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Microencapsulation of honey as flavouring agent in milk powder

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Abstract

Honey is a natural product commonly used in milk powder for flavoring purposes. Currently, honey flavor is added to milk powder in powder form. This study aimed to investigate the effect of spray drying operating conditions on the quantity and quality parameters of the honey powder recovered. The influence of inlet air temperatures, feed flow rates, and type of microencapsulating agents upon the properties of obtained powders was examined. Spray drying of honey with the addition of maltodextrin DE 18 and 12 was carried out at inlet air temperatures of 150, 170, 190°C and feed flow rates of 330 and 660 mL/h. The properties of the powders obtained were quantified in terms of moisture content (MC), hygroscopicity (HG) and color. Results of experiments showed higher inlet air temperature and lower feed flow rate led to a significant increase ($p<0.05$) in powder recovery. The total powder recoveries found in this study were between 39.42±2.35% to 71.92±4.17%, which indicates that this process has the potential to be applied on an industrial scale. The decrease in DE of maltodextrin promoted a higher powder recovery. Furthermore, it was found that the browning index increased with the decreasing inlet air temperature and increasing feed flow rate.

1. Introduction

Honey is a natural product commonly used in milk powder for flavoring purposes. It has been consumed all over the world since the earliest times. Currently, honey flavor is added to milk powder in powder form. Consumer's love for natural flavors drives the increasing demand for honey powder in the global market. However, the drying of liquid honey to obtain honey powder poses multiple problems in handling and processing involving mass production due to its high viscosity and stickiness (Nurhadi and Andoyo, 2012).

Honey drying is generally carried out through the spray drying method with various carrier agents. (Nurhadi and Andoyo, 2012) reported honey powder produced with spray drying and maltodextrin as the carrier agent with a ratio of honey to carrier agent 1: 1 had gained a recovery of 9.72%. In addition, a similar study conducted by Cuevas-Glory *et al*. (2017) showed a higher honey powder recovery of 39.6% with the use of different ratios of honey to carrier agent 1: 1.5. Despite a higher recovery of powder, the number is still relatively too low in order to meet the increasing honey powder demand. This means further research is still needed to apply an improved technique in order to obtain honey powder with higher recovery and better physical properties more achievable.

In this study, the microencapsulation technique was used to attain the previously mentioned honey powder quantity and quality objectives. Microencapsulation is a technique used to coat bioactive material (core) with a coating material (microencapsulating agent) to produce microcapsules in the form of solid particles (powder) (Gharsallaoui *et al*., 2007). Microencapsulation can provide protection of bioactive material during process and storage, and control the slow-release mechanism when needed (Özkan and Bilek, 2014; Shofinita *et al.,* 2020). Previous studies with various types of microencapsulating agents and operating conditions have been carried out by several authors. Avila *et al*. (2015) used a spray drying method to perform drying on sugar cane juice with maltodextrin DE 19-20 and was able to recover powder up to 91.3%. This was successfully achieved due to a higher amount of coating material used than the one performed by Nurhadi and Andoyo (2012)

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and Cuevas-Glory *et al*. (2017). Maltodextrins of different dextrose equivalent (DE) are commonly used as microencapsulating agents owing to their relatively lower cost, high solubility, low viscosity, and its colourless solution (Jafari *et al*., 2008; Kandansamy and Somasundaram, 2012). These properties make them useful ingredients in the food industry, especially in milk powder production (Avaltroni *et al*., 2004).

Nevertheless, no efforts have been made to study microencapsulation techniques on honey to date. Therefore, the objectives of this research were to produce honey powder by encapsulating honey with maltodextrin through spray drying and study the effect of the inlet air temperatures, feed flow rates, and types of microencapsulating agents on the quantity and quality parameters of powder.

2. Materials and methods

2.1 Materials

Honey (MC = 19.22±0.21%) was from Indonesia and kept at 4°C in a refrigerator until used. Maltodextrin DE 12 with MC of 5.12±0.85% and Maltodextrin DE 18 with MC of 4.35±0.09%, were used as microencapsulating agents.

