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Microencapsulation of Phenolic Compounds from Waste Mango Seed Kernel Extract by Spray Drying Technology

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ABSTRACT

Mango seed kernel, a byproduct of the mango processing industry, is known to be a rich source of phenolic compounds. Phenolic compounds are bioactive in nature and highly valuable due to a number of potential health and therapeutic benefits making it a good component in functional food formulations and for the nutraceutical industry. Through spray drying, the bioactive fraction from an industrial waste mango seed kernel extract was recovered through encapsulation using maltodextrin (MD), gum Arabic (GA), and starch (ST) as encapsulating agents. The encapsulating agent type used alongside inlet drying air temperature was varied and observed to have an influence on encapsulation yield and on the powder qualities measured such as total phenolic content, antioxidant activity, moisture content, bulk density and water solubility index. Among the encapsulating agents used, MD was found to be the most desirable encapsulating material with regards to the desirable properties of spray-dried powders in terms of functionality and applicability.

INTRODUCTION

Mango (*Magnifera indica* L.) is a tropical fruit abundant in the country. It comes third to bananas and pineapples as major fruit exports in the Philippines (Briones 2013). Aside from its nutritional value as fruit, it is industrially processed into puree, slices in syrup, nectar, pickles, canned slices and chutney. During the processing of mango, the by-product waste generated includes peels and seeds of the fruit. Several studies revealed that mango peels and seeds are potential sources of valuable bioactive compounds making these fruit waste a promising target for commercial valorization (Jahurul et al. 2014).

Mango seed kernels contain 6.7% crude protein, 12.3% total lipid, 2.7% crude fibre, 2.5% ash and 8.5% moisture. Moreover, it also contains high levels of amino acids and phenolic compounds (112 mg/100 g dry powder) (Abdalla et al. 2006). In another study, the proximate analysis of the mango seed kernel showed 6.57% moisture, 1.46% ash, 8.15% fat, 7.76% protein, 0.26% crude fibre, and the total phenolic content and scavenging activity were 23.90 mg gallic acid per g of dried sample and 95.07%, respectively (Ashoush & Gadallah 2010).

Among the different high value components of mango seed kernel, phenolic compounds were given regard for its potential health benefits and its antioxidant properties (Fang & Bhandari 2010). These compounds were found to have inhibited the mTOR (mechanistic target of rampamycin), a pathway responsible for age-degenerative diseases (Jakobek 2015). In a different study, biomarkers for CVD (cardiovascular diseases) such as total cholesterol and low-density lipoprotein cholesterol, were reported to have decreased as a result of a polyphenol-rich diet (Xie et al. 2017).

Phytochemicals present in the mango seed kernel can be co-extracted through screw pressing (Rombaut et al. 2014, Rabadan et al. 2018). After the extraction of phenolic compounds, its preservation is given importance since the presence of water in the recovered extract causes high water activity, which can be related to faster degradation of the bioactive components as a consequence of faster biochemical reactions (Quek et al. 2007). Therefore, extracts with high phenolic content can suffer from severe browning, loss of nutritional value and antioxidant activity during storage under ambient conditions (Fang & Bhandari 2010). In order to preserve the quality of such bioactive compounds, suitable environmental conditions must be met with and drying is a viable process that can be done in order to lengthen their storage life at ambient conditions making them available and usable as food. To address such an issue,

spray drying has been the prevailing technique employed in the pharmaceutical, food and flavour industry for it has shown advantages over other drying techniques like conventional air drying, freeze-drying and spouted bed drying (Patel et al. 2014, Chaul et al. 2017).

Spray drying is a microencapsulation technique involving a three-step unit operation, atomization, dehydration and powder collection, that transforms a feed solution or suspension from liquid to a dried powder state which is appropriate for heat sensitive materials like phenolic compounds (Gharsallaoui et al. 2007, Sansone et al. 2011, Chaul et al. 2017). During drying, the feed is atomized into droplets, which are then dried almost instantaneously through contact with a drying medium at an elevated temperature (Patel et al. 2014, Chaul et al. 2017). Powders formed after spray drying have superior benefits and economic potential over their liquid forms such as ease in handling, translating to supply and logistical advantages, and most especially a reduction in biological degradation prolonging shelf life (Fang & Bhandari 2010).

The type of encapsulating material affects the physicochemical properties of powders and target bioactive retention and recovery (Bayram et al. 2010, Sun-Waterhouse et al. 2013, Mishra et al. 2014). Among the different types of encapsulating materials, maltodextrins are commonly used (Sansone et al. 2011). A study by Mishra et al. (2014) observed phenolic contents of 150-350 mg gallic acid equivalence per gram of amla (*Emblica officinalis)* juice powder using maltodextrin as encapsulating agent. Gums, such as gum Arabic have also been widely used in encapsulation due to its effectivity in retaining and stabilizing phenolic compounds (Tonon et al. 2010, Ravichandran et al. 2012, Ballesteros et al. 2017). Starches, on the other hand, have also seen increased utilization in food ingredients due to its bland taste, it is generally recognized as safe (GRAS), cheap and non-allergenic (Zhu 2017). Encapsulation of gallic acid using starch systems through spray drying resulted in bioactive release patterns, greater than nine hours, suitable for the design of functional foods (Robert et al. 2012).

