Characteristics of Chemical and Functional Properties of Modified Cassava Flour (*Manihot esculenta*) by Autoclaving-Cooling Cycles Method

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Abstract. The modified cassava flour can be made using the method of the autoclaving cooling cycle (AAC). The stability of the warming can be seen from the decreasing value of breakdown viscosity, while the stability of the stirring process can be seen by the decreasing value of setback viscosity. The stages of research include: (1) the making of cassava flour, (2) the making of modified cassava flour by the method of treatment of ACC with a variety of flour concentration and autoclaving time, (3) chemical analysis of the moisture, ash, fat, protein, carbohydrate; The functional properties of the pasting characteristics to the initial temperature of the pasting, peak viscosity, hot paste viscosity, breakdown viscosity, cold paste viscosity and setback viscosity. The result shows that cassava flour modified by treatment of flour concentration 16% and autoclaving time 41 minutes having pasting code and pasting viscosity which is resistant to high temperature. Flour with this character is flour that is expected to maintain the texture of processed products with a paste form that remains stable. Utilization of modified cassava flour by the ACC method can be applied to the pasting product such as noodle and spaghetti, hoping to support for food diversification program to reduce dependence on wheat flour in Indonesia.

1. Introduction
In Indonesia, tubers are very potential to be developed as raw materials and supporting material for food and nonfood industry. One of the tubers that have potential as a source of carbohydrates for food diversification and as a functional food ingredient is cassava (*Manihot esculenta*). Part of cassava tuber contains about 25-28% starch and 10% fiber. In the processing of cassava into starch, obtained tapioca 20-25% and 11% cassava [1], whereas tapioca containing fat 0.08 to 1.54%, 0.03 to 0.06 % protein, and 0.02 to 0.33% ash [2]. One way to increase the efficiency of cassava is to create a modified cassava flour in order to obtain the properties of flour suitable for a particular application. Modified flour is given special treatment so resulted in better properties to correct previous properties and other properties, mainly physicochemical and functional properties. According to [3], the treatment can be classified physically and chemically. Chemically modified is a modification of starch or flour which the hydroxyl group has been converted through a chemical reaction (esterification or oxidation) or by disrupting its original structure. Modification of starch or flour physically involves several factors including: temperature, pressure, cutting, and the water content of the starch. In this study, cassava flour is done by physical modification using the method of autoclaving cooling cycles (ACC). According [4] heating treatment by using the method of autoclaving can increase the production of resistant starch to 9%.

The processing of starch modification can increase a value of functional food and has better quality. One of the food ingredients that are the result of starch modification and can be used as raw material for the manufacture of functional food is resistant starch [4]. The benefit of modified starch in...
the form of resistant starch can make the product more crispy, better in terms of mouthfeel, color and flavor if compared with traditional ingredients added products such as insoluble dietary fiber. Resistant starch also has low calory value, which amounted to 11.7 kJ / g RS or 1.9 kcal / g, so it can be used as an ingredient for low-calorie food [5]. The purpose of this study is to determine the characteristic of the chemical and functional modified cassava flour (Manihot esculenta) by using autoclaving-cooling cycles.

2. Research methods

2.1. Materials and tools

The study is conducted at the Laboratory of Post-Harvest Development, the center for Appropriate Technology Development, Indonesian Institute of Sciences, Subang. The raw material used is cassava Manggu variety with harvesting age of 9 months, obtained from cassava farmer in the village district of Gandasoli, Tanjung Siang, Subang district. Supporting material is a chemical used for chemical analysis and functional properties. Tools used include slicer; autoclave; cabinet dryer; vibrator screen; refrigerator; and tools for the analysis of moisture, ash, fat, protein; and Rapid Visco Analyzer (RVA) Newport RVA with Thermo Haake K20 for profile analysis of modified cassava flour paste.

