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Microencapsulation of Sweet Potato Leaf (*Ipomoea Batatas* L.) Extract with Different Concentrations of Glucomannan Konjac and Maltodextrin Using Spray Drying Method

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ABSTRACT: A large amount of phenolic compounds in sweet potato leaves are a potential source of antioxidants. However, polyphenol is sensitive under certain food processing, thereby needs innovation such as microencapsulation to maintain its stability. This study aimed to determine the encapsulation efficiency and characterization of microencapsulation of sweet potato leaf extract using maltodextrin (10% and 20%) combined with Konjac glucomannan (0.5%; 0.75% and 1%). The method used was spray drying using 120 ºC inlet temperature. The result showed that the total phenolic compound of sweet potato leaves was 685.06 GAE mg/g extract. The antioxidant activity of sweet potato leaf extract with the DPPH method was 52.80% with an IC50 value of 26.73 ppm. The highest antioxidant activity of the microencapsulated powder sample (10% maltodextrin: 0.75% glucomannan) was 66.84% at a concentration of 100 ppm which had the greatest percent encapsulation efficiency (89.91%). The particle size distribution showed that encapsulated sweet potato leaf extract has a micro-size of around 0.296 µm (78.3%) with good homogeneity of the particle size which can be seen from the Pdi value of 0.304. Therefore, encapsulated sweet potato leaf with 0,75% glucomannan and 10% maltodextrin has the highest encapsulation efficiency and good characteristic.

Keywords: Glucomannan Konjac, maltodextrin, microencapsulation, polyphenolic compounds, sweet potato leaf

INTRODUCTION

Sweet potato leaf (*Ipomoea batatas* L.) is one of the tropical plants' rich in bioactive compounds. Sweet potato leaf contains high content of polyphenolics that consist of caffeic acid, chlorogenic acid, 3,4-di-O-caffeoylquinic acid, 3,5-di-O-caffeoylquinic acid, 4,5-di-O-caffeoylquinic acid, and 3,4,5-tri-O-caffeoylquinic acid (Kurata *et al.*, 2007). Recently, many researchers have reported that sweet potato leaves have phytochemical benefits such as antioxidative and radical-scavenging activity (Islam, 2006; Truong *et al.*, 2007; Sun *et al.*, 2014;). In Indonesia, the production of sweet potato leaves reached 2,029,353 tons in 2017 (Ministry of Agriculture RI, 2018). However, during the harvesting period, more than 90% of the leaves are discarded while less than 5% is used for animal feeds (Ghasemzadeh *et al.*, 2012). Therefore, the utilization of sweet potato leaves needs to be improved as natural antioxidant sources. However, antioxidants are substances that are sensitive and easy to degrade by external factors such as pH, temperature, light, and many others.

Microencapsulation is a method to protect sensitive active ingredient components against external factors and to ensure the protection of nutritional content and even taste or aroma (Poshadri and Kuna, 2010). Generally, microcapsule coatings are formed from carbohydrate-based ingredients such as gum Arabic, maltodextrin, glucomannan, inulin, and other modified starches. Microencapsulation with maltodextrin coating material effectively produces high-efficiency values in maintaining the bioactive compound (Balasubramani *et al.*, 2015; Nambiar *et al.*, 2017; Lima *et al.*, 2019).

Maltodextrin is one of the polysaccharides that is mostly used for coating material. Maltodextrin is a coating that can increase the encapsulation efficiency, this is probably because of the ability of maltodextrin to form the encapsulant surface during the drying process (Balasubramani *et al.*, 2015). In addition, glucomannan is a source of polysaccharides that are generally extracted from the Konjac plant that have good characterization to be used for encapsulation material. Glucomannan from *Amorphophallus konjac* has linear random copolymers (1 → 4) β–D-mannose and β–D-glucose bonds composed of glucose and mannose units at a molar ratio of 1.6:1 with group-level low acetyl (Zhang *et al.*, 2014). Previous research revealed that the physical properties of glucomannan from Konjac tubers were not much different from Konjac glucomannan so, in this study, glucomannan was used as an encapsulation material for the polyphenol extract of sweet potato leaves (Harmayani *et al.*, 2014). Glucomannan has desirable properties of good film-forming, high water solubility, edibility, and biodegradation (Wattanapraset *et al.*, 2016).

