Ph.D. Dissertation

RECOVERY OF STARCH FROM SAGO PITH WASTE TO INCREASE SAGO STARCH PRODUCTION IN INDONESIA

インドネシアにおけるサゴ抽出残渣からの
澱粉回収効率向上に関する研究

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COMMONLY USED ABBREVIATIONS

CM	Commercial method
cm	centimetre
DSC	Differential scanning calorimetry
D(v,0.1)	Cumulative volume of 10%
D(v,0.5)	Cumulative volume of 50%
D(v,0.9)	Cumulative volume of 90%
E	Electrical energy
Es	Specific energy consumption
F.N	Fraction Number
F1	Fraction 1
F2	Fraction 2
g	gram
GPC	Gel permeation chromatography
h	hour
IC	Ice-water coolant
J	Joule
kg	kilogram
kJ	kilojoule
kHz	Kilo Hertz
kW	kilowatt
kV	kilovolt
l	litre
mA	milliampere
MPM	Micro powder mill
MPM-T1	Micro-powder-milled sago starch of T1 treatment
MPM-T2	Micro-powder-milled sago starch of T2 treatment
MPM-T3	Micro-powder-milled sago starch of T3 treatment
MPM-T4	Micro-powder-milled sago starch of T4 treatment
mg	milligram
min	minute
mL	millilitre
mm	millimetre
RC	Relative crystallinity
SEM	Scanning electron microscopy
SPW	Sago pith waste
t	tons
T0	Untreated sago starch
T1	Wide disc clearance treatment
T2	Wide-medium disc clearance treatment
T3	Medium-narrow disc clearance treatment
T4	Narrow disc clearance treatment

T_o	Onset temperature
T_p	Peak temperature
T_c	Conclusion temperature
U	Unit
WC	Water coolant
XRD	X-ray diffraction
ΔH	Enthalpy of gelatinization
×	Multiplication
%	percentage
°C	degrees Celsius
µL	Microlitre
µg	microgram

CHAPTER 1. GENERAL INTRODUCTION

1.1. Food security

According to Food and Agriculture Organization (FAO) of the United Nation (2006), food security exists when all people, at all times, have physical and economic access to sufficient, safe and nutritious food that meets their dietary needs and food preferences for an active and healthy life. Furthermore FAO discussed that this widely accepted definition points to the following dimensions of food security as follow : (1) *Food availability* : the availability of sufficient quantities of food of appropriate quality, supplied through domestic production or imports (including food aid); (2) *Food access* : Access by individuals to adequate resources (entitlements) for acquiring appropriate foods for a nutritious diet. Entitlements are defined as the set of all commodity bundles over which a person can establish command given the legal, political, economic and social arrangements of the community in which they live (including traditional rights such as access to common resources); (3) *Utilization* : Utilization of food through adequate diet, clean water, sanitation and health care to reach a state of nutritional well-being where all physiological needs are met. This brings out the importance of non-food inputs in food security; and (4) *Stability* : To be food secure, a population, household or individual must have access to adequate food at all times (FAO, 2006). They should not risk losing access to food as a consequence of sudden shocks (e.g. an economic or climatic crisis) or cyclical events (e.g. seasonal food insecurity). The concept of stability can therefore refer to both the availability and access dimensions of food security.

The concept of food security is well-suited to facilitate the discussion and guide action on promising pathways out of hunger and malnutrition. However, as the consequences of the recent food crisis unfold, the concept of food security may require a stronger focus on nutrition outcomes. Over time, Ecker and Breisinger (2012) said that the concept of food security and related approaches to address food insecurity have been developed and modified in accordance with the common understanding of the nature of the food problem and the evolution of the global food system. Figure 1.1 presents a diagrammatic overview of the food and nutrition security (FNS) system. The framework shows the main factors of FNS on the macro and micro levels and their linkages across sectors and levels that, in combination, determine nutrition outcomes. It also illustrates the major channels through which external shocks/stress and interventions at the macro and micro levels sequentially translate into individual nutritional status and how this, in turn, affects the economic and social developments in countries and households (and their individual members).

Nowadays, food security becomes serious topic in the world because growth of population rate continuous increase. According to United States Census Bureau (2014) that the number of world population around 6.1 billion in 2000 and then increase around 13 % (6.9 billion) in 2010 (Fig. 1.2). Furthermore, it is projected that the population of the world with an average growth rate of about 0.8 %, the world population will increase to 8.3 billion by 2030; 8.9 billion by 2040 and 9.4 billion by 2050. This indicates that the world needs to produce at least 50% more food to feed 9 billion people by 2050.

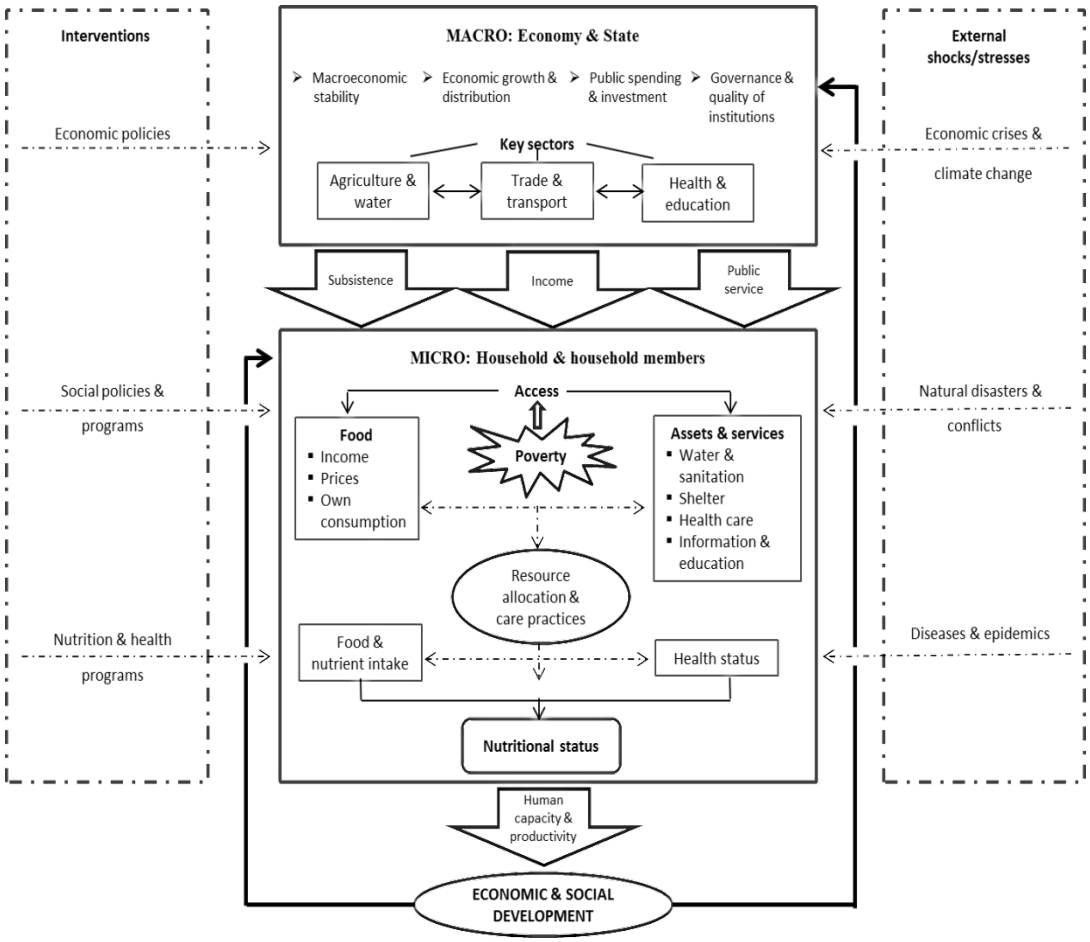

Figure 1.1. Overview of the food and nutrition security (FNS) system (Ecker and Breisinger, 2012)

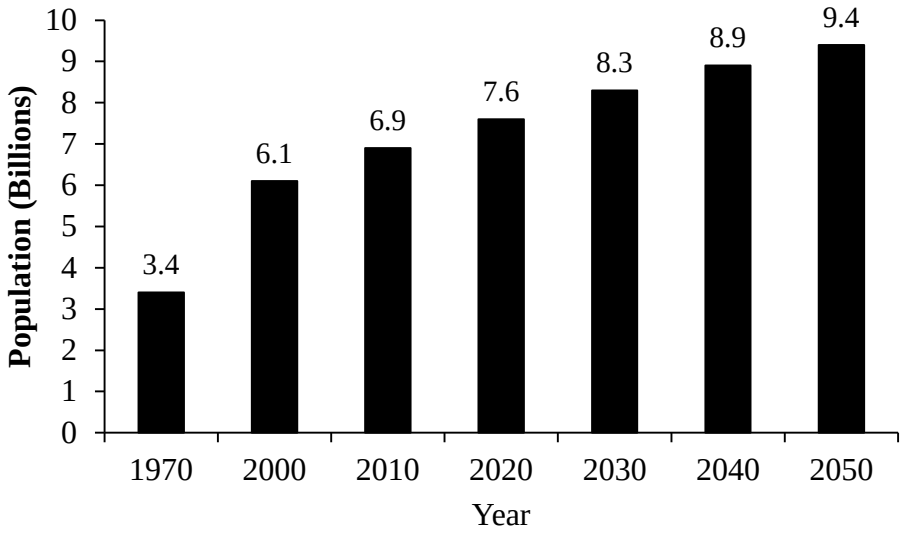

Figure 1.2. Prediction of the world population

(United States Census Bureau, 2014)

In Indonesia, based on the Indonesian statistics data, the number of Indonesian population in 2010 is about 237 million (Statistics Indonesia, 2010). Furthermore, According to the World Bank, the annual population growth rate of Indonesia from 2010 to 2013 is an average of 1.2 % (World Bank, 2014), and then is predicted that the Indonesian population will be reach about 252 million in 2015. This number will continue increase in the future. Fig. 1.3 presents the trend of Indonesian population from 2000 to 2015 increase around 47,238 people. According to projection by the United Nations (UN) with regard to the future absolute population, Indonesia is expected to have a population that exceed 270 million by 2025 and exceed 290 million by 2045 (Priyambada et al.,2015). Although the family planning program has been declared for more than three decades, however Indonesia continues to be unable to control population increase.

Up to the present time, the main staple food of Indonesia is rice. In the last decade in 2001, the production of rice was at amount of 31,132,083 t. This rice production of Indonesia was equal to 5.2 % of the global rice production at that time. Regarding to Indonesia population growth during period until 2022 it will require more 3,905,000 t of milled rice as staple food (Ahmad, 2013). Nowadays, Indonesia government faces serious problem due to overpopulation.

As we known, when the population increases, the housing facility will increase too, so the agricultural land will converted to fulfill housing and other facility needs. On the other hand, in East and Southeast Asia the impact of climate change might decrease by up to 30 % (Lobell, 2008). Its means the challenge is to provide the world's growing population with a sustainable, secure supply of safe, nutritious, and

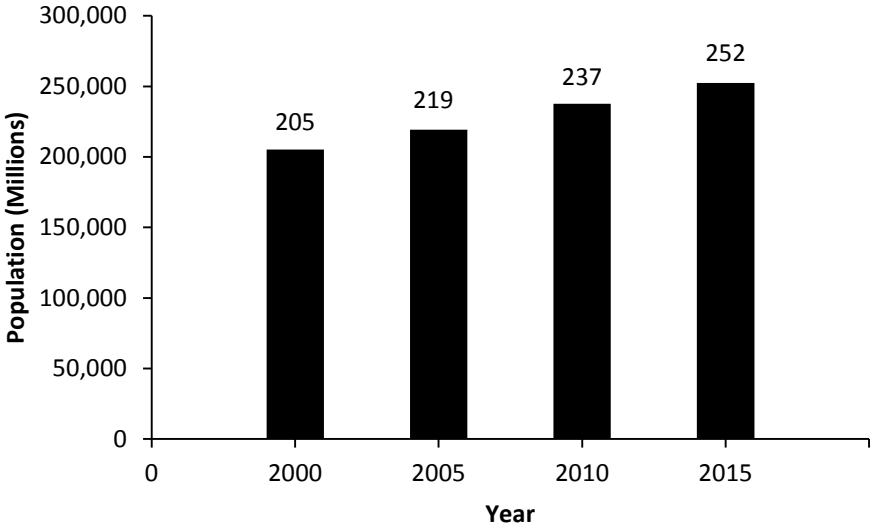

Figure 1.3. Trend of Indonesian population

(Priyambada et al., 2015)

affordable high-quality food using less land, with lower inputs, and in the context of global climate change, other environmental changes and declining resources. Therefore, all countries in the world including Indonesia should be anticipate food insecurity by seeking and utilizing a new source of food which resistant to environmental changes and diseases as well as has high production.

1.2. Potency of sago

The palm (*palmae*) are a family of some 2700 species of monocotyledonous plant. At least, there are 14 species of palms belonging to 8 genera are exploited for sago production, but of these only *Metroxylon* and *Arenga pinnata* are of major importance as palm starch sources. Comparing to *Arenga pinnata*, sago palm (*Metroxylon sagu* Rottb.) could be used far more extensively particularly as a source of starch and the true sago palm has been described as humankind's oldest food plant (Cecil, 1992 and Flach, 1983). The sago palm has important role in socioeconomic crop in Southeast Asia. The sago palm is one of the potential underutilized food palm and grows well in the tropical rain forest. The sago palm can well adapted to the tropical areas with an average temperature of 25 °C and approximate humidity of 70 %, in an extremely hardy plant, thriving in swampy, acidic peat soils and saline soils where other crops cannot survive (Flach, 1997; Flach and Schuilling, 1989). Sago palm are environmentally friendly and promote a socially stable agroforestry (Karim et al. 2008; Singhal et al., 2008).

Fig. 1.4 shows the densest distribution of sago appears to be in the South Pacific Island and then extending through Melanesia into South East Asia such as Indonesia,

Figure 1.4. Distribution of Metroxylon spp. in the Asia – Pacific region

(Greenhill, 2006 adapted from www.gcir.org)

Malaysia and Thailand (Flach, 1997). In the world, the total sago areal is around 2.5 million ha and which Indonesia has the largest natural sago area. The three leading world producers (Fig. 1.5) are Indonesia, Papua New Guinea, and Malaysia are 1,284,000 ha (56.5 %), 1,020,000 ha (41.2 %) and 45,000 ha (1.8 %), respectively (Flach, 1997). Even though Indonesia has the biggest sago area but the utilization of sago starch is only around 1 % of their potency (Samad, 2002). While Malaysia has the smallest sago area but as the biggest exporter of sago starch in the world, annually about 25,000–40,000 t of sago products around (Abd-Aziz, 2002; Bujang, 2011; Singhal et al. 2008).

In Indonesia, distribution of sago palm tree spread in all islands, and the largest sago area is found in Papua and West Papua Provinces around 90 % of 1.2 million ha (Flach, 1997) and also has high genetic variation of sago palm tree (Maturbongs and Luhulima, 2007). Totally 61 accessions were reported in Papua and the West Papua State, Indonesia (Yamamoto, 2011 adapted from Wijono, 2000).

Sago starch accumulated in the trunk of the sago palm until the flowering stage with maximum starch content occurring just before the onset of the palm flowers and dies shortly thereafter. The plant reaches commercial maturity at 9–12 years of age. At the mature stage, it possesses a huge trunk and may reach a height of 6–25 m and a diameter of 40 cm (Flach, 1997). There is a little information on optimal ecological condition (Flach, 1983).

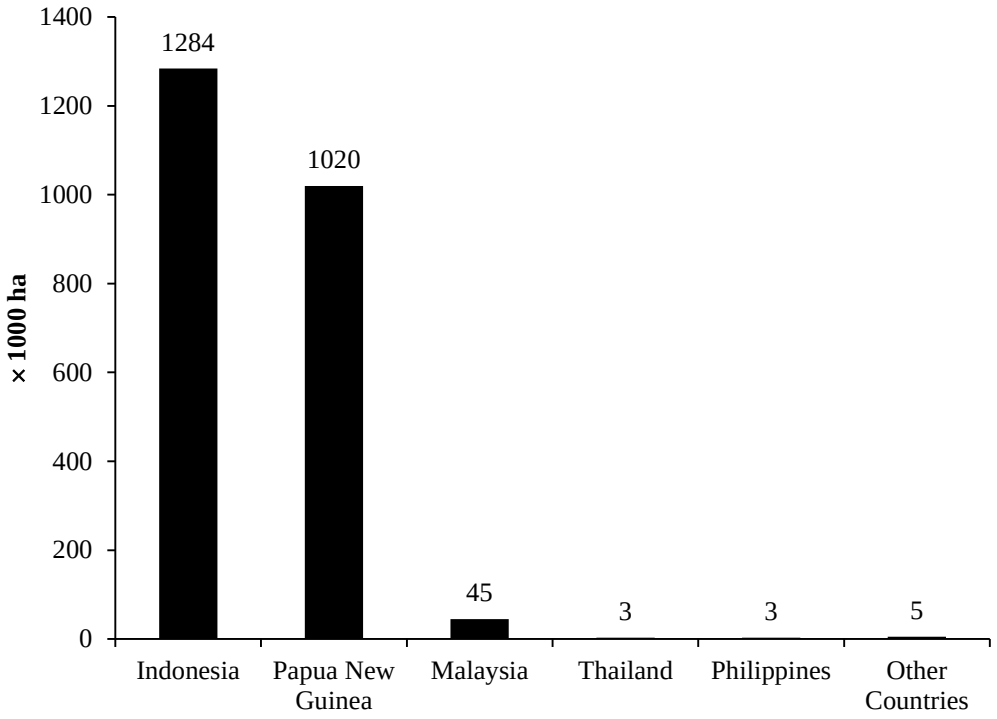

Figure 1.5. Estimation of sago palm in the world

(Flach, 1997)

Starch yield per sago palm tree is varied depending on the location and the intensity of cultivation. The amount of the extractable starch various depending on the efficiency and sophistication of the methods employed (Mohd et al., 2001). According to Cecil (1992) that sago starch content in the sago trunk is around 220 kg (29 %) of 750 kg total weight of sago palm trunk, but the extractable starch is only 150 kg (68 %) and the remaining starch around 32 % is still trapped within the parenchyma cells or the sago fibres. In Sarawak, the starch yield to be between 216 to 219 kg/palm (Pei Lang et al., 2006). While in Indonesia, the sago starch yield is average of 375 kg/palm (Yamamoto et al., 2007). Sago starch yield per hectare obtained in sago plantation (semi-cultivated) is higher than natural sago forest, due to in natural sago forest has high plant density. In sago plantation is grown in pattern ranging from 6 x 6 m square (280 clumps/ha) to 7.5 x 7.5 m triangular (205 clumps/ha) and the total sago starch yield by around 25 tons of air-dried starch/ha. However in natural sago forest is around 15 t of air-dried starch/ha (Flach, 1997; Ishizaki, 1998).

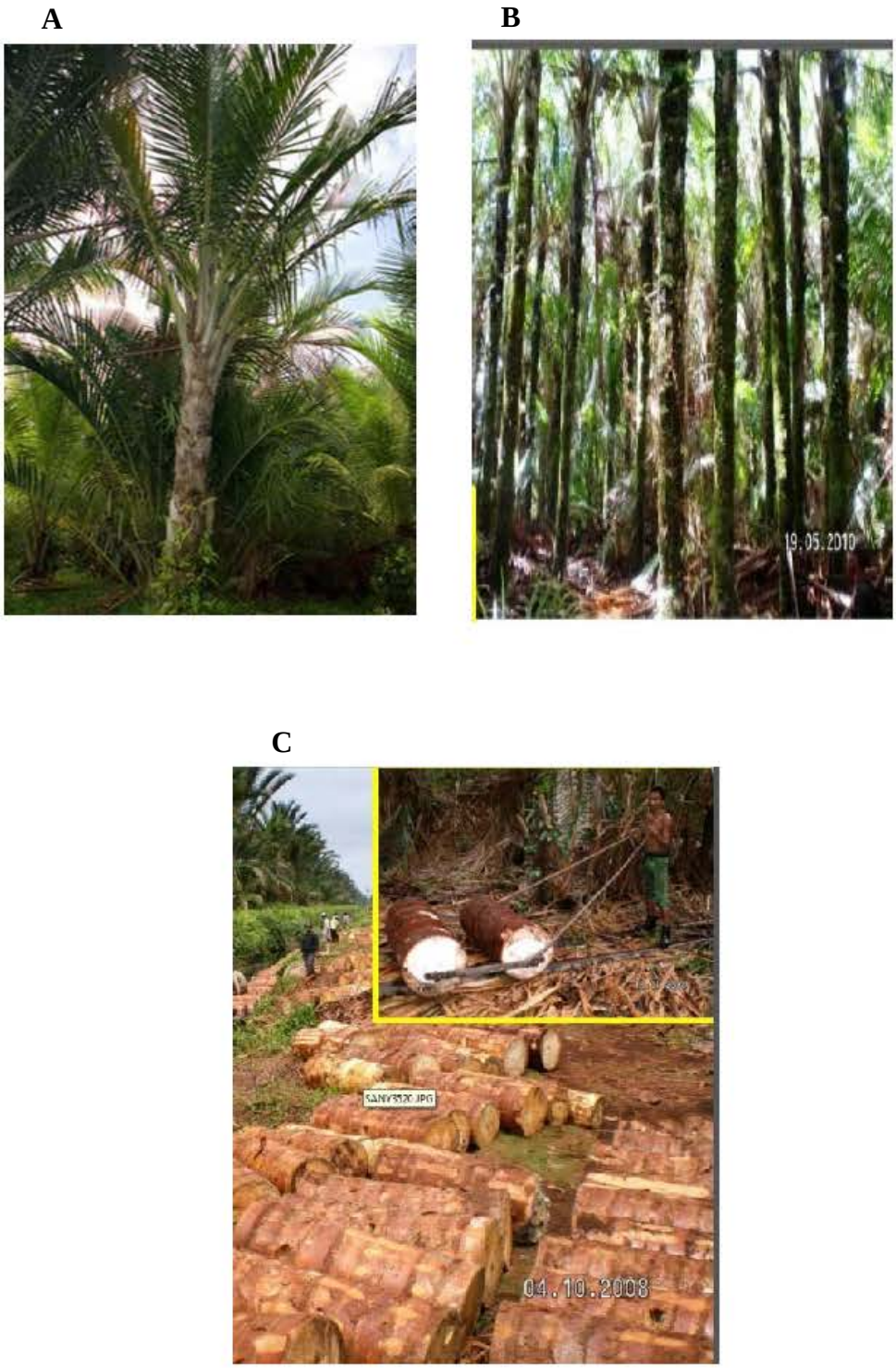

Figure 1.6. Sago palm tree (A, B) and sago palm log (C)

Photos were adapted from Jong, 2012

1.3. The role of sago starch for food security

Starch is the main storage carbohydrate and abundant storage of plants that is used for food. In the world, it is found in many different plant organs, including seeds, fruits, tubers and roots, where it is used as a source of energy during periods of dormancy and regrowth. Many of these starch-storing organs - for example, the grains of maize and rice or the tubers of cassava and potatoes - are staple foodstuffs in the human diet. Among all kinds of starches, wheat and maize starch is a valuable ingredient in the production of food, making up more than 80% of the world market for starch (Jobling, 2004). However, climate change might be because decreasing of agriculture and food supply. On the other hand, the number of the world population continues increase by the year and resulting in limited availability of food and rising food prices. According to United States Global Change Research Program (2009) that warmer temperatures may make many crops grow more quickly, but warmer temperatures could also reduce yields. Crops tend to grow faster in warmer conditions. However, for some crops (such as grains), faster growth reduces the amount of time that seeds have to grow and mature. This can reduce yields (i.e., the amount of crop produced from a given amount of land). More extreme temperature and precipitation can prevent crops from growing. Extreme events, especially floods and droughts, can harm crops and reduce yields (United State Environmental Protection Agency, 2013). Therefore, all countries in the world should diversify of food to anticipate food insecurity.