2.2. Making cassava flour

The processing of making cassava flour refers to method Cassava [6] peeled and washed to remove sap and grime. Furthermore, the downsizing of the tubers sample use a slicer. Cassava that has been thinly sliced dried using a cabinet dryer at a temperature 50 °C to a moisture content up to 12%. The next phase, sliced tubers are dry powdered using a disk mill, and then sieved using a screen vibrator until the degree of fineness of 70 mesh. Flow diagram of the process of making cassava flour can be seen in figure 1.

2.3. Making Modified Cassava Flour

Manufacture of modified cassava flour refers to a method [6] arrowroot starch modified. Manufacture of cassava starch modified by the method of autoclaving cycles cooling is done by 3 cycles. Cassava flour is suspended with the addition of water (table 1) according to treatment. Then the suspension is gelatinized at a temperature of 70-85 °C. Further heated by using an autoclave at 121 °C 1 atm pressure and adjusted for the treatment time. The heating temperature used is 121 °C referring to previous research [7]. The suspension is heated further cooled at room temperature until the temperature drops, followed by storage at 4 °C for 12-16 hours. The next stage is drying using a cabinet dryer at temperature 50 °C to a maximum moisture content of 12%. Modified cassava flour which has been dried subsequently crushed and sieved use 70 mesh sieving. In figure 2 can be seen a flow diagram of the process of making cassava modified starch.

The resulting modified cassava flour then testing the characteristics of the chemical properties include: analysis of water content used gravimetry method; ash content used method gravimetry; protein content used Kjeldahl analysis method; fat content used soxhlet analysis methods [8], analysis of carbohydrates by the difference. Analysis of functional properties pasting characteristics includes initial pasting temperature; peak viscosity; hot paste viscosity; breakdown viscosity; cold paste viscosity; and setback viscosity, using the Rapid Visco Analyzer (RVA) Newport RVA with Thermo Haake K20.
Cassava

Cutting

Washing to remove sap and grime

Slicing

Drying with cabinet dryer at temperature of 50 °C

Drying with cabinet dryer at temperature of 50 °C

Flouring with disk mill and sieving vibrator until fineness 70 mesh screen

Cassava flour

Cassava flour

Suspending in water 10-20%

Glatinisation at 70-85 °C

Autoclaving on 121 °C, 15-60 minutes

Cooling to 4 °C for 12-16 hours

Drying to a moisture content of ≤ 12 %

Milling

Sieving at 70 mesh

Modified cassava flour

Figure 1. Flow diagram of the processing of making cassava flour [6].

Figure 2. Flow diagram of the processing of making cassava flour [6].

2.4. Treatment and data analysis

The research design that used in this study uses software Design Expert 7. System (DX-7, DX 7 system obtained 16 experimental design. The system generates design data of the input concentration and time autoclave. In table 1 we can see the design of experiment using Design Expert 7.

| Treatment | Concentration (%) | Autoclaving time (minute) |
|-----------|-------------------|--------------------------|
| 1         | 13                | 15                       |
| 2         | 20                | 60                       |
| 3         | 10                | 60                       |
| 4         | 10                | 18                       |
3. Research methods

3.1. Proximate analysis results of modified cassava flour

The proximate analysis result of modified cassava flour is presented in table 2. The modified cassava flour has a moisture content of 3.20-11.4%, according to standard for products processed from cassava edible (edible cassava flour) with a maximum moisture content of 13% [9].

The ash content in cassava modified starch is 1.54-2.3%. The ash content indicates total mineral in food. Mineral and inorganic salt in a small amount expressed as ash which is a food residue after the combustion process into carbon-free [10]. In the quantitative, value of ash content in flour and starch derived from mineral in the fresh tuber, fertilizer use, and can also come from the soil and air contamination during processing [11]. Maximum ash content is 3%, which is still allowed in edible cassava flour [9].

