

OPTIMIZATION OF pH VALUE AND GEL VISCOSITY IN PORANG TUBER CAPSULE SHELL FORMULATION

Riza Maulana¹, Muhtadi^{2*}, Andi Suhendi², Farid Adi Prasetya³, Sabtanti Harimurti⁴, Hasyrul Hamzah⁵

¹Department of Pharmaceutics, Faculty of Pharmacy, Universitas Muhammadiyah Surakarta, Surakarta, Indonesia, 57144.

²Department of Chemical Pharmacy, Faculty of Pharmacy, Universitas Muhammadiyah Surakarta, Surakarta, Indonesia, 57144.

³Department of Management, Faculty of Economics and Business, Universitas Muhammadiyah Surakarta, Surakarta, Indonesia, 57144.

⁴School of Pharmacy, Faculty of Medicine and Health Sciences, Universitas Muhammadiyah Yogyakarta, Indonesia, 55183.

⁵Faculty of Pharmacy, Universitas Muhammadiyah Kalimantan Timur, Samarinda, Indonesia, 75124.

*Email Corresponding: Muhtadi@ums.ac.id

Submitted: March 12, 2024 Revised: August 18, 2024 Accepted: August 22, 2024

ABSTRACT

The *porang* tuber contains glucomannan compounds that can expand into a gel so that it has the potential to be used as the main ingredient of capsule shells. This research aims to optimize the pH and viscosity values that can affect the consistency of the capsule shell produced. The method used in the manufacture of capsule shells is the dipping pen method, while the optimization method carried out is trial and error by making 3 formulas by combining glycerin and PEG 400. The results obtained by the gel mass formed by the glycerin-PEG 400 gelling agent have a viscosity value of 586.67 dPas and a pH value of 6.02 can provide a gel mass that is easy to mold capsule shells and obtain capsule shells with elastic characters. The results indicate optimal viscosity and pH values to obtain a gel mass that is easy to mold capsule shells with good organoleptic characteristics.

Keywords: Glucomannan, pH, viscosity, capsule shell.

INTRODUCTION

The *porang* (*Amorphophallus muelleri* Blume) is a type of tuberous plant that grows in tropical and subtropical regions (Dewanto and Purnomo, 2009). It belongs to the *Amorphophallus* family and is commonly used in the food and non-food industries as an alternative source of carbohydrates, due to its glucomannan compounds (Figure 1). *Porang* tubers are utilized in the food industry to produce traditional Japanese dishes like noodles (shirataki) and tofu (konnyaku) (Sumadi, 1979).

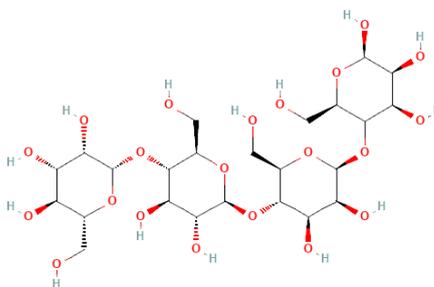


Figure 1. Glucomannan chemical structure

Due to its high glucomannan content, it can create a solid film layer and has good biocompatibility, biodegradability, and gelling properties, making it an ideal material for

preparing capsule shells (Riyanto *et al.*, 2020). Capsule shells typically comprise gelatin, but non-halal sources of gelatin are frequently exploited, thereby necessitating an alternative ingredient in the form of polysaccharides (Sumarwoto, 2007; Rosmalasari, 2018). Prior research indicates that *porang* flour has exceptional qualities as a material for producing hard capsule shells, being plant-based, halal, similar to gelatin in terms of characteristics, and possessing biological activity.

The process of formulating a *porang* tuber capsule shell involves creating an "intermediate preparation" in the form of a gel. This gel formation is crucial for dipping the capsule molding tool, or dipping pen, to ensure adhesion before proceeding to the drying process (Rosmalasari, 2018).

Gel formation is a crucial consideration in the formulation of capsule shells to support the tablet molding process and enhance the elasticity of the resulting capsules. This study introduces a novel approach using glycerin, and PEG 400 as thickening agents, which play a vital role in enhancing the elasticity of the capsule shells during their formation.

