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26 27

#### Psychochemical Characteristics of Black Rice Powder Based on Maltodextrin and Skimmed Milk Powder Ratio as Encapsulant

#### ABSTRACT

9 Anthocyanins are bioactive components in black rice. Black rice anthocyanins are potent 10 antioxidants, so they have the potential to be developed into functional food products. As 11 a bioactive component, anthocyanins in extracts have low stability to environmental 12 conditions such as light, temperature, and pH. Encapsulation process using the spray 13 drying technique is known to protect and increase the stability of anthocyanin bioactive 14 compounds. This study's general objective was to determine the effect of the ratio of 15 maltodextrin (MDE) and skim milk powder (SMP) as an encapsulant on black rice extract 16 powder's physical and chemical characteristics. The research method is an experimental 17 type using a monofactor Completely Randomized Design (CRD), which consists of 6 18 treatments, namely the MDE:SMP ratio (100:0, 90:10, 80:20, 70:30, 60:40, and 50:50). 19 The results showed that there was a very significant effect of the ratio of MDE and SMP 20 on the physical and chemical characteristics of black rice powder. MDE and SMP ratios 21 of 50:50 resulted in black rice powder's best physical and chemical characteristics. 22

*Keywords:* black rice extract, encapsulation, coating material, physicochemical
 characteristics.

#### ABSTRAK

28 Antosianin merupakan komponen bioaktif pada beras hitam. Antosianin beras hitam 29 bersifat antikosidan kuat sehingga sangat potensial dikembangkan menjadi produk 30 pangan fungsional. Sebagai komponen bioaktif antosianin dalam bentuk ekstrak memiliki 31 stabilitas yang rendah terhadap kondisi lingkungan seperti cahaya, suhu dan pH. Proses 32 enkapsulasi menggunakan Teknik spray drying diketahui mampu melindungi dan 33 meningkatkan stabilitas senyawa bioaktif antosianin. Tujuan umum penelitian yaitu untuk 34 mengetahui pengaruh rasio maltodekstrin (MDE) dan susu bubuk skim (SMP) sebagai 35 enkapsulan terhadap karakteristik fisik dan kimia serbuk ekstrak beras hitam. Metode 36 penelitian berjenis eksperimen menggunakan Rancangan Acak Lengkap (RAL) 37 monofaktor, yang terdiri dari 6 perlakuan yaitu rasio MDE:SMP (100:0, 90:10, 80:20, 38 70:30, 60:40 dan 50:50). Hasil penelitian menunjukkan ada pengaruh yang sangat nyata 39 dari rasio MDE dan SMP terhadap karakteristik fisik dan kimia serbuk beras hitam. Rasio 40 MDE dan SMP 50:50 menghasilkan karakteristik fisik dan kimia serbuk beras hitam 41 terbaik.

42 Kata Kunci: ekstrak beras hitam, enkapsulasi, bahan penyalut, karakteristik fisikokimia.
43

**Commented [H1]:** Please add briefly why MDE and SMP were chosen as treatmets

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1 2 3

#### 1. INTRODUCTION

Black rice is pigmented rice with an endosperm covered with black aleurone. Black rice
cultivation in Indonesia is increasing, but its utilization in the food sector is not optimal.
However, black rice has anthocyanin bioactive components, potent antioxidants, so it can
potentially be developed as an ingredient in functional food products (Hosoda et al.,
2018).

10 Anthocyanin pigments in black rice can be obtained by extraction using the 11 maceration method. Generally, ethanol solvent is used in the maceration method. Ethanol 12 has advantages over other solvents such as methanol and acetone, namely it is more 13 economical and has low toxicity (Nour et al., 2013). Acidified ethanol has been confirmed 14 to increase yield and anthocyanin content in black rice extract (Pedro et al., 2016). 15 Extraction with heat-assisted maceration technique based on a recent study (Nurhidajah 16 et al., 2022) was reported to be very effective in preparing black rice anthocyanin extract. 17 Black rice anthocyanin extract is volatile to light, temperature and pH (Cassidy, 2018). 18 Encapsulation techniques can overcome this weakness using a freeze dryer, spray dryer, 19 and others (Bao et al., 2019; Fang & Bhandari, 2010; Howard et al., 2013). 20 Encapsulation using a spray dryer is an effective solution to protect and increase 21 the stability of anthocyanin bioactive compounds (Kalušević et al., 2017). This technique 22 has been widely used in the food industry. In addition to being applicable, the spray drying

(SD) method for thermosensitive products is due to a short contact time with a heat source
(Lourenço et al., 2020). The success of SD is influenced by several things, including inlet
and outlet temperatures, air flow rate, feed flow rate, speed of the atomizer, to the type

**Commented [H4]:** revise these two sentences.

**Commented [H5]:** Are light and pH influence the volatility od a substance? If so.. please add some more references. "susceptible' is more appropriate word instead of using "volatile"

1	and concentration of coating material used (Patil-Gadhe & Pokharkar, 2014). The coating	
2	material is one of the factors that will affect the encapsulated powder.	
3	Mahdavi et al. (2014) reported maltodextrin (MDE) as the most optimal coating	
4	material in anthocyanin encapsulation using the SD technique. MDE is a coating material	
5	with various advantages, including high solubility, low viscosity, and maintaining the	
6	stability of bioactive compounds (Suhag & Nanda, 2016). Recent research by Kalušević	
7	et al. (2017) reported that skimmed milk powder (SMP) coating material produced higher	
8	anthocyanin levels than maltodextrin. As a coating material, SMP has non-stick	
9	properties and high encapsulation efficiency. Apart from being a source of protein,	
10	lactose, vitamins, and minerals, this ingredient provides additional nutritional value	
11	(Bylaitë et al., 2001; Jing & Giusti, 2005; Shamaei et al., 2016). However, using SMP to	
12	encapsulate anthocyanin extracts resulted in powders with lower phenolic/flavonoid	
13	content (Kalušević et al., 2017).	
14	Until now, there has been no research report on the combination of MDE and SMP	
15	as a coating material in the encapsulation of black rice extract. Based on this, it is	
16	necessary to review further the effect of the MDE-SMP combination as a coating material	
17	for black rice extract, the right combination is expected to produce black rice extract	

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#### 2. MATERIALS AND METHODS

powder with the best physical and chemical characteristics.

## 22 2.1. Materials23

24 The research material was black rice of the Jeliteng variety collected from organic rice 25 farmers in the Karanganyar region, Central Java Province, Indonesia, food-grade citric 26 acid from PT Gunacipta Multirasa Indonesia, and aquadest. Pro-analytical chemicals Commented [H6]: Most optimal?

Commented [H7]: Recheck this sentence: (IN FACT coating material DOES NOT PRODUCE ANTHOCYANIN)

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#### **4** J. T E P (2017)

1 from Merck include ethanol, potassium chloride, sodium acetate, folin-ciocalteu, sodium 2 carbonate, gallic acid (C7H6O5), and 2,2-Diphenyl-1-picrylhydrazyl (DPPH). 3 2.2. Black rice anthocyanin extraction (Nurhidajah et al., 2022) 4 Black rice is ground into flour. 100 g of black rice flour was added with ethanol (56% 5 v/v) and citrate (4.5% w/v) solvent in a ratio of 1:10 (w/v). The extraction process was 6 7 carried out in a thermostatic water bath with a controlled temperature (50 C) for 120 8 minutes with constant stirring (500 rpm). The following process separates the liquid from 9 the residue using 400 mesh filter paper. The ethanol in the extract was then evaporated using a rotary evaporator at 55 °C. The anthocyanin extract of black rice was stored in a 10 11 dark glass bottle at 4°C until used. 12 2.3. Anthocyanin extract encapsulation (Nurhidajah et al., 2022) 13 14 MD and SMP coating materials emulsion solutions with different ratios (100:0, 90:10, Commented [H13]: Emulsion and solution are different in 15 80:20, 70:30, 60:40 and 50:50) were prepared at a concentration of 20% (w/v) with 16 deionized water at room temperature. Anthocyanin extract was mixed with each coating 17 material in a ratio of 1:1 (v/v). Each mixture was homogenized separately for 15 min at 18 3000 rpm. It was dried using a laboratory-scale spray dryer with an inlet air temperature 19 of  $120 \pm 1$  C, an outlet air temperature of  $80 \pm 5$  C, and a feed flow rate of 6.0 mL/minute 20 at a pressure of 1.5 bar. The obtained powder was collected and stored at -20°C until 21 analyzed. 22 2.3. Anthocyanin content (Yamuangmorn et al., 2018)

23

24 A differential pH method was used to determine black rice powder's anthocyanin content.

25 First, 1 g of powder was dissolved with 1 mL of ethanol in a test tube, then mixed

26 separately with two buffer solutions (1 mL of potassium chloride buffer pH 1.0 and 1 mL Commented [H9]: was Commented [H10]: Black rice powder sample (100 g). don't put number at the beginning of the sentence.

Commented [H11]: How was the separation process (easy to flow or hard?) because 400 mesh size is very dense

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molecular size

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1 sodium acetate buffer pH 4.5), and incubated at room temperature (± 25 °C) for 15 2 minutes. The absorbance was measured by spectrophotometer at 520 and 700 nm. The 3 absorbance value was obtained by subtracting difference in absorbance at a wavelength 4 of 520 nm and 700 nm at pH 1.0 with the difference in absorbance at pH 4.5. Anthocyanin content was obtained by multiplying the absorbance value by the molecular weight of 5 cyanidin-3-glucoside (448.8 g/mol) and the amount of dilution, then divided by the 6 7 coefficient of molar absorptivity of cyanidin-3-glucoside (26900 l/mol cm) and the width 8 of the cuvette (1 cm). The anthocyanin content of black rice extract was expressed in 9 mg/100g.

**Commented [H16]:** Please give the formula for calculation in order readers can understand more easily

#### 10 2.4. Total phenolics

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21

12 The Folin-Ciocalteu method (Pedro et al., 2016), with slight modifications, was used to 13 analyze total phenolics content (TPC). 0.5 g of microcapsules were prepared in a dark 14 tube, then 5 mL of Folin-Ciocalteu 10% (v/v) reagent was added. The solution was 15 homogenized for 5 mins and added 4 mL of 7.5% Na2CO3 (w/v). The mixture was incubated for 60 mins at room temperature ( $25 \pm 1$  °C). Ethanol is used as a blank solution. 16 17 Gallic acid in ethanol with a concentration of 100-500 ppm is used as a standard solution. 18 Next, the absorbance was measured using a UV-Vis spectrophotometer with a wavelength 19 of 765 nm. TPC of black rice extract was expressed as mg GAE/100g.

#### 20 2.5. Flavonoid content

- 22 Cai et al. (2016) adopted a slight modification method in determining the flavonoid
- 23 content. A sample of 0.5 ml black rice extract was prepared in a dark tube, to which 1.5
- 24 ml of ethanol, 0.1 ml of 10% AlCl3, 0.1 ml of 1 M CH3COOK, and 2.8 ml of distilled
- 25 water were added. The solution was homogenized and incubated at room temperature (25

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Commented [H18]: Please revise this sentence

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± 1 °C) for 30 minutes. The absorbance of the sample was measured using a UV-Vis
spectrophotometer with a wavelength of 415 nm, the blank solution using aquadest. The
standard curve uses a quercetin solution in aquadest with a concentration range of 20-100
ppm. Therefore, the flavonoid content of black rice extract was expressed as mg QE/100
g.

#### 6 2.6. Antioxidant activities

7

Antioxidant activity was determined according to Pedro et al. (2016), with slight modifications. A total of 1.5 ml of 0.2 mM DPPH in ethanol was mixed with 0.2 ml of the sample into a test tube, and ethanol was added to a final volume of 3.5 ml. The tubes were tightly closed, homogenized, and incubated at room temperature ( $25 \pm 1$  °C) for 60 minutes. The absorbance was measured at a wavelength of 517 nm, and the ability of the extract to scavenge DPPH was obtained by reducing the absorbance of the blank to the sample, then compared with the absorbance of the blank and expressed in % of inhibition.