| No | Treatment | Water | Ash | Fat | Protein | Carbohydrate |
|----|-----------|-------|-----|-----|---------|--------------|
| 1  | 1         | 6.21  | 1.70| 1.17| 0.20    | 90.72        |
| 2  | 2         | 7.13  | 1.65| 0.44| 0.96    | 89.82        |
| 3  | 3         | 11.4  | 1.64| 1.23| 0.98    | 84.75        |
| 4  | 4         | 6.81  | 1.70| 1.10| 0.20    | 90.19        |
| 5  | 5         | 7.60  | 1.54| 1.05| 0.40    | 89.41        |
| 6  | 6         | 8.10  | 1.60| 1.14| 0.40    | 88.76        |
| 7  | 7         | 7.90  | 1.80| 1.15| 0.60    | 88.55        |
| 8  | 8         | 5.70  | 1.60| 0.66| 0.60    | 91.44        |
| 9  | 9         | 6.20  | 1.90| 0.51| 0.98    | 90.41        |
| 10 | 10        | 5.50  | 2.20| 0.60| 0.60    | 91.10        |
| 11 | 11        | 3.70  | 2.30| 0.16| 0.40    | 93.44        |
| 12 | 12        | 5.50  | 1.90| 1.13| 0.98    | 90.49        |
| 13 | 13        | 5.40  | 1.80| 1.76| 1.76    | 89.28        |
Modified cassava flour is produced from edible cassava. Therefore, the quality requirement of modified cassava flour may refer to CODEX STAN 176-1989 (Rev. 1-1995) of edible cassava flour (can be seen in table 3). Moisture content and ash from modified cassava flour is according to the requirements of CODEX STAN 176-1989 (Rev.1-1995).

| No | Parameter       | Unit | Quality Requirements |
|----|-----------------|------|----------------------|
| 1  | Water           | %    | Max 13               |
| 2  | Ash             | %    | Max 3                |
| 3  | Crude fiber     | %    | Max 2                |
| 4  | HCN             | mg/kg| Max 10               |
| 5  | Pesticide residues | - | Confirm              |
| 6  | Heavy metal     | -    | Not detectable       |
| 7  | Food additive   | -    | Not detectable       |

Source: [9]

Fat level obtained from the analysis of modified cassava flour is 0.16% -1.76%. According [12] the fat content is low and is at the boundary between 0.1-1.14%, except for treatment of 13 which has relatively high fat content. High-fat content is able to form a complex of amyllose-fat that can inhibit the release of starch granules.

Levels of a protein produced in modified cassava flour are relatively low, 0.20 to 2.45%. Generally bulbs - not including the root source of protein, so the value of its protein content is relatively low inversely proportional to the value of carbohydrate level. The test result shows that carbohydrate content of modified cassava flour has a high value ranged between 84.75-93.44%, according to research results [7] that cassava is a crop that contains high carbohydrate with the low level of amyllose and high amyllopectin, so it can be used as food ingredient source of carbohydrate instead of rice. According to [13], in addition to carbohydrate, fat and protein in the flour are very important to the formation of pasta, which will affect the texture of the food produced.

3.2. Analysis results of pasting characteristics for modified cassava flour

Cassava starch pasting profile changes before and after the modification can be seen in Table 4, includes initial pasting temperature, peak viscosity, hot paste viscosity, breakdown viscosity during heating, cold paste viscosity and setback viscosity.

Early temperature formation of pasta provides information on the required temperature of the starch granules begin to swell in the presence of water by the starch binding; time and peak viscosity can provide information related to the water holding capacity of starch and trends disintegration; breakdown viscosity (BDV) to provide information regarding resistance to warming; setback viscosity (SBV) to provide information regarding the potential retrogradation and syneresis, and the final viscosity to provide information regarding the potential ability to form gels [14]. Change of profile manioc starch paste is described as follows:
3.2.1. Early temperature pasting. Table 4 shows the effect of concentration and time ACC modification processing to changed pasting temperature from cassava flour pasta. Pasting temperature decreases from natural cassava flour, from 61.20 °C into 50.30 °C after ACC processes that occur at 20% concentrations and 20 minutes autoclaving. Delay the process of pasting shows that modified starch granules ACC is more stable and resistant to the heating process. This increase in pasting temperature is thought to be due to increasingly rigid granule due to changes in the crystalline starch region. The combination of concentration and time of heating used provide Significant pasting temperature changes on 4 and 11 of treatment compared with natural cassava flour. The results of this study is different from [15], that conducts research on starch amorphophalus, where the initial temperature pasting after modification HMT increases from 82.13 °C to be 86.54 - 88.56 °C. The same trend is happened also in sago starch [16], corn starch, peas, and lentils [17].

