# Characterization of *Kimpul* flour after process to remove itching sensation

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Abstract The results showed interacted between treatments on the chemical parameters of water, ash, protein, fat, carbohydrate, fiber, starch, amylose, and amylopectin content and physical parameters of gelatinization temperature, gel strength, and water absorption. Ca treatment was no interaction between treatments. The selected treatment of the expected value was B2G4 (blanching at 70 °C for 10 minutes and soaking in a 5% salt concentration), with total expected value of 6.01. The characteristics of treatment B2G4 resulted as water 12.35%, ash 2.70%, protein 2.71%, fat 0.22%, carbohydrate 82.05%, Ca 0.23%, crude fiber 3.43%, starch 56.26%, amylose 22.03%, amylopectin 34.27%, gelatinization temperature 90.67 °C, gel strength 0.23 N, and water absorption 7.95%. It found that *kimpul* flour with low Ca content revealed itching sensation was removed.

Keywords: Kimpul flour, Oxalate, Itching sensation

# Introduction

Assessing the potential of regional resources, Indonesia's natural resources have the potential of diverse food availability. Carbohydrate food sources usually come from cereals, tubers, and fruits. As alternative carbohydrate food sources and as a substitute for rice, the above food ingredients can be served in the daily diet, as long as they are enriched with high protein food sources (Widowati, 2009). *Kimpul* can be developed as a potential non-rice carbohydrate product. Another advantage of *kimpul* is its superior nutritional content, especially digestible protein and minerals such as calcium, phosphorous, and magnesium (Aniekwe, 2015; Boakye *et al.*, 2018; Wada *et al.*, 2019). But it also has a weakness, namely the content of antinutritional substances such as phytates and tannin (Wada *et al.*, 2019), hydrocyanic acid (HCN) (Vela-Guti érrez *et al.*, 2022), and oxalate content. Total oxalate ranged from 127 to 831 mg/100 g DM while the soluble oxalate

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ranged from 59.7 to 483 mg/100 g DM. The proportion of soluble oxalate ranged from 7.0% to 70.0% (overall mean 38%) of the total oxalate content of the tubers. The overall mean calcium content was 155 mg/100 DM and the percentage of insoluble calcium bound as calcium oxalate ranged from 24% to 88.9% of the total calcium content (overall mean 55%) (Hang, 2020). According to Sefa-Dedeh and Agyir-Sackey (2004), the oxalate content varies in the different cultivars and in different parts of the plant. Oxalate content can cause the itching sensation of *kimpul*.

This weakness of *kimpul* can be overcome by treatment in the *kimpul* processing to reduce anti-nutritional substances. The product resulting from the treatment is generally in the form of flour. *Kimpul* flour will have a wide use as a food ingredient, however in the form of tubers, *kimpul* consumption is still limited to chips and steamed tubers. *Kimpul* flour can also be made into composite flour to increase its nutritional value and can be made into processed products, such as biscuits (Puspitasari *et al.*, 2015), wet noodles (Revitriani *et al.*, 2013), and food bars (Rejeki *et al.*, 2019).

Various studies have been carried out to reduce the anti-nutritional substances contained in *kimpul* by various treatments, namely soaking, boiling, and roasting. Results showed that boiling effected the highest oxalate reduction (Iwuoha and Kalu, 1995; Owuamanam *et al.*, 2013; Vela-Guti érrez *et al.*, 2022). Reducing oxalate levels can also be done with acetic acid (Agustin *et al.*, 2017), using baking soda solution (Kumoro *et al.*, 2014), and a combination of physical treatment (boiling and steaming) and immersion treatment (Sulaiman *et al.*, 2021). Therefore, it is necessary to study other processing methods to relieve the itching sensation that are more natural, namely using salt combined with blanching. The aim was to determine the process for eliminating the itching sensation of *kimpul* flour; and the physical and chemical characteristics of *kimpul* flour.