Recently, attention to sago starch as a new food and a food-security crop continue to increase, especially due to the anticipated increase in human population and potential environmental disasters in future (Yamamoto, 2011). There are

several reasons sago starch can be considered as source of food to anticipate food insecurity. Flach (1983) stated that sago is one of the excellent of carbohydrate source plant which potential to develop as food and estimated that there is close to 6 million hectares of good quality natural sago stand in Southeast Asia. Sago grows well in the extensive peat swamps of Malaysia, Papua New Guinea and Indonesia. Fig. 1.7 shows the natural sago forest in West Papua Province, Indonesia. Sago forest serve as a sink of carbon dioxide similar to other tropical rain forests, thus contributing to slowing down the pace of global warming (Quat Ng, 2007).

There are no records of serious diseases and few predators in sago palm and also naturally sago palm tree is tolerant with extreme condition which a few other plant can be grow (Flach , 1997). This indicates that sago palm is no need intensive cultivated using any fertilizer or pesticide. Fig. 1.8 shows that compared to other starches, sago starch has high yields per land area ranging from 15 to 25 t of air-dried starch/ha. The yield of sago starch could be about 3 to 4 times higher than those of rice, corn or wheat, and about 17 times higher than that of cassava (Singhal et al, 2008). Sago palm tree grows in clumps (Fig. 1.9). Each clump is composed of 1-8 stems sago, at each base of the stems grow 5-7 suckers. the sago growing groups to form clumps ranging from seedlings to mature trees (Harsanto, 1986; Flach, 1983). Hence sago palm tree can be harvested throughout the year.

Figure 1.7. Natural sago palm tree in Papua, Indonesia

Photos were adapted from Jong, 2012

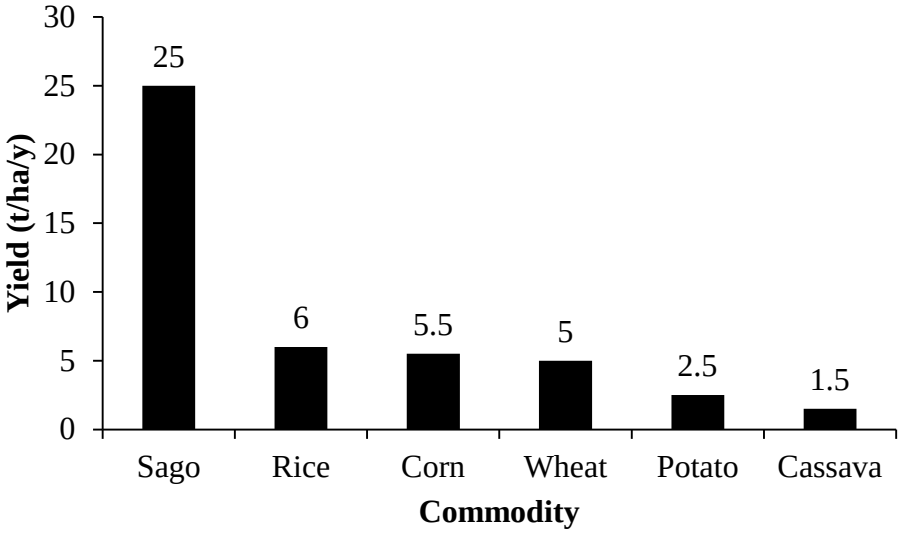

Figure 1.8. Sago starch yield compared to other commodities

(Ishizaki, 1998)

Figure 1.9. Sago palm tree grows in clumps

Photo was adapted from Jong, 2012

In east part of Indonesia, there are several staple foods made from sago starch. However, there are at least four main staple foods. *First*, one of the staple foods produced from sago starch is called “Papeda” (Fig. 1.10). This food is made by mixing sago starch with hot water and stirring constantly until a gel is formed. The gel has a glue-like consistency and texture, and it is eaten with fish and vegetable dishes. *Second*, it is also common to bake sago starch into sago lempeng (sagu lempeng) (Fig. 1.11A and B), in forms made of baked in a mold. The mold made by clay and called “forna”. The sago lempeng are usually made entirely of starch, but they may occasionally contain other foods as well, such as ground peanuts or other pulses. They are dipped in tea, coffee or milk before consumption (Flach, 1997). *Third*, we called sago sep (sagu sep) (Fig. 1.12). Sago starch is sprinkled with water to dampish. After that the sago starch dough is placed on a banana leaf and mixed with various ingredients such as meat, coconut, coconut milk and etc., then wrapped and baked on a stone or clay heat until cooked. There are five kind of sago sep based on their ingredients: Kumobo (sago is mixed with coconut and meat), Wanggilamo (sago is mixed with roasted meat), Nggalamo (sago is mixed with cubes of coconut and meat), Kaka (sago is mixed with coconut, meat and coconut milk), Siu (sago is mixed with bananas). *Fourth*, another staple food is called *sagu sinole* (Fig.1.13). This food is made by mixing hot grated coconut, dried and sieved sago starch, sugar and salt in a pan. All ingredients are stirred evenly for a few minute.

In recent year, however, Application of sago starch for food industries is an undeniable. Sago starch has been used in some small-scale industries, such as noodles, biscuits, chips, bread, vermicelli, and sago sohun. And also due to its viscous property upon gelatinization, starch has the potential to be used as thickener

in the production of soup and baby food, as well as an additive in various food products (Karim et al. 2008). Sago starch is also widely used together with rice, corn, and potatoes, in manufacture of noodles. Sago starch behaves in much the same way as sweet potato starch paste and that sago starch could be used for making vermicelli and other noodles in which sweet potato starch is currently used. Furthermore, sago starch is suitable for making noodles in Japan because the gels are of similar firmness and has lower adhesiveness and higher cohesiveness compared with other starches (Lee et al., 2002; Hamanishi et al., 2002).

Figure 1.10. Papeda, sago porridge as staple food in east part of Indonesia

Photo was adapted from Rana, 2012

Figure 1.11A. Sago lempeng

Photos were adapted from wikimedia.org:

http://commons.wikimedia.org/wiki/File:Sago_starch_product_sagu_lempeng_from_Maluku,ID_feb2002.jpg

Figure 1.11B. Sago lempeng mold

Photos were adapted from wikimedia.org:

http://catatansipemimpi.blogspot.jp/2012/06/hmmm-lezatnya-bubur-sagu-dan-tuna.html

Figure 1.12. Sagu sep (sep sago)

Photos were adapted from wordpress.com:

http://infopublik.id/album/113/foto-dalam-negeri-bulan-oktober-2012/

Figure 1.13. Sagu sinole

Photo was adapted from wordpress.com:

http://resepmasakanindonesia.info/resep-sinole/

1.4. The role of sago starch for biofuel production

Total consumption of energy in the world presented in Fig. 1.14. Based on both of pictures shows that the total consumption of fuel in the world is dominated by fossil fuel and only a small portion is renewable fuel (International Energy Agency, 2014). However, globally, fossil-fuel dependence resulted in different environmental problems, which include global warming, air quality deterioration, oil spills and acid rain among others. The combustion of fuel emits carbon dioxide (CO_2), one of the most significant greenhouse gases that trap heat in the earth's atmosphere. The heightened awareness of global warming as well as other environmental issues has increased interest in the development of methods to mitigate greenhouse gas (GHG) emissions and lessen the production of pollutants.

In 2011, the consumption of energy in Indonesia (Fig. 1.15) is still dominated by fossil fuel around 57 %, and then followed by natural gas (22 %) and coal (16 %), while for other energy sources such as hydrothermal heat and hydropower are less than 5 % (Karno et al., 2012). Therefore, government of Indonesia is committed to reducing the use of fossil fuel and develops a renewable energy.

Renewable energy is energy that comes from resources which are continually replenished, such as biofuel. Biofuels include a wide range of fuels which are derived from biomass. One of the liquid biofuel is bioethanol. Bioethanol is an alcohol made by fermenting the sugar components of plant materials and it is made mostly from sugar and starch crops, as well can be used as a gasoline additive to increase octane and improve vehicle emissions.

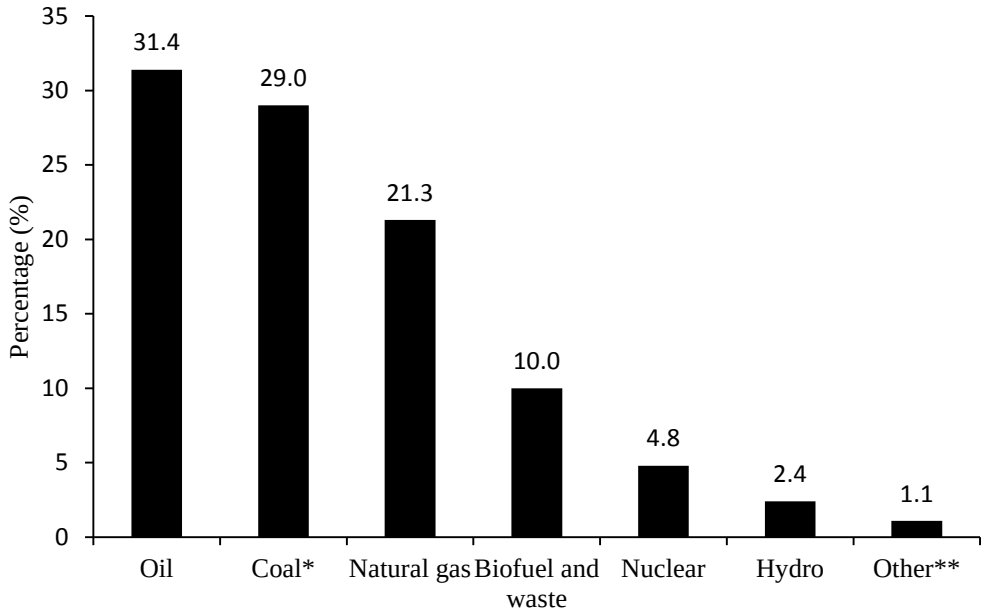

Figure 1.14. Total primary energy supply in the world, 2012

(International Energy Agency, 2014)

* Peat and oil shale are aggregated with coal

** Includes geothermal, solar, wind, heat, etc.

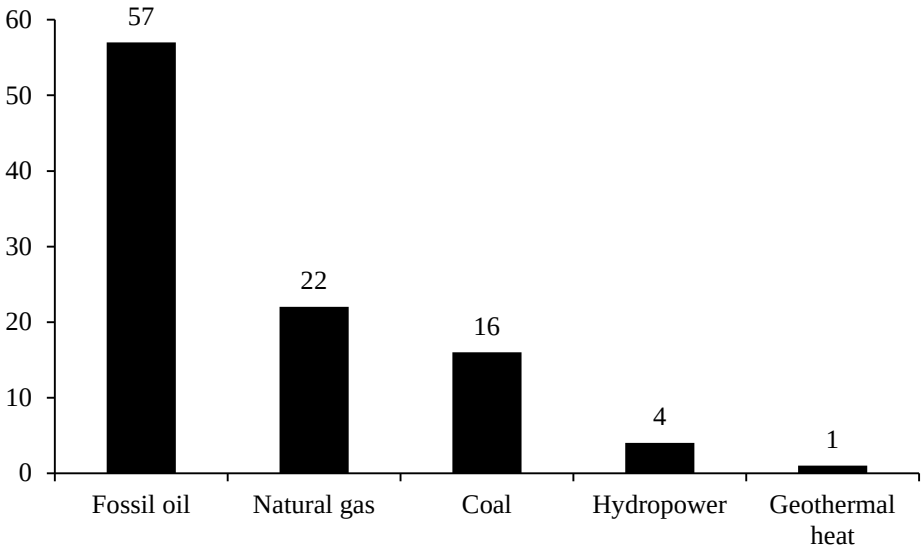

Figure 1.15. Energy source in Indonesia, 2011

(Karno et al., 2012**)**

In early 2006, the government of Indonesia enacted Presidential Regulation No. 5/2006 which formally established Indonesia's National Energy Policy (Nahatands, 2006). In that regulation formalized the development of biofuels in Indonesia, to include ethanol and biodiesel (Table 1.1), and established a five percent biofuel mandate by 2025. According to the Regulation biofuel development, as well as other new and renewable energies, will help to diversify and secure energy supplies and support sustainable economic development. This regulation established more detailed biofuel mandates through a progressive set of targeted biofuel mandates during the 2008-2025 time frames.

Sago starch is not only potential for foodstuff, but also can be used as a bio-resource, such as ethanol (Ishizaki, 1998). There are two technologies available for ethanol production: traditional yeast (*Saccharomyces cerevisiae* or others) fermentation and a newly developed bacterial ethanol fermentation technology using *Zymomonas mobilis* (Doelle et al., 1993). Recently, there has been active research aimed at increasing the ethanol yield by immobilized biocatalyst techniques (Singhal et al., 2008). In a study on simultaneous saccharification and fermentation (SSF) of ethanol from sago starch with coimmobilized *amyloglucosidase* (AMG) (immobilized on powdered chitin) and *Zymomonas mobilis* MTCC 92 by submerged fermentation, a maximum ethanol concentration of 55.3 g/L was obtained using a starch concentration of 150 g/L (Bandaru et al., 2006). Continuous ethanol production from sago starch was also carried out using immobilized *amyloglucosidase* (AMG) and *Z. mobilis* cells in a packed-bed reactor (Lee et al., 1987).

Table 1.1. Targeted biofuel mandates in Indonesia during the 2008-2025

Bioethanol (minimum)						
Sector	**2008**	**2009**	**2010**	**2015**	**2020**	**2025**
Transportation (Public service obligation, PSO)	3 %	1 %	3 %	5 %	10 %	15 %
Transportation (Non PSO)	5 %	5 %	7 %	10 %	12 %	15 %
Industry		5 %	7 %	10 %	12 %	15 %
Biodiesel (minimum)						
Transportation (Public service obligation, PSO)	1 %	1 %	2.5 %	5 %	10 %	20 %
Transportation (Non PSO)		1 %	3 %	7 %	10 %	20 %
Industry	2.5 %	2.5 %	5 %	10 %	15 %	20 %
Electricity	0.1 %	0.25 %	1 %	10 %	15 %	20 %

(Slette and Wiyono, 2013)

1.5. Sago starch extraction

Extraction process of sago starch can be divided into two methods, namely the traditional method and the modern one. The principles of sago extraction for both methods are similar, the differences is only in the scale of operation (Karim *et al*. 2008) and the tools that are used for processing (Greenhill, 2006). Traditional method divided into two levels, that are the domestic level and the small-scale processing plant level. Generally, domestic level using simple tools, such as axe, adze, and woven bags, as well sago palms are felled and processed in the garden (Fig. 1.16). Domestic level of sago starch processing was a time and labor intensive process, especially for pith disintegration and starch separation (Singhal et al., 2008). The total time required to process one trunk is 41 hours or around 6 days of work (Darma and Istalaksana, 2011). While the small-scale level of sago starch extraction using engine-powered of some processors (Fig. 1.17). Sago trunks are cut into sago logs (1 – 1.2 m) and then debarking is done. In some operations, a rotating mesh washer made of metal or screen washer is used to separate the starch. The starch slurry is channeled to a small settling pond made of board. Drying of wet starch is done mostly in the sunlight. For the modern method of extraction involves some modification to that of the small-scale level processing plant. New technologies for extracting starch are being adopted by the large-scale factories and these factories are now fully mechanized (Karim et al. 2008).

Extracting starch in sago trunk according to Nishimura et al. (2010) involves tree felling, cutting the trunk into logs, log splitting, pith crushing, starch filtering, starch extraction, drying and packaging. However, the method of extraction in different location is quite diverse depending on starch use, local resource use and economic

factors among different ethnic groups. Furthermore, Nishimura et al. (2010) stated that in Malaysia, the pith is ground or shaved by grating or rasping. Spinning drum-shaped graters are used for pith shaving. Motor-powered versions of these graters have appeared in recent years to make the task faster and easier. While in New Guinea, eastern Indonesia and the Philippines, the crushing tool is a handmade chipping axe (hammer) made of wood or bamboo. After pith crushing, crushed pith is extracted to obtain the sago starch. In Indonesia and Malaysia, the starchy pith fibers are placed into a vessel that is lined with a strainer or a net which is large enough for a worker to step into. The worker stomps the pith fibers while pouring water on them to extract starch. Alternatively, the bottom of a shallow square box is covered with a net on which raw material is placed and kneaded with feet while water is poured over it. Natural materials such as young coconut bark fibers (similar to palm stem fibers) were traditionally used as the filter, but nylon netting is widely used these days. The settled layer of starch is taken out of the container and packaged in the form of drained and semi-dried starch. Starch is packed in containers made from locally available materials such as the woven epidermis of the sago palm trunk or banana leaves which are tied with string. Starch may be processed further by drying again and grinding into flour. Recently, the Malaysian state of Sarawak is one of the areas that is actively pursuing intensive sago starch production, and the production reached of 300–500 tons per month. In Indonesia, starch refineries are found in Irian Jaya (Papua), Halmahera and Sumatra.

Generally, the sago starch which produced by traditional method has low both in quantity and quality (Flach, 1983 and Karim et al., 2008). In the trunk of a mature sago palm tree contains about 250 kg of starch (dry basis), 425 kg of water, and 175 kg of other materials (Flach, 1983). However, many studies have found that the

sago starch can be extracted only up to 55 – 75 % (dry basis) of the total starch in the sago trunk. The remaining starch is still trapped within the parenchyma cells or the sago fibres in sago pith waste (Awg-Adeni, 2013; Mohd et al., 2001,).

Figure 1.16. Domestic level of sago starch extraction

A: debarking, B: rasping, C: kneading, D: settling

Photos were adapted from wordpress.com:

https://www.google.co.id/search?q=tokok+sagu

A

B

C

Figure 1.17. Small-scale level of sago starch extraction

A: debarking & splitting, B: rasping, C: extraction and settling

Photos were adapted from Darma and Istalaksana, 2011

1.6. The general purpose of the study

As it has mentioned previously that in sago starch processing is a lot of starch cannot be extracted from sago pith, where around 25 – 45 % (dry basis) still trapped in sago pith waste. This indicates that not all potential sago starch in the sago trunk can be obtained. Related to the matter, the purpose of this study was to minimize the loss of starch through recovery of sago starch from sago pith waste to increase sago starch production.

1.7. References

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CHAPTER 2. RECOVERY OF SAGO STARCH FROM SAGO PITH WASTE BY A MICRO POWDER MILL

2.1. Background

Sago (*Metroxylon sagu* Rottb.) starch can be obtained by wet extraction of sago pith, because sago starch is present in trunk pith. The amount of starch obtained depends to a great extent on the fineness of the grated pulp, the efficiency of washing the starch out of the grated pulp (Cecil, 1992) and the efficiency and sophistication of the methods used (Mohd et al., 2001). The mechanical process currently used to extract sago starch is inefficient and often fails to dislodge residual starch embedded in the fibrous portion of the trunk (Karim et al., 2008).

Previous studies have found that the amount of sago starch trapped in sago pith waste (SPW) is around 25 – 45% on a dry weight basis (Awg-Adeni, 2013). However, Lai et al. (2013) reported that the amount of sago starch in SPW varies between 58 % and 67 % on a dry weight basis. Several papers have been published on the use of SPW for the production of food and non-food products, such as glucose (Asben et al., 2011; Linggang et al., 2012), fructose syrup (Mishima et al., 2011), bioethanol (Awg-Adeni, 2011; Peristiwati, 2011) and biobutanol (Awg-Adeni, 2013; Lingang et al., 2011). The extraction of residual starch from sago waste residues using an enzyme (Pectinex Ultra SP-L) was introduced by Mohd et al. (2001).

