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Microwave treatment to optimize physicochemical properties of modified Busil (*Xanthosoma sagittifolium*) starch

[1,2]Hakim, L., [2]Triwitono, P., [2]Supriyanto and [2,*]Marseno, D.W.

[1]*Department of Agroindustry, Banjarnegara Polytechnic, Jl. Raya Kenteng-Madukara Km. 02, Madukara 53482, Banjarnegara, Central Java, Indonesia*
[2]*Department of Food and Agricultural Product Technology, Faculty of Agricultural Technology, Gadjah Mada University, Jl. Flora No. 1, Bulaksumur 55281, Yogyakarta, Indonesia*

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Abstract

COVID-19 pandemic encourages the utilization of local food sources to ensure food availability. Busil (*Xanthosoma sagittifolium*) was readily available and affordable in Banjarnegara Regency in the Province of Central Java in Indonesia. However, the busil starch utilization was still rare due to the low functional properties of the native busil starch. The objective of this study was to explore busil starch physicochemical characterization enhancement after microwave irradiation treatment, especially on the stability of heat processing. This research was conducted in two steps. First, microwave treatment (with a variation of energy and irradiation time) of native busil starch (NBS), and the second was modified busil starch (MBS) physicochemical characterization. A rise in amylose was observed on MBS. SEM analysis was shown MBS granules are breakdown. Through viscosity, final viscosity, setback viscosity, peak time, and the pasting temperature of MBS generally were increased. Meanwhile, peak viscosity and breakdown viscosity of MBS was decreased. Thermal properties of MBS like onset (To), peak (Tp), and conclusion (Tc) temperatures were also increased. The degree of whiteness index (DW) of MBS was decreased. FTIR analysis has shown that microwave treatment did not cause functional group alteration. XRD analysis has also demonstrated no change in the diffraction pattern but a slight change in the crystallinity index. Generally, microwave treatment leads to MBS thermal stability and potentially broaden MBS utilization on food processing product.

1. Introduction

The COVID-19 pandemic condition makes people and goods mobility all over the world restricted. This condition encourages local food sources to ensure food availability as a part of the food security policy system. Busil (*Xanthosoma sagittifolium*), a local food source in Banjarnegara, Indonesia (Rudyatmi and Rahayu, 2014), locally had long been used as a daily snack food (Hakim *et al*., 2021). Many know *Xanthosoma* spp. of names in different countries, including *taro* in Cameroon, y*autia* in the Dominican Republic, *malanga* in Cuba, and *cocoyam* in Ghana and other South Africa. Although it can be found in many countries and known by various names, it is originally from northern South America and Asia (Calle *et al*., 2019). From its location of growth, the name of busil will be used throughout this research. Traditionally it was eaten as steamed and fried busil. This traditional processing is not much different (Olayemi *et al.*, 2008; Odeku, 2013; Adeyanju *et al.*, 2019; Coronell-Tovar *et al.*, 2019). Although busil was readily available and affordable, busil utilization, mainly as native busil starch (Figure 1), was still low. Generally, low utilization of native starches due to deficiencies properties that make native starch inappropriate as a food ingredient, like tight peak viscosity, low thermal process tolerance, form a viscous paste when processed, and unstable gels when cooled or during storage (Deka and Sit, 2016)

Figure 1. Native busil starch

*Corresponding author.
Email: *djagal@ugm.ac.id*

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Starch modification is a method that can be taken to improve native starch properties. Physical modification is the most studied starch modification method in the last decade, mainly due to its simplicity and safety (Ashogbon, 2018). Microwave treatment is one of them that has special attention due to its various advantages, including homogenous heating operation, higher heating rates, precise process control, ease to use, environmental-friendly energy processes, economist, and rapidity (Anderson and Guraya, 2006; Agriculture *et al.*, 2012; Fan *et al.*, 2014; Lewicka *et al.*, 2015; Chizoba Ekezie *et al.*, 2017; Li *et al.*, 2020; Li, Hu, Wang *et al.*, 2019; Lu *et al.*, 2020). Microwave irradiation has been considerably used for starch modification due to the dielectric heating and electromagnetic polarization of hydroxyl groups of structured water and starch (Zhong *et al.*, 2019). Recently, no research has been published on the physicochemical property changes of busil starch after microwave irradiation treatment. This study was focused on exploring busil starch physicochemical properties characterization after microwave irradiation and the effect of different energy and time. This information is essential to find the best microwave modification method and recommended utilization of microwaved busil starch.