2.2 Spray drying feed preparation

The feed solution was prepared by dissolving maltodextrin powder in distilled water to obtain a concentration of 70% (w/w-%DS). Honey was added to the solution in the ratio of honey to maltodextrin 1: 11 (w/w-%DS) and heated at 60±1°C at constant stirring. The solution was diluted to produce a concentration of 50±1°Brix with distilled water. Each preparation was homogenized with RW20 overhead stirrer (IKA, China).

2.3 Microencapsulation by spray drying

The feed solutions were prepared by pre-heating the solution and maintained at 60±1°C. The feed solutions were fed to a SD-Basic Laboratory Scale Spray Dryer (LabPlant, UK). The spray dryer was operated at an inlet air temperature of 150, 170 and 190°C, feed flow rates of 330 mL/h and 660 mL/h. The drying air flow rate and nozzle size were 0.3 m^3/min and 1 mm, respectively, for both types of microencapsulating agents. The powders obtained were stored in a sealed sampling bag and kept in a desiccator containing silica gel to prevent moisture uptake. All variations were performed in duplicate.

2.4 Powder recovery

The powder recovery (R_P) was calculated as the ratio of the mass of solids collected in the collecting vessel after spray drying to the mass in the feed solution on a dry basis. The total powder recovery ($R_{P,\ total}$) from the drying chamber, cyclone and collecting vessel was also calculated. The process of collecting powder in the drying chamber, cyclone, and other parts was carried out by directing a compressed air hose to the walls of components in order to blow all deposited particles to the new collecting vessel.

2.5 Characterization of powder

2.5.1 Analysis of moisture content

Approximately 1 g samples of powder were dried in the Universal Thermal Oven (Memmert, Jerman) at 105°C for 24 hrs (Chegini and Ghobadian, 2005). The samples were weighed before and after the drying process. Analysis was performed in triplicate.

2.5.2 Analysis of hygroscopicity

Approximately 1.5 g samples of powder were placed in a desiccator under the following conditions: 25°C and 75% relative humidity (saturated NaCl solution) (Tonon *et al*., 2009). The samples were weighed periodically during the equilibration process. Hygroscopicity was expressed as g of adsorbed moisture per 100 g of dry solids of powder. Analysis was performed in duplicate.

2.5.3 Analysis of color

Color measurements were performed by direct reading on a CR–400 Chromameter (Konica Minolta, Japan) using CIE Lab system. The chromameter was calibrated with a white plate before measurement. In this system, L^* values indicate lightness; a^* values indicate redness and greenness; b^* values indicate yellow and blueness. The parameter of L^*, a^*, and b^* were used to calculate the total color difference (ΔE^*) and Browning Index (BI). Analysis was performed in duplicate.

2.6 Statistical analysis

All data were reported as the mean ± standard deviation (SD) for every replication. Analysis of Variance (ANOVA) was carried out to determine the effect of spray dryer operating conditions and types of microencapsulating agents on all quantity and quality parameters studied.

3. Results and discussion

3.1 Powder recovery

The main evaluation of spray drying performance was done by calculation of powder recovery (drying yield) (Katarzyna Samborska, 2019). Only powder originating from the collecting vessel was used in the calculation, while powder collected from the chamber, cyclone, and other parts (i.e. connecting tubes) were disused to avoid unnecessary calculation errors (Fang

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and Bhandari, 2012). During spray drying, the feed solution (honey-MD mixture) may show sticky behavior by sticking to the wall of the drying chamber due to being rich in sugars (Bhandari and Howes, 1999). This can affect powder recovery or even cause problems during operation (Samborska *et al.*, 2015; Suhag and Nanda, 2016).