The yield of sago starch could be increased by extraction of SPW. A micro powder mill is a type of food processor that is commonly used for milling cereals and grains, but can be used to separate the sago starch physically from SPW. This

method is practical, because industrial operation of a micro powder mill is easy. Therefore, the objective of this study was to recovery of sago starch from sago pith waste by using a micro powder mill.

2.2. Materials and methods

The main material in this study was sago (*Metroxylon sagu* Rottb.) trunk (Fig. 2.1) which was obtained from a sago processor in Bogor, West Java, Indonesia. All chemicals used in the study were of analytical grade.

2.2.1. Sago starch extraction

The sago trunk was cut into pieces weighing around 200 g. The pieces were suspended in 800 mL of tap water and homogenized using a blender. The homogenate was sieved through two layers of muslin cloth and allowed to settle. The precipitate was washed three times. The starch and SPW were air dried and weighed until the water content was around 10 %, measured using an infrared moisture balance (FD-600-$_2$, Kett, Tokyo, Japan). The process of sago starch extraction showed in Fig. 2.2.

The true starch yield of sago pith was measured using the following method. After separating the sago starch and SPW from the sago pith, SPW (3 g) was soaked in deionized water (120 mL) in a 200 mL Erlenmeyer flask. The sample was autoclaved at 120 °C for 30 min. After cooling to 37 °C, 6 U α-amylase from porcine pancreas (Sigma-Aldrich, St Louis , MO, US) was added, and the sample was kept at 37 °C for 24 h. After hydrolysis, the sample was filtered using a glass

Figure 2.1. The sago pith after debarking of sago trunk

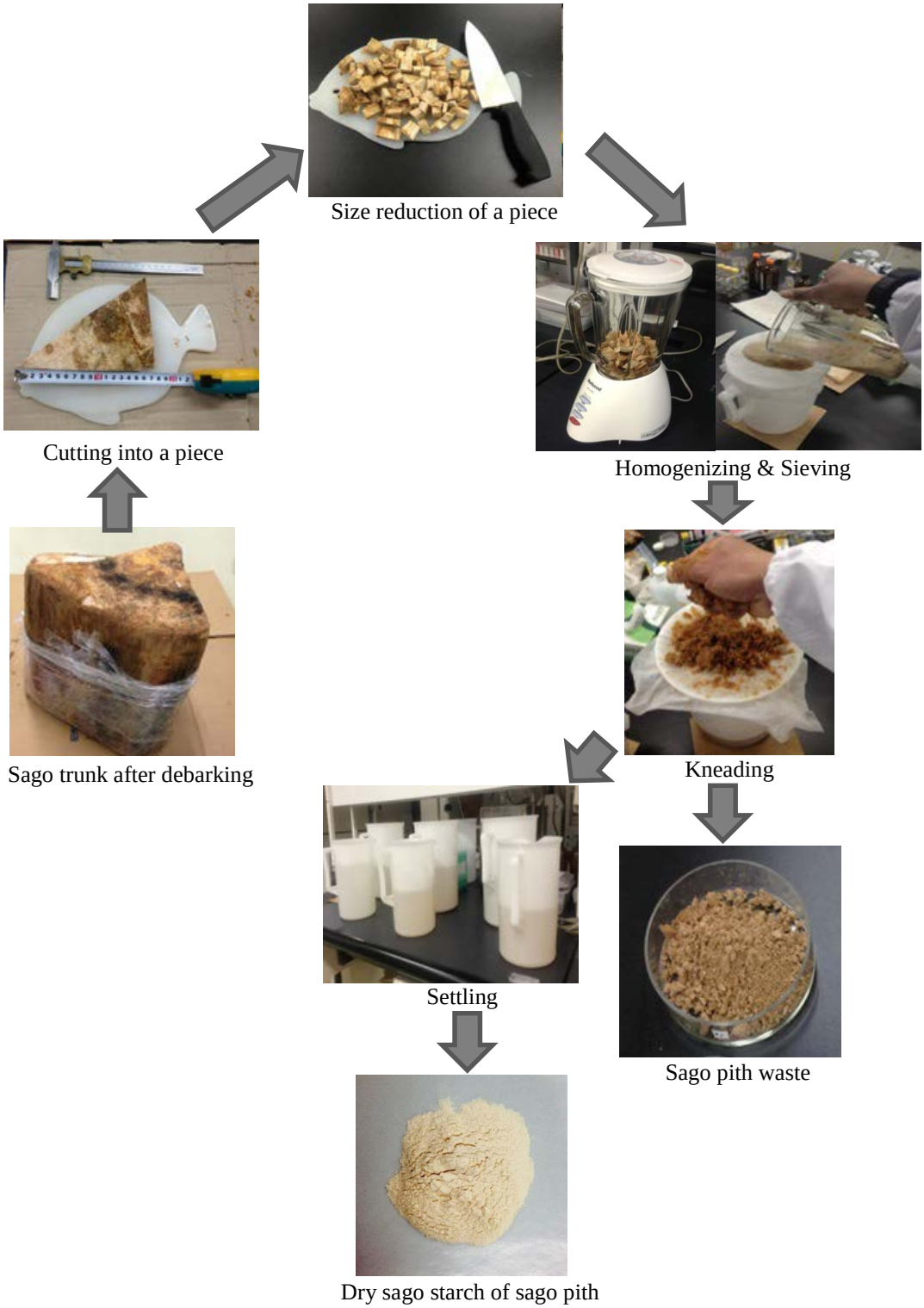

Figure 2.2. Flow diagram of process of sago starch production

filter and the solution was collected. The sugar content of the solution was determined using the phenol–sulfuric acid method. The true sago starch content was calculated according to Eq. 1.

$$\text{True starch of sago pith (\%)} = \frac{\text{Yield of starch from pith (g)} + \text{sugar content of solution (g)}}{\text{Sago pith (g)}} \times 100 \ldots (1)$$

The sago starch yield produced from sago pith, without the water content, was calculated according to Eq. 2. The amount of sago starch obtained from sago pith, called untreated sago starch (T0), was used as a comparison.

$$\text{Sago starch yield (\%)} = \frac{\text{Weight of sago starch (g)}}{\text{Weight of sago pith (g)}} \times 100 \ldots (2)$$

2.2.2. Micro-powder milling of sago pith waste

The main material for micro-powder milling was sago pith waste (SPW) (Fig.2.3). The SPW generated from sago starch production (section of 2-2-1). The dry SPW was milled using a micro powder mill (G-008; West Co., Ltd., Niigata, Japan) (Fig. 2.4). The milling process was conducted using either a water coolant (WC) or ice-water coolant (IC) to reduce the heat generated by the micro powder mill. Milling was performed at different levels of disc clearance, namely wide (T1), wide–medium (T2), medium–narrow (T3), and narrow (T4). The starch and fiber

were separated by decantation. The sago starch yield produced from SPW was calculated according to the following equation:

$$\text{Micro} - \text{powder} - \text{milled sago starch yield } (\%) = \frac{\text{Weight of sago starch (g)}}{\text{Weight of SPW (g)}} \times 100 \ldots (3)$$

After milling of the SPW, the starch of SPW was extracted using similar method of sago starch production (Fig. 2.5).

Figure 2.3. Sago pith waste

Photo was adapted from Darma & Istalaksana, 2011

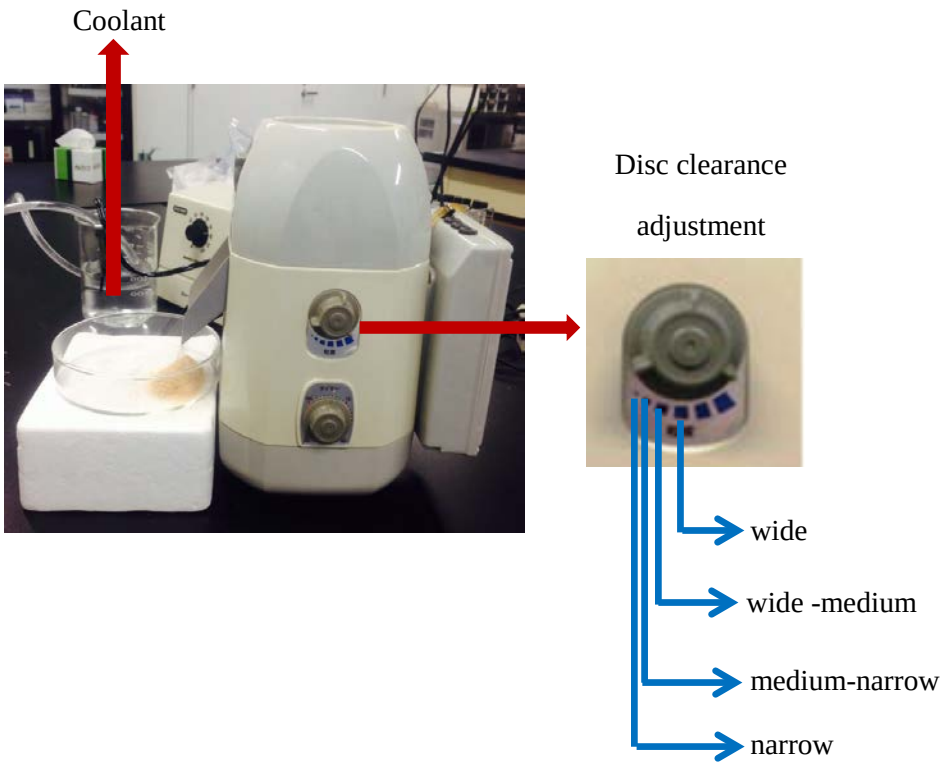

Figure 2.4. Sago pith waste milling process using micro-powder-mill (MPM)

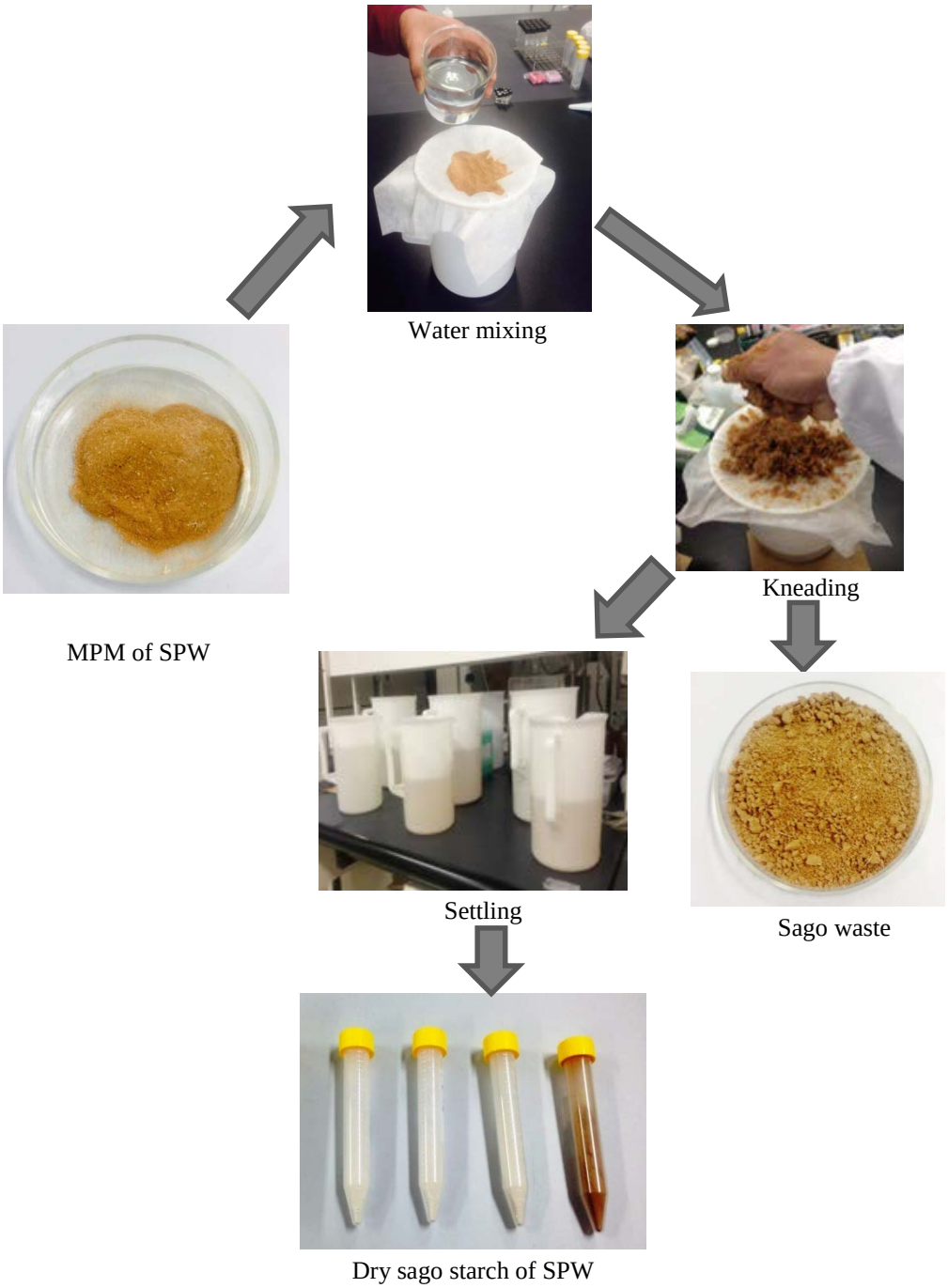

Figure 2.5. Flow diagram of process of sago starch recovery from sago pith waste

2.2.4. Scanning electron microscopy

Sago pith waste samples after micro-powder-milling were spread thinly and evenly on circular metal stubs using double-sided adhesive tape and then observed using a SEM (Miniscope TM-1000, Hitachi High-Technologies Corp., Tokyo, Japan), at an accelerating voltage of 15 kV.

2.2.5. Soluble starch analysis

The soluble starch content of the supernatant of the micro-powder-milled SPW was analyzed in triplicate. The micro-powder-milled SPW samples (about 100 mg) were diluted with 2 mL of distilled water and then centrifuged at 1,912×g for 10 min. An aliquot of the supernatant was collected and centrifuged at 4,302×g for 10 min. The final supernatant was collected and the soluble starch as the total sugar content was determined, using the phenol–sulfuric acid method (DuBois, 1956). The apparent total sugar content was measured from a calibration curve obtained using a similarly treated glucose solution.

2.3. Results and discussion

2.3.1. Sago starch yield. The true yield of sago starch from pith (Fig. 2.6), calculated using Eq. 1, was 65 %. The yields of sago starch obtained using commercial methods, calculated using Eq. 2, were around 32 %. The obtained results agree with those reported by Pei-Lang et al. (2006). The yields of sago starch obtained using WC and IC by micro powder milling at various disc clearances increased by around 10 %, 15 %, 17 %, and 18 % for T1, T2, T3, and T4, respectively.

The sago starch yield at T1 was lower, because this treatment produced large particles of SPW (Fig. 2.7A); therefore most of the starch was not released. According to Cecil (1992) and Mohd et al. (2001) that the sago starch in SPW is trapped within the parenchyma cells or fibers. To liberate the sago starch, it is necessary to break down the SPW. T4 treatment (Fig. 2.7D) produced smaller SPW particles than T3 treatment did (Fig. 2.7B and C); the sago starch yields were similar.

Separation process of sago starch for T1 to T3 treatments (Fig. 2.8) were easier compared with T4 treatment. Because of T4 treatment produces finer sago pith and also formed a gel when kneading process (Fig. 2.9), it becomes difficult to separate the starch and fibers. Cecil (1992) reported that further grating (secondary grating) gives little further increase in the yield and makes separation of the starch and fibers difficult. And also, low starch yield and purity in wet fractionation may be due to in adequate breakage of cell wall (fiber), which prevents the separation of starch granules from fibre (Naguleswaran and Vasanthan, 2010). The color of sago starch obtained using commercial method and micro-powder-milled sago starch from SPW showed in Fig. 2.10. The color of micro-powder-milled sago starch for all treatments was similar with untreated sago starch, except T4 of both water and ice water coolant treatments were brown. The brown color of starch in T4 treatment caused by the starch mixed with fiber. The fiber color was brown as shown in Fig. 2.11. T4 treatment produced finer fiber and passed through a sieve then settled together with the starch. The fiber was difficult to separate from starch after settling process. According to Naguleswaran and Vasanthan (2010), contamination of fiber component in starch sedimentation process associated to produce undesirable brownish. The soluble starch in the supernatant of micro-powder-milled SPW (Table 2.1) was very low, ranging from 0.13 % to 0.38 % and from 0.13 % to 0.35 % for

WC and IC, respectively. This shows that micro-powder-milling treatment hardly breaks the covalent bonds in the amylose and amylopectin chains.

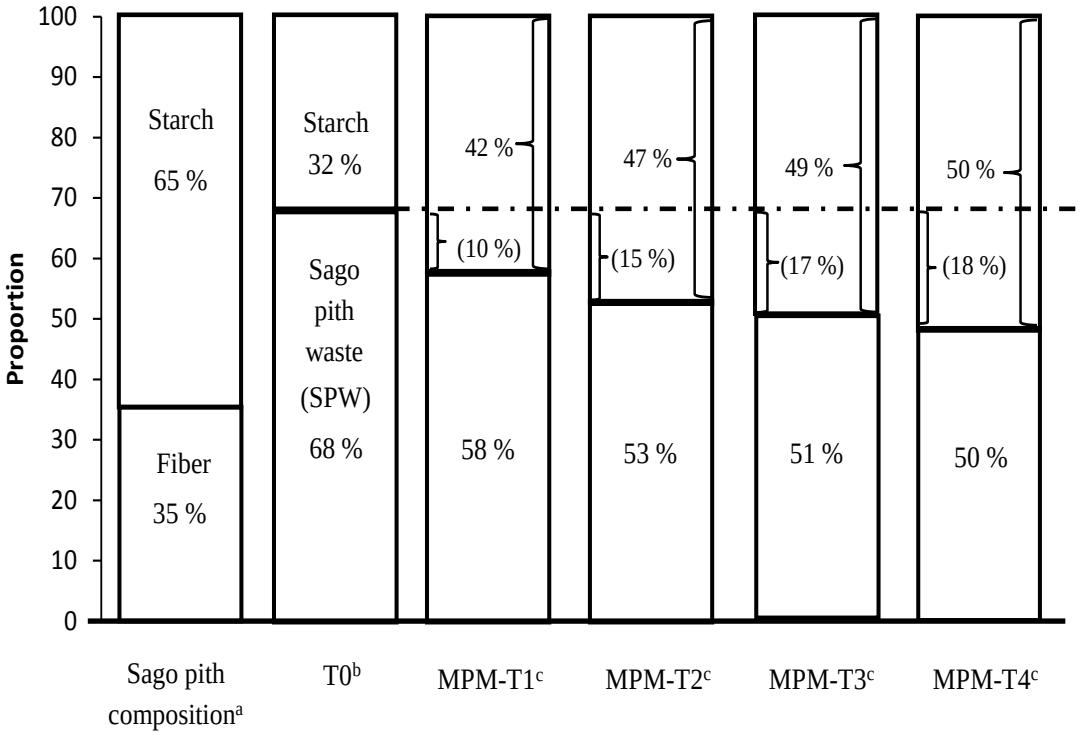

Figure 2.6. Sago starch yield obtained from sago pith (T0) and average of sago starch yields obtained from micro-powder-milled sago pith waste (SPW) at different disc clearance treatment with either water coolant (WC) or ice-water coolant (IC)

[a] True sago starch content in sago pith evaluated using Eq. 1

[b] Average sago starch yield obtained using a commercial method, evaluated using Eq.2

[c] Average sago starch yield, evaluated using Eq. 3, from SPW by micro powder milling using WC or IC.

MPM-T1: micro-powder-milled sample with wide clearance setting (T1);

MPM-T2: with wide-medium clearance setting (T2);

MPM-T3: with medium-narrow clearance setting (T3); and

MPM-T4: with narrow clearance setting (T4).

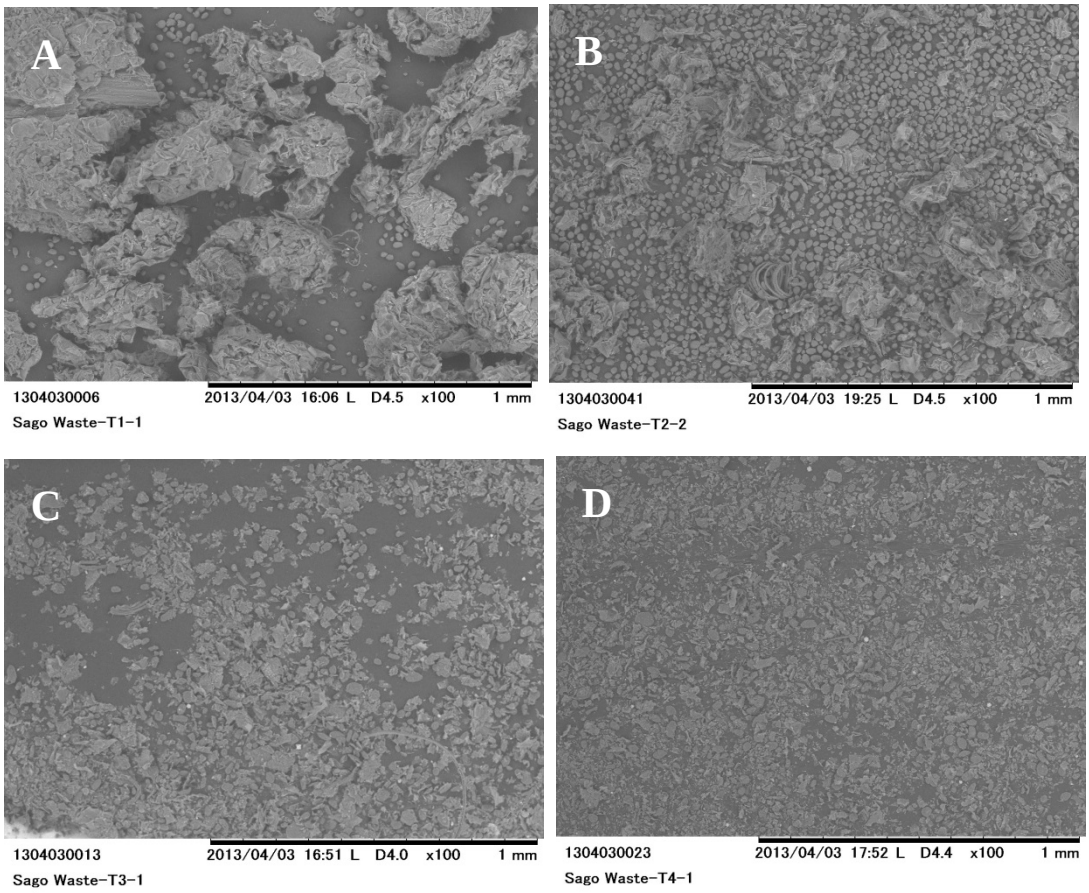

Figure 2.7. Scanning electron microscopy images of starch obtained from sago pith waste by micro powder milling with water coolant

Magnification is ×100.