2. Materials and methods

2.1 Materials

A local variety of Cocoyam (*Xanthosoma sagittifolium*) or busil was obtained from the farmer in Kali Bening District, Banjarnegara Regency, Central Java, Indonesia, at 11 months of age. Busil that has good quality and without physical defects were selected as sample research. Analytical grade reagents were used in this research.

2.2 Starch isolation

The wet extraction method was used to obtain busil starch. Busil was peeled, washed, and then grated. The grated material was then collected, added with water (1:3), and mixed manually until form a homogenous busil slurry. The filtrate was collected by busil slurry filtration using a clean filter cloth. Overnight sedimentation was done to obtain busil starch on the bottom of the container. Busil starch was decanted, dried on the cabinet dryer at 50°C overnight, grounded, and then sieved through 100 mesh sieve. For further treatment and analysis, native busil starch was packed and kept in a sealed container.

2.3 Microwave treatment

Native busil starches (NBS) were adjusted to 25% (wet basis) moisture content by adding appropriate distilled water. Starches were placed into a sealed container overnight to balance the moisture content before treatment. Microwave energy was used on 0.5 1.0 and 1.5 W/g (labelled with A, B, C respectively) and was carried out for 3, 5, and 7 mins (labelled by 1, 2, and 3 respectively). After microwave treatment was finished, each sample (next called modified busil starch/MBS) was dried on the cabinet dryer (50^{o}C for 12 hrs), milled using a conventional blender, and sieved (100 mesh). The MBS was packed individually and kept in dry places for further analysis.

2.4 Amylose and amylopectin content.

NBS and MBS amylose and amylopectin sample content were analyzed according to AOAC standard method (Horwitz, 2010). Data is displayed on a % dry basis.

2.5 Pasting properties

Pasting properties were determined using a Rapid Visco Analyzer (RVA 4500, Perten Instruments, Sweden) as described by (Ratnaningsih *et al.*, 2020). NMB and MBS (3.5 g) samples were weighed into a sample container, and 25 g of distilled water was added. Starch slurries were held at 50°C for 1 min, heated to 95° C at a rate of 6°C/min, held at 95°C for 5 mins, cooled to 50°C at a rate of 6°C/min, and held at 50°C for 2 mins. The speed was 960 rpm for the first 10 s, then 160 rpm for the remainder of the experiment.

2.6 Differential scanning calorimeter analyses

DSC TA-60WS, Shimadzu, Japan, was used to evaluate the thermal properties of NBS and selected MBS. DSC analysis method adapted from (Ratnaningsih *et al.*, 2020) with slight modification. The sample pan was filled with a starch sample (2 mg) and then added with 8 µL of distilled water. Another pan containing alumina powder (6 mg) was used as a reference. The sample and reference pan was heated at the rate of 10^{o}C/ min from 30^{o}C to 300^{o}C. Onset temperature (To), peak temperature (Tp), and enthalpy of gelatinization (DH) were obtained.

2.7 Colour

Colour parameters were measured with Chroma Meter (Konica Minolta CR-400, Japan). There were L* (lightness), a* (redness to greenness), and b* (yellowness to blueness) parameters of NBS and MBS were observed. The colour analysis method was done as described by (Yanuriati *et al.*, 2017). L*, a*, and b* values were then used to obtain the degree of whiteness (%DW) of NBS and MBS with the equation described by Ratnaningsih *et al.* (2020).

2.8 FT-IR Spectroscopy analysis

The FT-IR spectra of NBS and selected MBS were obtained with IR Prestige-21 (Shimadzu Co., Japan) spectrometer described by (Ratnaningsih *et al.*, 2020). Starch samples (2 mg) were dispersed in 200 mg of KBr (pellet procedure). Samples were analysed in the wave range of 4000–400 cm^{-1} at 25ºC. All spectra were displayed using Origin 2016 software.

2.9 X-ray diffraction studies

XRD patterns from NBS and selected MBS were determined using Rigaku MiniFlex600 (Japan), operated at 40 kV and 30 mA with a scan rate of 4°C/min. The diffraction angle (2θ) observed ranged from 5° to 50°. All spectra were displayed using Origin 2016 software.

2.10 Granule morphology and size

The granule morphology of selected native and modified busil starch was determined by scanning electron microscopy (SEM) using Hitachi SU 3500 (Japan). Starch samples were coated with gold-palladium. The magnification, accelerating voltage, and other crucial technical observation data were displayed on the micrograph. The size distribution of selected native and modified busil starch granules was measured using a Horiba laser particle size analyzer (Horiba LA-960, Japan). Water was used as a dispersing medium. The refractive index values used were 1.33. Dv (50) value was used as a mean size of sample starch.

