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# Statistic Evaluation of Extrusion Variabels on The Changing of The Pregelatinized Starch Characteristics

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**Abstract**
Most of the raw materials for pharmaceutical drugs including excipients are still imported products, whereas Indonesia has a large potential source of starch, such as cassava starch which can be used as pharmaceutical drug excipients. For this application, some modification needed to improve the characteristic of cassava starch. Physical modification using extruder is choosed for this research, because its easy to control the parameter during process. The effect of extrusion variables (frequency of feeder screw (KU), extrusion temperature (SUHU) and frequency of extruder screw (KS)) on change of characteristics of partially pregelatinized starch was analyzed using Minitab 15. The result analysis showed that the extrusion process variables had a significant effect on changes in starch viscosity, bulk density, tapped density and compressibility, and also starch flow rate. But it did not significantly influence the value of the angle of repose. Optimization of the extrusion variables analyzed using the counter plot overlaid graph. It shows that optimum variable area at KU = 3.5 Hz. All responnses at at KU = 3.5 Hz visible slice area feasible (at range of maximum and minimum value). So that the process conditions for production of pregelatinized starch from cassava starch by extrusion are recommended to be carried out in the area.
***Keywords:*** *Pregelatinized Starch, Cassava Starch, Statistic, Extrusion Variable.*

## 1. Introduction

The dependence of pharmaceutical raw materials is still felt by pharmaceutical industry in Indonesia. The data shows that almost 95% of the raw materials for medicines are still imported. While the market potential of Indonesian pharmaceutical drugs is growing rapidly along with the increase in population and public awareness of health. This condition caused a big trade deficit.

Various efforts have been made by the government to encourage the independence of pharmaceutical raw materials, one of which is to develop cassava starch products as excipients in pharmaceutical drugs. But characteristic of cassava starch still has some weaknesses that must be corrected, such as viscosity, flow rate and compressibility and hard to dissolved in cold water[1]. Characteristic improvement of cassava starch can be done with physical, chemical and biological modifications. One of the modified starches that is widely used as an excipient is pregelatinized starch. Its characteristic such as low viscosity can facilitate the distribution of the binder into the tablet period[2]. In addition pregelatinized starch has a better flow rate and compressibility when compared to native starch, so it can be used as a filler, binder and tablet disintegrant using wet granulation method at 5-10% concentration[2]. The characteristics of pregelatinized starch are very interesting to be studied further. Pregelatinized starch from cassava starch can be obtained so it can be applied as raw materials for pharmaceutical drugs and able to compete with imported excipient products.

One of technology that is used to produce pregelatinized starch is extrusion. This process modified the native starch by heat. Native starch with moisture content 25 -30% feeded to the extruder and then heated and pressed continuesly by screw and dye system. Some of granule of the starch will be broken, and produced gelation paste. After gelation process, the extrusion product (flakes) will be cooled and grinding to obtain the powder of pregelatinized starch. Partial gelatinization and retrogradation during this process changed the characteristic of starch. The extrusion variabels influence this process, especially temperature, residence time and pressure. The statistic evaluation of extrusion variabels on the changing of the product characteristic has not been found in references. Its very important to analysed because it can give a prediction for the optimal process.

The objective of this research was to obtain statistic evaluation of extrusion variabels on the changing of the product characteristic. The specific purpose in this research was to determine the effect of extrusion variables (frequency of feeder screw, extrusion temperature and frequency of extruder screw) on the changes of characteristics of partial pregelatinized starch from cassava starch.

## 2. Materials And Methods

### 2.1 Materials

Cassava starch was obtained from Tapioca Miniplant, National Laboratory of Starch Technology.

## 2.2 Methods

Cassava starch with 25-30% moisture content was steamed 15 minutes. And then it was cooled until room temperature. After that, It was fed to the extuder by hooper. The rotation frequency of the feeder screw, extrusion temperature and the frequency of extruder screw was set according to the test design in Table 1. Extrusion products (flakes) was dried and powdered using mill dryer. And then the characteristic of product will be analysed.