A: Micro-powder-milled sample with wide clearance setting (T1); B: with wide–medium clearance setting (T2); C: with medium–narrow clearance setting (T3); and D: with narrow clearance setting (T4).

Figure 2.8. Kneading process of T1, T2 and T3 treatments

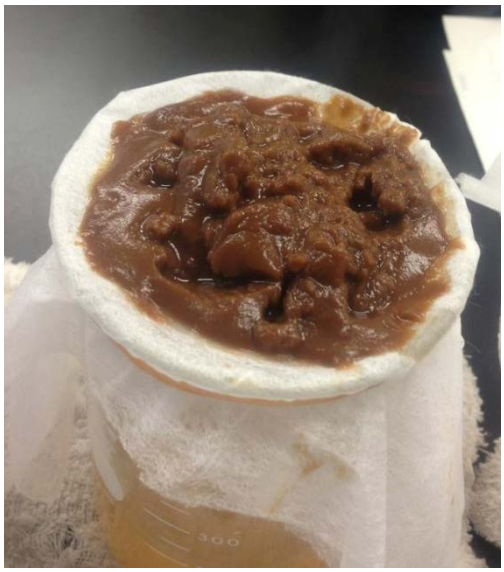

Figure 2.9. Kneading process of T4 treatment,

SPW formed a gel

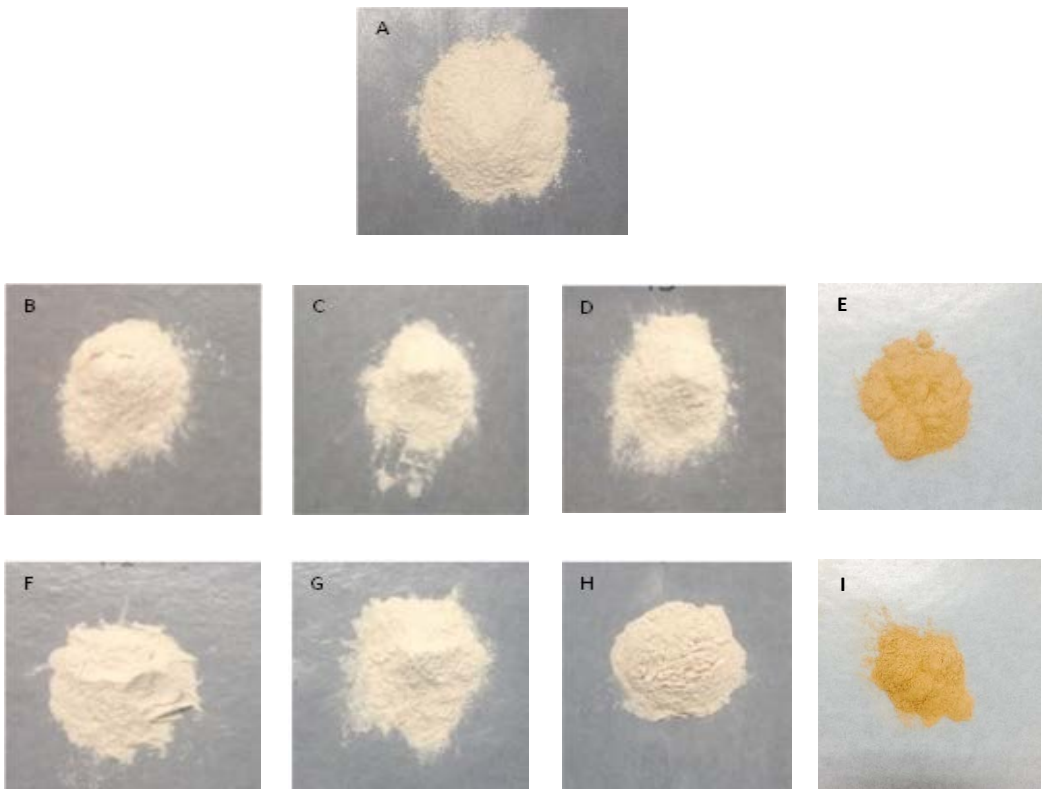

Figure 2.10. Micro-powder- milled of sago starch from sago pith waste

A: untreated sago starch sample; B: micro-powder-milled sample with water coolant (WC) and wide clearance setting (T1); C: with WC and wide–medium clearance setting (T2); D: with WC and medium–narrow clearance setting (T3); E: with WC and narrow clearance setting (T4); F: with ice-water coolant (IC) and wide clearance setting (T1); G: with IC and wide–medium clearance setting (T2); H: with IC and medium–narrow clearance setting (T3); and I: with IC and narrow clearance setting (T4).

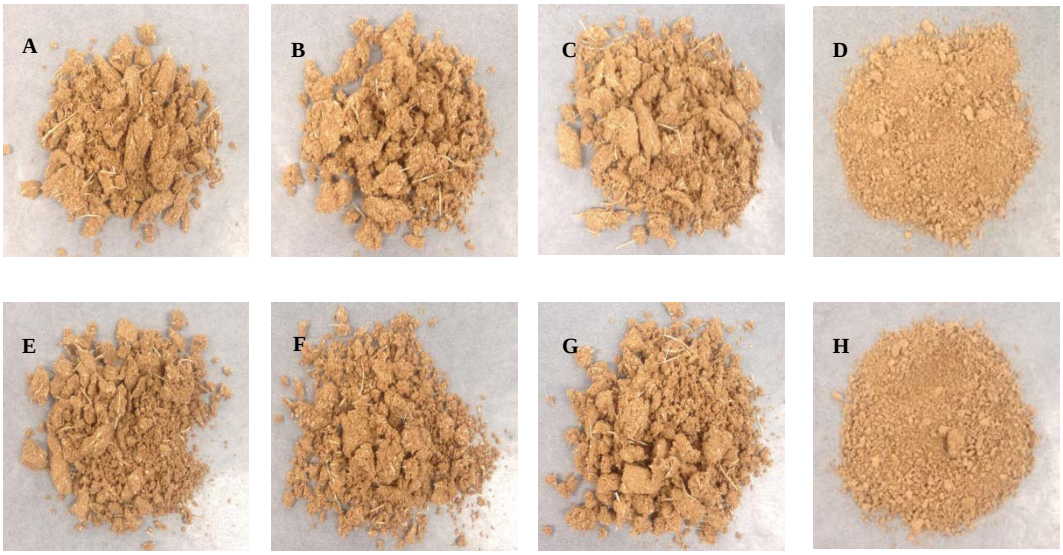

Figure 2.11. Fiber color of micro powder milling

A: micro-powder-milled sample with water coolant (WC) and wide clearance setting (T1); B: with WC and wide–medium clearance setting (T2); C: with WC and medium–narrow clearance setting (T3); D: with WC and narrow clearance setting (T4); E: with ice-water coolant (IC) and wide clearance setting (T1); F: with IC and wide–medium clearance setting (T2); G: with IC and medium–narrow clearance setting (T3); and H: with IC and narrow clearance setting (T4).

Table 2.1. Soluble starch contents of sago starch obtained using various micro-powder-milling treatments[a]

Treatment	Soluble starch (%)[b]
T0	-
WC-T1	0.13 ± 0.0
WC-T2	0.16 ± 0.0
WC-T3	0.16 ± 0.0
WC-T4	0.38 ± 0.0
IC-T1	0.13 ± 0.0
IC-T2	0.15 ± 0.0
IC-T3	0.16 ± 0.0
IC-T4	0.35 ± 0.0

[a]Parameters are given as average value ± standard deviation.

[b]The amountof soluble starch was calculated from the supernatant of the micro-powder-milled sago pith waste. WC: water coolant; IC: ice-water coolant; T0: untreated; T1: wide clearance; T2 and T3: medium clearances; T4: narrow clearance.

2.4. Summary

The sago starch from SPW which left behind after starch extraction can be extracted by micro powder milling. Industrial-scale micro powder milling is more practical and efficient than other milling processes because it has a continuous system and requires a short milling time. The highest yield of micro-powder-milled sago starch was T4 (18%) and then followed by T3, T2 and T1 were 17, 15 and 10 %, respectively.

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CHAPTER 3. PHYSICOCHEMICAL PROPERTIES OF MICRO-POWDER-MILLED SAGO STARCH

3.1. Background

The composition and structure of starch granules affecting the properties and functions of starches from different crops (Nadiha et al., 2010). The composition and structure can be changed by mechanical forces during the grinding process (Hatcher et al., 2002). Micro powder milling is a dry mechanical treatment. Dhital et al. (2010) reported that mechanical treatment alters the starch granular structure, physicochemical properties, and digestibility. The physical damage caused by milling results in starch granules with irregular, rough, and less uniform surfaces, and in reduced granular crystallinity (Barrera, 2013). Al-Rabadi et al. (2012) reported that for the same grain type and grinding under similar conditions, the particle size distribution may differ as a result of variations in the mechanical properties. The effects of mechanical treatments of starch have been investigated, e.g., the effects of cryogenic and hammer milling on rice grains (Devi et al. 2009; Hasjim et al. 2013) of ball milling on maize starch (Liu et al. 2011) of ultrasonication on wheat and rice starch (Sujka and Jamroz,2013) and of cryomilling on potato and maize starch (Dhital et al., 2010; Dhital et al., 2011). Most of these studies found that physical treatment led to changes in the crystallinity and internal granular architecture of the starch. However, there is a lack of information on the properties of sago starch produced from SPW by micro powder milling.

The objective of this study was to investigate the physicochemical properties of sago starch obtained by micro powder milling of SPW. The changes in micro-powder-milled sago starch were investigated using light microscopy, scanning electron microscopy (SEM), particle size analysis, X-ray diffraction (XRD), differential scanning calorimetry (DSC), and gel permeation chromatography (GPC).

3.2. Materials and methods

The main materials in this experiment were untreated sago starch (T0); micro-powder-milled sago starch sample with water coolant (WC) and wide clearance setting (T1); with WC and wide–medium clearance setting (T2); with WC and medium–narrow clearance setting (T3); with WC and narrow clearance setting (T4); with ice-water coolant (IC) and wide clearance setting (T1); with IC and wide–medium clearance setting (T2); with IC and medium–narrow clearance setting (T3); and with IC and narrow clearance setting (T4). All chemicals used in the study were of analytical grade.

3.2.1. Scanning electron and light microscopy

The morphologies of powder samples of T0, micro-powder-milled sago starch, and SPW samples were examined using SEM (Miniscope TM-1000, Hitachi High-Technologies Corp., Tokyo, Japan), at an accelerating voltage of 15 kV. The samples were spread thinly and evenly on circular metal stubs using double-sided adhesive tape. The birefringences of the untreated and micro-powder-milled starch were determined using polarized light microscopy (Olympus BX51, Olympus Optical Co., Ltd., Tokyo, Japan). A camera was used for image capture and micrographs were obtained at 40× magnification for each sample.

3.2.2. Particle size analysis

The particle size distribution of the T0 and micro-powder-milled sago starch were measured using laser diffraction (Microtrac MT3300EX2, Nikkiso Co., Ltd., Tokyo, Japan). The sample (ca. 5 mg) was dispersed in 25 mL of 96 % ethanol solution and then ultrasonicated at 20 kHz for 20 s. From each distribution, D(v,0.1), D(v,0.5), and D(v,0.9) were obtained, representing the particle diameters at cumulative volumes of 10 %, 50 %, and 90 %, respectively. The size dispersion was evaluated from the dispersion indexes, using the span equation 4 (Liu et al. 2011).

$$\text{Span} = \frac{\text{D(v, 0.9)} - \text{D(v, 0.1)}}{\text{D(v, 0.5)}} \ldots (4)$$

3.2.3. XRD analysis

XRD was performed using an Ultima IV X-ray diffractometer (Rigaku Corp., Tokyo, Japan). T0 and micro-powder-milled sago starch (ca. 50 mg) were placed in a square glass cell (2 cm × 2 cm) and exposed to an X-ray beam, operated at 40 kV and 40 mA, with Cu Kα radiation (Ni filter). The 2θ scanning range was 3–33°, which covers all the significant diffraction peaks of starch crystallites. The diffraction slit was operated at 0.5 - 10 mm and the scanning rate was 3°/min.

The method developed by Cheetham and Tao (1998) was used to calculate the percentage crystallinity of the sample from the X-ray measurements. The area above the smooth curve corresponded to the crystalline portion, and the lower area, between the smooth curve and a linear baseline, which connected the intensities at 2θ = 33° and 3°, corresponded to the amorphous section of the sample. The

percentage crystallinity was calculated from the ratio of the crystalline peak area to the total crystalline area (Frost et al., 2009; Htoon et al., 2009).

3.2.4. DSC analysis

Differential scanning calorimetry (DSC) was performed using a DSC 6100 SII calorimeter (Seiko Instruments Inc., Chiba, Japan). Samples of T0 and micro-powder-milled sago starch (ca. 12 mg) were weighed into silver pans of known weight; distilled water (45 µL) was added (starch/water, 1 : 3.75, w/w), and then the silver pans were sealed hermitically. An empty sealed silver pan was used as a reference. The sample was heated from 10 to 120 °C at a rate of 1 °C/min. The characteristic transition temperatures, i.e., onset temperature (T_o), peak temperature (T_p), and conclusion temperature (T_c), and the enthalpy of gelatinization (ΔH) were determined using the built-in software. The enthalpy of gelatinization was calculated from the peak area of the endothermic curve and was expressed in joules per gram of dry starch. The experiments were performed in duplicate.

3.3. Results and discussion

3.3.1. Starch granule shape

The starch granule is nature's chief way of storing energy over long periods in green plants. Starch granules are mainly found in seeds, roots and tubers, but are also found in stems, leaves, fruits and even pollen. Starch granules occur in all shapes and sizes (spheres, ellipsoids, polygons, platelets, irregular tubules); their long dimensions range from 0.1 to at least 200 µm, depending on the botanical source. Differences in external granule morphology are generally sufficient to

provide unambiguous characterization of the botanical source, via optical microscopy (Pérez et al., 2009), also all starch granules might contain pores and channels that are unobservable with SEM but that are large enough for water, reagents or enzymes to pass through (Fannon et al., 1992).

Sago starch granules are generally bigger than those of rice (3-10 µm), corn (5-20 µm), wheat (22-36 µm), or cassava (5-25 µm), but smaller than those of potato (15-80 µm) (Nadiha et al., 2010). The SEM micrographs of T0 and micro-powder-milled sago starch are shown in Fig. 3.1. The surfaces of the granules in T0 and those obtained using T1 treatment were smooth, without any pores and fissures. T0 granules are basically smooth and oval, with diameters in the range 20–40 µm (Ahmad, 1999) but the granule size can reach 20–60 µm (Cecil, 1992). T2 and T3 treatments gave slightly rough granules, and T4 treatment resulted in loss of smoothness, with both WC and IC. This might be because friction in the T4 treatment was higher than in the other treatments. Dhital et al. (2010) reported that the level of disruption of starch granules by mechanical treatment depends on the treatment type and severity, as well as the starch source.

Images of the sago starch observed under normal light microscopy (Fig. 3.2) clearly shows that the sago granules were mainly oval, but some were spherical and with pores. The surfaces of T0 granules were undamaged, except in the cases of the T2, T3, and T4 treatments. When the starch was observed under polarized light (Fig. 3.3), T0 and starch subjected to T1, T2, and T3 treatments showed birefringence, with strong patterns at the granule centers; these typical maltese crosses were uniform, with two crossed lines and a dark line at the center. However, the birefringence of the sago granules subjected to T4 treatment almost disappeared. According to Ambigaipalan et al. (2011) that the birefringence patterns indicate that

amylopectin crystallites are arranged radially within the granules at right angles to the surface, with their single reducing end group towards the hilum. Huang et al. (2007) found that ball-milled starch granules are less birefringent than native starch granules with reduction in crystallinity, number of double helices, and gelatinization enthalpy, suggesting that semi-crystalline granules are progressively converted into an amorphous state. Tester (1997) stated that waxy starch granules are damaged more easily than normal starch granules, probably because amylose in the amorphous regions of non-waxy starches acts as a shock absorber and provides a cushioning effect limiting amylopectin breakdown during milling.

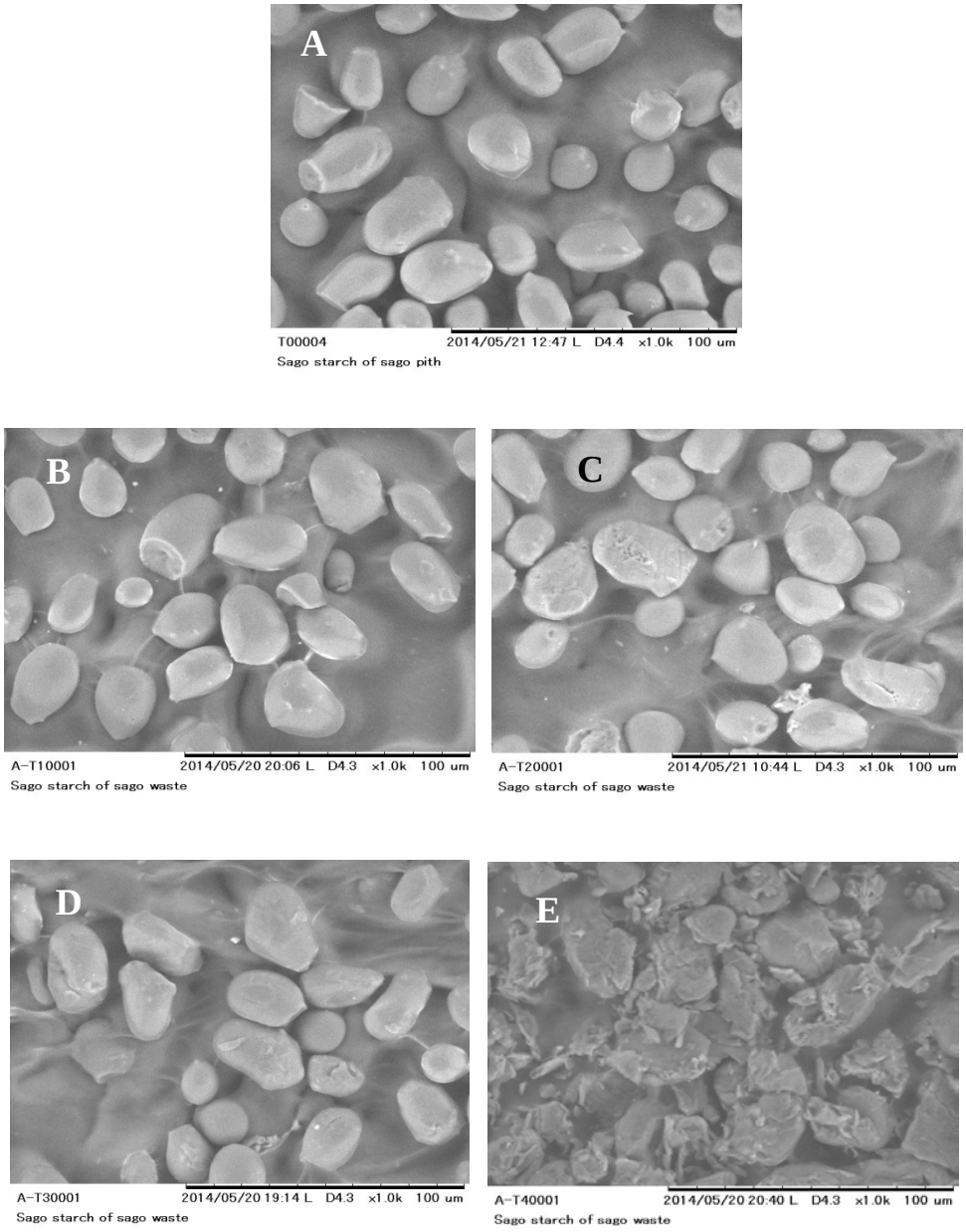

Figure 3.1.1. Scanning electron microscopy images of sago starch, untreated sago starch, and micro-powder-milled sago starch

Magnification is ×1000.

A: untreated sago starch sample; B: micro-powder-milled sample with water coolant (WC) and wide clearance setting (T1); C: with WC and wide–medium clearance setting (T2); D: with WC and medium–narrow clearance setting (T3); E: with WC and narrow clearance setting (T4).

