

New coating material for producing virgin coconut oil (VCO) microcapsules

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Abstract

The aim of this work was to investigate the microencapsulation efficiency (MEE) of different grades of broken rice (RB) and breadfruit (BB)-based maltodextrin as a coating material, using virgin coconut oil (VCO) as a model system. The VCO was generally found to be well microencapsulated using BB, RB or commercial (COM) maltodextrin at a core/wall material ratio of 1:3. In comparison to a different dextrose equivalent (DE) group, both RB and BB maltodextrins with DE values of 10-14 showed higher MEE values (84.81-94.39%) than maltodextrins with DE value of 15-19 (78.23-79.65%). Low DE value maltodextrins were shown higher glass transition temperatures than high DE value maltodextrins under the same moisture content. Both RB and BB maltodextrins were found to be compatible with COM maltodextrin as shown in the microstructure appearance when viewed with a scanning electron microscope (SEM).

1. Introduction

VCO is high value functional oil that has recently received public attention due to its nutraceutical benefits. Compared with other edible oils, VCO has unique characteristics, such as crystal clarity, a pleasant odor, high resistance to rancidity, a narrow range of melting temperature, easy digestibility and absorbability, and a good potential for high use as spraying oil (Marina *et al.*, 2009). Due to its therapeutic value, the world demand for VCO is rapidly increasing annually. The health benefits of VCO include weight loss, hair and skin care, boosting the immune system, antioxidant activity and maintenance of the cholesterol level (Bawalan and Chapman, 2006). Furthermore, the presence of medium-chain fatty acids in VCO elevates the metabolism and energy expenditure of the body. VCO is directly converted to energy in the liver and is not stored in adipose tissue, which is useful in weight management treatment. According to Nevin and Rajamohan (2012), VCO has a beneficial effect by lowering the level of lipid components in the body. VCO reduced the levels of total cholesterol, triglycerides, phospholipids, low-density lipoprotein (LDL) and very low-density lipoprotein (VLDL) cholesterol and increased the level of high-density lipoprotein (HDL) cholesterol in the serum and

tissues. The shorter chain length and smaller molecules present in VCO compared with those in other edible oils (long-chain triglycerides) allow it to be rapidly absorbed and hydrolyzed in the body (Nayak and Rastogi, 2010). Many investigations of VCO have been conducted, including sensory-evaluation studies (Wang *et al.*, 2000) and research into its antioxidant activities (Phisut, 2012) and chemical properties (Nayak and Rastogi, 2010). A recent study of Khor *et al.* (2014) reported the transformation of VCO into a palatable emulsion to overcome its oily taste and strong aroma, thereby encouraging consumers to enjoy this beneficial oil. One of the natural characteristics of VCO is that it solidifies at a temperature below 24°C, which makes it difficult to consume and store, particularly in cold countries. Therefore, the development of microencapsulated VCO using a spray drying technique would solve this problem without changing the condition or quality of the oil. Many studies of the microencapsulation of fish oil (Heinzelmann *et al.*, 1999), orange oil (Junxia *et al.*, 2011) and sunflower-seed oil (Cerdeira *et al.*, 2007) have been conducted but few studies have investigated techniques for microencapsulating VCO. The aim of this study was to introduce a new approach to microencapsulating VCO for future food applications.

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Microencapsulation is a technology that physically packs sensitive ingredients (liquid droplets or solid particles) inside a protective “wall” material to provide protection to the active ingredients or “core” materials from any cause of deterioration (Cerdeira *et al.*, 2007; Hogan *et al.*, 2011). Essentially, the microencapsulation process consists of two steps; the first step often involves the emulsification of the core material with a dense solution of shell material. The second step is the spray drying of the emulsion into dry powders (Madene *et al.*, 2006). The spray drying approach is the most common method because its cost is approximately 30-50 times lesser than other methods such as freeze drying (Cerdeira *et al.*, 2007). A good wall material is able to encapsulate the core material within the spherical, protect sensitive ingredients against unfavorable ambient conditions, mask or preserve flavors and aromas, reduce the volatility and reactivity and provide attractiveness for the merchandising of food products (Gharsallaoui *et al.*, 2007; Raja *et al.*, 2011).

