

Surface Modification To Reduce Deposition Flux of Spray Dried Noni (*Morinda Citrifolia L.*) Juice

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Spray drying is widely spread technique for drying of liquid products especially fruit juices such as noni juice. However, during spray drying of noni juice stickiness problem commonly occurs due to low molecular weight sugars (sucrose, glucose and fructose). The aim of the present research was to determine the impact of Teflon surface modification on the yield of spray dried Noni juice and deposition flux. Different exposure time of feed spray was undertaken prior to adhesion flux weight determination. Three different sets of inlet temperature (100°C, 150°C and 190°C) and exposure times (of 15 min, 30 min, 60 min, 75 min, 90 min and 115 min) were tested. Samples were evaluated for contact angle, weight of adhesion flux, hygroscopicity and moisture content. There was a sudden increased in deposition flux ($p < 0.05$) as the temperature increased. At 150 and 190°C, the weight of deposition flux was not significant ($p > 0.05$) for both plate materials. The borosilicate plates (BP) registered up to 80% deposit removal whilst Teflon plate (TP) recorded only 20%. Physical observation of the Teflon plates showed that some areas of the plate (100 °C) were fully clear from any particles. Nevertheless, for the borosilicate glass plates, the primary adhered particles tend to retain and smeared along the plates. In addition, there was a substantially negative relationship between the contact angle and adhesion weight, ($r = -0.924, p < 0.01$). The negative connection suggests that by increasing the contact angle, the adhesion weight will decrease and vice versa. The contact angle of the Teflon (PTFE) surface was approximately $117.2 \pm 1.4^\circ$, which provided an excellent hydrophobic surface. In this research, the powder with the lowest amount of hygroscopicity was gained at 190°C using the Teflon plate. For that reason, surface modification was indeed amplified the surface hydrophobicity thus decreasing the deposition flux.

Keywords: Spray drying, stickiness, Teflon, deposition flux, adhesion, yield.

Morinda citrifolia is described as a small or medium sized tree that usually grows in tropical lands. It is commonly known as Noni or Indian mulberry which has been used as both food and medicine¹. Extract of *M. citrifolia* has been used for generations in traditional therapy². Noni has been presented in the popular press as a supplementary treatment for various medical conditions, including

cancer³. *Morinda citrifolia* L. (Noni) is from the Rubiaceae family, and it is a supply of natural molecules that has been used as a healing plant by the Polynesians for more than 2,000 years⁴. However, many people avoid consuming the juice because of its unpleasant odor. Deodorization process will reduce the undesirable odor of Noni juice which was contributed by medium chain fatty

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acids such as decanoic acid, hexanoic acid and octanoic acid². Studies done by Haslaniza *et al.* 2015 has shown that deodorization of Noni extract has been successfully achieved using ion exchange resin with promising results⁵.

Spray drying is a method which depends on the properties of wall structures, nature of material to be retained and location. Indirectly, the combination will influence the parameters measured including moisture content, particle size, and density⁶. Spray drying is a technique to produce a dry powder from a liquid by rapidly spraying the feed into a hot drying medium⁷. Powder recovery in spray drying method is a big challenge in food technology. This is primarily because of the nature of food which contains low molecular weight sugars and organic acids, which have a low glass transition temperature⁸.

Spray drying of sugars and high acid food materials is associated with stickiness problem. In order to obtain successful drying, numerous methods have been studied such as use of high molecular weight aids to increase glass transition temperature of feed mixture, use of low humidity and low temperature conditions, scrapping of dryer surfaces, cooling of dryer wall by using dehumidified air and surface modification by proteins to encapsulate sugar⁹. The growth of wall depositions in the spray dryer decreases the production of the products and hence raises the costs of manufacturing and maintenance. Powder properties namely, moisture content, bulk density, particle size, hygroscopicity and morphology were influenced by inlet temperature. It was discovered that at a continuous feed flow level, raised the inlet air temperature decreased the residual moisture content. The same activity was found in the different fruit juice powders such as tomato juice, acai juice and pineapple juice¹⁰. This research is attempted to increase the efficiency of spray dryer by increasing its product recovery (Noni powder) through physico-chemical treatment on the surface of drying chamber of spray dryer by using different inlet temperature.