Table 1: The Design of Production Test of Partial Pregelatinized Starch by Extrusion

| Frequency of feeder screw = 3 Hz | | Frequency of ekstruder screw (Hz) | | | |
|---|---|---|---|---|---|
| | | 5 | 7,5 | 10 | 12,5 |
| Extrusion Temperature ($^0$C) | 40 | A1 | A2 | A3 | A4 |
| | 50 | C1 | C2 | C3 | C4 |
| | 60 | E1 | E2 | E3 | E4 |

| Frequency of feeder screw = 3,5 Hz | | Frequency of ekstruder screw (Hz) | | | |
|---|---|---|---|---|---|
| | | 5 | 7,5 | 10 | 12,5 |
| Extrusion Temperature ($^0$C) | 40 | F1 | F2 | F3 | F4 |
| | 50 | H1 | H2 | H3 | H4 |
| | 60 | J1 | J2 | J3 | J4 |

## 2.3 Evaluation of Characteristics of Partial Pregelatinized Starch

Evaluation was carried out by observing physical, and functional characteristics. The observed parameters includes: pH, water content, viscosity, bulk density, tap density, compressibility, flow rate, angle of repose and flow properties.

## 2.4 Data Analysis

The test results of the characteristics of pregelatinized starch in various extrusion variable according to the test design were statistical analyzed using MiniTab 15.

# 3. Result And Discussion

## 3.1 Physical Characteristic of extrusion Product (Partially Pregelatinized Starch/PGS)

The observation of changes in pH in each extrusion variable can be seen in Figure 1. Observation of pH on various extrusion variables does not show a significant pH changes. This is because the pH value depends on the pH of raw material and the pH of process water. If the pH of raw materials and process water are stable at normal pH (6.0-7.0), the pH value of PGS will also be relatively stable.

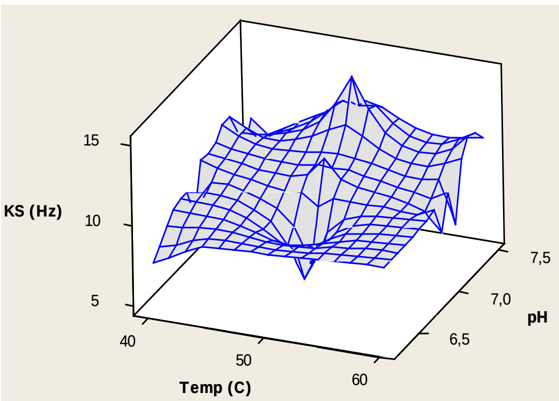


Fig. 1 Graph of changes on extrusion variables to pH values.

Observation of water content of PGS showed results in the range of 5.0-11%. The water content of PGS from cassava starch is more influenced by the conditions of the drying process. Figure 2 shows fluctuations in changes in water content but still in accordance with British Pharmacopeia[3] requirements ( maximum 14% ).

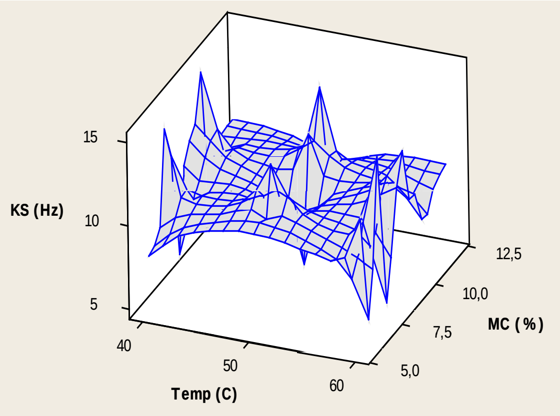


Fig. 2 Graph of changes in extrusion variables on the value of water content.

