International Food Research Journal 23(Suppl): S225-S230 (December 2016)

Journal homepage: *http://www.ifrj.upm.edu.my*

Effects of formulation on flowability of selected herbal powders using compendial methods and powder flow analyser

[1,3]Etti, C. J., [1*]Yusof, Y.A., [1]Chin, N. L. and [2]Mohd Tahir, S.

[1]*Department of Process and Food Engineering, Faculty of Engineering,Universiti Putra Malaysia*
[2]*Mechanical and Manufacturing Engineering Department, Faculty of Engineering, University Putra Malaysia, 43400 UPM, Serdang, Selangor, Malaysia*
[3]*Department of Agricultural and Food Engineering, Faculty of Engineering, University of Uyo, Uyo, Nigeria*

Article history

Received: 6 September 2016
Received in revised form: 16 September 2016
Accepted:20 September 2016

Keywords

Flowability,
Caking strength,
Formulation,
Powder flow analyser

Abstract

This study was aimed at investigating the effects of formulation on flowability of selected herbal powders which include Labisia *pumila*, *Ortosiphon stamineus*, *Eurycoma longifolia* and *Andrographis paniculata* using compendial methods and a recently available powder flow analyser. The material properties such as moisture content, particles sizes, tap and bulk densities of the pure herbal powders and the formulated powders were measured to determine Carr index and Hausner ratio which were indices of the compendial methods. Cohesion index and caking strength were used as basis of flowability indicator using the powder flow analyser. The flowability of the herbal powders were improved when they were formulated into beverages mix by mixing each herbal powders with other ingredients like sugar and nondairy creamer before analyzed using both the compendial methods and powder flow analyser. Both methods of flowability measurements adopted in this work complemented each other in the understanding and characterization of powder flowability.

Introduction

The necessity for reliable predictions of flowability behaviour in an array of unit operations such as blending, compression, filling, transportation and in scale up operations is high and these reliable predictions require proper information on the flowability and processing behaviour of the concerned powders (Rasanen *et al*., 2003). Understanding of the flowability behaviour of powders through a given process is very essential in the industry where the flow behaviour of powders can change resulting in stoppages or poor quality products (Rasanen *et al*., 2003). Generally, there are many methods available to measure powder flowability but no single test can be said to be a standard for all powders in flowability measurements (Shah *et al*., 2008). Some of the methods include the compendial methods such as angle of repose (USP 1174, 2007), bulk and tapped density (USP 616, 2007), Carr index (Carr, 1965), and Hausner ratio (Hausner, 1967). Some other technological advanced and innovative methods of powder flow characterization are cohesive determination (Faqih *et al*., 2006), avalanching determination (Hancock *et al*., 2004), shear cell (Jenike, 1964), dielectric imaging (Dyakowsk *et al*., 1999), atomic force microscopy (Weth *et al*., 2001), and penetrometry (Zatloukal and Sklubalova, 2007). These various methods have limitations such as reproducibility, predictability and performance conditions depending on the nature of the powders and the environmental conditions in which the measurements are being carried out (Shah *et al*., 2008) . Evaluation of these methods on a particular powder or powders can enable proper understanding and description of flowability characteristics. Herbal powders are very useful medicinally and they often have very small particle sizes making them to be very cohesive to flow (Fitzpatrick *et al*., 2004). Formulating the herbal powders by mixing with other materials which have relative higher particle sizes will improve the cohesive nature of the herbal powders hence, improving the handling and processing operations which are common challenges to process operatives (Fitzpatrick *et al*., 2004), making the nutritional benefits of the resulting products to be improved as well. The powder flow analyser can analyze properties like the caking strength and cohesion index of the powders. Mixing the herbal powders with other food products of high nutritional values will enhance the

*Corresponding author.
Email: *yus.aniza@upm.edu.my*

economic values of the herbal powders by making them to be of higher nutritional benefit for human consumption with safety assurance not undermining flow difficulties and with ease of human usage of the new products. The compendial methods and the powder flow analyser will help to complement each other and give a better understanding of flowability characterization of the selected herbal powders and their formulations.

