

Effect of operational conditions on physicochemical profiles of spray-dried powder of mango (*Mangifera indica* L.) juice from Tu Quy variety in Vietnam

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Abstract

The spray drying process is widely used to convert fruit juice to powder form, which improves the commercialization and consumption of fruit in powdered form. This research work was conducted to investigate the effects of spraying conditions including wall material concentration, inlet temperature, and feed flow rate on the physicochemical properties of the spray-dried mango powder from the Tu Quy variety in Vietnam. Maltodextrin as a wall material was determined at 10% which revealed an efficiently protective effect on the retention of polyphenol content (308.29 ± 8.62 mg/100 g) and vitamin C content (19.61 ± 0.35 mg/100 g) and exhibited even spherical particles with a smooth surface of the resulted powder. Increasing the inlet temperature was found to remarkably reduce the physicochemical properties of the resulted mango powder. The increase in the feed flow rate to 10 rpm resulted in better retention of polyphenols, and vitamin C content. The drying temperature and flow rate at 150°C and 10 rpm were found to obtain a good structural morphology of spray-dried powder. The X-ray diffraction analysis from the resulted powder indicated an amorphous material with a broad hump and no sharp diffraction peaks.

1. Introduction

Nowadays, health-conscious customers have been gaining interest in various foods with health benefits containing many bioactive compounds such as antioxidants, and antibacterial agents (Vivek *et al.*, 2020). This has increasingly attracted the attention of researchers to explore the beneficial effects of fruits. Rich sources of antioxidants have been successfully found in pomelo, butterfly pea flower, and watermelon (Mai and Tran, 2020; Nhi *et al.*, 2020; Tran *et al.*, 2020). Fresh fruits are an important constituent of a daily diet, composed of many biological compounds such as polyphenols, carotenoids, and anthocyanin (Cano-Higuera *et al.*, 2015).

Among tropical fruits, the mango (*Mangifera indica*)

is well-known as the most appreciated fruit for consumption due to its flavour, colour as well as nutritional values (Cano-Higuera *et al.*, 2015; Zotarelli *et al.*, 2017). Mango is a rich source of polyphenolic compounds promoting many beneficially biological effects. There are various types of polyphenols in mango pulp such as mangiferin, gallic acid, gallotannins, quercetin, isoquercetin in which gallic acid is a predominant constituent in mango pulp (Martin and He, 2009). According to the Ministry of Agriculture and Rural Development (MARD), Vietnam is the 13th biggest mango producer with a total mango production of 893.2 thousand tons in 2020. Mangoes are commonly cultivated in the Mekong Delta, accounting for about 48% of the total mango planted area in the country

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(MARD, 2021). Among these, the Tu Quy mango variety is popular and is very well adapted to adversely conditional cultivation such as drought and salinity as well as its ability in fruiting throughout the year. Mangoes, however, are easily susceptible to injuries during processing or storage (Zotarelli *et al.*, 2017). It is indicated that the physical damage and quality loss are accounted for 5-87% in a short period of time if mangoes are processed improperly (Serrano, 2005). Therefore, to effectively take an advantage of these benefits of mango fruit, the alternative approach should be applied to increase the storage time while preserving their beneficial effects. Powdered juice can be a possible option that is considered a convenient form due to the remarkable reduction in water content, and reduced transport cost (Caparino *et al.*, 2012).

Spray drying is a process of coating or entrapping food constituents in an appropriate carrying agent, allowing food from liquid form to be directly converted to powder form as a dehydrated product. Spray drying offers an operational condition at low temperatures and short exposure times, enhancing the retention of food properties such as nutrients, flavours, and bioactive compounds (Araujo *et al.*, 2020). The efficacy of the spray-drying process is dependent on operational conditions including inlet and outlet air temperatures, feed flow rate and concentration, pressure, or feed to wall material ratio (Caliskan and Nur Dirim, 2013). Maltodextrin, gum Arabic, and gelatin are successfully used as wall materials for spray drying (Mishra *et al.*, 2014). Maltodextrin derived from the hydrolysis of starch is considered to be a highly suitable wall material for application in the food and nutraceutical industry due to its low cost, high solubility, low viscosity at high concentration, and effective protection against oxidation (Labuschagne, 2018; Siacor *et al.*, 2020).