The aim of this study was to optimize the combination of glycerin-PEG 400 on two parameters, namely viscosity, and pH, which could impact the formation of a desirable gel mass in capsule shell formulations. Additionally, the study aimed to evaluate the organoleptic properties of the resulting capsule shell.

RESEARCH METHODS

Equipment and Materials

In this study the equipment that is used were glassware (Iwaki), Brookfield DV-1 viscosimeter (Ametek), pH-meter for semisolid preparations (Ohaus), water bath (Mettler), oven (Binder). The materials were *porang* tuber flour obtained from PT Bagaskara Konjac, glycerin (Mitra Medika), and PEG 400 (Mitra Medika).

Determination of Optimum Design

The process of the determination of optimum design was conducted through the trial and error method, utilizing three formula variations. The total weight for each formula was 50 g, as presented in Table I.

Table I. Capsule Shell Gel Optimization Design

Materials	Formulas		
	1	2	3
<i>Porang</i> tuber flour (g)	5	5	5
Glycerin (mL)	1.5	2.5	3.5
PEG 400 (mL)	0.5	0.5	0.5
Distilled water (mL)	48	47	46

Capsule Shell Molding and Drying

The materials were mixed by dissolving glycerin-PEG 400 in each formula in distilled water in a beaker and continuously stirred in a water bath at 60 °C. The *porang* tuber flour was added to the mixture while stirring until it formed mucilage and thickened into a gel-like consistency.

The dipping pen was prepared beforehand by placing it in the freezer, which rendered a cold surface for dipping. Subsequently, the pen was aerated for one minute before usage. The dipping pen was immersed in the gel mass immediately before it hardened and stiffened. Following the dipping process, the pen was dried in an oven at 60 °C for five hours. Organoleptic properties were evaluated for the capsule shells that formed.

Viscosity and pH Measurement of The Gel

On the other hand, the gel mass in the beaker glass was also measured for viscosity using a Brookfield DV-1 viscometer (Ametek) and a pH meter for semisolid preparations (Ohaus). Each measurement was done in 3 replications (triplo).

RESULTS AND DISCUSSION

An organoleptic test was conducted by visually observing the capsule shell formed, including color, odor, and characteristics of the resulting capsule shell. The test results are shown in [Table II](#).

Table II. Comparison of Results on All Formulas

Test	Aspects	F1	F2	F3
Organoleptic	Color	brownish-yellow	brownish-yellow	brownish-yellow
	Odor	typical of <i>porang</i> tuber	typical of <i>porang</i> tuber	typical of <i>porang</i> tuber
	The physical appearance of the capsule shell	very mushy	solid, elastic	solid, slightly stiff

There were no noticeable variations in the color and scent of the *porang* tuber capsule shells produced. F2 yielded the most elastic capsule shell with a glycerin-PEG 400 ratio of (2.5:0.5 mL). The elastic capsule shell offers a distinct advantage in the formulation process due to its ease of release from the mold (dipping pen), thus creating a desirable capsule shell shape.

Other formulations with F1 containing the lowest amount of glycerin result in a mushy shell after the drying process, making it sticky with the mold. F3, on the other hand, with the highest amount of glycerin, produces capsule shells with stiffer characteristics in comparison to those produced under F2. A stiff capsule shell will increase brittleness, potentially leading to losses during the molding process where the shell is prone to tearing and roughness. [Figure 2](#) displays the results of capsule shell molding in all formulas.



Figure 2. Results of capsule shell molding

The thickness of the capsule shell impacts its protective ability for the medication it encapsulates. Appropriate capsule shell thickness can preserve drug quality from environmental factors. The thickness of the capsule shell layer affects the weight of the capsule. If the capsule is made thicker, its weight increases.

Glucomannan demonstrates significant water expansion ability, with expansion capacity ranging from 138–200%. Glucomannan has the ability to expand (swell) up to 50% in water, which is significantly higher compared to starch's expansion ability (25%).

Through hydrogen bonding, glucomannan is able to combine with the molecular structure of water, forming two hydrogen atoms, which creates a very thick solution (Takigami, 2000; Chaplin, 2016). Additionally, the interaction between hydrocolloids and water occurs as a result of the reaction between glucomannan and water. This technique is employed for creating capsules using *porang* tuber and a distilled water solvent.

