UNDERGRADUATE THESIS

FORMULATION OF PEEL-OFF GEL MASK FROM RED RICE PREGELATINIZED STARCH AS THE GELLING AGENT

by :

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PHARMACY DEPARTMENT

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FORMULASI SEDIAAN MASKER GEL PEEL OFF DENGAN PATI PRAGELATINISASI BERAS MERAH SEBAGAI GELLING AGENT

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ABSTRAK

Pati merupakan salah satu bahan yang berpotensi untuk dijadikan sebagai gelling agent. Hal ini disebabkan karena didalam pati terdapat kandungan amilosa dan amilopektin. Namun, pati mempunyai sifat alami yang dapat menyebabkan beberapa kendala jika dipakai sebagai bahan baku dalam industri pangan maupun non pangan. Salah satunya pasta yang dibentuk membutuhkan waktu yang lama, bentuknya keras dan tidak bening. Oleh karena itu perlu dilakukan modifikasi salah satunya dengan cara gelatinisasi. Penelitian ini bertujuan untuk mengetahui formulasi yang sesuai untuk membuat masker gel peel off dengan pati pragelatinisasi beras merah sebagai gelling agent dan bagaimana karakteristik masker yang dihasilkan serta mengetahui berapa konsentrasi terbaik dari pati yang digunakan dalam formulasi. Formulasi masker dibuat dengan 3 formula dengan konsentrasi pati 5%, 10% dan 15%. Evaluasi sediaan masker gel peel off meliputi pengujian organoleptis, homogenitas, viskositas, daya sebar, waktu mengering, dan elastisitas. Uji stabilitas sediaan dilakukan dengan menyimpan sediaan selama 21 hari pada suhu ruang dan pengujian dilakukan pada hari ke 1, 7, 14 dan 21. Data yang diperoleh dari selama penyimpanan dianalisis menggunakan Paired Samples t Test. Hasil dari penelitian menunjukkan bahwa formulasi yang sesuai untuk membuat masker gel peel off adalah menggunakan bahan PVA 9%, propilenglikol 15%, Natrium benzoat 0,2% dan aquades. Karakteristik yang dihasilkan adalah masker berwarna coklat muda, beraroma khas pati, berbentuk kental, memiliki nilai pH 5,7-5,9, viskositas 1077-5082 cPs, daya sebar 7,3-5,5 cm, waktu mengering 31-14 menit, elastisitas 9,5-11 cm dan ketiga sediaan stabil selama penyimpanan. Konsentrasi pati pragelatinisasi terbaik yang digunakan dalam formulasi masker gel peel off adalah sediaan F(2) dengan konsentrasi pati 10%.

Kata kunci : Beras Merah, Formulasi, Gelling Agent, Masker Gel Peel Off, Pati Pragelatinisasi.

FORMULATION OF PEEL-OFF GEL MASK FROM RED RICE PREGELATINIZED STARCH AS THE GELLING AGENT

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ABSTRACT

Starch is one of the ingredients that has the potential to be used as a gelling agent. This is because the starch contains amylose and amylopectin. However, starch has natural properties that can cause several obstacles if used as raw materials in food and non-food industries. One of them is paste that requires a long time to form, whose shape is hard and not clear. Therefore it is necessary to modify and one of the ways to modify it by gelatinization. This study aims to determine the appropriate formulation to make peel-off gel masks with red rice pregelatinized starch as a gelling agent, how the characteristics of the masks are produced and to find out the best concentration of starch used in the formulation. Mask formulations are made with 3 formulas with 5%, 10% and 15% starch concentrations. Evaluation of peel-off gel masks includes organoleptic testing, homogeneity, viscosity, dispersion, drying time, and elasticity. Stability test was carried out by storing preparations for 21 days at room temperature and testing was carried out on days 1, 7, 14 and 21. Data obtained during storage were analyzed using Paired Samples t Test. The results of the study showed that the appropriate formulation for making peel-off gel masks was using 9% PVA, 15% propylenglycol, 0.2% sodium benzoate and distilled water. The characteristics produced are light brown colored masks, typical of starch, thick in shape, have a pH value of 5.7-5.9, viscosity of 1077-5082 cPs, spread of 7.3-5.5 cm, drying time of 31-14 minutes , 9.5-11 cm elasticity and all the formulation are stable during storage. The best concentration of pragelatinized starch used in the formulation of peel off gel masks is the preparation of F (2) with a starch concentration of 10%.

Keyword : *Red Rice, Formulation, Gelling Agent, Peel-Off Gel Mask, Pregelatinized Starch.*

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CHAPTER I

INTRODUCTION

1.1. Background of The Study

Cosmetics is a substance or mixture of substances that are applied to human skin for the purpose of cleaning, maintenance, transfiguring and improving an appearance but they are not included in the class of drugs. One example of cosmetics used for facial care is a mask (Sriwidodo, 1986). There are various kinds of mask products on the market including powder masks, cream masks, gel masks, and paper masks. Among the types of masks in the market, one type of mask that is practically used is a gel mask that can be immediately peeled off after drying or commonly called by peel-off gel mask (Muliyawan dan Suriana, 2013).

The peel-off gel mask is usually in a gel or pastes that are applied by spreading it to the facial skin. After 15-30 minutes, the mask can be removed by peeling (Slavtcheff, 2000). One of the advantages of peel off gel masks compared to other types of masks is the easy use and cleansing that is by lifting or releasing like an elastic membrane (Harry, 1973). In this case, the composition of the ingredients has an important role in determining the physical quality of the preparation of peel off gel masks such as gelling materials that affect the viscosity, dispersion and drying time of the preparation (Vieira, 2009).

هُوَ ٱلَّذِىٓ أَنزَلَ مِنَ ٱلسَّمَآءِ مَآءً لَّكُم مِّنْهُ شَرَابٌ وَمِنْهُ شَجَرٌ فِيهِ تُسِيمُونَ ﴿١٠﴾

Allah SWT says in An-Nahl verse 10 :

"It is He who sent down rain from the sky, from it is drinking and from it is foliage in which you pasture (animals). (Kementrian Agama RI, 2012)

According to the interpretation of al-Misbah, the verse above explains the essence of Allah SWT and explains the details of the blessings that Allah SWT has given to servants. This verse also describes plants as the main requirement for humans and animals. This verse can be used as a reminder for humans to always be grateful for His blessings and to take advantage of the gifts that have been given well (Shihab, 2002). Allah SWT has grown various types of plants on earth to be utilized by humans as well as possible. Likewise with red rice plants. During this time, red rice has only been used to fulfill what food needs. Therefore, the application of red rice starch as a gelling agent in peel-off gel mask preparations is one of the efforts to optimize the utilization of red rice in the world of the pharmaceutical industry.

Starch is a complex carbohydrate that is insoluble in water, a powdery white, tasteless and odorless. In the industrial world, starch can be used as a raw material or as an additional ingredients such as coagulation (thickening agent), gel-forming (gelling agent), film-forming (filming agent) and as a stabilizer (stabilizing agent). Starch has hydrocolloid characteristics so it can be used as a gelling agent and film. The hydrocolloid characteristics of starch are due to the amylose content in starch. Starch is a complex carbohydrate consisting of amylose and amylopectin (Jacobs and Delcour, 1998). Amylose is commonly used to make films and gels because amylose has a structure that makes it possible to form hydrogen bonds between glucose molecules during heating. Garcia et al. (1998) reported that high amylose content would make the film more compact because amylose was in charge of the formation of the film matrix.

Amylose content in rice can be viewed from the properties of cooked rice. Santika and Rozakurniati (2010) said that levels of amylose in rice is one of the indicators that determine the flavor of the rice. Rice which had a low amylose levels (10-20%) will produce rice that is too sticky. Rice which has amylose levels with moderate (20-25%) will produce fluffier rice and is generally preferred by the consumers. Meanwhile, the rice with high

amylose levels (>25%) will produce rice with hard texture and only some consumers prefer it. Red rice is one type of rice that has hard texture and just partially people who want to consume it. This shows that the levels of amylose in red rice is greater than in white rice. According to the study by Umar et al., (2011) about the physicochemical of red rice it was found that levels of amylose in red rice is 23.83%. Therefore, red rice has the potential to be used as a gelling agent and the film.

In the industrial world, native starch has several obstacles if used as food and non-food raw materials. When cooked, starch will take a long time, the paste formed is too hard and not clear. In addition, it is too sticky and cannot stand the acid treatment (Koswara, 2006). Meanwhile, the important characteristics of starch desired by the industry include higher brightness (whiter starch), lower retrogradation, lower viscosity, clearer gel formed, softer formed gel texture and easier starch granules broke (Jane, 1995). This is the reason for starch modification physically, chemically, and enzymatically or a combination of these methods. One method of modifying starch that can be done to change the properties of starch is by making pregelatinized starch using the gelatinization technique.

Pregelatinized starch is starch that has been gelatinized by heating the starch under the temperature of the gelatinization and then dried (Wurzburg, 1989). Gelatinization is the process of breaking starch granules with water and heat so that each surface layer of the molecule can absorb water or dissolve and react with other materials (Smith, 1985). The breakdown of starch granules is caused by the presence of water and heat so that amylose is able to diffuse out of the granule. Pregelatinized starch will dissolve if mixed with cold water and thicken. However, the viscosity produced from the gelatinized starch and pregelatinized starch is different. The viscosity of starch that is gelatinized will be higher than pragelatinized starch. Therefore, modification of starch used in this study was pragelatinized starch.

The gelatinization process can be carried out using a drum dryer, spray dryer or extruder (Collona et al., 1984). In addition to using these 3

devices, gelatinization can also be done using an oven. Research conducted by Lukman *et al.* (2012) on the use of pregelatinized glutinous rice starch as a loose tablet matrix of sodium diclofenac, the gelatinization of glutinous rice starch was carried out using an oven at 50 ^{o}C. The study of the use of modified starch as a gelling agent material was also carried out by Sulastri et al., (2016) on the effect of black rice pregelatinized starch as a gelling agent on the physical quality of the peel-off gel mask. The results of the study showed that the addition of pregelatinized starch can affect the characteristics of peel-off gel masks including lowering the pH value, increasing viscosity, lower dispersion power and shorten drying time. Based on the background above, this study was conducted to find out the appropriate formulations for making peel off gel masks from red rice pregelatinized starch as gelling agent.

1.2. Research Problem

The Research problem in this research are :

1. How is the appropriate formulation for making peel off gel mask from red rice pregelatinized starch as gelling agent ?
2. How is the physical characteristics of peel off gel mask from red rice pregelatinized starch as gelling agent ?
3. What is the best concentration of pregelatinized starch used for peel off gel mask ?