#### 15 2.7. pH value

16

20

17 The pH value of black rice powder was measured with the instrument Hanna HI 2211

18 bench pH meters (Hanna instruments Ltd.), calibration solution using standard buffer.

#### 19 2.8. Yield, moisture content and water activity (a<sub>w</sub>)

Yield was obtained from the final weight percentage of black rice powder (g) with the total solids of the sample (g). Determination of the moisture content of the powder using a moisture analyzer (Shimadzu MOC63u, Japan), while the aw of the powder was determined using a water activity analyzer (Rotronic Hygropalm-HP23-Aw-A, Switzerland) at 25 C. Commented [H22]: please write the formula for calculation .. same as above comment. , also why the IC 50 was not determined?

**Commented [H23]:** please write the sample preparation for pH measurement

Commented [H21]: was read

#### 1 2.9. Color measurement

Color characteristics of black rice powder were measured using a calibrated Minolta CR310 Chromameter (Konica Minolta Business Solution Asia Pte Ltd). The hue angle, H°
[tan-1(b\*/a\*)] and chroma, C° [(a\*2 + b\*2)1/2] were also determined. The H° is used to
identify colors (red, yellow, green, and blue), whereas C° distinguishes between bright
and dull colors (Caparino et al., 2012).

8 2.10. Statistical analysis

All experiments were carried out in 4 repetitions. Results are presented in terms of mean
± standard deviation. The effect of treatment factors was analyzed using one-way
ANOVA, and Duncan's post hoc test was used to detect differences between treatments.
The significance level used is p<0.05. Statistical analysis was carried out with the help of</li>
SPSS 22.0 software.

15 16

18

#### 3. RESULTS AND DISCUSSION

#### 17 **3.1.** Anthocyanin and pH value of capsules

19 The mean anthocyanin content of black rice extract powder obtained was 51.22-84.59 20 mg/100g (Figure 1a). This amount is higher than the research results reported by 21 Pramitasari and Angelica (2020), which only ranged from 2.00-12.00 mg/100g. The 22 highest anthocyanin levels were obtained at a coating ratio of 50:50 (84.59 mg/100g). 23 There was an increase in anthocyanin levels of 65% (51.22mg/100g) compared to a ratio of 100:0. SMP generally contains β-casein components and has been reported to interact 24 25 with Cyanidin-3-glucoside compounds from black soybean extract through reversible hydrogen bonds supported by an optimum pH (Kalušević et al., 2017). Previous studies 26 27 also reported that casein protein interacts with malvidin-3-glucoside from grape extract Commented [H24]: coated powder?

1 through complex hydrogen bonds, which then form a hydrophobic reaction and 2 effectively increase thermal stability so that the rate of anthocyanin degradation during 3 drying can be minimized (He et al. 2016). The pH value of black rice powder ranged from 2.60-2.95 (Figure 1b). The pH value of the black rice extract powder increased with the 4 addition of SMP in the coating material ratio because SMP had a neutral pH value. 5 6 However, the increase in pH value is still in the anthocyanin stability range, at a value of 7 2-3 (Sipahli et al., 2017). Anthocyanins are more stable under acidic conditions in the 8 form of a flavylium cation structure (Khoo et al., 2017).

#### 9 3.2. Total phenolics, flavonoid, and antioxidant activities

9 10

11 Total phenolic and flavonoid content of black rice powder is presented in Table 1. From 12 the data, it is shown that the total phenolic and flavonoid content increases with the 13 addition of SMP in the ratio of the coating material. B-casein in protein-derived 14 encapsulation forms a more substantial complex with polyphenols, and this is due to the 15 more hydrophobic nature of B-casein. (Hasni et al., 2011). The increased flavonoids are 16 associated with the structure and bonds formed from proteins with flavonoid compounds. 17 This result has previously been described by Bohin et al. (2012) on the flavonoid 18 encapsulation of tea drinks. This underlies the increase in flavonoids when the ratio of 19 SMP to coating material increases. In a previous study, Kalušević et al. (2017) reported 20 the results of high antioxidant activity in coating black rice extract using SMP. Therefore, 21 the antioxidant activity of black rice powder is thought to be derived from the 22 encapsulated anthocyanin, phenolic, and flavonoid components. In addition, the increase 23 in antioxidant activity is also potential from the maillard reaction, which produces 24 melanoidin compounds with antioxidant activity. This reaction occurs in the protein

Commented [H25]: Maillard

4

component of SMP due to the high temperature during the drying process (Wang et al.,
 2011).

#### 3 **3.3.** Aw, mouisture content, and yield of microcapsules

5 All samples showed an Aw value lower than 0.6 (0.49-0.58), so the black rice extract 6 powder in this study had an excellent aw value (Goyal et al., 2015). A low aw value tends 7 to result in extended shelf life. The water content values ranged from 0.91 to 1.13% (Table 8 2). The product moisture content was low to prevent particle agglomeration, avoid the 9 caking process, and improve powder dispersion and flowability (Daza et al., 2016). 10 Therefore, the powder from spray drying obtained both the aw level and the moisture 11 content in this study met the requirements and was suitable for inclusion in the dry food 12 matrix.

The highest yield was obtained from powders with a 50:50 coating material, which is in line with the results reported in a study comparing milk protein-based microparticles with other carriers (Belščak-Cvitanovic et al., 2015). A combination of MDE and SMP coating materials resulted in good physical characteristics so that the coating material can be proposed as a coating material for the bioactive component of black rice extract.

#### 18 **3.4. Color characteristic**

19

20 Color is one of the indicators in assessing the quality of food products. During the 21 production process, the color of black rice extract is expected to be maintained until a 22 powder is obtained from drying. Therefore, observing the color measurement of black 23 rice extract powder after the encapsulation process is necessary. The results of chromatic 24 color measurements are shown in Table 3. Based on the parameters a\*, b\*, and hue angle, 25 it can be concluded that the color of the MD-SMP-based powder is red-violet with varying Commented [H26]: moisture

**Commented [H27]:** please be consistent in using the name of the sample studied

1 brightness levels. Among all SBHs, the coating with a 50:50 ratio had the lowest 2 brightness value (40.58  $\pm$  0.83) and the highest values for a\* and C\*, indicating that this 3 ratio produced the darkest powder with the highest portion of red color. Many reports have investigated the molecular level of the bond formed between casein-based coating 4 5 materials and black rice anthocyanins on their stability from a thermodynamic point of 6 view. The casein-anthocyanin bond is mainly stabilized through hydrophobic interactions 7 and hydrogen bonds involving the aglycone and glucosyl moieties of the encapsulated 8 molecule so that the color can be maintained during the drying process (Aprodu et al., 9 2019).

10 11

#### 4. CONCLUSIONS

12 Based on the results of the research that has been carried out, it can be concluded that the 13 best combination of coating materials in black rice encapsulation was obtained at MDE: 14 SMP ratio of 50:50 with total anthocyanin levels obtained 84.59 mg/100g, with total 15 phenol 0.41 mg GAE/g, total flavonoid 0.21 mg QE/g and antioxidant activity 31.71% RSA. Has an average value of L\* 40.5; a\* 43.64; b\* 12.75; Chroma 45.47; and Hue 16.31 16 17 with red-purple color types. Anthocyanin stability with a pH value of 2.95. Furthermore, 18 it has an aw of 0.49%, moisture content of 0.91%, and a yield of 51.29%. The interaction 19 between the combination of MDE and SMP as a coating material in the encapsulation 20 process of black rice extract had a very significant effect on total anthocyanin levels, 21 phenolics, total flavonoids, antioxidant activity, pH, color, aw, water content, and yield.

**Commented [H28]:** please not to use 'interaction' because this experiment was designed as a single factor experiment.

#### 5. ACKNOWLEDGMENTS

2 The authors would like to thank all parties involved and those who contributed to this

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4 research scheme on behalf of Dr Nurhidajah, M.Si.

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1 Table 1. Total phenolic, total flavonoid and antioxidant activity of black rice extract 2 powder based on MD and SMP ratio

F · · · · · · · · · · · ·			
MD-SMP Patio	Total Phenolic (mg	Total Flavonoid	Antioxidant
	GAE/g)	(mg QE/g)	Activity (%)
100:0	$0.27\pm0.01^{a}$	$0.14\pm0.02^{a}$	$25.07\pm0.39^a$
90:10	$0.30\pm0.01^{\text{b}}$	$0.16\pm0.01^{ab}$	$26.52 \pm 0.35^{b}$
80:20	$0.33 \pm 0.01^{\circ}$	$0.17 \pm 0.01^{bc}$	$29.23 \pm 0.22^{\circ}$
70:30	$0.37\pm0.01^{\text{d}}$	$0.18 \pm 0.03^{bcd}$	$30.37\pm0.38^{d}$
60:40	$0.38\pm0.01^{d}$	$0.19 \pm 0.02^{cd}$	$31.18 \pm 0.48^{e}$
50:50	$0.41\pm0.05^{\rm e}$	$0.21 \pm 0.02^d$	$31.71 \pm 0.94^{e}$
Information:			

3 Ir

1. All values are mean  $\pm$  standard deviation 4 times repetition

 Different superscript values showed significant differences with a 95% confidence level based on the ANOVA difference test and the LSD post hoc test.

3456789

10 Table 2. Physical properties of black rice powder based on MD and SMP ratio

MD:SMP Ratio	Yield (%)	Moisture content (%)	a <sub>w</sub> (%)
100:0	$33.51\pm0.82^a$	$1.13\pm0.03^{e}$	$0.58\pm0.58^{\rm c}$
90:10	$35.11 \pm 0.75^{a}$	$1.08\pm0.01^{de}$	$0.55 \pm 0.55^{bc}$
80:20	$31.14\pm0.68^a$	$1.04\pm0.09^{cd}$	$0.54 \pm 0.54^{bc}$
70:30	$34.06\pm0.82^a$	$0.99\pm0.01^{bc}$	$0.52\pm0.52^{ab}$
60:40	$47.64 \pm 0.73^{b}$	$0.96 \pm 0.02^{cb}$	$0.51 \pm 0.51^{ab}$
50:50	$51.29 \pm 0.49^{b}$	$0.91\pm0.05^a$	$0.49\pm0.49^{a}$

# 11 Information: 12 1. All v 13 2. Diffe 14 base

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1. All values are mean  $\pm$  standard deviation 4 times repetition

2. Different superscript values showed significant differences with a 95% confidence level

based on the ANOVA difference test and the LSD post hoc test.

Table 3. Color parameters of black rice powder based on MD and SMP ratio

MD:SMP	I *	.*	<b>b</b> *	C*	**0	Color
Ratio	L'	a	0.	C.	H	Color
100:0	$56.06 \pm 0.79^{d}$	$26.83\pm0.66^a$	$4.43\pm0.31^{a}$	$27.20\pm0.62^a$	$9.39\pm0.81^{a}$	Red-violet
90:10	$55.46\pm1.46^{c}$	$28.48\pm0.12^{b}$	$4.30\pm0.51^a$	$28.80\pm0.16^{b}$	$8.58\pm0.99^{a}$	Red-violet
80:20	$52.91 \pm 0.61^{\circ}$	$28.71 \pm 0.39^{b}$	$5.34\pm0.50^{ab}$	$29.20\pm0.35^{b}$	$10.55 \pm 1.06^{a}$	Red-violet
70:30	$49.07\pm0.82^{b}$	$35.78\pm1.45^{c}$	$6.36 \pm 1.56^{\text{b}}$	$36.36\pm1.65^c$	$10.03\pm2.13^a$	Red-violet
60:40	$48.06\pm0.54^a$	$39.90 \pm 0.47^{d}$	$9.71 \pm 0.39^{\circ}$	$41.06 \pm 0.39^{d}$	$13.68 \pm 0.65^{b}$	Red-violet
50:50	$40.58\pm0.83^a$	$43.64\pm1.43^{e}$	$12.75\pm0.83^{d}$	$45.47\pm1.16^{\text{e}}$	$16.31\pm1.50^{\rm c}$	Red-violet

18 Information:

1. All values are mean  $\pm$  standard deviation 4 times repetition

2. Different superscript values showed significant differences with a 95% confidence level

based on the ANOVA difference test and the LSD post hoc test.