The importance of viscosity as a quality parameter for liquid and viscous substances to evaluate and control their quality has been emphasized by Sinurat *et al.*, (2006). The extraction process of *porang* tuber flour from *porang* tubers enhances its glucomannan content. The *Porang* tuber flour, when dissolved in water, can form a highly viscous gel with increased thickness (Rosmalasari, 2018).

The results of the viscosity measurement are illustrated in Figure 3. Statistical analysis was performed using SPSS software version 20. The results obtained for viscosity data were normally distributed and homogeneous. One-way ANOVA test results showed that glycerin had no significant effect on the viscosity of the gel produced with a significance value of $P = 0.269 > 0.05$. Likewise, when the Post Hoc test was conducted, there was no significant effect on the viscosity value between formulas.

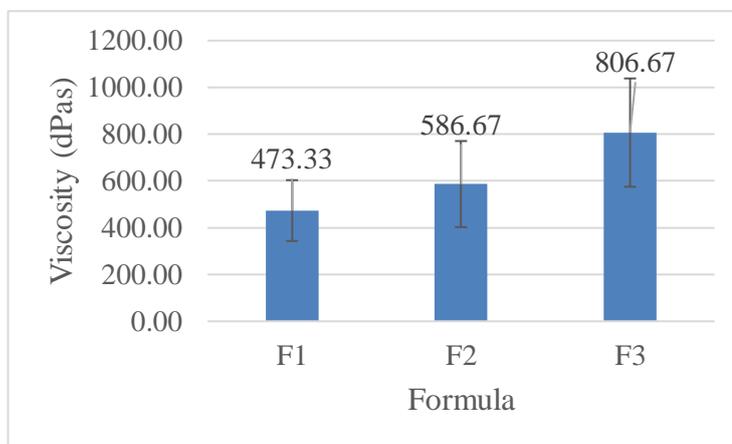


Figure 3. Comparison of viscosity test results (n=3)

Siswanti (2008) noted that the thickness of the surface of the glucomannan film can be impacted by the concentration of glucomannan. Higher levels of glucomannan generally result in higher tensile strength, which in turn causes the film to be thicker. This can affect the molding of the capsule shell.

However, to better observe the effect of varying concentrations of glycerin-PEG 400, the concentration of glucomannan was held constant in all formulas used in this study. The impact of viscosity arises from varying levels of thickening agent concentration. Asngad *et al.*, (2018) confirmed that glycerin also boosts the gel's viscosity.

Viscosity, a crucial aspect of gel strength (Permata *et al.*, 2016), describes a liquid's resistance to flow. Viscosity impacts the flow of a liquid aided by adsorbs (Iqbal *et al.*, 2015). Temperature, pH, and the concentration of thickener used are among the factors that can affect viscosity (Febriana *et al.*, 2021).

It is crucial to measure the pH value because it can affect the viscosity value and gel strength (Mardawati *et al.*, 2018), which then influences the characteristics of the resulting capsule shell.

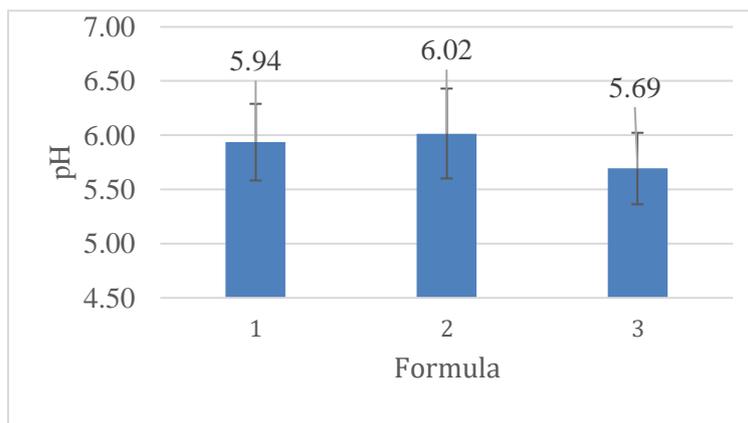


Figure 4. Comparison of pH test results (n=3)

Based on the physical properties of the capsule shell, it was found that F2 has the best elasticity compared to other formulas, leading to the highest pH value as presented in

Figure 4. According to [Sukmawati et al. \(2017\)](#), glycerin does not affect the pH value production. The results of statistical analysis showed that the data were normally distributed and homogeneous, the results of the One-way ANOVA test showed that glycerin had no significant effect on the pH value of the gel with a significance value of $P = 0.675 > 0.05$. Likewise, when the Post Hoc test was conducted, there was no significant effect on the viscosity value between formulas.