Microencapsulation can be prepared by the spray drying method. Technically, spray drying is changing samples from liquid to powder particles by spraying feed into a hot

drying medium. In recent years, spray drying techniques have been developed for the encapsulation process of food compound components, such as bioactive content, flavor, and also antibacterial compounds. Microencapsulation using glucomannan as the coating material has been reported to be able to protect bioactive compounds in spray drying encapsulation (Wattanapraset *et al.*, 2016). This study aimed to study the encapsulation efficiency of microencapsulation of sweet potato leaf extract through the combined amount of maltodextrin and glucomannan in a microencapsulation agent using the spray drying method.

MATERIALS AND METHOD

Materials

The materials used in this study include sweet potato leaves obtained from sweet potato farmers in Bantul Regency, Indonesia, Glucomannan Konjac (Faculty of Agricultural Technology, Universitas Gadjah Mada), maltodextrin DE 10-12 (Chemipan Co, LTD), Folin-Ciocalteu from 1.1-diphenyl-2-picrylhydrazyl (DPPH), Gallic Acid, sodium carbonate (Na_2CO_3), sodium nitrite ($NaNO_2$), aluminium chloride ($AlCl_3$), Sodium hydroxide (NaOH). All other chemicals were purchased from Sigma-Aldrich (USA) and Merck (Germany).

Preparation of sweet potato leaf extract

The sample preparation process refers to previous research (Ghasemzadeh *et al.*, 2012). The sweet potato leaves were then washed clean to remove dirt on the leaves before further separating the leaves from the stems that are still attached from the harvesting process. After that, the clean leaves were stored in the freezer at -80 ^oC for approximately 48 hours. After the leaves were frozen, the drying process was carried out using a freeze dryer for 48 hours. The dried leaf samples were then mashed and using 80 mesh-sieved. The leaf samples that had been powdered were then stored in the refrigerator in a tight packaging until the next analysis process.

Coating material preparation

The coating materials used in this study were glucomannan Konjac and maltodextrin. Glucomannan with a concentration of 0.5; 0.75; or 1% w/w total yield was dissolved in 50 mL aquabidest at 700 rpm in the heated water at 60-90 ^oC slowly. The second encapsulant material is maltodextrin with a concentration of 10 or 20% w/w total yield dissolved in 200 mL aquabidest. Furthermore, the maltodextrin and glucomannan solutions were mixed to obtain the total solid content (Cilek *et al.*, 2012).

Microencapsulation by spray drying

After the coating sample had been prepared, the next step was the mixing process. Sweet potato extract mixed with a coating solution (1:20 w/w). The sample was then homogenized using a high-speed homogenizer at 4000 rpm for 5 minutes. Furthermore, spray drying is carried out in the Mini Spray Dryer (BÜCHI B-290, BÜCHI Labortechnik AG, Flawil, Switzerland) with an inlet temperature of 120 °C and an outlet of 85-90 °C by adjusting the flow rate conditions (6 mL/min) (Cilek *et al.*, 2012; Sakawulan *et al.*, 2019).

Characterization of Microencapsulation

Total phenol analysis

Analysis of total phenol refers to the research of Ghasemzadeh *et al*. (2012). The total phenol content was carried out by the Folin-Ciocalteu method. Sweet potato leaf extract powder was weighed as much as 0.1 g to obtain a stock sample of 1000 ppm diluted in 100 mL of pro-analysis methanol using a volumetric flask. After that, a sample of 1 ml was taken and diluted in 10 mL of methanol and 1 mL of Folin. After 5 minutes, 20% sodium carbonate was added to the mixture. The solution was incubated in a dark room at room temperature for one hour and then the absorbance was measured at 750 nm using a spectrophotometer UV-Vis (Thermo Scientific Genesys 10S UV-Vis).