This study investigated the usage of an industrial waste stream of mango seed kernel extract as a potential source of phenolic compounds, and the recovery of such phytochemicals through spray drying by using maltodextrin, gum Arabic, and soluble starch as carriers or encapsulating agents. The effect of the type of encapsulating agent used, carrier concentration as observed by using maltodextrin, and inlet drying air temperature on powder yield, as well as, on the quality of the powder through its relevant physicochemical properties such as total phenolic content, antioxidant activity, bulk density, moisture content, and water solubility index were studied.

MATERIALS AND METHODS

Mango seed kernel extract (MSKE) was obtained from Green Enviro Management Systems, Inc. (Bankal, Lapu-Lapu City, Cebu, Philippines), which processes byproducts from the local mango processing industries and converts them into high-value products (Hlaing et al. 2015). MSKE was generated from the screw pressing of steeped mango seed kernels from washing to rid the MSK with water. Absolute ethanol 99.5% (RCI Labscan, Thailand); chemical reagents: folinciocalteau phenol reagent, 2,2-diphenyl-1-picrylhydrazyl, Sigma-Aldrich, St. Louis, MO; sodium carbonate 99.5% (Scharlau, Spain), and reference standards: 6-hyhydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Sigma-Aldrich, St. Louis, MO), and gallic acid (Scharlau, Spain) were of analytical grade and obtained from local suppliers. Encapsulating agents, maltodextrin, gum Arabic and soluble starch were of technical grade and sourced from local suppliers.

Feed storage and preparation: About 5 litres of MSKE was obtained 24 hours before spray drying. Upon acquisition, MSKE was filtered using qualitative filter paper (Whatman, United Kingdom) and was stored in glass amber containers at 4°C for further use. The total phenolic content and total solids of the extract were determined before spray drying. An hour before the experiment, MSKE was left to stand at ambient conditions until room temperature (25°C) was achieved. Appropriate proportions of encapsulating agent (EA) and MSKE were mixed until dissolution/dispersion to produce appropriate EA concentrations.

Spray drying operation: Solutions of MSKE with appropriate EA concentrations and termed feed extract were fed by means of a peristaltic pump where the flow rate was changed by varying the rotational speed. A Yamato DL410 lab scale spray dryer (Yamato Sci. Co. Ltd, Japan) with a cocurrent drying mode of operation, 457 mm (diameter), 975 mm (height) drying chamber dimensions, and a two-fluid nozzle system with a 0.7 mm orifice diameter was used in the production of powder samples by spray drying. A 3^2 full factorial design was used where inlet drying air temperature (T_i) was varied at 3 levels (130, 150 and 170°C) and 3 types of encapsulating agents, maltodextrin (MD), gum Arabic (GA), and soluble starch (ST), were used at a working concentration of 10% (w/w). The effect of encapsulating agent concentration, using MD, was also investigated at 3 levels (10, 15 and 20% w/w). Drying air flow rate (0.6 m^3/hr), atomizing pressure (0.1 MN/m^2), feed flow rate (25 $g.min^{-1}$) and feed temperature (25°C) were maintained throughout the duration of the experiment. Outlet temperature was moni-

tored and recorded during operation. After spray drying, mango seed kernel extract powder (MSKEP) was obtained in a pre-weighed product collecting vessel and the total mass was weighed. The efficiency of the process was assessed by the encapsulation yield (%EY) as described in the work of Shu et al. (2006) and was calculated using Eq. 1.

$$encapsulation\ yield\ (\%) = \frac{TPC_P \times W_p}{TPC_F \times V_F} \times 100 \quad \ldots(1)$$

Where, W_p is the weight of powdered product in grams, V_F is the feed volume in mL, and TPC_P and TPC_F are the total phenolic contents of MSKE powder in mg gallic acid equivalents (GAE) per gram of MSKEP and feed extract in mg GAE per mL of feed extract, respectively. Final product obtained during the experiment was stored in a desiccator for further analyses. The quality of the product was assessed by quantifying the total phenolic content (TPC), antioxidant activity quantified using DPPH radical scavenging capacity (AOAc), moisture content (%MC), bulk powder density (P_B) and water solubility index (WSI).

Characterization and analyses of spray-dried mango seed kernel extract powder (MSKEP): A method by Vieira et al. (2011) using folin-ciocalteau phenol reagent with modifications was used to quantify total phenolic content (TPC). Samples for analysis were prepared by dissolving about 50 mg powder in 50 mL distilled water. The sample solution was mixed thoroughly for about 2 minutes using an LSE vortex mixer (Corning, NY) and filtered by vacuum using a Whatman 42 filter paper (Whatman, UK). An aliquot (1 mL) was taken from the filtrate, diluted up to 7 mL and mixed well. Afterwards, 0.5 mL of concentrated folin-ciocalteau phenol reagent was added and the solution was left to react for 5 minutes. Saturated sodium carbonate solution (1.5 mL) was added to the solution and the contents were made up to 10 mL with distilled water. The resulting mixture was incubated for 2 hours at 30°C and the absorbance was read at 765 nm using a SpectroquantPharo 100 spectrophotometer (Merck, Kenilworth, NJ). Gallic acid was used as standard and result were expressed as mg gallic acid equivalence (GAE) per gram of MSKEP for the powdered MSKE samples and mg gallic acid equivalence (GAE) per mL of extract for the feed extract.

