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Production and properties of spray dried *Clinacanthus nutans* using modified corn starch as drying agent

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Abstract

Clinacanthus nutans leaves is a medicinal plant with promising therapeutic effect. The objective of this study was to produce *C. nutans* powder using a spray dryer and to evaluate its physicochemical properties, followed by physical observation after 3 months of storage at room temperature (21±1°C). *C. nutans* juice with and without the addition of 5% (w/v) of modified corn starch (MCS) was spray dried at a feed flow rate of 23 rpm, inlet air temperature of 160°C and outlet air temperature of 100°C. Results showed that significantly lower ($p<0.05$) values of water activity, moisture content, flowability, particle size, solubility, wettability and dispersibility times, while significantly higher ($p<0.05$) values of hygroscopicity, bulk and tapped densities were recorded as compared to the spray dried powder without the addition of MCS. These indicated that MCS as drying agent has significantly improved the powder properties and the physical appearance during storage. Furthermore, produced *C. nutans* powder had a significantly higher ($p<0.05$) IC_{50} values, percentage of inhibition and total phenolics content. Also, a strong correlation was observed between total phenolic and antioxidant activity, indicated that encapsulation using MCS successfully protected the thermally sensitive compounds which contributed to high antioxidant activity.

1. Introduction

Tremendous advances have been made in the discovery of many synthetic drugs in recent years, however, the demand for medicinal plants is still high among a vast majority of the world population (Anand *et al*., 2019), due to the increase in health awareness among consumers associated with vegetable consumption (Nor Hasni *et al*., 2020). The World Health Organization (WHO) estimates that around 80% of the world population in developing countries use plant-based products for primary health care (Yahaya *et al*., 2015). Besides, the increasing cost of modern medicines paved the way for rural practitioners to rely upon medicinal plants based on their traditional information.

Clinacanthus nutans or known as 'Daun Belalai Gajah' in Malaysia (Figure 1) has diverse and potential medicinal uses in traditional herbal medicine for treating skin rashes, insects and snake bites, lesions caused by herpes simplex virus, diabetes and gout (Aslam *et al*., 2015). According to Shim *et al*. (2013), varied contents of bioactive compounds have been documented in *C. nutans*, including protocatechuic acid, chlorogenic acid, ferulic acid, caffeic acid, gendarucin A, gendarucin A isomer, 3,3'-Di-O-methylellagic acid, schaftoside, arabinosyl-glucosyl apigenin isomer, ascorbic acid and two isomeric oxoprolinates (Zulkipli *et al*., 2017).

Figure 1. *C. nutans* leaves

However, *C. nutans* is highly perishable which results in a microbial deterioration within a few days after post-harvesting. Worse yet, boiling water during juice preparation degraded the heat-sensitive bioactive compounds. To overcome this problem, spray drying is a suitable approach to encapsulate and stabilize the *C. nutans* juice. Besides extending shelf life through high

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moisture removal rate, this method also reduces the cost of packaging, storage and transportation. However, stickiness is one of the issues in spray drying due to the build-up of powder deposit at the outlet of the spray drying chamber which resulting in possible blockage, downtime and loss of product. Another problem which may arise is the spray drying being unfavourable to heat-sensitive compounds due to high thermal treatment. Thus, the drying agent is commonly employed during the spray drying process to facilitate powder production.

During the spray drying process, drying agent or also known as wall material, coating the bioactive compounds with the technique called encapsulation. The goals of encapsulation are to entrap and protect the bioactive compounds from nutrient loss or oxidation reactions and to hide sensory characteristics that are not appreciated by consumers. Other than that, the drying agent can increase the glass transition temperature of the product, thereby reduce stickiness and wall deposition during the spray-drying process, producing small solid particles (1–500 µm in size) (Amaral *et al*., 2019).

Starch is a polysaccharide carbohydrate consisting of a large number of glucose units joined together by glycosidic bonds, comprising of amylose and amylopectin as its macromolecules (Noranizan, 2002). Modified starches have been developed for a very long time and its applications in the food industry are really significant nowadays. Currently, there are no studies reported on the spray dried *C. nutans* using MCS as a drying agent. Hence, the aim of this study is to determine the effect of MCS on properties of spray dried *C. nutans*, followed by the physical observation of powder after 3 months of storage at room temperature.