## 3.2 Functional Characterization of PGS

To determine the effect of extrusion variables on changes of PGS functional properties, experimental data was analyzed using Minitab 15. The result of ANOVA analysis results (at $\alpha = 0.05$) on each response observed (PGS functional characteristics) to extrusion variables : frequency of feeder screw (KU), frequency of screw extruder (KS) and extrusion temperature (SUHU) can seen in Tables 2 and 3.

Table 2: The Effect of Frequency of Feeder Screw (KU), Frequency of Screw Extruder (KS) and Extrution Temperature (SUHU) on The Functional Properties of PGS

| Response / Variable | Viscosity | Bulk Density | Tapped Density | Compressibility | Flow rate | Angle of repose |
|---|---|---|---|---|---|---|
| KU | -10,996 | 1,096 | -8,748 | 293,824 | -225,648 | 35,956 |
| KS | -0,179 | 0,289 | -1,699 | -66,305 | -70,904 | 17,204 |
| SUHU | -1,957 | 0,086 | -0,756 | -23,998 | -15,878 | 1,834 |
| Interaction KU-SUHU | 0,241 | -0,027 | 0,151 | 5,761 | 4,216 | -0,986 |
| Interaction KU-KS | -0,182 | -0,079 | 0,540 | 18,641 | 20,796 | -4,471 |
| Interaction SUHU-SUHU | 0,011 | 4,619e-5 | 0,003 | 0,048 | 0,015 | 0,018 |
| Interaction SUHU-KS | 0,016 | -0,008 | 0,030 | 1,275 | 1,299 | -0,431 |
| Interaction KS-KS | 0,003 | -1,028e-4 | -0,002 | 0,225 | 0,067 | -0,049 |
| Interaction KU-SUHU-KS | -0,0002 | 0,002 | -0.009 | -0,388 | -0,387 | 0,122 |

Table 3: Results of ANOVA Analysis at A = 0.05 Variable to Response

| Response / Variable | Viscosity | Bulk Density | Tapped Density | Compressibility | Flow rate | Angle of repose |
|---|---|---|---|---|---|---|
| Regression | 0,042 | 0,002 | 0,015 | 0,0004 | 0,0005 | 0,175 |
| KU | 0,463 | 0,036 | 0,013 | 0,0004 | 0,0025 | 0,599 |
| KS | 0,969 | 0,089 | 0,099 | 0,0030 | 0,0009 | 0,392 |
| SUHU | 0,072 | 0,021 | 0,004 | 0,0001 | 0,0012 | 0,707 |
| Interaction KU-SUHU | 0,416 | 0,011 | 0,027 | 0,0003 | 0,0014 | 0,443 |
| Interaction KU-KS | 0,897 | 0,128 | 0,087 | 0,004 | 0,0007 | 0,447 |
| Interaction SUHU-SUHU | 0,021 | 0,732 | 0,013 | 0,0037 | 0,0546 | 0,225 |
| Interaction SUHU-KS | 0,858 | 0,024 | 0,133 | 0,0024 | 0,0005 | 0,252 |
| Interaction KS-KS | 0,869 | 0,869 | 0,593 | 0,0029 | 0,0768 | 0,455 |
| Interaction KU-SUHU-KS | 0,995 | 0,035 | 0,131 | 0,0021 | 0,0004 | 0,279 |

## 3.3. Analysis of the effect of frequency of feeder screw (KU), frequency of screw extruder (KS) and extrusion temperature (SUHU) on the functional properties of PGS

### 3.3.1 Viscosity

Based on the p-value regression, results of ANOVA analysis showed that the frequency of feeder screw (KU), the frequency of screw extruder (KS) and extrusion temperature (SUHU) have a significant effect on changes in PGS viscosity (tables 2 and 3). The effect of KU, KS da SUHU will reduce the viscosity of PGS, as well as the interaction between KU-KS and KU-KS-SUHU. Conversely the interaction of SUHU such as KU-SUHU and SUHU-KS will increase the viscosity of PGS. Therefore, SUHU is an important factor to increase the viscosity of PGS. This is related to the degree of starch gelatination, where the viscosity pattern of starch tends to rise along with the increase in extrusion temperature (SUHU).