MATERIALS AND METHODS

Fruit Juice Extraction

The Noni fruit were obtained from a consistent local sources at UKM-MTDC

Technology Centre, Bangi, Selangor. The fruits were allowed to ripen to a soft consistency for 3 days. The Noni juice was prepared by selecting the fruits of stage 4 maturity index as shown in the Figure 1¹¹. The fruits were then being cut into smaller size and were added with distilled water in ratio of 1:1 (w/w) and blended using a food blender (7011S, Waring Blender, Torrington, USA). After that, the juice was filtered with muslin cloth and centrifuged at 8000 rpm for 15 minutes (Sorvall HS 23, Thermo Electron Corporation, USA). Centrifugation helps in removing the fruit pulp from the extract.

Conditioning of resin

Approximately of 0.5 g Amberlite IRA-67 resin was soaked in 5.0 ml HCl (5%) for 45 minutes^{12, 13}. Later, the resin was filtered with filter paper (Sartorius 1288) then re-soaked in 15 ml deionized water for 2 hours. To ensure the removing of residue, the resin was washed again with deionized water. Finally, the resin was filtered and was dried in desiccator for 24 hours

Deacidification of Noni Juice using Ion Exchange Resin

Noni juice extract was mixed with 0.5% (w/v) Amberlite IRA-67 resin; 0.1 g resin = 0.02 L sample in conical flask and was agitate in orbital shaker (WiseCube, Daihan Scientific, Korea) at 120 rpm for 327 minutes^{14,15,16}. The sample was then filtered with filter paper (Sartorius 1288) and kept in -20°C in Scott bottle before being used for further studies.

Spray Dryer Operating Conditions

The sample was fed into a lab-plant spray-dryer B-290 (Buchi Mini Spray Dryer, Flawil, Switzerland) by using a peristaltic pump. A cyclone air separator/powder recovery system was utilized. The dryer was designed as such that the outlet and inlet air temperature are the result of a combination of the inlet temperature, the aspirator setting, the pump setting and feed concentration⁷. After spray drying process, the powders deposited were collected and then weighed. The dried powders were collected from the cyclone separator and stored in desiccators at room temperature until further analysis.

Preparation of hydrophobic film

Microscope slides (Borosilicate, 76 mm x 26 mm x 1 mm) were obtained from Quasi-S Technology (M) Sdn. Bhd. and were used to mimic

the chamber wall of a spray dryer (BUCHI Mini Spray Dryer B-290). To measure of any particles agglomeration occurs at the walls of the spray dryer (rather than in the bulk of the dryer), the sample plates were left in the spray dryer for 1 hour to allow of any noni powder sticks to treated surfaces. Two sets of plates were involved in the preparation of the hydrophobic film; borosilicate glass plates (BP) and Teflon plates (TP). The TP were fabricated by covering the borosilicate glass involved in the experiment by using *Polytetrafluoroethylene* preparation (60 wt % dispersion in H₂O). Films were fabricated by dip-coating the PTFE emulsion onto glass slides, which were then placed in an oven at 100°C for 20 minutes to remove any residual water¹⁷.

Contact Angle (CA) Measurement

The water contact angle (WCA) was measured using an Automated Contact Angle Goniometer (Model 100) from Rame-Hart Inc. with MB-Ruler Software. The contact angle measurement was done using a static contact angle technique (Drop Tensile Analyzer). An amount of 0.5 ml deionized water was dropped at three separate points on the glass surface of the treated and untreated slides^{18,19}. All measurements were carried out in triplicates ($n=3$).