In this study, the Tu Quy mango variety was selected to formulate the spray-dried mango powder. The effects of operational conditions of the spray-drying process on the quality of resulted powder were evaluated including the inlet temperature, wall material: feed ratio, and feed flow rate. The result can provide an optimal condition for the process of spray-dried mango powder.

2. Materials and methods

2.1 Sample preparation

Mango (*Mangifera indica L.*) with Tu Quy variety was supported from Thanh Phu province, Ben Tre in July. After washing, the mangoes were preliminarily blanched in 90°C water for 4 mins, followed by a peeling process. The samples were then pureed by pressing processor (Phillips HR1863, Koninklijke Philips N.V,

Netherlands). The obtained mango juice was repeatedly filtered to remove insoluble solids using a 1 mm mesh.

2.2 Investigation of maltodextrin ratio on the quality of mango juice

The mango juice was mixed with maltodextrin, ranging from 5-20% (w/w). Tricalcium phosphate was added as an anti-caking agent to the mixture at the concentration of 0.003% to prevent the formation of lumps. The mixture was then spray-dried using a spray dryer (SD-06 spray dryer, Labplant UK Ltd, UK) at the temperature of 150°C with a flow rate of 10 rpm. The physicochemical properties of the resulted powder were evaluated. The spray drier was designed with a downward co-current drying mode of operation, 600 mm (diameter) × 1110 mm (height) × 825 mm (weight) in drying chamber dimensions, and a 0.5 mm jet assisted by a de-blocking to prevent the jet nozzle from becoming blocked.

2.3 Effect of spray-drying temperature on the quality of mango juice

The mango juice was mixed with maltodextrin and 0.003% of tricalcium phosphate. The maltodextrin ratio was selected upon the optimized condition from section 2.2. The effect of temperature was investigated in the range from 130 to 160°C at the flow rate of 10 rpm. The quality of mango juice including polyphenol content, antioxidant activity, and vitamin C was recorded at different processed temperatures.

2.4 Effect of the flow rate of the spray-drying system on the quality of mango juice

Similarly, the mixture composed of mango juice, maltodextrin, and temperature at optimized conditions was spray-dried at different flow rates, ranging from 7 to 10 rpm. The quality of mango juice including polyphenol content, antioxidant activity, and vitamin C was recorded at different processed flow rates of the mango juice.

2.5 Determination of polyphenol content in spray-dried mango powder

The polyphenol content of spray-dried powder was conducted following the Folin-Ciocalteu method. The sample of 0.25 g was mixed with 25 mL of methanol (50%), which was shaken for 3 hrs. The sample was then centrifuged at 13000 rpm for 10 mins (Hermle Z 446 Universal Centrifuge, Germany). The supernatant was collected and used for the analysis process. An aliquot of the supernatant (1 mL) was mixed with 5 mL of Folin – Ciocalteu (10%). After 5 mins, 4 mL of sodium carbonate was added to obtain the final volume of 10 mL. The sample was kept in the dark for 1 hr and the

absorbance was read at 675 nm using a photospectrometer (PhotoLab 6100 VIS, Germany). A standard calibration curve using gallic acid was also established to obtain the total polyphenol content (Siacor et al., 2020).

2.6 Determination of vitamin C content in spray-dried mango powder

The vitamin C content was determined following the method of Kapur et al. (2012). The mixture of metaphosphoric acid-acetic acid was prepared by mixing 15 g of metaphosphoric acid with 40 mL of acetic acid, which was volumed up to 500 mL. The sample (5 g) was homogenized with 25 mL of metaphosphoric acid – acetic acid solution. The obtained solution was filtered and centrifuged at 4000 rpm for 15 mins. The supernatant (4 mL) was mixed with 0.23 mL of 3% bromine water to oxidize ascorbic acid to dehydroascorbic acid, followed by the addition of 0.13 mL of 10% thiourea to remove the excess bromine. An aliquot (1 mL) of 2,4 DNP was then added to the mixture to form osazone, kept for 3 hrs at 37°C in a thermostatic bath. After cooling in an ice bath for 30 mins, 5 mL of 85% H₂SO₄ was added to the mixture and stirred constantly. The absorbance of the coloured solution was recorded at 521 nm. The calibration curve was created by using a stock solution of 500 ppm of vitamin C.