The aim of this study was to formulate the various selected herbal powders such as *Labisia pumila* (*L. pumila*), *Ortosiphon stamineus* (*O. stamineus*), *Eurycoma longifolia* (*E. longifolia*) and *Andrographis paniculata* (*A. paniculata*) of smaller particle sizes (less than 100 µm), into herbal powder mix by mixing them with other materials like sugar and creamer which have bigger particle sizes (see Table 1) to improve the cohesive nature of the herbal powders and hence, improving the flowability characteristics of the powders, because, smaller particle size makes flow to be difficult (Fitzpatrick *et al*., 2007). Also, to evaluate the herbal powders and the formulations using compendial methods and powder flow analyser.

Materials and Methods

The herbal powders such as *L. pumila*, *O. stamineus*, *E. longifolia* and *A. paniculata* powders and a freeze dried Labisia *pumila* extract (*L. pumila* ext.) powders were used in this study. These herbal materials were acquired from Ethno Resources Sdn. Bhd., Sungai Buloh, Malaysia. The powders were produced after the herbs were oven dried and ground to smaller standard particle sizes and then the freeze dried *L. pumila* ext. powder was made by water extraction after which the extract was freeze dried to obtain the powder. The material properties used in this work are summarized in Table (1). Sugar and the nondairy creamer were bought from a supermarket (KK) in Malaysia with particle size (D0.5) of 349.1 ±1.88 µm and 190.7 ±2.15 µm respectively. The 3 in 1 commercial coffee acquired had the particle size (D0.5) of 131.5 ±1.45 µm.

Bulk and tapped densities

Bulk density can be identified as the volume occupied by the solid plus the volume of voids when divided into powder. Whereas, tap density is a different type of bulk density obtained by tapping or vibrating the container in a particular method to achieve more effective particle parking and therefore, it is usually higher than bulk density (Lowell and Shields, 1984);

$$\rho_b = \left(\frac{w_t}{V_b}\right) \quad (1)$$

where w_t is weight of the powder, V_b is volume of the powder obtained from tarred graduated cylinder without tapping. The tapped density (ρ_{tap}) of the powders was calculated by using the following equation;

$$\rho_{tab} = \left(\frac{w_t}{V_{ta}}\right) \quad (2)$$

where w_t is the weight of powder, V_{ta} is the volume of the powder bed after 500 taps.

Carr index and Hausner ratio

Carr Index and Hausner Ratio are used in describing the flowability of powder. Carr Index (*CI.*) can be determined as the ratio of the difference of the tapped and the bulk densities to the tapped density (Carr, 1965). According to Carr (1965), who introduced the flowability index, an excellent flowability is between the Carr Index of 5% to 15% while Carr Index of above 25% normally shows poor flowability.

$$CI. = \frac{\rho_{tap} - \rho_b}{\rho_{tap}} \quad (3)$$

Hausner Ratio (*HR*) was also used to characterize the flowability of the powder, which can be determined by the ratio of the tapped density to that of bulk density (Hausner, 1967). *HR* of 1.0 to 1.1, powder was considered as free flowing, *HR* greater than 1.1 to 1.25, powder was classified as medium flowing, *HR* greater than 1.25 to 1.4, the powder was classified as difficult to flow and *HR* higher than 1.4, powder was considered to be very difficult to flow (Hayes, 1987);

$$HR = \frac{\rho_{tap}}{\rho_b} \quad (4)$$

Powder flow analyser

The flowability characteristics such as caking strength and cohesion index of the herbal powders and formulation were determined using a powder flow analyzer (TA.HD plus, Stable Micro Systems, Surrey, UK). The powder flow analyser was made up of a vertical glass container (120mm height and 50mm internal diameter) and a rotating blade (48mm diameter and 10mm height), which can move up and down, in right or left cycle (Landillon *et al*., 2008). The flowability characteristics of the samples were determined by the movement of the cycling blade inside the container that contained the powder sample (Mukherjee and Bhattacharya, 2006; Etti *et al*., 2014)

Particle size

Particle size and particle size distribution for all the herbal materials were measured by a particle size analyzer (Malvern instruments Ltd, Worcestershire, UK). Approximately 5ml of powder was used for each measurement and particle size distributions were recorded. The samples were measured and recorded in triplicate, and the results are shown in Tables 1 and 2.