2. Materials and methods

2.1 Sample preparation of Clinacanthus nutans juice

C. nutans Lindau or Sabah Snake Grass leaves were freshly harvested from a commercial farm. Only the top stems were removed from the mother plants. The leaves were hand-plucked from the stems and washed twice under running tap water and tossed. The juice was extracted using a juicer (Phillips, Malaysia) with a fixed ratio, 1:3 of water to leaf. The obtained juice was filtered and utilized right away for spray drying. MCS (Brown and Polson, Malaysia) was purchased from a local supplier.

2.2 Preparation of Clinacanthus nutans powder by spray drying

The drying was performed using a spray dryer (Büchi Mini Spray Dryer B-290, Switzerland), a typical laboratory-size model spray dryer at inlet/outlet air temperatures (160°C/100°C) respectively, as according to Koç and Dirim (2018). The control of outlet air temperature was regulated by adjusting the feed flow rate at 23 rpm. The obtained powder was stored at ambient temperature (21±1°C) in a sealed polythene bag under dry and dark conditions for further analysis of powder properties.

2.3 Moisture content and water activity of Clinacanthus nutans powder

The moisture content of the powder was measured using moisture analyser (MX-50, Tokyo, Japan) whereas the water activity was measured using Water Activity Meter (Aqualab, Washington, USA).

2.4 Bulk density

The bulk density of *C. nutans* powder was measured using a graduated measuring cylinder. Approximately 100 g of the sample was passed through a sieve with apertures greater than or equal to 1.0 mm to break up agglomerates that may have formed during storage. The sample was gently introduced into a dry graduated measuring cylinder of 250 mL, carefully levelling the powder without compacting. The bulk density (ρ) was calculated using the following formula:

$$\rho = \frac{m}{V}\left(\frac{g}{mL}\right)$$

Where m is the weight of *C. nutans* powder and V is the unsettled apparent volume (Etti *et al*., 2016).

2.5 Tapped density

The tapped density of *C. nutans* powder was obtained by mechanically tapping the graduated measuring cylinder containing the sample. After observing the initial powder volume or mass, the measuring cylinder was mechanically tapped, and volume or mass readings were taken until little further volume or mass change is observed. The mechanical tapping was achieved by lifting the cylinder and allowing it to drop, under its mass and a specified distance. The tapped density in (g/mL) was calculated using the same formula m/V_t, as bulk density (Etti *et al*., 2016).

2.6 Flowability and cohesiveness

The flowability and cohesiveness of *C. nutans* powder were evaluated in terms of Carr index (CI) and Hausner ratio (HR) respectively (Jinapong *et al*., 2008). Both CI and HR were calculated from the bulk and tapped densities of the powder as shown in equations below.

$$CI = \frac{\rho tapped - \rho bulk}{\rho tapped} \times 100$$

$$HR = \frac{\rho tapped}{\rho bulk}$$

2.7 Solubility

The solubility of *C. nutans* powder was determined by adding 5 g of the sample to 150 mL of distilled water at room temperature (25±1°C) in a 400 mL beaker. the mixture was immediately stirred using a magnetic stirrer at 1000 rpm to assure systematic stirring, meanwhile the time taken for complete solubility was measured using a stopwatch (Elfalleh *et al*., 2009).

2.8 Wettability

The wettability of *C. nutans* powder was determined by weighing the sample and transferring it into a funnel that is positioned on top of a beaker containing 100 mL of deionized water. A pestle was placed inside the funnel to block the lower opening. The time is monitored when the pestle was lifted to allow the powder to drop onto the water surface. The time taken until all the powder is visually wetted was recorded (Szulc and Lenart, 2016).

2.9 Dispersibility

The dispersibility of *C. nutans* powder was determined by mixing the sample with water at room temperature (21±1°C) in a beaker until there were no visible lumps at the bottom of the beaker. The time taken to disperse the powder was measured using a stopwatch (Szulc and Lenart, 2016).