Determination of Flux Adhesion Weight

Flux adhesion weight was determined using an analytical balance SHIMADZU (model: ATX224) by assessing the weight of the plates before and after the feed spray¹⁹. The deposition flux was obtained at three different temperatures. The feed spray was carried out 30 min prior to deposition flux determination. For each set of plates, three inlet temperatures (120, 150 and 170°C) were analyzed²⁰. In selecting these two plate materials, only the surface energy properties were taken into account. The plates were put on the stage in the drying chamber randomly as shown in Figure 2 and the weight of deposition flux on the plates were determined ($n = 3$).

Hygroscopicity (H)

Hygroscopicity of the powders was decided based on the method illustrated by Cai and Corke (2000), with some modification. About 0.5 g of each powder were put in a desiccator containing a saturated solution of NaCl (RH 75.3%). The results were performed as the mass

of water absorbed per 100 g of sample after seven days²¹ in triplicates ($n=3$).

Moisture Content (M)

The moisture content of the spray dried samples was determined by using a moisture analyzer. Samples with predetermined masses were placed in an oven (Mettler) at 70 °C until consecutive weighing, which was done at 2 h intervals, provided less than 0.3% variation⁹. The product moisture content was determined from the weight loss by averaging three measurements ($n=3$).

Statistical Analysis

The experimental results in the single factor experiments were analyzed using Statistical Package for the Social Science SPSS version 23.0 (SPSS Inc., Chicago, IL, USA). The data was analyzed by analysis of variance (ANOVA) method. Significance differences between the means were determined by One-Way ANOVA (Tukey's multiple comparison) and independent t-test ($p<0.05$).

RESULTS AND DISCUSSION

Result of Duration Treatment on Contact Angle

In the present research, the duration of treatment involved was between 15-60 minutes. Figure 3 shows a substantially significant difference ($p<0.05$) between the value of the contact angle of the untreated slide (Borosilicate plate) with the slide that was treated (Teflon plate) ($p<0.05$). This showed that 15 minutes treatment time was not sufficient to raise the contact angle value. An increase in the contact angle value also can be observed in this study on Teflon coated plate with increasing duration time. The value of contact angle on Teflon coated plate (TP) achieved the highest on the 45-minute treatment period, while the Borosilicate plate (untreated) start to maintain its value from 30-minute treatment until 60-minute of treatment. The interfacial surface energy of the equipment and product plays a vital role in controlling the adhesion phenomenon, while the interphase diffusion and viscoelastic behavior control the cohesive phenomenon. In this study, the contact angle value indicated that the BP had a more hydrophilic character as compared with TP. The contact angle of TP (117°) was relatively

high, showing its poor hydrophilicity significantly ($p < 0.05$) compared to BP.

Effect of Weight Adhesion Flux and Duration of Treatment

According to this result in this study, there is an increment ($p < 0.05$) in weight of adhesion flux on Teflon coated plate and borosilicate plate. A rise in the inlet air temperature from 100°C to 190°C resulted in a significant rise ($p < 0.05$) in the particle size distribution of the powder. By increasing the inlet air temperature, particles changed into a largersize because of the greater swelling of particles¹⁰. General trend for the plate at different inlet temperature clearly indicated that usage of Teflon coated plate (TP) resulted with less deposit when compared to Borosilicate plate (BP) show higher value of weight adhesion flux at 190 °C ($p < 0.05$) as compared to Teflon coated

plate. At 150°C and 100°C of inlet temperature, it seems to be in significant value between Teflon and Borosilicate plate. Drying at higher temperatures resulted in faster drying rates, which led to the early formation of a structure and that did not allow the particles to shrink during drying²¹. When the inlet air temperature is low, the particle remains more shrunk and smaller. However, at 100 °C, both plates have low weight of adhesion compare to 150 °C and 190°C. Figure 5 show the fine particle deposited



