THE MAKING OF DYE POWDER OF DUWET (*Syzygium cuminii*) RIND BY USING SPRAY DRYER METHOD AND THE STABILITY DURING THE STORING

Suci Nurdiastuti[1] ; Sukatiningsih; and Siti Hartanti

Abstract

Along with the development of food processing industry, the use of dye at food processing industry also increases. Most of the dye used is synthetic, both liquid and powder, which can generate the problem of health. To reduce the risk, the use in foodstuff begins omitted and changes over into the use of natural dye. However, the limited amount of natural substance is one of the reasons to look for natural dye source of Indonesian typical plant, such as duwet. All this time, duwet (*Syzygium cuminii*) is not optimally exploited yet, so by using this fruit as the staple of dye will be able to improve the economic value of it. The most economic and efficient powder drying method is dryer spray method with the addition of filler substance of maltodextrin 10%, 15%, and 20%. It is caused the storing of anthocyanins in a long time and high temperature is easy spoiled. This research is conducted to know the process of making duwet rind dye powder, the characteristic of dye powder which is produced, and the stability level during the storing. In this research, the extraction of duwet rind is conducted by using the combination of solvent water : etanol (1:1). The obtained extract is combined with filler substance of maltodextrin 10%, 15%, and 20%. This mixture is dried using spray dryer. The result of this process is dye powder. Then, its characteristics are observed, enclose total yield, water content, ash content, solubility, hygroscopicity, colour, quantity of anthocyanins, losing of anthocyanins during drying and analyzing the stability during the storing. The results of research of dye powder are: total yield 80.35%-88.33%; water content 5.46%-6.74%; ash content 0.22%-0.48%; temperatures solubility 27^0C (98.82%-99.58%) and temperatures 40^0C (99.49%-99.89%); hygroscopicity 22.02 g / 100g-25.36 g / 100g; the colour tends to reddish blue (purple); quantity of anthocyanins 3.72 mg / mg g-8.93 / g; and drying loss 16.73%. For the stability of dye powder during the storing which is done at frozen temperature and room temperature (RH 32%) indicate that the endurance stability of anthocyanins during the storing at frozen temperature more stable than at RH 32%. Degradation rate ranged 0.002 week^{-1} x (10^{-1}) - 0.030 week^{-1} x(10^{-1}) at frozen temperature and 0.003-0.016 week^{-1} x (10^{-1}) at room temperature (RH 32%). Half-life 22.796 - 407.647 week at frozen temperature and 44.710-238.966 week at room temperature (RH 32%).

Keywords: *Anthocyanin; Duwet rind; Maltodextrin; Dye powder; Stability of storing.*

Introduction

Colour is the first sensory parameters which are observed when consumer look at the food product, food dyes also have important role to the food quality (Winarno, 2002). Most

[1] Agricultural Technology Department, Agricultural Technology Faculty, Jember University, e-mail: suci_silver@yahoo.co.id

commonly used food dyes are synthetic dyes. Synthetic dyes are cheap and better stability than natural dyes. But the use of these dyes can cause the health problem (Tranggono ,1990). The limited number of stocks of natural dyes is one of the reasons have to do the research sources of natural dyes from plants unique to Indonesia. One of the plants that can be utilized is Duwet (Syzygium cuminii). With the fruit make Duwet as raw material for natural dye powder, it can increase the economic value of the fruit. By Anonymous (2001) noted that duwet contain anthocyanin pigments which sianidin ramno-glucoside, petunidin and malvidin which is the red pigment formation.

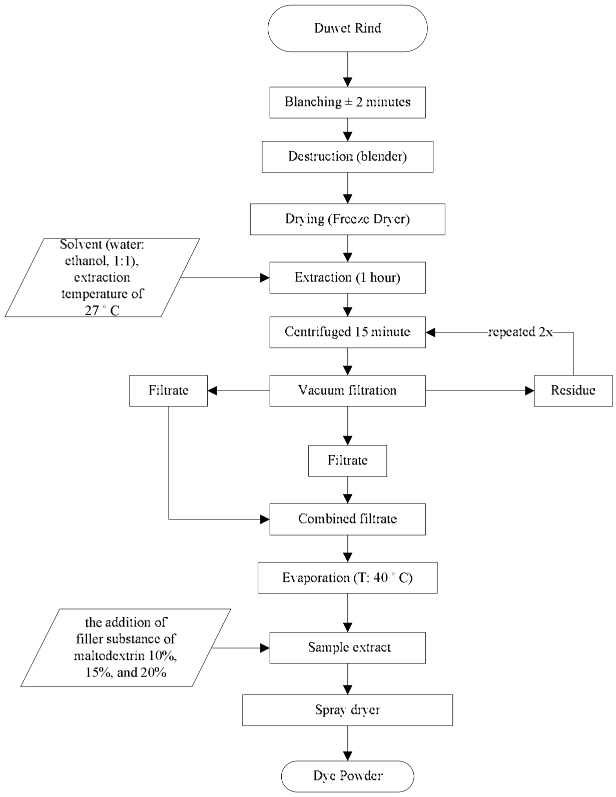
The most common method used in the manufacture of instant powder is a spray dryer because the process is efficient and economical (Kjaegaard, 1974). Use of this method especially for products that are sensitive to high temperatures. Anthocyanin at high temperatures and long time will decline as well as damaged if in contact with oxygen and light (DeMan, 199). To maintain stability the anthocyanin pigments must be trapped in the filler material so that it can be protected from the cause of the malfunction at the time of the process and storing.