The DPPH assay by Vieira et al. (2011) with modifications was used to quantify the total antioxidant activity. Ethanol-DPPH (0.1 mM) solution was prepared fresh daily. Ethanol-DPPH solution (2.9 mL) was pipetted in 10-mL reaction tubes and the initial absorbance ($A_{initial}$) was read at 515 nm using a spectrophotometer. Shortly after, 100 µL of dissolved sample (as prepared in total phenolic content assay) was added, mixed thoroughly and incubated in the dark for 30 minutes. The final absorbance (A_{final}) was read after incubation and the reduction of DPPH radical was calculated using Eq. 2. Trolox (6-hyhydroxy-2,5,7,8-tetramethy lchroman-2-carboxylic acid) was used as standard and antioxidant activity was expressed as mmol Trolox equivalence per 100 g MSKEP

$$DPPH\ reduction\ (\%) = \left[100 - \left(\frac{A_{initial}}{A_{final}}\right)\right] \times 100 \quad \ldots(2)$$

A method from AOAC (2000) was used for moisture content determination. About 1 g of powder samples (W_i) were weighed in pre-dried and weighed crucibles (W_c). Samples were then dried at 105°C in a Memmert UN75 natural convection oven (MemmertGmBH, Germany) until constant weight (W_F). The moisture content was then calculated with Eq. 3:

$$moisture\ content\ (\%, w/w) = \frac{W_i - W_F}{W_i - W_C} \times 100 \quad \ldots(3)$$

A method by Goula & Adamopoulos (2004) was used to determine the bulk density of the powder. The powder sample was freely flowed and weighed (W_p) until reaching the 100 mL mark in a 100 mL graduated cylinder. The bulk density was determined by weighing the sample as it was transferred. Both bulk densities were calculated using Eq. 4.

$$bulk\ density\ (g/cm^3) = \frac{W_P}{V_P} \quad \ldots(4)$$

Where, W_p is the powder weight and V_p is the volume occupied by the powder.

Water solubility index (WSI) was determined using a modified method by Jafari et al. (2017). About 1 g of spray dried powder was dissolved/dispersed in 30 mL distilled water. The resulting mixture was heated in a water bath at 37°C for 30 min and centrifuged at 11,000 rpm for 6 min. The supernatant was decanted into pre-weighed evaporating dishes and dried at 105°C until constant weight. WSI was calculated using Eq. 5.

$$WSI\ (\%) = \frac{dried\ supernatant\ weight}{supernatant\ weight} \times 100 \quad \ldots(5)$$

Statistical analysis: All data were analysed using the *R* statistical environment and its corresponding base packages. Analysis of variance (ANOVA) was used to test for significance for investigated variables and a Tukey's-HSD (Honest Significant Difference) test was used for post-hoc analysis. Duplicates were made for all process conditions exam-

ined and triplicates were done for each analysis. Results were reported as means with standard deviations.

RESULTS AND DISCUSSION

Total phenolic content and antioxidant activity of spray dried extract: The effect of the inlet air drying temperature and encapsulant type on the total phenolic content (TPC) and antioxidant activity (AOAc) of the mango seed kernel extract powder (MSKEP) produced were investigated at a constant encapsulating agent concentration of 10% (w/w) for the three carriers used: maltodextrin (MD), gum Arabic (GA) and soluble starch (ST), as shown in Fig. 1. The presence and content of phenolic compounds and other antioxidants serve as a metric of its potential usability as a nutraceutical or functional food ingredient. Spray drying of the MSKE with such process conditions produced MSKEP with TPC values ranging from 77-127 mg per g of MSKEP and AOAc values ranging from 86,740 to 217,700 mmol Trolox per 100 g of MSKEP.

An analysis of variance (ANOVA) revealed that the encapsulating agent (EA) type, inlet drying air temperature (T_i) and the corresponding EA to T_i (EA T_i) interaction had a significant effect ($p < 0.05$) on the TPC of the powder and its corresponding AOAc. A detailed assessment of these effects using Tukey-HSD test revealed that inlet air drying temperature in the experimental range (130-170°C) used in the study did not have a significant effect ($p > 0.05$) on both TPC and AOAc for MD and GA microencapsulated powders. These results were found to be in contrast with several studies which observed a decrease in bioactive content in MD microencapsulated extracts as inlet temperature was increased (Goula & Adamopoulos 2004, Shu et al. 2006, Kha et al. 2010, Fazaeli et al. 2012).

The observed trend in this study may have been due to process conditions that were not enough to enact a significant change in bioactive content when using MD and GA. This can be seen in the higher outlet temperatures used in literature which ranged from 83°C to 125°C (Tonon et al. 2008, Kha et al. 2010) as compared to outlet temperatures ranging from 64-80°C observed in this work. A higher outlet temperature, in a spray-drying perspective, usually denotes a high residence time of the product within the vessel at constant inlet temperature due to a lower feed rate and a higher drying air rate which induces flow recirculation within the vessel (Goula & Adamopoulos 2005). This lengthy contact with heat increases the extent of product degradation and will result to higher losses of thermally sensitive bioactive compounds. In contrast, a lower outlet temperature indicates a lower residence time, giving lesser contact time with heat and thus a lower extent of product degradation.

