution. The process involves the hydronium ions breaking the glycosidic bonds in starch molecules and attracting water molecules, leading to the fragmentation of the molecules into smaller fragments. The reactive hydroxyl groups in starch are substituted by acetate groups through an addition-elimination mechanism (Kusumaningsih *et al.*, 2023; Nawaz *et al.*, 2020; Subroto *et al.*, 2023). The reaction can be seen in Figure 6.

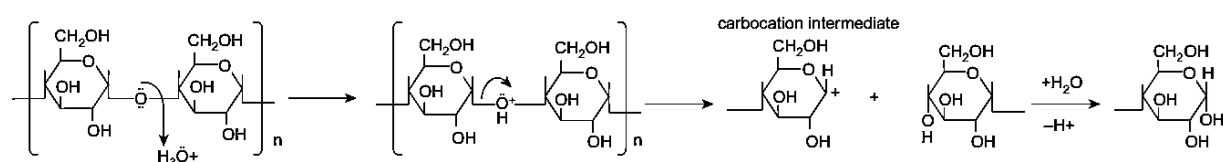


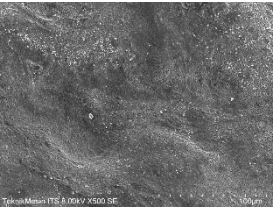
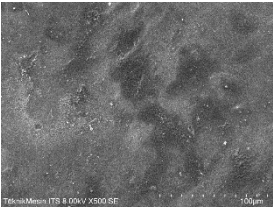
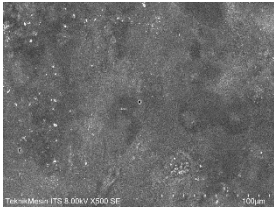
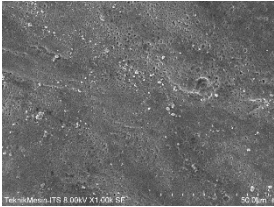
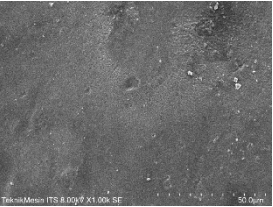
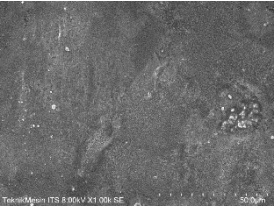
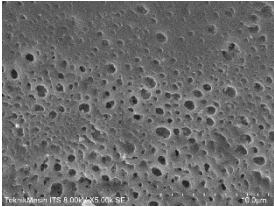
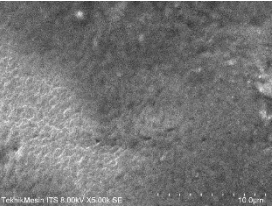
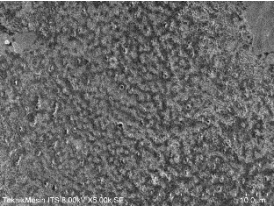
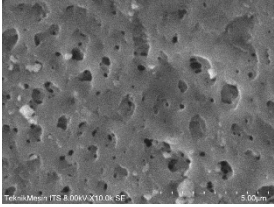
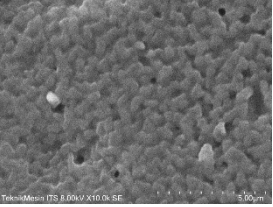
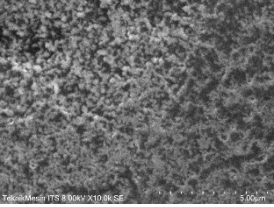
Figure 6. The process of breaking the glycosidic bonds in starch molecules.

Scanning Electron Microscope (SEM)

A scanning electron microscope (SEM) was conducted to observe the effect of modified *E. guineensis* starch on the microscopically surface morphology of the capsule shell. The results of morphological observations shown in Table 7 that the F1 capsule shell had many pores and was larger when compared to other formulation capsule shells, observations of the F2 capsule shell had the least pores with a bumpy surface, while the morphology of the F3 capsule shell had an irregular surface and smaller pores compared to other capsule shells. The correlation between SEM and FTIR analysis affects the characterization of capsule shells. For instance, the modified capsule shells have faster dissolution time than unmodified ones, resulting in stronger capsule shells and better morphology.

The large pores found in the F1 shell can be caused by the ratio of materials used being unable to cover all parts of the capsule shell. This can occur because the formula used has not thickened completely. The F2 capsule shell has the best morphology, closely resembling the polyamide capsule shell from the Essawy and Tauer (2010) research, with a bumpy surface. This similarity is caused by the ratio of modified starch and HPMC being able to bind each other perfectly, which reduces the number of pores and ensures complete coverage of the capsule shell surface. The irregular surface and smaller pores in F3 can be caused by the difference in shell thickness, which closes the pores gradually in some parts, resulting in an irregular shape and fewer visible pores (Essawy and Tauer, 2010).

Table 7. The result of SEM on capsule shell formulations.

Magnificent	Morphology of Capsule Shell		
	F1 (1:1)	F2 (2:1)	F3 (3:1)
500x			
			
			
			

CONCLUSION

The best concentration of modified *E. guineensis* starch and HPMC with a ratio of 2:1 based on its disintegration time an average of 6 minutes and 47.06 seconds, tensile strength of 2.565 MPa, and the best morphological surface of capsule shell compared to the other formulations. Modified starch concentration affects organoleptic characteristics, capsule shell weight, disintegration time, tensile strength, FTIR, and SEM. However, there is no effect on the capsule size. Suggestions for future research are to use a pin bar in the manufacturing process to get more uniform results. Additionally, further research is needed to modify the method used to make capsule shells using only modified *E. guineensis* starch.

CONFLICT OF INTEREST

There is no conflict of interest in this article.

AUTHOR CONTRIBUTION

AK: Manuscript Drafting, Manuscript Review, Characterization (tensile strength, FTIR, and SEM); NK: Manuscript Review, Formulation, Decolorization; PV: Manuscript Review, Preparation sample, Extraction; DAA: Manuscript Drafting-Review, Modification of palm starch.

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