This research is conducted to know the process of making duwet rind dye powder, the characteristic of dye powder which is produced, and the stability level during the storing. In addition, research is expected to provide information on how to make to dye powder of duwet rind and stability during the storing and increase the economic value of Duwet.

Research Materials and Method

The materials used are Duwet got from Rambipuji Market, Jember. The research is conducted in Chemical Laboratory and Bio-Chemical of Agriculture, Department of Technology of Agriculture, Faculty of Agriculture Technology, Jember University and Pilot Plan PAU Laboratory, Agriculture Institute of Bogor. The research starts in October 2005 up to July 2006.

A. Dye Powder Making Process



Picture 1. Diagram of the making process of dye powder from Duwet rind

B. Yield Total Test (Wijaya, *et al.*, 2001)

The formulation applicated was:

$$YieldTotal\,(\%) = \frac{Output}{Input}\,x\,100\%$$

Where: Output = fnal solids (dye powder which is produced) (gram)

Input = weight of sample + filler (gram)

C. Water Content Test (Oven method; AOAC, 1995)

The water content is measured using the following equation:

$$WaterContent\,(\%b/b) = \frac{x-y}{x-a}\,x\,100\%$$

Where: x = weigh bottle and sample before drying (gram)

y = weigh bottle and sample before drying (gram)

a = empty weigh bottle (gram)

D. Ash Content Test (Apriantono, 1989)

Ash content is measured using the following equation:

$$AshContent = \frac{C - A}{B - A}\ x\ 100\%$$

E. Solubility (Gravimetri Method; Rahayu, 1988)

The powder solubility is measured using the following equation:

$$Solubility\ (\%) = 100 - \frac{(a-b)}{\frac{(100 - \%KA)\ x\ c}{100}}\ x\ 100\%$$

Where: a = weight of filter paper dan residue (gram)

b = weight of filter paper (gram)

c = weight of sample (gram)

KA = water content of samples (% b/b)

F. Hydro-absorbability (AOAC, 1990)

Hydro-absorbability is measured using the difference calculation of powder initial weight and powder weight after one week per 100 grams solids (gram/100 grams).

G. Color Measurement

The color measurement is conducted using color reader (Minolta CR - 10). The parameter of the color is measured using the following pattern:

L = 94,35 + dL

a* = -5,75 + da

b* = 6,51 + db

C = $\{(a^*)^2 + (b^*)^2\}^{0,5}$

$H^o = \tan^{-1} \frac{b^*}{a^*}$

The parameters observed are:

L = brightness, the value is around 0-100, which shows black up to white

a* = shows green up to red, the value is around -80 –(100)

b* = shows blue up to yellow, the value is around -80 –(70)

C = chrome (color intensity), C = 0 means colorless, the higher the value of C, the bigger the value of color intensity.

H° = Hue, color angle (0° = neutral color, 90° = yellow, 180° = green, 270° = blue)

H. Anthocyanin Concentration (Prior et al., 1998)

The measurement method of anthocyanin concentration is based on the method of *pH* differential. The absorbance measurement uses spectrophotometer at the wave length 520 nm and 700 nm. The absorbance value of each pH is measured with pattern:

A= [$(A_{520} - A_{700})$ pH 1 – $(A_{520} - A_{700})$ pH 4,5]. The anthocyanin total is measured as cyanidins 3-glikoside using molar extinction coefficient as high as 26900 L cm^{-1} and molecular weight as high as 449,2.

