ORIGINAL ARTICLE

Thermal Effect of Spray Drying Process on the Quality of *Ficus deltoidea* Extract

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ABSTRACT

Introduction: Spray dry is a single step of drying method to transform the fluid materials to dry particles. Common practice for producing the solid form is by using the freeze dry technique. However, the existing freeze dry process was associated with longer drying process, high maintenance and costly. Alternatively, researchers used spray drying during extraction process, yet, an elevated drying temperature applied may incur some effects on the quality and quantity of the extract. Therefore, the present study was aimed to investigate the thermal effect of spray drying process on the quality of spray dried *Ficus deltoidea* (*F.deltoidea*). **Methods:** The thermal effects of spray drying were identified at three different inlet air temperatures (160 °C, 191 °C and 220 °C) which are minimum, optimum and maximum of inlet air temperature, respectively. The Box-Behnken Design through response surface methodology was utilized to identify the optimum operating conditions at these temperatures. The quality of *F. deltoidea* in terms of yield, moisture content, marker compound (vitexin), total saponins, total protein and total polysaccharides were studied. **Results:** From the study, total saponins and polysaccharides exhibited better retentions during the spray drying process. Meanwhile, vitexin and total protein was found decreasing by 30% and 50% respectively, during the spray drying process. **Conclusion:** High operating of air inlet temperatures in spray drying process contribute to higher process yield, produce non-sticky particles with lower moisture contents compared to drying process at 160 °C.

Keywords: Bioactive compounds, *Ficus deltoidea* extract, Spray dry, Thermal effect, Vitexin

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INTRODUCTION

F. deltoidea or Mas Cotek belongs to the Moraceae family. It is an epiphytic shrub plant that can be found in several countries throughout Southeast Asia. *F. deltoidea* plant is divided into two plant types which are male and female plant. Since the ancient time, the leaves of *F. deltoidea* have been used as herbal remedies in strengthening the uterus for women after childbirth, increase the blood circulation, enhance and recover the sexual desire, lessen the cholesterol, remove the toxin in body, reduce the level of blood sugar, treat migraine and delay menopause (1). It also contains phytochemical compounds namely flavonoids, tannins, phenols, triterpenoids, proanthocyanins and saponin (2-3). The studies done by researchers have revealed that *F. deltoidea* possess pharmacological effects like an anti-inflammatory (4-5), antioxidant (6-9), anti-diabetic (10-11), anti-melanogenic (2), anti-photoaging (12), antithrombotic and antinociceptive (1), enhance wound healing (13) and antihyperglycemic activity (14).

Since the shelf life and quality of the end product is an important aspect in herbal processing; therefore, the water content in the *F. deltoidea* extract should be removed before the product will be commercialized. Spray drying process is the most practice for finished product of *F. deltoidea*. It is a method of transforming liquid or slurry into a dry powder form using a hot air as a drying media. Nevetherless, the retention of phytochemicals of dried *F. deltoidea* after spray dry was questioned especially when it was operated at elevated temperature as it might cause some of thermolabile compound to be degraded.

According to our previous work on optimization of spray drying Nor Rashidah (2016), (32), the inlet air temperature was found to be the most important variables for the quality of bioactive compounds of *F. deltoidea*. This was supported in the study of the retention of anthocyanin, 6-gingerol, diadzein and

genistein, tannin and vitamin C in spray dry powder (15-18). The inlet air temperature has the capability to affect the efficiency of heat and mass transfer process. The current study focused to identify the effects of spray drying temperature towards the quality of spray dry of *F. deltoidea* extract mainly through screening process of the selected conditions. At the selected optimum conditions of spray dying, the effect of inlet air temperature on moisture content and biactive compounds retentions were determined. The range of selected conditions used was mainly to screen for the early effects on the extract before proceeding to optimizations study using Response Surface Methodology.

MATERIALS AND METHODS

Materials

HPLC - grade ortho-phosphoric acid, methanol, acetonitrile, ethanol, sulphuric acid and phenol were purchased from Merck®, USA. Escin, D-glucose and vanillin were purchased from Sigma-Aldrich®, USA. Bovine Serum Albumin, sodium hydroxide (NaOH), sodium carbonate (Na^2CO^3), copper sulphate hydrated (CuSO4.5H2O), potassium sodium tartarate ($KNaC_4H^4O_{6.}4H_2O$) and Folin-Ciocalteau purchased from Merck®, USA. The green leaves of *F. deltoidea* leaves were procured from Moro Seri Utama Enterprise, Batu Pahat, Johor (particle size 24 mesh). The leaves were identified and authenticated by a botanist and a voucher specimen (SK 2310/13) was sent to Herbarium Institute of Bioscience, Universiti Putra Malaysia, Serdang, Selangor.