1.3. Purpose of The Study

The purpose of the study are :

1. Knowing the appropriate formulation for making peel off gel masks from red rice pregelatinized starch as gelling agent
2. Knowing the characteristics of peel off gel masks from red rice pregelatinized starch as gelling agent
3. Determine the best concentration of pregelatinized starch used for peel off gel mask

1.4. Significance of The Study

1.4.1. Theoretical Significance

The benefit of this study is to provide scientific information about the potential of red rice starch as gel forming and film forming in preparation of peel off gel mask with gelatinization techniques and to know the characteristics of peel off gel masks produced form pregelatinized starch.

1.4.2. Practical Significance

The benefit of this reaserch is that it can be used as a reference for further reaserch and as a reference for making peel of gel masks with pregelatinized starch as a gelling agent.

CHAPTER II

LITERATURE REVIEW

2.1. Previous Research

This research was conducted referring to several previous studies. The study of the use of starch as a gelling agent was carried out by Soebagio et al., (2009) with the title The Utilization Of Arrowroot Starch (Amylum Marantae) as a Gelling Agent on 10% Urea Gel Preparations. The study used a variation of 4.5%, 6.5%, 8.5%, 10.5% and 12.5% starch concentrations. The conclusion of this study is that urea gel preparations can be made using arrowroot starch as a gelling agent which is safe to use.

Research on pregelatinized starch was also carried out by Lukman et al. (2012) with the title The Utilization of Pregelatinized Glutinous Rice Starch as a Matrix of Tablets Released Slowly Diclofenac Sodium. In this study, the starch was gelatinized using an oven at a temperature of 50 ^{o}C. The gelatinized starch is then formulated by combining pregelatinized starch and sodium carboxyl methyl cellulose. The conclusion of this study is that the combination of pregelatinized glutinous rice starch with sodium carboxyl methyl cellulose in the tablet matrix was slowing down the dissolution rate of diclofenac sodium.

Evi Sulastri et al., (2016) conducted a study entitled "The Effect of Black Rice Pregelatinized Starch as Gelling Agent on Physical Quality of Peel Off Gel Mask". This study used 5 types of formulations with concentration of pregelatinized starch with each formulas of F (1) 3%, F (2) 4%, F (3) 5%, F (4) 6% and F (5) 6%. The amount of starch used in the 4th and 5th formula is the same. The difference is, the fourth formulation uses HPC while the fifth formulation does not use HPC. The results of the study indicate that the administration of starch in the formulation of peel off gel masks can affect the physical characteristics of the mask. Increasing the concentration of pregelatinized starch will increase the viscosity of peel-off

gel mask produced. Increasing the viscosity of mask will increase the time of mask to dry out and decrease the value of preparation to spread. Thus, from the results of the research it can be concluded that the value of the starch concentration that can give the best characteristics of the peel off gel mask according to the standard is 3%.

2.2. Theoretical Basis

2.2.1. Red Rice Plants

The staple food in Indonesia besides white rice is red rice. Classification and morphology of red rice according to the Indonesian Ministry of Health (2005) is as follows :

Kingdom	: Plantae
Sub kingdom	: Tracheobionta
Super division	: Spermatophyta
Division	: Magnoliophyta
Class	: Liliospida
Sub class	: Commelinidae
Ordo	: Poales
Family	: Poaceae
Genus	: Oryza
Species	: *Oryza nivara*

The morphology of red rice plant is that it has fiber roots. The red rice plant is round, the surface is slippery, it has sections and hollow, and the direction of the stem is straight up. The red rice paddy stem is green but the base of the stem is red. The stem length can reach 2 meters. Red rice leaves are classified as incomplete leaves because they only have leaves and leaf midribs. They have ligula membrane type. The leaves have ribbon-shaped with pointed ends in which the base leaves are flat and flat-edged. The middle of the leaf is green but the edges of the leaf are red. Red rice is a single fruit that is dry with

hard and woody outer parts like dry skin. Red rice fruit is included in thin-walled fruit. It has only one seed and the fruit skin is attached to the seed coat. The red rice plant morphology can be seen in Figure 2.1 below.

Figure 2.1. Red Rice Plant

In general, red rice is consumed without going through the process of ignition, but it is only ground into broken rice and the epidermis of red rice is still attached to the endosperm (Santika and Rozakurniati, 2010). This causes red rice contains fiber, protein, essential fatty acids, various vitamins, iron, magnesium and polyphenols (Wang et al., 2013). Red rice has more fiber content than white rice. This can be seen in Table 2.1 below.

Table 2.1. The Comparison of Nutrient in Red Rice and White Rice

Parameter	Red Rice	White Rice
Calories	232	232
Protein	4,88 g	4,10 g
Carbohydrates	49,7 g	49,6 g
Fat	1,17 g	0,205 g
Fiber	3,32 g	0,74 g
Thiamin (B1)	0,223 mg	0,176 mg
Riboflavin (B2)	0,039 mg	0,021 mg
Niacin (B3)	2,730 mg	2,050 mg

Vitamin B6	0,294 mg	0,103 mg
Folate	10 mcg	4,1 mcg
Vitamin E	1,4 mg	0,462 mg
Magnesium	72,2 mg	22,6 mg
Phosphorus	142 mg	57,4 mg
Potassium	137 mg	57,4 mg
Selenium	26 mg	19 mg
Zinc	1,05 mg	0,841 mg
Iron	1,9 mg	0,5 mg

High fiber in red rice can be used to prevent gastrointestinal diseases. High levels of B vitamins and minerals can be used to prevent beri-beri. High fat content in red rice can act as a source of energy in the body. Red rice has a low glycemic index which can reduce the risk of type 2 diabetes (Babu et al., 2009). In addition, the anthocyanin content of red rice is higher than anthocyanin content in white rice (Murdiati and Amaliah, 2013). In red rice, anthocyanin is a red pigment found in the skin layer of rice or grain (Chang and Bardenas, 1965). The anthocyanin content in red rice is from 0.34 to 93.5 µg (Herani and Rahardjo, 2005). Anthocyanin has functions as an antioxidant that can be used to treat liver disease (hepatitis), colon cancer, stroke, and diabetes (Kristamini and Purwaningsih, 2009).

In terms of health, red rice has higher nutrient content compared to the nutrient content in white rice. Unfortunately, red rice is rarely in demand because it has a hard and fluffy texture when cooked. Unlike the characteristics of fluffier white rice, many people prefer consuming white rice to red rice. The difference in the taste of rice produced is influenced by the content of amylose and amylopectin contained in starch. Santika and Rozakurniati (2010) stated that amylose content in rice is one indicator that determines the taste of rice. Rice that has low amylose content (10-20%) will produce rice that is too fluffier, while rice with moderate amylose content (20-25%) will produce fluffier rice and it is generally preferred by consumers. Meanwhile rice with

high amylose content (> 25%) will produce pure rice and only some consumers want to consume it. From the statement above, it can be concluded that the cause of red rice having a hard flavor is the high content of amylose contained in red rice starch. For this reason, red rice starch has a high potential to be used as a gelling agent because one component that is important in gel forming is amylose.

2.2.2. Pregelatinized Starch

Starch is a polysaccharide produced from the synthesis of green plants through photosynthesis. Starch is a food storage found in grains or tubers. Natural sources of starch can be obtained from corn, pumpkin, potatoes, sweet potatoes, bananas, sago, rice, cassava and sorghum (Herawati, 2010). Starch is a carbohydrate consisting of amylose and amylopectin. Amylose is a part of linear polymer with α- (1-> 4) glucose units. Meanwhile amylopectin is a polymer α- (1-> 4) glucose units with side chains α- (1-> 6) glucose units (Jacobs and Delcour, 1998). When viewed from the structure of starch granules, amylose and amylopectin are arranged and form rings. The number of rings in a starch granule are about 16 piece, which consist of an amorphous layer and a semicrystal layer (Hustiany, 2006).

Starch in its original form is small granules often called granules (Hill and Kelley, 1942). In pure conditions, starch granules are white, shiny, odorless and tasteless. If viewed microscopically, starch granules are seen to be formed by molecules that form a thin and centralized layer. Starch granules have varied shapes and sizes in which some are round, oval, or have irregular shapes. Likewise with its size, some is measured less than 1 micron to 150 microns (Fennema, 1976).

Pregelatinized starch is starch that has been gelatinized by heating the starch under the temperature of the gelatinization and then dried (Wurzburg, 1989). Gelatinization is the process of breaking starch granules with water and heat so that each surface layer of the molecule can absorb water or dissolve and react with other materials

(Smith, 1985). The mechanism of gel formation begins when the starch solution is heated. Starch grains will expand so that the hydrogen bonds in the amorphous unit will be damaged and at certain temperatures the granules will break (Hodge and Osman, 1976). The gelatinization temperature begins with the swelling of starch granules which are irreversible in hot water and terminated when starch has lost its crystalline properties (McCready, 1970). The gelatinization phase begins when water slowly or alternately alternates into the granule, then the granule will expand rapidly and eventually lose its "birefringence" nature and if the temperature continues to rise, the starch molecules will diffuse out of the granule (Uhi, 2006).

Birefringence is a characteristic of intact starch granules that can form two crossed colors on the surface due to being passed on polarized rays because of the differences in refractive indexes in starch granules (Cui, 2009). The tool that can be used to see birefringence from starch granules is a polarized microscope. If starch granules do not have birefringence or cannot form two crossed colors, it can be said that all the granules in the starch have broken. The rupture of all starch granules indicates that the starch has been gelatinized perfectly and has reached the peak temperature of gelatinization. Whereas if there is still birefringence in the starch, it can be said that the starch still has complete granules or it has not been gelatinized perfectly because there is still a whole portion of starch granules (Anwar et al., 2006).

According to Swinkles (2006), there are two kinds of pregelatinization of starch, namely partial pregelatinization starch and perfect pregelatinization starch. Partial pregelatinization still has whole starch granules while in perfect pregelatinization there is no longer a whole starch granule. The difference in the process of making partial and perfect pregelatinized starch is at the temperature used. The characteristics produced from partial and perfect pregelatinized starch are different too. Perfect pregelatinized starch has a clear color and has

a high degree of viscosity compared to partial pregelatinized starch. For this reason, the temperature must be measured and maintained so that the temperature used does not exceed the pregelatinization temperature of the starch. Because if the temperature used exceeds the temperature of pregelatinization, the starch will run into gelatinization and not pregelatinization.

2.2.3. Gelling Agent

The gel is a semi-solid system which consists of a dispersion composed of either small inorganic particles or large organic molecules and is impregnated with each other (Ansel, 1989). Some of the characteristics possessed by gel include (Lieberman et al., 1996):

1. Can expand because it has a gel-forming component that can absorb the solution which results in additional volume.
2. Sineresis, a process that occurs due to contraction during the gel period. If the gel is left there will be shrinkage and the liquid is forced out of the capillary leaving the surface wet.
3. Having a structure that is resistant to change so that the gel structure can vary depending on the gelling component.