Maltodextrin is primarily used to encapsulate difficult-to-dry products such as oils, flavors, fruit juices and sweeteners. It is a good coating material because it reduces stickiness, agglomeration, improves storage stability and protects against oxidation (Villarino *et al.*, 2007). Maltodextrin is a product of starch hydrolysate that is produced via the aids of acids or enzyme treatment (Abbas *et al.*, 2010). It contains both simple sugar and polymer of saccharides with lower molecular-weight (Hadnadev *et al.*, 2011). Several chemical, physical and enzymatic modification of starch have been studied to improve their functional properties, allowing a wide range of applications including carriers for synthetic sweeteners, fat replacers, bulking agents, flavor enhancers, high-tech film formers and spray drying aids for flavors, oils and other sensitive ingredients (Cheirsilp *et al.*, 2012; Khor *et al.*, 2014). The demand on maltodextrins in food application is increasing due to their low hygroscopicity, low sweetness and bland taste (Ahn *et al.*, 2008). Over the years, commercial maltodextrin has been widely produced from various starchy sources such as corn, potatoes, sago, tapioca and wheat (Abbas *et al.*, 2010). However, there has been no full spectrum study on the physico-chemical characteristics of the maltodextrin that is produced from BB and RB starchy sources. The differences in the starch structure will influence the physico-chemical properties of maltodextrin that eventually defines its specific functional role in the microencapsulation process. Therefore, the objective of this work is to investigate the MEE of different grades of RB and BB-based maltodextrin as a coating material, using VCO as a model system.

2. Materials and methods

2.1 Preparation of BB and RB maltodextrin

Mature but un-ripened BB and RB were purchased from a local market (Selangor, Malaysia). The starch was prepared at a laboratory scale (Akanbi *et al.*, 2009; Koh *et al.*, 2012). The suspension of starch (20%) was prepared with 40 g starch to 200 mL of distilled water. An amount of 0.04 g calcium chloride was added. The pH was adjusted to 6.5 with sodium hydroxide (0.1 N) before hydrolysis using enzyme Ban 480L. Different dextrose equivalent (DE) value of BB and RB maltodextrin was prepared according to the specific enzymatic hydrolysis process condition (MARDI in-house method). Enzyme inactivation was done by adding hydrochloride acid (0.1 N) to achieve pH value of 3.0 before being transferred to centrifugation to separate clear supernatant from crude maltodextrin under the condition of 4°C, 10,000 rpm for 10 mins. The liquid maltodextrin was spray dried using optimized parameter setting as in previous studies: inlet temperature (170°C); feed flow (9%) and aspirator rate (95%). A total of 10% commercial (COM) maltodextrin (cassava base, DE 10-14) was used as a reference material because it is known to be of high quality (Koh *et al.*, 2012).

2.2 Determination of dextrose equivalent (DE) value

The Lane and Eynon method was used to analyze the DE value of BB and RB maltodextrin samples. Fehling Solutions were standardized against standard dextrose obtained from the Bureau of Standards. To determine the Fehling Factor, 0.5 g anhydrous grams of dextrose was weighed per 200 mL of distilled water and used as the test solution. Fehling Factor was calculated as follow:

$$\text{Fehling Factor} = \frac{(100) \times (\text{mL used in titration}) \times \left(\frac{\text{g dextrose}}{\text{mL}}\right)}{100}$$