Table 1 Material properties of pure herbal powders used

Materials	Moisture content (% dry basis)	Particle size (D0.5)	Density (kg/m^3)	
			Bulk	Tap
O. stamineus	8.2 (±0.2)	56.3 (±0.0)	288.4 (±0.6)	496.4 (±1.4)
E. longifolia	7.6 (±0.2)	57.7 (±0.0)	287.6 (±1.5)	441.0 (±1.1)
L. pumila	8.2 (±0.1)	27.5 (±0.7)	306.4 (±1.1)	523.3 (±8.4)
L. pumila ext.	6.7 (±0.1)	80.7 (±1.0)	645.1 (±1.8)	798.6 (±3.7)
A. paniculata	9.1 (±0.5)	50.3 (±0.4)	332.7 (±1.5)	541.6 (±1.4)
Sugar	0.7 (±0.0)	349.1 (±1.9)	881.5 (±1.8)	1004.3 (±0.5)
Non-dairy creamer	-	190.7 (±2.15)	647 (±1.5)	834.6 (±0.9)
Commercial 3 in 1 Coffee	2.3(±0.5)	131.5 (±1.45)	532.0 (±1.6)	652.0 (±1.8)

(* Standard deviation values are placed in the bracket)

Table 2 Material properties of formulated herbal powder mix

Herbal powder mix	Moisture content (% dry basis)	Particle size (D0.5)	Bulk density (kg/m^3)	Tap density (kg/m^3)
O. stamineus	2.09 (±0.32)	253.7 (±0.1)	657.4 (±0.6)	803.7 (±2.8)
L. pumila	2.29 (±0.15)	251.7 (±8.8)	640.2 (±1.3)	818.7 (±0.7)
L. pumila ext	2.15 (±0.23)	256.4 (±0.7)	617.2 (±0.9)	761.2 (±2.7)
A. paniculata	2.12 (±0.09)	255.7 (±3.5)	661.2 (±2.1)	799.0 (±3.1)
E. longifolia	2.51 (±0.09)	131.5 (±1.5)	647.2 (±1.3)	789.1 (±2.2)

Results and Discussion

The herbal powders were formulated into beverages mix by mixing the herbal powders with sugar and nondairy creamer. The taste, flowability, nutritional benefits, level of toxicity of the herbal powders and the therapeutic benefits of the beverages were considered in the course of the herbal beverage formulation. The formulation of the herbal powders into herbal powder mix actually enhanced the particle sizes and the material properties of the herbal powder mix products. Considering the compendial method, Table 2 shows material properties of herbal powder mix and Table 3 shows the flow behaviour of pure herbal powder and the herbal powder mix. It was observed that based on Carr index (1965) and Hausner ratio (1967), all the formulated powder mix were "Free flowing" in their flowability properties compared with the material properties of pure herbal materials used (see Table 3) which were mostly highly cohesive to flow. The free flowing nature of the formulated beverage will reduce the cost of controlling caking problems in the industry which is also the target of this research.

The material properties of pure herbal powders, sugar, non-dairy creamer and commercial 3 in 1 coffee are presented in Table 1. Analysis of Table 1 with the compendial methods showed the pure herbal powders to be mostly "Cohesive", except for *L. pumila* ext. and commercial 3 in 1 coffee which were both "Free flowing" (see Table 3). Tables 1 and 2 also show the result of particle size analysis (D0.5) of pure herbal powders, sugar, creamer and the formulated herbal powder beverage mix products.