Surface phenolic content of capsules

For the determination of surface phenolic compounds of the microcapsule, 0.3 g was treated with 100 mL methanol Pro analysis (3000 ppm). These dispersions were agitated in a Vortex at room temperature for 1 min and filtered (0.45 µm Millipore filter). The amounts of phenolic compounds were quantified by Folin-Ciocalteu (Saénz *et al.*, 2009).

Antioxidant capacity using the DPPH method

The antioxidant activity was carried out using 1.1-diphenyl-2-picrylhydrazyl (DPPH) as a free radical test regarding the method carried out by previous studies (Suárez *et al.*, 2020). 0.1 ml of the extract then mixed with 1.9 mL of DPPH 0.1 mM in methanol PA. The solution was then incubated for 30 minutes, and the absorbance was calculated using a spectrophotometer (Thermo Scientific Genesys 10S UV-Vis) at a wavelength of 517 nm. The antioxidant activity using the DPPH method is then calculated using the following formula:

$$DPPH = \frac{\text{blank absorbance} - \text{sample absorbance}}{\text{blank absorbance}}\ x\ 100\%$$

The extract concentration providing 50% inhibition (IC_{50}) was calculated from the graph of radical scavenging activity percentage against extract concentration.

Encapsulation efficiency

Encapsulation efficiency analysis was carried out based on research (Cilek *et al.*, 2019), which is the ratio of the encapsulated phenolic content minus the phenolics

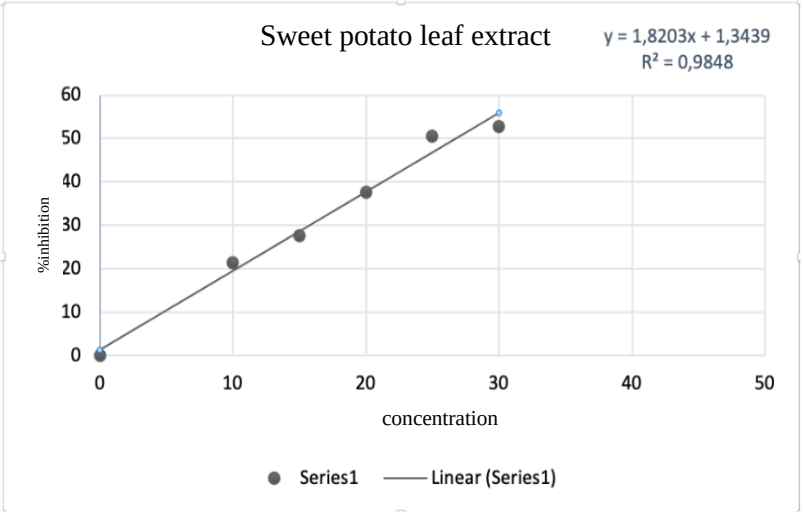

Figure 1. Correlation between % inhibition and concentration of sweet potato leaf extract

outside the encapsulant to the total phenolic content. The total encapsulation efficiency is presented in the following formula:

$$EE\% = \frac{\text{total phenolic compound} - \text{surface phenolic compound}}{\text{Total phenolic compound}} \; x \; 100\%$$

Particle size distribution

Analysis of the particle size distribution of the sweet potato leaf extract encapsulation was measured by a 2000 laser diffraction particle size analyzer (Malvern Instrument Ltd, UK). The encapsulated powder of sweet potato leaf extract was dispersed using aquabidest as a dispersing agent (Santiago-Adame *et al.*, 2015).

Attenuated total reflectance-Fourier transform infrared (ATR–FTIR) spectroscopy

ATR-FTIR (Agilent cary-630) analysis of microencapsulates were recorded at room temperature in the range of wave number from 4000 to 650 cm^{-1}.