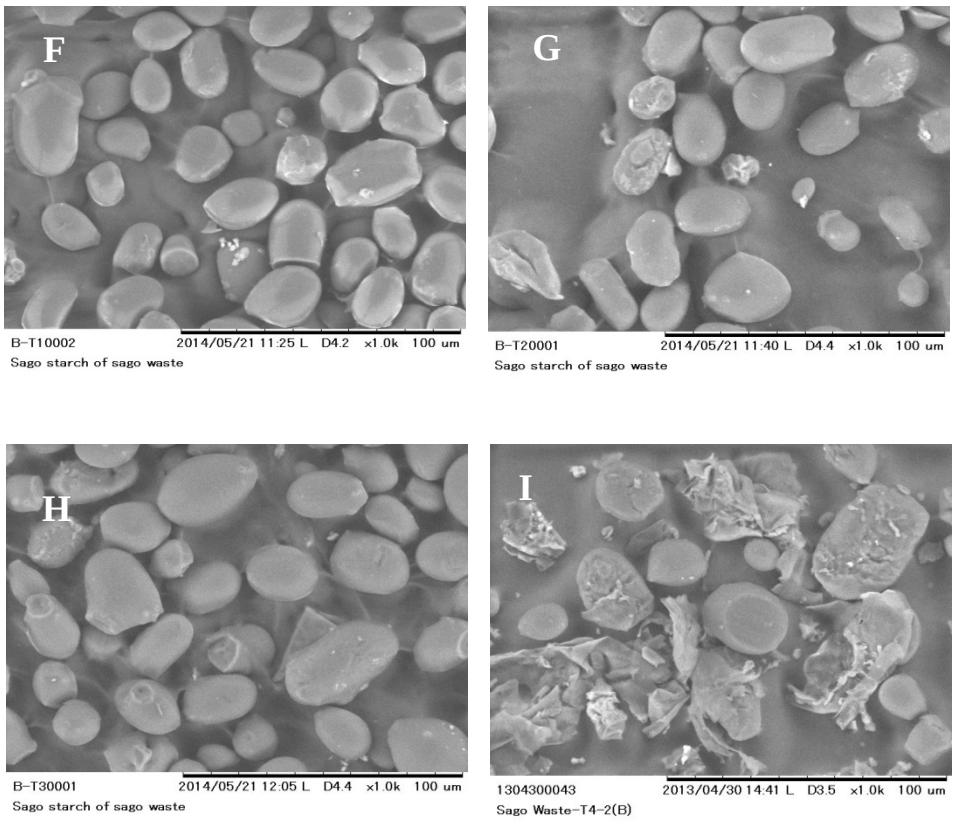

Figure 3.1.2. Scanning electron microscopy images of sago starch, untreated sago starch, and micro-powder-milled sago starch

Magnification is ×1000.

F: micro-powder-milled sample with ice-water coolant (IC) and wide clearance setting (T1); G: with IC and wide–medium clearance setting (T2); H: with IC and medium–narrow clearance setting (T3); and I: with IC and narrow clearance setting (T4).

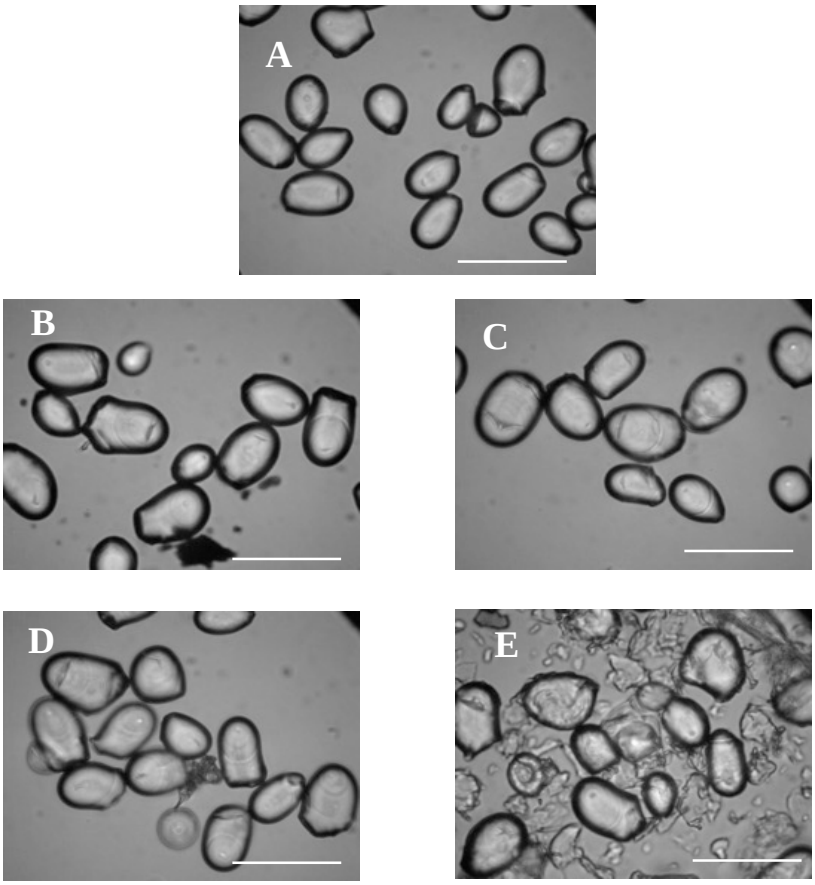

Figure 3.2.1. Granules shape of untreated sago starch and micro-powder-milled sago starch of WC under normal light microscopy

Magnification is ×40 and bar indicates 50 μm.

A: untreated sago starch sample; B: micro-powder-milled sample with water coolant (WC) and wide clearance setting (T1); C: with WC and wide–medium clearance setting (T2); D: with WC and medium–narrow clearance setting (T3); E: with WC and narrow clearance setting (T4).

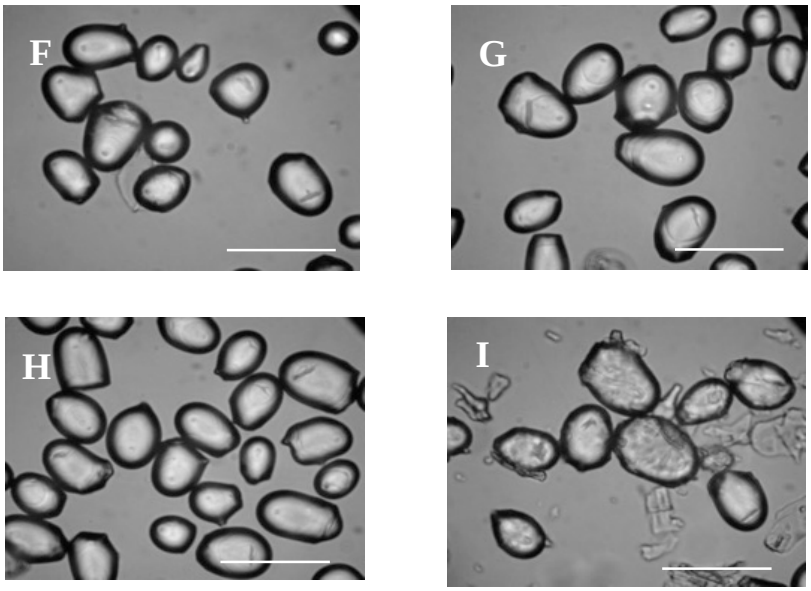

Figure 3.2.2. Granules shape of untreated sago starch and micro-powder-milled sago starch of IC under normal light microscopy

Magnification is ×40 and bar indicates 50 μm.

F: micro-powder-milled sample with ice-water coolant (IC) and wide clearance setting (T1); G: with IC and wide–medium clearance setting (T2); H: with IC and medium–narrow clearance setting (T3); and I: with IC and narrow clearance setting (T4).

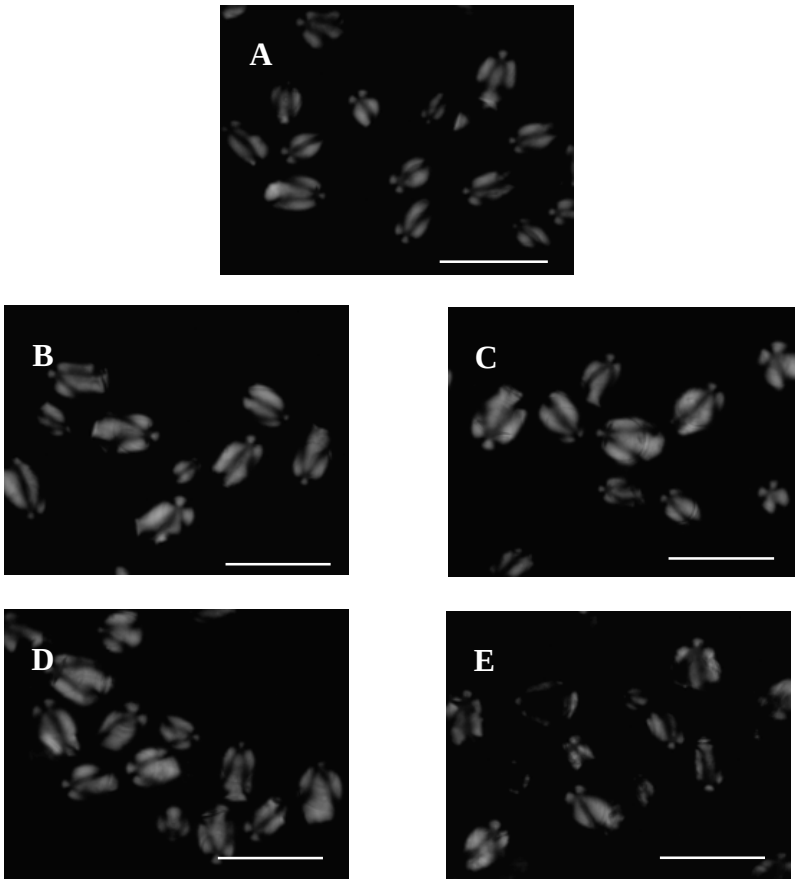

Figure 3.3.1. Birefringence changes of untreated sago starch and micro-powder-milled sago starch of WC under polarized light

Magnification is ×40 and bar indicates 50 μm.

A: untreated sago starch sample; B: micro-powder-milled sample with water coolant (WC) and wide clearance setting (T1); C: with WC and wide–medium clearance setting (T2); D: with WC and medium–narrow clearance setting (T3); E: with WC and narrow clearance setting (T4).

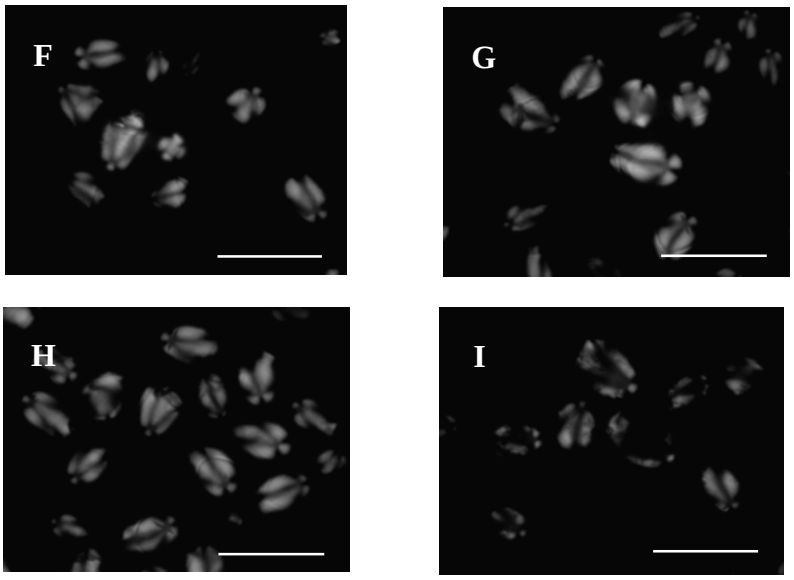

Figure 3.3.2. Birefringence changes of untreated sago starch and micro-powder-milled sago starch of IC under polarized light

Magnification is ×40 and bar indicates 50 μm.

F: micro-powder-milled sample with ice-water coolant (IC) and wide clearance setting (T1); G: with IC and wide–medium clearance setting (T2); H: with IC and medium–narrow clearance setting (T3); and I: with IC and narrow clearance setting (T4).

3.3.2. Particle size

Micro powder mill is a mechanical process which working under the frictional force of discs to disrupt cell-matrices of the SPW in order to liberated sago starch. Starch granules can be damage during milling process, particularly particle size of starch granules because the disc friction generates heat. Changes in the size of the starch granules can be analyzed using laser diffraction.

Graphic of particle size distribution of the sago starch obtained using different milling treatments are shown in Fig. 3.4 and the mean diameters and spans summarized in Table 3.1. The average size of T0 granules varied from 17.3 to 36.4 µm. The average sizes of all the granules of sago starch milled at T1, T2, and T3 were similar, and slightly higher than that of T0. T4 treatment gave larger granules, of average sizes from 18.0 to 108.2 µm and from 17.9 to 109.4 µm for WC and IC treatments, respectively. The $D_{(v,0.5)}$ value increased, indicating increasing particle size. The effect of micro powder milling on the size distribution of starch granules was not greater than that of ball milling. This might be because micro powder milling only involves friction, whereas ball milling involves friction, collisions, impingement, shearing, and other mechanical actions (Huang et al., 2008).

The span reflects the width of the starch granules. The particle size distribution index of T0 was 0.80 µm and did not differ from those obtained using wide and medium clearance (T1, T2, and T3) milling. For the T4 treatment, however, the particle size distribution was twice as broad, around 2.2 µm, with WC and IC. This might be caused by swelling of the starch granules during milling because micro powder mill produce heat in milling action.

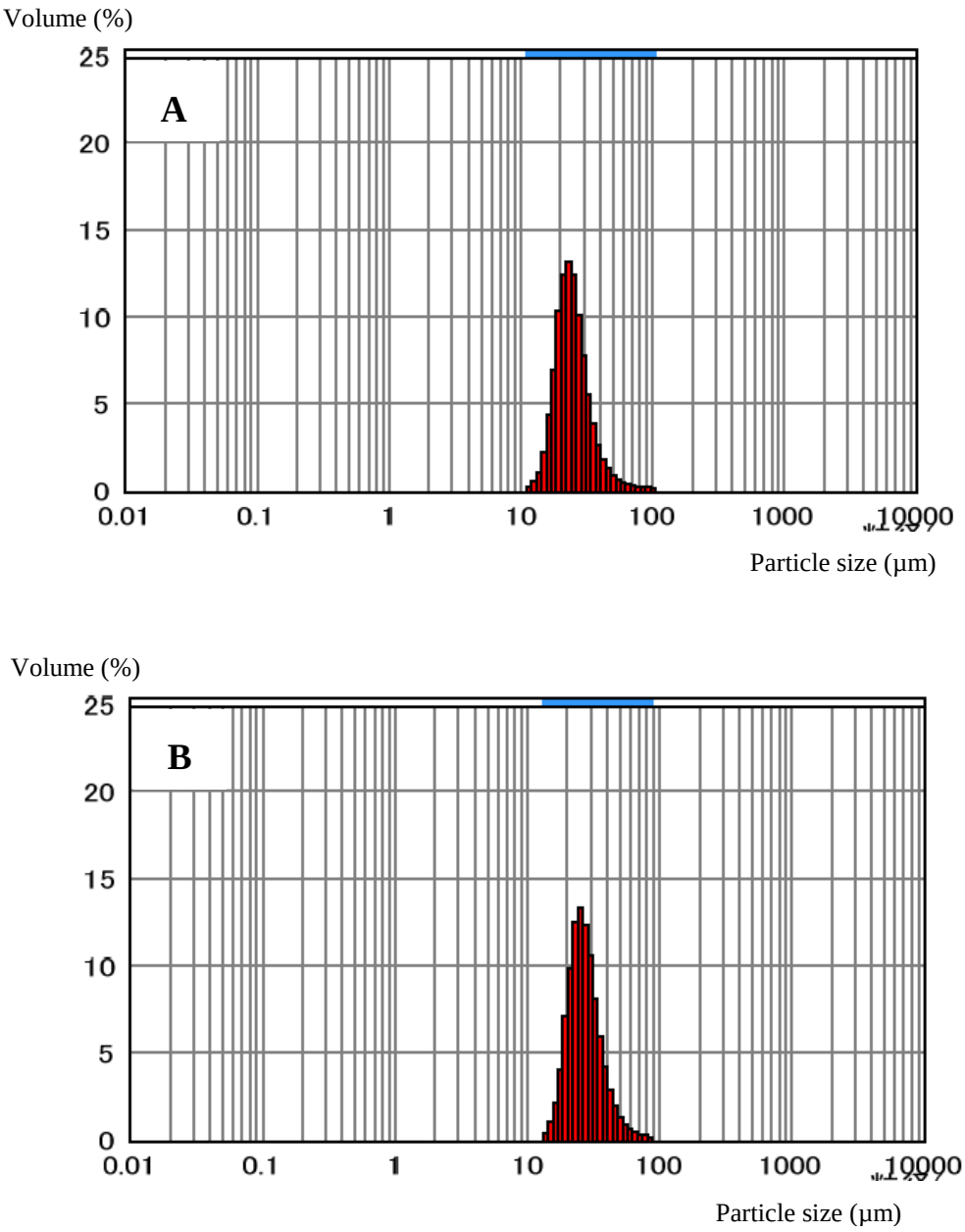

Figure 3.4.1. Particle size distribution of untreated and micro-powder-milled sago starch T1 of WC

A: untreated sago starch sample; B: micro-powder-milled sample with water coolant (WC) and wide clearance setting (T1).

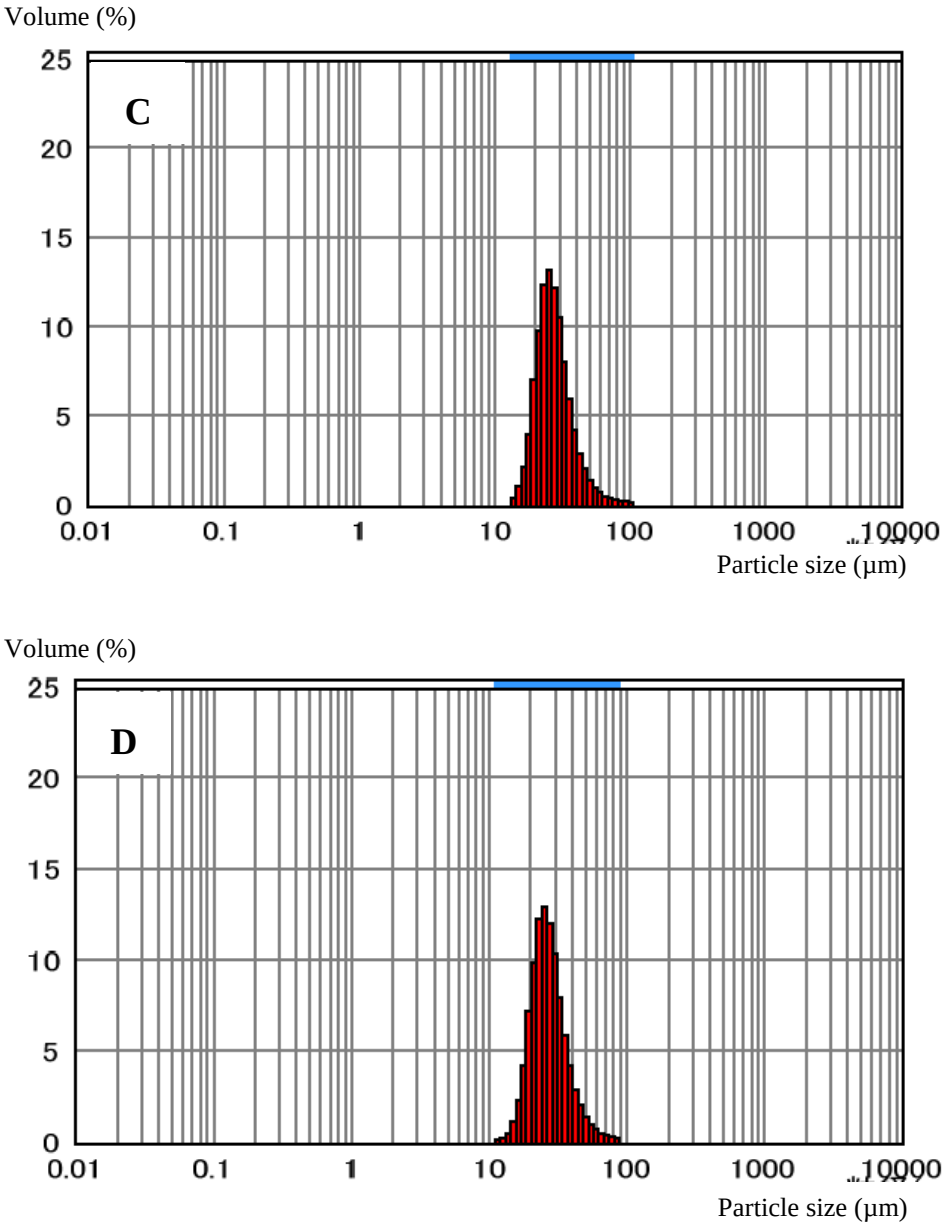

Figure 3.4.2. Particle size distribution of micro-powder-milled sago starch T2 and T3 of WC

C: micro-powder-milled sample with water coolant (WC) and wide–medium clearance setting (T2); D: micro-powder-milled sample with water coolant (WC) and medium–narrow clearance setting (T3).