Fig. 1. Freshly harvested Noni fruits at stage 4 maturity



Fig. 2. The Borosilicate and Teflon plates were placed horizontally in the bottom of drying chamber. The adhesion flux deposited at plates were observed and weighed ($n=3$)

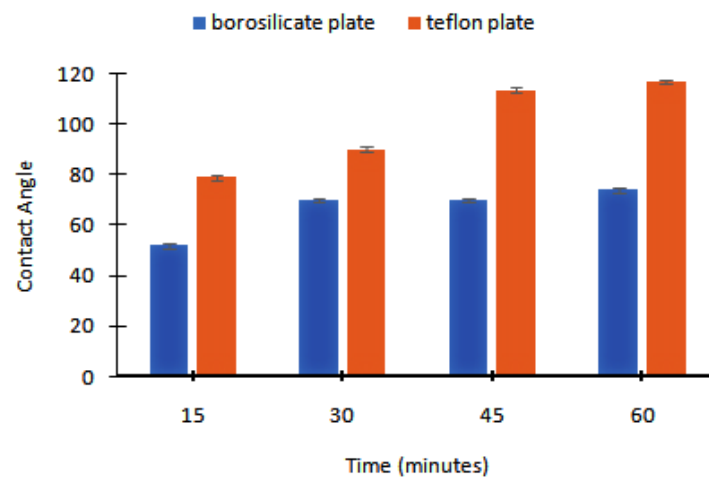
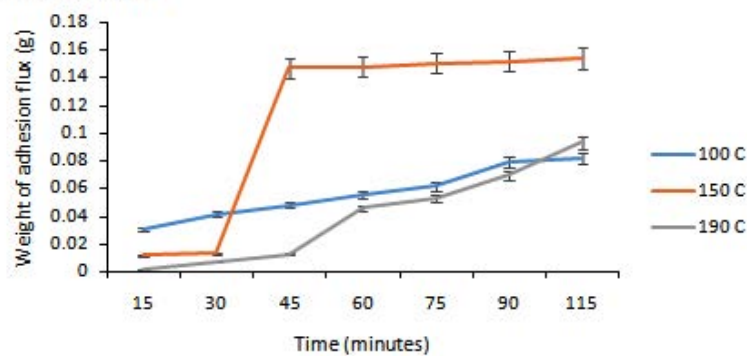


Fig. 3. Contact angle variation with respect to time duration

at Teflon coated plate, meanwhile the Borosilicate plate observed the deposition of hard crust. This is linked with the different drying rates, which has the highest rate at higher temperatures, resulting in the faster water evaporation and then it led to the formation of smooth and hard crust¹⁰. However, particles at 120°C and 150°C showed a rigid with wrinkled structures due to the lower operating temperature⁸. From this observation, if the inlet

temperature is low, the heat does not have enough time to dry the noni juice and thus the highmoisture content in the powdery product affect its stickiness at the drying chamber wall and eventually decrease the product yield. Furthermore, when the drying time too long, an accumulation of underdried large particles on the chamber wall may occur, which leads to low yield values end product. In average, at 150°C and 170°C, the deposition fluxes were obviously lower for TP.

a) Teflon coated plate (TP)



a) Borosilicate plate (BP)

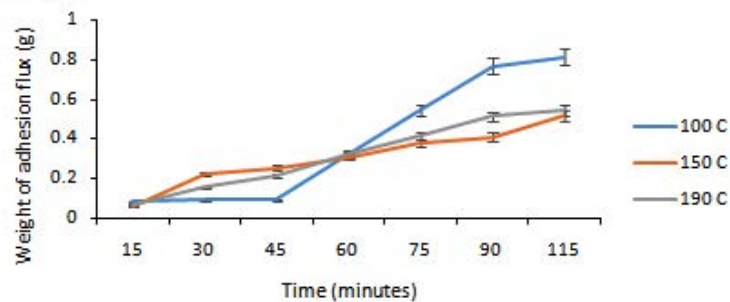


Fig. 4. Weight of adhesion of a) Teflon coated plate (TP) and b) Borosilicate plate (BP) with respect to duration of time at different temperature (100 °C, 150 °C and 170 °C)