Preparation of the extract

The dried leaves of *F. deltoidea* were boiled at 87°C with ratio of water to solid content 19.12:1 (w/w), agitation speed of 400 rpm for 2.71 hours on the hot plate. The filtrate was then filtered using Whatman No. 1 filter paper and the extract was kept at 4 °C until further use.

Spray Drying Conditions

The process was performed by using a lab-scale spray dryer Buchi (Model SD-04, Switzerland) with co-current flow between sprayed *F. deltoidea* extract and hot air as shown in Fig. 1. The diameter of nozzle was 0.5 mm. The feed solution was continuosly stirred and fed into the chamber through peristaltic pump.

Process Yield

In order to determine the successful of the spray drying process, the process yield was calculated using Equation 1. The total of solid content was determined in reference to AOAC standard [31].

$$\% \text{ Process yield} = \frac{\text{Mass of powder recovered}}{\text{Mass of solid content in feed solution}} \times 100 \quad (1)$$

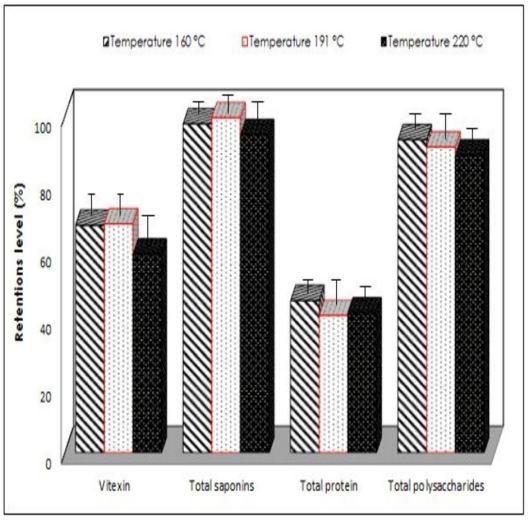

Fig. 1 : *F. deltoidea* marker compounds

Moisture Content

The moisture content was determined according to Erham (19). Ten mg of sample was weight in crucible dish and placed in vacuum oven at 105°C until a constant weight was achieved.

Vitexin Determination

Quantitative analysis of vitexin was carried out using HPLC with operating conditions that matched the previous study with slight modification (4). The HPLC used was equipped with Waters 2690 Separation Module auto sampler and Waters 996 Photodiode-array detector (Water Corporation, Milford, Massachusetts, USA). The chromatographic separation of vitexin was performed using Kinetex 5 µ Biphenyl 100 A column (150 mm x 4.6 mm, 5 µm). The column temperature was controlled at 35°C. A gradient elution consisting of solvent A (acetonitrile) and solvent B (0.1% ortho-phosphoric acid) was run at flow rate of 1 mL/min as: 0 – 11 min (5 – 35% A), 11 – 12 min (35 – 5% A), 12 – 14 min (5% A). The injection volume was 10 µl and the detection wavelength was set at 335 nm.

Total Saponins Measurement

The total saponins content of *F. deltoidea* leaves was examined using colorimetric method with slight modification (20-21). The sample was mixed with 0.5 mL of 8% (w/v) vanilin and 5 mL of 72% sulfuric acid. The mixtures were then incubated in a water bath for 10 minutes at 60°C and cooled for another 15 minutes in an ice bath. The samples were analysed using UV-Vis Spectrophotometer (Model UV-1800, Shimadzu, Japan) at absorbance of 560 nm. Escin was served as standard.

Total Protein Measurement

Determination of total protein was measured according to The Lowry's method (22). Burette reagent (3 mL) was prepared by mixing solution A (50 ml of 2% Na_2CO_3in 0.1 N NaOH) and B (1 ml of 0.5% $CuSO_4.5H_2O$ in

1% $KNaC_4H_4O_6.4H_2O$) with ratio volume of 50:1, respectively. Then, the sample or standard (1 mL) was added into burette reagent. The mixture vortexed and incubated at room temperature for 20 minutes. Then, 200 µl of Folin-Ciolcateau reagent was added and reaction mixture reincubated for another 30 minutes. Bovine Serun Albumin (BSA) was used as a standard. The samples were analyzed using UV-Vis Spectrophotometer (Model UV-1800, Shimadzu, Japan) with the absorbance reading at 750 nm.