A maltodextrin solution (10 g/ 200 mL) with the known concentration of an anhydrous starch basis was prepared. The maltodextrin solution was transferred to a 50 mL burette. To 50 mL of distilled water in a 500 mL Erlenmeyer flask, 5 mL of each Fehling A and Fehling B were added in. The contents of the flask were brought to boil over a hot plate. When the water starts boiling, 2 drops of methylene blue indicator was added and stirred continuously. The titration was completed by adding the maltodextrin solution drop-wise until the blue colour disappears. The volume of maltodextrin solution used was recorded. The DE was calculated based on below stated formula:

$$DE = \frac{(\text{Fehling Factor})}{(\text{g/mL maltodextrin concentration}) \times \text{mL maltodextrin solution}} \times 100$$

2.3 Preparation of microencapsulated VCO powder

The emulsion of 200 mL was prepared at a core/wall material ratio of 1:3. A small amount of gum Arabic (2.0%, w/v) was added together with maltodextrin (15.0%, w/v) and dispersed thoroughly in deionized water with a controlled temperature of 40°C water bath. The VCO (5.0%, w/v) was added drop wise to the continuous stirring phase to form the emulsion. These coarse emulsions were homogenized in a shear homogenizer (Silverson L4R, Buckinghamshire, UK) for 1.5 min at 7,000 rpm to produce a finer emulsion before subjecting it to the spray drying process. The emulsions were spray dried with a mini-spray dryer (model: Büchi B-290, Büchi Labortechnik AG, Switzerland) equipped with a standard 0.7 mm standard diameter nozzle. The inlet and outlet temperatures of the spray dryer were maintained at 170±2°C and 80±2°C, respectively.

2.4 Microencapsulation efficiency in VCO microcapsules

The total oil contents of VCO microcapsules were determined by Lim *et al.* (2012) with minor modifications. Five grams of VCO microcapsules was mixed with 20 mL of water at 50°C in a 250 mL Erlenmeyer flask with a stopper. A total of 15 mL of de-emulsification reagent was then added to the mixture and vortex before leaving it in a 70°C water bath for 6 min. The resulting mixture was then centrifuged at 3,000 x g for 10 min, and the total oil was collected. To prepare the de-emulsification reagent, 10 g of sodium salicylate and 10 g of sodium citrate were dissolved separately in double-distilled water, and the two solutions were mixed together with 18 mL of n-butanol, and brought up to 90 mL with double-distilled water.

The surface oil was measured by adding 200 mL of hexane to 5 g of VCO microcapsules and hand shaken for 1.5 min. The solvent mixture was then passed through filter paper. The surface oil was collected after hexane evaporation. The MEE of different maltodextrin bases produced VCO microcapsules were calculated as follows:

$$\text{MEE\%} = [(\text{total oil} - \text{surface oil}) \times 100] / \text{total oil}$$

2.5 Moisture content

The moisture contents of the VCO microcapsules were determined by following AOAC official methods. Approximately 2 g of VCO microcapsules was placed in an aluminum dish and dried at 40°C

until a constant weight was obtained.

2.6 Differential scanning calorimeter analysis

A differential scanning calorimeter (DSC), (Perkin Elmer DSC7, Waltham, MA, USA) was used to determine the thermal behavior properties of various maltodextrin-based VCO microcapsules. One milligram of the microencapsulated powder was weighed directly into the DSC aluminum sample pan and sealed with a lid. An empty pan with a lid was used as a reference. The purge gas used was dry nitrogen with the flow rate of 20 mL/min. The temperature range was from -20 to 80°C with a heating rate of 10°C/min.

2.7 Scanning electron microscopy

A scanning electron microscope was used to examine the morphology and surface appearance of the VCO microcapsules. The VCO microcapsules samples were attached to a specimen stub with carbon paint. The coated microcapsules were examined in a Hitachi Hi-Tech FE-SEM model SU8000 Series (Schaumburg, IL, USA) at 15.0 kV.