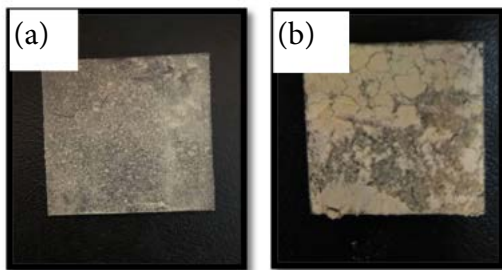


Fig. 5. The flux in fine particle deposited at borosilicate plate (a) and become hard crust at Teflon coated plate (b)

Effect of Hygroscopicity and Moisture Content

Hygroscopicity refers to the ability of a material to soak up moisture from the surrounding, and it is an important property to be taken into account in food stability. Low moisture inhibits the agglomeration of particles preventing the caking of powders, which could lessen retention of active properties and other properties such as flow and dispersion of powders²². With arise in the inlet air temperature, powders with lower moisture

Table 1. Experimental design with coded and real values, moisture content (*M*) and hygroscopicity (*H*) of spray-dried *Noni* powders using Teflon plate. Different superscripts letters in the same column mean significantly differences ($p < 0.05$)

Test	Inlet Temperature (°C)	Moisture Content (%)	Hygroscopicity
1	100	5.07 ± 0.1 ^g	15.5 ± 0.3 ^b
2	100	3.75 ± 0.2 ^{ef}	13.9 ± 0.1 ^a
3	100	4.67 ± 0.2 ^g	13.2 ± 0.0 ^a
4	150	3.34 ± 0.1 ^{cd}	17.3 ± 0.4 ^d
5	150	3.56 ± 0.1 ^{de}	17.6 ± 0.2 ^c
6	150	4.07 ± 0.2 ^f	16.5 ± 0.1 ^c
7	190	2.66 ± 0.1 ^a	18.9 ± 0.2 ^c
8	190	2.8 ± 0.1 ^{ab}	18.5 ± 0.3 ^{de}
9	190	3.1 ± 0.1 ^{bc}	17.6 ± 0.2 ^c

Table 2. Experimental design with coded and real values, moisture content (*M*) and hygroscopicity (*H*) of spray-dried *Noni* powders using Borosilicate plate. Different superscripts letters in the same column mean significantly differences ($p < 0.05$)

Test	Inlet Temperature (°C)	Moisture Content (%)	Hygroscopicity
1	100	3.07 ± 0.1 ^{cde}	17.5 ± 0.3 ^d
2	100	3.15 ± 0.3 ^{de}	15.9 ± 0.2 ^{bc}
3	100	3.67 ± 0.2 ^f	13.7 ± 0.0 ^a
4	150	2.54 ± 0.2 ^{bc}	17.3 ± 0.4 ^e
5	150	3.42 ± 0.2 ^{cd}	15.6 ± 0.2 ^c
6	150	1.87 ± 0.3 ^{ef}	14.5 ± 0.1 ^b
7	190	2.37 ± 0.1 ^a	19.9 ± 0.2 ^f
8	190	2.63 ± 0.1 ^{ab}	17.5 ± 0.3 ^d
9	190	8.4 ± 0.1 ^{bc}	33.6 ± 0.5 ^b

content were obtained. On the contrary, rising the air flow rate usually causes powders to have higher moisture content on the both plates ($p < 0.05$). This might be due to the shorter staying of particles/droplets in the chamber and smaller values of vapor pressure of water ⁶. From this research, the addition of Teflon coated on the plates leads to a significant decrease ($p < 0.05$) of the hygroscopicity compared to Borosilicate plates (non-coating) at 100 °C. This demonstrated the ability of Teflon to improve the hygroscopicity values of *Noni* fruit extract powders. Moreover, temperature of inlet also affected the hygroscopicity values of powders obtained using Teflon coating. Temperature at 100 °C was the variable that most affected ($p < 0.05$) compare to 150 °C and 190 °C. It is important to note that for samples with low moisture contents the hygroscopicity values were higher. This can be justified because higher temperatures may produce smaller particles, expanding the exposure area of the particles to the medium²².