Particles that were dried at different inlet air temperatures and feed flow rates showed different powder recovery (Figure 1). At fixed inlet air temperatures, powder recovery indicated a significant decrease ($p<0.05$) as the feed flow rate increased from 330 mL/h to 660 mL/h. The powder recovery was negatively influenced by the feed flow rate where a lower feed flow rate obtained more powder in the collecting vessel. This trend might occur as a result of a higher feed flow rate that increases the number of droplets. This resulted in an increased risk of droplet collision that led to an agglomeration, which subsequently created a larger droplet and thus a larger surface. Moreover, a larger surface requires greater energy (heat from the drying air) to remove water content and more drying time, therefore resulting in inadequate droplet drying. Consequently, the resulting wetter powder mainly stuck to the wall of the drying chamber, and hence the powder recovery was significantly reduced. Additionally, a similar effect has been reported by several other researchers (Fazaeli *et al.*, 2016; Can Karaca, 2019).

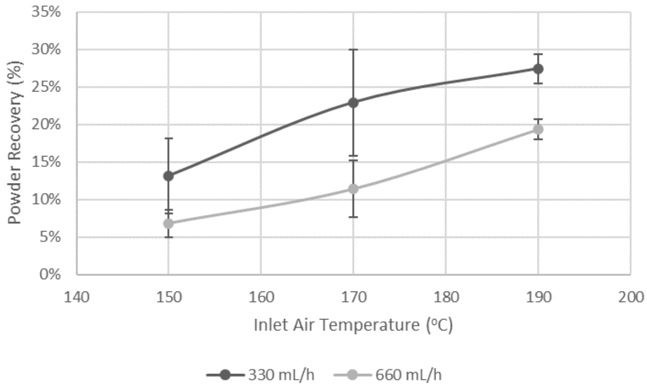

Figure 1. The effect of different inlet air temperatures and feed flow rates (330 and 660 mL/h) on powder recovery.

The effect of inlet air temperatures on powder recovery was also observed. Inlet air temperatures are known to have various effects on powder recovery with sugar-rich feed solutions. Studies conducted by other researchers noted some positive effects (Gouaou *et al.*, 2019), negative effects (Langrish and Premarajah, 2013), or even some reported no relationship between the two (Islam, 2009). Figure 1 shows the increase in inlet air temperatures resulted in significant increases in powder recovery at a constant feed flow rate. These observations were in agreement with those of Bansal *et al.* (2014) and Suhag and Nanda (2016). This can be an effect of more water contained in the droplet that was being evaporated by the heat from the drying air when the inlet air temperature was increased. Furthermore, the lowest powder recovery was obtained at an inlet air temperature of 150°C, both at feed flow rates of 330 mL/h and 660 mL/h. More powder obtained at higher inlet air temperature is associated with the low glass transition temperature (T_g) of low molecular sugars in the feed solution and is probably due to the diffusivity phenomenon.

During a spray drying experiment, when a droplet surface largely consists of low molecular sugars, a greater adhesive bond between droplets and the surface of the drying chamber wall will be formed hence resulting in more particle deposits generated on the wall (Bhandari and Howes, 2005). This is further supported by the low T_g value of fructose and glucose which mainly constitute honey sugars in the feed solution. Fructose is known to have a very low T_g of 5°C, whereas glucose has a T_g of 31°C (Roustapour *et al.*, 2006; Jaya and Das, 2009). According to Onwulata and Huth (2009), the stickiness point (T_{st}) occurs when the surface temperature ($T_{surface}$) is 10 – 20°C above the T_g. Studies indicated that the stickiness point decreases with the decrease in T_g of feed solution. On that account, $T_{surface}$ must be maintained at temperature below the T_{st} when in contact with the drying chamber wall to avoid stickiness. Therefore, the addition of maltodextrin with high T_g, 257.5°C (Castro *et al.*, 2016), was carried out to increase the T_g of the feed solution hence reducing the stickiness while also increasing the final powder recovery.