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### Psychochemical Characteristics of Black Rice Powder Based on Maltodextrin and Skimmed Milk Powder Ratio as Encapsulant

#### ABSTRACT

9 Anthocyanins are bioactive components in black rice. Black rice anthocyanins are potent 10 antioxidants, so they have the potential to be developed into functional food products. As 11 a bioactive component, anthocyanins in extracts have low stability to environmental 12 conditions such as light, temperature, and pH. Encapsulation process using the spray 13 drying technique is known to protect and increase the stability of anthocyanin bioactive 14 compounds. This study's general objective was to determine the effect of the ratio of 15 maltodextrin (MDE) and skim milk powder (SMP) as an encapsulant on black rice extract 16 powder's physical and chemical characteristics. The research method is an experimental 17 type using a monofactor Completely Randomized Design (CRD), which consists of 6 18 treatments, namely the MDE:SMP ratio (100:0, 90:10, 80:20, 70:30, 60:40, and 50:50). 19 The results showed that there was a very significant effect of the ratio of MDE and SMP 20 on the physical and chemical characteristics of black rice powder. MDE and SMP ratios 21 of 50:50 resulted in black rice powder's best physical and chemical characteristics. 22

*Keywords:* black rice extract, encapsulation, coating material, physicochemical
 characteristics.

#### ABSTRAK

28 Antosianin merupakan komponen bioaktif pada beras hitam. Antosianin beras hitam 29 bersifat antikosidan kuat sehingga sangat potensial dikembangkan menjadi produk 30 pangan fungsional. Sebagai komponen bioaktif antosianin dalam bentuk ekstrak memiliki 31 stabilitas yang rendah terhadap kondisi lingkungan seperti cahaya, suhu dan pH. Proses 32 enkapsulasi menggunakan Teknik spray drying diketahui mampu melindungi dan 33 meningkatkan stabilitas senyawa bioaktif antosianin. Tujuan umum penelitian yaitu untuk 34 mengetahui pengaruh rasio maltodekstrin (MDE) dan susu bubuk skim (SMP) sebagai 35 enkapsulan terhadap karakteristik fisik dan kimia serbuk ekstrak beras hitam. Metode 36 penelitian berjenis eksperimen menggunakan Rancangan Acak Lengkap (RAL) 37 monofaktor, yang terdiri dari 6 perlakuan yaitu rasio MDE:SMP (100:0, 90:10, 80:20, 38 70:30, 60:40 dan 50:50). Hasil penelitian menunjukkan ada pengaruh yang sangat nyata 39 dari rasio MDE dan SMP terhadap karakteristik fisik dan kimia serbuk beras hitam. Rasio 40 MDE dan SMP 50:50 menghasilkan karakteristik fisik dan kimia serbuk beras hitam 41 terbaik.

42 Kata Kunci: ekstrak beras hitam, enkapsulasi, bahan penyalut, karakteristik fisikokimia.
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#### 1. INTRODUCTION

Black rice is pigmented rice with an endosperm covered with black aleurone. Black rice
cultivation in Indonesia is increasing, but its utilization in the food sector is not optimal.
However, black rice has anthocyanin bioactive components, potent antioxidants, so it can
potentially be developed as an ingredient in functional food products (Hosoda et al.,
2018).

10 Anthocyanin pigments in black rice can be obtained by extraction using the 11 maceration method. Generally, ethanol solvent is used in the maceration method. Ethanol 12 has advantages over other solvents such as methanol and acetone, namely it is more 13 economical and has low toxicity (Nour et al., 2013). Acidified ethanol has been confirmed 14 to increase yield and anthocyanin content in black rice extract (Pedro et al., 2016). 15 Extraction with heat-assisted maceration technique based on a recent study (Nurhidajah 16 et al., 2022) was reported to be very effective in preparing black rice anthocyanin extract. 17 Black rice anthocyanin extract is volatile to light, temperature and pH (Cassidy, 2018). 18 Encapsulation techniques can overcome this weakness using a freeze dryer, spray dryer, 19 and others (Bao et al., 2019; Fang & Bhandari, 2010; Howard et al., 2013). 20 Encapsulation using a spray dryer is an effective solution to protect and increase 21 the stability of anthocyanin bioactive compounds (Kalušević et al., 2017). This technique 22 has been widely used in the food industry. In addition to being applicable, the spray drying 23 (SD) method for thermosensitive products is due to a short contact time with a heat source

24 (Lourenço et al., 2020). The success of SD is influenced by several things, including inlet

and outlet temperatures, air flow rate, feed flow rate, speed of the atomizer, to the type

**Commented [U2]:** Explain the weakness of anthocyanin extract on storage. so the sentence is one of the weaknesses of black rice anthocyanin

extract which is unstable and volatile to light, temperature and  $\rm pH$  (Cassidy, 2018)

1 and concentration of coating material used (Patil-Gadhe & Pokharkar, 2014). The coating 2 material is one of the factors that will affect the encapsulated powder. 3 Mahdavi et al. (2014) reported maltodextrin (MDE) as the most optimal coating material in anthocyanin encapsulation using the SD technique. MDE is a coating material 4 5 with various advantages, including high solubility, low viscosity, and maintaining the 6 stability of bioactive compounds (Suhag & Nanda, 2016). Recent research by Kalušević 7 et al. (2017) reported that skimmed milk powder (SMP) coating material produced higher 8 anthocyanin levels than maltodextrin. As a coating material, SMP has non-stick 9 properties and high encapsulation efficiency. Apart from being a source of protein, 10 lactose, vitamins, and minerals, this ingredient provides additional nutritional value 11 (Bylaitë et al., 2001; Jing & Giusti, 2005; Shamaei et al., 2016). However, using SMP to 12 encapsulate anthocyanin extracts resulted in powders with lower phenolic/flavonoid 13 content (Kalušević et al., 2017). 14 Until now, there has been no research report on the combination of MDE and SMP

as a coating material in the encapsulation of black rice extract. Based on this, it is necessary to review further the effect of the MDE-SMP combination as a coating material for black rice extract, the right combination is expected to produce black rice extract powder with the best physical and chemical characteristics.

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#### 2. MATERIALS AND METHODS

## 22 2.1. Materials23

24 The research material was black rice of the Jeliteng variety collected from organic rice 25 farmers in the Karanganyar region, Central Java Province, Indonesia, food-grade citric 26 acid from PT Gunacipta Multirasa Indonesia, and aquadest. Pro-analytical chemicals

#### **4** J. T E P (2017)

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1 from Merck include ethanol, potassium chloride, sodium acetate, folin-ciocalteu, sodium 2 carbonate, gallic acid (C7H6O5), and 2,2-Diphenyl-1-picrylhydrazyl (DPPH). 3 2.2. Black rice anthocyanin extraction (Nurhidajah et al., 2022) 4 5 Black rice is ground into flour. 100 g of black rice flour was added with ethanol (56% 6 v/v) and citrate (4.5% w/v) solvent in a ratio of 1:10 (w/v). The extraction process was 7 carried out in a thermostatic water bath with a controlled temperature (50 C) for 120 8 minutes with constant stirring (500 rpm). The following process separates the liquid from 9 the residue using 400 mesh filter paper. The ethanol in the extract was then evaporated 10 using a rotary evaporator at 55 °C. The anthocyanin extract of black rice was stored in a 11 dark glass bottle at 4°C until used.

#### 12 **2.3**. Anthocyanin extract encapsulation (Nurhidajah et al., 2022)

14 MD and SMP coating materials emulsion solutions with different ratios (100:0, 90:10, 15 80:20, 70:30, 60:40 and 50:50) were prepared at a concentration of 20% (w/v) with 16 deionized water at room temperature. Anthocyanin extract was mixed with each coating 17 material in a ratio of 1:1 (v/v). Each mixture was homogenized separately for 15 min at 18 3000 rpm. It was dried using a laboratory-scale spray dryer with an inlet air temperature 19 of  $120 \pm 1$  C, an outlet air temperature of  $80 \pm 5$  C, and a feed flow rate of 6.0 mL/minute 20 at a pressure of 1.5 bar. The obtained powder was collected and stored at -20°C until 21 analyzed.

#### 22 **2.3.** Anthocyanin content (Yamuangmorn et al., 2018)

A differential pH method was used to determine black rice powder's anthocyanin content.
First, 1 g of powder was dissolved with 1 mL of ethanol in a test tube, then mixed
separately with two buffer solutions (1 mL of potassium chloride buffer pH 1.0 and 1 mL

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1 sodium acetate buffer pH 4.5), and incubated at room temperature (± 25 °C) for 15 2 minutes. The absorbance was measured by spectrophotometer at 520 and 700 nm. The 3 absorbance value was obtained by subtracting difference in absorbance at a wavelength 4 of 520 nm and 700 nm at pH 1.0 with the difference in absorbance at pH 4.5. Anthocyanin 5 content was obtained by multiplying the absorbance value by the molecular weight of cyanidin-3-glucoside (448.8 g/mol) and the amount of dilution, then divided by the 6 7 coefficient of molar absorptivity of cyanidin-3-glucoside (26900 l/mol cm) and the width 8 of the cuvette (1 cm). The anthocyanin content of black rice extract was expressed in 9 mg/100g.

#### 10 2.4. Total phenolics

The Folin-Ciocalteu method (Pedro et al., 2016), with slight modifications, was used to 12 13 analyze total phenolics content (TPC). 0.5 g of microcapsules were prepared in a dark 14 tube, then 5 mL of Folin-Ciocalteu 10% (v/v) reagent was added. The solution was 15 homogenized for 5 mins and added 4 mL of 7.5% Na2CO3 (w/v). The mixture was incubated for 60 mins at room temperature ( $25 \pm 1^{\circ}$ C). Ethanol is used as a blank solution. 16 17 Gallic acid in ethanol with a concentration of 100-500 ppm is used as a standard solution. 18 Next, the absorbance was measured using a UV-Vis spectrophotometer with a wavelength 19 of 765 nm. TPC of black rice extract was expressed as mg GAE/100g.

#### 20 2.5. Flavonoid content

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## 22 Cai et al. (2016) adopted a slight modification method in determining the flavonoid

23 content. A sample of 0.5 ml black rice extract was prepared in a dark tube, to which 1.5 ml of ethanol, 0.1 ml of 10% AlCl3, 0.1 ml of 1 M CH3COOK, and 2.8 ml of distilled 24 25 water were added. The solution was homogenized and incubated at room temperature (25

± 1 °C) for 30 minutes. The absorbance of the sample was measured using a UV-Vis
spectrophotometer with a wavelength of 415 nm, the blank solution using aquadest. The
standard curve uses a quercetin solution in aquadest with a concentration range of 20-100
ppm. Therefore, the flavonoid content of black rice extract was expressed as mg QE/100
g.

#### 6 2.6. Antioxidant activities

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8 Antioxidant activity was determined according to Pedro et al. (2016), with slight 9 modifications. A total of 1.5 ml of 0.2 mM DPPH in ethanol was mixed with 0.2 ml of 10 the sample into a test tube, and ethanol was added to a final volume of 3.5 ml. The tubes 11 were tightly closed, homogenized, and incubated at room temperature ( $25 \pm 1$  °C) for 60 12 minutes. The absorbance was measured at a wavelength of 517 nm, and the ability of the 13 extract to scavenge DPPH was obtained by reducing the absorbance of the blank to the 14 sample, then compared with the absorbance of the blank and expressed in % of inhibition.

#### 15 2.7. pH value

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17 The pH value of black rice powder was measured with the instrument Hanna HI 2211

18 bench pH meters (Hanna instruments Ltd.), calibration solution using standard buffer.

#### 19 2.8. Yield, moisture content and water activity (a<sub>w</sub>)

Yield was obtained from the final weight percentage of black rice powder (g) with the total solids of the sample (g). Determination of the moisture content of the powder using a moisture analyzer (Shimadzu MOC63u, Japan), while the aw of the powder was determined using a water activity analyzer (Rotronic Hygropalm-HP23-Aw-A, Switzerland) at 25 C.