2.10 Hygroscopicity

The hygroscopicity of *C. nutans* powder was determined by placing about 1 g of sample in a desiccator with a saturated solution of sodium chloride (NaCl). After one week, the sample was weighed and the hygroscopicity was expressed as the amount of adsorbed moisture per 100 g of sample (g/100 g) (Cai and Corke, 2000).

2.11 Total phenolic content

The total phenolic content of *C. nutans* powder was determined according to the Folin-Ciocalteu procedure. Different concentrations (50, 100, 150, 250 and 500 mg/mL) of spray dried *C. nutans* with (5% MCS) and without drying agent (control) were prepared in ethanol. For each concentration, 2 mL was added into test tubes contained 1.0 mL of Folin-Ciocalteu's reagent and 0.8 mL of sodium carbonate (7.5%). The tubes were mixed and allowed to stand for 30 min. Absorption at 765 nm was measured using spectrophotometer (Thermo Electron Corporation, USA). The total phenolic content was then expressed as gallic acid equivalents (GAE) in milligrams per gram dry material (Kamath *et al*., 2015).

2.12 Total antioxidant activity

The total antioxidant activity of *C. nutans* powder was determined according to 2, 2- Diphenyl-1-picrylhydrazyl (DPPH) assay. 0.1 mM of DPPH solution was prepared in methanol. Different concentrations (4, 8, 12, 16 and 20 mg/mL) of spray dried *C. nutans* with (5% MCS) and without drying agent (control) were prepared in ethanol. For each concentration, 1 mL of sample was added into 1 mL of DPPH solution. Control was prepared with an equal volume of methanol and DPPH without adding the sample. The mixture in the test tubes was shaken vigorously and left to stand in the dark for incubation for 30 mins at room temperature. Then, absorbance was measured at 517 nm using a spectrophotometer (Thermo Electron Corporation, USA) against methanol as blank. Ascorbic acid at various concentrations was used as the standard reference (Basumatary *et al*., 2015). The percentage of DPPH inhibition was calculated using the following equation:

$$\%\ of\ inhibition = \frac{Ac\ - As}{Ac} \times 100$$

where *Ac* is the absorbance value of the control and *As* is the absorbance value of the added test samples solution.

2.13 Particle size of the powder

The Malvern Panalytical Mastersizer 2000 (Worcestershire, UK) laser diffraction testing instrument was used to evaluate the particle size of the powder. Sample powder was passed through a laser beam resulting in the laser light scattered at a wide range of angles. The detectors measured the intensity of light scattered at that position. A mathematical model (Mie or Fraunhoffer Theory) was applied to generate the particle size of the powder. The final result was reported on an equivalent spherical diameter volume basis.

2.14 Visual observation of powder's physical appearance

The powder obtained was sealed in a polyethylene bag and stored in the dark and dry condition at room temperature (21±1°C) for 3 months. The powder was kept for a storage period of 3 months as any prolonged storage would result in undesirable effects in the quality of the powder. The powder was then visually observed for any formation of clumps or caking of the powder before and after storage.

2.15 Statistical analysis

All statistical analyses were conducted by one-way analysis of variance (ANOVA). Tukey's multiple range tests were employed with a probability of $p<0.05$. The linear Pearson correlation was also used to evaluate correlations between total phenolics and antioxidant activity. The software for statistical analysis was Minitab V.16 (Minitab Inc., State College, Pennsylvania, USA).

3. Results and discussion

The initial total soluble solid content of the *C. nutans* juice was found to be 7.0°Brix prior to the addition of 5% MCS as a drying agent. Initially, about 2.5% and 5% of MCS were used in the juice respectively prior to spray drying in order to determine the percentage of MCS that is able to produce high yield and good quality of the powder. It was found that when 2.5% of MCS used, the *C. nutans* powder produced was quite sticky and clumps were also observed in the powder. Thus, the concentration of MCS was increased to 5% which resulted in powder with good flowability. The total soluble solid content of the juice increased to 10.0°Brix after the addition of 5% MCS.