2.8 Particle size analysis

A particle size analyzer was used to determine the particle size distribution of the VCO microcapsules. Each measurement time was set at 12 s, and the background time was 10 s. All the VCO microcapsules samples were sieved at size of <100 µm before being subjected to particle size determination to avoid particle agglomeration from the coverage of surface fat. The particle size distribution of VCO microcapsules was determined by using a Scirocco 2000 dry powder system provided with a Mastersizer 2000 using laser diffraction (Malvern, Worcestershire, UK). Measurements were performed in triplicate and the results were reported as the means.

2.9 Statistical analysis

Data were statistically analyzed by a one-way analysis of variance (SPSS statistics version 16). Significant differences (p<0.01) between means were determined by Duncan's multiple range test.

3. Results and discussion

3.1 Moisture content of microencapsulated VCO powder

Table 1 shows the moisture content of all microencapsulated VCO powders that were coated with different wall materials. In general, there were no significant differences (p<0.01) in the moisture contents between BB, RB and COM maltodextrin

microencapsulated products. The moisture content of each sample was maintained within a range of 4.6 to 5.2%. Similar to a study carried out by Nayak and Rastogi (2010), the microencapsulated powders using maltodextrin of various DE values as a wall material, possessed moisture content ranges between 5.6 to 5.8% with no significant differences between them. The range of moisture content in our study was suitable to yield high microencapsulation efficiency. The ranges of moisture content in our microcapsules were closely related to the study of Gharsalloui *et al.* (2007), which was able to obtain the highest microencapsulation efficiency (84.95%) when the moisture content was 5.7% compared to lower moisture contents (0.9-4.0%) of the wall material. Moisture content is one of the crucial parameters for entrapped oil powders because high moisture content can cause membrane rupture by promoting lipid oxidation (Lim *et al.*, 2012). According to Hogan *et al.* (2001), the type of wall materials used did not affect moisture content of the powder produced. Similar findings were resulted in our studies on VCO microcapsules using different coating materials, as the moisture contents showed no significant differences between them.

Table 1. Wall material matrixes of spray-dried microencapsulated virgin coconut oil (VCO) powder and its moisture content.

Wall materials	DE value	Core/Wall ratio	Moisture Content (%)
BB	10-14	1:3	4.77±0.16 ^a
RB	10-14	1:3	5.16±0.27 ^a
COM	10-14	1:3	5.03±0.09 ^a
BB	15-19	1:3	4.67±0.04 ^a
RB	15-19	1:3	4.68±0.35 ^a

^aData were expressed as mean ± standard of triplicate determinations. Mean values with different superscripts in the same column are significantly different $p < 0.01$

(Abbreviations: BB: Breadfruit Maltodextrin; RB: Broken rice Maltodextrin; COM: Commercial Maltodextrin; DE: Dextrose Equivalents)

3.2 The MEE and particle size distribution of microencapsulated VCO powder

The particle size distribution and MEE of microencapsulated VCO samples that were produced from different wall materials are shown in Table 2. Particle size distribution has a major role in processing, handling, shelf-life and the microstructure, which is related to powder functionality, stability and flow ability (Raja *et al.*, 2011). Based on the results, the type of wall materials with specific DE values were shown to have a significant influence on the MEE of the VCO microcapsules. Maltodextrins consist of 7 mixtures of oligosaccharides standard (DP1