This observation, also denotes the relative bioactive stability when using MD and GA compared to ST as encapsulating agents. ST microencapsulated MSKE powders exhibited a decrease in TP and AOAc as inlet temperature increased. This result could be due to the nature of the phenolic compounds present in the extract which affects its interaction with the encapsulant (Robert et al. 2010). The phenolic fraction of mango seed kernel is typically abundant with tannins and phenolic acids (Masibo & He 2008). This would consequently affect the diffusion rate of phenolic compounds in the droplet matrix, which may result to its enrichment on the droplet surface and lead to an increased susceptibility to heat and oxidation.

In this study, antioxidant activity was measured through the capacity of the microencapsulated MSKE powders to inhibit the DPPH radical. Fig. 1b shows the effect of inlet temperature and encapsulating agent on the antioxidant activity of the MSKEPs after spray drying. It was noted that the inlet drying air temperature only had a significant effect on the AOAc of the MSKEP when ST was used as encapsulant. This observation was similar to that with TPC since most of its antioxidant capacity may be attributed to the phenolic compounds present in the extract. A Pearson's correlation test was done between TPC and AOAc values and it was found out that both values were moderately correlated ($\rho = 0.638$). This result was in contrast with reported studies that noted strong positive correlations between TPC and AOAc (Kha et al. 2010, Mishra et al. 2014). Results suggest that the different encapsulating agents may have recovered different fractions of compounds exhibiting antioxidant properties. Moreover, total phenolic content does not account for all antioxidants and that compounds exhibit a synergistic antioxidant effect which is likely dependent on its chemical structure (Djeridane et al. 2006, Georgetti et al. 2008).

The effect of encapsulating agent concentration on TPC and AOAc as observed by varying MD concentration is shown in Fig. 2. Increasing concentration from 10% to 20% significantly affected and had a negative effect on both TPC and AOAc. With the feed volume being constant, an increase in MD addition would lead to dilution of the total bioactive compound content and consequently, a decrease in its efficacy. Results were in agreement with other studies where MD concentration had an inversely proportional effect on total phenolic content (Kha et al. 2010, Fazaeli et al. 2012).

Encapsulation yield of the spray dried extract: Bioactive retention during spray drying is affected by factors such as the affinity of the target compound to the encapsulating material and environmental factors such as temperature, oxi-

dation and light (Goula & Adamopoulos 2004, Ravichandran et al. 2012, Ballesteros et al. 2017). As given in Table 1, only the encapsulating material type had a significant effect (p <0.01) on the encapsulation yield (%EY). Inlet drying air temperature did not have a significant effect possibly due to the benign process conditions relative to the amount of encapsulating material used which may have prevented the degradation of the bioactive fraction. A study by Garofulic et al. (2017), on the encapsulation of sour cherry juice with varying inlet temperatures and encapsulating materials, only observed a significant effect of inlet temperature when encapsulant-to-core ratio was low.

Among the encapsulating materials used, as shown in Fig. 3, the use of MD resulted in the highest encapsulation yield, while GA and ST had more or less similar values. This observation may infer that the bioactive fraction of MSKE may have had a strong affinity to MD. This inference is confirmed in literature involving the spray drying of polyphenol-rich extracts where it was also observed that MD provided the highest encapsulation efficiency among other alternative carriers (Ballesteros et al. 2017, Garofulic et al. 2017). On the other hand, the similarity in encapsulation yield of GA and ST may be attributed to the hydrophobicity of both materials. GA contains 2% protein linked covalently to its carbohydrate chains which is responsible for its hydrophobicity (Dickinson 2003). While, unmodified ST is relatively less soluble in water-rich extract. These hydrophobic properties respectively mentioned for GA and ST may have been responsible for their inability to encapsulate bioactive compounds and instead acted as a drying aid only (Tonon et al. 2010). This consequently led to lower retentions as it led to an increase in exposure of the phenolic compounds to environmental factors such as heat, oxidation and light.

An increase in MD concentration did not have a significant effect (p >0.05) on the polyphenol encapsulation efficiency (%EY), as seen in Fig. 4. This observation denotes that a proportional amount of phenolic compounds were encapsulated as MD concentration increased. A similar result was observed by Ortiz-Basurto et al. (2017) in the retention of anthocyanins from *Eugenia uniflora* L. juice using MD as encapsulating material. This result may have been due to the relative stability of the droplet formation within the range of MD concentrations used during spray drying. In a study by Shu et al. (2006) on the spray drying of lycopene, the increase of the wall-to-core ratio from 1/4 (25% MD) to