During feed solution preparation, the added honey will diffuse into the maltodextrin solution to form an equilibrium mixture. When atomized, both sugbar solids from maltodextrin and honey have the tendency to migrate preferentially to the air-droplet interface (Adhikari *et al.*, 2007). However, due to a much larger amount of maltodextrin being mixed in the feed solution (honey: MD = 1: 11, DS%), the outer layer of the droplet will probably be occupied by maltodextrin. When contacted with drying air, maltodextrin will form a glass-like layer while the sugar solids (i.e. fructose) from honey remain inside. This phenomenon occurs alongside the shrinking of droplet size caused by rapid evaporation of water in the droplet by heat from the drying air. The glass-like layer formed on the droplet-air interface is capable of withstanding sticky interactions between sugar solids and the drying chamber wall (Adhikari *et al.*, 2007). For that reason, at higher inlet air temperature, drying of droplets occurs faster which then holds the solids sugar in the inner part of the droplet from diffusing completely. This provides a lower chance for sugar solids to migrate to the air-droplet interface thus reducing the stickiness or even eliminating it. As a result, a larger amount of powder was able to be recovered.

The total powder recovery/$R_{P,total}$ (Figure 2) varied from 39.42±2.35% to 71.92±4.17%. These values were relatively much higher than those obtained from collecting vessels only (Figure 1). This probably occurred due to a small-scale spray dryer being used in the experiment where the nozzle was very close to the wall and/or the bottom of the drying chamber. Thus, the drying of droplets must be carried out in the shortest time possible before the particles are able to reach the wall and form deposits.

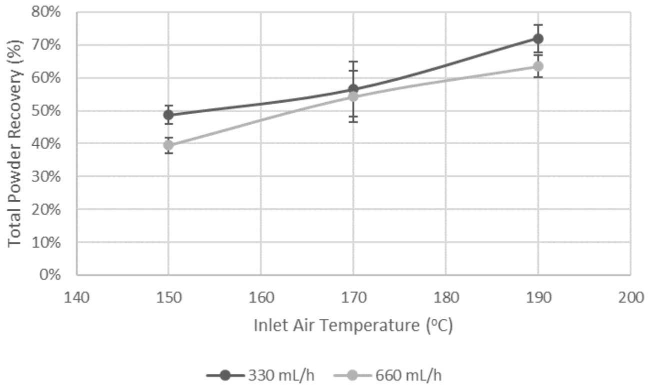

Figure 2. The effect of different inlet air temperatures and feed flow rates (330 and 660 mL/h) on total powder recovery.

The total powder recovery appeared to be more relevant to be used as a basis in the effort to scale up this experiment. An industrial-scale spray dryer typically has a greater capacity and is equipped with a pneumatic hammer to hit the wall of the drying chamber. Consequently, all deposited particles will be decayed and blown to the final product collector chamber. This indicated that although not every spray dryer is able to calculate the total powder recovery ($R_{P,total}$), as reported by Shi *et al*. (2013), an industrial-scale spray drying process is expected to produce a higher powder recovery than those reported in the small-scale process, including this experiment. It was also verified by Islam *et al*. (2013) who claimed that a small-scale spray drying process with powder recovery of 60 - 80% can be considered a successful process, with the potential of a much higher powder recovery when being processed in an industrial-scale (Shofinita *et al.,* 2021).

Apart from the varied operating conditions, the effect of different types of maltodextrin (MD DE18 and MD DE12) on powder recovery was also observed. A study by Perez-Alonso *et al*. (2003) reported that the addition of maltodextrin in the feed solution is known to be capable of modifying the balance of hydrophilic-hydrophobic groups of powder particles while reducing the amount of water which may be absorbed. As shown in Figure 3, the decrease in DE of maltodextrin promoted a higher powder recovery. Possibilities might be a result of two main factors: (1) physicochemical properties and (2) glass transition temperature (Tg) of sugar contained in maltodextrin (Lee *et al*., 2018).