# Materials and methods

## Materials and tools

The research was conducted in the Industrial Product Analysis Laboratory of the Agro-industrial Technology, Engineering Faculty, Universitas Wijaya Kusuma Surabaya. The materials were included the main raw materials, auxiliary materials, and chemicals for analysis. The main raw material was "*Kimpul*" tuber (*Xanthosoma sagitifolium* sp.), while the auxiliary material was salt (NaCl). Chemicals for analysis included aquadest, diethyl ether (Merck, GR), K<sub>2</sub>SO<sub>4</sub> (Merck, GR), HgO (Merck, GR), H<sub>2</sub>SO<sub>4</sub> (Merck, GR), NaOH

(Merck, GR),  $Na_2S_2O_3$  (Merck, GR),  $H_3BO_3$  (Merck, GR), BCG-MR (Merck, GR), HCl (Merck, GR), ethanol, HClO<sub>4</sub> (Merck, GR), Cu (Merck, GR), Nelson (Merck, GR), and filter paper (Whatman). The tools included processing tools and chemical analysis tools. The tools for processing included a paring knife, chopper, blanching pan, dryer, flour machine, and sieve. The tools for analysis included an analytical balance (Ohaus), drying oven (Binder), muffle furnace (Wisd), water bath (Memerth), and glassware (Pyrex).

## Experimental design

The experiment design was used 2 factors factorial in randomized complete block design. Factor I was blanching with 3 levels, namely: B1: no blanching, B2: blanching at 70  $^{\circ}$ C for 10 minutes, and B3: blanching at 70  $^{\circ}$ C for 20 minutes. Factor II was soaked in salt solution with 4 levels, namely: G1: no soaked, G2: soaked in 1% salt concentration, G3: soaked in 3% salt concentration, and G4: soaked in 5% salt concentration.

# Kimpul flour preparation

*Kimpul* was obtained from a local traditional market (Sidoarjo, Indonesia). The tubers were peeled to remove the brown, slightly hairy skin of the knots. Stripping was done using a stain-less steel knife. Then, washing was carried out to reduce mucus from the peeled *kimpul* as well as to remove other impurities that may be attached to the peeled *kimpul* tuber. The peeled tubers were cut to reduce the size and to increase the surface area. Furthermore, the itching relief treatment was carried out according to the research treatment, namely by blanching with 3 levels, and soaking in salt solution with 4 levels, which was repeated 3 times. The *kimpul* tubers were treated to relieve the itching sensation which drained to remove the remaining water. Furthermore, drying was carried out by means of artificial drying using a cabinet dryer. The dried tubers were crushed by grinding to produce *kimpul* flour, which was sifted using an 8 Mesh sieve to make the size uniform.

# Parameters of the chemical and physical and characteristics of kimpul flour

The characteristics of *kimpul* flour, including the chemical and physical properties of *kimpul* flour were investigated. Chemicals parameters testing was carried out on *kimpul* flour, as water content test using the gravimetric method (Nielsen, 2010), ash content using the ashing method (Nielsen, 2010), protein content test using the micro kjehldal method (AOAC, 1990), fat content with

Soxhlet (Nielsen, 2010), carbohydrate content test using the by difference method (Nielsen, 2010), Ca content using the spectrophotometer method, and crude fiber content test (AOAC International, 1995), starch content using the acid hydrolysis method (AOAC, 1990), and amylose content test using the standard curve method (AOAC International, 1995). Further, the physical parameters were observed, the gelatinization temperature, gel strength, and water absorption.

#### Data analysis

Analysis of data on chemical and physical parameters were carried out by Analysis of Variance (ANOVA); Duncan Multiple Range Test was carried out with a 95% confidence level.

## Results

## **Chemical characteristics**

The results for the chemical characteristics of *kimpul* flour showed that the water content of *kimpul* flour ranged from  $10.82 \pm 0.03\%$  to  $13.57 \pm 0.02\%$  (Table 1). The analysis of variance for the water content parameter showed interaction between the treatments (p<0.05). The B2G1 treatment had the lowest water content (10.82  $\pm 0.03\%$ ), while the highest water content was in the B3G3 treatment (13.57  $\pm 0.02\%$ ).

The analysis of variance for the ash content parameter showed interaction between the treatments (p<0.05). The ash content ranged from  $1.68\pm0.01\%$  to  $3.85\pm0.02\%$ , with B3G1 treatment had the lowest ash content ( $1.68\pm0.01\%$ ) and the highest ash content was in B3G4 treatment ( $3.85\pm0.02\%$ ).

The protein content ranged from  $2.36\pm0.02\%$  to  $3.00\pm0.02\%$ . The lowest protein content was in the B3G3 treatment ( $2.36\pm0.02\%$ ), while the highest protein content was in the B2G1 treatment ( $3.00\pm0.02\%$ ). The analysis of variance for the protein content parameter showed interaction between the treatments (p<0.05).