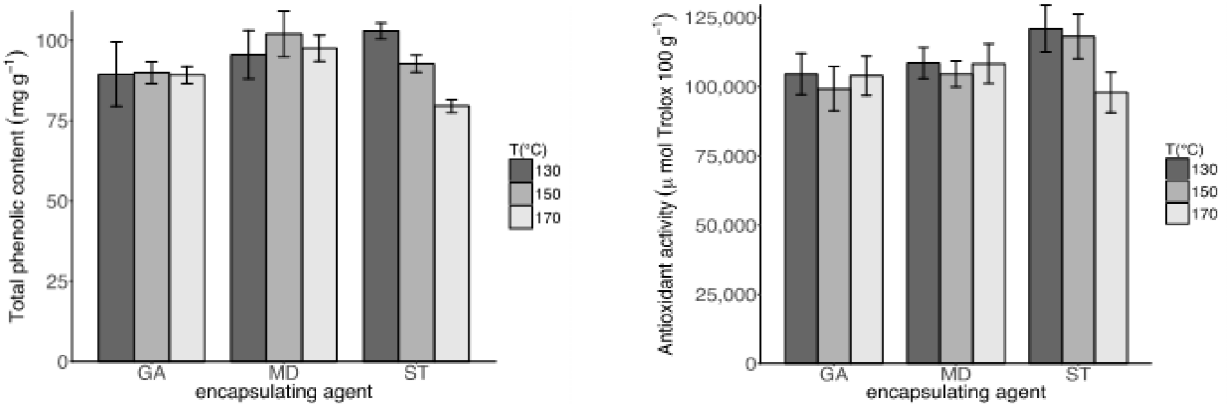
Table 1: Analysis of variance (p-values) of investigated responses in the spray drying of MSKE.

	Term			
	EA	T_i	$EA \times T_i$	% wt. (MD)
Encapsulation yield	<0.0001	0.5501	0.1772	0.0607
Total polyphenol content	0.0001	0.0007	<0.0001	<0.0001
Antioxidant activity	0.0034	0.0055	0.0004	<0.0001
Moisture content	<0.0001	<0.0001	<0.0001	0.0111
Bulk density	<0.0001	<0.0001	0.1318	0.0018
WSI	<0.0001	0.6103	0.0155	0.3550

Table 2: Physical properties of spray dried mango seed kernel powders (MSKEP).

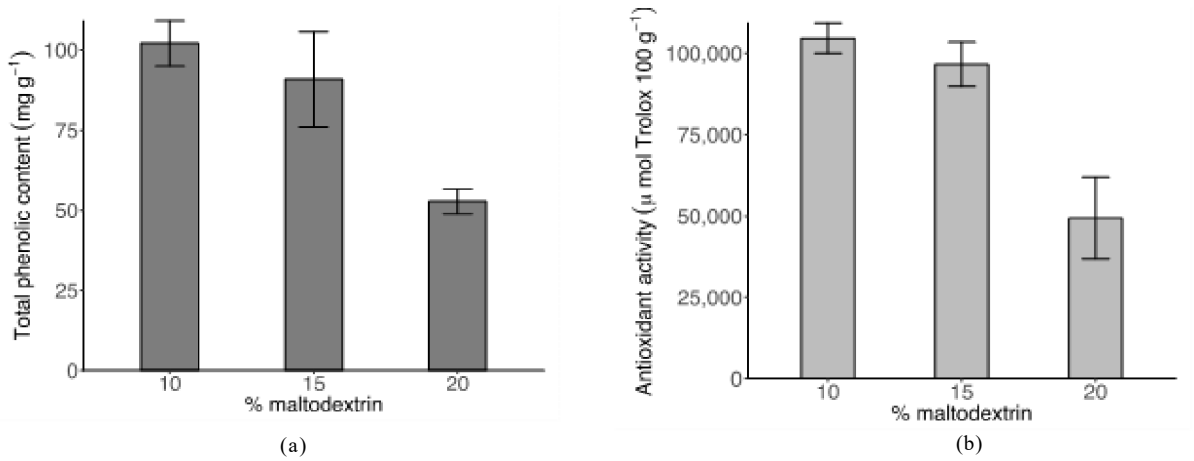
Encapsulating agent		Moisture content (%)	Bulk density (g.cm^{-3})	Water solubility index (%)
10% MD	130°C	4.00 ± 0.47^a	0.32 ± 0.01	92.23 ± 1.59
	150°C	3.17 ± 0.15^b	0.32 ± 0.02	88.75 ± 3.33
	170°C	2.79 ± 0.05^c	0.30 ± 0.02	94.94 ± 2.42
10% GA	130°C	3..65 ± 0.28^a	0.37 ± 0.02^a	84.25 ± 2.81
	150°C	2.68± 0.29^b	0.38 ± 0.01^a	88.28 ± 1.68
	170°C	2.74 ± 0.14^b	0.31 ± 0.01^b	88.14 ± 1.34
10% ST	130°C	5.80 ± 0.06	0.43 ± 0.02^a	38.19 ± 4.32
	150°C	5.83 ± 0.34	0.42± 0.01^a	33.01 ± 1.31
	170°C	5.54 ± 0.20	0.38 ± 0.06^b	34.71 ± 6.47
MD (T_i = 150°C)	10%	3.17 ± 0.15a	0.32 ± 0.02a	88.75 ± 3.33
	15%	3.54 ± 0.26b	0.34± 0.04b	90.96 ± 2.13
	20%	3.98 ± 0.80b	0.36 ± 0.02b	90.08 ± 0.36

Means with different superscripts (a-c) indicate parameter significance (p< 0.05).



(a) (b)

Fig. 1: Influence of inlet drying air temperature and encapsulating agent on (a) total phenolic content and (b) antioxidant activity MSKEP.



(a) (b)

Fig. 2: Influence of maltodextrin concentration on (a) total phenolic content and (b) antioxidant activity of mango seed kernel extract powders

1/2 (50% MD) decreased the encapsulation yield. This was due to the delamination and instability of the emulsion at this ratio.