#### 1 2.9. Color measurement

Color characteristics of black rice powder were measured using a calibrated Minolta CR310 Chromameter (Konica Minolta Business Solution Asia Pte Ltd). The hue angle, H°
[tan-1(b\*/a\*)] and chroma, C° [(a\*2 + b\*2)1/2] were also determined. The H° is used to
identify colors (red, yellow, green, and blue), whereas C° distinguishes between bright
and dull colors (Caparino et al., 2012).

8 2.10. Statistical analysis

All experiments were carried out in 4 repetitions. Results are presented in terms of mean
± standard deviation. The effect of treatment factors was analyzed using one-way
ANOVA, and Duncan's post hoc test was used to detect differences between treatments.
The significance level used is p<0.05. Statistical analysis was carried out with the help of</li>
SPSS 22.0 software.

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#### 3. RESULTS AND DISCUSSION

18 19 The mean anthocyanin content of black rice extract powder obtained was 51.22-84.59 20 mg/100g (Figure 1a). This amount is higher than the research results reported by 21 Pramitasari and Angelica (2020), which only ranged from 2.00-12.00 mg/100g. The 22 highest anthocyanin levels were obtained at a coating ratio of 50:50 (84.59 mg/100g). 23 There was an increase in anthocyanin levels of 65% (51.22mg/100g) compared to a ratio of 100:0. SMP generally contains β-casein components and has been reported to interact 24 25 with Cyanidin-3-glucoside compounds from black soybean extract through reversible hydrogen bonds supported by an optimum pH (Kalušević et al., 2017). Previous studies 26 27 also reported that casein protein interacts with malvidin-3-glucoside from grape extract

3.1. Anthocyanin and pH value of capsules

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1 through complex hydrogen bonds, which then form a hydrophobic reaction and 2 effectively increase thermal stability so that the rate of anthocyanin degradation during 3 drying can be minimized (He et al. 2016). The pH value of black rice powder ranged from 2.60-2.95 (Figure 1b). The pH value of the black rice extract powder increased with the 4 addition of SMP in the coating material ratio because SMP had a neutral pH value. 5 6 However, the increase in pH value is still in the anthocyanin stability range, at a value of 7 2-3 (Sipahli et al., 2017). Anthocyanins are more stable under acidic conditions in the 8 form of a flavylium cation structure (Khoo et al., 2017).

#### 9 3.2. Total phenolics, flavonoid, and antioxidant activities

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11 Total phenolic and flavonoid content of black rice powder is presented in Table 1. From 12 the data, it is shown that the total phenolic and flavonoid content increases with the 13 addition of SMP in the ratio of the coating material. B-casein in protein-derived 14 encapsulation forms a more substantial complex with polyphenols, and this is due to the 15 more hydrophobic nature of B-casein. (Hasni et al., 2011). The increased flavonoids are 16 associated with the structure and bonds formed from proteins with flavonoid compounds. 17 This result has previously been described by Bohin et al. (2012) on the flavonoid 18 encapsulation of tea drinks. This underlies the increase in flavonoids when the ratio of 19 SMP to coating material increases. In a previous study, Kalušević et al. (2017) reported 20 the results of high antioxidant activity in coating black rice extract using SMP. Therefore, 21 the antioxidant activity of black rice powder is thought to be derived from the 22 encapsulated anthocyanin, phenolic, and flavonoid components. In addition, the increase 23 in antioxidant activity is also potential from the maillard reaction, which produces 24 melanoidin compounds with antioxidant activity. This reaction occurs in the protein 4

component of SMP due to the high temperature during the drying process (Wang et al.,
 2011).

#### 3 3.3. Aw, mouisture content, and yield of microcapsules

5 All samples showed an Aw value lower than 0.6 (0.49-0.58), so the black rice extract 6 powder in this study had an excellent aw value (Goyal et al., 2015). A low aw value tends 7 to result in extended shelf life. The water content values ranged from 0.91 to 1.13% (Table 8 2). The product moisture content was low to prevent particle agglomeration, avoid the 9 caking process, and improve powder dispersion and flowability (Daza et al., 2016). 10 Therefore, the powder from spray drying obtained both the aw level and the moisture 11 content in this study met the requirements and was suitable for inclusion in the dry food 12 matrix.

The highest yield was obtained from powders with a 50:50 coating material, which is in line with the results reported in a study comparing milk protein-based microparticles with other carriers (Belščak-Cvitanovic et al., 2015). A combination of MDE and SMP coating materials resulted in good physical characteristics so that the coating material can be proposed as a coating material for the bioactive component of black rice extract.

#### 18 **3.4. Color characteristic**

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20 Color is one of the indicators in assessing the quality of food products. During the 21 production process, the color of black rice extract is expected to be maintained until a 22 powder is obtained from drying. Therefore, observing the color measurement of black 23 rice extract powder after the encapsulation process is necessary. The results of chromatic 24 color measurements are shown in Table 3. Based on the parameters a\*, b\*, and hue angle, 25 it can be concluded that the color of the MD-SMP-based powder is red-violet with varying

#### **10** J. T E P (2017)

1 brightness levels. Among all SBHs, the coating with a 50:50 ratio had the lowest 2 brightness value (40.58  $\pm$  0.83) and the highest values for a\* and C\*, indicating that this 3 ratio produced the darkest powder with the highest portion of red color. Many reports have investigated the molecular level of the bond formed between casein-based coating 4 5 materials and black rice anthocyanins on their stability from a thermodynamic point of 6 view. The casein-anthocyanin bond is mainly stabilized through hydrophobic interactions 7 and hydrogen bonds involving the aglycone and glucosyl moieties of the encapsulated 8 molecule so that the color can be maintained during the drying process (Aprodu et al., 9 2019).

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#### 4. CONCLUSIONS

12 Based on the results of the research that has been carried out, it can be concluded that the 13 best combination of coating materials in black rice encapsulation was obtained at MDE: 14 SMP ratio of 50:50 with total anthocyanin levels obtained 84.59 mg/100g, with total 15 phenol 0.41 mg GAE/g, total flavonoid 0.21 mg QE/g and antioxidant activity 31.71% RSA. Has an average value of L\* 40.5; a\* 43.64; b\* 12.75; Chroma 45.47; and Hue 16.31 16 17 with red-purple color types. Anthocyanin stability with a pH value of 2.95. Furthermore, 18 it has an aw of 0.49%, moisture content of 0.91%, and a yield of 51.29%. The interaction 19 between the combination of MDE and SMP as a coating material in the encapsulation 20 process of black rice extract had a very significant effect on total anthocyanin levels, 21 phenolics, total flavonoids, antioxidant activity, pH, color, aw, water content, and yield.

#### 5. ACKNOWLEDGMENTS

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4 research scheme on behalf of Dr Nurhidajah, M.Si.

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1 Table 1. Total phenolic, total flavonoid and antioxidant activity of black rice extract 2 powder based on MD and SMP ratio

F · · · · · · · · · · · · ·			
MD-SMP Patio	Total Phenolic (mg	Total Flavonoid	Antioxidant
WID.SIVII Katio	GAE/g)	(mg QE/g)	Activity (%)
100:0	$0.27\pm0.01^{a}$	$0.14\pm0.02^{a}$	$25.07\pm0.39^a$
90:10	$0.30\pm0.01^{\text{b}}$	$0.16\pm0.01^{ab}$	$26.52\pm0.35^{b}$
80:20	$0.33 \pm 0.01^{\circ}$	$0.17 \pm 0.01^{bc}$	$29.23 \pm 0.22^{\circ}$
70:30	$0.37\pm0.01^{\text{d}}$	$0.18 \pm 0.03^{bcd}$	$30.37\pm0.38^d$
60:40	$0.38\pm0.01^{d}$	$0.19 \pm 0.02^{cd}$	$31.18 \pm 0.48^{e}$
50:50	$0.41\pm0.05^{\rm e}$	$0.21 \pm 0.02^{d}$	$31.71 \pm 0.94^{e}$
Information:			

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1. All values are mean  $\pm$  standard deviation 4 times repetition

 Different superscript values showed significant differences with a 95% confidence level based on the ANOVA difference test and the LSD post hoc test.

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10 Table 2. Physical properties of black rice powder based on MD and SMP ratio

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	MD:SMP Ratio	Yield (%)	Moisture content (%)	a <sub>w</sub> (%)
	100:0	$33.51\pm0.82^{a}$	$1.13\pm0.03^{e}$	$0.58\pm0.58^{\rm c}$
	90:10	$35.11 \pm 0.75^{a}$	$1.08 \pm 0.01^{de}$	$0.55\pm0.55^{bc}$
	80:20	$31.14\pm0.68^a$	$1.04 \pm 0.09^{cd}$	$0.54\pm0.54^{bc}$
	70:30	$34.06\pm0.82^a$	$0.99 \pm 0.01^{bc}$	$0.52\pm0.52^{ab}$
	60:40	$47.64\pm0.73^{b}$	$0.96\pm0.02^{cb}$	$0.51\pm0.51^{ab}$
	50:50	$51.29\pm0.49^{b}$	$0.91\pm0.05^a$	$0.49\pm0.49^{a}$

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1. All values are mean  $\pm$  standard deviation 4 times repetition

2. Different superscript values showed significant differences with a 95% confidence level

based on the ANOVA difference test and the LSD post hoc test.

Table 3. Color parameters of black rice powder based on MD and SMP ratio

MD:SMP	Ι*	0*	<b>b</b> *	C*	110	Color
Ratio	L.	a	0.	C.	н	COIOI
100:0	$56.06 \pm 0.79^{d}$	$26.83\pm0.66^a$	$4.43\pm0.31^{a}$	$27.20\pm0.62^{a}$	$9.39\pm0.81^{a}$	Red-violet
90:10	$55.46\pm1.46^c$	$28.48\pm0.12^{b}$	$4.30\pm0.51^{a}$	$28.80\pm0.16^{b}$	$8.58\pm0.99^{a}$	Red-violet
80:20	$52.91 \pm 0.61^{\circ}$	$28.71 \pm 0.39^{b}$	$5.34\pm0.50^{ab}$	$29.20\pm0.35^{b}$	$10.55 \pm 1.06^{a}$	Red-violet
70:30	$49.07 \pm 0.82^{b}$	$35.78 \pm 1.45^{\circ}$	$6.36 \pm 1.56^{b}$	$36.36 \pm 1.65^{\circ}$	$10.03 \pm 2.13^{a}$	Red-violet
60:40	$48.06\pm0.54^a$	$39.90 \pm 0.47^{d}$	$9.71 \pm 0.39^{\circ}$	$41.06\pm0.39^d$	$13.68\pm0.65^{\text{b}}$	Red-violet
50:50	$40.58\pm0.83^a$	$43.64\pm1.43^{e}$	$12.75\pm0.83^{d}$	$45.47\pm1.16^{\text{e}}$	$16.31\pm1.50^{\rm c}$	Red-violet

18 Information:

1. All values are mean  $\pm$  standard deviation 4 times repetition

2. Different superscript values showed significant differences with a 95% confidence level

based on the ANOVA difference test and the LSD post hoc test.





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#### Physicochemical Characteristics of Anthocyanins Extract Powder from Black Rice Based on Maltodextrin and Skimmed Milk Powder Ratio as Encapsulant

#### ABSTRACT

10 Anthocyanins are bioactive components in black rice. Black rice anthocyanins are potent 11 antioxidants, so they have the potential to be developed into functional food products. As 12 a bioactive component, anthocyanins in extracts have low stability to environmental 13 conditions such as light, temperature, and pH. Encapsulation process using the spray 14 drying technique is known to protect and increase the stability of anthocyanin bioactive 15 compounds. Maltodextrin (MDE) is widely used as a coating material in the 16 encapsulation of anthocyanins using the spray drying technique with many advantages. 17 However, skim milk powder (SMP) was found to encapsulate black rice anthocyanins 18 efficiently. This study's general objective was to determine the effect of the ratio of MDE 19 and SMP as an encapsulant on black rice extract powder's physical and chemical 20 characteristics. The research method is an experimental type using a single factor 21 Completely Randomized Design (CRD), which consisted of 6 treatments, namely the 22 MDE:SMP ratio (100:0, 90:10, 80:20, 70:30, 60:40, and 50:50). The results showed that 23 there was a very significant effect of the ratio of MDE and SMP on the physical and chemical characteristics of coated anthocyanin powder. MDE and SMP ratios of 50:50 24 25 resulted in coated anthocyanin powder's best physical and chemical characteristics. 26

*Keywords:* black rice extract, encapsulation, coating material, physicochemical
 characteristics.
 characteristics.

