

The mixing of cohesive and flowable powder materials using a common laboratory powder mixer

¹Mohd Radzuan, N., ^{1,*}Anuar, M.S. and ²Tahir, S.M.

¹Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia.

²Department of Mechanical and Manufacturing Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia.

Article history:

Received: 10 April 2020

Received in revised form: 22 May 2020

Accepted: 15 July 2020

Available Online: 3 January 2021

Keywords:

Cohesive,
Mixing,
Flowability,
Tumblers,
Homogeneity,
Drum mixers

DOI:

[https://doi.org/10.26656/fr.2017.5\(S1\).004](https://doi.org/10.26656/fr.2017.5(S1).004)

Abstract

This study presented the homogeneity obtained when mixing cohesive and flowable powder materials using a laboratory powder mixer. The mixing process parameters studied were the mixing time and the mixer rotational speed (20 rpm, 40 rpm and 60 rpm) at the different ratios (95%: 5%, 50%: 50% and 5%: 95%) of the cohesive cocoa and flowable mannitol powder materials. The homogeneity sampled at the powder bed surface showed that only at the highest rotational speed of 60 rpm used in this work yield acceptable homogeneity at the two extremes of the powder mass ratios; 95%: 5% and 5%: 95% of mannitol: cocoa for some of the locations on the powder bed surface, especially near the wall of the mixer. Other combinations of the experimental conditions did not yield acceptable mixture homogeneity. These results showed the difficulties in obtaining a homogeneous powder mix when mixing cohesive powder materials, especially in academic teaching and research laboratories using a simple powder mixer apparatus.

1. Introduction

Powder mixing is a frequently encountered unit operation in many processes (Asachi and Nourafkan, 2018). Powder mixing is a complex process that is dependent upon the type of equipment used and the raw material to be mix (Bridgwater, 2012). Recently, a number of works attempted to elucidate the complex mechanisms of powder mixing using computer simulations to gain further understanding of the individual particle movements during the mixing process (Gui *et al.*, 2010; Zhang *et al.*, 2017; Zhang *et al.*, 2019). This is to further support the needs of the industry where uniformity of the powder mixture is crucial such as in the pharmaceutical industry (Muzzio *et al.*, 2002). The industrial use of continuous in-line noninvasive techniques to monitor powder mixture uniformity (Gerich *et al.*, 2017) reflects the importance of the mixing stage for commercial production to ensure the final product uniformity is within the acceptable regulatory limits (Cholayudth, 2017). Meanwhile, all of these were seen to be more geared towards the industrial needs, one must not neglect the importance of powder mixture preparation for smaller-scale use such as for academic teaching and research laboratories. These

laboratories sometimes were not equipped with sophisticated equipment for powder mixing purposes. In some cases, powder mixing was conducted manually through the use of dilution, manual shaking of containers containing the powder mix or simply hand mixing using a spatula inside a container. In our laboratory, we utilized a laboratory powder rotator for the mixing of the powder materials where these powder mixtures were subsequently compacted into tablet form.

The powder flowability has a large influence on the mixing performance of the powder (Chaudhuri *et al.*, 2006). Cohesive powders are normally difficult to mix and need special equipment for mixing. Therefore, this work is aimed to highlight the difficulties in achieving homogeneity when mixing with a cohesive powder using a common laboratory powder mixer.

2. Materials and methods

2.1 Raw material

Mannitol and cocoa powders were used in this mixing experiment. The source of the material could not be disclosed in this work due to proprietary reasons.

*Corresponding author.

Email: mshamsul@upm.edu.my

2.2 Raw material physical properties characterization

2.2.1 Particle size

The particle sizes of the cocoa and mannitol powders materials were determined by using a laser light diffraction equipment (Malvern Mastersizer 2000, Malvern, Malvern Instrument Ltd, UK). The median particle size (d_{50}), was used as the parameter to represent the particle size of the powders used in this work.

2.2.2 Tapped density

About 2 g of each cocoa and mannitol powder samples was weighed and inserted into the 10 mL graduated cylinder and dropped 120 times on a flat rubber mat from a height of 15 cm. Afterwards, the sample was then inserted into the tap density analyzer (GeoPyc 1360, Micromeritics, Norcross, USA) to obtain the tapped density of the powder sample (Shamjuddin *et al.*, 2014).

2.2.3 Bulk density

About 2.5 g of each cocoa and mannitol powder samples were loaded into 15 mL graduated cylinder and placed onto a flat and smooth surface. The bulk density was then calculated using by dividing the mass of the powder with the volume of the powder bed (Shamjuddin *et al.*, 2014).