2.11 Data analysis

Data were analyzed using IBM-SPSS software version 26 with the variance analysis method (ANOVA), followed by Duncan Test to detect differences. Significance was confirmed at P values < 0.05.

3. Results and discussion

3.1 Amylose and amylopectin content

Amylose and amylopectin are significant constituents of starch that govern the characteristics and functionality of starch (Shah *et al.*, 2016; Shevkani *et al.*, 2017; Biduski *et al.*, 2018). A rise in amylose and a decrease in amylopectin content were observed (Table 1). Both microwave energy and irradiation time variation treatment have shown an amylose parabolic trend line. Amylose content tends to increase at an initial condition, then decrease when both are being raised, and then would be increased again at the final state. Microwave power and irradiation time significantly take effect on amylose and amylopectin content.

Generally, MBS showed higher amylose contents than NBS. This condition was also reported by (Deka and Sit, 2016; Mutlu *et al.*, 2017) and is potentially due to amylopectin breakdown as stated by the previous researcher (Brasoveanu and Nemtanu, 2014; Deka and Sit, 2016; Li, Hu, Zheng *et al.*, 2019; Oyeyinka *et al.*, 2019; Tao *et al.*, 2020). MBS granules were heated homogenously and rapidly, making MBS absorb optimal microwave energy, which reacts with hydrogen bonding forces within busil starch granules and stimulates amylopectin chains breakdown and resulting in linear amylose chains (Brasoveanu and Nemtanu, 2014). The highest amylose content was reached by the B3 sample treated with 1 W/g microwave energy and irradiated for 7 mins. As stated before, there was an amylose parabolic trend line. This condition is likely due to microwave treatment, which leads to amylopectin breakdown at the initial condition and amylose breakdown at the increased microwave energy and irradiation time.

3.2 Pasting properties

The pasting properties of NBS and MBS starch are shown in Table 2. Through viscosity, final viscosity, setback viscosity, peak time, and pasting temperature of MBS were generally increased. Meanwhile, peak viscosity and breakdown viscosity of MBS was decreased. This condition is in line with what was reported by (Brasoveanu and Nemtanu, 2014; Ashogbon and Oluwafemi, 2018; Arinola, 2019). MBS with high amylose content tended to have higher final viscosity and pasting temperature values. This condition could be due to greater hydrogen bonding interactions (Biduski *et al.*, 2018). MBS also has a higher peak time and setback viscosity value, which indicates that MBS was more stable in heat processing and storage.

3.3 Thermal properties

Thermal properties and % crystallinity from NBS and selected MBS sample were shown in Table 3. There was a linear correlation among pasting properties from

Table 1. Amylose and amylopectin from native and modified busil starch

	NBS	A1	A2	A3	B1	B2	B3	C1	C2	C3
Amylose (%)	15.15^{a}	34.41^{i}	29.24^{e}	30.89^{h}	29.67^{g}	16.06^{b}	34.57^{j}	19.25^{c}	27.91^{d}	29.38^{f}
Amylopectin (%)	80.21^{i}	58.61^{b}	64.95^{e}	58.71^{b}	63.12^{b}	77.43^{h}	56.82^{a}	74.41^{g}	66.34^{f}	64.57^{d}

Values with different superscripts within the same row are significantly different ($p<0.05$) among the variation. A, B and C is 0.5, 1.0 and 1.5 W/g microwave energy, respectively. 1, 2 and 3 is the irradiation time at 3, 5 and 7 mins respectively. NBS: native busil starch.

Table 2. Pasting properties from native and modified busil starch

	NBS	A1	A2	A3	B1	B2	B3	C1	C2	C3
Peak visc. (cP)	6153	6001	6217	5722	5944	5788	5627	5852	5658	5799
Trough visc. (cP)	3438	3485	3863	4027	3659	4027	4193	3954	4520	4382
Breakdown visc. (cP)	2715	2516	2354	1695	2285	1761	1434	1898	1138	1417
Final visc. (cP)	4748	5175	5788	6143	5501	6150	6802	6108	7222	7310
Setback visc. (cP)	1310	1690	1925	2116	1842	2123	2609	2154	2702	2928
Peak time (min)	7.1	8.3	8.2	8.5	8.5	8.5	8.3	8.4	8.5	8.4
Pasting temp. (°C)	74.30	75.1	75.8	78.4	76.2	77.6	79.5	77.2	79.1	79.5

A, B and C is 0.5, 1.0 and 1.5 W/g microwave energy, respectively. 1, 2 and 3 is the irradiation time at 3, 5 and 7 mins respectively. NBS: native busil starch.