#### ABSTRAK

32 Antosianin merupakan komponen bioaktif pada beras hitam. Antosianin beras hitam 33 bersifat antikosidan kuat sehingga sangat potensial dikembangkan menjadi produk 34 pangan fungsional. Sebagai komponen bioaktif antosianin dalam bentuk ekstrak memiliki 35 stabilitas yang rendah terhadap kondisi lingkungan seperti cahaya, suhu dan pH. Proses 36 enkapsulasi menggunakan Teknik spray drying diketahui mampu melindungi dan 37 meningkatkan stabilitas senyawa bioaktif antosianin. Maltodekstrin (MDE) banyak 38 digunakan sebagai bahan pelapis dalam enkapsulasi antosianin menggunakan teknik 39 spray drying dengan banyak keunggulan. Namun, susu bubuk skim (SMP) ditemukan 40 mampu mengenkapsulasi antosianin beras hitam secara efisien. Tujuan umum penelitian 41 yaitu untuk mengetahui pengaruh rasio MDE dan susu SMP sebagai enkapsulan terhadap 42 karakteristik fisik dan kimia serbuk ekstrak beras hitam. Metode penelitian berjenis 43 eksperimen menggunakan Rancangan Acak Lengkap (RAL) monofaktor, yang terdiri 8

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dari 6 perlakuan yaitu rasio MDE:SMP (100:0, 90:10, 80:20, 70:30, 60:40 dan 50:50).
 Hasil penelitian menunjukkan ada pengaruh yang sangat nyata dari rasio MDE dan SMP
 terhadap karakteristik fisik dan kimia serbuk beras hitam. Rasio MDE dan SMP 50:50
 menghasilkan karakteristik fisik dan kimia serbuk beras hitam terbaik.

5 Kata Kunci: ekstrak beras hitam, enkapsulasi, bahan penyalut, karakteristik fisikokimia.
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#### 1. INTRODUCTION

Black rice (Oryza sativa L.) is a pigmented rice that has black bran covering the endosperm. Consumption of pigmented rice, such as black rice is currently starting to increase because people have begun to change their food consumption patterns towards foods that are beneficial to health, namely functional foods. Black rice has potential as a functional food because it contains bioactive components, namely polyphenolic compounds, flavonoids, and anthocyanins that act as antioxidants, anti-inflammatory and have other important health benefits (Kong et al., 2012; Hosoda et al., 2018).

18 Anthocyanin pigments in black rice can be obtained by extraction using the 19 maceration method. Generally, ethanol solvent is used in the maceration method. Ethanol 20 has advantages over other solvents such as methanol and acetone, namely it is more 21 economical and has low toxicity (Nour et al., 2013). Acidified ethanol has been confirmed 22 to increase yield and anthocyanin content in black rice extract (Pedro et al., 2016). 23 Extraction with heat-assisted maceration technique based on a recent study (Nurhidajah 24 et al., 2022) was reported to be very effective in preparing black rice anthocyanin extract. 25 Anthocyanin extracts can be degraded during the storage process. This is due to its low 26 chemical stability to environmental conditions including temperature, light intensity, and 27 pH (Cassidy, 2018; Fernandes, 2014). The anthocyanin decomposition process occurs 28 faster at high temperatures (Patras et al., 2010). Anthocyanins are unstable at high light 29 intensity. The release of the sugar group causes the anthocyanin aglycones that are formed

to fade quickly when exposed to light (Janna et al., 2006). Encapsulation techniques can
 overcome this weakness using a freeze dryer, spray dryer, and others (Bao et al., 2019;
 Fang & Bhandari, 2010; Howard et al., 2013).

4 Encapsulation using a spray dryer is an effective solution to protect and increase the stability of anthocyanin bioactive compounds (Kalušević et al., 2017). This technique 5 6 has been widely used in the food industry. In addition to being applicable, the spray drying 7 (SD) method for thermosensitive products is due to a short contact time with a heat source 8 (Lourenço et al., 2020). The success of SD is influenced by several things, including inlet 9 and outlet temperatures, air flow rate, feed flow rate, speed of the atomizer, to the type 10 and concentration of coating material used (Patil-Gadhe & Pokharkar, 2014). The coating 11 material is one of the factors that will affect the encapsulated powder.

12 Mahdavi et al. (2014) reported maltodextrin (MDE) is extensively used as a coating 13 material in anthocyanin encapsulation using the SD technique. MDE as a coating material 14 with various advantages, including high solubility, low viscosity, and maintaining the 15 stability of bioactive compounds (Suhag & Nanda, 2016). Recent research by Kalušević 16 et al. (2017) reported that skimmed milk powder (SMP) reported that SMP coating 17 material could encapsulate anthocyanin components at higher levels than maltodextrin. 18 As a coating material, SMP has non-stick properties and high encapsulation efficiency. 19 Apart from being a source of protein, lactose, vitamins, and minerals, this ingredient 20 provides additional nutritional value (Bylaitë et al., 2001; Jing & Giusti, 2005; Shamaei 21 et al., 2016). However, the use of SMP as a coating material in the encapsulation of black 22 rice extract still has a weakness, namely the coating efficiency of phenolic and flavonoid 23 components is still very low (Kalušević et al., 2017).

#### **4** J. T E P (2017)

1 Until now, there has been no research report on the combination of MDE and SMP 2 as a coating material in the encapsulation of black rice extract. Based on this, it is 3 necessary to review further the effect of the MDE-SMP combination as a coating material 4 for black rice extract, the right combination is expected to produce coated anthocyanin 5 powder with the best physical and chemical characteristics.

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#### 2. MATERIALS AND METHODS

## 9 2.1. Materials

The research material was black rice of the Jeliteng variety collected from organic rice farmers in the Karanganyar region, Central Java Province, Indonesia, food-grade citric acid from PT Gunacipta Multirasa Indonesia, and aquadest. Pro-analytical chemicals from Merck include ethanol, potassium chloride, sodium acetate, folin-ciocalteu, sodium carbonate, gallic acid (C7H6O5), and 2,2-Diphenyl-1-picrylhydrazyl (DPPH).

#### 16 **2.2. Black rice anthocyanin extraction (Nurhidajah et al., 2022)**

18 Black rice was ground into flour. Black rice powder sample (100 g) was added with 19 ethanol (56% v/v) and citrate (4.5% w/v) solvent in a ratio of 1:10 (w/v). The extraction 20 was carried out for 120 minutes in a thermostatic water bath at a regulated temperature 21 (50 C) with steady stirring (500 rpm). Using 100 mesh filter paper, the following 22 procedure separates the liquid from the residue. The extract's ethanol was then evaporated 23 using a rotary evaporator set at 55 °C. The evaporation procedure was repeated until the 24 ethanol solvent ceased pouring, yielding a final volume of 44% black rice extract. The 25 black rice anthocyanin extract was kept at 4°C in a dark glass bottle.

## 26 **2.3.** Anthocyanin extract encapsulation (Nurhidajah et al., 2022)

1 MD and SMP coating materials emulsion with different ratios (100:0, 90:10, 80:20, 2 70:30, 60:40 and 50:50) were prepared at a concentration of 20% (w/v) with deionized 3 water at room temperature. Anthocyanin extract was mixed with each coating material in a ratio of 1:1 (v/v). Each mixture was homogenized separately for 15 min at 3000 rpm. It 4 5 was dried using a laboratory-scale spray dryer with an inlet air temperature of  $120 \pm 1$  C, 6 an outlet air temperature of  $80 \pm 5$  C, and a feed flow rate of 6.0 mL/minute at a pressure 7 of 1.5 bar. The obtained coated anthocyanin powder was collected and stored at -20°C 8 until analyzed.

#### 9 2.4. Anthocyanin content (Yamuangmorn et al., 2018)

11 A differential pH method was used to determine coated anthocyanin powder. First, 12 sample (1 g) was dissolved with 1 mL of ethanol in a test tube, then mixed separately 13 with two buffer solutions (1 mL of potassium chloride buffer pH 1.0 and 1 mL sodium 14 acetate buffer pH 4.5), and incubated at room temperature (± 25 °C) for 15 minutes. The 15 absorbance was read by spectrophotometer at 520 and 700 nm. The absorbance value was 16 obtained by subtracting difference in absorbance at a wavelength of 520 nm and 700 nm 17 at pH 1.0 with the difference in absorbance at pH 4.5. The absorbance value of the extract 18 sample was calculated using the equation: 19  $A = [(A_{520} - A_{700}) \text{ pH1} - (A_{520} - A_{700}) \text{ pH4.5}]$ 

20 Then anthocyanin content of black rice extract was expressed in mg/100g:

21 Anthocyanin content (mg/100 g) = 
$$\frac{A \times MW \times DF \times V \times 100}{E \times 1 \times W}$$

22 A = absorbance

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23 MW = molecular weight of cyanidin-3-glucoside (448.8 g/mol)

24 DF = dilution factor

- 1 V = volume of the sample mother liquor
- 2 E = coefficient of molar absorptivity of cyanidin-3-glucoside (26900 l/mol cm)
- 3 W = sample weight (g)

#### 4 **2.5. Total phenolics**

5 6 Total phenolics content (TPC) used the Folin-Ciocalteu technique based on (Pedro et al., 7 2016) with minor modifications. In a dark tube, coated anthocyanin powder (0.5 g) was 8 made, and 5 mL of Folin-Ciocalteu 10% (v/v) reagent was added. After homogenizing 9 the solution for 5 minutes, 4 mL of 7.5% Na<sub>2</sub>CO<sub>3</sub> (w/v) was added. The mixture was 10 incubated at room temperature (25 1C) for 60 minutes. As a blank solution, ethanol is employed. A standard solution of gallic acid in ethanol with a concentration of 100-500 11 12 ppm is utilized. The absorbance was read with a UV-Vis spectrophotometer set at 765 13 nm. TPC was calculated as mg GAE/100g of black rice extract.

#### 14 **2.6. Flavonoid content**

Flavonoid content was determined according to Cai et al., (2016), with modifications. In 16 17 a dark tube, 0.5 ml of black rice extract was mixed with 1.5 ml of ethanol, 0.1 ml of 10% 18 AlCl<sub>3</sub>, 0.1 ml of 1 M CH<sub>3</sub>COOK, and 2.8 ml of distilled water. The solution was 19 homogenized and incubated for 30 minutes at room temperature (25  $\pm$  1 °C). The 20 absorbance of the sample was read with a UV-Vis spectrophotometer at 415 nm, and the 21 blank solution was aquadest. A quercetin solution in aquadest with a concentration range 22 of 20-100 ppm is used in the standard curve. The flavonoid concentration of black rice 23 extract was therefore reported as mg QE/100 g.

#### 24 2.7. Antioxidant activities

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1 Antioxidant activity was determined according to Pedro et al. (2016), with slight 2 modifications. A total of 1.5 ml of 0.2 mM DPPH in ethanol was mixed with 0.2 ml of 3 the sample into a test tube, and ethanol was added to a final volume of 3.5 ml. The tubes 4 were tightly closed, homogenized, and incubated at room temperature ( $25 \pm 1$  °C) for 60 5 minutes. The absorbance was read at a wavelength of 517 nm and calculated using the 6 equation:

7 % RSA =  $\frac{\text{Abs blanko x Abs test}}{\text{Abs blanko}} \times 100\%$ 

#### 8 2.8. pH value

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The pH value was measuring according to Park et al., (2016). Coated anthocyanin powder
sample (4g) was mixed with 16 mL of distilled water in a vortex mixer for 1 min. The pH
was recorded using a instrument Hanna HI 2211 bench pH meters (Hanna instruments
Ltd.), calibration solution using standard buffer.