Gelling agent is a hydrocolloid substance that gives tixotropy consistency in the gel. In general, these substances are also known as solidifiers or stabilizers and thickening agents (Lieberman et al., 1996). Based on its origin, hydrocolloids can be classified into three groups, namely natural hydrocolloids, naturally modified hydrocolloids and hydrocolloid syntheses. Natural hydrocolloids are hydrocolloids that come from natural sources and do not experience changes in chemical properties during the processing. Examples of natural hydrocolloids are plant exudates, seed gum, pectin, seaweed extract, starch and gelatin. Modified natural hydrocolloids are hydroxoid which are obtained from the modification of natural materials both hydrocolloid and not hydrocolloid to form new hydrocolloids with the desired properties. Modified hydrocolloids are usually obtained from starch derivatives

and cellulose derivatives. Meanwhile, what is meant by synthetic hydrocolloid is hydrocolloid obtained from the chemical synthesis process. Examples of synthetic hydrocolloids are polyvinyl pyrrolidine (PVP), carboxyvinyl polymers (carbopol) and polyethylene oxide (polyox) polymers (Whistler, 1973).

2.2.4. Peel-Off Gel Mask

The peel-off gel mask is one type of gel mask that is used by being applied on the face for 15-30 minutes. One of the advantages of peel-off gel masks compared to other types of masks is their practical use and easy to clean (Harry, 1973). Some of the benefits of using peel-off gel masks include relaxing facial muscles, cleansing, refreshing, moisturizing and softening facial skin (Viera, 2009). Regular use of peel-off mask can reduce fine wrinkles on the face. In addition, the peel-off gel mask also has a different way of working from other masks. When the mask is released, the dirt and epidermis of the dead are also lifted (Septiani, 2011). The ingredients used in making peel off gel masks include:

1. Propylene Glycol

   Propylene glycol is a thick liquid, colorless, odorless, has slightly sweet taste, and has a slightly sharp taste resembling glycerin. It is soluble in acetone, chloroform, ethanol (95%), glycerin and water, insoluble with light mineral oil or fixed oil, but will dissolve some essential oils. Propylenglokol concentrates commonly used as humectants are 15% (Rowe et al., 2009). In the formulation of the peel off gel mask, propylenglycol is added as a humectant which serves to maintain the stability of the preparation from the absorption of moist environment and reduce the evaporation of water from the preparation. In addition, humectants also have a role in maintaining skin moisture (Rowe et al., 2006).

2. Sodium Benzoate

   Sodium Benzoate is a granule or white powder, white, odorless and stable in the air. It dissolves easily in the water, is rather difficult to dissolve in ethanol, and is more easily soluble in 90% ethanol. Sodium benzoate is commonly used as a preservative to prevent microbial growth with concentrations used between 0.02% - 0.5% (Depkes RI, 2014).

3. Polivynil Alcohol (PVA)

   Polyvinyl alcohol is a white to cream powder and it does not smell. It is soluble in hot water, slightly soluble in 95% ethanol and insoluble in organic solvents (Depkes RI, 2014). Polyvinyl alcohol is a non-toxic material and is non-irritant in concentrations up to 10%. PVA is commonly used in cosmetics at a concentration of 7%. One of the advantages of PVA is its ability to form gels that can dry quickly and can form very strong and plastic films to provide good contact between drugs and skin (Rowe *et al*., 2009).

4. Aquadest

   Aquadest is pure water obtained by distillation. Apart from distillation, this pure water can be obtained through ion exchange, reverse osmosis, or in an appropriate manner. Pure water is free from dirt or microbes (Ansel, 1989).

2.2.5. Evaluation of Peel-Off Gel Mask

Several tests to analyze the characteristics of peel-off gel masks include:

1. Organoleptic Test

   Organoleptic testing was carried out to determine the physical form of the preparation that had been made by observing the preparation in terms of shape, color and smell (Ansel, 1989).

2. Homogeniety Test

   This test is carried out to determine the level of homogeneity of a preparation. Homogeneous preparations indicate that the ingredients in the mask have spread evenly. A preparation is homogeneous when no coarse grain is found in the preparation (Depkes RI, 1995).

3. pH Test

   The pH test is carried out to prevent skin irritation during use. Preparations that have a pH too low will cause skin irritation when used. Whereas preparations that have too high pH will cause the skin to dry out (Schueller, 1999).

4. Viscosity Test

   Viscosity is a statement of how resistant a liquid to flow. This test is carried out to determine the viscosity value of the preparation.

5. Spreadability Test

   Spreadability test is carried out to see the ability of the mask to spread on the skin surface when used. Spread is one of the characteristics that can be used to calculate the ease of preparations use. A preparation is classified into a semi-rigid preparation (Semistiff) if the diameter produced from this test is less than 5 cm. If the diameter produced is between 5-7 cm, then the preparation is classified as semi-liquid preparation (Semifluid) (Garg *et al*., 2002).

6. Drying Time Test

   The drying time test is carried out to know how long it takes for the mask to dry and form a film layer on the surface of the skin. This test is done by calculating the time needed by the mask starting from the application to form a dry layer (Vieira, 2009).

7. Elasticity Test

   The elasticity test is carried out to determine the strain strength of a preparation. The elasticity of a preparation will affect the comfort of a preparation when exfoliating the mask from the face (Vieira, 2009).

8. Stability Test

   Stability test is carried out to determine the stability or physical resistance of the preparation during storage (Syarifah, 2015).

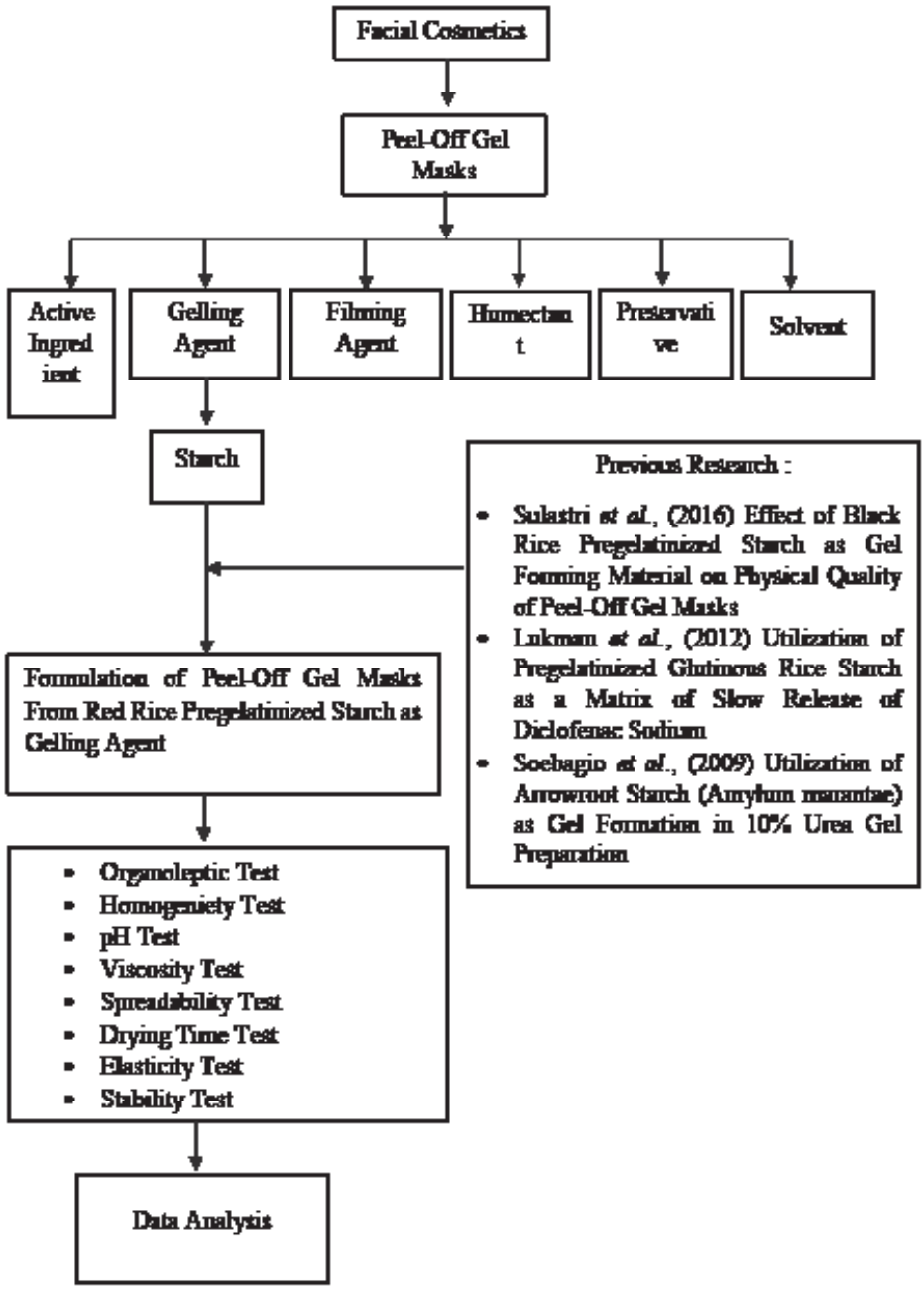
2.2.6. Halal Product Analysis

Haram halal analysis of a product is done by paying attention to the 3P elements, namely Person, Process and Product. 'Person' includes behavior carried out by the researcher. 'Process' includes treatment of post-harvest material, extraction and formulation of preparation. 'Product' includes tools and materials used in making preparations. The *halal* analysis of peel-off gel masks in this study according to the principle of evaluating the *halal* guarantee system are on the basic ingredients, tools used and processing (LPPOM MUI, 2008).

A preparation is said to be *halal* if the basic ingredients used also come from *halal* sources. Basically everything contained in nature is *halal* for consumption except for several types of animals and plants that are forbidden in the Qur'an, such as: carcasses, blood, meat, sacrifices in names other than Allah, strangled animals which are struck, which fall , which was gore and which the wild beast ripped apart except those that had been slaughtered. The vegetable material which is

forbidden in the Quran is khamr. In addition to the materials used, the product processing process also affects the halalness of the products produced. When the processing takes place, the material must not be mixed with objects or animals that are *haram*. Additional materials and auxiliary materials used must also be halal and hygienically produced and fulfill a good production process. Likewise the equipment used in the production process must be made from halal material (Departemen Agama RI, 2010)

2.3.Conceptual Framework



CHAPTER III

RESEARCH METHODS

3.1. Setting Research

This research was conducted for 5 months from September until January 2019 at the Pharmacy Technology Laboratory, Pharmacy Department, Faculty of Health Sciences, University of Darussalam Gontor.