CONCLUSION

The present study established a simplified model of spray drying based on fundamental parameter concentrating on application in food industry. The development of this model allows

the estimation of some parameters for the design of processes preventing stickiness with lead to waste with successive laboratory tests throughout the development of new alternative by spray drying. These findings in this study showed that the parameters studied, inlet temperature and duration of treatment (minute) showed significant effects ($p < 0.05$) on the contact angle (°) and weight of adhesion (g). Compared to untreated surface (BP), the contact angle rose and the weight of the adhesion decreased significantly ($p < 0.05$). This study revealed there was a rise in the contact angle on TP, and the value of the contact angle is considered as hydrophobic ($117.2 ± 1.4°$). The borosilicate plates registered up to 80% deposit removal while Teflon plate recorded only 20%. Physical observation on the Teflon plates revealed that some regions on the plate (190 °C) were totally clear from any particles. However, for the borosilicate glass plates, the primary adhered particles tend to retain and smeared along the plates. The powder with the lowest hygroscopicity was obtained at 190°C using Teflon coated plate. Further work can be done using different plasma system which were easier to manage such as atmospheric pressure plasma system that do not need the use of vacuum chamber.

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REFERENCES

- West, B. J., Deng, S., & Jensen, C. J. *Morinda citrifolia* (Noni) Fruit Juice Inhibits Endocannabinoid Degradation Enzymes. *Journal of Biosciences and Medicines*, 2019; **7**(07), 22.
- Nur Hafiza, Z., Maskat, M. Y., Liew, S. L., & Mamot, S. *Fermentation of Morinda citrifolia extract by Saccharomyces cerevisiae as affected by substrate concentration, inoculum size, temperature and fermentation time. International Food Research Journal*, 2013; **20**(4): 1889-189.
- Amy, B. *Anticancer activity of Morinda citrifolia (Noni) fruit: A review. Phytotherapy Research*, 2012; **26**(10), 1427-1440.
- Coutinho de Sousa, B., Reis Machado, J., da Silva, M. V., da Costa, T. A., Lazo-Chica, J. E., Degasperi, T. D. P. & Freire Oliveira, C. J. *Morinda citrifolia (noni) fruit juice reduces inflammatory cytokines expression and contributes to the maintenance of intestinal mucosal integrity in DSS experimental colitis. Mediators of inflammation*, 2017.
- Haslaniza, H., Wan Yaacob, W.A., Osman, H. and Maskat, M. Y. Interaction of antioxidants and organic acid from noni (*Morinda citrifolia* L.) juice with ion exchange resins during deodorization via deacidification. *Der Pharma Chemica*, 2015; **7**(9): 9-21.
- Kalantari, M., Niakousari, M., Haghighi Manesh, S., & Rasouli, M. Fig extract drying: The relationship between the main operating parameters of a pilot scale spray dryer and product specifications. *Food science & nutrition*, 2018; **6**(2): 325-333.
- Kiritsakis, K., Goula, A. M., Adamopoulos, K. G., & Gerasopoulos, D. Valorization of olive leaves: spray drying of olive leaf extract. *Waste and biomass valorization*, 2018; **9**(4): 619-633.