3.1 Moisture content and water activity of powder

The moisture content of the powder should be <10% and water activity in the range of 0.1-0.4 depending on the product type. Water activity is defined as the vapor pressure of water in a sample divided by the vapor pressure of pure water at the sample temperature (Nielsen *et al*., 2012). Water activity measures the availability of free water in a food system that is responsible for biochemical reactions during storage. Therefore, this property provides valuable information about microbial spoilage, chemical stability, and physical stability of the *C. nutans* powder. Moisture content is another important powder property, which is related to the drying efficiency. In addition, moisture content provides information about the texture of the powder since increasing levels of moisture provide water mobility. Water activity and moisture content together provide a complete moisture analysis.

Based on the result obtained in Table 1, the water activity and moisture content of spray dried *C. nutans* (control) were found to be 0.47% and 7.31% respectively. The addition of MCS resulted in a significant decrease ($p<0.05$) in the water activity and moisture content of *C. nutans* powder, which were 0.34% and 4.18%, respectively. The addition of MCS has increased the total solid content in the feed as shown by an increase from 7.0°Brix to 10.0°Brix and thus, reducing the moisture content of the powder. Apart from an increase in inlet air temperature, 160°C and decrease in outlet air temperature, 100°C, the addition of high molecular weight additives such as MCS to the extract before atomizing is widely used as an alternative way to increase glass transition temperature, Tg of powder (Truong *et al*., 2005). Food products generally formed in a method of rapid heat exchange environment and are not at thermodynamic equilibrium. Depending on their water activity level and molecular weight, they will undergo the shift from a glassy crystallizing temperature to rubbery state matter (Lee *et al*., 2018). In this research, the nature of high molecular weight of the carbohydrate additive helped to increase the glass transition temperature of the spray drying process. Increasing the glass transition temperature contribute to powder stability, reduce caking and stickiness problem. These results showed that MCS was a useful drying aid in the spray drying process of *C. nutans* extract and as a result it has improved the yield of powder.

Table 1. The properties of spray dried *C. nutans* powder with and without the addition of MCS

Powder properties	0% MCS (Control)	5% MCS
Water activity (a_w)	0.47±0.04^a	0.34±0.01^b
Moisture content (%)	7.31±0.58^a	4.18±0.07^b
Bulk density (kg/m^3)	500.07±0.21^b	595.82±0.55^a
Tapped density (kg/m^3)	556.18±0.63^b	618.19±0.22^a
Flowability (CI)	14.61±0.03^a	11.92±0.12^b
Cohesiveness (HR)	1.26±0.04^a	1.11±0.04^a
Solubility (s)	20.33±1.53^a	15.33±0.58^b
Wettability (s)	147.00±0.58^a	106.00±2.65^b
Dispersibility (s)	34.33±0.58^a	31.67±0.58^b
Hygroscopicity (g/100 g)	15.39±0.03^b	19.84±0.30^a
Particle size (μm)	613.376^a	383.664^b

MCS: Modified corn starch. Results are expressed as mean±standard deviation (n=3). Values with the same superscript in each column are not significantly different ($p>0.05$).

3.2 Bulk density and tapped density of powder

The physical characteristics of powders are not independent, including those of food powders. For example, a change in the distribution of particle size can result in a simultaneous change in bulk density, flowability, and appearance (Micha, 2009). Bulk density includes the volumes of the solid and liquid materials, all pores and is generally used to characterize a final product obtained by drying (Kurozawa *et al*., 2009). The bulk density of powders is determined by particle density, which in turn is determined by solid density and particle internal porosity, and also by spatial arrangement of the particles in the container.

The bulk density and tapped density of spray dried *C. nutans* (control) were found to be 500.07 kg/m^3 and 556.18 kg/m^3 respectively. The increase in the drying air temperature usually causes a decrease in the apparent density since there is a greater tendency to hollow particle formation (Walton, 2000). In this research, the inlet drying air temperature was 160°C, and the outlet drying air temperature was 100°C. Besides, the moisture content of the control *C. nutans* powder is higher than that of *C. nutans* powder with MCS. The high moisture content of powder causes their gathering into larger aggregations, which causes more empty voids between particles, resulting in a reduction of bulk density (Janiszewska *et al*., 2008).