3.2. Tools and Materials of Research

The tools used in this research are analytical scales (Acis BC-5000), oven (memmert), blender (Philips HR2116), viscolead adv (fungilab), pH meter (Ohaus ST10), magnetic stirrer (Cimarec), filter paper, cloth filter (batis), funnel (pyrex), stirring rod (pyrex), glass jar, beaker glass (pyrex), watch glass (pyrex), measuring cup (pyrex), sieve, tube, mortar and stamfer.

The main material used in this research is red rice from the Cirebon, West Java. The material used for the formulation of peel-off gel masks consisted of propylene glycol (PT Brataco), sodium benzoate (Cap Merak), polyvinyl alcohol (PT Brataco), and distilled water.

3.3. Research Design

This research is an experimental study with the following formulation design :

Table 3.1. Formulation Design of Peel-Off Gel Masks

No	Materials	Composition (%)			Function
		F(1)	**F(2)**	**F(3)**	
1.	Red Rice Pregelatinized Starch	5	10	15	Active Ingredient and Gelling Agent
2.	Propylene glicol	15	15	15	Humectant
3.	Sodium Benzoat	0,2	0,2	0,2	Preservative
4.	Polivynil Alcohol (PVA)	9	9	9	Filming Agent
5.	Aquadest	Add 100	Add 100	Add 100	Solvent

The dependent variable in this study was the characteristic of peel-off gel mask from red rice pregelatinized starch as gelling agent. The independent variable of this study is the variation of the concentration of red rice pregelatinized starch. The concentration variations used in this study were 5%, 10% and 15%.

3.4. Stages of Research

3.4.1. Determination of Plant

Determination of red rice is done at the Faculty of Biology of University of Gajah Mada, Yogyakarta. The purpose of the determination of the plant is to know the species of plants to be used in research. Determination of results can be seen in Appendix 1.

3.4.2. The Extraction of Starch

The starch extraction process begins by weighing 1 kg of red rice then washing it using water and draining it. After that, the clean rice is blended using a blender and mixed with 2 L of distilled water. The filtration process is carried out using Batiste until the residue left behind does not remove the water anymore. The filtrate is obtained then silenced for 24 hours to get the large deposits of starch. To separate the large deposits of starch with water, the sediment is filtered using the filter paper and drying it in the oven with a temperature of 50 ^{o}C for 24 hours. After it dries, the starch deposits are crushed and sieved with 100 mesh sieve. In this extraction process the amount of red rice used are 1 kg and get as many as 280 grams of starch.

3.4.3. Manufacture of Pregelatinized Starch

Preparation of pregelatinized starch consists of two stages, they are :

1. Determination of Gelatinization Temperature

   The starch suspension is made in water with a concentration of 50% b/v. The mixture is slowly heated by stirring it and observed

until a thick period is formed. The temperature when the suspension changes to the thick period is determined as the temperature of gelatinization (Lukman, 2012). The results from determining the gelatinization temperature of starch found that red rice starch began to be gelatinized at 77 ^{o}C. This is consistent with the results of research conducted by Indrasari et al., (2010) which stated that red rice begins to gelatinize at a temperature of >74 ^{o}C while at a temperature of <70 ^{o}C red rice has not yet been gelatinized.

2. Manufacture of Pregelatinized Starch

Preparation of pragelatinized starch is done by cooking starch paste at a temperature of 2 ^{o}C below the gelatinization temperature. It is known that the temperature of red rice gelatinization in this study is 77 ^{o}C so the temperature used to make pregelatinized starch is 75 ^{o}C. This is consistent with the results of a study conducted by Chandra et al., (2014) which showed that granules of red rice starch began to swell at 70 ^{o}C and the granules began to break down at 80 ^{o}C. Starch paste that has been made with a concentration of 50% b/v is cooked at a temperature of 75 ^{o}C until it thickens. The temperature used to cook starch must be maintained and controlled. If starch paste has reached a temperature of 75 ^{o}C, then the starch must be removed and cooled immediately. This is done to keep the starch cooked at the pragelatinization temperature, not at the gelatinization temperature. The cooled starch is then dried in the oven at 50 ^{o}C for 24 hours. Starch obtained is smoothed using a blender and sifted with a 100 mesh sieve (Lukman, 2012).

3.4.4. Manufacture of Peel-Off Gel Mask

Before making a peel-off gel mask, the ingredients to be used are weighed first according to the required dose. The initial step of making masks is done by dissolving starch into distilled water (1: 2) at 50 ^{o}C. Sodium benzoate which has been dissolved in distilled water is added little by little to the etched starch while continuing to stir until

it becomes homogeneous. In separate containers, PVA is developed in hot water with a temperature of 80 °C. Then the mixture of starch and sodium benzoate is added to the inflated PVA while stirring it until it becomes homogeneous. Mixing is carried out at 80 °C. Propylene glycol is added gradually to the mixture. Then the remaining water is added little by little and stirred until all the ingredients are mixed.

2.4.5. Evaluation of Peel-Off Gel Mask

The evaluation of peel-off gel masks consist of :

1. Organoleptic Test

   All the gels that have been made are tested by organoleptic test which includes shape, colour and aroma (Farmakope IV, 1995).

2. Homogeniety Test

   The testing is done by placing the sample on top of a glass object and closing it using another glass object, then the two glass objects are pressed and observed. A preparation is homogeneous when no coarse grain is found in the preparation (Farmakope IV, 1995).

3. pH Test

   PH testing was carried out using a digital pH meter dipped in a mask preparation. The number shown at the pH meter is the pH value of the preparation (Farmakope V, 2014). According to SNI number 16-4399-1996, the appropriate pH value for the skin ranges from 4.5-8 (BSN, 1996)

4. Viscosity Test

   Viscosity testing is done by using 100 grams of sample which was measured using a viscolead adv fungilab with a L4 number spindle and a speed of 100 rpm. The viscosity of mask will appear on the scale in the tool after the stability has been achieved (Rahmawaty et al., 2015). A good viscosity value for peel-off gel masks is 2000-4000 cps (Garg *et al*., 2002).

5. Spreadability Test

   Place 1 gram of mask carefully on a 20x20 cm glass. Then put another glass on it and give the ballast until the weight load reaches 125 grams, then measured the diameter after 1 minute. The best value for spreadability test is about 5-7 cm (Voigt, 1994).

6. Drying Time Test

   Dry time testing is carried out by applying peel-off gel mask to the back of the hand and observing the time required for the mask to dry. The time calculated from the peel-off gel mask is applied until it forms a dry and elastic layer that can be peeled off from the skin surface without leaving the gel mass. The best time needed for masks to dry is no more than 30 minutes (Slavtcheff, 2000).

7. Elasticity Test

   Elasticity testing is done by applying masks on 5x50 mm glass objects. After drying, the preparation is withdrawn and measured to determine the maximum pull that can be achieved until the film survives before breaking (Farmakope V, 2014). The highest percent value of stretch strength will provide comfort in the use of masks because the film layer formed will not easily break when pulled (Ningsih, 2016).

8. Stability Test

   Stability testing is carried out by storing the masks at room temperature for 21 days. Tests were carried out on days 1, 7, 14 and 21. Observations made included organoleptic, homogeneity, viscosity, pH, spreadability, drying time and elasticity (Daswi *et al*., 2018).

2.5. Data Analysis

To find out the characteristics of the preparation, the data obtained in the organoleptic and homogeneity test were analyzed by descriptive method while the data on pH, viscosity, spreadability, drying time and elasticity

test were analyzed by comparing data with the standards for peel-off gel masks. To determine the stability of the preparation, data obtained during 21 days storage were statistically analyzed using the SPSS program. The data obtained were tested for distribution first using the Shapiro-Wilk Test. Normal distributed data is tested using the Paired Samples t Test and data that is not normally distributed is tested using Wilcoxon.

CHAPTER IV

RESULTS AND DISCUSSION

4.1. The Results of Making Peel-Off Gel Masks

At the beginning of the research, the design of the peel-off gel mask formulations used were as follows:

No	Materials	Composition (%)			Function
		F(1)	F(2)	F(3)	
1.	Red Rice Pregelatinized Starch	2	3	4	Active Ingredient and Gelling Agent
2.	Propylene glicol	15	15	15	Humectant
4.	Sodium Benzoat	0,2	0,2	0,2	Preservative
5.	Hydroxypropyl Cellulose (HPC)	4	4	4	Filming Agent
6.	Aquadest	Add 100	Add 100	Add 100	Solvent

Description : F(1) 1st Formula of peel-off gel mask, F(2) 2nd Formula of peel-off gel mask, F(3) 3rd Formula of peel-off gel mask, (Add) Add to

However, apparently the mask produced from the formulation does not meet the standard. All of the masks produced are too liquid and cannot form a film layer. The masks that are too liquid may be caused by a lack of pregelatinized starch used in the formulation. Because the pregelatinized starch in this formulation has a function as a thickening agent (gelling agent) so its utilization can influence the viscosity of the preparation. This is in accordance with the statement of Jacobs and Delcour (1998) who said that starch has amylose content which can form a gel. In addition to gelling agents, materials that can affect the thickness of the preparation are filming agents. The film-forming material used in this formulation is HPC with a concentration of 4%. The cause of not forming the film layer in this formulation is that the HPC used cannot form the film layer on the mask preparation. Therefore, the mask produced cannot form the film layer.

Because of these constraints, changes were made in the formulation of the peel-off gel mask and produced a formula with the ingredients as follows:

No	Materials	Composition (%)			Function
		F(1)	F(2)	F(3)	
1.	Red Rice Pregelatinized Starch	5	10	15	Active Ingredient and Gelling Agent
2.	Propylene glicol	15	15	15	Humectant
4.	Sodium Benzoat	0,2	0,2	0,2	Preservative
5.	Polivynil Alcohol (PVA)	9	9	9	Filming Agent
6.	Aquadest	Add 100	Add 100	Add 100	Solvent

Description : F(1) 1st Formula of peel-off gel mask, F(2) 2nd Formula of peel-off gel mask, F(3) 3rd Formula of peel-off gel mask, (Add) Add to

The concentration of pregelatinized starch used in the above formulations was increased to 5%, 10% and 15%. Because the HPC in the previous formulation could not form the film layer on the preparation, the use of HPC was changed to PVA which according to Ogur (2005), PVA was biodegradable and biocompatible so that PVA was widely used as a film-forming material in topical preparations.