Scanning electron microscopy

The morphological analysis of the obtained microparticles was performed by Scanning Electron Microscopy, SEM (Fei Quanta 400 FEG ESEM/EDAX Pegasus X4M). Samples were coated with gold by pulverization under vacuum in a Jeol JFC 100 apparatus and analyzed by SEM for surface structure observation.

Statistical analysis

The independent variables were the Maltodextrin (0.5%; 0,75%; 1% b/b): glucomannan (10%; 20%) ratio. Analysis of variance (ANOVA) was conducted for the determination of differences between treatments by using SPSS Inc., USA. If a significant difference was found, Duncan's multiple comparison test was used for comparison ($p < 0.05$). All the results represent the means of at least two replications

RESULT AND DISCUSSION

Total phenolic content

The results showed that the total phenolic content in sweet potato leaves was 685.06 GAE mg/g of dried extract. This is much higher compared with previous research, sweet potato leaf extract 112.98 mg GAE/g of dried extract, using ethanol as the extraction solvent (Zhang *et al.*, 2019). The results of the total phenolic content are influenced by several things, including the age of the sweet potato leaves used in the study. Padda and Picha (2007) reported that differences in the harvest age of sweet potato leaves used resulted in different total phenolic content. Young sweet potato leaves had a higher phenolic content using chlorogenic acid equivalent (CAE) (87.29 CAE mg/g dw) when compared to mature sweet potato leaves (27.77 CAE mg/g dw). In this study, the sweet potato leaf used was younger age. Sweet potato leaves taken at the top contained higher total phenolic than in the middle or at the bottom. Previous research stated that sweet potato leaf shoots had a higher total phenolic that was 91 CAE mg/g dw, while the middle part was around 70 CAE mg/g dw and the bottom has the lowest phenolic content, which is 60 CAE mg/g dw (Suárez *et al.*, 2020).

Antioxidant activities

The antioxidant activity of sweet potato leaf extract before being given microencapsulation treatment resulted in % RSA (Radical Scavenging Activity) 52.80% at a concentration of 20 ppm sweet potato leaf extract. Based on the inhibitory data (shown in Figure 1), a graph of the relationship between %inhibition and sample concentration was made to calculate the IC_{50} value. The results showed that sweet potato leaf extract had an antioxidant activity with an IC_{50} value of 26.73 ppm, while the gallic acid compound had a lower IC_{50} value of 2.48 ppm. This showed that the gallic acid compound has

greater antioxidant activity when compared to sweet potato leaf extract. Ghasemzadeh *et al.*, (2012) reported that at 200 ppm concentration, the scavenging activity of methanol extract sweet potato leaf (Vardaman variety) reached 62.12%, while other varieties of sweet potato leaf showed different % RSA (49.3% (Beauregard), 41.7% (Jewell), 39.64% (Bush Porto Rico), 36.73% (Georgia Jet) and 32.8% (Centennial). The value of IC_{50} Vardaman variety has the highest (184.3 ppm) while Centennial was the lowest (450.46 ppm). Moreover, the high scavenging activity of sweet potato leaf was further confirmed by the study conducted by Yang *et al.*, (2005) in which among 23 commonly consumed vegetables in Taiwan, sweet potato leaf ranked in the first place with the highest % RSA.

On the other hand, the value of antioxidant activity in encapsulated sweet potato leaf extract samples determined were 51.012% to 66.66%. It is shown in Table 1.

DPPH radical scavenging activity (Table 1) of encapsulated sweet potato leaf extract significantly has a slight difference value. The antioxidant activity using the DPPH method showed the results of % RSA in the range of 50.53-66.84% at a concentration of 100 ppm encapsulated extract powder. Statistically, the results of the antioxidant activity of microencapsulated sweet potato leaf extract showed that the addition of 10% and 20% maltodextrin did not have a significant effect, while in the treatment with the addition of glucomannan, the percent inhibition increased even though the difference was not large, this was indicated by the notation on the average % RSA. It can be seen that antioxidant activity significantly increased with the addition of glucomannan (0.5%; 0.75%; and 1%) compared with control (maltodextrin 10% and 20% without glucomannan). Thus, the result showed that the microencapsulation treatment of sweet potato leaf extract with glucomannan can maintain antioxidant compounds quite well until after the heating process. It can be observed that glucomannan has good characterization as a coating material and good film-forming ability, also it tends to create a fine dense network upon drying (Zhang *et al.*, 2005; Wattanaprasert *et al.*, 2016).