The analysis of variance for the fat content parameter showed interaction between the treatments (p<0.05). The fat content ranged from  $0.22\pm0.02\%$  to  $0.47\pm0.03\%$ . The lowest fat content was in the B2G4 treatment ( $0.22\pm0.02\%$ ), while the highest fat content was in the B1G4 treatment ( $0.47\pm0.03\%$ ).

The carbohydrate content using the by difference analysis of *kimpul* flour ranged from  $80.61\pm0.01\%$  to  $83.13\pm0.02\%$ . The analysis of variance for the carbohydrate content parameter showed interaction between the treatments

(p<0.05). The lowest carbohydrate content was in the B2G1 treatment ( $80.61\pm0.01\%$ ), while the highest carbohydrate content was in the B2G2 treatment ( $83.13\pm0.02\%$ ).

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3 +0.02 +0.03 -0.02 -0.02 -2 -0.01 -1 +0.02 -0.02 -0	30.73 <sup>b</sup> ±0.
	)2
$B3G  13.53^{j}  3.85^{h}  2.69^{e} \pm  0.31^{b} \pm  80.67^{a} \pm 0.0  0.15^{ab} \pm  4.17^{j} \pm 0.0  62.88^{l}  22.89^{d} \pm  4.13^{l} \pm 0.0  0.15^{ab} \pm  0.13^{l} \pm 0.0  0.15^{l} \pm 0.0  0.05^{l} \pm 0.05$	$40.04^{1}\pm0.$
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 Table 1. Chemical characteristics of kimpul flour

Note: different letters in the same column indicate a significant difference (p < 0.05)

The calcium (Ca) content of *kimpul* flour ranged from  $0.12\pm0.01\%$  to  $0.23\pm0.02\%$ . The lowest calcium content  $0.12\pm0.01\%$  were shown in treatments (B1G1, B3G1, and B3G3), while the highest calcium content was in the B2G4 treatments ( $0.23\pm0.02\%$ ). The analysis of variance for calcium content showed no interaction between treatment (p>0.05), and the both of factors (blanching factor and soaking in salt solution factor) were no significantly affected the calcium content.

The analysis of variance for the crude fiber parameters showed interaction between treatments (p<0.05). The crude fiber content ranged from 2.91  $\pm$ 0.01% to 4.17  $\pm$ 0.01%. The lowest crude fiber content 2.91  $\pm$ 0.01% was in the B1G1 treatment, while the highest crude fiber content was in the B3G4 treatment (4.17  $\pm$ 0.01%.).

The starch content of *kimpul* flour ranged from  $51.23\pm0.02\%$  to  $62.88\pm0.04\%$ . The lowest starch content was in the B2G3 treatment ( $51.23\pm0.02\%$ ), while the highest starch content was in the B2G4 treatment ( $62.88\pm0.04\%$ ). The analysis of variance for the starch content parameters showed interaction between treatments (p<0.05).

The analysis of variance for the amylose content parameters showed interaction between treatments (p<0.05). The amylose content ranged from 20.95±0.04% to 24.61±0.01%. The lowest amylose content was in the B3G2 treatment (20.95±0.04%), while the highest amylose content was in the B1G1 treatment (24.61±0.01%).

Furthermore, the amylopectin content ranged from  $27.93\pm0.02\%$  to  $40.04\pm0.01\%$ . The lowest amylopectin content was in the B2G3 treatment ( $27.93\pm0.02\%$ ), while the highest amylopectin content was in the B3G4 treatment ( $40.04\pm0.01\%$ ). The analysis of variance for the amylopectin content parameters showed interaction between treatments (p<0.05).