4.2. The Results of Evaluation of Peel-Off Gel Mask

4.2.1. The Results of Organoleptic Test

The results of organoleptic test on the three formulas can be seen in Table 4.1. below :

Table 4.1. The Result of Organoleptic Test

Formula	Observation		
	Colour	Odor	Consistency
F(1)	Light Brown	Aroma of starch	Thick Liquid
F(2)	Light Brown	Aroma of starch	Thick
F(3)	Light Brown	Aroma of starch	Very Thick

Description : F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration F(3) mask formulation with a 15% starch concentration

From the data above it can be seen that the result of organoleptic test on F(1), F(2) and F(3) showed that all of peel-off gel masks has the same color, namely light brown. However, there are differences in the consistency of the colors produced in the three preparations. The dosage color produced in F (3) is thicker than the color produced in F (1) and F (2). This is because red rice has a red pigment called anthocyanin (Chang and Bardenas, 1965). The higher starch concentration used, the higher the amount of anthocyanin contained in the mask and the color of the mask produced is also increasingly thick. Likewise with the level of thickness produced by the mask. All of the three masks have a thick consistency, but the viscosity level at F(3) is higher than the level of thickness in F(1) and F(2). This is because starch has amylose which can form hydrogen bonds when interacting with water (Jacobs and Delcour, 1998). If the concentration of starch used is increasing, the hydrogen bonds between water and amylose will increase too. The more water that is bound in amylose, the thicker preparation is produced. While for aroma, all three have the same aroma namely the distinctive aroma of starch. The distinctive aroma of starch which is owned by masks comes from the 2-acetyl-1-pyroline compound in rice. 2-acetyl-1-pyroline compounds are the main fragrance components possessed by various rice varieties in the world (Buttery et al., 1983). These compounds that make preparations have a distinctive aroma of starch.

4.2.2. The Results of Homogeneity Test

The results of Homogeneity test on the three formulas can be seen on Table 4.2. below :

Table 4.2. The Result of Homogeneity Test

Formula	Homogeneity
F(1)	+
F(2)	+
F(3)	+

Description : (+) homogenous, (-) inhomogenous, F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration, F(3) mask formulation with a 15% starch concentration

The homogeneity tests performed on the three mask preparations showed that all of the masks had good homogeneity. This is indicated by the absence of coarse particles on the mask when applied to transparent glass. Homogeneous preparations indicate that the ingredients used are appropriate because there is no interaction between the ingredients that cause the clumping of materials. In addition, the homogeneity of the preparations also shows that the process of mixing or making masks carried out is in accordance with the procedure because there are no more ingredients that are still clumped or not dissolved. The mask making process is very influential on the homogeneity of the preparation because the wrong manufacturing process will produce a non-homogeneous preparation. The important thing that must be considered in the process of making masks is the stirring process and the use of temperature. The stirring process must be done evenly and constantly so that the ingredients used can be perfectly mixed and the temperature used must also be suitable so that the ingredients used can be dissolved and mixed with other ingredients. Homogeneity of mask preparations is important because it will affect the activity of the active substance contained in the mask (Cahyani *et al*., 2017).

4.2.3. The Results of pH Test

pH testing is carried out to determine the level of safety of the masks when used on the skin. The results of pH on the three formulas can be seen on Table 4.3. below :

Table 4.3. The Results of pH Testing

Formula	Avarage Value ± SD
F(1)	5.9 ± 0.000
F(2)	5.7 ± 0.057
F(3)	5.8 ± 0.000

Description : F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration F(3) mask formulation with a 15% starch concentration (SD) Standard deviation

From the data above, it can be seen that the pH values in preparations F(1), F(2) and F(3) are 5.9; 5.7; 5.8 respectively. The standard pH value for topical preparations that are safe to use for skin according to SNI number 16-4399-1996 is about 4.5-8. Thus it can be concluded that the pH value of the three preparations is still included in the range of permissible pH values. Although there are differences in pH values between preparations, all of the pH value of the mask are still at the same number, which is 5. The pH value of the preparation is influenced by the material used in the formulation. Sulastri et al., (2016) stated that starch has an acidic pH so that the mask produced tend to be acidic. According to Fennema (1996) acidity in starch can be caused due to the cycle of TCA or glycosides that accumulate in plant vacuoles. In addition, acidity can also be caused by the length of the deposition process carried out during the extraction process of starch so that microbes can carry out the fermentation process which produces organic acids and affects the pH of the starch produced.

4.2.4. The Results of Viscosity Test

Viscosity testing is carried out to determine the viscosity value of a preparation. The higher the value of the viscosity that the preparation has, the more resistance of the preparation to flow. The results of the viscosity test performed on the three formulas can be seen in The table 4.4.

Table 4.4. The Results of Viscosity Test

Formula	Average Value ± SD
F(1)	1077 ± 60.11
F(2)	2128 ± 24.75
F(3)	5082 ± 30.23

Description : F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration F(3) mask formulation with a 15% starch concentration (SD) Standard deviation

From the test results, it can be seen that the viscosity values of the three preparations were 1077 cPs, 2128 cPs and 5082 cPs respectively. The standard viscosity for peel off gel masks is at a range of 2000-4000 cPs (Garg et al., 2002). From the test data can be concluded that the viscosity of masks which is suitable with the standard is F(2). While the viscosity of F(1) and F(3) are not in accordance with the standard because the preparation of F(1) is too liquid and the preparation of F(3) is too thick. The difference in viscosity that occurred in the three preparations was due to the difference in concentration of the pregelatinized starch used.

In this formulation, pregelatinized starch has a function as a gelling agent so its utilization can influence the viscosity of the preparation. The characteristic of pregelatinized starch which can directly form a gel when dissolved in water is because the granules contained in it have undergone swelling so that the starch can immediately expand and form a gel when dissolved into water. The higher the concentration of pregelatinized starch used, the higher the

amount of water that will be bound. Moreover, the higher the water that is bound in the starch, the thicker the preparation will produce. Likewise, conversely the less the amount of pragelatinized starch used, the less the amount of water that will be bound and the less the water that is bound in the starch, the more liquid the preparation will produce. The relationship between the addition of pregelatinized starch and viscosity in this study is in accordance with the results of research conducted by Sulastri et al., (2016) which states that the higher the concentration of pregelatinized starch used, the thicker the preparations produced and vice versa. Yuliani (2010) also said that the viscosity in the preparation was also influenced by the increase the concentration of humectants and gelling agents.

4.2.5. The Results of Spreadability Test

Spreadability testing is carried out to determine the ability of masks to spread when applied to the skin. Good gel preparations only need a short time to spread and have a high spread value (Shai et al., 2009). The results of spreadability test on the three formulas can be seen in the Table 4.5.

Table 4.5. The Results of Spreadability Test

Formula	Average Value ± SD
F(1)	7.3 ± 0.288
F(2)	6.0 ± 0.500
F(3)	5.5 ± 0.500

Description : F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration F(3) mask formulation with a 15% starch concentration (SD) Standard deviation

From the table above, it can be seen that the spread value of the three formulas are 7.3 cm, 6 cm and 5.5 cm respectively. A good standard of dispersion values for peel-off gel masks ranges at 5-7 cm (Voigt, 1994). Therefore, it can be concluded that the spreadability of F(2) and F(3) are suitable with the standard criteria while the spreadability of F(1)

is not suitable with the criteria. The difference in spreadability value from the three formulas was due to the different concentration of the pregelatinized starch used. The utilization of pregelatinized starch as a gelling agent can affect the viscosity of the preparation and the viscosity of the preparation can affect the spreadability of the preparation. Madan and Singh (2010) stated that viscosity is a factor that can affect the spreadability and release of active substances from the gel. The results of the research conducted by Ameliawati (2012) also stated that the higher viscosity of the preparation will decrease the spreadability of the preparation and the lower viscosity of the preparation will increase the spreadability of the preparation. According to Wylie (1992), the relationship that occurs between viscosity and spreadability is due to the cohesion between particles in liquid. The higher the viscosity of preparation, the higher cohesion force that occurs in the preparation. The higher the cohesion force that occurs in the preparation, the longer time needed for the preparation to spread.

4.2.6. The Results of Drying Time Test

Testing of drying time is done to find out how long it takes for the mask to dry and form a film layer. The test results of the drying time of the three formulas can be seen in the Table 4.6. below :

Table 4.6. The Results of Drying Time Test

Formula	Average Value ± SD
F(1)	31 ± 3.605
F(2)	16 ± 2.645
F(3)	14 ± 1.000

Description : F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration F(3) mask formulation with a 15% starch concentration (SD) Standard deviation

From the test results it can be seen that the drying time of the three preparations were 31 minutes, 16 minutes and 14 minutes, respectively. The standard drying time for mask preparations is no more

than 30 minutes (Slavtcheff, 2000). Hence, it can be concluded that the drying time of preparations which is suitable with the criteria are F(2) and F(3). Meanwhile the drying time on F(1) is not in accordance with the criteria. The difference in drying time of the three formulas was due to the difference in concentration of the pregelatinized starch used. The higher the concentration of pragelatinized starch used, the higher viscosity of the preparation produced. The higher the viscosity of the preparation, the less water contained in the preparation. The less water contained in the preparation, the faster time needed for the preparation to dry out and vice versa. The relationship between the addition of pregelatinized starch and drying time in this study is in accordance with the results of research conducted by Sulastri et al., (2016) which states that the addition of pregelatinized starch to the formula affects the time of preparation to dry.

4.2.7. The Results of Elasticity Test

The elasticity testing is done to determine the strain ability of the mask so that exfoliating the mask from the face will not make the person feel pain. The results of the elasticity test performed on all three formulas can be seen in the Table 4.7. below :

Table 4.7. The Results of Elasticity Test

Formula	Average Value ± SD
F(1)	9.5 ± 0.500
F(2)	11 ± 2.783
F(3)	10 ± ٠,٧٦٣

Description : F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration F(3) mask formulation with a 15% starch concentration (SD) Standard deviation

From the table above, it can be seen that the three preparations have stretch elasticity of 9.5 cm, 11 cm and 10 cm. For testing elasticity there is no specific standard that determines whether the elasticity of a preparation is good or not. However, the higher the value of elasticity

of the preparation, the higher the comfort that is given when using masks because the mask that has high elasticity values will not be easily broken when exfoliated (Ningsih, 2016).

The results of this study are different from the results of a study conducted by Cahyani and Putri (2017) on the effectiveness of using carbopol in peel-off gel masks from white tumeric extracts. In this study it was found that the higher the concentration of gelling agent used, the more elastic the preparation produced. The gelling agent used in the Cahyani and Putri (2017) study was carbopol 940 while in this study the gelling agent used was pregelatinized starch. This difference in the use of gelling agent causes differences in the results of the elasticity produced. Carbopol 940 has an elastic gel form so that if the concentration of its use is increased it can affect the elasticity of the preparation. Pregelatinized starch has a gel form that is not elastic so that the addition of the concentration of its use also does not affect the elasticity of the preparation.

Nofiandi et al., (2016) said that the use of starch as a natural polymer has limitations such as producing poor mechanical properties. For this reason, its use needs to be combined with other ingredients. One of them is PVA which has good mechanical properties and is able to cover the deficiency of starch. The results of this study indicate that the formulation that can produce the best elasticity is preparation F(2). This shows that the ratio of the concentration of the material used in F(2) is appropriate so it can produce preparations that have good elasticity. The preparation of F(1) produces elasticity with a shorter extension compared to F(2) because the concentration of pregelatinized starch is less so that the resulting preparation is too liquid and has a large water content that can affect the elasticity of the preparation. Meanwhile the preparation F(3) uses pregelatinized starch with more concentration and produce preparations that are thick and have little water content so that the resulting elasticity is not good.