#### 14 **2.9.** Yield, moisture content and water activity (a<sub>w</sub>)

Yield was obtained from the final weight percentage of coated anthocyanin powder (g)
with the total solids of the sample (g). Determination of the moisture content of the
powder using a moisture analyzer (Shimadzu MOC63u, Japan), while the aw of the
powder was determined using a water activity analyzer (Rotronic Hygropalm-HP23-AwA, Switzerland) at 25 C.

#### 21 2.10. Color measurement

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Color characteristics of coated anthocyanin powder were measured using a calibrated
Minolta CR-310 Chromameter (Konica Minolta Business Solution Asia Pte Ltd). The hue
angle, H° [tan-1(b\*/a\*)] and chroma, C° [(a\*2 + b\*2)1/2] were also determined. The H°

Commented [H1]: please write the formula for calculation .. same as above comment. , also why the IC 50 was not determined?

**Commented [MOU2R1]:** Thank you very much for the input. We will consider it for future research.

is used to identify colors (red, yellow, green, and blue), whereas C° distinguishes between
 bright and dull colors (Caparino et al., 2012).

3 2.11. Statistical analysis

3.1. Anthocyanin and pH value of capsules

All experiments were carried out in 4 repetitions. Results are presented in terms of mean
± standard deviation. The effect of treatment factors was analyzed using one-way
ANOVA, and Duncan's post hoc test was used to detect differences between treatments.
The significance level used is p<0.05. Statistical analysis was carried out with the help of</li>
SPSS 22.0 software.

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#### 3. RESULTS AND DISCUSSION

13 The mean anthocyanin content of black rice coated anthocyanin powder obtained was 14 15 51.22-84.59 mg/100g (Figure 1a). This amount is higher than the research results reported 16 by Pramitasari and Angelica (2020), which only ranged from 2.00-12.00 mg/100g. The 17 highest anthocyanin levels were obtained at a coating ratio of 50:50 (84.59 mg/100g). 18 There was an increase in anthocyanin levels of 65% (51.22mg/100g) compared to a ratio 19 of 100:0. SMP 50% as a coating material was able to trap more anthocyanins, resulting 20 in a powder with the highest anthocyanin content, significantly different from all 21 treatments. SMP generally contains  $\beta$ -casein components and has been reported to 22 interact with Cyanidin-3-glucoside compounds from black soybean extract through 23 reversible hydrogen bonds supported by an optimum pH (Kalušević et al., 2017). Previous 24 studies also reported that casein protein interacts with malvidin-3-glucoside from grape 25 extract through complex hydrogen bonds, which then form a hydrophobic reaction and 26 effectively increase thermal stability so that the rate of anthocyanin degradation during

8

drying can be minimized (He et al. 2016). The pH value of coated anthocyanin powder
ranged from 2.60-2.95 (Figure 1b). The pH value increased with the addition of SMP in
the coating material ratio because SMP had a neutral pH value. However, the increase in
pH value is still in the anthocyanin stability range, at a value of 2-3 (Sipahli et al., 2017).
Anthocyanins are more stable under acidic conditions in the form of a flavylium cation
structure (Khoo et al., 2017).

#### 7 3.2. Total phenolics, flavonoid, and antioxidant activities

9 Total phenolic and flavonoid content of coated anthocyanin powder is presented in Table 10 1. From the data, it is shown that the total phenolic and flavonoid content increases with 11 the addition of SMP 50% in the ratio of the coating material, and significanly different 12 with all treatments. B-casein in protein-derived encapsulation forms a more substantial 13 complex with polyphenols, and this is due to the more hydrophobic nature of B-casein. 14 (Hasni et al., 2011). The increased flavonoids are associated with the structure and bonds 15 formed from proteins with flavonoid compounds. This result has previously been 16 described by Bohin et al. (2012) on the flavonoid encapsulation of tea drinks. This 17 underlies the increase in flavonoids when the ratio of SMP to coating material increases. 18 In a previous study, Kalušević et al. (2017) reported the results of high antioxidant activity 19 in coating black rice extract using SMP. Therefore, the antioxidant activity of coated 20 anthocyanin powder is thought to be derived from the encapsulated anthocyanin, 21 phenolic, and flavonoid components. In addition, the increase in antioxidant activity is also potential from the Maillard reaction, which produces melanoidin compounds with 22 23 antioxidant activity. This reaction occurs in the protein component of SMP due to the 24 high temperature during the drying process (Wang et al., 2011).

1 2	3.3. Aw, moisture content, and yield of coated anthocyanin powder
3	All samples showed an Aw value lower than 0.6 (0.49-0.58), so the coated anthocyanin
4	powder in this study had an excellent aw value (Goyal et al., 2015). A low aw value tends
5	to result in extended shelf life. There is a positive tendency to increase the ratio of SMP
6	as a coating material, SMP 50% is able to increase the aw of the product significantly.
7	The water content values ranged from 0.91 to 1.13% (Table 2), the use of SMP 50%
8	significantly made anthocyanin powder with the lowest water content. On the other hand,
9	coatings with high MDE content produce high water content due to the nature of MDE,
10	which quickly releases water (Kustyawati et al., 2022). The product moisture content was
11	low to prevent particle agglomeration, avoid the caking process, and improve powder
12	dispersion and flowability (Daza et al., 2016). Therefore, the powder from spray drying
13	obtained both the aw level and the moisture content in this study met the requirements
14	and was suitable for inclusion in the dry food matrix.
15	The highest yield was significantly obtained from powders with a 50:50 coating
16	material, which is in line with the results reported in a study comparing milk protein-
17	based microparticles with other carriers (Belščak-Cvitanovic et al., 2015). A combination
18	of MDE and SMP coating materials resulted in good physical characteristics so that the

coating material can be proposed as a coating material for the bioactive component ofblack rice extract.

#### 21 **3.4. Color characteristic**

22

Color is one of the indicators in assessing the quality of food products. During the production process, the color of black rice extract is expected to be maintained until a powder is obtained from drying. Therefore, observing the color measurement of coated anthocyanin powder after the encapsulation process is necessary. The results of chromatic

#### **11** J. T E P (2017)

1 color measurements are shown in Table 3. Based on the parameters a\*, b\*, and hue angle, 2 it can be concluded that the color of the MD-SMP-based powder is red-violet with varying 3 brightness levels. Among all coated anthocyanin powders, coating with a ratio of 50:50 differed significantly by having the lowest brightness value ( $40.58 \pm 0.83$ ) and the highest 4 5 value for a\* and C\* with significant differences with other treatments. Indicating that this 6 ratio produced the darkest powder with the highest portion of red color. Many reports 7 have investigated the molecular level of the bond formed between casein-based coating 8 materials and black rice anthocyanins on their stability from a thermodynamic point of 9 view. The casein-anthocyanin bond is mainly stabilized through hydrophobic interactions 10 and hydrogen bonds involving the aglycone and glucosyl moieties of the encapsulated 11 molecule so that the color can be maintained during the drying process (Aprodu et al., 12 2019).

13 14

#### 4. CONCLUSIONS

15 It can be concluded that the best combination of coating materials in black rice 16 encapsulation was obtained at MDE: SMP ratio of 50:50 with total anthocyanin levels 17 obtained 84.59 mg/100g, with total phenol 0.41 mg GAE/g, total flavonoid 0.21 mg QE/g and antioxidant activity 31.71% RSA. Has an average value of L\* 40.5; a\* 43.64; b\* 18 19 12.75; Chroma 45.47; and Hue 16.31 with red-purple color types. Anthocyanin stability 20 with a pH value of 2.95. Furthermore, it has an aw of 0.49%, moisture content of 0.91%, 21 and a yield of 51.29%. The combination of MDE and SMP as a coating material in the 22 encapsulation process of black rice extract had a very significant effect on total 23 anthocyanin levels, phenolics, total flavonoids, antioxidant activity, pH, color, aw, water 24 content, and yield.

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## Table 1. Total phenolic, total flavonoid and antioxidant activity of coated anthocyanin powder based on MD and SMP ratio

MD:SMD Patio	Total Phenolic (mg	Total Flavonoid	Antioxidant
WID.SWIF Katio	GAE/g)	(mg QE/g)	Activity (%)
100:0	$0.27\pm0.01^{a}$	$0.14\pm0.02^{a}$	$25.07\pm0.39^a$
90:10	$0.30 \pm 0.01^{b}$	$0.16 \pm 0.01^{ab}$	$26.52 \pm 0.35^{b}$
80:20	$0.33 \pm 0.01^{\circ}$	$0.17 \pm 0.01^{\rm bc}$	$29.23 \pm 0.22^{\circ}$
70:30	$0.37\pm0.01^{d}$	$0.18\pm0.03^{bcd}$	$30.37\pm0.38^d$
60:40	$0.38\pm0.01^{d}$	$0.19 \pm 0.02^{cd}$	$31.18\pm0.48^{e}$
50:50	$0.41\pm0.05^{e}$	$0.21\pm0.02^{d}$	$31.71\pm0.94^{e}$
Information:			

#### 3 Ir

1. All values are mean  $\pm$  standard deviation 4 times repetition

 Different superscript values showed significant differences with a 95% confidence level based on the ANOVA difference test and the LSD post hoc test.

10 Table 2. Physical properties of coated anthocyanin powder based on MD and SMP ratio

MD:SMP Ratio	Yield (%)	Moisture content (%)	a <sub>w</sub> (%)
100:0	$33.51\pm0.82^a$	$1.13\pm0.03^{e}$	$0.58\pm0.58^{\rm c}$
90:10	$35.11\pm0.75^a$	$1.08\pm0.01^{de}$	$0.55\pm0.55^{bc}$
80:20	$31.14\pm0.68^a$	$1.04\pm0.09^{cd}$	$0.54\pm0.54^{bc}$
70:30	$34.06\pm0.82^a$	$0.99\pm0.01^{bc}$	$0.52\pm0.52^{ab}$
60:40	$47.64 \pm 0.73^{b}$	$0.96\pm0.02^{cb}$	$0.51\pm0.51^{ab}$
50:50	$51.29\pm0.49^{b}$	$0.91\pm0.05^a$	$0.49\pm0.49^{a}$

#### 11 Information:

1. All values are mean  $\pm$  standard deviation 4 times repetition

2. Different superscript values showed significant differences with a 95% confidence level

based on the ANOVA difference test and the LSD post hoc test.

12 13 14 15 16 17

MD:SMP	Ι*	o*	h*	C*	T TO	Color
Ratio	L	a	0	C	п	COIOI
100:0	$56.06 \pm 0.79^{d}$	$26.83\pm0.66^a$	$4.43\pm0.31^{a}$	$27.20\pm0.62^a$	$9.39\pm0.81^{a}$	Red-violet
90:10	$55.46 \pm 1.46^{c}$	$28.48 \pm 0.12^{b}$	$4.30\pm0.51^{a}$	$28.80\pm0.16^{\text{b}}$	$8.58\pm0.99^{\rm a}$	Red-violet
80:20	$52.91 \pm 0.61^{\circ}$	$28.71 \pm 0.39^{b}$	$5.34\pm0.50^{ab}$	$29.20\pm0.35^{b}$	$10.55\pm1.06^a$	Red-violet
70:30	$49.07\pm0.82^{b}$	$35.78 \pm 1.45^{\circ}$	$6.36 \pm 1.56^{\text{b}}$	$36.36\pm1.65^c$	$10.03 \pm 2.13^{a}$	Red-violet
60:40	$48.06\pm0.54^b$	$39.90 \pm 0.47^{d}$	$9.71 \pm 0.39^{\circ}$	$41.06 \pm 0.39^{d}$	$13.68 \pm 0.65^{b}$	Red-violet
50:50	$40.58\pm0.83^a$	$43.64\pm1.43^{e}$	$12.75\pm0.83^d$	$45.47\pm1.16^{\text{e}}$	$16.31\pm1.50^{\rm c}$	Red-violet

18 Information:

1. All values are mean  $\pm$  standard deviation 4 times repetition

2. Different superscript values showed significant differences with a 95% confidence level

based on the ANOVA difference test and the LSD post hoc test.