2.2.4 Flowability assessment

The flowability of each cocoa and mannitol powder materials was assessed using the simple angle of repose and Carr index values (Carr, 1965; Anuar *et al.*, 2019). These methods are commonly used to characterize the flowability of powders (Shah *et al.*, 2008). For the measurement of the angle of repose, the powder was poured inside a funnel at a fixed height of 10 cm. The powder would then flow through the funnel to form a heap on the flat horizontal surface below the funnel. The angle of the heap was then measured and taken as the angle of repose for the powder. Meanwhile, the Carr index was calculated using the values of the tapped and bulk densities by using:

$$CI = 100 \times \frac{\rho_t - \rho_b}{\rho_t} \quad (1)$$

Where CI is the Carr index, ρ_t is the tapped density, and ρ_b is the bulk density.

2.3 Raw material preparation for mixing experiment

Cocoa and the mannitol powders were mixed in three different mass ratios (Table 1) consisting of 50%: 50% mass ratio of the mannitol powder with the cocoa powder, 95%: 5% mass ratio of the mannitol powder with the cocoa powder and finally 5%: 95% mass ratio of the mannitol powder with the cocoa powder.

Table 1. Raw material preparation.

Mass ratio of mixture	Mass of pure material (g)	
	Mannitol powder	Cocoa powder
50% mannitol: 50% cocoa	100	100
95% mannitol: 5% cocoa	190	10
5% mannitol: 95% cocoa	10	190

2.4 Mixing experiment

The powder samples were then placed into a laboratory powder mixer (Dry Powder Rotator, Glas-Col, Terre Haute, USA) according to the required mass ratios as stated in Table 1. The cylindrical mixing container has a diameter of approximately 8 cm with a height of 20 cm and was filled approximately at 30% capacity. The mixing action of the laboratory powder mixer involved the rotation of the mixing container at an axis thus the powder mixture was tossed from end-to-end inside it. The mannitol powder was inserted first into the mixing container. This was then followed by the insertion of the cocoa powder on top of the mannitol powder. The total mass of the powder mixture in the mixer was 200 g for each mixing run (Table 1). The mixing process was conducted for a total of 5 minutes mixing time at various constant rotational speeds of 20, 40 and 60 rpm. The mixing process was interrupted every one minute in order to examine the homogeneity of the mixture. Samples were collected at four sampling positions (Figure 1) within the mixer to ensure a thorough characterization of the homogeneity of the powder mixture. Position 1 represented the sampling position taken in the middle of the mixer. Position 2 and 3 were sampling positions near the wall of the mixer. Meanwhile, position 4 was the area between the middle and wall of the mixer. The samples were drawn at the surface of the powder bed at these positions as depicted in Figure 1. These positions were chosen by considering areas that might have different shear conditions during the mixing process. Three samples having a mass of approximately 1 g each were withdrawn from each position at one-minute time interval during the 5-minute mixing process.

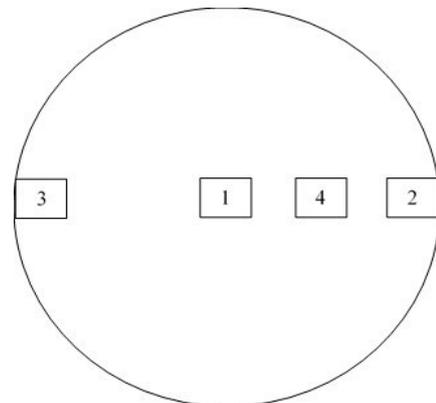


Figure 1. Sampling position in the mixer (top view).

2.5 Mixture homogeneity quantitative measurement

The homogeneity of the powder mixture was determined by analyzing the homogeneity of samples taken at the different positions in the mixer during the mixing process as discussed in Section 2.4 previously. Sample (1 g) taken was then dissolved in a 15 mL distilled water and stirred for 2 mins. The dissolved mixture was filtrated using a dried filter paper (Permula Chemical, Malaysia). The dried filter paper was weighed before conducting the filtration process. The undissolved sample was trapped on the dry filter paper during this filtration process. Subsequently, the drying of the filter paper containing the undissolved sample was conducted in an oven (Memmert, Germany) at the temperature of 105°C for 24 hrs. After drying, the dried filter paper along with the dried undissolved sample was weighed. Mass of the undissolved sample was calculated by:

$$m_{us} = (m_{wf} - m_{df}) \quad (2)$$

Where m_{us} is the mass of the undissolved sample after drying, m_{wf} is the total mass of the dried filter with the dried filtered sample on it and m_{df} is the mass of the dried filter paper prior to the filtration process.

