Preliminary Comparison of Pre-gelatinization vs Ultrasound Modified Sweet Potato Starch for Tablet Formulation

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Abstract: Starch is a common material for tablet formulation as disintegrant, binder, or filler, however, it needs to be modify to improve its native properties. Physical modification by heat is one of the easiest way to modify the starch in tablet formulation. Sweet potato starch were pre-gelatinized and ultrasound to improve some physical properties of starch. The aim of this study was to compare the properties of starch produce using two different treatment. The swelling power (SP), solubility (S), and water binding capacity (WBC) of starches are the properties which important in the usage of starch in tablet formulation. The increase of all parameters measured could be important for controlled drug delivery. The granule of starches were also analysed using scanning electron microscopy to determine the effect of different treatments toward starch granules.

1 INTRODUCTION

Indonesia is one of the main producer of sweet potato, however their use is still short compare to other tuber such as cassava or potato. According to FAOSTAT (2017) the trend of sweet potato production increases from 101,813,946 tonnes in 2012 up to 105,190,501 tonnes in 2016 with the total yield and production in 2015 are 160,533 hg/ha and 2,297,634 ton in Indonesia. Sweet potato's leaves and tubers are used as a source of nutrient for humans and livestock, whereas its usage can be expanded to increase the value.

Many industrial and pharmaceutical industry can use sweet potato starch as their ingredient, however it still have many shortages in the native form. Native starch has shortages in application due to its limited properties towards heat, easily break by shear forces, and the viscosity is low (Chi *et al.*, 2008; Das *et al.*, 2010), usually modification is used to overcome those shortages (Das *et al.*, 2010; Frank and Adebowale, 2010; Krishnakumar and Sajeev, 2018). Pre-gelatinization is physical modification known to produce easily soluble starch which improves its flow ability due to its loss in crystallinity (Visavarungroj and Remon, 1991; Freitas *et al.*, 2004).

Some research about pre-gelatinized starch for tablet formulation were done (Visavarungroj and Remon, 1991; Alebiowu and Itiola, 2002; Odeku, Schmid and Picker-freyer, 2008; Adedokun and Itiola, 2010; Jubril, Muazu and Mohammed, 2015). Physical modification is preferred due to less amount of by products and the possibility of chemical residue, so that this approach more sustainable (Krishnakumar and Sajeev, 2018).

Ultrasound is the sound above the threshold of human ear that can be used in some food processing, include the following: extraction, emulsification, homogenization, and separation (Jambrak *et al.*, 2010). Very limited studied have been reported on the effect of ultrasound treatment on starches (Jambrak *et al.*, 2010; Zhu, 2015; Krishnakumar and Sajeev, 2018). Ultrasound will induce the formation of cavitation filled with gas which can increased the temperature locally, modifying the physical and chemical conditions of the system (Jambrak *et al.*, 2010; Zhu, 2015). The aim of this research was to compare the properties of starch produce by pre-gelatinization and ultrasound.

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2 MATERIALS AND METHODS

2.1 Preparation of Starch

Sweet potato tubers were obtained from local market in Yogyakarta, Indonesia. The tubers were washed to remove the dirt, peeled, and grated using machine. The water was added into grated tuber to obtain the starch and the suspension filtered. The filtrate was precipitated and the water were removed. This process were repeated three times. The sediment of starch obtained then dried in the dryer and sieved. The starch was secured with silica gel and kept for further analysis.

2.2 Pre-gelatinization Modification

The design described by Adedokun and Itiola (2010) with modification was done to pre-gelatinized the starch. A quantity (100 ml) water was added into 100 g starch to make the suspension. The suspension was heated at 55˚C with constant stirring for 10 minutes. The paste then dried, pulverized, and sieved. The starch then kept with silica gel for further analysis.

2.3 Ultrasound Modification

The method of Jambrak *et al.* (2010) with modification was done to treat the starch. Prepared samples of 500 ml volume were place in the ultrasound bath and treated with ultrasound 24 kHz frequency. The temperature was set to 55˚C for 10 minutes. The paste then dried, pulverized, and sieved. The starch then kept with silica gel for further analysis.

2.4 Physical Characteristics

2.4.1 Swelling Power and Solubility Test

SP and S was measured by using Leach method with some modification (Kaur *et al.*, 2011; Lee and Yoo, 2011; Grace *et al.*, 2019). A mixture of starch was prepared in centrifuge tube then heated at 90˚C for 30 minutes and cooled down into room temperature. After that, the tube were centrifuged and the fraction obtained was separated. The sediment and liquid part then dried and counted.

2.4.2 Water Binding Capacity Test

WBC was measured by using Medcalf method with some modification (Robertson *et al.*, 2000; Iheagrawa, 2013; Grace *et al.*, 2019). A mixture of starch was prepared in tube and stirred for 1 hour and centrifuged. After that, the excess water was separated and the sediment part obtained was counted.

2.5 Morphological Characteristics

Native, pre-gelatinized, and ultrasound modified starches were observed for its morphological characteristics conducted with Scanning Electron Microscopy (SEM) (Hitachi TM3000, Japan). The accelerating potential used was 15 kV and the samples were coated with palladium.