<sup>3456789</sup> 







4. Paper Accepted (15-11-2022)

## [JTEP-L] Editor Decision External Inbox ×



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Yth Penulis,

Berdasarkan revisi yang kami terima, dengan gembira kami sampaikan bahwa naskah Saudara yang berjudul: Psychochemical Characteristics of Black Rice Powder Based on Maltodextrin and Skimmed Milk Powder Ratio as Encapsulant dinyatakan ACCEPTED dan dapat dipublikasikan di JTEP.

 $\times$ 

Selain itu kami sampaikan kabar gembira bahwa JTEP telah naik peringkatnya menjadi SINTA 2 (<u>https://sinta.kemdikbud.go.id/journals</u> ketik Teknik Pertanian pada Source) yang berlaku mulai Vol. 10, No. 2, September 2021. Tidak hanya itu, manajemen JTEP akan meningkatkan lagi ke arah jurnal terindeks scopus, sehingga mulai Vol. 11 No. 1, Maret 2022, JTEP akan mempublikasikan paper dalam Bahasa Inggris.

Sehubungan dengan ini semua kami sampaikan bahwa biaya APC (Article Processing Charge) JTEP naik menjadi 750.000. Biaya agar ditransfer ke rekening BNI, No. 0698202763 a/n Elhamida Rezkia Amien selambat-lambatnya Tanggal 21 November 2022.

Demikian surat ini disampaikan, atas perhatiannya kami ucapkan terima kasih.



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November 15<sup>th</sup>, 2022

Number : 117/J.TEP-L/XI/2022 subject : Accepted Paper

To :

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## Physicochemical Characteristics of Anthocyanin Extract Powder from Black Rice Based on Maltodextrin and Skimmed Milk Powder Ratio as Encapsulant

Nurhidajah<sup>1⊠</sup>, Ali Rosidi<sup>2</sup>, Diode Yonata<sup>1</sup>, Nurrahman<sup>1</sup>, Boby Pranata<sup>3</sup>

<sup>1</sup>Department of Food Technology, University of Muhammadiyah Semarang, Semarang, INDONESIA <sup>2</sup>Department of Nutrition, University of Muhammadiyah Semarang, Semarang, INDONESIA <sup>3</sup>Postgraduate Student of Food Technology, Soegijapranata Catholic University, Semarang, INDONESIA

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#### ABSTRACT

Anthocyanins are bioactive components in black rice. Black rice anthocyanins are potent antioxidants, so they have the potential to be developed into functional food products. As a bioactive component, anthocyanins in extracts have low stability to environmental conditions such as light, temperature, and pH. Encapsulation process using the spray drying technique is known to protect and increase the stability of anthocyanin bioactive compounds. Maltodextrin (MDE) is widely used as a coating material in the encapsulation of anthocyanins using the spray drying technique with many advantages. However, skim milk powder (SMP) was found to encapsulate black rice anthocyanins efficiently. This study's general objective was to determine the effect of the ratio of MDE and SMP as an encapsulant on black rice extract powder's physical and chemical characteristics. The research method is an experimental type using a single factor Completely Randomized Design (CRD), which consisted of 6 treatments, namely the MDE:SMP ratio (100:0, 90:10, 80:20, 70:30, 60:40, and 50:50). The results showed that there was a very significant effect of the ratio of MDE and SMP on the physical and chemical characteristics of coated anthocyanin powder. MDE and SMP ratios of 50:50 resulted in coated anthocyanin powder's best physical and chemical characteristics.

<sup>™</sup>Corresponding Author: nurhidajah@unimus.ac.id

#### **1. INTRODUCTION**

Black rice (*Oryza sativa* L.) is a pigmented rice that has black bran covering the endosperm. Consumption of pigmented rice, such as black rice is currently starting to increase because people have begun to change their food consumption patterns towards foods that are beneficial to health, namely functional foods. Black rice has potential as a functional food because it contains bioactive components, namely polyphenolic compounds, flavonoids, and anthocyanins that act as antioxidants, anti-inflammatory and have other important health benefits (Kong *et al.*, 2012; Hosoda *et al.*, 2018).

Anthocyanin pigments in black rice can be obtained by extraction using the maceration method. Generally, ethanol solvent is used in the maceration method. Ethanol has advantages over other solvents such as methanol and acetone, namely it is more economical and has low toxicity (Nour et al., 2013). Acidified ethanol has been confirmed to increase yield and anthocyanin content in black rice extract (Pedro et al., 2016). Extraction with heat-assisted maceration technique based on a recent study (Nurhidajah et al., 2022) was reported to be very effective in preparing black rice anthocyanin extract. Anthocyanin extracts can be degraded during the storage process. This is due to its low chemical stability to environmental conditions including temperature, light intensity, and pH (Cassidy, 2018; Fernandes et al., 2014). The anthocyanin decomposition process occurs faster at high temperatures (Patras et al., 2010). Anthocyanins are unstable at high light intensity. The release of the sugar group causes the anthocyanin aglycones that are formed to fade quickly when exposed to light (Janna et al., 2006). Encapsulation techniques can overcome this weakness using a freeze dryer, spray dryer, and others (Bao et al., 2019; Fang & Bhandari, 2010; Howard et al., 2013).

Encapsulation using a spray dryer is an effective solution to protect and increase the stability of anthocyanin bioactive compounds (Kalušević *et al.*, 2017). This technique has been widely used in the food industry. In addition to being applicable, the spray drying (SD) method for thermosensitive products is due to a short contact time with a heat source (Lourenço *et al.*, 2020). The success of SD is influenced by several factors, including temperatures of inlet and outlet, feed flow rate, speed of the atomizer, air flow rate, and type as well as concentration of coating material (Patil-Gadhe & Pokharkar, 2014). One of the elements that will influence the encapsulated powder is the coating substance.

Mahdavi *et al.* (2014) reported maltodextrin (MDE) is extensively used as a coating material in anthocyanin encapsulation using the SD technique. MDE as a coating material with various advantages, including high solubility, low viscosity, and maintaining the stability of bioactive compounds (Suhag & Nanda, 2016). Recent research by Kalušević *et al.* (2017) reported that skimmed milk powder (SMP) reported that SMP coating material could encapsulate anthocyanin components at higher levels than maltodextrin. As a coating material, SMP has excellent encapsulation effectiveness and non-stick characteristics. This ingredient adds extra nutritional value in addition to being a source of protein, lactose, vitamins, and minerals (Bylaitë *et al.*, 2001; Jing & Giusti, 2005; Shamaei *et al.*, 2016). However, the use of SMP as a coating material in the encapsulation of black rice extract still has a weakness, namely the coating efficiency of phenolic and flavonoid components is still very low (Kalušević *et al.*, 2017).

Until now, there has been no research report on the combination of MDE and SMP as a coating material in the encapsulation of black rice extract. Based on this, it is necessary to review further the effect of the MDE-SMP combination as a coating material for black rice extract, the right combination is expected to produce coated anthocyanin powder with the best physical and chemical characteristics.

#### 2. MATERIALS AND METHODS

#### 2.1. Materials

The main ingredient used is black rice of the jellyteng variety obtained from organic rice farmers in Central Java. While the chemicals used are citric acid, gallic acid (C7H6O5), ethanol, 2,2-Diphenyl-1-picrylhydrazyl (DPPH), potassium chloride, folin-

ciocalteu, sodium acetate, and sodium carbonate are some of the pro-analytical substances used. manufactured by Merck.

#### 2.2. Black Rice Anthocyanin Extraction (Nurhidajah et al., 2022)

Flour was made from pulverized black rice. Black rice flour sample (100 g) was mixed in a 1:10 (w/v) ratio with the solvents ethanol (56% v/v) and citrate (4.5% w/v). The extraction was carried out for 120 minutes with constant agitation (500 rpm) in waterbath at 50 °C. Using 100 mesh filter paper, the following procedure separates the liquid from the residue. The extracted ethanol was then evaporated at 55 °C using a rotary evaporator. The evaporation procedure was repeated until the ethanol solvent ceased pouring, yielding a final volume of 44% black rice extract. The black rice anthocyanin extract was kept at 4 °C in a dark glass bottle.

### 2.3. Anthocyanin Extract Encapsulation (Nurhidajah et al., 2022)

MD and SMP coating materials emulsion with different ratios (100:0, 90:10, 80:20, 70:30, 60:40 and 50:50) were prepared at a concentration of 20% (w/v) with deionized water at room temperature. Anthocyanin extract was mixed with each coating material in a ratio of 1:1 (v/v). Each mixture was homogenized separately for 15 min at 3000 rpm. It was dried using a lab-scale spray drier with  $120 \pm 1$  °C inlet air temperatures, and  $80 \pm 5$  °C outlet air temperatures, 6.0 mL/min feed flow rate, and 1.5 bar feed pressure. The obtained coated anthocyanin powder was gathered and kept at -20°C until analyzed.

### 2.4. Anthocyanin Content (Yamuangmorn et al., 2018)

To measure covered anthocyanin powder, a differential pH technique was employed. First, the sample (1 g) was dissolved in 1 mL of ethanol in a test tube. Next, two buffer solutions 1 mL of potassium chloride buffer, pH 1.0, and 1 mL of sodium acetate buffer, pH 4.5 were combined independently, and the test tube was incubated at room temperature (±25 °C) for 15 min. The absorbance was read by spectrophotometer at 520 and 700 nm. The following equation was used to determine the extract sample's absorption value (A):

$$A = [(A_{520} - A_{700})_{pH1} - (A_{520} - A_{700})_{pH4.5}]$$
(1)

Then anthocyanin content (AC) of black rice extract was calculated according to Equation (2) and was expressed in mg/100g:

$$AC (mg/100 g) = \frac{A \times MW \times DF \times V \times 100}{E \times 1 \times W}$$
(2)

where MW = 448.8 g/mol is molecular weight of cyanidin-3-glucoside, DF is dilution factor, V is volume of the sample mother liquor, E = 26900 l/mol cm is the measure of molar absorptivity of cyanidin-3-glucoside, and W is sample weight (g).

#### 2.5. Total Phenolics

The Folin-Ciocalteu method, which was modified slightly from Pedro et al. (2016), was used to measure the total phenolic content (TPC). Sample (0.5 g) was mixed with 5 ml of 10% Folin-Ciocalteu reagent. The addition of 7.5% Na2CO3 as much as 4 mL then homogenized. The solution was incubated for 60 minutes at 25  $\pm$  1 °C. Reference solution used gallic acid at a strength of 100–500 ppm. The absorbance was measured using a UV-Vis spectrometer set at 765 nm. TPC was calculated as mg GAE/100g of black rice.

#### 2.6. Flavonoid Content

Flavonoid content was determined according to Cai *et al.*, (2016), with modifications. Black rice extract was combined with 2.8 milliliters of purified water, 0.1 milliliters of 10% AlCl3, 1.5 milliliters of ethanol, and 0.1 milliliters of 1 M CH3COOK in a dark tube. The mixture was homogenized and kept for 30 minutes at room temperature (25  $\pm$  1 °C). The blank solution was aquadest, and the sample's absorbance was measured with a UV-Vis spectrometer at 415 nm. The reference curve uses a quercetin solution in aquadest with a concentration range of 20-100 ppm. Thus, mg QE/100 g was stated as the black rice extract's flavonoid content.

#### 2.7. Antioxidant Activities

Antioxidant activity was determined according to Pedro et al. (2016), with slight modifications. The sample (0,2 ml) was mixed with 1.5 ml DPPH (0.2 mM), then diluted by adding 3.5 ml of ethanol. The solution was incubated at 25  $\pm$  1 °C for 60 minutes, then read at 517 nm. The following equation was used to determine it:

$$\% RSA = \frac{Abs \, b \, lanko \, x \, Abs \, test}{Abs \, b \, lanko} \, x \, 100\% \tag{3}$$

#### 2.8. pH value

The pH value was measuring according to Park & Kim (2016). Coated anthocyanin powder sample (4g) was mixed with 16 mL of distilled water in a vortex mixer for 1 min. The pH was recorded using instrument Hanna HI 2211 bench pH meters (Hanna instruments Ltd.), calibration solution using standard buffer.