3.2.2. Peak viscosity. Peak viscosity is the ability of starch granules to inflate optimally when the flour pasta is heated. Cassava flour that undergoes ACC modification at various concentrations of flour and heating time decreased the peak viscosity value compared with natural cassava flour. The higher the concentration and the time of the modification process, the more decreased peak viscosity. The highest decrease in the viscosity of the peak occurs in modified cassava flour on treatment 3 (concentration of 10% flour, 60 minutes), and 14 (concentration of 15% flour, 60 minutes). According to [18], this peak viscosity indicates that HMT starch granules have inflated power and lower water absorption. This is influenced by the structure of the increasingly rigid starch granule as result of inter interaction and intramolecular granules that are getting stronger and tighter which will inhibit the penetration of water so that the swelling is limited.

Based on the research results [21], a presence of amylose-amylose chain interactions with the amylose-amylopectin chain occurring during the HMT process, causing the bond between the molecules are becoming tighter and water is increasingly difficult to penetrate into the granules. According [10] that the decrease in peak viscosity is influenced by the presence of fat amylose bond during the HMT process which can limit the interaction of starch molecules with other molecules outside the granule. This is thought to occur because cassava starch still contains small amounts of fat.

3.2.3. Hot pasta viscosity and breakdown viscosity. Measurement of hot paste viscosity and breakdown viscosity is to determine the effect of treatment ACC process to the stability of the starch paste during the heating process. Changes hot paste viscosity and breakdown viscosity of ACC cassava flour. The rate of the decrease occurred in various concentrations of cassava flour in variations of heating time. The result shows that the starch of ACC treatment has better thermal stability than natural starch. Breakdown viscosity value is getting lower with increasing the heating time. [19] suggested that the warming effect of HMT on white sorghum flour has a breakdown value which decreased significantly. The breakdown viscosity is one of the important factors when starch is applied to a product that demonstrates the stability of starch paste on heating. The smaller the value of the breakdown viscosity then starch will be more stable on heating condition. The Result of research [20] states that all of the parameters on the characterization of commodity starch paste on Indian water chestnut, which includes peak viscosity, hot paste viscosity, viscosity breakdown, and setback viscosity shows a declin.

| Table 4. Result of pasting profile analysis of modified cassava flour |
|-------------------------------------------------|
| Treatment          | Parameter of Cassava pasting profile |
| Native cassava     | Pasting Temp. (°C) | Peak Viscosity (cP) | Hot Paste Viscosity (cP) | Breakdown Viscosity (cP) | Cold Paste Viscosity (cP) | Setback Viscosity (cP) |
|                    | 61.20              | 2810                | 1138                   | 1672                    | 1741                     | 603                     |


### Cassava Flour

| ACC | 63.20 | 1822 | 1314 | 508 | 1759 | 445 |
|-----|-------|------|------|-----|------|-----|
| 2   | 59.60 | 2543 | 1068 | 1475| 1560 | 492 |
| 3   | -     | 87   | 64   | 23  | 127  | 63  |
| 4   | 51.10 | 3746 | 1325 | 2421| 1983 | 658 |
| 5   | 57.95 | 2029 | 1182 | 847 | 1669 | 487 |
| 6   | 50.20 | 2273 | 1130 | 1143| 1720 | 590 |
| 7   | 61.60 | 1922 | 1282 | 640 | 1710 | 428 |
| 8   | 61.95 | 30.82| 1254 | 1828| 1934 | 680 |
| 9   | -     | 582  | 329  | 253 | 631  | 302 |
| 10  | 59.60 | 2130 | 1089 | 1041| 1621 | 532 |
| 11  | 50.30 | 4590 | 1282 | 3308| 2033 | 751 |
| 12  | 57.55 | 3425 | 1296 | 2129| 2055 | 759 |
| 13  | 62.75 | 1863 | 1061 | 802 | 1586 | 525 |
| 14  | -     | 89   | 57   | 32  | 102  | 45  |
| 15  | 58.00 | 2829 | 1250 | 1579| 1993 | 743 |
| 16  | 77.25 | 748  | 470  | 278 | 740  | 270 |