Physical properties of spray dried extract: The moisture content of powders influences important aspects on its usage such as its shelf-life, usability and functionality (Arogba 1999, Chiou & Langrish 2007). As given in Table 2, increasing inlet drying temperature for 10% MD and GA microencapsulated mango seed kernel extracts had a significant effect (p <0.05) on its moisture content. A faster drying rate was expected when inlet temperature was increased due to a higher temperature gradient between the drying medium and the feed droplets resulting to a lower moisture content. Numerous studies reported similar trends regarding the effect of inlet temperature on the moisture content of spray-dried powders (Goula & Adamopoulos 2005, Tonon et al. 2008, Kha et al. 2010, Tonon et al. 2010). On the other hand, MSKE encapsulated with 10% w/w ST exhibited no significant difference on moisture content as the inlet drying temperature was increased. This could be related to the influence of the chemical structure of starch on the equilibrium moisture content achieved during spray drying. With the given residence time, drying may have reached equilibrium at the experimental temperature range resulting to comparable moisture contents for ST microencapsulated extracts.

The encapsulating material used during spray drying also had a significant effect (p <0.01) on the moisture content. This is clearly shown in Fig. 5a where ST microencapsulated extracts had the highest moisture content compared to MD and GA in all inlet drying air temperature settings. This may be explained by the difference in physicochemical properties of each encapsulating agent which would likely affect the achievable moisture content

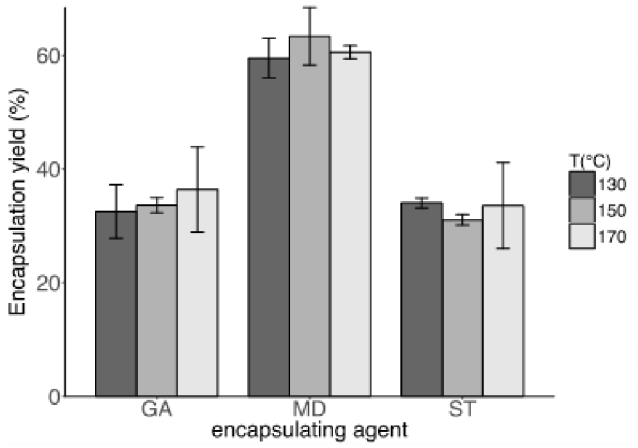


Fig. 3: Influence of inlet drying air temperature and encapsulating agent type on the encapsulation yield.

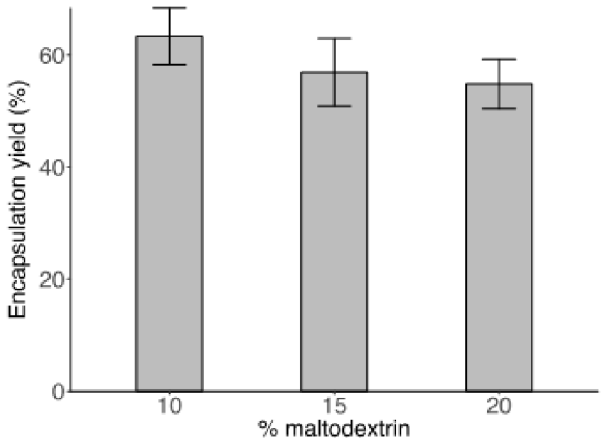


Fig. 4: Influence of maltodextrin concentration on the encapsulation yield.