4.2.8. The Results of Stability Test Peel-Off Gel Mask

Stability testing is carried out to determine the stability or physical resistance of the preparation during storage. Preparation storage was carried out for 21 days with tests carried out on days 1, 7, 14 and 21 (Daswi et al., 2018). The results of organoleptic stability testing and homogeneity of preparations can be seen in Table 4.8. and the results of testing the stability of pH, viscosity, spreadability, elasticity and drying time can be seen in the Table 4.9.

Table 4.8. The Result of Stability of Organoleptic and Homogeneity Test

Formula	Parameter	Duration of Storage (Days)			
		1	7	14	21
F(1)	Colour	Light Brown	Light Brown	Light Brown	Light Brown
	Odor	Aroma of Starch	Aroma of Starch	Aroma of Starch	Aroma of Starch
	Consistency	Liquid Thick	Liquid Thick	Liquid Thick	Liquid Thick
	Homogeneity	Inhomogenous	Inomogenous	Inhomogenous	Inhomogenous
F(2)	Colour	Light Brown	Light Brown	Light Brown	Light Brown
	Odor	Aroma of Starch	Aroma of Starch	Aroma of Starch	Aroma of Starch
	Consistency	Thick	Thick	Thick	Thick
	Homogeneity	Homogenous	Homogenous	Homogenous	Homogenous

F(3)	Colour	Light Brown	Light Brown	Light Brown	Light Brown
	Odor	Aroma of Starch	Aroma of Starch	Aroma of Starch	Aroma of Starch
	Consistency	Very Thick	Very Thick	Very Thick	Very Thick
	Homogeneity	Homogenous	Homogenous	Homogenous	Homogenous

Description : F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration F(3) mask formulation with a 15% starch concentration

The results of organoleptic testing showed that the color, odor and consistency of mask before and after storage for 21 days are the same. It has light brown colour, aroma of starch and thick in consistency. It shows that the preparation of peel-off gel mask is physically stable during storage for 21 days. The stability of a preparation can be influenced by the effectiveness of the preservatives used. The preservative used in this formulation is sodium benzoate which has been proven effective because no fungus grows in the preparation and it does not damage the color and aroma of the preparation.

Homogeneity testing was carried out on the three formulas. It obtained the results that preparations F(2) and F(3) were stable during storage and they are homogeneous. Meanwhile, the preparation F(1) has a precipitation or called flocculation. Flocculation began to occur on day 2 of storage with deposition length of 0.5 cm. Precipitation continues until the 7^{th} day and the length of precipitation reaches 1.5 cm. Flocculation only occurs in preparations F(1). According to Priyambodo (2007), one of the factors causing flocculation is the lack of viscosity of the preparation. The higher the viscosity of a preparation, the smaller rate of precipitation in the preparation. To make the preparation homogeneous and mixed again, it needs to be stirred for about 3 minutes.

Table 4.9. The Results of pH, Viscosity, Spreadability, Drying Time, and Elasticity Stability Test

Evaluation	Duration of Storage (Days)	Average Value ± SD		
		F(1)	F(2)	F(3)
pH	1	5.9 ± 0.000	5.7 ± 0.057	5.8 ± 0.000
	7	6.0 ± 0.000	5.8 ± 0.057	5.8 ± 0.057
	14	6.1 ± 0.000	5.9 ± 0.057	5.8 ± 0.057
	21	6.0 ± 0.000	5.9 ± 0.000	5.7 ± 0.000
Viscosity (cP)	1	1077 ± 60.11	2128 ± 24.75	5082 ± 30.23
	7	1054 ± 14.04	2028 ± 25.53	5023 ± 25.23
	14	1131 ± 26.76	2154 ± 21.38	5049 ± 11.67
	21	1050 ± 32.18	2030 ± 30.50	5039 ± 19.03
Spreadability (cm)	1	7.3 ± 0.288	6.0 ± 0.500	5.5 ± 0.500
	7	6.5 ± 0.300	6.0 ± 0.513	6.1 ± 0.288
	14	6.8 ± 0.288	6.2 ± 0.251	5.4 ± 0.100
	21	6.6 ± 0.100	6.0 ± 0.152	5.4 ± 0.057
Drying Time (minute)	1	31 ± 3.605	16 ± 2.645	14 ± 1.000
	7	28 ± 2.886	18 ± 1.527	15 ± 1.154
	14	22 ± 2.516	20 ± 1.527	15 ± 1.527
	21	23 ± 2.645	18 ± 1.527	15 ± 2.516
Elasticity (cm)	1	9.5 ± 0.500	11 ± 2.783	10 ± ٠,٧٦٣
	7	10 ± 2.136	4.3 ± 0.288	12 ± 5.658
	14	10 ± 2.466	10 ± 3.055	6.6 ± 2.081
	21	10 ± 1.527	9.3 ± 2.007	11 ± 2.081

Description : F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration F(3) mask formulation with a 15% starch concentration

Based on the results of pH testing during storage for 21 days, the pH values in the three preparations ranged from 5.7 to 6.1 (Table 4.9). Although there are differences in pH values during storage, all the pH value are still included in the range of pH values that are allowed for topical preparations. Topical preparation should not have a pH value that is too acidic or too alkaline, because a pH value that is too

acidic will irritate the skin and a pH value that is too alkaline will make the skin dry.

Table 4.10. The Statistic Analysis Results for pH Stability

Formula	Comparation	Sig
F(1)	1st Day vs 7th Day	0.102
	1st Day vs 14th Day	0.083
	1st Day vs 21st Day	0.083
F(2)	1st Day vs 7th Day	0.083
	1st Day vs 14th Day	0.102
	1st Day vs 21st Day	0.102
F(3)	1st Day vs 7th Day	0.317
	1st Day vs 14th Day	0.317
	1st Day vs 21st Day	0.083

Description : F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration F(3) mask formulation with a 15% starch concentration

Data of pH values during storage for 21 days then analyzed statistically to determine the effect of storage on the pH stability of the preparation. Before testing, the data were tested first for normality to find out whether the data was normally distributed or not. Data that is normally distributed will be tested using the Paired Samples t Test and data that are not normally distributed will be tested using Wilcoxon. The statistical analysis results for pH stability test can be seen in Table 4.10. The results of the test showed that the data of pH value for all preparation during storage does not differ significantly ($p > 0.05$) which means that the pH of the three preparations was stable for storage 21 days at room temperature.

Table 4.11. The Statistical Analysis Results for Viscosity Stability Test

Formula	Comparation	Sig
F(1)	1st Day vs 7th Day	0.507
	1st Day vs 14th Day	0.165
	1st Day vs 21st Day	0.658

F(2)	1st Day vs 7th Day	0.070
	1st Day vs 14th Day	0.421
	1st Day vs 21st Day	0.059
F(3)	1st Day vs 7th Day	0.244
	1st Day vs 14th Day	0.364
	1st Day vs 21st Day	0.301

Description : F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration F(3) mask formulation with a 15% starch concentration

The results of viscosity test during storage for 21 days showed that the value of the viscosity of the preparation F(1) ranged between 1050-1131 cPs, preparations F(2) ranged from 2128-2030 cPs and preparations F(3) ranged from 5023-5082 cPs (table 4.9) Data obtained during storage for 21 days then analyzed statistically to determine the effect of storage on the stability of the viscosity of the preparation. Before testing, the data were tested first for normality to find out whether the data was normally distributed or not. Data that is normally distributed will be tested using the Paired Samples t Test and the data that are not normally distributed are tested using Wilcoxon. The statistical analysis results for viscosity stability test can be seen in Table 4.11. The results of the tests showed that the data on the viscosity of the three preparations during storage did not differ significantly ($p > 0.05$) which means that the viscosity of the three preparations was stable for storage 21 days at room temperature.

Table 4.12. The Statistical Analysis Results for Spreadability Stability Test

Formula	**Comparation**	**Sig**
F(1)	1st Day vs 7th Day	0.109
	1st Day vs 14th Day	0.180
	1st Day vs 21st Day	0.109

F(2)	1st Day vs 7th Day	0.900
	1st Day vs 14th Day	0.371
	1st Day vs 21st Day	0.914
F(3)	1st Day vs 7th Day	0.157
	1st Day vs 14th Day	0.593
	1st Day vs 21st Day	0.655

Description : F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration F(3) mask formulation with a 15% starch concentration

The results of spreadability test during storage for 21 days indicate that the spread value of F(1) ranges from 6.5-7.3 cm, F(2) ranges from 6-6.2 cm and F(3) ranges from 5.4 -6.1 cm (Table 4.9). Data obtained during storage then analyzed statistically to determine the effect of storage on the stability of spreadability preparation. Before testing, the data were tested first for normality to find out whether the data was normally distributed or not. Data that is normally distributed will be tested using the Paired Samples t Test and data that are not normally distributed are tested using Wilcoxon. The statistical analysis results of the spreadability stability test can be seen in Table 4.12. The results of the tests showed that the data of the spreadability of the three preparations during storage did not differ significantly ($p > 0.05$). It means that the spreadability of the three preparations was stable during storage for 21 days at room temperature.

Table 4.13. The Statistical Analysis Results for Drying Time Stability Test

Formula	Comparation	Sig
F(1)	1st Day vs 7th Day	0.276
	1st Day vs 14th Day	0.109
	1st Day vs 21st Day	0.109
F(2)	1st Day vs 7th Day	0.109
	1st Day vs 14th Day	0.109
	1st Day vs 21st Day	0.285

F(3)	1st Day vs 7th Day	0.102
	1st Day vs 14th Day	0.317
	1st Day vs 21st Day	0.276

Description : F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration F(3) mask formulation with a 15% starch concentration

The results of drying time test during storage for 21 days showed that the value of drying time preparation of F(1) ranged from 22-31 minutes, preparation F(2) ranged from 16-20 minutes and preparation F(3) ranged from 14-15 minutes (table 4.9). Data obtained during storage then analyzed statistically to determine the effect of storage on the stability of drying time the preparation. Before testing, the data were tested first for normality to find out whether the data was normally distributed or not. Data that is normally distributed will be tested using the Paired Samples t Test and data that are not normally distributed are tested using Wilcoxon. The statistic analysis results for drying time stability test can be seen in table 4.13. The results of the tests showed that the data on the drying time of the three preparations during storage did not differ significantly ($p > 0.05$) which means that the time to dry the three preparations was stable for storage 21 days at room temperature.