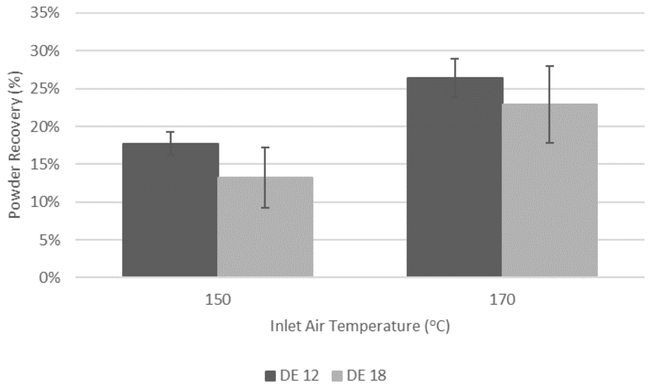

Figure 3. The effect of different types of microencapsulating agents on powder recovery.

Saavedra *et al*. (2015) reported a linear relationship between DE and molecular weight distribution (MWD) as well as the degree of polymerization (DP) of maltodextrin. This contributes to the number of branches in saccharides which can be formed. Previously, Wang and Wang (2000) observed that higher molecular weight leads to a higher branching of the main chain of saccharides. Maltodextrin with low molecular weight (MD12) has fewer branches than maltodextrin with high molecular weight (MD18) (Saavedra-Leos *et al*., 2019). Thus, maltodextrin with less branch structure provides fewer aldehyde functional groups to interact (i.e. hydrogen bonding and van der Waals force) with water molecules from the feed solution and/or water vapor from the drying chamber during the experiment. As a consequence, a higher Tg of feed solution was produced due to less water of low Tg (-138°C) bound to the maltodextrin in the feed solution. In addition, lower DE of maltodextrin indicated fewer saccharides were being hydrolyzed to DP1 sugar (glucose) during the production of maltodextrin, resulting in higher Tg of feed solution thus a higher stickiness point. Therefore, powder with higher recovery was obtained. An experiment conducted by Saavedra-Leos *et al*. (2019) showed a similar result. The authors reported that low molecular weight maltodextrin (DE 10) produced higher powder recovery than those of DE 20 and DE 40.

3.2 Moisture content

The moisture content observed in Figure 4 varied from 1.01±0.40% to 3.74±0.26%. An increase in feed flow rate resulted in a higher moisture content of the powder obtained. According to Tan *et al*. (2011), when the feed flow rate is increased, a larger droplet will be produced due to the high amount of feed solution being atomized. A larger droplet caused the distance that the heat must go through to get to the centre of the droplet to be even farther. Therefore, there was a possibility that the moisture content inside the droplet was not evaporated completely, thus powder with higher moisture content was produced.

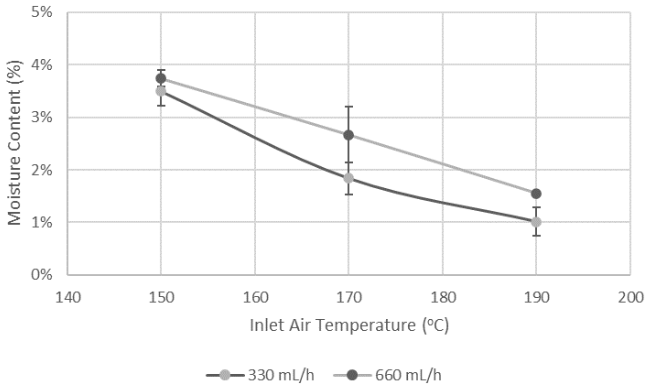

Figure 4. The effect of different inlet air temperatures and feed flow rates (330 and 660 mL/h) on moisture content.