However, with addition of MCS as drying agent, there was a significant increase in the bulk density and tapped density of the *C. nutans* powder ($p<0.05$), which were 595.82 kg/m³ and 618.19 kg/m³ respectively. The higher the initial concentration of the feed solution, the greater the values of apparent density of powders (Janiszewska and Witrowa-Rajchert, 2007). Thus, apart from the low moisture content of the *C. nutans* powder with MCS, the high concentration of the feed solution prior to spray drying also contributed to the increased densities of the powder. A higher bulk density is associated with better powder flow (Niazi, 2013). Tapped density is another important factor related to packaging, transport, and commercialization of powder thus, this value can be useful in terms of weight and amount of material that will fit into a container (Finney *et al*., 2002). A high-density dry product can be stored in smaller containers in comparison with a low-density product. The heavier material accommodates itself more easily in the spaces among the particles, resulting in higher density (Tonon *et al*., 2010).

3.3 Flowability and cohesiveness of powder

Flow characteristic of powder is judged by the free flowing, semi-free flowing, and cohesive flow. Cohesive flow is considered as the more severe resistance to flow. Both flowability and cohesiveness are significant quality attributes for the manufacturers and end users for packaging, measuring, handling, transportation, bag filling, emptying storage, dosing and mixing (Koç and Dirim, 2017). The flowability and cohesiveness properties of the *C. nutans* powder were evaluated in terms of CI and HR.

Based on the results obtained, both flowability and cohesiveness of spray dried *C. nutans* (control) were 14.61 and 1.26 respectively. On the contrary, the addition of 5% MCS as drying agent to the *C. nutans* powder resulted in the flowability and cohesiveness of 11.92 and 1.11, respectively. Thus, the addition of MCS has significantly decreased the value of the two properties ($p<0.05$). Both flowability and cohesiveness of sample powder were lower than that of control powder, probably due to the lower moisture content of the sample powder in which MCS was added as drying agent. Factors which influence the flow behavior of powder include, particle size, particle distribution, morphology, hygroscopic nature, drying aid and moisture content of material (Seth *et al*., 2017).

According to Jinapong *et al*. (2008), the classification of the powder flowability and cohesiveness based on the CI and HR are very good (<15), good (15-20), fair (20-35), bad (35-45) and very bad (>45), and low (<1.2), intermediate (1.2 – 1.4) and high (>1.4), respectively. The lower the flowability and cohesiveness, the powder will be less cohesive and more able to flow freely. Food powders obtained by spray drying might have some problems with their properties, such as solubility, flowability and cohesiveness, due to the presence of low molecular weight chemicals which have a low glass transition temperature (Jittanit *et al*., 2010). They can therefore stick on the dryer chamber wall during spray drying, resulting in low product yield and operational issues. These issues can be fixed before being atomized by adding certain drying agents with high molecular weight such as carbohydrate additives, to the feed.

3.4 Solubility time of powder

Food powders constitute a wide range of different powder components in their chemical and physical composition. Solubility, a main determinant of powder quality, is a significant variable in assessing wettability and dispersibility of powder in aqueous solution. The raw materials and additives used, as well as the characteristics of the powder (humidity content and particle size), affect the solubility index of spray dried powder.

The solubility time of spray dried *C. nutans* (control) was 20.33 s. However, there was a significant decrease ($p<0.05$) in the solubility time of the *C. nutans* powder with MCS, 15.33 s. This may be due to the type of carrier agent added as there is variance in powder solubility. Starch is generally difficult to solubilize in water, however modified starch and maltodextrin are more soluble in water. This explains the short time required to solubilize MCS in water, 11.33 s. Literature reviews have stated that the functionality of carbohydrates contribute to the increase of solubility of powder with increase of drying agent used. During rapid heat exchange environment in the drying process, crust formation occurs where the least soluble substance starts to precipitate and form crust at the droplet surface (Lee *et al*., 2018). The formed crust is mainly constituted of MCS that is highly soluble in nature. This explains why the solubility time of *C. nutans* powder with MCS as drying agent is shorter than that of the control powder.