Table 4.14. The Statistical Analysis Results for Elasticity Stability Test

Formula	**Comparation**	**Sig**
F(1)	1st Day vs 7th Day	0.432
	1st Day vs 14th Day	0.625
	1st Day vs 21st Day	0.423
F(2)	1st Day vs 7th Day	0.109
	1st Day vs 14th Day	1.000
	1st Day vs 21st Day	0.109

F(3)	1st Day vs 7th Day	0.222
	1st Day vs 14th Day	0.086
	1st Day vs 21st Day	0.050

Description : F(1) mask formulation with a 5% starch concentration, F(2) mask formulation with a 10% starch concentration F(3) mask formulation with a 15% starch concentration

The results of elasticity test during storage for 21 days showed that the elasticity of preparation F(1) ranged from 9.5-10 cm, preparations F(2) ranged from 4.3 to 11 cm and preparations F(3) ranged from 6.6- 12 cm (see Table 4.9). Data obtained during storage then analyzed statistically to determine the effect of storage on the stability of the elasticity preparation. Before testing, the data were tested first for normality to find out whether the data was normally distributed or not. Data that is normally distributed will be tested using the Paired Samples t Test and data that are not normally distributed are tested using Wilcoxon. The results of elasticity stability testing can be seen in Table 4.14. The statistic analysis results for elasticity stability test showed that the elasticity data of the three preparations during storage did not differ significantly ($p > 0.05$) which means that the elasticity of the three preparations was stable for storage 21 days at room temperature.

From the results of stability test for three preparations, it showed that the peel-off gel mask was stable for 21 days of storage at room temperature both from organoleptic, homogeneity, pH, viscosity, spreadability, drying time and elasticity. This indicates that the mask material used in the formulation was dissolved and completely mixed especially the preservatives used that are sodium benzoate which has been proven to be effective because there are no fungi that grow in the preparation. In addition to preservatives, other ingredients that help maintain the stability of the preparation are humectants. Rowe et al., (2006) said that in the formulation, propylenglycol was added as a

humectant to maintain the stability of the preparation through moist absorption from the environment and prevent evaporation of water from the preparation so that the viscosity will be maintained. If the viscosity of the preparation is stable, the spreadability and drying time of the preparation will also be stable. Some other things that can also affect the stability of the preparation are the storage temperature and the interaction between preparation with air (O_2 and CO_2).

4.3. The Best Formulation of Peel-Off Gel Masks

The best formulation of peel-off gel mask is determined from the results of tests performed on all three preparations. The test results showed that the best formulation for making peel off gel masks was F(2) preparations using pregelatinized starch with a concentration of 10%. This can be seen from the characteristics possessed by the preparation F(2) in Table 4.15.

Table 4.15. The Characteristics of F(2)

Evaluation	**Result**	**Standard**
Organoleptic : Colour Odor Consistency	 Light Brown Aroma of Starch Thick	
Homogeneity	Homogenous	Homogenous
pH	5.7	4,5 – 8
Viscosity	2128 cPs	2000-4000 cPs
Spreadability	6 cm	5-7 cm
Drying Time	16 minute	15-30 minute
Elasticity	11 cm	More longer is better

The result of organoleptic test for preparations F(2) shows that the preparations have a light brown colour, aroma of starch and thick consistency. The results of the pH test indicate that this preparation has a pH of 5.7 which means that the preparation of F(2) is safe to use for the skin because the pH value is in accordance with the physiological pH of the skin. The viscosity possessed by preparation F(2) is also in accordance with the standard, which is 2128 cPs. The preparation of a peel-off gel mask should not be too thick because the preparations that are too thick will affect the diffusion of the active substance to get out of the mask base. It is in accordance with research conducted by Cahyani et al., (2017) which stated that the higher the viscosity of the preparation, the antibacterial activity produced will be lower. The amount of dispersion possessed by preparation F (2) is also appropriate, which is equal to 6 cm. The smaller the value of the

spread of a preparation, the more difficult the preparation to spread when applied to the skin and vice versa. The time needed for preparation F (2) to dry for 16 minutes. The preparation F (2) has elasticity with a strain value of 11 cm.

4.4. The Results of Halal Identification of Peel-Off Gel Masks

The peel-off gel mask can be said to be a halal product if the materials and tools used and the manufacturing process that is carried out are not in contact or comes from haram ingredients. Halal identification results of peel-off gel mask can be seen in Table 4.16 below :

Table 4.16. Halal Identification of Peel-Off Gel Mask

Identification		Qualification Result	
		Halal	**Haram**
Materials of Mask	Red Rice Starch	√	
	Polivynil Alcohol	√	
	Propilene glicol	√	
	Sodium Benzoat	√	
	Aquades	√	
Manufacturing Process	Extraction of Starch	√	
	Manufacture of Pregelatinized Starch	√	
	Manufacture of Mask	√	
Tools	Extraction of Starch	√	
	Manufacture of Pregelatinized Starch	√	
	Manufacture of Mask	√	

Based on the table above, it can be concluded that the peel-off gel mask is made from halal ingredients. The basic material for making masks comes from brown rice which is included in plants that are halal. Brown rice is obtained from the Cirebon area, West Java. All ingredients like Polyvinyl alcohol (PVA), Propilene glicol, Sodium benzoate and distilled water are halal ingredients for use in Islam. This is according to the decision letter of the MUI Food, Drug and Cosmetics Study Number: SK07 / Dir / LPPOM MUI / I / 13-rev1 2015 which discusses the list of non-critical

materials. In this letter it has been stated that propylene glycol, polyvinyl alcohol, sodium benzoate and aquades are included in non-critical and halal materials for use.

In the manufacturing process, starting from the extraction process to making masks there is nothing that involves or is mixed with haram ingredients. The solvents used in the extraction process, starch pregelatinization and making masks also came from distilled water which is halal material. The tools used are also not contaminated with haram ingredients. The process of making masks is not mixed with haram ingredients. Hence, it can be concluded that the peel-off gel mask which is made from pregelatinized starch as a gelling agent can be qualitified as halal.

CHAPTER V

CLOSING

5.1. Conclusions

The conclusions that can be drawn from this study are:

1. The appropriate formulations for making peel-off gel masks with red rice pregelatinized starch as gelling agent are using 9% PVA, 15% propylene glycol, 0.2% sodium benzoate and distilled water.
2. The characteristics produced from the formulation of peel-off gel masks with red rice pregelatinized starch as a gelling agent has a light brown colour, aroma of starch, and thick in shape. In F(1), the preparation had a pH value of 5.9, the viscosity of 1077 cPs, spreadability of 7.3 cm, drying time of 31 minutes and elasticity of 9.5 cm. In F (2), the preparation has a pH value of 5.7, viscosity of 2128 cPs, spreadability of 6 cm, drying time of 16 minutes and elasticity of 11 cm. In F (3), the preparation has a pH value of 5.5, the viscosity of 5082 cPs, the spreadability of 5.5 cm, the drying time of 14 minutes and the elasticity of 10 cm.
3. The best concentration of pregelatinized starch used in the formulation of peel off gel masks is the preparation of F (2) with a starch concentration of 10%.

5.2. Suggestions

Further testing needs to be carried out such as the sticky test and the hedonic test to improve the quality of the mask produced. In addition, antioxidant testing is also needed to determine the activity of antioxidant compounds found in red rice starch.

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APPENDICES

Appendix 1. Determination of Plant

PEMERINTAH PROVINSI JAWA TIMUR
DINAS KESEHATAN
UPT LABORATORIUM HERBAL MATERIA MEDICA BATU
Jalan Lahor No.87 Telp. (0341) 593396
KOTA BATU 65313

Nomor : 074/ 344A/ 102.7/ 2019
Sifat : Biasa
Perihal : **Determinasi Tanaman Beras Merah**

Memenuhi permohonan saudara :

Nama : MAFAZATIEN NAILIYAH ISNA
NIM : 362015711132
Fakultas : ILMU KESEHATAN, PROGRAM STUDI FARMASI
UNIVERSITAS DARUSSALAM GONTOR

1. Perihal determinasi tanaman padi (beras merah)
   - Divisi : Spermatophyta
   - Sub divisi : Angiospermae
   - Kelas : Monocotyledonae
   - Bangsa : Poales
   - Suku : Graminae
   - Marga : Oryza
   - Jenis : *Oryza nivara* S .D. Sharma & Shastry
   - Nama Umum : Beras merah.
   - Kunci determinasi : 1b-2b-3b-4a-5a-2c-18b-20a-21b-22b.
2. Morfologi : Terna semusim, berakar serabut, batang sangat pendek, struktur serupa batang terbentuk dari rangkaian pelepah daun yang saling menopang daun sempurna dengan pelepah tegak, daun berbentuk lanset, warna hijau muda hingga hijau tua, berurat daun sejajar, tertutupi oleh rambut yang pendek dan jarang, bagian bunga tersusun majemuk, tipe malai bercabang, satuan bunga disebut floret yang terletak pada satu spikelet yang duduk pada panikula, tipe buah bulir atau kariopsis yang tidak dapat dibedakan mana buah dan bijinya, bentuk hampir bulat hingga lonjong, ukuran 3mm hingga 15mm, tertutup oleh palea dan lemma yang dalam bahasa sehari-hari disebut sekam, struktur dominan padi yang biasa dikonsumsi yaitu jenis endospermium.
3. Nama Simplisia : Oryzae nivarae Amylum / Pati Beras Merah.
4. Kandungan : Serat, karbohidrat, vitamin B, magnesium, fosfor, kalsium, kalium, zinc, dan selenium.
5. Penggunaan : Penelitian.
6. Daftar Pustaka
   - Van Steenis, CGGJ. 2008. *FLORA, untuk Sekolah di Indonesia*. Pradnya Paramita, Jakarta.

Demikian surat keterangan determinasi ini kami buat untuk dipergunakan sebagaimana mestinya.

Batu, 22 April 2019
Kepala UPT Lab. Herbal Materia Medica Batu

Dr. Husin R.M., Drs., Apt., M.Kes.
NIP. 199103 1 003

Appendix 2. Extraction of Starch

Wheighing the rice

Wash the rice with water

Blend the red rice

Mix with aquadest

Filter with batiste

Wait until 24 hours

Filter the sediment with filter paper

Oven it at 50°C

The dried starch

Appendix 3. Manufacture of Pregelatinized Starch

Determine of Gelatinization Temperature

Suspension of starch 5% b/v

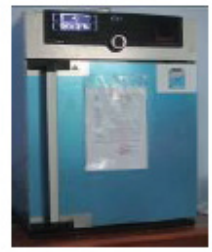
Heat the paste until thick

Measuring the temperature

Manufacture of Pregelatunized Starch

Suspension of starch 5% b/v

Heat the suspension at 75°C



Drying the starch in oven at 50°C

Smooth the star

Pregelatinized starch

Appendix 4. The Statistical Analysis Result of pH Stability

a. pH Stability of F(1)

Tests of Normality[b,c,d]

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 7	385.	3	.	750.	3	000.

a. Lilliefors Significance Correction
.b. Day 1 is constant. It has been omitted
.c. Day 14 is constant. It has been omitted
.d. Day 21 is constant. It has been omitted

The Result of Wilcoxon Test

Test Statistics[b]

	Day 7 - Day 1	Day 14 - Day 1	Day 21 - Day 1
Z	-1.633[a]	-1.732[a]	-1.732[a]
(Asymp. Sig. (2-tailed	102.	083.	083.