2.7 Antioxidant activity

The supernatant collected in the total polyphenol content determination step was used to evaluate the scavenging ability of antioxidants present in the sample. Briefly, the supernatant solution was concentrated to 20 mL in total volume. An aliquot of concentrated supernatant (0.15 mL) was added to 2.85 mL of DPPH (0.1 mM), and kept in the dark for 30 mins. The absorbance was recorded using Photolab 6600 Vis spectrophotometer at 517 nm. The percentage of DPPH inhibition was expressed as followed:

$$\% \text{ DPPH inhibition} = \frac{A_0 - A_1}{A_0} \times 100$$

Where: A₀ is the absorbance of the control sample and A₁ is the absorbance of the analysed sample

2.8 Scanning Electron Microscopy (SEM)

The morphologies of spray-dried mango powder were observed by a field emission scanning electron microscope (JEOL JSM-6480LV, Tokyo, Japan) at an accelerating voltage of 5 kV. All samples were coated with a thin layer of gold to improve the quality of captured images.

2.9 X-ray powder diffraction

The crystallinity of powder was evaluated using an X-ray diffractometer (XRD) (Rigaku Ultima IV, Rigaku Co., Ltd., Japan). The spray-dried mango powder was placed into a sample holder for the powder X-ray diffraction and the surface was smoothed with a glass slide. The measurement was carried out with the powder X-ray diffraction meter using Co (K_{α1+2}) radiation at 40 kV of potency and 30 mA, which was scanned between 4 and 50°- in 2θ, with a step-by-step increase of 0.02°- 2θ/0.5 s (Cano-Chauca et al., 2005).

2.10 Statistical analysis

Each experiment was conducted in three replicates. One-way analysis of variance (ANOVA) and Tukey's HSD tests (p<0.05) were performed to determine significant differences from each other using SPSS 22.0 software (Chicago, USA).

3. Results and discussion

3.1 Effect of maltodextrin concentration on physicochemical properties of spray-dried mango powder

The effect of maltodextrin concentration on the quality of the resulted mango powder is presented in Figure 1. Maltodextrin at 10% was found to better retain the polyphenol content (308.29±8.62 mg/100 g) and vitamin C (19.61±0.35 mg/100 g) while the maltodextrin concentration at 15% resulted in the highest DPPH reduction (20.36±0.84%), p≤0.05. Maltodextrin as a wall material was found to induce a favourable condition by protecting bioactive compounds under spray-drying. Maltodextrin was previously reported to interact with phenolic compounds to form a stable complex, enhancing the stability of polyphenols under the spray-drying process (Zhang et al., 2020). Increasing the maltodextrin led to the reduction in the polyphenol content. This could be due to the dilution effect of maltodextrin when its concentration was increased in the feed slurry (Mishra et al., 2014). In terms of vitamin C content, similarly, maltodextrin at 10% possibly contributed to the protection of vitamins C against operational conditions of the spray-drying process. The DPPH method evaluates the antioxidants that can scavenge DPPH radicals by donating hydrogen molecules from antioxidants. It has been noted that the DPPH assay is *in vitro* model and can not evaluate all of the antioxidant activities in foods including hydrophilic and hydrophobic polyphenols. Besides, the DPPH assay uses only radicals preferably dissolved in organic media which is suitable for hydrophobic antioxidants (Floegel et al., 2011). Thus, the antioxidants could probably not be extracted to the full extent in the DPPH assay. In this