	Parameter								
Samples	Gelatinization	Gel Strength (N)	Water Absorption (%)						
	Temperature ( $^{\circ}$ C)								
B1G1	83.67 <sup>k</sup> ±0.58	0.53°±0.058	10.16 <sup>b</sup> ±0.02						
B1G2	82.33 <sup>j</sup> ±0.58	$0.37^{b} \pm 0.058$	9.91 <sup>a</sup> ±0.02						
B1G3	$84.67^{h}\pm0.58$	$0.27^{ab} \pm 0.058$	9.23 <sup>b</sup> ±0.02						
B1G4	86.33 <sup>e</sup> ±0.58	0.33 <sup>ab</sup> ±0.058	8.02 <sup>c</sup> ±0.01						
B2G1	86.67 <sup>1</sup> ±0.58	$0.27^{ab} \pm 0.058$	10.53 <sup>c</sup> ±0.04						
B2G2	88.33 <sup>f</sup> ±0.58	$0.27^{ab} \pm 0.058$	8.46 <sup>d</sup> ±0.04						
B2G3	84.33 <sup>i</sup> ±0.58	0.23 <sup>a</sup> ±0.058	9.85 <sup>b</sup> ±0.02						
B2G4	90.67 <sup>d</sup> ±0.58	0.23 <sup>a</sup> ±0.058	7.95 <sup>f</sup> ±0.04						
B3G1	89.33 <sup>b</sup> ±0.58	0.23 <sup>a</sup> ±0.058	7.37 <sup>e</sup> ±0.02						
B3G2	86.67 <sup>g</sup> <u>±</u> 0.58	0.37 <sup>b</sup> ±0.058	9.09 <sup>c</sup> ±0.02						
B3G3	84.67 <sup>a</sup> ±0.58	$0.57^{\circ} \pm 0.058$	6.91 <sup>b</sup> ±0.01						
B3G4	86.33 <sup>c</sup> ±0.58	$0.23^{a}\pm0.058$	$7.76^{\circ} \pm 0.02$						
Sig.	0.00	0.00	0.00						

Table 2. Physical characteristics of kimpul flour	Table 2.	Physical	characteristics	of kimpu	<i>l</i> flour
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Note: different letters in the same column indicate a significant difference (p < 0.05)

# **Physical characteristics**

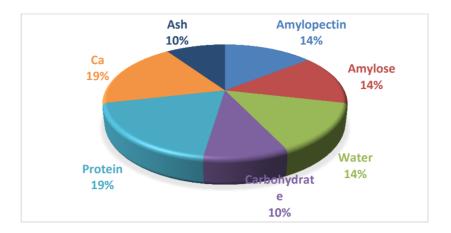
The physical characteristics showed that the gelatinization temperature ranged from 82.33  $\pm 0.58 \,^{\circ}$ C to 90.67  $\pm 0.58 \,^{\circ}$ C (Table 2). The lowest gelatinization temperature 82.33  $\pm 0.58 \,^{\circ}$ C was in B1G2 treatment, while the highest gelatinization temperature was in the B2G4 treatment (90.67  $\pm 0.58 \,^{\circ}$ C). The analysis of variance for the gelatinization temperature parameter showed interaction between treatments (p<0.05). The gel strength of *kimpul* flour ranged from 0.23  $\pm 0.058$ N to 0.57  $\pm 0.058$ N, with the lowest gel strength 0.23  $\pm 0.058$ N were shown in (B2G3, B2G4, B3G1, and B3G4), and the highest gel strength was in the B3G3 treatment (0.57  $\pm 0.058$ N). The analysis of variance for the gel strength parameters showed interaction between treatments (p<0.05). The water absorption ranged from 6.91  $\pm 0.01\%$  to 10.53  $\pm 0.04\%$ . The lowest water absorption 6.91  $\pm 0.01\%$  was in B3G3 treatment, while the highest water absorption was in the B2G1 treatment (10.53  $\pm 0.04\%$ ). The analysis of variance for the water absorption of *kimpul* flour showed interaction between treatments (p<0.05).

# Alternative selection

Alternative selection was done to determine the best treatment in the processing of *kimpul* flour. The basis of calculation for alternative selection was the product quality result for each parameter and the probability of each parameter. The concept of the expected value decision was to choose a decision that has the maximum payoff (profit or use) or minimum cost (loss or sacrifice). The quality parameters of *kimpul* flour products that used to select the best alternative process were the contents of amylopectin, amylose, water, carbohydrate, protein, calcium, and ash.

The first step in the alternative selection stage was determined the weight of the importance of each product quality parameter. Based on conditions for quality include amylopectin, amylose, water, carbohydrate, protein, calcium, and ash content, the importance weight for each quality parameter is listed in Figure 1.

Analyze to alternative selection used to expected value method. Result of expected value for each treatment displayed on Table 3.