= glucose; DP2 = maltose; DP3 = maltotriose; DP4 = maltotetraose; DP5 = maltopentaose; DP6 = maltohexaose; DP7 = maltoheptaose). RB maltodextrin exhibited a significantly higher degree ($p < 0.01$) of MEE in comparison with BB and COM maltodextrins. However, maltodextrin with high DE values from 15-19 presented a significantly lower degree ($p < 0.01$) of MEE than maltodextrin with low DE values from 10-14. This result can be explained through the composition of oligosaccharide mixtures in different DE of maltodextrin. Maltodextrins with higher DE value (15-19) contained higher amount of smaller molecular structure of oligosaccharides ranging from DP 1 to DP 7, indicating the least amount of longer glucose chain length ($DP > 7$). DP 1-7 was the total oligosaccharides with smaller chain length of sugar molecules (from glucose DP1 up to maltoheptaose DP7), while $DP > 7$ was the glucose molecule chain length longer than maltoheptaose. The characteristics of maltodextrin DE 15-19 resulted in a more viscous solution as it contained higher sugar composition compared to DE 10-14 which resulted in its lower MEE %. Maltodextrin with the same DE values can possess different functional and physico-chemical properties, which is dependent on the starch molecular structure itself (Villa-Vélez *et al.*, 2012). According to a micrograph study conducted by Koh *et al.* (2012), COM starch possessed the largest molecule size compared to RB and BB starches. This statement was confirmed by the significant differences ($p < 0.01$) in the MEE values obtained for RB (94.39%), BB (84.81%) and COM maltodextrins (81.15%) under the same grade of maltodextrin (DE 10-14) when applied as a coating material to produce VCO microcapsules. According to Shiga *et al.* (2004), the surface area of the carrier increased with an increase in the molecular-weight. This finding confirmed that the higher molecular-weight of maltodextrins with the DE value of 10-14 provide better encapsulation property (81.15-94.39%) than lower molecular-weight of maltodextrins with the DE value of 15-19 (78.23-79.65%). Based on our findings, our RB maltodextrin (DE 10-14) was able to achieve higher MEE than other studies which also used maltodextrin as a microencapsulating agent including 84.95% MEE (Gharsallaoui *et al.*, 2007), 42.35% MEE (Calvo *et al.*, 2010), 84.25% MEE (Rosenberg *et al.*, 1990) and 81.1% MEE (Marina *et al.*, 2009). A higher percentage of MEE values indicated better protection of the core material by the wall material (Lim *et al.*, 2012). Among all maltodextrins tested, the VCO microcapsules produced using BB maltodextrin with high DE value of 15-19 exhibited the lowest MEE value, which was 79.65%.

Table 2. Characterization of microencapsulated VCO with different wall materials in terms of their dextrose equivalent value, particle size distribution and microencapsulation efficiency.

Wall materials	DE value	Powder particle size D4,3 (μm)	MEE (%)
BB	10-14	7.21 \pm 0.03 ^a	84.81 \pm 1.2 ^c
RB	10-14	13.38 \pm 0.00 ^b	94.39 \pm 1.40 ^d
COM	10-14	13.44 \pm 0.00 ^b	81.15 \pm 1.21 ^b
BB	15-19	19.46 \pm 2.19 ^b	79.65 \pm 0.52 ^{ab}
RB	15-19	15.90 \pm 0.19 ^c	78.23 \pm 0.39 ^a

^aData were expressed as mean \pm standard of triplicate determinations. Mean values with different superscripts in the same column are significantly different $p < 0.01$

(Abbreviations: BB: Breadfruit Maltodextrin; RB: Broken rice Maltodextrin; COM: Commercial Maltodextrin; DE: Dextrose Equivalents)

The particle size of microencapsulated VCO powder is presented as D4,3 (the volume-weighted mean or volume mean diameter) after spray drying. Wall materials based on RB and BB maltodextrins with DE values of 10-14 showed significantly ($p < 0.01$) smaller particle sizes than other wall materials with DE values of 15-19. Microencapsulated VCO powders coated with RB maltodextrin exhibiting DE values of 10-14, possessed the highest MEE values with a particle size distribution of 13.38 μm . However, VCO microcapsules that were produced with either BB or RB maltodextrin with high DE values of 15-19 tend to form bigger particle sizes with low MEE values. The similar phenomena observed by Ahn *et al.* (2008), confirmed that the particle size of the microcapsules formed had an influence on MEE value of encapsulated powder.