3 RESULTS

3.1 Physical Characteristics

The SP, S, and WBC of native and treated starches are shown in Table 1. SP represent the interaction between amorphous and crystalline area inside the granule (Takizawa, Oliveira and Konkel, 2004). During the gelatinisation process, there is an increase in the SP and a large amount of starch content leaks from the granule (Mat *et al.*, 1992). The solubility of starch increased along with the increasing of process's temperature (Paterson *et al.*, 1994). The increase of S indicated an increase of solubilized amylopectin and this increase was enormous after the granule start to rupture (Srichuwong *et al.*, 2005).

Table 1: Physical characteristics of native, pre-gelatinized, and ultrasound modified starches (n=3).

Parameters	Native	Pre-gelatinized	Ultrasound
Swelling power (g/g)	17.95±0.55	30.31±1.06	36.05±0.11
Solubility (%)	1.48±0.07	26.55±1.18	2.41±0.09
Water binding capacity (%)	89.32±1.00	232.39±1.83	166.63±1.14

Pre-gelatinization could increase the solubility of starch in water probably due to the rupture of granule during process (Adedokun and Itiola, 2010; Grace *et al.*, 2020). The WBC of starch is observed higher due to the increasing of water binding sites inside the granules which represent the availability of the hydroxyl groups. The disruption of granule during gelatinization also increase the hydophilicity of the granule enabling the increasing of hydration of starch (Wootton and Bamunuarachchi, 1978).

The increase of SP and S in ultrasound modified starch also happen due to the loss of granule integrity after the swelling process happened. This loss could be happened due to the amorphous area become more damaged so that the hydrophylicity of starch increases (Herceg *et al.*, 2010). The increasing of SP and S value probably happened due to the weaken bonding within the crystalline area and the availability of hydrogen bonds (Luo *et al.*, 2008; Sujka and Jamroz, 2013). The increasing of starch WBC might be due to the result of gelatinisation that happen after the weaken bond inside amorphous area happened (Wootton and Bamunuarachchi, 1978). The higher availaibility of water penetration into the granule due to the disruption by mechanically damages of ultrasound process could leads to a higher water retention (Manchun *et al.*, 2012).

The increasing of physical characteristics was higher in conventional pre-gelatinization than in ultrasound process, even though the temperature of process were set at the same temperature. It might be due to there is an engagement of hydroxyl groups to form covalent and hydrogen bonds between starches in ultrasound starch. Apart from that, maybe there was an excessive amount of energy in ultrasound treated starch that could trigger the retrogradation happen higher than in pre-gelatinization.

3.2 Morphological Characteristics

The SEM of native, pre-gelatinized, and ultrasound modified starches are shown in Figure 1. The sweet potato starch consisted of mixed size granule from small to large and in various shapes (Das *et al.*, 2010). Polygonal is the most shape shown in the most of sweet potato starch granule, however there are also round and irregular shapes. Modification process can change the shape of granules (Babu, Parimalavalli and Jagannadham, 2014).

All the treatments of starch showed the improvement of starches' granule size. This improvement is related to the ability of the starch to trap the water inside the granule so the SP of starches was also higher in treated starch. From the pictures, pre-gelatinized starch has relatively larger granules than other starches. It indicates the swelling process happened inside the granule due to the process.

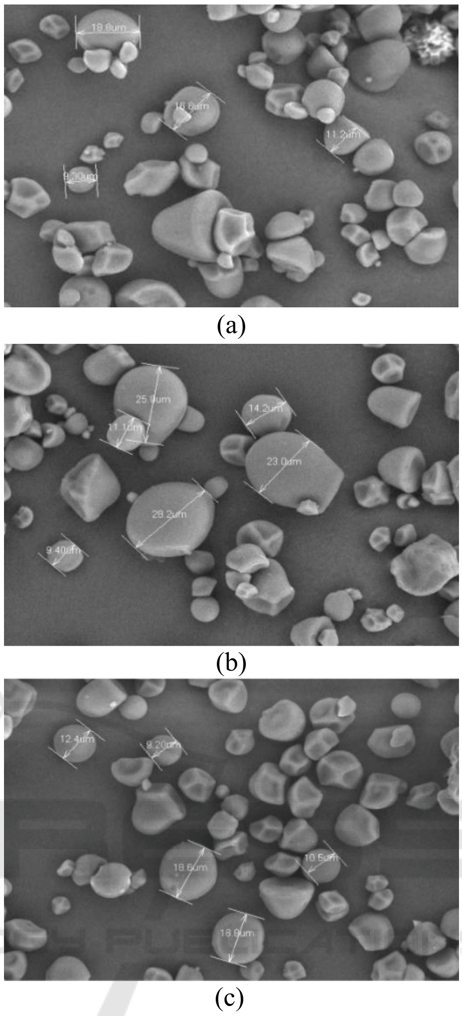

Figure 1: SEM of a) native; b) pre-gelatinized; c) ultrasound modified starch (1200x)

Ultrasound starch also has larger granules than native starch due to the process even though not as big as pre-gelatinized starch. These morphological characteristics was in accordance to the result of SP, S, and WBC earlier.

4 CONCLUSIONS

Compared to ultrasound starch, pre-gelatinized starch has resulted in greater changes in SP, S, WBC, and the size of granule. However, both modification results are better than native starch. These results could be adjusted according to industrial and pharmaceutical needs. The effect of pre-gelatinization or ultrasound treatment of sweet potato starch on the tablet formulation have not been studied enough yet. Some parameters should be optimized to gain the best results accordance with the needs.

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