Concentarion of antosianin (mg/L) = (A x BM x FP x 1000)/ (€ x 1)

Where:

A = absorbance

BM = Molecular weight (449,2)

FP = dilution factor

€ = molar extinction (26900 L cm^{-1})

I. Drying Loss of Pigment Anthocyanin During Drying using Spray Dryer (Cai and Corke, 2000)

Drying loss of pigment anthocyanin (%) is expressed as (the difference of anthocyanin concentration of freeze drying from the anthocyanin concentration of spray drying) x 100/ (anthocyanin concentration of freeze drying).

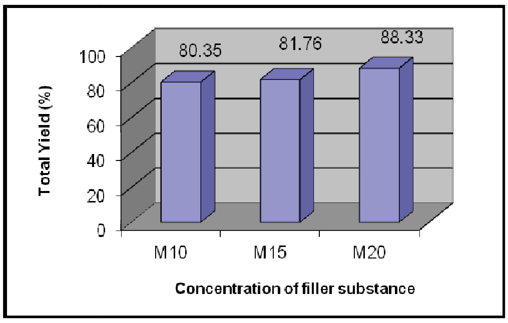
J. The stability Test of Anthocyanin during Storing

The stability test is conducted by measuring dye powder as much as 0,5 grams sample and the storing is conducted at RH 32% ($MgCl_2$) and storing at solidification temperature (-20ºC). The monitoring is conducted once a week from day 0 up to 16 week by measuring the anthocyanin concentration. The determination of anthocyanin stability during storing uses first order reaction equation where Ln (anthocyanin concentration) = -kt + c which is reached by plotting Ln (anthocyanin retention) with the storing time. the regression equation is Y = -kx + c where Y= ln(anthocyanin retention); k= slope/constant of reaction speed and half time is measured using the first order reaction equation, that is to say $T_{½}$ = 0,693/k.

Result and Discussion

A. Yield Total

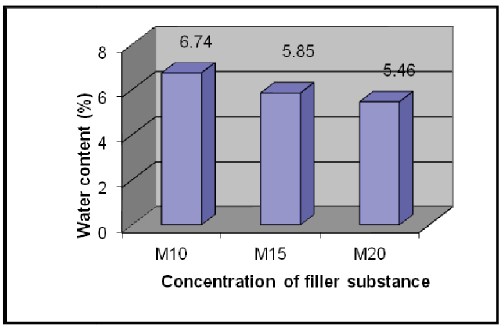
The highest Yield is achieved at the 20% maltodextrin addition. In **Picture 2**, it is shown that the higher the maltodextrin addition concentration, the higher the Yield. The filler materials functions to increase the density total, increase the volume, fasten the drying process and prevent the damage of the materials due to heat (Master in Sari et al.., 2004).



Picture 2. Total yield of Dye powder of duwet rind on concentration of filler substance 10%, 15%, and 20%

B. Water Content

The highest water content is achieved at the 10% maltodextrin addition. The powder which has high water content will be easily damaged because it can cause the growth of mildew and mushroom in the powder. For powder product, the water content must be less than 8% (Buckle, 1987).

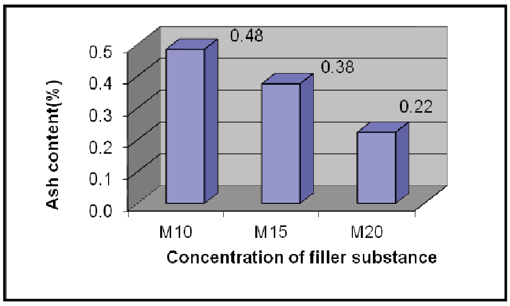


Picture 3. Water content of Dye powder of duwet rind on concentration of filler substance 10%, 15%, and 20%

From **Picture 3**, it is clear that the higher the maltodextrin addition concentration, the lower the water content. The increase of filler materials added makes the proportion of the density total in dye powder of Duwet rind bigger than the free-water total in the materials (Winarno, 2002).

C. Ash Content

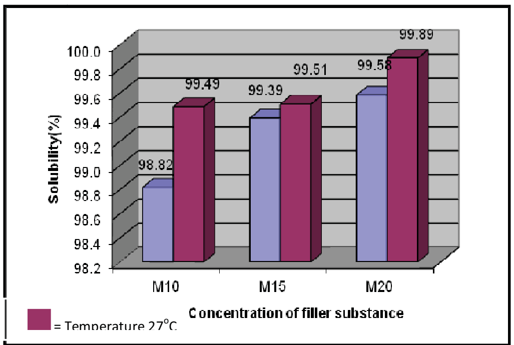
The highest ash content is reached at the 10% maltodextrin addition concentration. **Picture 4** shows that the maltodextrin addition in some concentration makes the decrease of ash content of dye powder. This is due to the filler materials such as Arabic gum, dextrin and maltodextrin have low content of ash. Maltodextrin composition consists of glucose (0,6%), maltose (4,0), maltotriose (7,0%) and the other saccharide (88,4%), while the ash content of maltodextrin as 0,5% (Kennedy dkk., 1995).