The particle size of the formulated herbal powder mix were enhanced unlike that of the pure powders (see Tables 1 and 2) which were made up of smaller particle sizes. The smaller the particle size, the more difficult it is for the particles to flow (Fitzpatrick *et al*., 2007). All the pure herbal powders had their particle sizes below 100µm which made them to be cohesive as seen in Table 1, and this result is in line with the work of Fitzpatrick *et al*. (2004), on flow property measurement of food powders which they observed that the difficult flow nature of many powders may be because of smallness in powders particle sizes. Hence, the small particles will tend to form binding thereby making the flow to be difficult (Li *et al*., 2004).

Table 4 shows the Cohesion index and flow behaviour of pure herbal powders and herbal powder mix using the powder flow analyser. The flowability properties for the different herbal powders were improved after the formulation of the powders into beverage mix. From Table 4, it was noticed that *O. stamineus*, *E. longifolia* and *L. pumila* powders that were "cohesive to flow" based on (Benkovic and Bauman, 2009) categorization scale became "Free flowing" after they were formulated into herbal powder mix (see Table 4). These improvements in flowability came mainly as a result of improvement

Table 3 Flow behavior of pure herbal powder and herbal powder mix using the compendial method of measurements

	Pure herbal powder			Herbal powder mix		
	Carr index (1965) (CI) (%)	Hausner ratio (1967) (HR)	Flow behaviour	Carr index (1965) (CI) (%)	Hausner ratio (1967)(HR)	Flow behaviour
O. stamineus	41.91 (±0.0)	1.72 (±0.03)	Highly cohesive	18.20 (±0.09)	1.22 (±0.53)	Free flowing
L. Pumila ext.	19.22 (±0.5)	1.24 (±0.02)	Free flowing	18.92 (±0.03)	1.23 (±0.65)	Free flowing
A. paniculata	38.58 (±0.0)	1.63 (±0.01)	Cohesive	17.25 (±1.02)	1.21 (±0.98)	Free flowing
E. longifolia	34.78 (±0.3)	1.53 (0.01)	Cohesive	17.98 (±0.33)	1.22 (±0.76)	Free flowing
L. pumila	41.44 (±0.8)	1.71 (±0.06)	Highly cohesive	21.81 (±0.75)	1.28 (±0.32)	Free flowing
Commercial 3in1 Coffee	18.41 (±0.8)	1.23 (±0.97)	Free flowing	-	-	-

(* Standard deviation values are placed in the bracket, Flow behavior was from categorization scale based on Cohesion Index by Benkovic and Bauman, 2009)

Table 4 Cohesion index and flow behaviour of pure herbal powders and herbal powder mix using the Powder flow analyser

	Pure herbal powder		Herbal powder mix	
	Cohesion index	Flow behaviour	Cohesion index	Flow behaviour
O. stamineus	15.4 (±1.2)	Cohesive	12.5(±0.8)	Free flowing
L. Pumila ext.	9.9(±0.9)	Free flowing	10.2(±0.5)	Free flowing
A. paniculata	13.7(±1.9)	Easy flowing	12.2(±1.1)	Free flowing
E. longifolia	15.2(±0.9)	cohesive	11.2(±0.9)	Free flowing
L. pumila	15.7 (±0.5)	cohesive	13.3(±0.5)	Free flowing
Commercial 3in1 Coffee	13.0(±1.2)	Free flowing	-	-