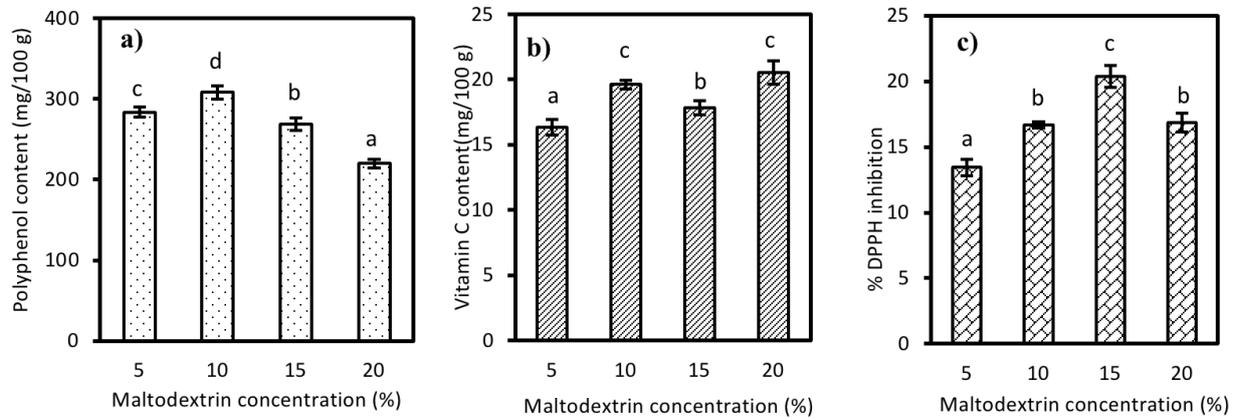


Figure 1. The effect of maltodextrin concentration on a) polyphenol content, b) vitamin C content, and c) antioxidant activity of spray-dried mango powder. Bars with superscripts indicate the significant difference in each treatment, $p < 0.05$.

study, the maltodextrin at 15% could better protect the hydrophobic polyphenols in the sample rather than at 10% regardless of the higher total polyphenols content at 10% of maltodextrin, resulting in the higher antioxidant activity at 15% of maltodextrin. The DPPH radical scavenging capacity of spray-dried powder is dependent on the feed concentration, type of wall materials, and inlet temperature (Gowdhaman and Ponnusami, 2015). Zhang *et al.* (2018) showed that the retention of antioxidant capacity was observed under the inlet air temperature of 120–160°C at 24% of maltodextrin or the maltodextrin content of 18–36% at 160°C (Zhang *et al.*, 2018). Different types of maltodextrin based on different DE values revealed different capacities in stabilizing the antioxidant activity of phenolic compounds (Negrão-Murakami *et al.*, 2017). Thus, the lower concentration of maltodextrin (10%) used for this study was possibly explained by the properties of feed slurry (Tu Quy variety) or the studied maltodextrin as compared to previous studies.

3.2 Effect of temperature on quality of mango powder

The inlet temperature of the spray-dryer has a significant impact on the quality of the resulted powder. The influence of temperature is presented in Figure 2. The physicochemical properties of spray-dried powder including polyphenol content, vitamin C content, and

antioxidant capacity were affected significantly by increasing the inlet temperature. The result indicated that polyphenols and vitamin C were susceptible to thermal processing. Larrauri *et al.* (1997) implied that the main mechanism for the loss of polyphenol content during drying was thermal degradation. Under thermal treatments, polyphenol oxidase is considered a factor in degrading the phenolic compounds (López-Vidaña *et al.*, 2017).

The polyphenol, vitamin C content, and antioxidant capacity were 374.26 ± 4.29 mg/100g, 21.13 ± 1.25 mg/100g, and $20.36 \pm 0.84\%$ which decreased to 247.29 ± 20.61 mg/100g, 13.44 ± 0.63 mg/100g, and 13.14%, respectively. These results were consistent with previously reported studies when increasing the inlet temperature (Mishra *et al.*, 2014; Zhang *et al.*, 2018). The polyphenol content of amla (*Embllica officinalis*) juice powder was noticeably reduced when increasing the inlet temperature from 125°C to 175°C, as reported by Mishra *et al.* (2018). Zhang *et al.* (2018) reported that the antioxidant capacity was found to decline from 76.57% to 66.63% when the inlet temperature was elevated from 120°C to 200°C (Zhang *et al.*, 2018). The decline in the antioxidant capacity was possibly due to the breakdown of the phenolic structure or the synthesis into a different form when they were exposed to higher temperatures (Mishra *et al.*, 2014).