3.5 Wettability time of powder

Wettability is the ability of powder to overcome the surface tension between themselves and water or the time required to make the powder wet completely. In order to attain high wettability values, high porosity or large pores for bigger particles are desirable (Koç and Dirim, 2017). Besides the effects of physical properties, the chemical composition of the powder also influences wettability depending on the content of fats, proteins, and carbohydrates on their surfaces.

According to the result obtained, the wettability time of spray dried *C. nutans* (control) was 147 s. However, there was a significant decrease ($p<0.05$) in the wettability time of the *C. nutans* powder with MCS, 106 s. In the present study, the wettability time was found to have a direct relationship with the solubility time. This is due to the increase in the dissolution of powder components that required less time to wet their surface. Moreover, MCS as drying agent prevented the particle agglomeration of spray dried powder, thus shortened the wettability time. Also, fast dissolution is appeared to be associated with the lower moisture content, which are in line with the study by Goula and Adamopoulos (2004) in the spray drying of tomato pulp. It is explained that powder with low moisture content was less sticky and can be associated with rapid rehydration (Goula and Adamopoulos, 2008).

3.6 Dispersibility time of powder

The term dispersibility refers to the dispersion of agglomerates with little stirring. It describes the ease with which the powder may be distributed as single particles over the surface and its ability to be wetted without formation of dry lumps in the reconstituting water. Clump formation reduces dispersibility and can be enhanced if the sink ability is high (Kothakota *et al.*, 2014). Based on the result obtained, the dispersibility time of *C. nutans* powder with 5% MCS was 31.67 s, significantly shorter ($p<0.05$) than that of control *C. nutans* powder which was 34.33 s. Additives have been reported to reduce the interfacial tension between particles and the dispersion media, resulting in enhanced level of dispersibility and shorter dispersibility time in sample powder. Besides, the absence of additives in the control feed produces spray dried powder with reduced particle size. When the size of the particles is small, the adhesive forces between them becomes larger, resulting in poorer flow properties and lower dispersibility in water (Sakurai *et al.*, 2017). Variations in spray drying process parameters and additives type, concentration and dispersant has shown potential improvements in functional and reconstitution properties such as solubility, wettability and dispersibility during food formulations (Yousefi *et al.*, 2011).

3.7 Hygroscopicity of powder

Water adsorption measured as hygroscopicity is a critical physical property in powder products as the presence of water influences the deterioration of vitamins, phenolic compounds and other powder properties such as flowability. Hygroscopicity of *C. nutans* powder with 5% MCS was 19.84 g/100 g, significantly higher ($p<0.05$) than that of control *C. nutans* powder, 15.39 g/100 g. The significant increase in the hygroscopicity of the sample powder was probably due to its low moisture content. It was found that the low moisture spray-dried powder has the greater capacity to absorb water from the surrounding air and hence is more hygroscopic (Tonon *et al.*, 2010). The lower the powder moisture content, the higher their hygroscopicity, the greater their capacity to adsorb ambient moisture, which is related to the greater water concentration gradient between the product and the moist air (Bringas-Lantigua *et al.*, 2016). During spray drying, the rapid removal of moisture in the sample powder with MCS results in either a fully amorphous product or some microcrystalline areas dispersed in the amorphous mass. The amorphous form is a meta-stable non-equilibrium. Amorphous products also show high degree of hygroscopicity, contributing to the hygroscopicity of powder (Kothakota *et al.*, 2014).

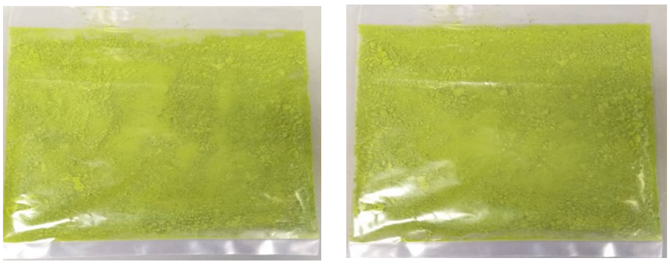
3.8 Particle size of powder

The reconstitution and flow properties of the powder depends on the particle size. Different processing parameters and feed characteristics can affect the distribution of particle size. By an increase in the inlet air temperature, particles became larger sized because of the greater swelling of particles (Tonon *et al.*, 2008). The inlet temperature used in this spray drying process was 160°C, higher than the outlet temperature, 100°C. As the inlet air temperature increases, the drying rate increases as well. Because of this, larger-sized particles are produced, due to the early formation of a structure, and would prevent them from shrinking during the drying process (Tonon *et al.*, 2008).