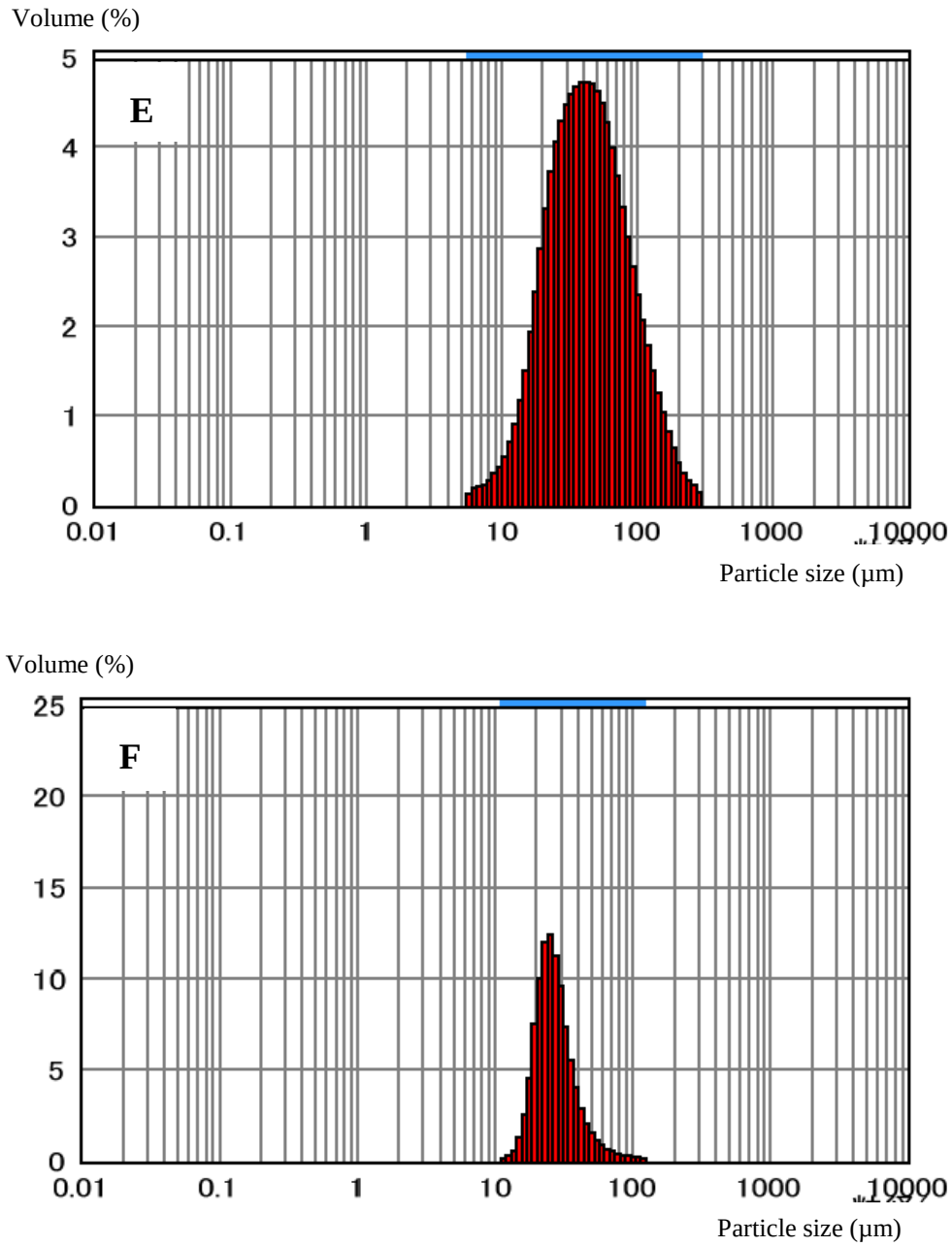

Figure 3.4.3. Particle size distribution of micro-powder-milled sago starch T4 of WC and T1 of IC

E: micro-powder-milled sample with water coolant (WC) and narrow clearance setting (T4); F: micro-powder-milled sample with ice-water coolant (IC) and wide clearance setting (T1).

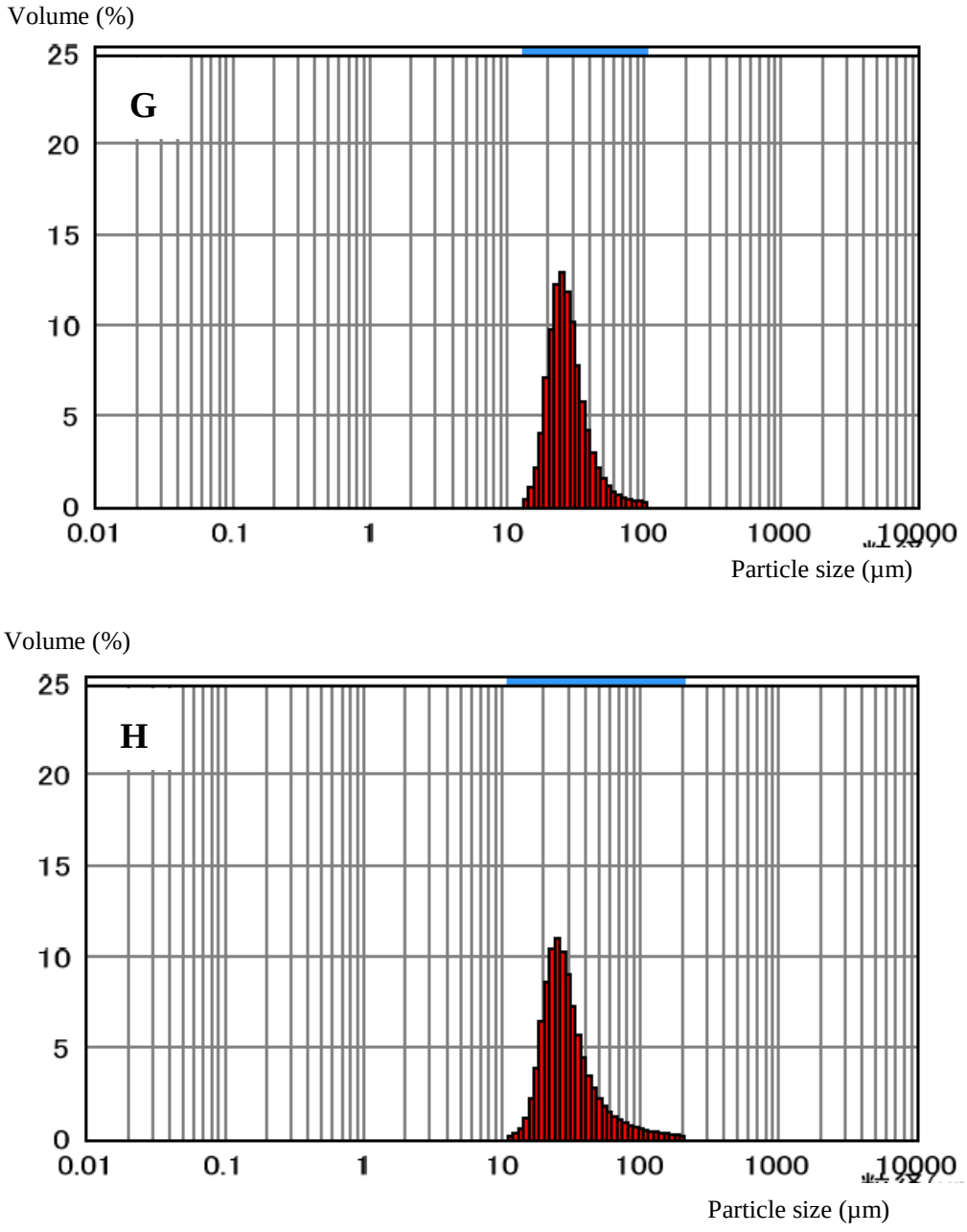

Figure 3.4.4. Particle size distribution of micro-powder-milled sago starch T2 and T3 of IC

G: micro-powder-milled sample with ice-water coolant (IC) and wide–medium clearance setting (T2); H: micro-powder-milled sample with ice-water coolant (IC) and medium–narrow clearance setting (T3).

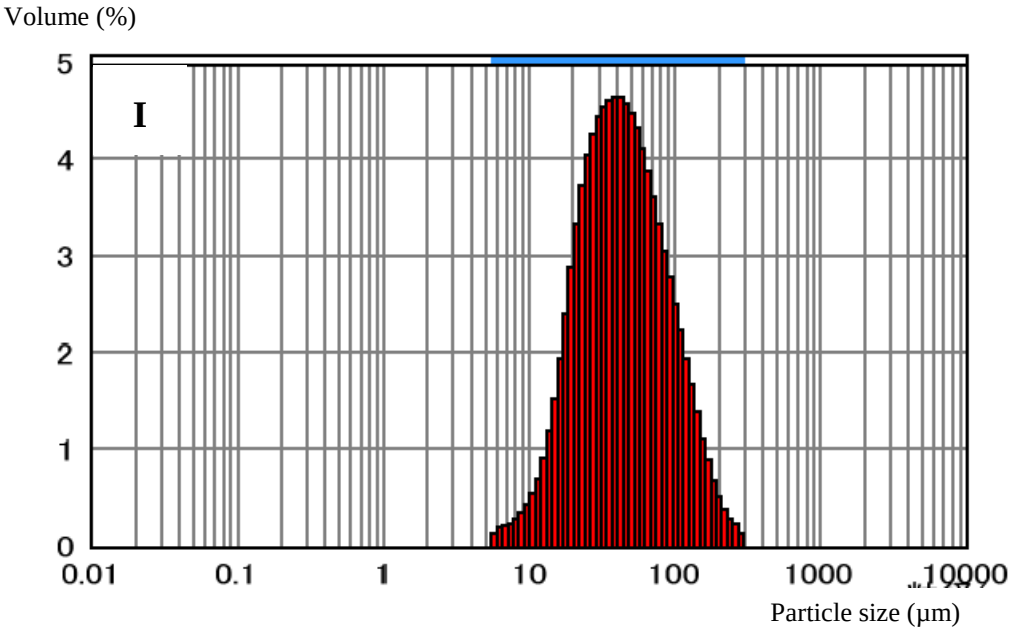

Figure 3.4.5. Particle size distribution of micro-powder-milled sago starch T4 of IC

I: micro-powder-milled sample with ice-water coolant (IC) and narrow clearance setting (T4).

Table 3.1. Size distributions of sago starch obtained using various micro-powder-milling treatments[a]

Treatment	Particle size			
	$D_{(v,0.1)}$ (µm)	$D_{(v,0.5)}$ (µm)	$D_{(v,0.9)}$ (µm)	Span[b]
T0	17.3 ± 0.1	23.8 ± 0.1	36.4 ± 0.2	0.8 ± 0.0
WC-T1	19.2 ± 0.0	26.1 ± 0.1	39.2 ± 0.1	0.8 ± 0.0
WC-T2	19.2 ± 0.0	26.1 ± 0.1	39.5 ± 0.7	0.8 ± 0.0
WC-T3	18.9 ± 0.0	26.1 ± 0.1	40.1 ± 0.6	0.8 ± 0.0
WC-T4	18.0 ± 0.1	42.0 ± 0.2	108.2 ± 1.3	2.2 ± 0.0
IC-T1	18.7 ± 0.0	25.9 ± 0.1	41.4 ± 0.2	0.9 ± 0.0
IC-T2	19.1 ± 0.0	26.2 ± 0.0	41.0 ± 0.1	0.8 ± 0.0
IC-T3	19.1 ± 0.0	27.4 ± 0.0	53.0 ± 1.2	1.2 ± 0.0
IC-T4	17.9 ± 0.0	42.2 ± 0.3	109.4 ± 1.2	2.2 ± 0.0

[a]Parameters are given as average value ± standard deviation.

[b]Span: size dispersion index (Eq. 4).

WC: water coolant; IC: ice-water coolant; T0: untreated; T1: wide clearance; T2 and T3: medium clearances; T4: narrow clearance; $D_{(v,0.1)}$ and $D_{(v,0.9)}$ represent particle diameters at cumulative particle volumes of 10% and 90%; $D_{(v,0.5)}$: median diameter.

3.3.3. XRD

Starch is deposited in semi-crystalline granular form. From a molecular perspective, it mainly consists of two types of α-linked D-glucosyl homopolymers, namely amylose and amylopectin. Amylose and amylopectin are packed into granules, which are part crystalline and part amorphous in structure. It is hypothesized that the crystallites are composed of parallel, left-handed double helices formed from the short external amylopectin chains (Wang et al., 2008).

Typically, native starch granules range in degree of crystallinity from about 15 % to 45 % (Zobel, 1988). X-ray pattern of the native starch granules have been used to categorize starches into A-, B-, and C-polymorphs (Imberty et al., 1991). A-type pattern is associated mainly with cereal starches and has main peaks at 15°, 17°, 18°, and 23° (O'Brien and Wang, 2008, Huijbrechts et al., 2008, and Hu et al., 2014). B-type pattern is usually obtained from tuber starches and shows main peaks at 5.6°, 15°, 17°, 22°, and 24° (O'Brien and Wang, 2008; Alvani et al., 2011). The C-type is mixture of both A- and B-type. The C-type formed when both A- and B-polymorphic arrangement coexist and characterized by diffraction peak at 2θ values of around 5° and strong peaks at around 15°, 17°, 18° and 23° (Karim et al., 2008; Wang et al., 2008). The main difference between A- and B-types is that the former adopt a close-packed arrangement with water molecules between each double helical structure, while the B-type is more open, there being more water molecules, essentially all of which are located in a central cavity surrounded by six double helices (Imberty and Paerez, 1998). There is a close relationship between the weight-average chain lengths of the amylopectins and crystal type of starch granules. Short chain lengths display A-type crystallinity, while long chain lengths show B-

type crystallinity and intermediate chain lengths is associated with C-type crystallinity (Hizukuri, 1985).

The XRD pattern and the relative crystallinities of T0 and sago starch micro powder milled at different levels of disc clearance are presented in Fig. 3.5, Fig. 3.6 and Table 3.2. As expected, T0 had a C-type diffraction pattern with peaks at 2θ values of ~15°, 17°, 18°, and 23°, and a small peak at 2θ = 5°. This result agreed with Ahmad et al. (1999). The relative crystallinity of T0 was around 30.5 %; this is within the range reported by Srichuwong et al. (2005) and Adawiyah et al. (2013) i.e., between 27.5 % and 32.9 %. The XRD patterns show that the crystallinity decreased with decreasing milling clearance, and the crystalline peaks almost disappeared for T4 treatment. The crystalline region decreased gradually from 30.5 % for T0 to 11.2 % and 13.3 % for T4 milling treatments with WC and IC, respectively.

Starch crystallinity affects the physical, mechanical, and technological properties of numerous starch-based products, and is therefore relevant for product development, quality and process control. In food, loss of native crystallinity through gelatinization influences apparent viscosity, gelation and matrix forming characteristics, whereas reordering of the starch during processing or storage has impact product texture, stability, quality, digestibility and functionality (Sajilata et al., 2006).

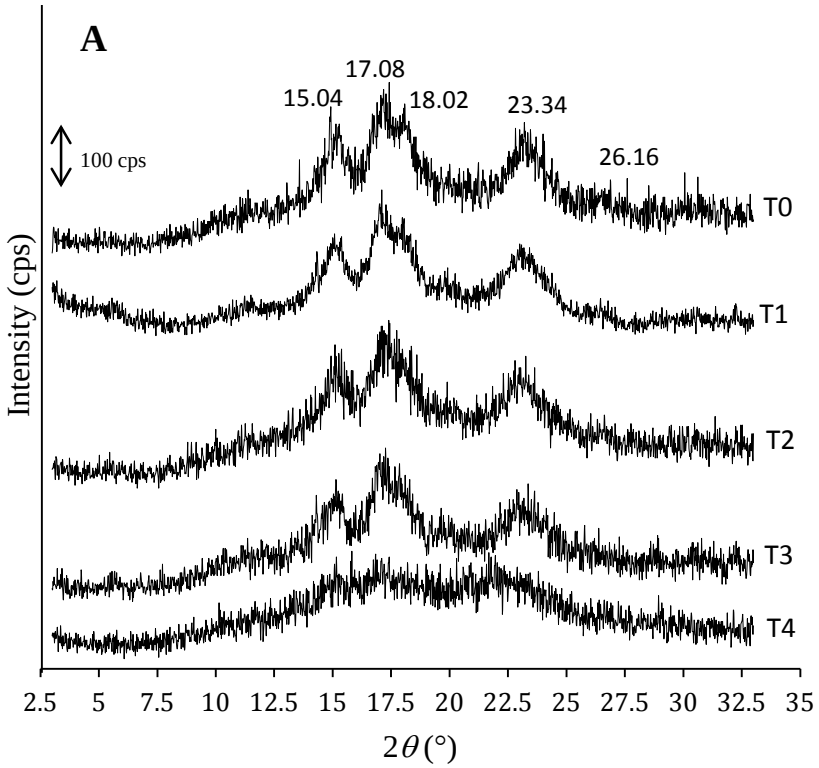

Figure 3.5. X-ray diffraction patterns of untreated sago starch and micro-powder-milled sago starch with water coolant (A)

T0: untreated sago starch sample; T1: micro-powder-milled sample with wide clearance setting; T2: with wide - medium clearance setting; T3: with medium - narrow clearance setting; and T4: with narrow clearance setting.

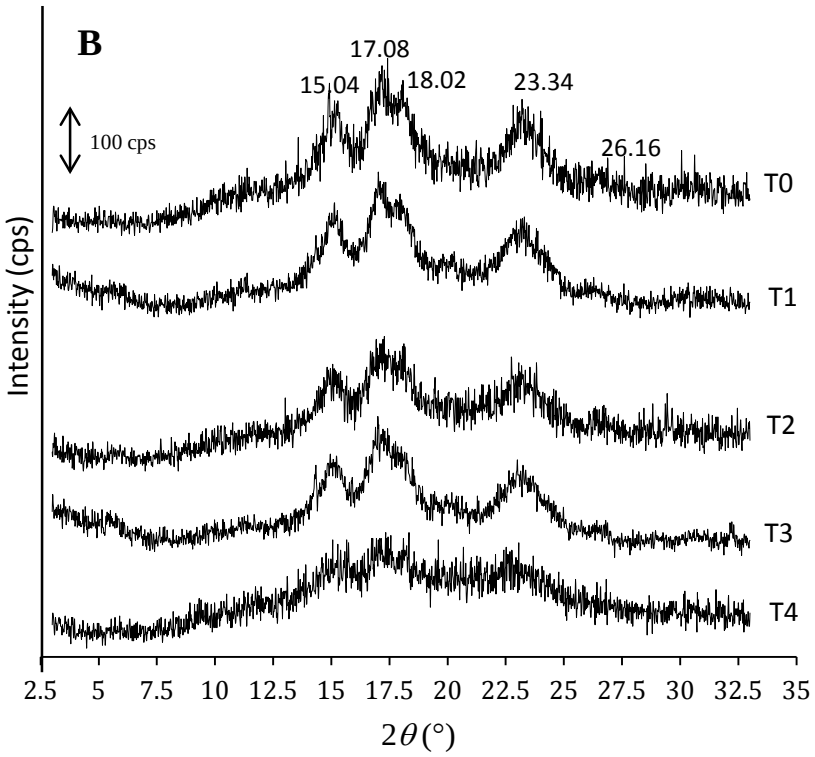

Figure 3.6. X-ray diffraction patterns of untreated sago starch and micro-powder-milled sago starch with ice-water coolant (B)

T0: untreated sago starch sample; T1: micro-powder-milled sample with wide clearance setting; T2: with wide–medium clearance setting; T3: with medium–narrow clearance setting; and T4: with narrow clearance setting.

Table 3.2. Relative crystallinities of sago starch obtained using various micro powder milling treatments[a]

Treatment	RC[b] (%)
T0	30.5 ± 1.8
WC-T1	30.1 ± 1.6
WC-T2	22.4 ± 0.7
WC-T3	22.1 ± 1.1
WC-T4	11.2 ± 1.1
IC-T1	29.8 ± 1.2
IC-T2	24.4 ± 0.9
IC-T3	23.2 ± 1.6
IC-T4	13.3 ± 0.9

[a]Parameters are given as average value ± standard deviation.

[b]RC: relative crystallinity.

3.3.4. DSC

When starch granules are heated in excess water to progressively higher temperatures, a point is reached where the polarization cross starts to disappear and the granules begin to swell irreversibly. These phenomena, associated with the disruption of granular structure, are called ‘gelatinization’. In this context, gelatinization can be defined as disruption of molecular orders (breaking of H-bonds) within the granule, along with all concomitant and irreversible changes in properties such as water uptake, granular swelling, crystallite melting, birefringence loss, starch solubilization and viscosity development (Beliaderis, 2009).

There are three main processes happen to the starch granule: *First*, water will penetrate into the amorphous space of starch, which leads to a swelling of starch granules. This phenomenon occurs during heating. *Second*, water continue to enters via amorphous regions the tightly bound areas of double helical structures of amylopectin. Heat causes such regions to become diffuse and the amylopectin chains begin to melt, to separate into an amorphous form and the number and size of crystalline regions decreases. *Third*, Penetration of water thus increases the randomness in the starch granule structure and this pressure caused by this swelling eventually rupture the granule and allows for leaching of amylose molecules to surrounding water (Donald, 2004). DSC method was used to study the gelatinization behavior of the native starches.

The DSC results and gelatinization temperatures for T0 and micro-powder-milled sago starch are presented in Table 3.3 and Fig. 3.7 and Fig. 3.8. The transition temperatures associated with the gelatinization and retrogradation, namely onset (T_o), peak (T_p) and conclusion (T_c). The onset temperature (T_o), peak temperature (T_p), and conclusion temperature (T_c) did not differ much for all

treatments. The average T_o, T_p, and T_c values of T0 were 56.3, 66.9, and 75.9 °C, respectively. For treated sago starch, the T_o values varied from 53.7 to 57.3 °C, the T_p values varied from 66.3 to 66.8 °C, and the T_c values varied from 71.5 to 75.9 °C.