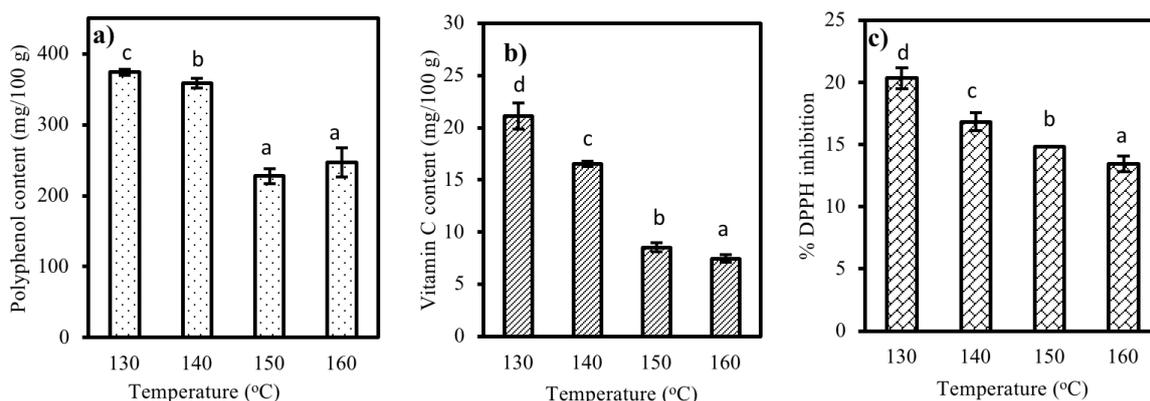


Figure 2. The effect of temperature on a) polyphenol content, b) vitamin C content, and c) antioxidant activity of spray-dried mango powder. Bars with superscripts indicate the significant difference in each treatment, $p < 0.05$.

3.3 Influence of flow rate on the quality of spray-dried mango powder

The influence of feed flow rate on the quality of mango powder is illustrated in Figure 3. The polyphenol content increased along with the increase in the flow rate from 7 rpm to 10 rpm (206.39 ± 2.05 mg/100 g to 361.12 ± 12.99 mg/100 g), $p \leq 0.05$. The low flow rate caused the extended time of polyphenols exposure to the drying temperature, resulting in the thermal degradation of polyphenols (López-Vidaña *et al.*, 2017). Increasing the flow rate meant to decrease the exposure of the sample to heat resulting in better efficacy in the retention of polyphenols, and vitamin C. In this study, the vitamin C content of spray-dried mango powder operated at the flow rate of 7 rpm was 11.09 ± 1.05 mg/100 g, increasing to 14.17 ± 0.57 mg/100 g at the flow rate of 10 rpm. The highest percentage in inhibition of DPPH was $14.31 \pm 0.07\%$ at the flow rate of 8 rpm, which slightly decreased at higher flow rates.

3.4 Structural morphology of spray-dried powder

Figure 4 presents the SEM micrographs of spray-dried mango powder with respect to different maltodextrin concentrations, inlet temperatures, and flow