Table 3. Thermal properties and % crystallinity from native and modified busil starch

	To (°C)	Tp (°C)	Tc (°C)	DH (J/g)	% crystallinity
NBS	69.39	74.45	80.41	11.09	11.54
B1	70.32	75.47	78.35	4.24	13.76
B2	71.44	77.05	88.14	15.18	12.62
B3	74.44	78.50	79.96	2.45	12.48

A, B and C is 0.5, 1.0 and 1.5 W/g microwave energy, respectively. 1, 2 and 3 is the irradiation time at 3, 5 and 7 mins respectively. NBS: native busil starch.

RVA analysis with thermal properties from DSC analysis. Increased the onset (To), peak (Tp), and conclusion (Tc) gelatinization temperatures on MBS showed that microwave treatment could produce stable MBS during the heating process. This condition could be due to stronger interaction between more amylose and amylopectin components, requiring a higher temperature to break down the crystalline regions. Increased To of MBS also indicated the formation of long-chain double-helical crystallites of amylose (Ratnaningsih *et al.*, 2020). Higher amounts of amylose will also decrease the energy for gelatinization (Shevkani *et al.*, 2017) and % crystallinity.

3.4 Colour properties

The value of L*, a*, and b* colour parameters of NBS and MBS were shown in Table 4. Microwave treatment decrease %DW and L* value meanwhile increase a* and b* value. MBS %DW was about 85.60% to 86.44%. According to (Widowati *et al.*, 1997), this value is still met with the Indonesian Industrial Standard for Starch (min 85%). Generally, MBS %DW values were lower than NBS. This condition could occur due heating process from microwave treatment, mainly due to the reduction in L* value because of the starch caramelization. Starch caramelization produces simple sugars by breaking down starch molecules that have a low L* value. These conditions are in line with those reported by (Barua, 2017; Kumar *et al.*, 2020).

3.5 FT-IR spectroscopy analysis

As shown in Figure 2, microwave treatment does not cause the generation or disappearance of functional groups. NBS and MBS FTIR spectra have shown a similar pattern. This condition is in line with those reported by (Chen *et al.*, 2015; Zeng *et al.*, 2016; Han *et al.*, 2020). A minor difference was observed in the O-H group peaks range of 3600–3000 cm^{-1}, where MBS peaks were widened than NBS. This result indicated that hydrogen bonds might be formed on the MBS sample between starch and water molecules, as described by (Shah *et al.*, 2016; Zeng *et al.*, 2016; Wang *et al.*, 2019). The changes in transmittance peak intensity in FTIR spectra showed the starch molecule's ability to absorb microwave energy and transform it into kinetic energy (Tao *et al.*, 2020).

3.6 XRD studies

Same as FTIR spectra, XRD spectra of NBS and

Table 4. Colour properties from native and modified busil starch

	NBS	A1	A2	A3	B1	B2	B3	C1	C2	C3
L*	89.53^{e}	86.42^{a}	86.55^{a}	87.11^{c}	86.89^{b}	86.93^{b}	87.28^{cd}	87.41^{d}	87.20^{c}	87.12^{c}
a*	4.96^{bcd}	5.01^{d}	4.99^{d}	4.99^{d}	4.93^{bc}	4.86^{a}	4.97^{cd}	4.92^{bc}	4.91^{ab}	4.86^{a}
b*	-2.40^{a}	-1.30^{b}	-1.30^{b}	-1.24^{bcd}	-1.26^{bc}	-1.38^{b}	-1.03^{e}	-1.10^{de}	-1.05^{e}	-1.12^{cde}
%DW	88.17^{g}	85.47^{a}	85.60^{a}	86.12^{cd}	85.94^{b}	85.99^{bc}	86.31^{ef}	86.44^{f}	86.25^{de}	86.19^{de}

Values with different superscripts within the same row are significantly different ($p<0.05$) among the variation. A, B and C is 0.5, 1.0 and 1.5 W/g microwave energy, respectively. 1, 2 and 3 is the irradiation time at 3, 5 and 7 mins respectively. NBS: native busil starch.