Based on the results, the particle size of control powder was 613.376 μm, whereas that of the sample powder was 383.664 μm. The size of particle formed during spray drying is strongly related to feed viscosity, as higher liquid viscosity, the larger the droplets formed during atomization produces larger particles obtained during spray drying (Jinapong *et al.*, 2008). The initial concentration of the control feed before spray drying process was 7.0°Brix, which increased to 10.0°Brix following the addition of 5% MCS as drying agent. In this research, although there was an increase in the concentration of the feed, the particle size of the sample powder produced was small compared to that of control powder. This may be due to the high moisture content of the control powder without MCS. The powder with higher moisture content tends to have a higher bulking weight, caused by the presence of water, which is very denser than the dry solid. This hardened skin prevents the moisture from exiting out of the droplet, and therefore, the particle size increases (Kalantari *et al*., 2018).

3.9 Physical appearance of powder before and after 3 months of storage at room temperature

The sample powder, spray dried with MCS was stored in a sealed polyethylene bag under dry and dark conditions at room temperature (21±1°C) for 3 months. The sample powder was stored in such a way that consumers would store at home. Based on the result obtained, clump formation can be seen in the powder in Figure 2.



Before storage After storage

Figure 2. The physical appearance of spray dried *C. nutans* with 5% modified corn starch before and after 3 months of storage at room temperature.

Initially, after exiting the spray dryer, there were no clumps seen and the powder did not appear to be sticky. However, the clump formation observed after the storage period must be due to increasing moisture content of the powder as a result of exposure to the humidity of the environmental conditions during storage (Samborska *et al*., 2015). The powder particles must have absorbed moisture from the environment. In addition, the moisture absorption by the particles during storage may develop stickiness and cake-like structure which can prevent wetting easily (Koç and Dirim, 2017).

3.10 Total antioxidant activity

The ethanolic extract of *C. nutans* leaves was reported to exhibit antioxidant activity and protective effect against free radical-induced hemolysis (Pannangpetch *et al*., 2007). Based on Table 2, the antioxidant activity of spray dried *C. nutans* were found to be concentration-dependent. The antioxidant activities observed can be attributed to either the different mechanisms exhibited by different polyphenolic compounds that are, tocopherols, flavonoids and other organic acids and to the synergistic effects of different compounds. Studies have shown that many polyphenols make a significant contribution to antioxidant activity and act as extremely efficient free radical scavengers, primarily owing to their redox properties. They play a significant role in adsorbing and neutralizing free radicals, quenching singlet and triplet oxygen or decomposing peroxides (Seth *et al*., 2017). The phenolics, sulfur-containing glucosides, sulfur-containing compounds, chlorophyll derivatives, and some phytosterol derivatives that are isolated from *C. nutans* leaves using solvent might be the contributors to the antioxidant activity exhibited (Khoo *et al*., 2018).

Table 2. Effect of different concentrations of ascorbic acid, spray dried *C. nutans* powder with and without the addition of MCS on the percentage of inhibition and IC_{50} values

Concentration (mg/mL)	% of Inhibition		
	Ascorbic acid	0% MCS (Control)	5% MCS
4	15.89 ± 0.79^{a}	11.26 ± 0.56^{b}	10.6 ± 0.53^{b}
8	27.15 ± 1.36^{a}	13.91 ± 0.70^{b}	14.57 ± 0.73^{b}
12	56.95 ± 2.85^{a}	20.53 ± 1.03^{c}	25.17 ± 1.26^{b}
16	66.23 ± 3.31^{a}	35.76 ± 1.79^{c}	48.34 ± 2.42^{b}
20	96.03 ± 4.80^{a}	50.99 ± 2.55^{c}	64.9 ± 3.25^{b}
	IC_{50} values (mg/mL)		
	11.51 ± 0.58^{c}	21.28 ± 1.06^{a}	16.86 ± 0.84^{b}

MCS: Modified corn starch. Results are expressed as mean±standard deviation (n=3). Values with the same superscript in each column are not significantly different ($p>0.05$).