Table 1. Antioxidant activity (% RSA) of microencapsulation of sweet potato leaf extract

Glucomannan (%)	Maltodextrin (%)	
	10	20
0	$51.5 \pm 0.823^{a*}$	$50.53 \pm 0.157^{a*}$
0,5	$65.51 \pm 0.901^{b*}$	$66.84 \pm 0.588^{b*}$
0,75	$65.13 \pm 0.353^{b*}$	$65.15 \pm 1.175^{b*}$
1	$66.48 \pm 0.157^{b*}$	$66.78 \pm 0.431^{b*}$

Data are means ± SD (n = 2). Values within the same letters (line) and (*=maltodextrin) are significantly no different (p > 0.05).

Encapsulation efficiency (EE)

It is known from Table 2 of the encapsulation efficiency that the treatment of 0.75% glucomannan concentration had the highest efficiency encapsulation (89.21%) compared to other glucomannan concentrations (0.5% and 1%). Encapsulation efficiency can indicate the success rate of the encapsulation process. The type of coating material and the amount of core material used as well as spray drying conditions greatly affect the results of encapsulation efficiency (Jafari *et al.*, 2008).

Table 2 shows that the addition of glucomannan concentrations (0.5%; 0.75%; and 1%) gave a significant effect on the encapsulation efficiency of sweet potato leaf extract. The addition of glucomannan concentrations 0.5%, 0.75%, and 1% resulted in an increasing encapsulation efficiency value. The encapsulation efficiency using glucomannan had higher value than without glucomannan. It can be concluded that microencapsulation using glucomannan can protect the effective phenolic compound from external degradation. This is probably because glucomannan has heat-stable characterization (Wang *et al.*, 2010). However, glucomannan 0.75% has significantly no difference from 1% as shown in Table 2. In addition, glucomannan has a fairly high molecular weight so the viscosity of glucomannan is also very high (Yanuriati *et al.*, 2017). The high viscosity will affect the results of encapsulation efficiency, this is due to the disruption of the spray drying process during drying so that it is not able to maintain the active compound properly in the capsule layer. The higher viscosity will also reduce the occurrence of porosity formed in the microcapsules.

Table 2. Encapsulation efficiency (%) of microencapsulated sweet potato leaf extract

Glucomannan (%)	Maltodextrin (%)	
	10	20
0	$80.21 \pm 2.229^{a*}$	$82.73 \pm 1.864^{a*}$
0,5	$86.46 \pm 0.594^{b*}$	$86.8 \pm 0.320^{b*}$
0,75	$88.73 \pm 0.110^{c*}$	$89.96 \pm 2.13^{b*}$
1	$87.89 \pm 0.004^{c*}$	$89.91 \pm 1.943^{b*}$

Data are means ± SD (n = 2). Values within the same letters (line) and * (column) are significantly no different (p > 0.05).

On the other side, it seems that maltodextrin (10% and 20%) significantly did not affect encapsulation efficiency. It is slightly different from the report by Balasuramani *et al.*, (2015) that mention the addition of the maltodextrin concentration can increase the percentage of encapsulation efficiency related to the density of the coating walls during the spray drying process. Moreover, a high percentage of EE was affected by the inlet temperature used. In this study, the inlet temperature used was slightly low (120°C) to decrease the degradation of bioactive compounds. The increase of inlet air

Table 3. Particle size and Pdi index

Sample	Diameter (µm)	Vol (%)	Polydispersity index (Pdi)
Maltodextrin 10%: glucomannan 0,75%	0.296	78.3	0.304
	0.05	17	
	1.9	4.7	
Maltodextrin 10%	0,32	76.4	0.334
	0.076	23.6	

temperature led to a decrease in phenolic compounds (Lourenço *et al.*, 2020).