Total polysaccharides measurement

Total polysaccharides content in F. deltoidea spray dried powder was quantified according to the phenol-sulphuric acid colorimetric assay method in which the glucose was used as a standard (21). 1 mL of extract was mixed with 4 mL of 99.5% ethanol in a plastic tube. The mixture was incubated in an ice bath for an hour. After incubation, the mixtures were centrifuged for 10 minutes at 13,000 rpm. The supernatant was removed without disturbing the pellet. The pellet was washed twice with 4 mL of 99.5% ethanol. The pellet was then air dried and dissolved in 3 mL of distilled water. Glucose stock solution (100 ppm) was prepared in distilled water and a series of dilutions of 20, 40, 60 and 80 ppm were prepared to final volume of 1 mL. For analysis, 0.5 mL of the standard and extracts solutions was mixed with 0.5 mL of 4% (w/v) of phenol. 2.5 mL of concentrated sulphuric acid was added into the mixture in ice bath. The reaction mixture was heated at temperature of 80°C for 30 minutes in water bath (Model WNE14, Memmert, Germany). After cooling to room temperature, the absorbance was measured at 492 nm against distilled water with reagent as a blank.

Statistical Analysis

The three level of Box-Behnken design with five replicates at the centre point was utilized to construct the experimental run for this study. The range of each independent variables and their coded level are displayed in Table I. Design Expert software (version 6.0.8) was used to simulate the analysis of variance (ANOVA) for all designed experiments with its statistical difference and its interactions from the p value and regression analysis of the study.

Table I : Spray drying factors and their coded level in Box-Behnken design for *F. deltoidea* extract

Factor	Unit	Factor levels		
		-1	0	+1
Inlet air temperature (A)	°C	160	191	220
Feed flow rate (B)	ml/min	2	4	6
Air pressure (C)	psi	20	40	60
Feed temperature (D)	°C	25	62.5	100

RESULTS

Optimum Conditions of Heat Treatment

According to the design developed by BBD, the operation conditions for three targeted inlet air temperatures at 160°C (minimum), 191°C (optimum) and 220°C (maximum) were determined as Table 2 where the optimum inlet air temperature provided highest process yield (10.01%), followed by minimum and highest inlet air temperature with 8.11% and 7.27% of process yield, respectively. A full quadratic model was developed in overall process from the response surface methodolgy acquire from Design Expert and is shown as in the equation below.

$$\text{Process yield} = 7.58 + 1.00A - 2.50B + 0.34C + 0.094D - 0.30A^2 + 0.22B^2 - 1.14C^2 + 0.018D^2 + 0.37AB + 0.17AC + 0.022AD - 0.083BC + 2.500E\text{-}03BD + 0.16CD$$

Powders Moisture Content

As shown in Table II, the moisture content of *F. deltoidea* spray dried powder at the inlet air temperatures of 220°C, 191°C and 160°C were 3.67%, 4.33% and 6.83%, respectively. It was demonstrated that the moisture content was lowest when it was introduced to the highest inlet air temperature as compared to the lowest inlet air temperature. Meanwhile, a full quadratic model was developed to show the moisture content interactions as shown in the equation below.

$$\text{Moisture content} = 5.30 - 1.47A + 0.47B - 0.28C + 0.19D + 0.23A^2 + 0.27B^2 - 0.66C^2 - 0.20D^2 + 0.12AB - 0.083AC + 0.21AD + 0.042BC - 0.17BD + 0.021CD$$

Table II : Operating conditions at selected inlet air temperatures of *F. deltoidea* extract spray drying process

Factor				Response		
A	B	C	D	Process yield (%)	Moisture content (%)	Total poly-saccharides (%)
160	2.00	20.00	25.00	8.11	6.83	99.5
191	2.00	28.00	25.00	10.01	4.33	97.1
220	4.00	24.00	25.00	7.27	3.67	88.5