(* Standard deviation values are placed in the bracket, Flow behavior was from categorization scale based on Cohesion Index by Benkovic and Bauman, 2009)

in particle size during the herbal powder formulation and which in turn influenced the cohesion index and physical properties of the herbal powders (see Tables 1 and 2). Both results from compendial methods and powder flow analyser agree with each other. Using the flow behaviour categorization scale based on Cohesion Index by (Benkovic and Bauman, 2009), *L. pumila* ext. was free flowing with Cohesion Index of 9.9 ± 0.9, followed by *A. paniculata* powder which was Easy flowing with 13.7 ± 1.9 Cohesion Index. The rest of the herbal powders were cohesive based on the categorization scale used. The results on flowability behavior of powders using powder flow analyser (see Table 4) was seen to have been influenced by the particle size and material properties of the powders. Particle size and moisture content sometimes have a combined effect on the flowability of the powder. Considering a particular powder, reducing the particle size may have a tendency to reduce flowability, because of particle surface area per unit mass increase, making available a greater surface area for surface cohesive forces to interact resulting in more cohesive flow (Li *et al*., 2004). This result is seen with *L. pumila* ext. having the highest particle size amongst the pure herbal powders becoming the freest with the least cohesion index of 9.9 ± 0.9 as can be seen from Table 4. Also, increasing moisture content may tend to make powder cohesive; nevertheless, above certain moisture contents the moisture may be acting as lubricant thereby improving the flowability. This may also show why *A. paniculata* with the highest moisture content amongst the pure herbs (9.1 ±0.5) % dry basis (see Table 1) was an easy flowing powder base on powder flow analyser (Tables 4). This result corresponds with the work of (Fitzpatrick *et al*., 2004), on flow property measurement of food powders and sensitivity of Jenike's hopper design methodology to the measured values. Their investigation result showed that difficult flow nature of many powders may be because of fine nature of the powders particle sizes and high moisture content even though significant relationship is not established between physical properties and flow index (Fitzpatrick *et al*., 2004). This un-established relationship makes it difficult to compare flowability of herbal powders based on their physical properties.

Figure 1 shows the result of the mean caking strength of the formulated herbal powder beverage mix and that of normal herbal powders analyzed from the powder flow analyser. The mean caking strengths were represented by the heights of the bar charts. The result showed that the bars of the normal herbal powders were significantly higher than that of their formulated beverage mix, meaning that the formulated herbal powder mix reduced the caking strength of the normal herbal powders. It also shows that the flowability of the formulated herbal powders mix was better than that of the pure herbal powders. Hence, the flowability characteristics of the powders were enhanced after formulating the powders into herbal powder mix. This result further support the findings of compendial methods of measurements and that of cohesion index from powder flow analyser.

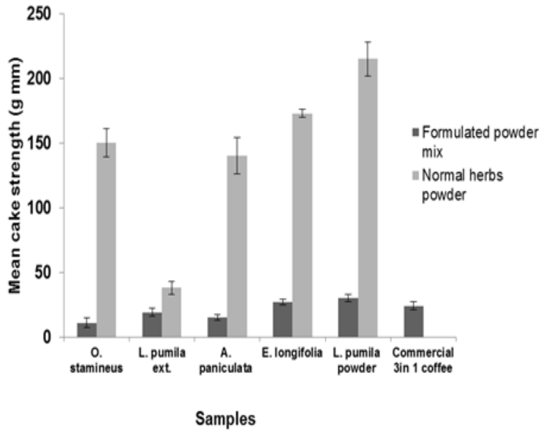

Figure 1. Mean caking strength of pure herbal powders and formulated powder mix using powder flow analyser

Conclusion

The cohesive flowability characteristics of Labisia *pumila*, *Ortosiphon stamineus*, *Eurycoma longifolia* and *Andrographis paniculata* powders were improved to free flowing powders after they were formulated with sugar and nondairy creamer. The evaluated result of flowability was similar in both the compendial methods and powder flow analyser. The caking strength of the pure herbal powders was reduced after their formulation making the formulated herbal powder mix products to be reliable in terms of flow.

Acknowledgements

The authors would like to thank Universiti Putra Malaysia for the Research University Grant Scheme with Vot Number: 937800. Thanks to University of Uyo, Nigeria for the scholarship provided to one of the authors.