- Woo, M., Daud, W. R. W., Tasirin, S. M., & Talib, M. Z. M. Controlling food powder deposition in spray dryers: wall surface energy manipulation as an alternative. *Journal of Food Engineering*, 2009; **94**(2): 192-198.
- Chasekioglou, A. N., Goula, A. M., Adamopoulos, K. G., & Lazarides, H. N. An approach to turn olive mill wastewater into a valuable food by-product based on spray drying in dehumidified air using drying aids. *Powder technology*, 2017; **311**: 376-389.
- Phisut, N. Spray drying technique of fruit juice powder: some factors influencing the properties of product 2012.
- Yien, Y.S., Hassan, O. & Zubairi, S.I. Deodorizing Mechanism of α -Cyclodextrin-Organic Acids Inclusion against Strong Odor of Morinda Citrifolia (Mengkudu) Juice. *Jurnal Teknologi (Sciences and Engineering)*, 2016; **79**:10; p67-75
- Abidin, N.Z., Janam, A. & Zubairi, S.I. Adsorption of Saponin Compound in Carica papaya Leaves Extract Using Weakly Basic Ion Exchanger Resin. In AIP Conference Proceedings (Vol. 1784, 030039) 2016; AIP Publishing.
- Hashim, H., Ahmad, W. Y. W., Zubairi, S. I. & Maskat, M. Y. Effect of pH on Adsorption of Organic Acids and Phenolic Compounds by Weakly Basic Ion Exchanger. *Jurnal Teknologi (Sciences and Engineering)*, 2019; **81**:1; p69-81.
- Amran, S. N. S., Abidin, A. Z. & Zubairi, S. I. (2018). Saponin Bitterness Reduction of Carica Papaya Leaves Extract through Weakly Basic Ion Exchange Resins Adsorption. *Journal of Food Quality*, 2018, Article ID 5602729, 11 pages
- Hashim, H., Zubairi, S. I., Mustapha, W. A. M. & Maskat, M. Y. Characterizing the Deacidification Adsorption Model of Organic Acids and Phenolic Compounds of Noni Extract using Weak Base Ion Exchanger. *Journal of Chemistry*, 2018, Article ID 637692
- Hashim, H., Ahmad, W. Y. W., Zubairi, S. I. & Maskat, M. Y. Potential of Amberlite IRA 67 Resin For Deacidification of Organic Acids In Noni Juice. *Der Pharma Chemica*. 2015; **7**:12 (2015) 62-69.
- Zhang, Y.-Y., Ge, Q., Yang, L.-L., Shi, X.-J., Li, J.-J., Yang, D.-Q., & Sacher, E. Durable superhydrophobic PTFE films through the introduction of micro- and nanostructured pores. *Applied Surface Science*, 2015; **339**: 151-157
- Ramlan, N., Zamri, N. W. M., Maskat, M. Y., Hoong, C. O., Theng, L. Y., & Zubairi, S. I. Effect of plasma treatment (He/CH₄) on glass surface for the reduction of powder flux adhesion in the spray drying process. In AIP Conference Proceedings, 2018a (Vol. 1940, No. 1, p. 020093). AIP Publishing
- Ramlan, N., Zamri, N. W. N., Maskat, M. Y., Aizad, S. & Zubairi, S. I. Effect of Plasma Treatment (He/CH₄) on the Glass Surface during Drying to Reduce Powder Flux Adhesion. *Sains Malaysiana*, 2018b; **47**(6): 1147-1155.
- Woo, M. W., Daud, W. R. W., Tasirin, S. M., & Talib, M. Z. M. Optimization of the spray drying

- operating parameters - A quick trial-and-error method. *Drying Technology*, 2007; **25**(10): 1741-1747
21. Reineccius, G. A. The spray drying of food ingredients. In *The Encapsulation of Food Ingredients* (pp. 151-184). Leatherhead Publ 2001.
 22. Daza, L. D., Fujita, A., Fávoro-Trindade, C. S., Rodrigues-Ract, J. N., Granato, D., & Genovese, M. I. Effect of spray drying conditions on the physical properties of Cagaita (*Eugenia dysenterica* DC.) fruit extracts. *Food and bioproducts processing*, 2016; **97**: 20-29.