A - Inlet air temperature (°C); B - Feed flow rate (ml/min); C - Air pressure (psi); D - Feed temperature (°C)

Marker Compounds Retention

To investigate the level of retentions of bioactive compounds after heat treatment, the bioactive compounds from *F. deltoidea* extract (feedstock) and spray dried powder were determined. The vitexin content was determined using linear regression equation ($Y = 44054x + 36625$, $R^2 = 0.9998$). Total saponins content was obtained using linear regression

equation (Y = 0.0012x + 0.175, R^2 = 0.9964), which was obtained from the calibration curve of Escin. Total protein obtained from the BSA standard curve was estimated by linear regression equation (Y = 0.002x + 0.2418, R^2 = 0.9983). Total polysaccharides were identified by linear regression equation (Y = 0.0118x + 0.0552, R^2 = 0.9967), which was obtained from glucose standard curve.

From Fig. 1, it can be seen that at all inlet air temperatures indicate that two constituents, namely saponins and polysaccharides were founds to have better percentage of retention which are vary from 88.5 to 99.5 %. On the other hand, the marker compounds of *F. deltoidea* namely vitexin was observed to degrade after sprays drying, which are 32.4%, 31.9% and 40.9% at the inlet air temperatures of 160°C, 191°C and 220°C, respectively. Same goes to the protein, which found to be denatured after spray drying process; with the retention level is ranging from 54.8% to 59.2%. The retention of protein was found the lowest in spray dried of *F.deltoidea.*

DISCUSSION

In spray drying process, operating at an optimum processing conditions that produces highest process yield is required. From this study, the optimum inlet air temperatures (191°C) is nearly in line as stated by Písecký (2005) that 180°C to 190°C of inlet air temperature is commonly used spray dryers at an industrial scale. From the ANOVA results (Table 3), the generated second order model shows the experimental data with the R^2 values of 0.9727. The significant *P*-value was achieved for this model (p<0.0001). Therefore, it means the operating parameters considered provided significant effect on the process yield of *F. deltoidea* spray dried.

Table III : ANOVA for process yield of *F. deltoidea* spray drying process

Source	SS	df	F-value	P-value	R^2
Regression	99.21	14	7.09	35.69	0.9727
Lack of fit	2.60	10	35.75	0.0532	-
Pure error	0.18	4	0.045	-	-
Residual error	2.8	14	0.20	-	-
Total	101.99	28	-	-	-

The moisture content determines the long shelf life of the product, affecting the physical properties of the product and the presence of chemical and microbiological stability of the spray dried products (23). At the higher inlet air temperature, there was a greater temperature gradient between the drying air and the atomized feed, leading to faster evaporation of water and thus producing powders with lower moisture content (15). For the pharmaceutical product including spray dried extract, the moisture content with values of lower than 5% (w/w) was considered adequate (24). Therefore, moisture content range of *F. deltoidea* spray dried was acceptable for pharmaceutical product and sufficient to say the *F. deltoidea* powder is microbiologically safe (25).

The findings of saponins and polysaccharides can be stated as thermo-resistant compound. It was proved that there was no apparent degradation occurred even subjected at elevated inlet air temperature during spray drying. This is possibly due to the evaporative cooling effect of co-current operation during the critical drying time (26). However, vice versa occur on the vitexin which are degraded over inlet air temperatures. The same results found that the flavonoid in *Rosmarinus officinalis* extract degraded up to 39.1% when 150°C of inlet spray drying temperature was used. This because of flavonoids substance exhibited antioxidant activity is highly reactive with oxygen (27). Hence, the oxidative condensation or decomposition of thermolabile compounds induced by heat may affect the degradation of flavonoids substances in *F. deltoidea*. In addition, the phytochemicals in plant might be affected by the thermal process which can affect the integrity the cell structure of plants and losses by various chemical reactions such as enzymes, oxygen and light (28).

The total protein content was found the most thermo-labile compounds among *F. deltoidea's* bioactive compounds. At an elevated temperature, the molecules of protein become flexible and increment the intermolecular collision among them cause greater probability of protein denaturation (29). Besides that, more than 70% of occurrence of protein denaturation could occur when the drying temperature was operated above 200°C due to occurrence of disruption and destruction of protein secondary and tertiary structure during the drying process (26). The other possible factor of denaturation of protein during spray drying is the atomization condition. High atomization could produce fine droplets with extremely high surface area which greatly influenced the denaturation of protein (30).