**Figure 1**. Graph of the importance weight of each quality parameter of *kimpul* flour

Paramete						Treat	ments					
rs	B1G	B1G	B1G	B1G	B2G	B2G	B2G	B2G	B3G	B3G	B3G	B3G
	1	2	3	4	1	2	3	4	1	2	3	4
Amylopec												
tin	0.93	1.08	0.67	0.70	0.85	1.44	0.00	0.79	0.64	0.36	0.35	1.50
Amylose	1.50	1.03	1.20	0.91	0.99	0.88	0.95	0.44	0.85	0.00	0.39	0.80
Water	0.16	0.20	0.59	0.78	0.00	0.71	0.75	0.83	1.17	1.20	1.50	1.48
Carbohydr												
ate	0.90	0.82	0.49	0.27	0.00	1.00	0.91	0.57	0.65	0.90	0.12	0.02
Protein	1.91	0.38	0.69	1.00	2.00	0.28	0.09	1.09	1.66	0.63	0.00	1.03
Ca	0.00	0.18	1.27	0.73	0.55	1.09	1.64	2.00	0.00	0.36	0.00	0.55
Ash	0.28	0.48	0.46	0.46	1.00	0.13	0.28	0.29	0.00	0.15	3.10	0.61
Total	5.67	4.17	5.37	4.84	5.39	5.53	4.62	6.01	4.96	3.60	5,46	5.98

Table 3. Expected value of each kimpul flour treatment

Based on the results of the alternative selection in Table 3, the highest expected value was obtained for the B2G4 treatment (blanching at 70  $^{\circ}$ C for 10 minutes and soaking in 5% salt concentration) with total expected value 6.01, so that the B2G4 treatment was selected.

# Discussion

Based on the results of the water content of *kimpul* flour ranged from  $10.82\pm0.03\%$  to  $13.57\pm0.02\%$ . This value was less than 14%, so it can prevent mold growth (Damayanti and Suwita, 2018). The water content of *kimpul* flour

as a type of tuber was ranged almost the same as the water content of other tuber flours as reported by Hasbullah *et al.* (2017) which compared to the water content of *kimpul* flour (Zuhro *et al.*, 2015), the water content of this research was still higher. It was presumably due to soak in a salt solution which supported by Sitanggang *et al.* (2019) who stated that the interaction between the concentration of salt and polyphosphate was significantly affected in the response to the difference in water holding capacity (Damez and Clerjon, 2008). According to Sopandi and Wardah (2014), the amount of water in the material affected the resistance of the material to damage which caused by microbes. Drying of flour and starch reduced the water content to inhibited microbial contamintaion and enzyme activity that damaged to flour and starch. The water content limit at which microbes can still grow is 14-15% (Sopandi and Wardah, 2014).

Result showed that the water content of *kimpul* flour products tendeds to increase in the blanching treatment with a longer time. The highest water content was in the B3G3 treatment (blanching at 70 °C for 20 minutes and soaking in 3% salt concentration), while the lowest water content was in the B2G1 treatment (blanching at 70 °C for 10 minutes and no soaking). Based on the results of Kusumawati *et al.* (2012) who reported that, the increase in water content due to the blanching process which causes the starch contained in the material to swell, and greatly increasing the ability to absorb water.

Quantitatively the value of ash content in flour can arise from the minerals in the raw materials. It is found that the higher the salt content in the soaking treatment, the higher the mineral content in the flour. It may due to immersion in NaCl solution causing the accumulation of sodium and chloride minerals in the treatment sample. The higher mineral content in the tubers caused the ash content of the *kimpul* flour increased. It is supported by Lim (2015). Desniar *et al.* (2009) stated that salt contains minerals such as sodium and chloride, there is an osmosis process where the hygroscopic NaCl solution absorbs and remove water from taro tubers, and some of the solids in the NaCl solution enter the tubers through a diffusion process.

The protein content of *kimpul* flour was still low when compared to previous research results (Richana and Sunarti, 2004; Hasbullah *et al.*, 2017), which the flour of *suweg*, cassava, and *gembili* had high protein contents, namely 5.22%; 6.66%; and 6.11%, respectively. Interaction between the blanching and soaking salt solution affected protein content. NaCl can interact with proteins, at low concentrations, the protein salts at high concentrations would occur, the protein salts out. In the salting-in process, the protein will be more soluble, on the contrary, in the salting-out event, the protein will settle and not dissolve easily. The protein content of *kimpul* flour tended to decrease

in the salt soaking treatment with increasing concentration. It is due to the salting out process so that protein solubility is reduced, and the protein separated as a precipitate. In addition, salt has a high osmotic pressure so that it can attract water from the material (Winarno, 1986).