In order to obtain the homogeneity of the samples taken from the mixer, the mass of the undissolved sample would then be compared with the mass of the undissolved sample in a known mass ratio of the mannitol and cocoa powders, which was set as the targeted homogeneity for comparison purposes. For approximately 1 g of mixing 50% of mannitol with 50% cocoa powder, the amount obtained for the targeted homogeneity was 0.570 g mass of the undissolved sample. Therefore, 0.570 g of undissolved sample was set as the target specification with an acceptable range of 0.570 g \pm 15%. Meanwhile, the target specification with an acceptable range 0.202 g \pm 15% was set for the mixing of 95% mannitol with 5% cocoa powder. For the mixing of 5% mannitol with 95% cocoa powder, the target specification with an acceptable range is 0.713 g \pm 15% was set. This was a first-order analysis of the homogeneity of the samples, which required no additional chemicals and sophisticated analytical tools.

3. Results and discussion

Generally, the cocoa powder used in this work had a smaller median particle size in comparison with the mannitol powder sample as indicated in Table 2. The cocoa powder exhibited very poor flowability characteristics that could be seen through the relatively higher angle of repose and Carr index values (Table 2) compared to the mannitol powder. In contrast, the

mannitol powder possessed better flowability characteristics represented by the lower values of the angle of repose and the Carr index compared to cocoa powder as shown in Table 2. These wide variations between both the powder materials, where the cocoa powder not just having relatively smaller particle sizes but also very cohesive in comparison to the mannitol powder was deemed suitable to be used as a model material to be mixed with the fairly flowable mannitol powder in this study.

Table 1. Raw material preparation.

Material properties	Cocoa powder	Mannitol powder
Bulk density (kg/m ³)	330.94 \pm 0.03	541.57 \pm 0.04
Tapped density (kg/m ³)	657.70 \pm 0.003	646.00 \pm 0.002
Median particle size (μ m)	12.27 \pm 0.90	345.89 \pm 0.60
Angle of repose (°)	44.33 \pm 0.05	36.33 \pm 0.08
Carr's index	49.68	16.17
Flowability	Extremely poor	Fairly flowable

Figures 2-10 illustrate the homogeneity of the cocoa-mannitol powder mixed at different mass ratios and mixer rotational speeds. It can be clearly observed from Figures 2-10, the best homogeneity that can be achieved was when there was a higher mass of the cohesive cocoa powder (95%) compared to the flowable mannitol powder (5%) at the highest rotational speed of 60 rpm. The homogeneity was achieved within the acceptable range in the initial duration of the mixing for some of the positions on the surface of the powder bed, where towards the end of the mixing process, none of the mixture sampled at the surface of the bed achieved homogeneity within the acceptable limit (Figure 2). Other conditions that achieved the required homogeneity was when the lowest amount of cohesive cocoa powder (5%) and highest amount of flowable mannitol powder (95%) were used also at the highest rotational speed of 60 rpm. The similarity for both conditions was that the acceptable homogeneity was observed for samples obtained from the powder bed surface near the wall of the mixer. This finding could be explained based upon a previous study, where the zone near the wall was found to experience relatively higher particle movements during mixing (Soni *et al.*, 2016) hence this might contribute to the observed homogeneity found for the powder bed surface near the wall. In addition, the use of the highest rotational speeds resulted in the mixture meeting the required homogeneity limit at both mixture compositions. For all other mixture composition and rotational speed combinations, none of the positions sampled on the powder bed surface achieved the acceptable homogeneity (Figures 3-10). Also, based upon the general trend observed, the increase in the mixing time led to a more 'uniform' powder bed,

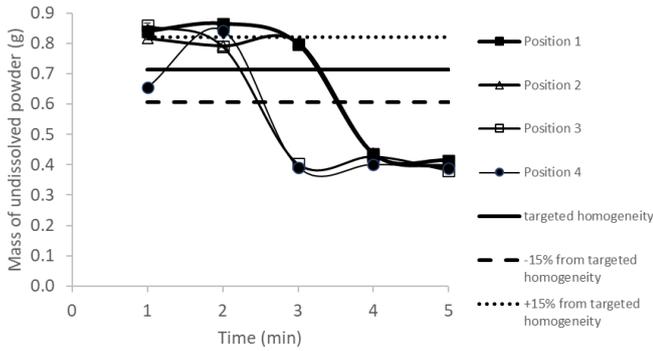


Figure 2. Homogeneity of 5% mannitol: 95% cocoa mixture at 60 rpm mixing speed.

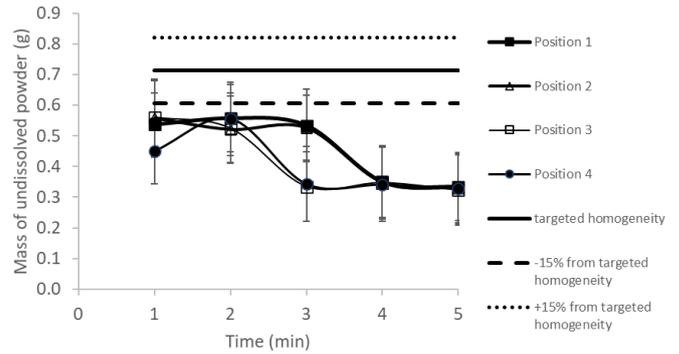


Figure 3. Homogeneity of 5% mannitol: 95% cocoa mixture at 40 rpm mixing speed.