CONCLUSION

From the result of this study, it can be concluded that there was no thermal degradation happened on the bioactive compounds in *F. deltoidea* extract, namely total saponins and total polysaccharides. However, the inlet air temperature of spray drying was found led to the degradation of biomarker compound (vitexin) about 32.4% to 40.9%. Same goes to the protein compound which degraded approximately more than 50%. The degradation problem of vitexin and protein

during the spray drying process might be overcome by microencapsulate the extract using adjuvant such maltodextrin. High operating inlet air temperatures could produce better process yield, produced non-sticky product with lower moisture content.

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REFERENCES

1. Sulaiman MR, Hussain MK, Zakaria ZA, Somchit MN, Moin S, Mohamad AS, Israf, DA. Evaluation of the Antinociceptive Activity of Ficus deltoidea aqueous Extract. Fitoterapia. 2008;79:557-561.
2. Oh MJ, Hamid MA, Ngadiran S, Seo YK, Sarmidi MR, Park CS. Ficus deltoidea (Mas cotek) Extract Exerted Anti-melanogenic Activity by Preventing Tyrosinase Activity in Vitro and by Suppressing Tyrosinase Gene Expression in B16F1 Melanoma Cells. Archives of Dermatological Research. 2011;303:161-170.
3. Shafaei A, Farsi E, Ahamed BMK, Siddiqui MJA, Attitalla IH, Zhari I, Asmawi MZ. Evaluation of Toxicological and Standardization Parameters and Phytochemical Investigation of Ficus deltoidea Leaves. American Journal of Biochemistry and Molecular Biology. 2011;1(3):237-243.
4. Abdullah Z, Hussain K, Ismail Z, Ali, RM. Anti-inflammatory Activity of Standardised Extracts of Leaves of Three Varieties of Ficus deltoidea. International Journal of Pharmaceutical and Clinical Research. 2009;1(3):100-105.
5. Zakaria ZA, Hussain MK, Mohamad AS, Abdullah FC, Sulaiman MR. Anti-inflammatory Activity of the Aqueous Extract of Ficus deltoidea. Biological Research for Nursing. 2012;14(1):90-97.
6. Hakiman M, Maziah M. Non Enzymatic and Enzymatic Antioxidant Activities in Aqueous Extract of Different Ficus deltoidea Accessions. Journal of Medicinal Plants Research. 2009;3(3):120-131.
7. Wahid S, Mahmud TMM, Maziah M, Yahya A, Rahim MA. Total Phenolics Content and Antioxidant Activity of Hot Water Extracts from Dried Ficus deltoidea Leaves. Journal of Tropical Agriculture and Food Science. 2010;38(1):115-122.
8. Omar MH, Mullen W, Crozier A. Identification of Proanthocyanidin Dimers and Trimers, Flavone C-glycosides, and Antioxidants in Ficus deltoidea, a Malaysian Herbal Tea. Journal of Agricultural and Food Chemistry. 2011;59(4):1363-1369.
9. Misbah H, Aziz AA, Aminudin N. Antidiabetic and Antioxidant Properties of Ficus deltoidea Fruit Extracts and Fractions. BMC Complementary and Alternative Medicine. 2013;13(118):1-12.
10. Adam Z, Hamid M, Ismail A, Khamis S. Effect of Ficus deltoidea Aqueous Extract on Blood Glucose Level in Normal and Mild Diabetic Rats. Jurnal Sains Kesihatan Malaysia. 2007;5(2):9-16.
11. Adam Z, Khamis S, Ismail A, Hamid M. Inhibitory Properties of Ficus deltoidea on α-glucosidase Activity. Research Journal of Medicinal Plant. 2010;4(2):61-75.
12. Hasham R, Choi HK, Sarmidi MR, Park CS. Protective Effects of a Ficus deltoidea (Mas cotek) Extract against UVB-Induced Photoageing in Skin Cells. Biotechnology and Bioprocess Engineering. 2013;18:185-193.
13. Abdulla MA, Ahmed KA, Abu-luhoom FM. Role of Ficus deltoidea Extract in the Enhancement of Wound Healing in Experimental Rats. Biomedical Research. 2010;21(3):241-245.
14. Adam Z, Ismail A, Khamis S, Mokhtar MHM, Hamid M. Antihyperglycemic Activity of F. deltoidea Ethanolic Extract in Normal Rats. Sains Malaysiana. 2011;40(5):489-495.
15. Tonon RV, Brabet, C, Hubinger MD. (). Influence of Process Conditions on the Physicochemical Properties of Açai (Euterpe oleraceae Mart.) Powder Produced by Spray Drying. Journal of Food Engineering. 2008;88:411-418.
16. Jangam SV, Thorat BN. Optimization of Spray Drying of Ginger Extract. Drying Technology. 2010;28:1426-1434.
17. Couto RO, Conceição EC, Chaul LT, Oliveira EMS, Martins FS, Bara MTF, Rezenda KR, Alves SF, Paula JR. Spray-dried Rosemary Extracts: Physicochemical and Antioxidant Properties. Food Chemistry. 2012;131:99-105.
18. Patil V, Chauhan AK, Singh RP. Optimization of the Spray-drying Process for Developing Guava Powder using Response Surface Methodology. Powder Technology. 2014;253:230-236.
19. Ehrman T. Standard Test Method for Moisture, Total Solids, and Total Dissolved Solids in Biomass Slurry and Liquid Process Samples. Chemical Analysis and Testing Task, Laboratory Analytical Procedure, LAP-012. 1994.
20. Hiai S, Oura H, Nakajima T. Color Reaction of Some Sapogenins and Saponins with Vanillin and Sulfuric Acid. Plant Medica. 1976;29:116-122.
21. Chen Y, Roan H, Lii C, Huang Y. Wang T. Relationship between Antioxidant and Antiglycation Ability of Saponins, Polyphenols, and Polysaccharides in Chinese Herbal Medicines used to treat Diabetes. Journal of Medicinal Plant Research. 2011;5(11):2322-2331.
22. Lowry OH, Rosebrough NJ, Farr L. Randall RJ.