#### 2.9. Yield, Moisture Content and Water Activity (a<sub>w</sub>)

Yield was obtained from the final weight percentage of coated anthocyanin powder (g) with the total solids of the sample (g). Determination of water content and water activity using analysis tools, each of which is a moisture analyzer (Shimadzu MOC63u) and a water activity analyzer (Rotronic Hygropalm-HP23-Aw-A) at room temperature.

#### 2.10. Color Measurement

An appropriately adjusted Minolta CR-310 Chromameter was used to test the coated anthocyanin powder's color characteristics (Konica Minolta Business Solution Asia Pte Ltd). Additionally, the chroma,  $C^{\circ} = [(a^{*}2 + b^{*}2)1/2]$ , and the hue angle,  $H^{\circ} = [tan-1(b^{*}/a^{*})]$ , were calculated. Red, yellow, green, and blue are the four primary hues, while the C° differentiates between bright and dull colors (Caparino *et al.*, 2012).

#### 2.11. Statistical Analysis

All experiments were performed in 4 repetitions. Results were presented in terms of average value  $\pm$  standard deviation. The effect of treatment factors was analyzed using one-way ANOVA, and differences among treatments was detected using Duncan's post hoc test. The significance level used is p < 0.05. Statistical analysis was conducted with the help of SPSS 22.0 software.

#### **3. RESULTS AND DISCUSSION**

#### 3.1. Anthocyanin and pH Value of Capsules

The mean anthocyanin content of black rice coated anthocyanin powder obtained was 51.22-84.59 mg/100g (Figure 1a). This amount is higher than the research results

reported by Pramitasari & Angelica (2020), which only ranged from 2.00-12.00 mg/100g. The highest anthocyanin levels were obtained at a coating ratio of 50:50 (84.59 mg/100g). There was an increase in anthocyanin levels of 65% (51.22mg/100g) compared to a ratio of 100:0. SMP 50% as a coating material was able to trap more anthocyanins, resulting in a powder with the highest anthocyanin content, significantly different from all treatments. SMP generally contains b-casein components and has been reported to interact with Cyanidin-3-glucoside compounds from black soybean extract through reversible hydrogen bonds supported by an optimum pH (Kalušević et al., 2017). Previous studies also reported that casein protein interacts with malvidin-3glucoside from grape extract through complex hydrogen bonds, which then form a hydrophobic reaction and effectively increase thermal stability so that the rate of anthocyanin degradation during drying can be minimized (He et al., 2016). The pH value of coated anthocyanin powder ranged from 2.60-2.95 (Figure 1b). The pH value increased with the addition of SMP in the coating material ratio because SMP had a neutral pH value. However, the increase in pH value is still in the anthocyanin stability range, at a value of 2-3 (Sipahli et al., 2017). Anthocyanins are more stable under acidic conditions in the form of a flavylium cation structure (Khoo *et al.*, 2017).



**Figure 1.** Anthocyanin and pH value coated anthocyanin powder based on MDE:SMP Ratio

#### 3.2. Total Phenolics, Flavonoid, and Antioxidant Activities

Total phenolic and flavonoid content of coated anthocyanin powder is presented in Table 1. From the data, it is shown that the total phenolic and flavonoid content increases with the addition of SMP 50% in the ratio of the coating material, and significanly different with all treatments. B-casein in protein-derived encapsulation forms a more substantial complex with polyphenols, and this is due to the more hydrophobic nature of B-casein. (Hasni et al., 2011). The increased flavonoids are associated with the structure and bonds formed from proteins with flavonoid compounds. This result has previously been described by Bohin et al. (2012) on the flavonoid encapsulation of tea drinks. This underlies the increase in flavonoids when the ratio of SMP to coating material increases. In a previous study, Kalušević et al. (2017) reported the results of high antioxidant activity in coating black rice extract using SMP. Therefore, the antioxidant activity of coated anthocyanin powder is thought to be derived from the encapsulated anthocyanin, phenolic, and flavonoid components. In addition, the increase in antioxidant activity is also potential from the Maillard reaction, which produces melanoidin compounds with antioxidant activity. This reaction occurs in the protein component of SMP due to the high temperature during the drying process (Wang et al., 2011).

MD:SMP Ratio	Total Phenolic (mg GAE/g)	Total Flavonoid (mg QE/g)	Antioxidant Activity (%)
100:0	$0.27 \pm 0.01^{a}$	$0.14 \pm 0.02^{a}$	25.07 ± 0.39 <sup>a</sup>
90:10	$0.30 \pm 0.01^{b}$	$0.16 \pm 0.01^{ab}$	$26.52 \pm 0.35^{b}$
80:20	$0.33 \pm 0.01^{\circ}$	$0.17 \pm 0.01^{bc}$	$29.23 \pm 0.22^{\circ}$
70:30	$0.37 \pm 0.01^{d}$	$0.18 \pm 0.03^{bcd}$	$30.37 \pm 0.38^{d}$
60:40	$0.38 \pm 0.01^{d}$	$0.19 \pm 0.02^{cd}$	$31.18 \pm 0.48^{e}$
50:50	$0.41 \pm 0.05^{e}$	$0.21 \pm 0.02^{d}$	$31.71 \pm 0.94^{e}$

**Table 1.** Total phenolic, total flavonoid and antioxidant activity of coated anthocyanin

 powder based on MD and SMP ratio

Note: All values are mean  $\pm$  standard deviation 4 times repetition. Different superscript values showed significant differences with a 95% confidence level based on the ANOVA difference test and the LSD post hoc test.

#### 3.3. AW, Moisture Content, and Yield of Coated Anthocyanin Powder

All samples showed an Aw value lower than 0.6 (0.49-0.58), so the coated anthocyanin powder in this study had an excellent aw value (Goyal *et al.*, 2015). A low aw value tends to result in extended shelf life. There is a positive tendency to increase the ratio of SMP as a coating material, SMP 50% is able to increase the aw of the product significantly. The water content values ranged from 0.91 to 1.13% (Table 2), the use of SMP 50% significantly made anthocyanin powder with the lowest water content. On the other hand, coatings with high MDE content produce high water content due to the nature of MDE, which quickly releases water (Kustyawati *et al.*, 2022). The product moisture content was low to prevent particle agglomeration, avoid the caking process, and improve powder dispersion and flowability (Daza *et al.*, 2016). Therefore, the powder from spray drying obtained both the aw level and the moisture content in this study met the requirements and was suitable for inclusion in the dry food matrix.

MD:SMP Ratio	Yield (%)	Moisture content (%)	a <sub>w</sub> (%)
100:0	$33.51 \pm 0.82^{a}$	$1.13 \pm 0.03^{e}$	$0.58 \pm 0.58^{\circ}$
90:10	$35.11 \pm 0.75^{\circ}$	$1.08 \pm 0.01^{de}$	$0.55 \pm 0.55^{bc}$
80:20	$31.14 \pm 0.68^{a}$	$1.04 \pm 0.09^{cd}$	$0.54 \pm 0.54^{bc}$
70:30	$34.06 \pm 0.82^{a}$	$0.99 \pm 0.01^{bc}$	$0.52 \pm 0.52^{ab}$
60:40	$47.64 \pm 0.73^{b}$	$0.96 \pm 0.02^{cb}$	$0.51 \pm 0.51^{ab}$
50:50	$51.29 \pm 0.49^{b}$	$0.91 \pm 0.05^{a}$	$0.49 \pm 0.49^{a}$

Table 2. Physical properties of coated anthocyanin powder based on MD and SMP ratio

Note: All values are mean  $\pm$  standard deviation 4 times repetition. Different superscript values showed significant differences with a 95% confidence level based on the ANOVA difference test and the LSD post hoc test.

The highest yield was significantly obtained from powders with a 50:50 coating material, which is in line with the results reported in a study comparing milk proteinbased microparticles with other carriers (Belščak-Cvitanovic *et al.*, 2015). A combination of MDE and SMP coating materials resulted in good physical characteristics so that the coating material can be proposed as a coating material for the bioactive component of black rice extract.

#### 3.4. Color Characteristic

Color is one of the indicators in assessing the quality of food products. During the production process, the color of black rice extract is expected to be maintained until a powder is obtained from drying. Therefore, observing the color measurement of coated anthocyanin powder after the encapsulation process is necessary. The results of chromatic color measurements are shown in Table 3. Based on the parameters a\*, b\*, and hue angle, it can be concluded that the color of the MD-SMP-based powder is redviolet with varying brightness levels. Among all coated anthocyanin powders, coating with a ratio of 50:50 differed significantly by having the lowest brightness value (40.58  $\pm$  0.83) and the highest value for a\* and C\* with significant differences with other treatments. Indicating that this ratio produced the darkest powder with the highest portion of red color. Many reports have investigated the molecular level of the bond formed between casein-based coating materials and black rice anthocyanins on their stability from a thermodynamic point of view. The casein-anthocyanin bond is mainly stabilized through hydrogen bonds and hydrophobic interactions involving the aglycone and glucosyl moieties of the encapsulated molecule so that the color can be maintained during the drying process (Aprodu et al., 2019).

MD:SMP Ratio	L*	a*	b*	С*	H°	Color <sup>#</sup>
100:0	56.06 ± 0.79 <sup>d</sup>	$26.83 \pm 0.66^{a}$	$4.43 \pm 0.31^{a}$	$27.20 \pm 0.62^{a}$	9.39 ± 0.81 <sup>a</sup>	R-V
90:10	55.46 ± 1.46 <sup>c</sup>	$28.48 \pm 0.12^{b}$	$4.30 \pm 0.51^{a}$	$28.80 \pm 0.16^{b}$	$8.58 \pm 0.99^{\circ}$	R-V
80:20	$52.91 \pm 0.61^{c}$	$28.71 \pm 0.39^{b}$	$5.34 \pm 0.50^{ab}$	29.20 ± 0.35 <sup>b</sup>	$10.55 \pm 1.06^{a}$	R-V
70:30	49.07 ± 0.82 <sup>b</sup>	35.78 ± 1.45 <sup>c</sup>	$6.36 \pm 1.56^{b}$	36.36 ± 1.65 <sup>c</sup>	$10.03 \pm 2.13^{a}$	R-V
60:40	$48.06 \pm 0.54^{b}$	$39.90 \pm 0.47^{d}$	$9.71 \pm 0.39^{\circ}$	$41.06 \pm 0.39^{d}$	$13.68 \pm 0.65^{b}$	R-V
50:50	$40.58 \pm 0.83^{\circ}$	$43.64 \pm 1.43^{e}$	$12.75 \pm 0.83^{d}$	45.47 ± 1.16 <sup>e</sup>	$16.31 \pm 1.50^{\circ}$	R-V

Table 3. Color parameters of coated anthocyanin powder based on MD and SMP ratio

Note: #) R-V = red-violet.

All values are mean ± standard deviation 4 times repetition. Different superscript values showed significant differences with a 95% confidence level based on the ANOVA difference test and the LSD post hoc test.

#### 4. CONCLUSIONS

It can be concluded that the best combination of coating materials in black rice encapsulation was obtained at MDE: SMP ratio of 50:50 with total anthocyanin levels obtained 84.59 mg/100g, with total phenol 0.41 mg GAE/g, total flavonoid 0.21 mg QE/g and antioxidant activity 31.71% RSA. The encapsulated product has an average value of L\* 40.5; a\* 43.64; b\* 12.75; Chroma 45.47; and Hue 16.31 with red-purple color types. Anthocyanin stability with a pH value of 2.95. Furthermore, it has an aw of 0.49%, moisture content of 0.91%, and a yield of 51.29%. The combination of MDE and SMP as a coating material in the encapsulation process of black rice extract had a very significant effect on total anthocyanin levels, phenolics, total flavonoids, antioxidant activity, pH, color, aw, water content, and yield.

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