3.3 Morphology study of microencapsulated VCO via SEM

The morphological appearances of various VCO microcapsule products are presented in Figure 1. SEM was performed to assess the MEE by determining the encapsulation ability of the wall materials as the integrity and porosity of the microcapsules (Shiga *et al.*, 2004). This morphological analysis demonstrated size, shape and agglomeration tendency of smaller particles between themselves and forms bigger particles. Although the outer surfaces of VCO microcapsules exhibited irregularities (dents), they showed no pores or cracks on the VCO microcapsules. The presence of these dents has an adverse effect on the flow behavior of microcapsules; however, they did not affect the stability of the encapsulation property (Finotelli *et al.*, 2005).

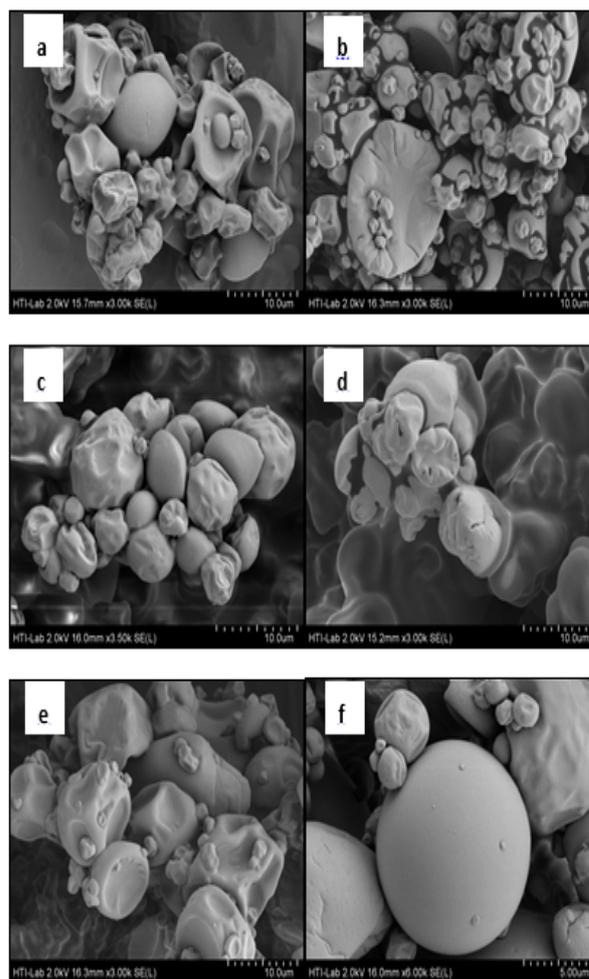


Figure 1. SEM images of microencapsulated VCO with different wall materials. a) BB DE 10-14 (10 μm); b) BB DE 15-19 (10 μm); c) RB DE 10-14 (10 μm); d) RB DE 15-19 (10 μm); e) COM DE 10-14 (10 μm); f) RB DE 10-14 (5.00 μm).

According to SEM observations, the surfaces of VCO microcapsule coatings with a maltodextrin grade DE 10-14 were generally smoother and less dented than VCO microcapsules coated with maltodextrin grade DE 15-19. The microcapsules coated with BB, RB and COM maltodextrins of grade DE 10-14 (Figures 1a, c and e) had spherical and smoother surfaces, whereas the VCO microcapsules produced from either BB or RB maltodextrin with DE values of 15-19 (Figures 1b and d) were found to be partially disrupted and were observed to have some oils on the surface of the microcapsules. In brief, the VCO microcapsules that were coated with RB maltodextrin DE 10-14 wall material exhibited the best morphology (Figures 1c and f) with % MEE of 94.4, confirming that this type of wall material has better functionality in encapsulating properties as opposed to other maltodextrin bases. The differences in the MEE between low and high DE value ranges of maltodextrin were found related to their sugar

compositions. According to Shiga *et al.* (2010), longer chains of glucose polymers (low DE group) increased the retention ability of the microcapsules. This statement was further confirmed with the findings observed by SEM analysis and MEE value of all VCO microcapsule samples.

3.4 The thermal behavior study by DSC

Table 3. The thermal behavior of microencapsulated VCO powder with different wall materials.