rates. Generally, the morphology was observed to have approximately spherical particles, which was compatible with previous reports (Cano-Chauca *et al.*, 2005; Mishra *et al.*, 2014; Us-Medina *et al.*, 2018). At the low maltodextrin concentration (5%), these particles appeared to aggregate while increasing the concentration of maltodextrin helped them to be scattered and more spherical, which was in an agreement with Zhang *et al.* (2018). The concentration of maltodextrin at 10% was observed to have an even spherical particle with a smooth surface. The surface of mango powder at the concentration of 15% and 20% presented some cavities and dents which were ascribed to the uneven shell shrinkage during spray-drying or colliding by small particles (Zhang *et al.*, 2018). It was indicated that the spray-dried particles with the rough surface could be susceptible to oxidation due to their increased area (Tolun *et al.*, 2016). Besides, the cracks and dents on the particle surface were also found to adversely affect the flowability and reconstitution properties of resulted powder (Kaushik and Roos, 2007). It has been also suggested that the particle with a smooth surface is considered to be appropriate for applications in the food industry (Zhang *et al.*, 2018). From this point of view, even though the maltodextrin at 15% revealed a higher

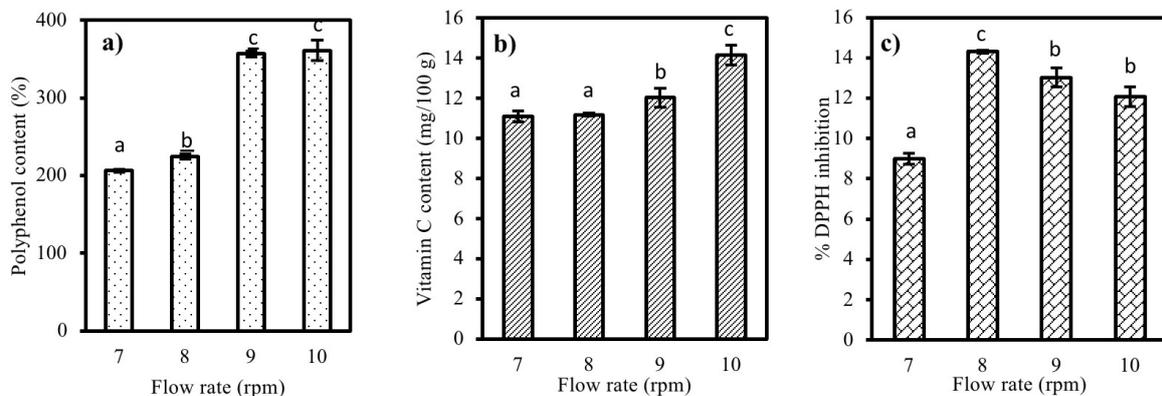


Figure 3. The effect of flow rate on a) polyphenol content, b) vitamin C content, and c) antioxidant activity of spray-dried mango powder. Bars with superscripts indicate the significant difference in each treatment, $p < 0.05$.

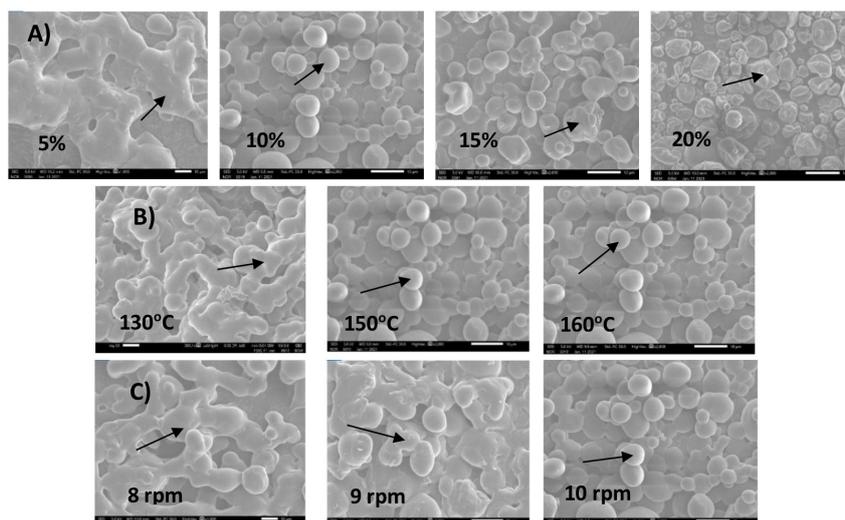


Figure 4. Structural morphologies of spray-dried mango powder under different operated conditions of A) maltodextrin concentration (5-20%), B) inlet temperature (130-160°C), and C) flow rate (8-10 rpm)

antioxidant capacity, the obtained morphology of the resulted powder appeared to have many cavities and dents which was an unfavourable option to be applied in the food industry.