Particle Size Distribution

Particle size distribution analysis was carried out on the sample with the best efficiency results (Maltodextrin 10% concentration: Glucomannan 0.75%) and the comparison of the control with 10% Maltodextrin concentration without the addition of glucomannan.

Particle size distribution of the microparticles of the encapsulated sweet potato leaf extract were distributed at 0.296 µm (78.3%), 0.05 µm (17%), and 1.9 µm (4.7%) while control is 0,32 µm (76.4%) and 0.076 µm (23.6%).

While the polydispersity index (Pdi) value on the microcapsules resulted in a value of 0.304 which proves that the diversity of particle sizes can be said that have good homogeneity. Pdi is one of the factors that can show the homogeneity of the particle size of a particle, if the Pdi value is close to 0 then the level of homogeneity is high, but if it is more than 0.5 then the particle is said to be heterogeneous (Avadi *et al.*, 2010). While the polydispersity index (Pdi) value on the microcapsules resulted in a value of 0.304 which proves that the diversity of particle sizes can be said that have good homogeneity. Pdi is one of the factors that can show the homogeneity of the particle size of a particle, if the Pdi value is close to 0 then the level of homogeneity is high, but if it is more than 0.5 then the particle is said to be heterogeneous (Avadi *et al.*, 2010).

Attenuated total reflectance-Fourier transform infrared (ATR–FTIR) spectroscopy

ATR-FTIR was determined to verify the interaction of phenolic with polysaccharides and to detect the structural changes in microcapsules due to encapsulation of sweet potato leaf extract within the wall material (Figure 2). Spectra of microencapsulation of a wide band 3400-3300 cm^{-1} were observed that included the contributions of OH stretching vibration of carbohydrates (Paramera *et al.*, 2011).

It was observed that these changes indicated the characteristic belonging to microcapsules: 3425.02-

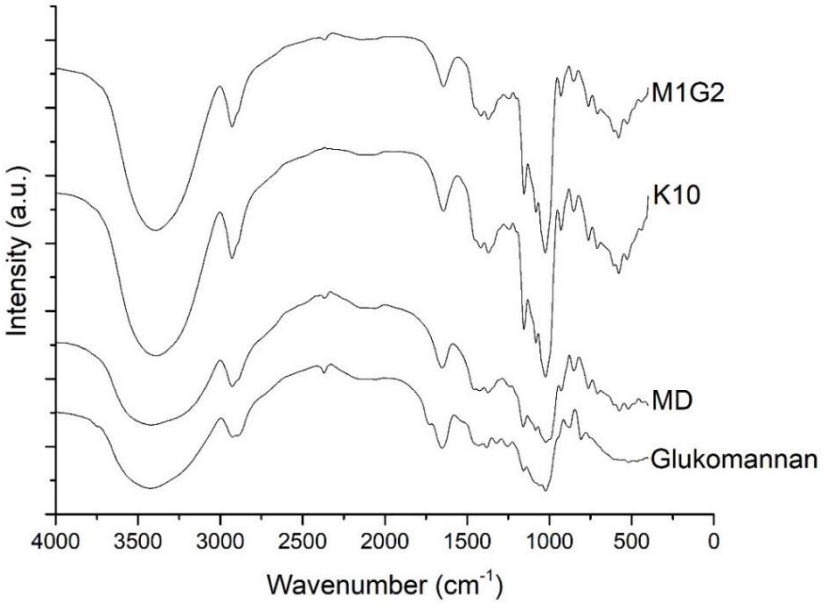

Figure 2. ATR-FTIR glucomannan, Maltodextrin, K10 (Encapsulated extract with 10% maltodextrin without glucomannan), M1G2 (Encapsulated extract with Maltodextrin 10%: Glucomannan 0.75%)