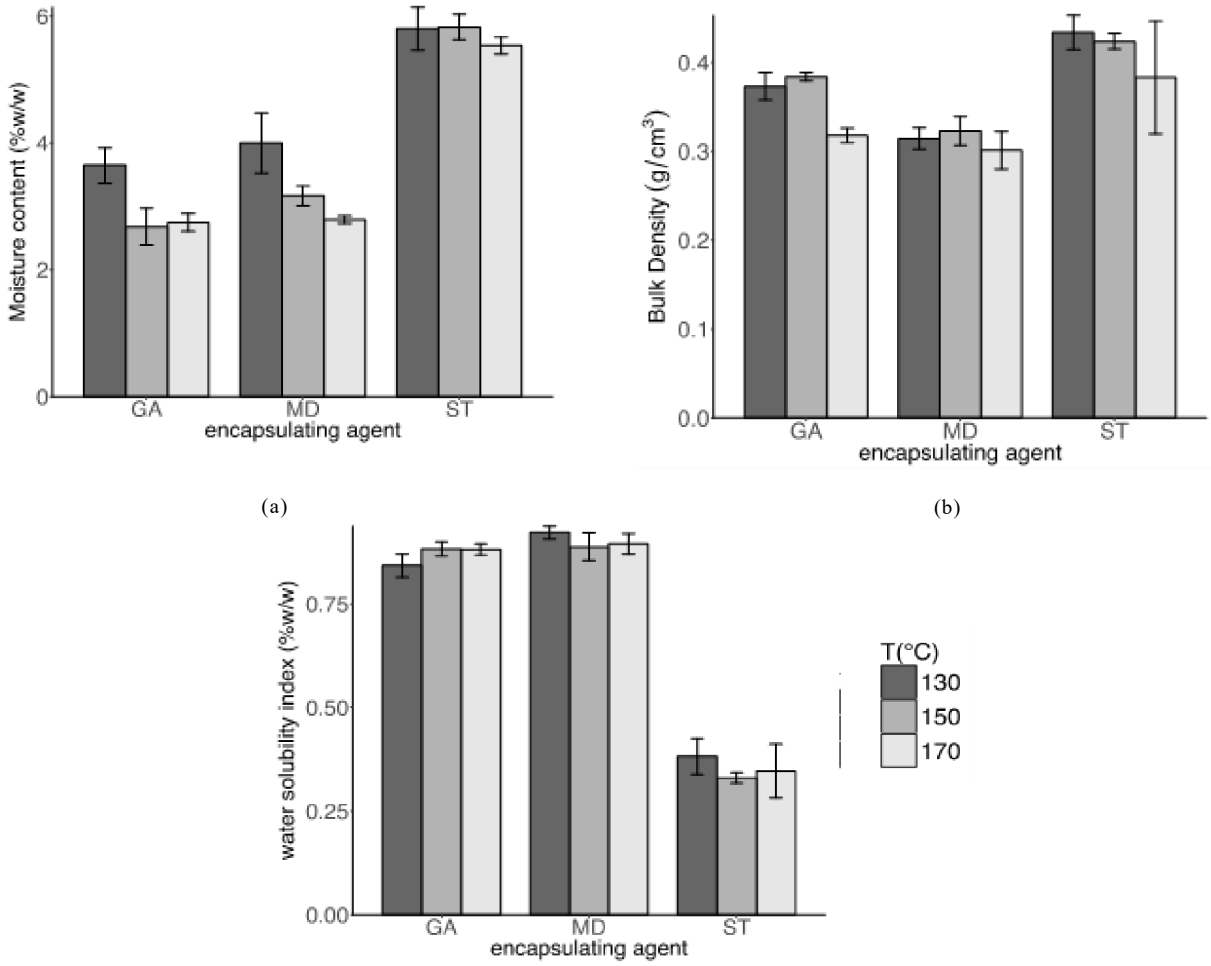


Fig. 5: Influence of encapsulating agent and inlet drying air temperature on moisture content (a), bulk density (b), and water solubility index (c).

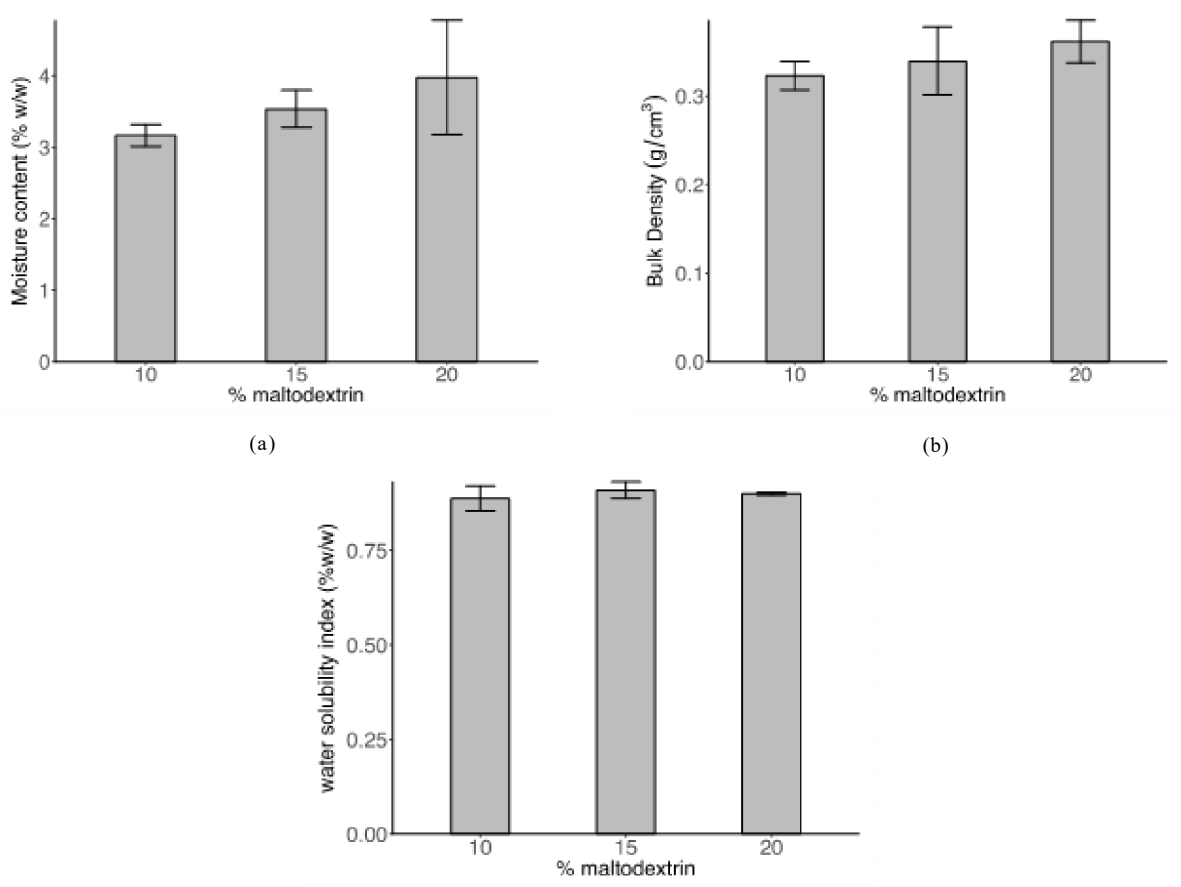


Fig. 6: Influence of MD concentration on moisture content (a), bulk density (b), and water solubility index (c).

for each specific set of process conditions during spray drying.