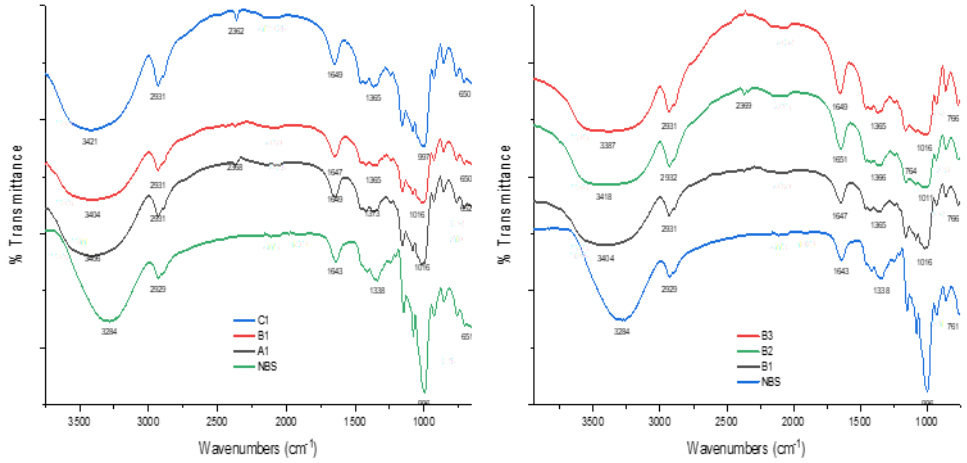

Figure 2. FTIR spectra of NBS and MBS after microwave irradiation at (a) different microwave energy and (b) different times. A, B and C is 0.5, 1.0 and 1.5 W/g microwave energy, respectively. 1, 2 and 3 is the irradiation time at 3, 5 and 7 mins respectively. NBS: native busil starch.