References

Benkovic, M. and Bauman, I. 2009. Flow Properties of Commercial Infant Formula Powders. World Academy of Science, Engineering and Technology 54: 495-499.

Carr, R. L. 1965. Evaluating flow properties of solids. Chemical Engineering 72: 163-168.

Dyakowski, T., Luke, S. P., Ostrowski, K. L. and Williams, R. A. 1999. On-line monitoring of dense phase flow using real time dielectric imaging. Powder Technology 104: 287-295.

Etti, C. J., Yusof, Y. A., Chin, N. L. and Tahir, S. M. 2014. Flowability Properties of Labisia *pumila* Herbal Powder. Agriculture and Agricultural Science Procedia 2: 120-127.

Faqih, A., Chaudhuri, B., Alexander, A. W., Davies, C., Muzzio, F. J. and Silvina, T. M. 2006. An experimental/computational approach for examining unconfined cohesive powder flow. International Journal of Pharmaceutics 324: 116–127.

Fitzpatrick, J., Barringer, S. and Iqbal, T. 2004. Flow property measurement of food powders and sensitivity of Jenike's hopper design methodology to the measured values. Journal of Food Engineering 61(3): 399-405.

Fitzpatrick, J., Hodnett, M., Twomey, M., Cerqueira, P. S. and O'flynn, J. 2007. Glass transition and the flowability and caking of powders containing amorphous lactose. Powder Technology 178: 119-128.

Hancock, B. C., Vukovinsky, K. E., Brolley, B., Grimsey, I., Hedden, D., Olsofsky, A. and Doherty, R. A. 2004. Development of a robust procedure for assessing powder flow using a commercial avalanche testing instrument. Journal of Pharmaceutical and Biomedical Analysis 35: 979– 990.

Hausner, H. H. 1967. Friction conditions in a mass of metal powder. International Journal of Powder Metallurgy 3(4): 7-13.

Jenike, A. W. 1964. Storage and flow of solids Bulletin No. 123. Utah, USA: Utah Engineering Station Bulletin, University of Utah.

Landillon, V., Cassan, D., Morel, M. H. and Cuq, B. 2008. Flowability, cohesive and granulation properties of wheat powders. Journal of Food Engineering 86: 178-193.

Li, Q., Rudolph, V., Weigl, B. and Earl, A. 2004. Interparticle van der Waals force in powder flowability and compactibility. International Journal of Pharmaceutics 280: 77-93.

Lowell, S. and Shields, J. E. 1984. Density measurement. In Scarlett, B. (Ed.) Powder Surface Area and Porosity, p. 217-221. New York: Chapman and Hall

Mukherjee, S. and Bhattacharya, S. 2006. Characterization of agglomeration process as a function of moisture content using a model food powder. Journal of Texture Studies 37: 35-48.

Rasanen, E., Antikainen, O. and Yliruusi, J. 2003. A new method to predict flowability using a microscale fluid bed. AAPS PharmSciTech 4: 418-424.

Shah, R. B., Tawakkul, M. A. and Khan, M. A. 2008. Comparative Evaluation of Flow for Pharmaceutical Powders and Granules. AAPS PharmSciTech 9(1): 250-258.

The United States Pharmacopieal Convention (USP). 2007. Powder flow (1174). Retrieved from http://www.pharmacopeia.cn/v29240/usp29nf24s0_c1174.html

The United States Pharmacopieal Convention (USP). 2007. Bulk density and tapped density (616). http://www.usp.org/sites/default/files/usp_pdf/EN/USPNF/revisions/m99375-bulk_density_and_tapped_density_of_powders.pdf

Weth, M., Hoffman, M., Kuhn, J. and Frick, J. 2001. Measurement of attractive forces between single aerogel powder particles and the correlation with powder flow. Journal of Non-Crystalline Solids 285: 236–243.

Zatloukal, Z. and Sklubalova, Z. 2007. Penetrometry and estimation of the flow rate of powder excipients. Pharmazie 62: 185–189.