In terms of temperature variation, even spherical particles and smooth surfaces resulted from operating the spray-drying process at the temperature of 150°C and 160°C. The inlet temperature at 130°C was not adequate to separate the particles due to the low rate of drying while a higher temperature with a rapid drying rate revealed the spherical particles with the increase in the number of particles (Mishra *et al.*, 2014). Feed flow rate had a noticeable effect on the morphology of spray-dried particles, as seen in Figure 4C. The flow rate at 10 rpm was found to be effective to obtain spherical particles while these particles tended to create agglomerates by glueing together at the flow rate of 8 rpm. A high flow rate was found to cause a high driving force to contribute to better droplet formation before exposure to the hot drying air, resulting in separately spherical particles rather than agglomerates (Vicente *et al.*, 2013). Therefore, the processed conditions at the temperature of 150°C and feed flow rate of 10 rpm also promoted the smooth surface of particles which was highly suggested apply for the production of spray-dried mango powder.

3.5 Analysis of X-ray powder diffraction

The XRD profile of spray-dried mango at different maltodextrin concentrations is described in Figure 5. X-ray diffraction profile of particles indicated an amorphous material by the presence of large, non-defined peaks with abundant noises. There was no significant difference in the XRD profile at different maltodextrin concentrations. The presence of amorphous material was due to inadequate time for crystallization. Besides, the amorphous solid demonstrated that the resulted powder had a high dissolution rate (Cano-Chauca *et al.*, 2005; Zhang *et al.*, 2018).

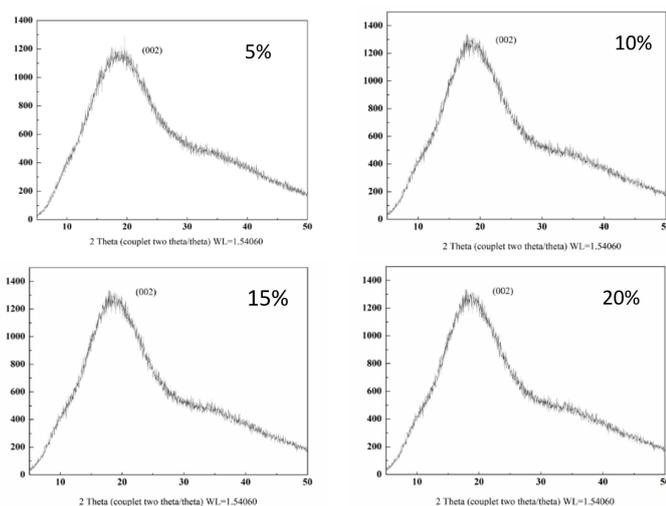


Figure 5. The XRD analysis of spray-dried mango powder

4. Conclusion

The final spray-dried mango powder was successfully developed. The operational process parameters including wall material (maltodextrin), temperature, and the feed flow rate were evaluated to obtain the spray-dried mango powder. The inlet temperature was found to significantly reduce the polyphenol, vitamin C content and antioxidant activity of the resulted powder. Polyphenol and vitamin C content were mostly retained at the maltodextrin of 10% and feed flow rate of 10 rpm. Although the higher remained antioxidant activity was obtained at the 15% of maltodextrin and the feed flow rate of 8 rpm, the smooth and spherical particles of the spray-dried powder processed at the 10% of maltodextrin, the inlet temperature of 150°C, and the feed flow rate of 10 rpm should be a priority selection to further applications in the industrial field. Besides, these selected parameters could be acceptable due to their high retention of the total polyphenol and vitamin C content. The amorphous behaviour revealed from the XRD analysis showed that the spray-dried mango powder had a high dissolution rate.

Conflict of interest

The authors declare no conflict of interest.

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