The Protein Measurement with the Folin Phenol Reagent. The Journal of Biological Chemistry. 1951;193:265-275.
23. Telang AM, Thorat BN. Optimization of Process Parameters for Spray Drying of Fermented Soy Milk. Drying Technology. 2010;28:1445-1456.
24. Couto RO, Martins FS, Chaul LT, Conceição EC, Freitas LAP, Bara MTF, Paula JR. Spray Drying of Eugenia dysenterica Extract: Effects of In-process Parameters on Product Quality. Brazilian Journal of Pharmacognosy. 2013;23(1):115-123.
25. Santhalakshmy S, Bosco SJD, Francis S, Sabeena M. Effect of Inlet Temperature on Physicochemical Properties of Spray-dried Jamun Fruit Juice Powder. Powder Technology. 2015;274:37-43.
26. Anandharamakrishnan C, Rielly CD, Stapley AGF. Effects of Process Variables on the Denaturation of Whey Proteins during Spray Drying. Drying Technology. 2007;25:799-807.
27. Souza CRF, Schiavetto IA, Thomazini FCF, Oliveira WP. Processing of Rosmarinus officinalis Linne Extract on Spray and Spouted Bed Dryers. Brazilian Journal of Chemical Engineering. 2008;25(1):59-69.
28. Davey MW, Montagu MV, Inzé D, Kanellis A, Sanmartin M, Smirnoff N, Kanellis A, Smirnoff N, Benzie IJ, Strain JJ, Favell D, Fletcher J. Review Plant L-ascorbic Acid: Chemistry, Function, Metabolism, Bioavailability and Effects of Processing. Journal of the Science of Food and Agriculture. 2000;80:825-860.
29. Haque MA, Adhikari B. Drying and Denaturation of Proteins in Spray Drying Process. In Mujumdar, A. S (Ed.). Handbook of Industrial Drying (pp. 971–983). London: CRC Press, Taylor & Francis Group. 2015.
30. Ameri M, Maa YF. Spray Drying of Biopharmaceuticals: Stability and Process Considerations. Drying Technology. 2006;24:763-76.
31. AOAC Standard, Official Method of Analysis, 14th Ed., Association of Official Analytical Chemists, Washington, D.C, 1990.
32. Nor Rashidah. Optimization of extraction and spray drying parameters on Ficus deltoidea extract, University Teknologi Malaysia, Johor, Malaysia. Retrieved from http://eprints.utm.my/id/eprint/77800/. 2016