### 3.3. Response surface method (RSM)

**3.3.1. RSM for treatment autoclaving cooling cycle and concentration of flour on parameter ash content.** Three-dimensional contour plot graph in Figure 3 illustrates the shape of the surface of the combination of the interaction between the component influences each other to test response value ash content. The difference in surface height demonstrates the value of different responses on any combinations of components. The low area shows low ash content test grade while the high area indicates that the ash content test value is high.

![Figure 3](image-url)
3.3.2. RSM for treatment autoclaving cooling cycle and concentration of flour on parameter ash content. Three-dimensional contour plot graph in Figure 4 illustrates the shape of the surface of the combination of the interaction between the components influence each other to test response value carbohydrate content. The difference in surface height demonstrates different response value for each combination of components. Low area indicates low carbohydrate test response rate while the high area indicates high carbohydrate test response rate.

![Figure 4](image1.png)

**Figure 4.** Three-dimensional graph of test results on the carbohydrate content response time autoclaving process optimization and concentration of cassava flour.

3.3.3. RSM for treatment autoclaving cooling cycle and concentration of flour on the breakdown viscosity (BV). Three-dimensional graph in figure 5 illustrates the results of a combination of surface response form two factors that influence the response of breakdown viscosity modified cassava flour. The difference in surface height indicates different response value for each combination of components. The low area shows a low breakdown viscosity value, whereas the high area shows a high viscosity breakdown value. The different colors also show the value of the response of breakdown viscosity. Blue color indicates the lowest breakdown viscosity, and green color indicates the highest viscosity breakdown response.

![Figure 5](image2.png)

**Figure 5.** The three-dimensional graph of test results Setback viscosity response at the time of autoclaving process optimization and concentration of cassava flour.

3.3.4. RSM for treatment autoclaving cooling cycle and concentration of flour on the setback viscosity. Contour three-dimensional graph plots can be seen in Figure 6. The lines consisting of dots on the graph plots contour show a combination of the two components with different amounts of certain desirability produce the same value. Point prediction in Figure 6 shows the combined concentration of cassava flour and autoclaving time that produce certain desirability value. Three-
dimensional graph shows the projection of contour plot. The low area on the three-dimensional graph shows the low desirability value, while the high area shows a high desirability value.

![Figure 6. The three-dimensional graph of test results Setback viscosity response at the time of autoclaving process optimization and concentration of cassava flour.](image)

4. Conclusions and recommendations
Modification of cassava flour from the varieties Manggu can be done with the method of autoclaving cooling cycle, so can increase the stability of the flour to the heating and stirring. Characteristics of the functional properties of cassava modified flour are determined by (1) the initial temperature of the formation of pasta which provides information about the required temperature of the starch granules to begin expanding in the presence of water binding by starch; (2) the time and peak viscosity that provide information related to the starch water binding and disintegration trends (3) breakdown viscosity (BDV) is to provide information regarding resistance to heating; (4) setback viscosity (SBV) providing information regarding potential retrogradation and syneresis, and (5) the final viscosity to provide information regarding the potential ability to form gel.

The research result shows that modified cassava flour with a flour concentration treatment of 16% and autoclaving time 41 minutes has to paste the code and paste viscosity which is resistant to high temperature. Flour with this character is a flour that is expected to maintain the texture of processed products with a paste form that remains stable. Utilization of modified cassava flour by this ACC method can be applied to the pasta product such as noodle and spaghetti, hoping to support food diversification program to reduce dependence on wheat flour in Indonesia

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