In addition to that, according to Figure 4, there was a decrease in moisture content with an increase in inlet air temperature at a fixed feed flow rate. This is in agreement with the fact reported by Samborska and Bieńkowska (2013). They reported that the increasing inlet air temperature from 160°C to 200°C negatively affects the moisture content from 2.1% to 1.4%. It was also confirmed by the theory of energy needs for water evaporation reported in a previous study by Kanojia *et al*. (2016), who reported that at higher inlet air temperatures, greater energy in the form of heat from drying air was being produced. Thus, a greater amount of water could be evaporated resulting in less moisture content. Powder with low moisture content has an unfavorable effect on product stability during storage. Powder with low moisture content may absorb more water than those with higher moisture content when exposed to an environment with high RH (Juarez-Enriquez *et al*., 2017).

3.3 Hygroscopicity

The values obtained for hygroscopicity (Figure 5) varied from 8.70±1.20% to 16.85±0.60%. A minimum value of hygroscopicity was found at an inlet air temperature of 150°C and a feed flow rate of 660 mL/h. Figure 5 showed the increase in feed flow rate promoted a decrease in hygroscopicity at constant inlet air temperature, which is consistent with some results from previous work. This relationship is likely to be associated with a decrease in pores formation at higher feed flow rates thus leading to a reduction of total particle surface area through which the water may be absorbed, similar to the findings of Chen *et al*. (2014) and Muzaffar and Kumar (2015).

On the other hand, results indicated a positive effect between inlet air temperature and hygroscopicity at a fixed feed flow rate. In addition, at a higher inlet air temperature, the powder produced tends to have a low moisture content thus a higher water concentration gradient between powder and the surrounding air, according to a study conducted by Fernandes *et al*. (2013). As a consequence, powder with lower moisture content had a greater capacity to absorb ambient moisture from the environment during storage, therefore powder with significant hygroscopic properties was produced. A similar phenomenon can also be found in research conducted by Goula and Adamopoulos (2010) and Bringas-Lantigua *et al*. (2016).

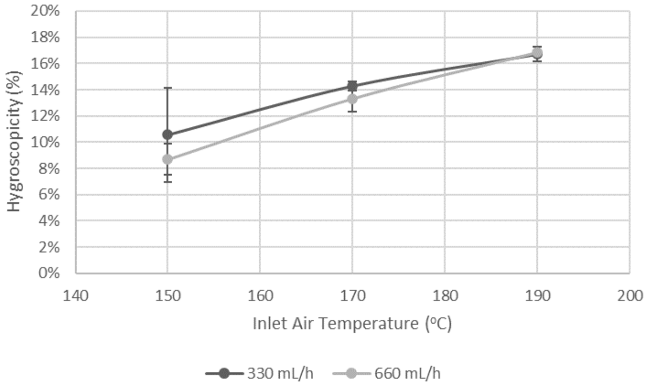

Figure 5. The effect of different inlet air temperatures and feed flow rates (330 and 660 mL/h) on hygroscopicity.

3.4 Color

Color measurement is an important quality indicator as it reflects sensory attractiveness and powder quality in the spray drying process (Quek *et al*., 2007). The L^* values showed insignificant changes (p>0.05) at different inlet air temperatures and feed flow rates in the range of 94.83±1.08 - 96.93±0.10 (Table 1). All varied operating conditions were observed to have produced a value of L^* close to 100, which indicated that the powders obtained were very light in color when compared to the one reported by Shi *et al*. (2013). This is due to a higher amount of maltodextrin with white color dominancy being used, resulting in a much lighter powder obtained.

Similarly, changes in inlet air temperature and feed flow rate also showed a negligible effect on b^* values of the powders produced. The b^* values varied from 3.29±0.07 - 3.77±0.08. These values were relatively lower in comparison to a study conducted by Nurhadi and Andoyo (2012) where they used a higher amount of honey in the feed solution resulting in a yellower powder.

Table 1 shows the use of all variations of operating conditions resulting in powders with the total color difference (ΔE^*) >5 when compared to honey powder control reported by Nurhadi and Andoyo (2012). These values indicated that the color difference was evident and easily distinguishable (Mokrzycki and Tatol, 2011). This

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Table 1. The effect of different inlet air temperatures and feed flow rates on color parameters.