The ΔH during gelatinization is the amount of energy needed to convert crystalline structure to amorphous structure. The ΔH values for gelatinization primarily reflect the loss of molecular (double-helical) order (Cooke and Gidley, 1992). The data in Table 3.3 show that the enthalpy of gelatinization sharply decreased from 15.4 J/g for T0 to 3.8 J/g and 4.9 J/g for starch subjected to T4 treatment with WC and IC, respectively. The decrease in the crystalline region was related to the particle size (Table 3.1) and ΔH value (Table 3.3). This suggests that the physical force in the T4 treatment is sufficient to disrupt the crystalline structure. The gelatinization temperature and enthalpy of the starch depends on the microstructure and degree of crystallinity within the granules, the granule size, and the amylose to amylopectin ratio (Ahmad et al., 1999). The results of this study showed that T4 treatment could lead to damage of the amylopectin crystalline structure, and the coolant temperature had no effect in this treatment. Normally the smaller the granule the higher will be the gelatinization temperature (Cowburn, 1989). Micro-powder-milling is a physical process. This process is similar with cryo-milling and ball-milling. According to Dhital et al. (2011) that the cryo-milled starches had qualitatively similar properties to those of ball-milled starches, suggesting that milling is predominantly a physical process rather than a thermal process.

Table 3.3. Gelatinization properties of sago starch obtained using various micro powder milling treatments[a]

Treatment	Gelatinization properties				
	T_o (°C)	T_p (°C)	T_c (°C)	Range ($T_c - T_o$) (°C)	ΔH (J/g)
T0	56.3 ± 0.3	66.9 ± 0.0	75.9 ± 0.1	19.6 ± 0.1	15.4 ± 0.5
WC-T1	56.1 ± 1.3	66.5 ± 0.0	74.9 ± 0.1	18.8 ± 1.4	14.5 ± 0.7
WC-T2	57.0 ± 0.2	66.8 ± 0.1	75.8 ± 1.6	18.8 ± 1.4	12.1 ± 0.0
WC-T3	56.3 ± 0.3	66.3 ± 0.1	74.6 ± 0.0	18.3 ± 0.3	11.9 ± 0.6
WC-T4	53.7 ± 1.6	65.3 ± 0.5	71.5 ± 1.3	17.8 ± 0.2	3.8 ± 0.6
IC-T1	57.3 ± 0.1	66.7 ± 0.1	74.5 ± 0.2	17.2 ± 0.3	13.0 ± 0.9
IC-T2	56.8 ± 1.8	66.8 ± 0.1	75.1 ± 0.6	18.3 ± 2.4	13.6 ± 1.0
IC-T3	55.8 ± 0.6	66.8 ± 0.1	73.9 ± 0.6	18.1 ± 1.3	12.5 ± 1.2
IC-T4	56.7 ± 2.4	65.7 ± 0.1	73.6 ± 0.2	17.0 ± 2.6	4.9 ± 0.9

[a]Parameters are given as average value ± standard deviation.

T_o: onset temperature; T_p: peak temperature; T_c: conclusion temperature; ΔH: enthalpy.

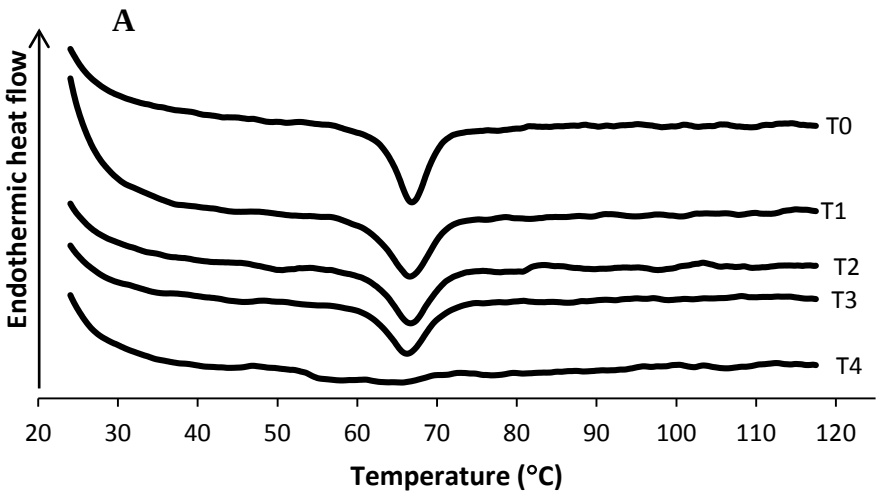

Figure 3.7. DSC thermograms of untreated sago starch and micro-powder-milled sago starch with water coolant (A)

T0: untreated sago starch sample; T1: micro-powder-milled sample with wide clearance setting; T2: with wide - medium clearance setting; T3: with medium - narrow clearance setting; and T4: with narrow clearance setting.

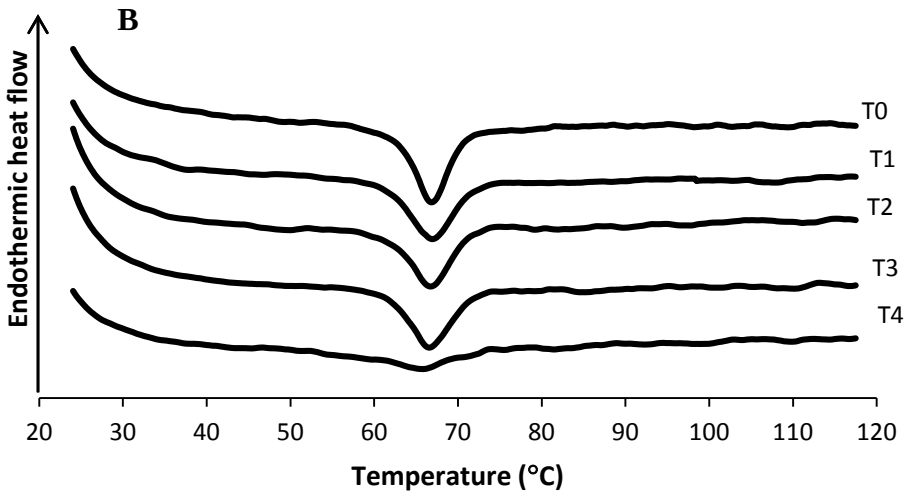

Figure 3.8. DSC thermograms of untreated sago starch and micro-powder-milled sago starch with ice-water coolant (B)

T0: untreated sago starch sample; T1: micro-powder-milled sample with wide clearance setting; T2: with wide–medium clearance setting; T3: with medium–narrow clearance setting; and T4: with narrow clearance setting

3.4. Summary

Micro-powder-milled sago starch obtained by wide treatment (T1), wide-medium treatment (T2) and medium–narrow treatment (T3) properties were similar to those of untreated sago starch (T0). Physical changes in the starch granules, such as changes in the starch surface, granule size, relative crystallinity and gelatinization properties, predominantly occurred during narrow treatment (T4). Based on the physicochemical properties and the sago starch yield, the best result of micro powder milling of SPW was achieved by medium–narrow treatment (T3).

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CHAPTER 4. ANALYSIS OF MOLECULAR WEIGHT DISTRIBUTION OF MICRO-POWDER-MILLED SAGO STARCH

4.1. Background

Starch contains two fraction were amylose and amylopectin chains. Amylose and amylopectin are substances that categorized as major component in the starch with composition 20-30 % and 70-80 %, respectively. Therefore, the contents and the structures of amylopectin and amylose play major roles in the functional properties of starch (Jane, 2009).

Amylose is a linear (1 → 4)- linked -α-D-glucan and amylopectin a highly branched molecule which consist of short chains of (1 → 4)- linked -α-D-glucose with (1 → 6)-α-linked branches. Chain lengths of amylose are commonly in excess of 6,000 D-glucopyranose units with molecular weight ranging from 150,000 to 600,000 Da. While, amylopectin is very branched with an average of 17 – 26 D-glucosyl units separating the α-(1 → 6) branch points.

Amylopectin is one of the largest naturally occurring macromolecules because it has molecular weight around 10^6 D-glucosyl residues per molecule (Pérez et al., 2009). In native sago starch contains amylopectin around 68.8 % and amylose around 31.2 % (Nadiha et al., 2010). In this study, we tried to milling of SPW using a micro powder mill to obtained the sago starch, and the micro powder mill is physical treatment which can be produce heat and also cause induce gelatinization of starch. The objective of this experiment was to analyze the effect of micro powder mill on degradation of either amylopectin or amylose chains.

4.2. Materials and methods

4.2.1. Materials

The main materials in this experiment were untreated sago starch (T0); micro-powder-milled sample with water coolant (WC) and wide clearance setting (T1); with WC and wide–medium clearance setting (T2); with WC and medium–narrow clearance setting (T3); with WC and narrow clearance setting (T4); with ice-water coolant (IC) and wide clearance setting (T1); with IC and wide–medium clearance setting (T2); with IC and medium–narrow clearance setting (T3); and with IC and narrow clearance setting (T4). *Flavobacterium odoratum* isoamylase was obtained from GODO-FIA, Godo Shusei Co.Ltd. (Japan). The gels of Toyopearl HW-75F was obtained from Tosoh Co., Ltd. (Japan). All chemicals used in the study were of analytical grade.

4.2.2. Gel chromatography

Molecular weight distribution of untreated and micro-powder-milled sago starch were analyzed using a gel permeation chromatography (GPC). About 50 mg of sample was gelatinized with 500 µL of 40 % $HClO_4$ in ice bath and stirred using a glass rod for 60 min. The solution added 1.7 mL of 7 % NaOH and diluted in 8 mL of distilled water, following by centrifugation at room temperature at 3,824×g for 10 min. The solution was neutralized with 1 M HCl and adjusted pH 6 – 7 and then diluted with distilled water up to 20 mL. Taken 5 mL and mixed with 5 mL of distilled water and then applied to column.

Each sample was applied on a column (2.6 cm I.D. × 100 cm) of Toyopearl HW-75F and eluted with 50 mM NaCl containing 0.02 % of NaN_3 by using

peristaltic pump on the flow rate of 126 mL/h (2.1 mL/min). Carbohydrate in each fraction (10 mL) was estimated by the phenol-H_2SO_4 method (DuBois et al. 1956).

4.3. Results and discussion

GPC has been used to study about distribution of molecular weight in starch polymer. GPC can be separate the compounds based on their molecular weight, especially for the compounds which have large molecular weight. GPC pattern and results of the sample are shown in Fig. 4.1 to 4.5 and Table 4.1, meanwhile the results of iodine-reaction of sample is shown in Table 4.2. Based on the GPC pattern of untreated sago starch (Fig. 4.1A) can be divided into two fractions. From fraction number 21 (F.N. 21) to fraction number 38 (F.N. 38) proposed as amylopectin equivalent fraction (F1) and fraction number 39 (F.N. 39) to fraction number 55 (F.N. 55) proposed as amylose equivalent fraction (F2). The fraction number 38 (F.N. 38) was a transition point of those amylopectin and amylose fractions. Untreated sago starch shows only one peak at F1 and then shows a little shoulder at F2. GPC pattern of untreated sago starch (T0) was not different with those of T1, T2 and T3 treatments for both water and ice-water coolant. However, the shoulder of amylose equivalent (F2) becomes peak at the pattern of T4 treatment (Fig. 4.3E and Fig. 4.5I). Increasing of the F2 area was only ranging from 2 – 5 % of T1, T2 and T3 than that of T0 treatment. Increasing of F2 area of T4 treatment was 8 % and around 12 % of amylopectin was transferred to amylose area. Iodine color (Table 4.2) shows the blue color drop of T1, T2 and T3 was fluctuating from 20 – 30 % of untreated sago starch. While declining the blue color of T4 was higher (50 %) compared with those of T1, T2 and T3 treatments which ranging from 20 – 30 %.

Because of the drops of the blue value was associated to amylopectin degradation, then this result shows that degradation of amylopectin was higher on T4 treatment.

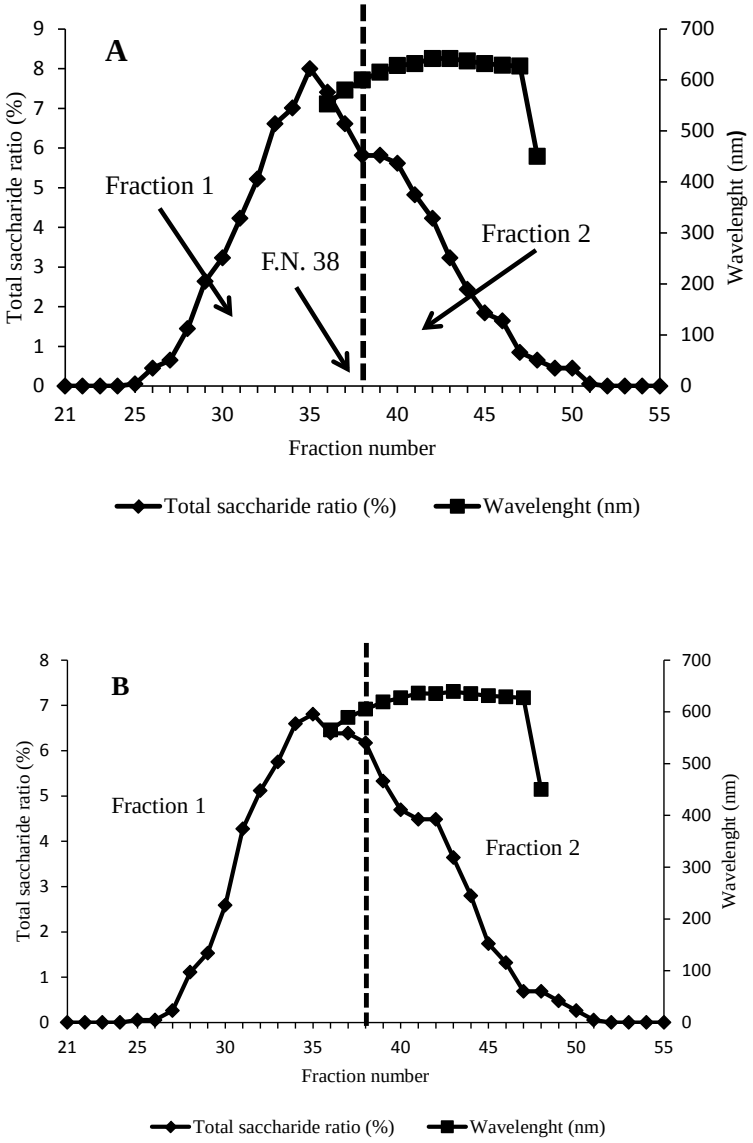

Figure 4.1. GPC profiles of of untreated and micro-powder-milled sago starch T1 of WC on Toyopearl HW-75F

A: untreated sago starch sample; B: micro-powder-milled sample with water coolant (WC) and wide clearance setting (T1).

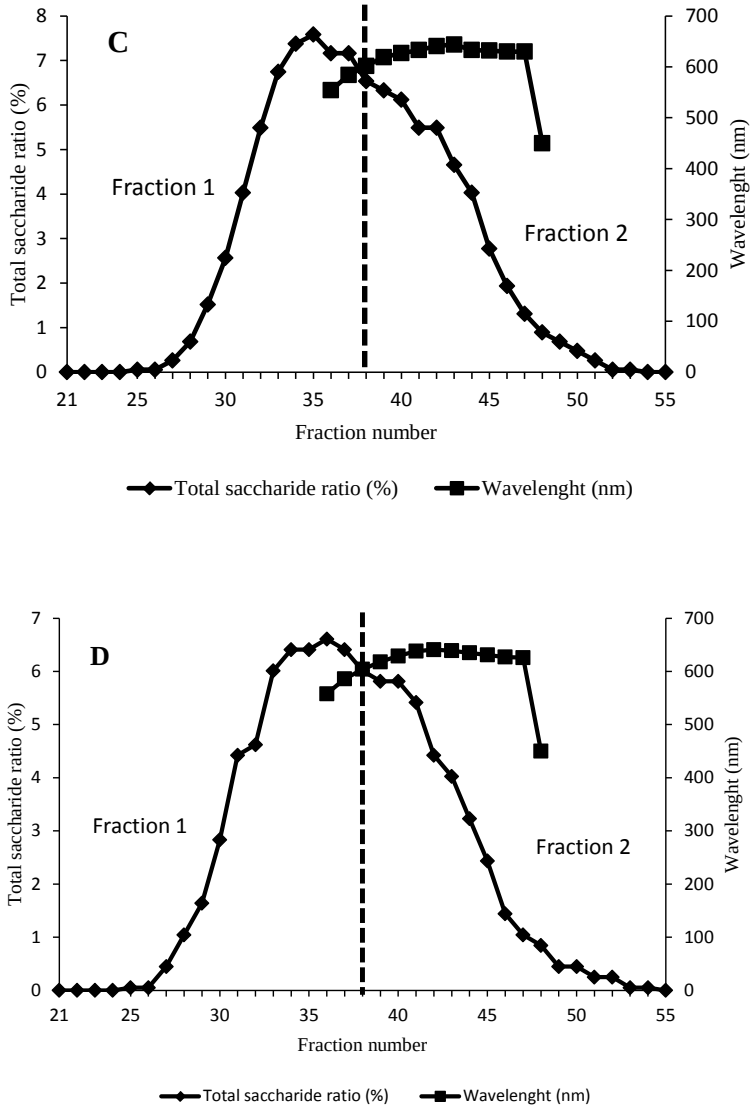

Figure 4.2. GPC profiles of micro-powder-milled sago starch T2 and T3 of WC on Toyopearl HW-75F

C: micro-powder-milled sample with water coolant (WC) and wide–medium clearance setting (T2); D: micro-powder-milled sample with water coolant (WC) and medium–narrow clearance setting (T3).

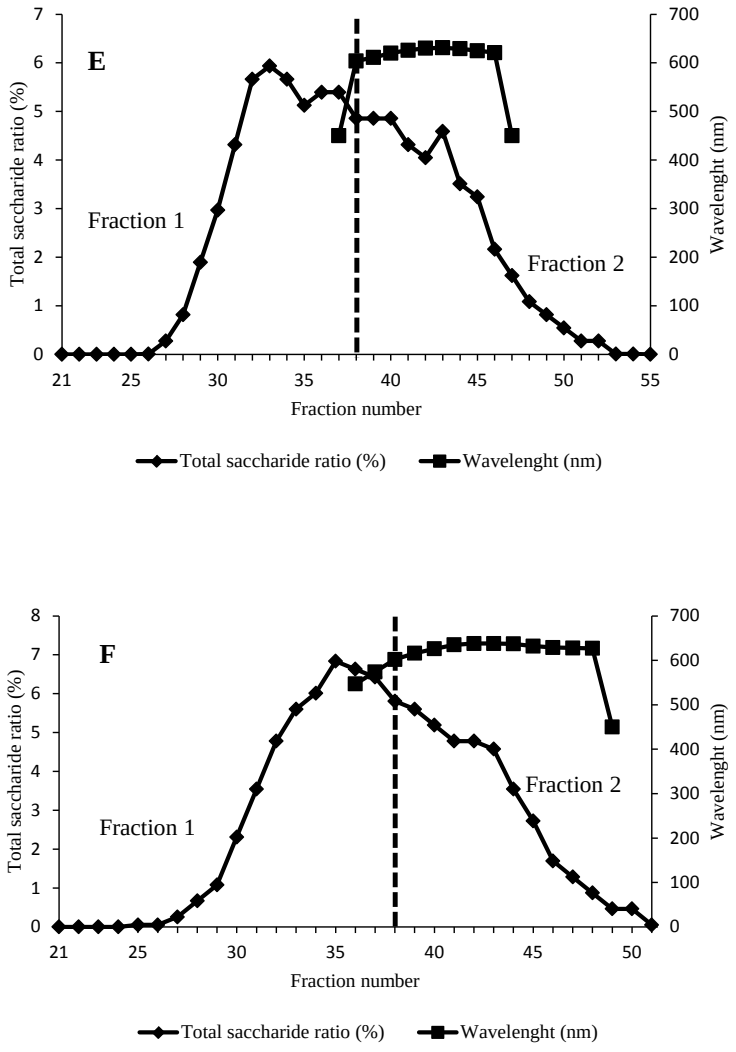

Figure 4.3. GPC profiles of micro-powder-milled sago starch T4 of WC and T1 of IC on Toyopearl HW-75F

E: micro-powder-milled sample with water coolant (WC) and narrow clearance setting (T4); F: micro-powder-milled sample with ice-water coolant (IC) and wide clearance setting (T1).

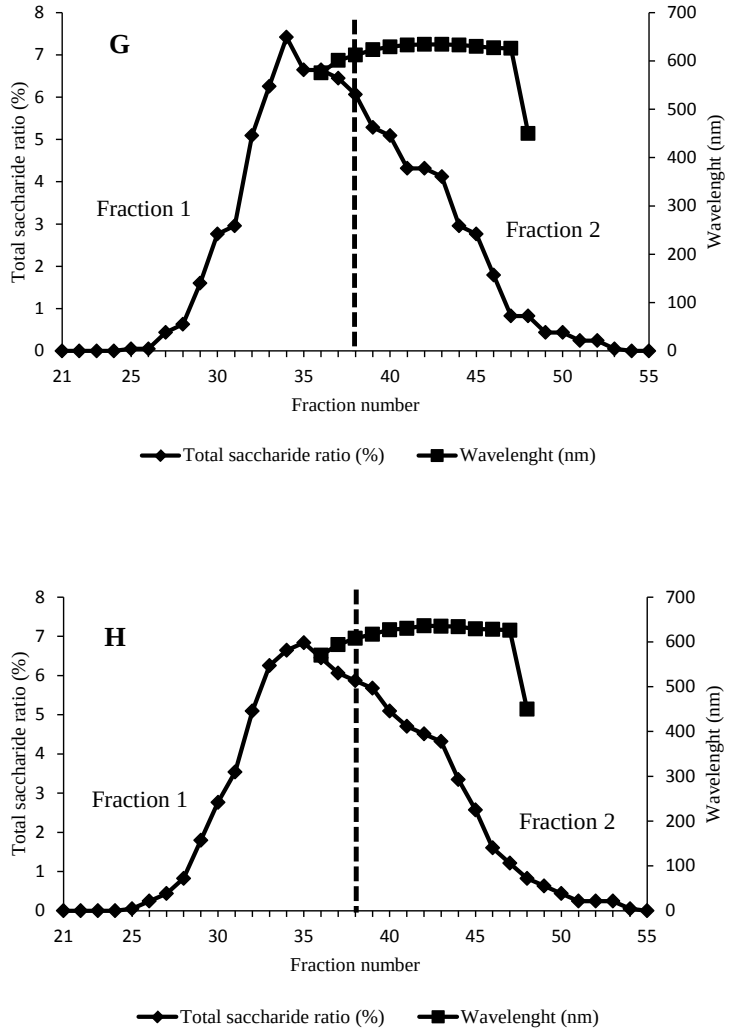

Figure 4.4. GPC profiles of micro-powder-milled sago starch T2 and T3 of IC on Toyopearl HW-75F

G: micro-powder-milled sample with ice-water coolant (IC) and wide–medium clearance setting (T2); H: micro-powder-milled sample with ice-water coolant (IC) and medium–narrow clearance setting (T3).