When compared to gelatin, which is commonly used for capsule shells, the strength of the formed gel depends on the presence of hydrogen bonds with molecular water and free amino acid groups. The viscosity can increase or decrease depending on the pH. Changes in pH can affect the viscosity within the pH range of 6–8 for pharmaceutical gelatin according to the Gelatin Manufacturers Institute of America (GMIA) standard ([Firlianty, 2016](#)).

CONCLUSION

Optimizing the thickening agent in the *porang* tuber capsule shell production process is crucial in achieving high-quality capsules. Alongside organoleptic evaluations for determining the shell's characteristics, monitoring the viscosity and pH levels of the gel preparation - an intermediate product - also provides valuable information that assists in the molding process and the final physical properties of the capsule shell.

ACKNOWLEDGMENT

Our gratitude goes to Universitas Muhammadiyah Surakarta and the Institute for Research & Innovation (LRI) for providing funding support for Productive Innovative Research - UMS Contract Number: 81.13/A.3-III/LRI/III/2023, and the Faculty of Pharmacy UMS for providing facilities in the implementation of this research.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest in the conduct of this research.

REFERENCES

- Asngad, A., Aprilia, B. R., and Nopitasari, 2018, Kualitas Gel Pembersih Tangan (*Hand Sanitizer*) dari Ekstrak Batang Pisang dengan Penambahan Alkohol, Triklosan dan Gliserin yang Berbeda Dosisnya, *Jurnal Bioeksperimen*. 4(2): 61–70.
- Chaplin, M, 2016, Hydrocolloids and Gum, *Water Structure and Science*.
- Dewanto, J. and Purnomo, B. H, 2009, Pembuatan Konyaku dari Umbi Iles-iles (*Amorphophallus oncophyllus*), *Skripsi*, Universitas Sebelas Maret. Surakarta.
- Febriana, L. G., Stannia, N. A. S., Fitriani, A. N., and Putriana, N. A, 2021, Potensi Gelatin dari Tulang Ikan sebagai Alternatif Cangkang Kapsul Berbahan Halal: Karakteristik dan Pra Formulasi, *Majalah Farmasetika*. 6(3): 223–233.
- Firlianty, I. R, 2016, Physico-chemical Characterization and Skin Gelatin Rheology of Four Freshwater Fish as Alternative Gelatin Source, *AAFL Bioflux*. 9(6) : 1196–1207.
- Iqbal, M. C., Anam, A. A., and Ridwan, 2015, Optimasi Rendemen dan Kekuatan Gel Gelatin Ekstrak Tulang Ikan Lele Dumbo (*Clarias gariepinus* sp.), *Jurnal Teknosains Pangan*. 9(4): 8–10.
- Mardawati, E., Sugandi, H., Kayaputri, I. L., Cahyana, Y., Wira, D. W., Pujiyanto, T., et al., 2018, Study and Characterization of Powder Mackerel (*Scomberomorus commerson*) Bone Gelatin through Hydrolysis of Hydrochloric Acid, *AIP Conference Proceedings*, Universitas Padjadjaran, Bandung, Indonesia.
- Permata, Y., Widiastri, F., Sudaryanto, and Anteng, A, 2016, Gelatin dari Tulang Ikan Lele (*Clarias batrachus*): Pembuatan dengan Metode Asam, Karakterisasi dan Aplikasinya sebagai *Thickener* pada Industri Sirup, *Jurnal Ilmiah Widya Teknik*. 15(2) : 146–152.
- Riyanto, R., Darma, G. C. E., and Aryani, R, 2020, Preparasi dan Karakterisasi Tepung Iles-Iles (*Amorphophallus muelleri* Blume.) serta Analisis Potensinya sebagai Bahan Pembuatan Cangkang Kapsul, *Prosiding Farmasi*, 6(2). <http://dx.doi.org/10.29313/v6i2.23214>.
- Rosmalasari, A. A, 2018, Pembuatan Cangkang Kapsul Halal Berbahan Dasar Umbi Porang (*Amorphophallus oncophyllus*), *Skripsi*, Fakultas Ilmu Alam, ITS Surabaya.
- Sinurat, E., Murdinah, and Bagus, S, 2006, Sifat Fungsional Formula Kappa dan Iota Karaginan dengan Gum, *Jurnal Pasca Panen dan Bioteknologi Kelautan dan Perikanan*. 1(1): 1–8.
- Siswanti, 2008, Karakteristik Edible Film Komposit dari Glukomanan Umbi Iles-Iles (*Amorphophallus muelleri* Blume) dan Maizena, *Skripsi*, Bogor: IPB Press.
- Sukmawati, A., Laeha, N-A., and Suprpto, 2017, Efek Gliserin sebagai Humectan Terhadap Sifat Fisik dan Stabilitas Vitamin C dalam Sabun Padat, *Pharmakon: Jurnal Farmasi Indonesia*. 14(2): 40–47.
- Sumadi, 1979, Beberapa Tinjauan Mengenai Iles-Iles, 30 – 37, Lembaga Kimia Nasional-LIPI, Bandung.
- Sumarwoto, 2007, Review: Kandungan Mannan pada Tanaman Iles-iles (*Amorphophallus muelleri* Blume.), *Bioteknologi*. 4(1): 28–32.
- Takigami, S, 2000, Konjac Mannan. (G. O. Phillip, and P. A. 68 Williams, Eds.) *Handbook of Hydrocolloids*. Washington DC: CRC Press.