Picture 4. Ash content of Dye powder of duwet rind on concentration of filler substance 10%, 15%, and 20%

D. Solubility

Based on the solubility test of dye powder, at 27ºC and 40ºC, the solubility content of dye powder is still above 99%. This suggests that the level of the solubility of dye powder high so that it is beneficial in the practice.

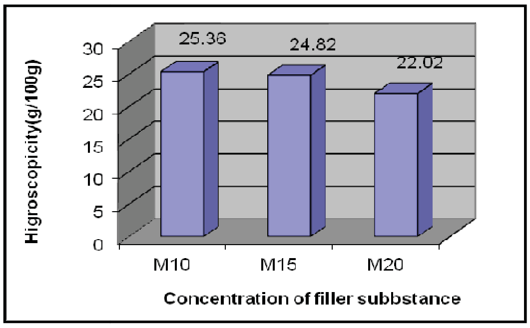


Picture 5. Solubility of Dye powder of duwet rind on concentration of filler substance 10%, 15%, and 20% at temperature 27^{0}C and

In **Picture 5**, the solubility of dye powder of Duwet rind at some filler materials concentration is at 27º and 40ºC. This result of the research shows that the higher the maltodextrin concentration, the higher the solubility level. According to National Standardization Council (1992 and 1989), maltodextrin has 97% solubility.

E. Hydro-absorbability

The highest hydro-absorbability at the treatment of the maltodextrin addition 10% is as high as 25,36 g/ 100g. **Picture 6** shows that the higher the maltodextrin concentration added, the lower the hydro-absorbability. Maltodextrin not only has a high solubility value but also has a low hydro-absorbability (hayati, 2004). The anthocyanin color consists of aglicone (antocyanidins) that is esterified by one sugar or more. The sort of sugar found in anthocyanin molecule is glucose, ramnose, galactose, xylose, arabinose (Francis, 1985).



Picture 6. Higroscopisity of Dye powder of duwet rind on concentration of filler substance 10%, 15%, and 20%

F. Color

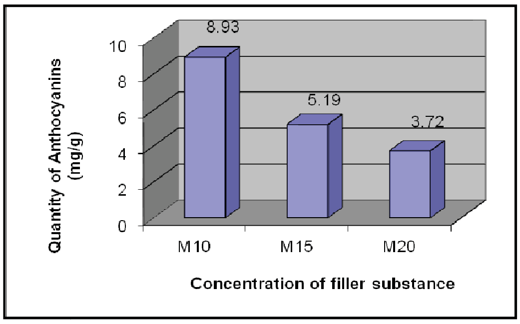
Color measurement is conducted using color reader (Minolta CR - 10). Table 1 shows that the higher the concentration of maltodextrin added, the brighter the color (L value is bigger). For a* (red level) is decreasing and b* is negative (blue chromaticity). Meanwhile, C is decreasing and makes the color getting fade. °H value (hue) shows blue corner.

Table 1 shows chromaticity data of dye powder of Duwet rind.

Treatment	L	a*	b*	C	°H
Maltodekstrin 10%	71,99± 0,23	8,85± 0,16	-6,95± 0,25	11,25± 0,25	269,33± 0,01
Maltodekstrin 15%	78,03± 0,08	5,01± 0,11	-4,93± 0,23	7,031 ± 0,14	269,22± 0,03
Maltodekstrin 20%	79,05± 0,51	3,53± 0,48	-4,01 ± 0,26	5,35± 0,48	269,15± 0,04

G. Quantity of Anthocyanin

In **Picture 7**, it is shown that with the addition of maltodextrin from concentration 10% up to 20%, there comes up the decrease of the anthocyanin concentration of dye powder of Duwet rind. Filler materials concentration addition causes the ratio of the filler materials bigger than anthocyanin in the dye powder of Duwet rind.



Picture 7. Quantity of anthocyanins of Dye powder of duwet rind on concentration of filler substance 10%, 15%, and 20%

H. Drying Loss

Drying loss value and anthocyanin retention using spray dryer method can be seen in **Table 2.**

Table 2. Shows the value of drying loss and anthocyanin retention during the spray drying at the addition of maltodextrin 10%.