Wall materials	DE value	A1(°C)	Delta H (J/g)
BB	10 -14	23.04±0.01 ^c	13.12±0.02 ^a
RB	10 - 14	22.87±0.01 ^b	13.75±0.01 ^c
COM	10 - 14	23.04±0.01 ^c	15.67±0.03 ^c
BB	15 - 19	22.87±0.01 ^b	13.30±0.10 ^b
RB	15 - 19	22.38±0.01 ^a	14.44±0.01 ^d

^aData were expressed as mean ± standard of triplicate determinations. Mean values with different superscripts in the same column are significantly different $p < 0.01$ (Abbreviations: BB: Breadfruit Maltodextrin; RB: Broken rice Maltodextrin; COM: Commercial Maltodextrin; DE: Dextrose Equivalents)

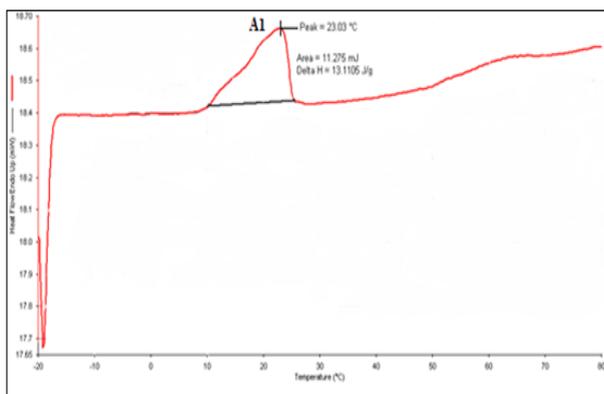


Figure 2. DSC thermogram of microencapsulated VCO powder; A1: Optimum temperature.

The thermodynamic properties of all VCO microcapsule samples are summarized in Table 3 and Figure 2. Figure 2 demonstrates an illustration of the microcapsules thermal behavior. Based on Table 3, the melting point (A1°C) of the higher DE (15-19) maltodextrins were significantly lower ($p < 0.01$) than low DE (10-14) maltodextrins. Maltodextrins DE 15-19 possessed melting points ranged in between 22.38 to 22.87°C, while maltodextrins DE 10-14 possessed higher melting points ranged in between 22.87 to 23.04°C. The low melting point of the dry solids produced sticky products which may stick on the dryer chamber wall during drying, leading to low product yield (Raja *et al.*, 2011). According to Cano-

chauca *et al.* (2005), stickiness may be related to the morphology of the particles (SEM morphology). As shown in Figure 1b and 1d, the microcapsules coated with maltodextrin DE 15-19 demonstrated an amorphous particle surface and the presence of smaller glucose polymer molecular chain length may influence the stickiness appearance and low melting point values.

The significant difference ($p < 0.01$) in the thermal behaviors between maltodextrin DE 10-14 and DE 15-19 were explained by the influences of their molecular weight. The higher molecular weight of maltodextrins (DE 10-14) required a higher temperature and required less enthalpy energy (Delta H) to achieve their optimum melting point (Nevin and Rajamohan, 2008).

4. Conclusions

All broken rice, breadfruit and commercial maltodextrins with the same DE value of 10-14 exhibited better microencapsulation properties than maltodextrins with DE value of 15-19. In summary, broken rice maltodextrin exhibited the highest MEE value, followed by breadfruit and commercial maltodextrins under the same DE group. The findings also showed that both broken rice and breadfruit maltodextrins had better microencapsulating properties than commercial maltodextrins and were further confirmed by observing its microstructure appearance. On top of these, broken rice maltodextrin DE 10-14 possessed the best microencapsulation efficiency relatively to the other maltodextrins. This study provides an opportunity to create value added products and explore the potential usage of maltodextrin from breadfruit and broken rice sources for future applications in food industries.

Conflicts of interest

The authors declare no conflict of interest.

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