The fat content in starch and flour can interfere with the gelatinization process because fat is able to form complexes with amylose thereby inhibiting the release of amylose from starch granules. In addition, most of the fat would be absorbed by the surface of the granule, so that it forms a hydrophobic fat layer around the granule. The fat layer will inhibit the binding of water by the starch granules. It causes the viscosity and stickiness of the starch to decrease due to the reduced amount of water for the development of starch granules (P érez *et al.*, 2007). The longer blanching resulted decrease in fat content; the decreased in fat content is due to the loss of total solids during blanching (Aminah and Hersoelistyorini, 2012). Starch content is one of the quality criteria for flour, both as food and non-food ingredients (Richana and Sunarti, 2004).

The carbohydrate content of *kimpul* flour products tended to decrease in the salt soaking treatment with higher concentrations. The highest carbohydrate content was in the B2G2 treatment while the lowest carbohydrate content was in the B2G1 treatment. The results for the carbohydrate content showed that there was an interaction between treatments on the carbohydrate content of *kimpul* flour. The blanching factor and soaking in salt solution were significantly affected the carbohydrate content of *kimpul* flour.

As comparison with the Ca content in *kimpul* tubers of 1%, there was decreased in Ca content in *kimpul* flour. The decrease in Ca content occurred due to the reaction between sodium chloride (NaCl) and calcium oxalate (CaC<sub>2</sub>O<sub>4</sub>). Salt (NaCl) dissolved in water decomposed into Na<sup>+</sup> and Cl<sup>-</sup> ions. These ions are like magnets. Na<sup>+</sup> ions attract negatively charged ions and Cl<sup>-</sup> ions attract positively charged ions. Meanwhile, calcium oxalate (CaC<sub>2</sub>O<sub>4</sub>) in water decomposes into Ca<sup>2+</sup> and C<sub>2</sub>O<sub>4</sub><sup>2-</sup> ions. Na<sup>+</sup> binds to C<sub>2</sub>O<sub>4</sub><sup>2-</sup> ions to form sodium oxalate (Na<sub>2</sub>C<sub>2</sub>O<sup>4</sup>). The Cl- ion binds Ca<sup>2+</sup> to form a white precipitate of calcium dichloride (CaCl<sub>2</sub>) which is easily soluble in water. Various studies have shown that processing methods such as boiling, fermentation, and roasting can significantly reduce anti-nutritional factors (phytates and tannin) to a low level (Adane *et al.*, 2013; Azene, 2017; Ramakrishna *et al.*, 2006). Boiling reduced 69% of the oxalate found in raw Boloso (Azene, 2017). The results obtained in this study agreed with others (Iwuoha and Kalu 1995 *in* Adane *et al.*, 2013) who reported as 65.7-82.1% reduction of oxalates by boiling.

Amylose and amylopectin affect the nature of the flour produced. The functional properties of starch in flour are also influenced by the variety, natural conditions, and the place of origin of the plant (Srichuwong *et al.*, 2005; Riley *et al.*, 2006). The tendency for retrogradation to cause crystallization is accompanied by small amylose molecules and long amylopectin chains (Peroni *et al.*, 2006). Amylopectin is a component that plays an important role in the gelatinization process. High levels of amylose can reduce the ability of starch to undergo gelatinization (Tester and Morrison, 1990; Srichuwong *et al.*, 2005). The amylose content affects gelatinization and retrogradation properties, swelling power, and enzymatic susceptibility of starches. High levels of amylose can reduce the ability of starch starches. High levels of amylose can reduce the ability of starch to undergo gelatinization (Tester and Morrison, 1990; Peroni *et al.*, 2006).

It concluded that the highest expected value was obtained for the B2G4 treatment (blanching at 70 °C for 10 minutes and soaking in 5% salt concentration) with total expected value 6.01, and the B2G4 treatment was selected. Characteristic of B2G4 treatment had content of amylopectin  $34.27 \pm 0.01$ , amylose  $22.03 \pm 0.02$ , starch  $56.26 \pm 0.02$ , crude fiber  $3.43 \pm 0.02$ , calcium  $0.23 \pm 0.02$ , carbohydrate  $82.05 \pm 0.01$ , fat  $0.22 \pm 0.02$ , protein  $2.71 \pm 0.02$ , ash  $2.70 \pm 0.01$ , and water  $12.35 \pm 0.01$ .

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