SUPPLEMENTARY DATA

Test	Replication	F1	F2	F3
Viscosity (dPas)	1	340	820	500
	2	430	370	1060
	3	650	570	860
	Average	473.33	586.67	806.67
	SD	130.21	184.09	231.71
pH	1	5.45	5.45	5.26
	2	6.28	6.43	5.76
	3	6.08	6.17	6.06
	Average	5.94	6.02	5.69
	SD	0.35	0.41	0.33

Tests of Normality

Formula	Kolmogorov-Smirnov ^a			Shapiro-Wilk			
	Statistic	df	Sig.	Statistic	df	Sig.	
Viscosity	Formula 1	.274	3	.	.945	3	.546
	Formula 2	.196	3	.	.996	3	.878
	Formula 3	.241	3	.	.974	3	.688
pH	Formula 1	.296	3	.	.918	3	.445
	Formula 2	.285	3	.	.932	3	.495
	Formula 3	.232	3	.	.980	3	.726

a. Lilliefors Significance Correction

➔ **Oneway**

[DataSet0]

Test of Homogeneity of Variances

	Levene Statistic	df1	df2	Sig.
Viscosity	.459	2	6	.652
pH	.162	2	6	.854

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
Viscosity	Between Groups	172355.556	2	86177.778	1.649	.269
	Within Groups	313600.000	6	52266.667		
	Total	485955.556	8			
pH	Between Groups	.170	2	.085	.419	.675
	Within Groups	1.217	6	.203		
	Total	1.388	8			

Post Hoc Tests

Multiple Comparisons

Bonferroni

Dependent Variable	(I) Formula	(J) Formula	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Viscosity	Formula 1	Formula 2	-113.333	186.667	1.000	-726.99	500.32
		Formula 3	-333.333	186.667	.373	-946.99	280.32
	Formula 2	Formula 1	113.333	186.667	1.000	-500.32	726.99
		Formula 3	-220.000	186.667	.850	-833.66	393.66
	Formula 3	Formula 1	333.333	186.667	.373	-280.32	946.99
		Formula 2	220.000	186.667	.850	-393.66	833.66
pH	Formula 1	Formula 2	-.08000	.36779	1.000	-1.2891	1.1291
		Formula 3	.24333	.36779	1.000	-.9657	1.4524
	Formula 2	Formula 1	.08000	.36779	1.000	-1.1291	1.2891
		Formula 3	.32333	.36779	1.000	-.8857	1.5324
	Formula 3	Formula 1	-.24333	.36779	1.000	-1.4524	.9657
		Formula 2	-.32333	.36779	1.000	-1.5324	.8857