Feed flow rate (mL/h)	Inlet air temperature (°C)	*L**	*a**	*b**	*ΔE**
330	150	96.46±0.13	-0.27±0.02	3.65±0.06	7.02±0.12
	170	96.93±0.10	-0.25±0.01	3.29±0.07	7.58±0.12
	190	96.42±0.17	-0.23±0.01	3.30±0.15	6.03±0.05
660	150	94.83±1.08	-0.28±0.07	3.73±0.02	6.26±0.35
	170	95.89±0.03	-0.18±0.01	3.77±0.08	6.62±0.08
	190	96.03±0.08	-0.26±0.01	3.53±0.01	6.89±0.05

can be attributed to the differences in feed solution composition and heating that led to the browning reaction during spray drying. Therefore, an analysis of the Browning Index (BI) was further carried out to determine the effect of heating on the final color of the powders produced.

The BI observed in Figure 6 varied from 3.19±0.08% to 3.78±0.09%. BI values increased with the decreasing inlet air temperature and increasing feed flow rate. According to Langrish and Fletcher (2001), the increase in feed flow rate will result in a higher risk of exposure at high temperatures due to the longer contact time needed for bigger droplets to evaporate. This statement is further supported by Yildiz (2009) and Shofinita and Langrish (2016), who reported that longer exposure to high temperatures provokes a higher oxidation and browning rate. Heating can cause browning which is most probably caused by Maillard reaction and caramelization. The Maillard reaction begins with the condensation of an amino group from a protein with a carbonyl group from reducing sugar contained in honey and maltodextrin in the feed solution and produces a Schiff base (Lund and Ray, 2017). This reaction can form a brownish melanoidin polymer compound which leads to the formation of brown color powder as a final product. Whereas caramelization occurred when pure sugars in the feed solution were exposed to heat. Caramelization is the process of dehydration followed by isomerization and polymerization that leads to the formation of brown-colored compounds (caramel) (Kroh, 1994).

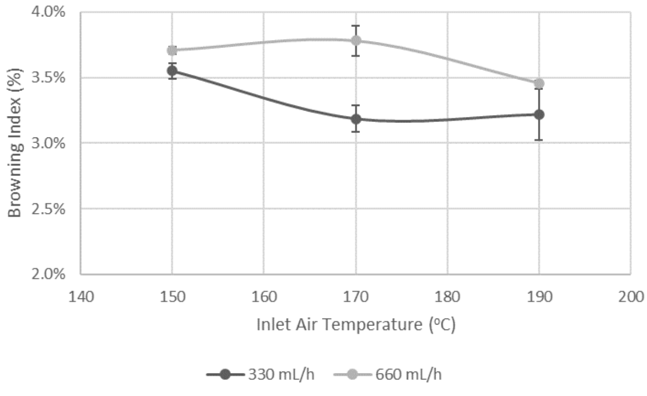

Figure 6. The effect of different inlet air temperatures and feed flow rates (330 and 660 mL/h) on browning index.

On the other hand, lower browning potential could also be obtained at the spray drying process with higher inlet air temperature. This is due to the resulting powders tend to have more free-flowing properties as more water is being evaporated hence powder with lighter weight is produced (Buma, 1971). Correspondingly, due to the lighter weight of the powder, it was easier to blow the product to get to the collecting vessel where a shorter time is needed thus no chance of excessive browning was allowed.

4. Conclusion

Results of experiments showed that higher inlet air temperature and lower feed flow rate led to a significant increase ($p<0.05$) in powder recovery. The powder recovery was in the range of 19.37±1.33% – 27.45±1.91%, and the total powder recovery was between 39.42±2.35% to 71.92±4.17, which indicates that this process has the potential to be applied on an industrial scale. The decrease in DE of maltodextrin promoted a higher powder recovery. Furthermore, it was found that the browning index increased with the decreasing inlet air temperature and increasing feed flow rate.

Conflict of interest

The authors declare no conflict of interest.

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