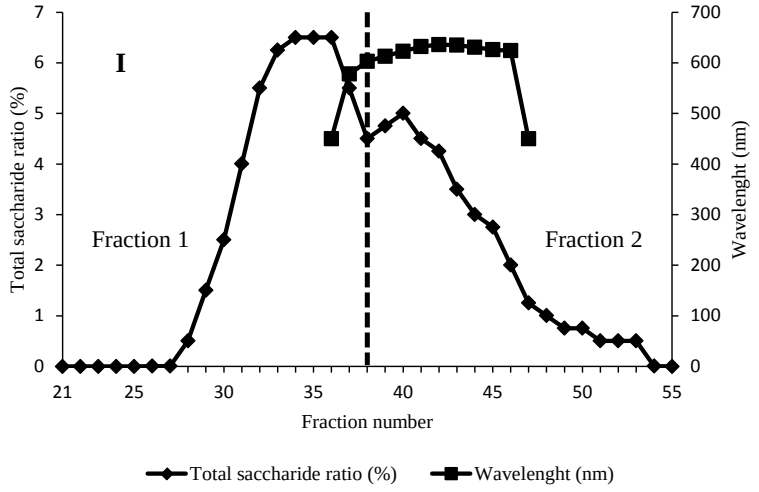

Figure 4.5. GPC profiles of micro-powder-milled sago starch T4 of IC on Toyopearl HW-75F

I: micro-powder-milled sample with ice-water coolant (IC) and narrow clearance setting (T4).

Table 4.1. Ratio of untreated and micro-powder-milled sago starch components separated by GPC on Toyopearls HW-75F

Treatment	Fraction 1 (%)	Fraction 2 (%)
T0	65 (100)	35
WC-T1	63 (97)	37
WC-T2	59 (91)	41
WC-T3	60 (92)	40
WC-T4	57 (88)	43
IC-T1	62 (95)	38
IC-T2	61 (94)	39
IC-T3	60 (92)	40
IC-T4	57 (88)	43

Table 4.2. Iodine color reaction of untreated and micro-powder-milled sago starch components separated by GPC on Toyopearls HW-75F

Treatment	Iodine color reaction	
	λ max (nm)	ε 680 (Abs.)
T0	642	1.309 (100)
WC-T1	635	0.916 (70)
WC-T2	645	1.050 (80)
WC-T3	641	0.975 (74)
WC-T4	631	0.635 (49)
IC-T1	638	1.053 (80)
IC-T2	634	0.933 (71)
IC-T3	636	0.950 (73)
IC-T4	632	0.680 (52)

4.4. Summary

In this study, GPC pattern of untreated sago starch (T0) was not different with those of T1, T2 and T3 treatments for both water and ice-water coolant. The high amylopectin degradation has occurred on narrow treatment (T4). This result agreed with iodine-reaction where the blue color of T4 treatment was decreases almost 50 % comparing with other treatments.

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CHAPTER 5. REGIONAL INNOVATION ASPECT

5.1. Sago starch production and energy consumption for milling process

Sago pith waste (SPW) is a starchy lignocellulosic by-product produced from pith of sago palm after extraction (Fig. 5.1). The amount of SPW released from the sago processing factory depends mostly on the efficiency and sophistication of the methods employed (Mohd et al., 2001). Bujang et al. (1996) estimated that approximately 7 t of SPW was produced daily from a single sago starch processing mill, and currently these residues were washed off into nearby stream together with waste water and deposited in the factory's compound, which can lead to serious environmental problems since the SPW contain high organic materials.

As mentioned above that the SPW which contained mostly starch and lignocellulosic materials. Lai et al. (2013) reported that the amount of sago starch in SPW varies between 58 % and 67 % on a dry weight basis. In this study, we managed to increase the sago starch yield from micro-powder-milled SPW by around 17% and 18% by T3 and T4 treatment, respectively.

According to Ehara (2010) Indonesia produces approximately 208,000 tons of sago starch per year and the amount of SPW is discarded around 68% (442,000 tons). Based on data of the micro-powder-milled SPW yield (Figure 2.6), we can increase the sago starch production is around 75,000 t per year for T3 treatment and around 80,000 t per year for T4 treatment (Figure 5.2). Schemes should be sought for taking advantage of this as it is an important industrial theme for overcoming shortages of this starch. The annual demand for sago starch continues to increase because sago starch gel is firmer, and has lower adhesiveness and higher cohesiveness compared

with other starches, and is suitable for food production (Hamanishi et al., 2002). And also, in this experiment, we managed to decrease around 17 – 18% of sago waste, thus we can reduce the pollution problems from sago starch processing plant.

Basically, sago starch has multitude of uses both singly or mixed with other starches. It can be consumed as staple food, but also it can be processed to produce several foods in small scale, such as bread, vermicelli, noodle, cracker, pearl sago and stabilizer. At recent time, however, sago starch widely used for agroindustry (glucose syrup and fructose syrup), as well as bio-pesticides, bio-ethanol, bio-degradable plastics, cosmetics and the pharmaceutical industry (Bintoro, 2011). Therefore, the utilization of each treatment of micro-powder-starch sago starch as shown in Table 5.1.

Micro-powder-milled sago starch can be utilized for all products (food and non-food products), except for T4 treatment cannot used for staple food and small scale food industry due to contain a lot of fiber and has occurred gelatinization (Table 3.3). Therefore, T4 treatment is suitable used for sweetener and bioethanol. In generally, bioethanol can be produced by using three kinds of raw materials, such as sugary materials (sugar cane, sugar beet, molasses and fruit liquid), starchy materials (grains and potatoes) as well as cellulosic materials (rice straw, wood, etc.) (Balat et al., 2008). However, bioethanol production using sago starch as raw material (first generation materials) has been debate, because at this time, the whole countries pay special attention to overcome food security problem. Therefore, the production of bioethanol is preferable to use non-starch (second generation) as raw materials, such as lignocellulosic biomass materials, and also does not compete with food needs.

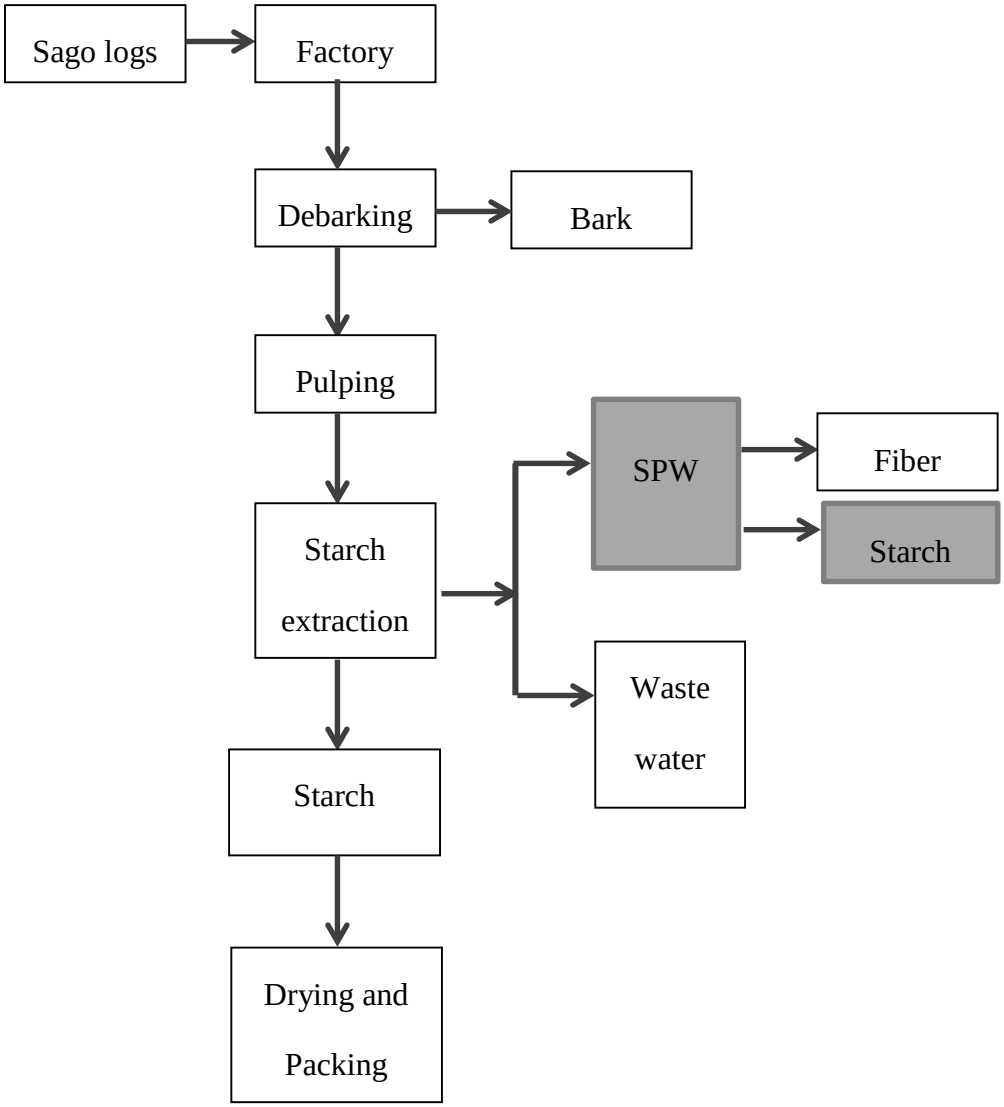
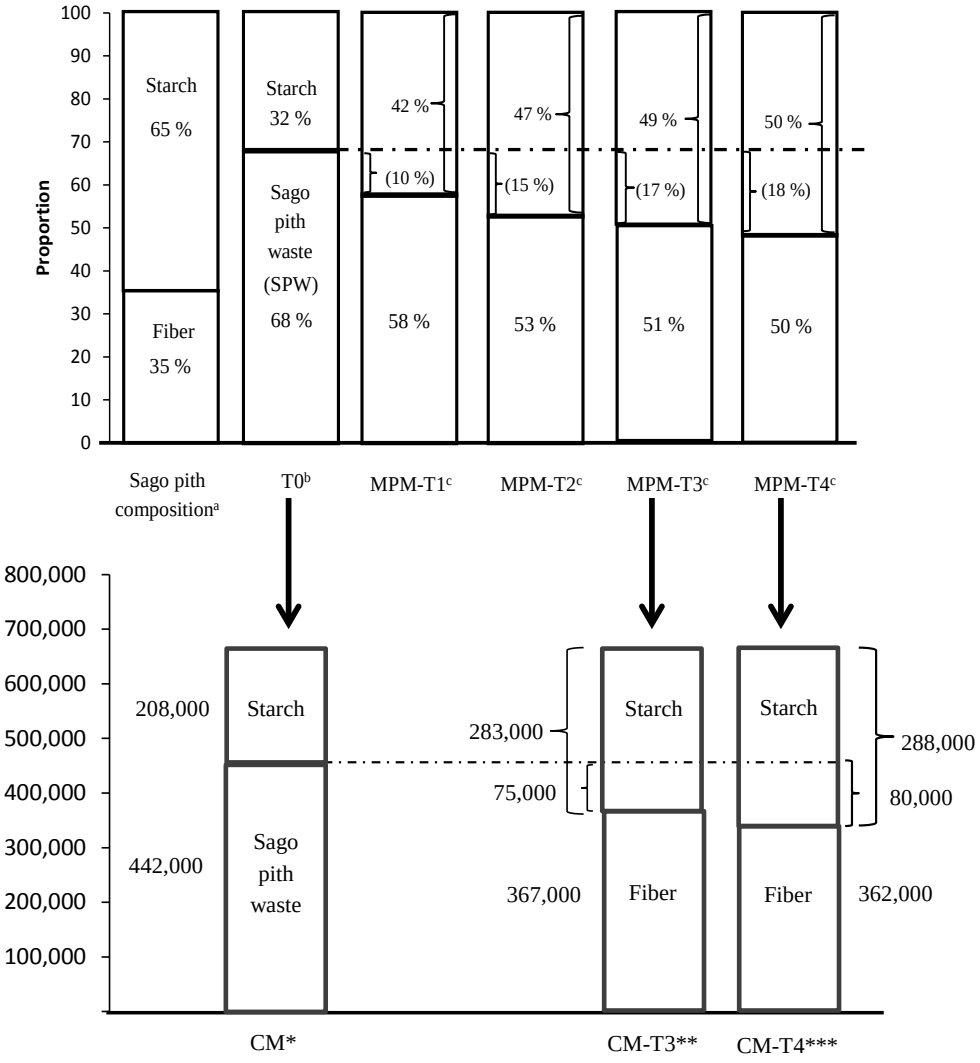


Figure 5.1. Flow diagram for sago processing

(Awg-Adeni et al., 2010 adapted from Yean and Lan, 1993)



*) Starch production in Indonesia by commercial method (Ehara, 2010)

**) Commercial method + MPM-T3

***) Commercial method + MPM-T4

Figure 5.2. Comparison sago starch yield between commercial method and micro-powder-milled SPW

Table 5.1. Utilization of micro-powder-milled sago starch

Treatment	Food product	Non-food product
T1, T2, T3	Staple food, small scale food industry, and agroindustry product	a variety of non-food products
T4	Agroindustry product, except pharmaceutical products	a variety of non-food products, except cosmetics and pharmaceutical products

*) Small scale industry: bread, vermicelli, noodle, cracker, pearl sago, etc.

*) Agroindustry: glucose syrup, fructose syrup, bio-ethanol, bio-pesticides, bio-degradable plastic, cosmetics and pharmaceutical industry.

Energy consumption was calculated using formula as described by Ngamnikom and Songsermpong (2011) as follows:

The electrical power for single phase was calculated using Eq. (5):

$$P = (A \text{ x } V \text{ x } P.F)/1000 \ldots\ldots(5)$$

The electrical energy consumption during grinding was calculated using Eq. (6):

$$E = P \times t \ldots\ldots(6)$$

The specific energy consumption was calculated using Eq. (7):

$$Es = E / WS \ldots\ldots(7)$$

The unit are P = electrical power (kW), A = electrical current (ampere), V = voltage (volt), $P.F$ = power factor (no unit), E = electrical energy (kJ), t = second, Es = specific energy consumption (kJ kg^{-1}), WS = weight sample (kg).

The micro powder mill used in this experiment is laboratory scale machinery with the power is 300 W (single phase). The milling time of T3 and T4 was 0.14 kg/h and 0.11 kg/h, respectively. Energy results, based on these equations, are shown in Table 5.2. T4 treatment requires more milling time because it passed to four steps, while T3 treatment was only passed three steps in milling process. Therefore, T4 treatment consumed more specific energy consumption (7,714.29 kJ kg^{-1}) than T3 treatment (9,818.18 KJ kg^{-1}). The specific energy consumption in this experiment was higher compared with other equipment, such as hammer mill (420 kg^{-1}), pin mill (795 kg^{-1}) and roller mill (801 kg^{-1}) (Ngamnikom and Songsermpong, 2011). Moreover, Mohd Rozalli et al. (2015) found that the specific energy consumption decreases with the decrease of milling time. The specific energy consumption was 85.68 kJ kg^{-1} for 5 min of milling time and then decrease to 39.40

kJ kg^{-1} for 2 min of milling time. The energy consumption during grinding/milling is varies depend on the ratio of initial and final particle size distribution of materials before and after milling, moisture content, hardness, the feed rate of material, pre-treatment before grinding as well as machine variables, i.e. type of machine, speed and screen size (Mohd Rozalli et al., 2015). Based on this data, in order to reduce the specific energy consumption of the micro powder mill, in the future, we should scale up the capacity of the micro powder mill. After that, we should investigate about the sago starch quality, especially particle size distribution, because according to Mohd Rozalli et al. (2015) that short milling time produced larger range of particle size distribution than long milling time.

Table 5.2. Energy used for milling process

Treatment	Electrical energy (kJ)*	Specific energy consumption (kJ kg^{-1})
T3 of micro powder mill	1080	7714.29
T4 of micro powder mill	1080	9818.18
Hammer mill**		420.00
Pin mill**		795.00
Roller mill**		801.00
Ultra-high speed grinding in 2 min***		39.40
Ultra-high speed grinding in 5 min***		85.68

*) Based on the micro powder mill specification

**) Ngamnikom and Songsermpong (2011)

***) Mohd Rozalli et al. (2015)

5.2. General conclusion

Recovery of starch from the sago pith waste (SPW) using a micro powder mill is a new innovation to increase the sago starch production. This innovation is not only valuable for sago farmers and industries, but also for government. Micro powder milling method is applicable for mass production in sago factory. This study represents a new opportunity to further investigate physicochemical and enzymatic properties of the micro-powder-milled sago starch, as well as utilization of the micro-powder-milled sago starch for food and non-food product is an aspect that worthy in further research.

5.3. References

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SUMMARY

Recently, food security and fossil fuel are hot issues in the world because the number of world population continue to increase. This indicates that the world needs to produce at least 50 % more food to feed 9 billion people by 2050. Therefore, all countries should be seeking and utilizing a new source of food to anticipate food insecurity in the world. Beside that, the world faces fossil fuel problem. In 2012, total consumption of energy in the world is dominated by fossil fuel and only a small portion is renewable fuel. One of an alternative to anticipate food insecurity and fossil fuel problems is sago palm tree. Recently, attention to sago starch as a new food and a food-security crop continue to increase, especially in order to the anticipated increase in human population and potential environmental disasters in future. Several studies have been reported that the sago starch can be extracted only up to 55 – 75% (dry basis) of the total starch in the sago trunk. The remaining starch is still trapped within the parenchyma cells or fiber in sago pith waste (SPW). This fact indicates that not all sago starch potential in the sago trunk can be obtained. The purpose of this study was to minimize the loss of starch through recovery of sago starch from sago pith waste to increase sago starch production.

Overall, this study consists of five consecutive experiments, where each of chapter was discussed in different topic. Chapter 1 was a general introduction which discussed about food security, potency of sago, the role of sago for food security and biofuel production, sago starch extraction process and the general purpose of the study.

Chapter 2 was about recovery of sago starch from SPW by a micro powder mill (MPM). The objective of this study was to recovery of sago starch from SPW by using a micro powder mill. In this experiment, sago starch from sago pith was

produced by using commercial method. Then the true starch yield of sago pith was measured through hydrolysis process using α-amylase from porcine pancreas. To produce micro-powder-milled sago starch, the SPW was milled using a MPM with two kind of coolant, that are water (WC) and ice-water (IC) coolant to reduce the heat from the MPW. Milling process was performed in four disc clearance levels, namely wide (T1), wide-medium (T2), medium-narrow (T3) and narrow (T4). The result shows that the sago starch from SPW which left behind after starch extraction can be extracted by micro powder milling. The highest yield of micro-powder-milled sago starch was T4 (18%) and then followed by T3, T2 and T1 were 17%, 15% and 10%, respectively. This method is more practical and efficient than other milling processes because it is a continuous system and requires a short milling time.

In chapter 3 discusses about physicochemical properties of MPM sago starch. The objective of this study was to investigate the physicochemical properties of sago starch obtained by MPM of SPW for all treatments. The properties of MPM sago starch were investigated using scanning electron microscopy (SEM), normal and polarized light microscopy, particle size analysis, X-ray diffraction (XRD), and differential scanning calorimetry (DSC). The result revealed that MPM sago starch obtained by both water coolant and ice-water coolant in wide treatment (T1), wide-medium treatment (T2) and medium–narrow treatment (T3) properties were similar to those of untreated sago starch (T0). Physical changes in the starch granules, such as changes in the starch surface, granule size, relative crystallinity, and gelatinization properties, predominantly occurred during narrow treatment (T4). Based on the physicochemical properties and the sago starch yield, the best result of micro powder milling of SPW was achieved by medium–narrow treatment (T3).

Chapter 4 is about the molecular weight distribution of MPM sago starch. The objective of this experiment was to analyze the effect of micro powder mill on degradation of either amylopectin or amylose chains. Based on the GPC pattern of untreated sago starch can be divided into two fractions. From fraction number 21 to fraction number 38 proposed as amylopectin equivalent fraction. While fraction number 39 to fraction number 55 proposed as amylose equivalent fraction. The GPC pattern of HW-75F column chromatography shows that untreated sago starch (T0) was not different with those of T1, T2 and T3 treatments for both water and ice-water coolant. However, degradation of amylopectin was higher on T4 treatment. This trend was followed by iodine color where a blue color was decrease around 50% on T4 treatment.

Regional innovation aspect (chapter 5) is further explanation regarding the result of previous experiment (chapter 2 to chapter 5). This chapter discusses about the benefit of this research to farmers, industry, government and environment. Indonesia produced approximately 208,000 t of sago starch and 442,000 t of sago pith waste per year. Using a micro powder mill (dry milling), the sago starch yield will increase around 283,000 t and 288,000 t for T3 and T4 treatments, respectively. While using a super mass colloider mill (wet milling), the sago starch will increase around 300,820 t. The specific energy consumption of a micro powder mill can be reduced with scale up of the capacity of the micro powder mill.

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