Drying loss (%)	Retention of anthocyanin (%)
16.731	83.269

Table 2 shows that the drying loss using spray dryer is relatively small. The value of pigment loss during drying only represents one of the treatments, that is to say during the addition of maltodextrin 10%. During the addition of maltodextrin more than 10% in frozen drying treatment, there is no any encapsulation. This is because before the drying process is conducted, the sample extract added to the filler materials is frozen first, during the freezing process there is a separation of filler materials and sample extract, where the filler materials precipated.

I. The Sustainability Stability of Anthocyanin during the Storing

The anthocyanin retention value of Duwet rind in some different addition concentration of maltodextrin filler materials during the 16-week storing is presented in **Table 3**.

Table 3. Shows the anthocyanin retention of dye powder during the 16-week storing.

Treatment	Retention of anthocyanin (%)	
	Frozen temperature	$MgCl_2$
Maltodekstrin 10%	100.93	106.57
Maltodekstrin 15%	100.09	101.10
Maltodekstrin 20%	101.03	98.17

At the storing at solidification temperature (-18ºC) and RH 32% using saturated solution $MgCl_2$, the stability of dye powder tends to be stable at some concentration of maltodextrin addition. However, the table above shows that storing treatment at solidification temperature is better than the storing treatment with RH 32% (saturated $MgCl_2$). Winarno and Laksmi (1973) explains that in storing conducted at 1ºC, the anthocyanin does not change within six months, but when it is stored at the room temperature or more, the change happens fast.

J. Degradation Speed and Half Time of Dye Powder of Duwet During the Storing

From the stability test during solidification temperature, what is reached is the speed value of degradation and half time as what presented in **Table 4**.

Table 4 Shows the speed of degradation and half time of the anthocyanin of dye powder of Duwet rind at solidification temperature storing

Concentration of Filler substance	Degradation rate week^{-1} x (10^{-1})	Half-life ($T_{1/2}$) (week)
Maltodekstrin 10%	0.030	23
Maltodekstrin 15%	0.008	87
Maltodekstrin 20%	0.002	408

Table 4 suggests that the higher the concentration of maltodextrin added, the smaller the damage speed and the bigger the value of half time during storing. The value of degradation speed of anthocyanin and half time of dye powder of Duwet rind during storing is presented in Table 5.

Tabel 5. Shows the speed of degradation and half time of the anthocyanin of dye powder of Duwet rind during the storing at RH 32% (saturated $MgCl_2$)

Concentration Of filler substance	Degradation rate week^{-1} x (10^{-1})	Half-life ($T_{1/2}$) (week)
Maltodekstrin 10%	0.016	45
Maltodekstrin 15%	0.015	44
Maltodekstrin 20%	0.003	239

Table 5 shows that the higher the concentration of maltodextrin added, the smaller the damage speed and the bigger the value of half time during storing. The bigger the maltodextrin addition, the more the anthocyanin encapsulated. According to Anonym (2006a), the function of the filler materials is to stabilize the saluted materials, to control the saluted materials in order not to get easy to be damage, to keep the sensitive food from the oxidation environment and heat, and to increase the storing age. The concentration increase of the filler materials makes the encapsulated anthocyanin get more so that the amount of anthocyanin that is damage due to contact with heat or oxygen can be reduced.

Conclusion

Dye powder of duwet rind can be obtained by using spray dryer method. During drying process by using spray dryer, the percentage of drying loss is 16.731% and retention of anthocyanins is 83.269%. The increasing of maltodextrin quantity which is added to dye powder of duwet rind can increase the total value of yield, solubility, and stability of (half-life) dye powder of duwet rind. Whereas the value of water content, quantity of anthocyanins, hygroscopicity, ash content, and degradation rate decrease during the storing. For colour measuring using colour reader, the addition of quantity of maltodextrin 10% showing small brightness, colour tends to reddish blue and large amount of colour intensity.

Degradation rate of anthocyanins of duwet rind dye powder at frozen temperature storing 0.002-0.030 (minggu^{-1}) x 10^{-1}, and at room temperature storing (RH 32%) ranged 0.003-0.016 (minggu^{-1}) x 10^{-1}. For the half-life at the frozen temperature storing 23-408 week and room temperature (RH 32%) ranged 44-239 week. Whereas the addition of maltodextrin (25 DE:10 DE=3:1) 20.5% in dye powder of Amaranthus betasianin by using spray dryer have degradation rate 0.0109 (minggu^{-1}) x 10^{-1} and half-life ($T_{1/2}$) 64 week. Therefore, the storing treatment of frozen temperature is better than room temperature (RH 32%).

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