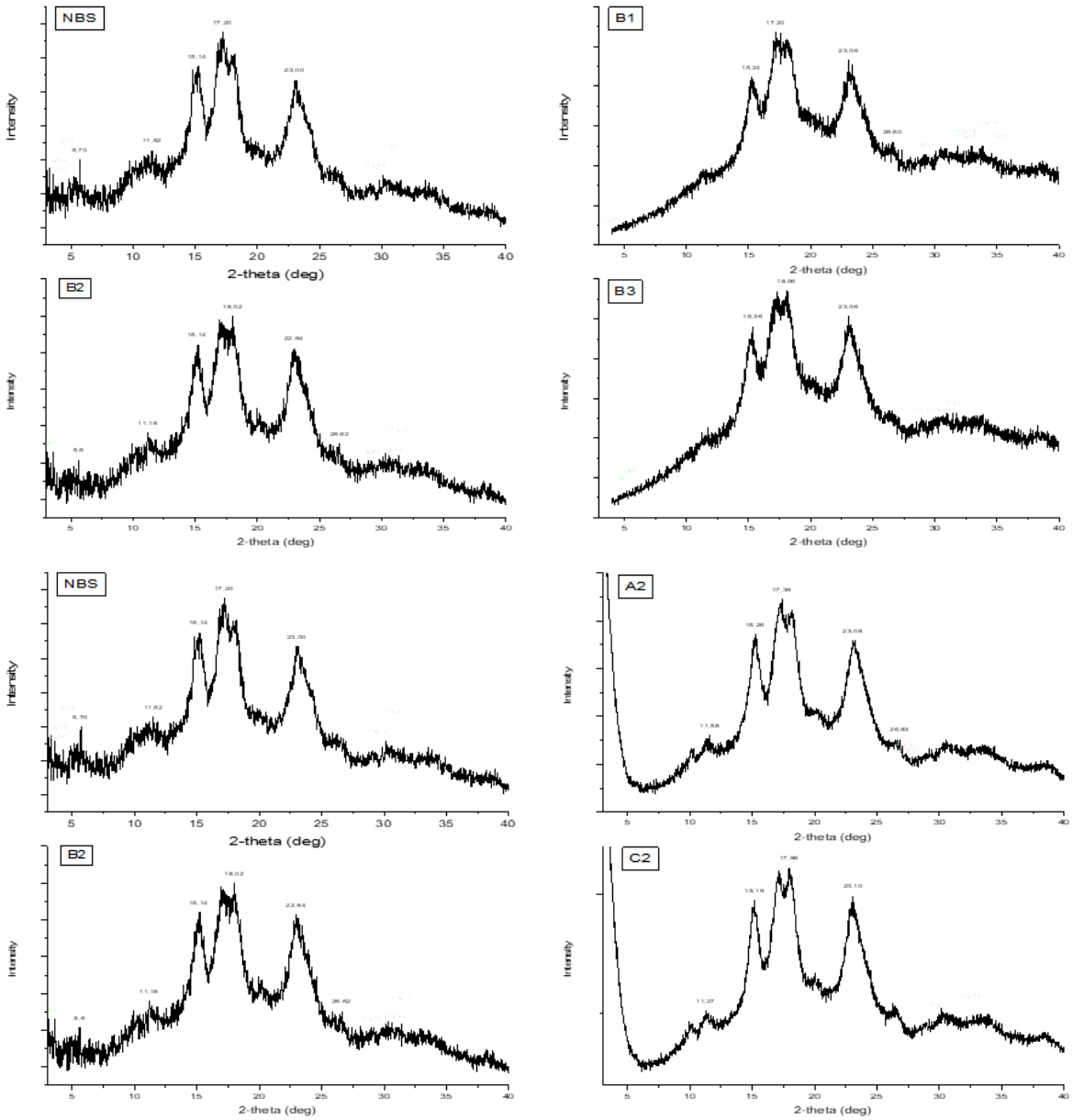

Figure 3. XRD spectra of native busil starch (NBS) and modified busil starch on different microwave irradiation times (1, 2, 3) and on different microwave irradiation energy (A, B, C). A, B and C is 0.5, 1.0 and 1.5 W/g microwave energy, respectively. 1, 2 and 3 is the irradiation time at 3, 5 and 7 mins respectively.

MBS (Figure 3) also have shown a similar pattern. All of XRD spectra have shown 2θ peak at 15°, 17°, 18°, and 23°. That peaks are characteristic of the "A" type of XRD pattern, in line with (Moorthy, 2002). An "A" XRD pattern starch has more density at the helix structure. It shows double helix found, and amylopectin short-chain proportion is higher (Faridah, 2009). Although XRD patterns from NBS and MBS have demonstrated similar, % crystallinity tends to decrease when prolonged irradiation time, as shown in Table 3. This condition showed that microwave treatment could cause crystalline regions breakdown, loss of the double helices, and degradation of the MBS granules, as stated by (Han *et al.*, 2020; Tao *et al.*, 2020). On the other hand, % crystallinity of MBS was higher than NBS. It could be due to the rewinding of amylose-amylose and amylose-amylopectin chains to generate double helix chains and form a crystalline array as an effect of microwave irradiation, which is more ordered than that in NBS (Yang *et al.*, 2017).

3.7 Granule morphology

Scanning electron micrographs of NBS and selected MBS (B3) granule morphology are shown in Figure 4. The NBS granule's shape tended to be oval or round with heterogonous size. This granules size diversity could occur due to the grating effect in the starch extraction method. The grafting method commonly produces heterogeneous starch granules but has maximal starch yield (Wanita, 2018). MBS granules looked more broken and became slightly bigger. MBS granule alteration could happen due to soaking and swelling. Previous research showed that microwave treatment, even for a shorter duration and lower power, proved effective in changing granule morphology (Agriculture *et al.*, 2012).

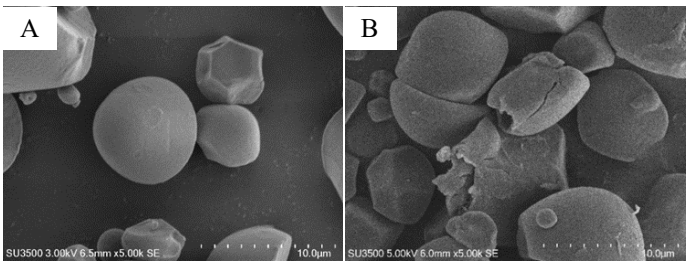

Figure 4. Scanning electron micrographs of NBS (A) and MBS (B) 1 W/g, 7 mins

4. Conclusion

Microwave treatment generally has some advantages to the busil starch. Microwave treatment could raise MBS amylose content, alter pasting, and thermal properties to be more stable on heat processing and storage, and also initiate granules breakdown that will enlarge the starch surface area. Meanwhile, microwave treatment also has a disadvantage in reducing the whiteness index of busil starch. However, this disadvantage can be ignored if MBS is used in the dark-coloured product. Microwave treatment also did not change the busil starch functional group and busil starch diffraction pattern. There was only a slight change in the crystallinity index. This research suggests that microwave treatment effectively improves the physicochemical properties of modified busil (*Xanthosoma sagittifolium*) starch, with 1 W/g microwave energy and 7 mins of irradiation time is recommended for further product development.

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