### 3.3.2 Bulk Density, Tapped Density, and Compressibility

Based on the p-value regression, results of ANOVA analysis showed that the frequency of feeder screw (KU), the frequency of extruder screw (KS) and extrusion temperature (SUHU) have a significant effect on changes in bulk density, tapped density and compressibility valaue (Tables 2 and 3). Interaction between two process variables ( KU-SUHU, KU-KS, SUHU-KS and SUHU-SUHU ) will increase the value of tapped density and compressibility value, but reduce the bulk density.

### 3.3.3 Flow Rate and Angle of Repose

The flow properties of powder is a critical factor in the production of solid dosage drugs. Because flow properties of powder have an affect on improving reproducibility of compression chamber filling in the manufacture of tablets and capsules. So that it can produce a better weight uniformity. Flow rate and angle of repose are two variables that determine the flow properties of powder. The higher the flow rate, the better the flow properties, where the flow rate of 4-10 g / s is included in the category of good flow characteristics. Whereas the lower the value of the stop angle, the better the flow properties of powder, where the angle of repose between 25-30 is included in the category of good flow characteristics[4], [5], [6].

Based on the p-value regression, results of ANOVA analysis showed that the frequency of feeder screw (KU), frequency of extruder screw (KS) and extrusion temperature (SUHU) have a significant effect on changes in flow rate but not significantly influence the value of the angle of repose. The interaction between two process variables ( KU-SUHU, KU-KS, SUHU-KS and SUHU-SUHU ) will increase the value of the flow rate. While the KU, KS and SUHU effects will decrease the flow rate. If thus PGS is compared with cassava starch and commercial PGS, the PGS flow rate has a much better value (classified as very good flow properties).

The angle of repose of PGS for all variations of the extrusion variables showed values between 19-25. Anyway, in general the the flow properties of the PGS was very good.

The next analysis is to create a regression equation. And then plot overlaid counter graphs can be made from each response. Regression equations of each response can be seen in table 4, while the plot overlaid counter graph is shown in Figure 5.

Table 4: Regression Equations For Each Response

| Response | Regression Equation |
|---|---|
| Viscosity | Viscosity (dPas) = 34,3872 - 5,49779 KU - 0,978605 SUHU - 0,0895804 KS + 0,120411 KU*SUHU - 0,0911085 KU*KS + 0,00529974 SUHU*SUHU + 0,00814634 SUHU*KS + 0,00158196 KS*KS - 8,21122e-005KU*SUHU*KS |
| Bulk Density | Bulk Density = -1,97493 + 0,547796 KU +0,0430204 SUHU + 0,144847 KS -0,0134692 KU*SUHU - 0,0393853KU*KS + 2,30935e-005 SUHU*SUHU -0,00377811 SUHU*KS - 5,14312e-005KS*KS + 0,00108696 KU*SUHU*KS |
| Tapped Density | Tapped Density = 15,7692 - 4,37416 KU - 0,378197 SUHU - 0,849819 KS + 0,0755751 KU*SUHU + 0,270202 KU*KS + 0,00130196 SUHU*SUHU + 0,0151141 SUHU*KS + 0,00117312 KS*KS - 0,00466563 KU*SUHU*KS |
| Compressibility | Compressibility (%) = 578,532 - 146,912 KU - 11,9989 SUHU - 33,1525 KS + 2,88034 KU*SUHU + 9,3203 KU*KS + 0,0240255 SUHU*SUHU + 0,637666 SUHU*KS + 0,112549 KS*KS - 0,193801 KU*SUHU*KS |
| Flow rate | Flow rate (g/s) = 411,064 - 112,824 KU - 7,93896 SUHU - 35,4518 KS + 2,10802 KU*SUHU + 10,398 KU*KS + 0,00732741 SUHU*SUHU + |

| | |
|---|---|
| | 0,649953 SUHU*KS + 0,0336708 KS*KS - 0,193251 KU*SUHU*KS |
| Angle of Repose | Angle of Repose = -26,691 + 17,9781 KU + 0,917237 SUHU + 8,60185 KS - 0,493193 KU*SUHU - 2,23562 KU*KS + 0,00897817 SUHU*SUHU - 0,215744 SUHU*KS - 0,0242653 KS*KS + 0,0607839 KU*SUHU*KS |