Standard ascorbic acid showed the highest DPPH radical scavenging activity, expressed as the concentration of a sample required to decrease DPPH absorbance by 50% (IC_{50} value). Moreover, the percentage of inhibition of *C. nutans* spray dried with MCS was found to be significantly ($p<0.05$) higher than that of control at the concentrations of 8 mg/mL and above. Also, the calculated IC_{50} value was significantly ($p<0.05$) lower for *C. nutans* spray dried with MCS as compared to the control sample. It showed better antioxidant potential when compared to control that was spray dried without any drying agent. This result could be explained by the fact that in the sample powder, MCS created a strong impermeable film around the droplets that helps protect the phytochemical compounds from hot drying air. The result showed that the entrapment of the phytochemical compounds in the presence of a drying agent helped prevent some compounds loss during spray drying. Besides, the high spray-drying temperature may associate with the activation of some phenolic compounds, resulting in an increase of antioxidant activity of the powder (Tran and Nguyen, 2018).

3.11 Total phenolic content

C. nutans are commonly rich in phenolic compounds such as flavonoids, phenolic acids, tannins, coumarins, and lignins (Kosai *et al*., 2016). Quantification was done based on the standard curve of the gallic acid and expressed as gallic acid equivalent (GAE) using the linear equation based. Numerous previous findings have reported the influence of factors such as sample preparation and processing, as well as the extraction procedures on the phenolics yield (Ji *et al*., 2012).

Based on Table 3, the total phenolic content of *C. nutans* spray dried with 5% MCS is significantly ($p<0.05$) higher than that of control at all concentrations which indicates higher phenolic content found in the sample powder. This result may possibly be due to the bound phenolic compounds disengaging from the cellular constituents and forming free phenolic compounds in the powder (Chang *et al*., 2006). In addition, the extract spray dried with MCS and resulting in low moisture content can cause greater cell disruption and rupture, which may result in more phenolic compounds being released. According to the result obtained, it can be concluded that spray drying performed with the addition of a drying agent has a significant effect on the total phenolic content of the powder. Also, a strong positive correlation with $R^2 = 0.8527$ ($p<0.05$) was detected between total phenolics and antioxidant activity, suggesting that spray drying with MCS was successfully produced *C. nutans* powder with less degradation of the heat-sensitive bioactive compounds and its antioxidant activity.

Table 3. Effect of different concentrations of spray dried *C. nutans* powder with and without the addition of MCS on the total phenolic content

Concentration (mg/mL)	Total Phenolic Content (mg GAE/g)	
	0% MCS (Control)	5% MCS
50	18.17 ± 0.91^b	26.5 ± 1.33^a
100	73.17 ± 3.66^b	101.5 ± 5.08^a
150	119.83 ± 5.99^b	154.83 ± 7.74^a
200	164.83 ± 8.24^b	191.5 ± 9.58^a
500	196.5 ± 9.83^b	233.17 ± 11.66^a

MCS: Modified corn starch. Results are expressed as mean±standard deviation (n=3). Values with the same superscript in each column are not significantly different ($p>0.05$).

4. Conclusion

The results in this study indicated that MCS was a useful drying aid to produce high yield and good quality of *C. nutans* powder. Moreover, the addition of 5% MCS has significantly improved the powder properties of *C. nutans* in terms of water activity, moisture content, flowability, solubility, wettability, dispersibility times and particle size compared to powder without the addition of MCS. Also, heat-sensitive compounds with high antioxidant activity were well protected through encapsulation using MCS as a drying agent. The physical appearance was still good after 3 months of storage at room temperature, however, there is a tendency for clump formation. Therefore, the application of modified atmosphere packaging (MAP) by removing the oxygen and replacing with the inert gas is recommended in order to control the humidity and maintain the *C. nutans* powder for a longer time.

Conflict of interest

The authors declare no conflict of interest.

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