.a. Based on negative ranks
b. Wilcoxon Signed Ranks Test

b. pH Stability of F(2)

Tests of Normality[b]

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 1	385.	3	.	750.	3	000.
Day 7	385.	3	.	750.	3	000.
Day 14	385.	3	.	750.	3	000.

a. Lilliefors Significance Correction
.b. Day 21 is constant. It has been omitted

The Result of Wilcoxon Test

Test Statistics[b]

	Day 7 - Day 1	Day 14 - Day 1	Day 21 - Day 1
Z	-1.732[a]	-1.633[a]	-1.633[a]
(Asymp. Sig. (2-tailed	083.	102.	102.

.a. Based on negative ranks
b. Wilcoxon Signed Ranks Test

c. pH Stability of F(3)

Tests of Normality[b,c]

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 7	385.	3	.	750.	3	000.
Day 14	385.	3	.	750.	3	000.

a. Lilliefors Significance Correction

.b. Day 1 is constant. It has been omitted

.c. Day 21 is constant. It has been omitted

The Result of Wilcoxon Test

Test Statistics[c]

	Day 7 - Day 1	Day 14 - Day 1	Day 21 - Day 1
Z	-1.000[a]	-1.000[a]	-1.732[b]
(Asymp. Sig. (2-tailed	317.	317.	083.

.a. Based on negative ranks

.b. Based on positive ranks

c. Wilcoxon Signed Ranks Test

Appendix 5. The Statistical Analysis Result of Viscosity Stability

a. Viscosity Stability of F(1)

Tests of Normality

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 1	367.	3	.	792.	3	095.
Day 7	204.	3	.	993.	3	843.
Day 14	275.	3	.	944.	3	542.
Day 21	289.	3	.	928.	3	480.

a. Lilliefors Significance Correction

Paired Samples Test

		Paired Differences					t	df	Sig. ((2-tailed
		Mean	Std. Deviation	Std. Error Mean	Confidence Interval of 95% the Difference Lower	Upper			
Pair 1	Day 1 - Day 7	2.30000E1	49.72927	28.71121	-100.53435	146.53435	801.	2	507.
Pair 2	Day 1 - Day 14	-5.40000E1	43.55456	25.14624	-162.19553	54.19553	-2.147	2	165.
Pair 3	Day 1 - Day 21	2.73333E1	91.88217	53.04820	-200.91463	255.58130	515.	2	658.

b. Viscosity Stability of F(2)

Tests of Normality

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 1	309.	3	.	901.	3	388.
Day 7	260.	3	.	959.	3	609.
Day 14	253.	3	.	964.	3	637.
Day 21	176.	3	.	1.000	3	982.

a. Lilliefors Significance Correction

Paired Samples Test

		Paired Differences					t	df	Sig. ((2-tailed
		Mean	Std. Deviation	Std. Error Mean	Confidence Interval 95% of the Difference Lower	Upper			
Pair 1	Day 1 - Day 7	1.00000E2	48.56954	28.04164	-20.65342	220.65342	3.566	2	070.
Pair 2	Day 1 - Day 14	-2.66667E1	46.04708	26.58529	-141.05395	87.72061	-1.003	2	421.
Pair 3	Day 1 - Day 21	9.76667E1	42.82912	24.72740	-8.72676	204.06009	3.950	2	059.

c. Viscosity Stability of F(3)

Tests of Normality

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 1	263.	3	.	956.	3	595.
Day 7	261.	3	.	957.	3	602.
Day 14	246.	3	.	970.	3	668.
Day 21	195.	3	.	996.	3	884.

a. Lilliefors Significance Correction

Paired Samples Test

		Paired Differences					t	df	(Sig. (2-tailed
		Mean	Std. Devi-ation	Std. Error Mean	Confidence Inter- 95% val of the Difference Lower	Upper			
Pair 1	Day 1 - Day 7	5.30000E1	56.20498	32.44996	-86.62092	192.62092	1.633	2	244.
Pair 2	Day 1 - Day 14	2.96667E1	44.07191	25.44493	-79.81403	139.14736	1.166	2	364.
Pair 3	Day 1 - Day 21	3.96667E1	49.69239	28.68991	-83.77607	163.10940	1.383	2	301.

Appendix 6. The Statistical Analysis Result of Spreadability Stability

a. Spreadability Stability of F(1)

Tests of Normality

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 1	385.	3	.	750.	3	000.
Day 7	175.	3	.	1.000	3	1.000
Day 14	385.	3	.	750.	3	000.
Day 21	175.	3	.	1.000	3	1.000

a. Lilliefors Significance Correction

The Result of Wilcoxon Test

Test Statistics[b]

	Day 7 - Day 1	Day 14 - Day 1	Day 21 - Day 1
Z	-1.604[a]	-1.342[a]	-1.604[a]
(Asymp. Sig. (2-tailed	109.	180.	109.

.a. Based on positive ranks

b. Wilcoxon Signed Ranks Test

b. Spreadability Staility of F(2)

Tests of Normality

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 1	175.	3	.	1.000	3	1.000
Day 7	269.	3	.	949.	3	567.
Day 14	219.	3	.	987.	3	780.
Day 21	253.	3	.	964.	3	637.

a. Lilliefors Significance Correction

Paired Samples Test

		Paired Differences					t	df	Sig. ((2-tailed
		Mean	Std. Deviation	Std. Error Mean	Confidence Interval of the Difference 95% Lower	Upper			
Pair 1	Day 1 - Day 7	06667.-	81445.	47022.	-2.08988	1.95655	142.-	2	900.
Pair 2	Day 1 - Day 14	26667.-	40415.	23333.	-1.27062	73729.	-1.143	2	371.
Pair 3	Day 1 - Day 21	03333.-	47258.	27285.	-1.20729	1.14062	122.-	2	914.

c. Spreadability Stability of F(3)

Tests of Normality

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 1	175.	3	.	1.000	3	1.000
Day 7	385.	3	.	750.	3	000.
Day 14	175.	3	.	1.000	3	1.000
Day 21	385.	3	.	750.	3	000.

a. Lilliefors Significance Correction

The Result of Wilcoxon Test

Test Statistics[c]

	Day 7 - Day 1	Day 14 - Day 1	Day 21 - Day 1
Z	-1.414[a]	535[b].-	447[b].-
(Asymp. Sig. (2-tailed	157.	593.	655.

.a. Based on negative ranks

.b. Based on positive ranks

c. Wilcoxon Signed Ranks Test

Appendix 7. The Statistical Analysis Result of Drying Time Stability

a. Drying Time Stability of F(1)

Tests of Normality

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 1	276.	3	.	942.	3	537.
Day 7	385.	3	.	750.	3	000.
Day 14	219.	3	.	987.	3	780.
Day 21	314.	3	.	893.	3	363.

a. Lilliefors Significance Correction

The Result of Wilcoxon Test

Test Statistics[b]

	Day 7 - Day 1	Day 14 - Day 1	Day 21 - Day 1
Z	-1.089[a]	-1.604[a]	-1.604[a]
(Asymp. Sig. (2-tailed	276.	109.	109.

.a. Based on positive ranks

b. Wilcoxon Signed Ranks Test

b. Drying Time Stability of F(2)

Tests of Normality

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 1	314.	3	.	893.	3	363.
Day 7	253.	3	.	964.	3	637.
Day 14	253.	3	.	964.	3	637.
Day 21	253.	3	.	964.	3	637.

a. Lilliefors Significance Correction

The Result of Wilcoxon Test

Test Statistics[b]

	Day 7 - Day 1	Day 14 - Day 1	Day 21 - Day 1
Z	-1.604[a]	-1.604[a]	-1.069[a]
(Asymp. Sig. (2-tailed	109.	109.	285.

.a. Based on negative ranks

b. Wilcoxon Signed Ranks Test

Appendix 8. The Statistical Analysis Result of Elasticity Stability

a. Elasticity Stability of F(1)

Tests of Normality

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 1	175.	3	.	1.000	3	1.000
Day 7	324.	3	.	877.	3	314.
Day 14	349.	3	.	832.	3	194.
Day 21	253.	3	.	964.	3	637.

a. Lilliefors Significance Correction

Paired Samples Test

		Paired Differences					t	df	Sig. ((2-tailed
		Mean	Std. Devi-ation	Std. Error Mean	Confidence Inter- 95% val of the Difference Lower	Upper			
Pair 1	Day 1 - Day 7	-1.43333	2.54231	1.46780	-7.74878	4.88211	977.-	2	432.
Pair 2	Day 1 - Day 14	66667.-	2.02073	1.16667	-5.68643	4.35309	571.-	2	625.
Pair 3	Day 1 - Day 21	83333.-	1.44338	83333.	-4.41888	2.75221	-1.000	2	423.

b. Elasticity Stability of F(2)

Tests of Normality

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 1	238.	3	.	976.	3	702.
Day 7	385.	3	.	750.	3	000.
Day 14	253.	3	.	964.	3	637.
Day 21	340.	3	.	849.	3	238.

a. Lilliefors Significance Correction

The Result of Wilcoxon Test

Test Statistics[c]

	Day 7 - Day 1	Day 14 - Day 1	Day 21 - Day 1
Z	-1.604[a]	000[b].	-1.604[a]
(Asymp. Sig. (2-tailed	109.	1.000	109.

.a. Based on positive ranks

.b. The sum of negative ranks equals the sum of positive ranks

c. Wilcoxon Signed Ranks Test

c. Elasticity Stability of F(3)

Tests of Normality

	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	Statistic	df	.Sig	Statistic	df	.Sig
Day 1	292.	3	.	923.	3	463.
Day 7	273.	3	.	945.	3	548.
Day 14	253.	3	.	964.	3	637.
Day 21	292.	3	.	923.	3	463.

a. Lilliefors Significance Correction

Paired Samples Test

		Paired Differences					t	df	Sig. ((2-tailed
		Mean	Std. Deviation	Std. Error Mean	Confidence Interval of the Difference 95% Lower	Upper			
Pair 1	Day 1 - Day 7	-6.10000	6.03987	3.48712	-21.10386	8.90386	-1.749	2	222.
Pair 2	Day 1 - Day 14	-4.00000	2.17945	1.25831	-9.41405	1.41405	-3.179	2	086.
Pair 3	Day 1 - Day 21	-5.16667	2.08167	1.20185	-10.33781	00448.	-4.299	2	050.