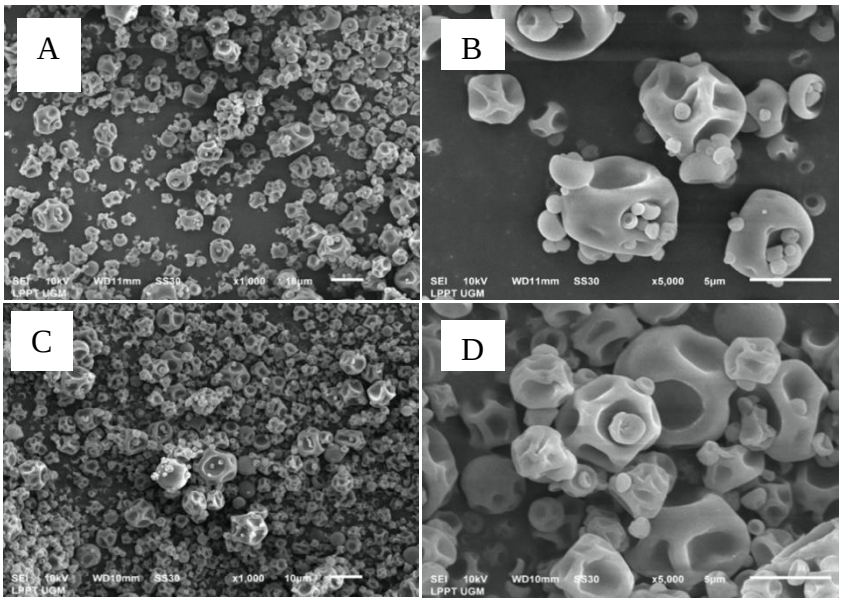

Figure 3. Micrograph of (a) control MD 10% 1000x (b) control MD 10% 5000x (c) glucomannan 0,75%:MD 10% 1000x (d) glucomannan 0,75%:MD 10% 5000x

3386,01 cm^{-1} which refers to O-H stretching vibration, 1642,64-1650,71 cm^{-1} is related to C-O vibration, and 1081,67-1020,91 cm^{-1} corresponds to C-O-C stretching vibration. The results showed that the bands from the encapsulated sweet potato leaf extract slightly shifted which indicated that the sweet potato leaf extract had been coated by matrix (glucomannan and maltodextrin) (Dachriyanus, 2017). Ahmad *et al* (2018) stated that a distinctive band in the microencapsulated extract confirms the incorporation of phenolic in the microparticles capsule.

Scanning Electron Microscope

Scanning electron micrograph of microencapsulation sweet potato leaf extract is shown in Fig. 3. There was no significant difference in the morphology of control and M1G2 treatment, the results showed that in both images there were wrinkles and agglomeration occurred

Agglomeration occurs due to the neutralization charge during the cross-linking process. In addition, the uneven surface is caused by the heating process with the spray dry method which results in a perforated surface, the formation of the basin is caused by shrinkage after inflating the outer surface of the ball followed by the process of expanding the air trapped in the particles, this process is called ballooning (Rigon and Noreña., 2016).

Based on the figure presented, the particle size of the microencapsulated powder was not completely uniform, approximately 78% of the particle size was 0.29 µm and the rest ranged between 1 µm and 0.05 µm, it can be shown by the figure the presence of particles of irregular shapes and different size, while the smaller particles adhere to the surface of larger ones.

CONCLUSION

From the result of this research, it can be concluded that microencapsulation of sweet potato leaf extract encapsulated by glucomannan and maltodextrin can be used as a natural source of antioxidants. Microencapsulation treatment of sweet potato leaf extract with a concentration of 10% maltodextrin combined with 0.75% glucomannan was the best treatment. In the best sample, it is known that the number of uniform particle size distributions is 0.296 µm (volume 78.3), and the Pdi value is 0.304 which indicates that the particle size is homogeneous. The results of phenolic dyes and SEM also showed that encapsulation of sweet potato leaf extract had occurred in the coatings (glucomannan and maltodextrin).

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