An increase in the MD concentration resulted in an increase in moisture content, as depicted in Fig. 6a, which is in agreement with the works of Goula & Adampoulos (2008), Akkaya et al. (2012), and Santhalakshmy et al. (2015). This is because the larger MD molecules hinders the diffusion of water molecules resulting to a slower rate of drying, and for sugar rich products, evaporation is dependent on the moisture diffusion in the crust (Roustapour et al. 2006, Santhalakshmy et al. 2015). In addition, Akkaya et al. (2012) reported that the drying of a drop occurs in two stages during spray drying. In the first stage, most of the drying occurs as a result of the vaporization of free moisture from the surface of the drop. However, in the second stage, crust formation happens at the surface of the droplet resulting in the concentration of unbound water in the inner part of the droplet.

Powder properties such as the bulk density is a strong indicator on how well powders can be handled, stored and processed (Jallo et al. 2012). The type of encapsulating agents had a significant effect (p <0.01) on the bulk density of spray-dried powders, as seen in Table 1. ST microencapsulated extracts resulted to powders with higher bulk densities compared to MD and GA. This is due to the inherent higher molecular weight of starch, a material composed of two polymers namely amylose and amylopectin. A high molecular weight would make it easier for the particles to be packed, resulting in a lesser occupied space (Tonon et al. 2010). Bulk densities of GA and ST microencapsulated extracts were observed to have significantly decreased when inlet temperature was increased. This was possibly due to bubble nucleation happening within the given residence time during the spray drying process which arises when droplets expand due to internal pressure build-up, increasing particle volume (Vehring et al. 2007). This result was in agreement with findings in literature where pomegranate juice and amla juice were encapsulated with MD (Mishra et al. 2014, Jafari et al. 2017). Relative to the other encapsu-

lating agents used, the MD microencapsulated extract was observed to have no significant difference in bulk density as inlet temperature was increased. This observation may be associated to the encapsulating material's solubility in water or which relates to the strength of the bonds created with water. The chemical structure of MD, which consists of -OH terminal functional groups, can create hydrogen bonding with water. Furthermore, MD encapsulation may have had a relatively stable droplet structure within the temperature range which hindered droplet expansion as opposed to the other encapsulating materials.

Increasing MD concentration also significantly increased (p <0.01) the bulk density of micro-encapsulated extracts, as shown in Fig. 6b. This increase in MD concentration added more encapsulant material per volume of extract resulting to an increase in the density of the powder produced. This result was found to be in agreement with the findings of Bae & Lee (2008), and Caliskan & Dirim (2016).

In product applications, the water solubility index (WSI) of powders is a strong factor to be considered in its proper reconstitution in aqueous matrices (Jafari et al. 2017). This property is strongly affected the choice of process parameters and the encapsulating agent to be used. In this study, it was observed that inlet drying air temperature did not have a significant effect on the WSI of microencapsulated extracts. This result was in agreement with the studies of Kha et al. (2010) and Jafari et al. (2017) where it was also observed through inlet drying air temperature variations. Furthermore, as shown in Fig. 5c, MD and GA powders were found to have higher WSI compared to ST. Interestingly, Fig. 6c shows that MD concentration has no effect on the WSI of the powders formed. GA in this case was found to have high WSI in spite of the inability of the material to dissolve completely in water. Again, this is mainly attributed to the presence of a small percentage of hydrophobic groups (2% protein) in its chain, with a large portion of it being hydrophilic (Dickinson 2003). This chemical structure is the reason for its emulsifying property and thus its considerable WSI. ST on the other hand is largely composed of amylose and amylopectin folded into helices which would result in a low solubility in water (Buléon et al. 1998).

CONCLUSIONS

Using spray-drying technology, the waste MSKE was successfully spray dried into powders using MD, GA and ST as encapsulating agents. Increase on the inlet drying air temperature (130°C to 170°C) had varying effects on the product quality parameters at different encapsulating agents; where it generally did not have a significant effect on %EY and WSI but significantly affected TPC, AOAc, moisture content and bulk density. On the other hand, the type of encapsulating agent (maltodextrin, gum Arabic and starch) had a significant effect on all of the quality parameters measured. These noted effects were heavily influenced by the physicochemical properties of the encapsulating agent as it interacts with the components of the extract and the spray-drying environment. The effect of encapsulating material concentration, as evaluated using MD in this work, influenced TPC and AOAc through a dilution effect. However, it should be noted that the opposite effect to moisture content was observed, also, increasing MD concentration had a null effect on %EY and WSI.

Basing on the results and complying the desirable traits of microencapsulated powders, MD is recommended as an encapsulating agent for the spray drying of MSKE due to it exhibiting superior yields, high bioactive contents and antioxidant activities. Moreover, it has also exhibited desirable physical properties such as low moisture content, considerable bulk density and high WSI for excellent reconstitution and dispersion of bioactive compounds.

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