The quality requirements for PGS as pharmaceutical drug excipients do not yet exist in the Indonesian Pharmacopoeia, therefore this research uses the quality test of commercial PGS as a reference. After each contour plotted response, the maximum and minimum limits of the desired response are determined based on measurements of commercial PGS as a reference. Functional properties such as compressibility and flow properties are determined based on good flow properties of powder[4], [5], [6]

After the minimum and maximum limits of each desired response are determined, the optimum process conditions can be determined. For this reason, all of the counter plots that have been set minimum and maximum limits, overlap each other (counter plot overlaid) so that the sliced area will be obtained. The slice area is the optimum process variable area (feasible for all response variables) as shown in Figure 5 and 6.

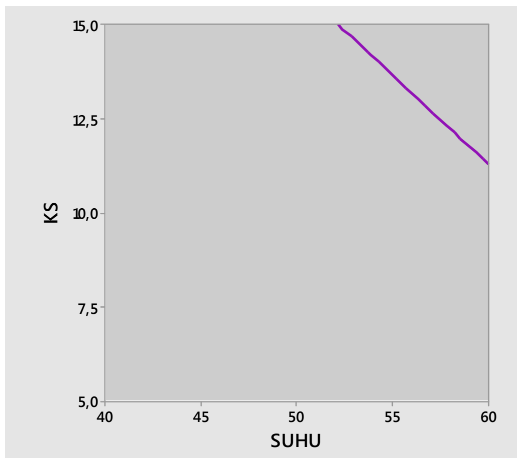


Fig. 5 Overlaid Plot Counter Graph for KU = 3.0

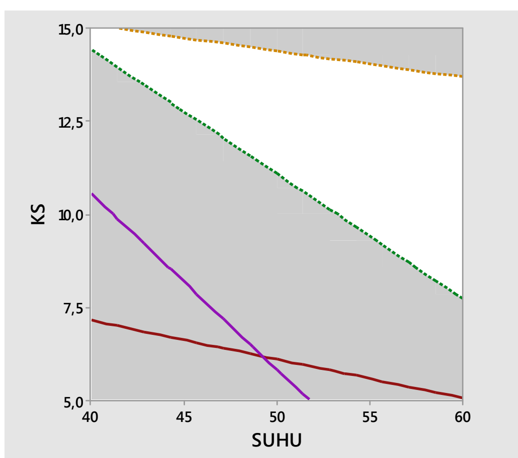


Fig. 6 Overlaid Plot Counter Graph for KU = 3.5

The counter plot graph, overlaid for KU = 3.0 Hz is not obtained by the slice area. This is because the response to water content, viscosity and pH is in the area of the maximum and minimum limits that have been set. However, on the counter plot graph, overlaid for KU = 3.5 Hz shows the slice area which is the optimum process variable area (white area). This shows that all responses are at maximum and feasible. So that the production of PGS by extrusion are suggested to be carried out in the area, in order to obtain a product that meets the specified requirements.

## 4. Conclusion

1. Extrusion variables includes frequency of feeder screw (KU), frequency of extruder screw (KS) and extrusion temperature (SUHU) have a significant effect on changes in PGS viscosity and PGS compressibility.
2. Extrusion variables includes frequency of feeder screw (KU), frequency of extruder screw (KS) and extrusion temperature (SUHU) have a significant effect on changes in flow rate but have no significant effect on angle of repose.
3. The optimum process variable area is obtained on the counter plot graph overlaid for KU = 3.5 Hz.

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