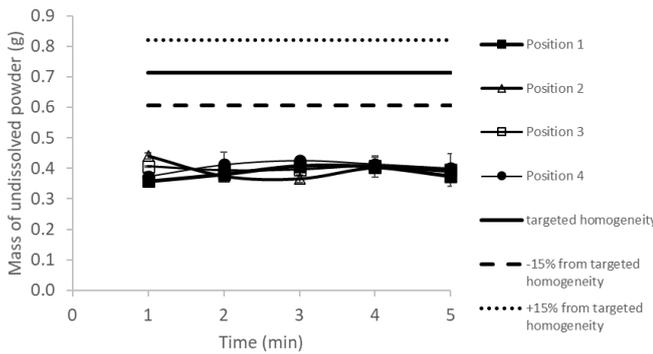


Figure 4. Homogeneity of 5% mannitol: 95% cocoa mixture at 20 rpm mixing speed.

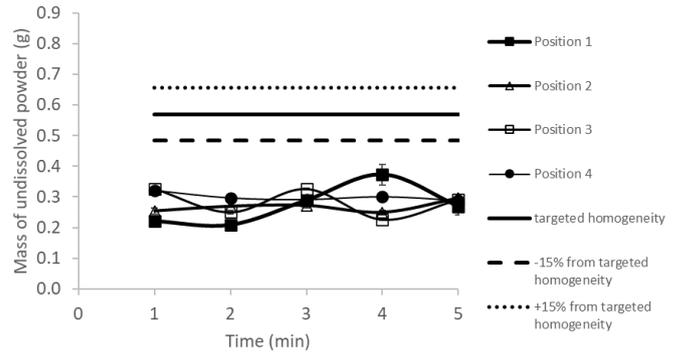


Figure 5. Homogeneity of 50% mannitol: 50% cocoa mixture at 60 rpm mixing speed.

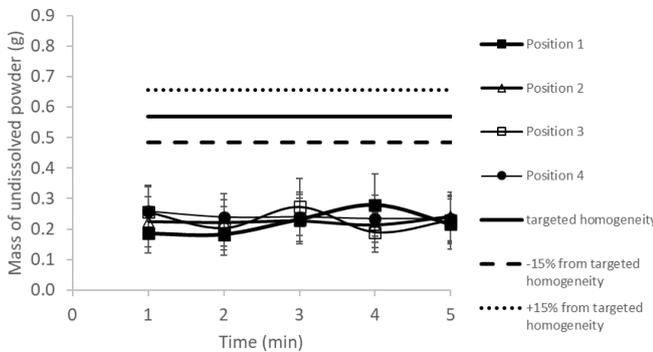


Figure 6. Homogeneity of 50% mannitol: 50% cocoa mixture at 40 rpm mixing speed.

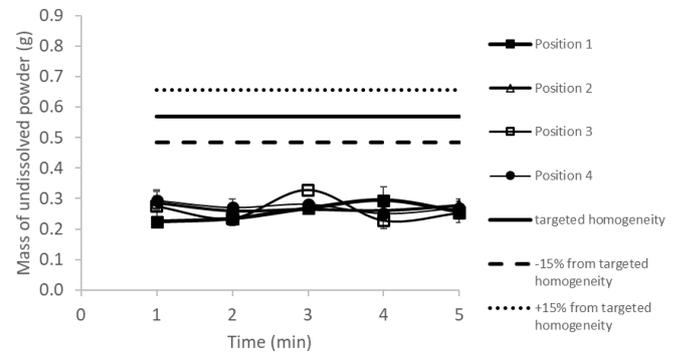


Figure 7. Homogeneity of 50% mannitol: 50% cocoa mixture at 20 rpm mixing speed.

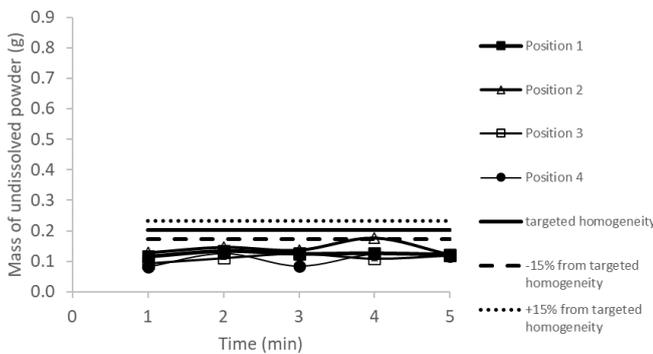


Figure 8. Homogeneity of 95% mannitol: 5% cocoa at 60 rpm mixing speed.

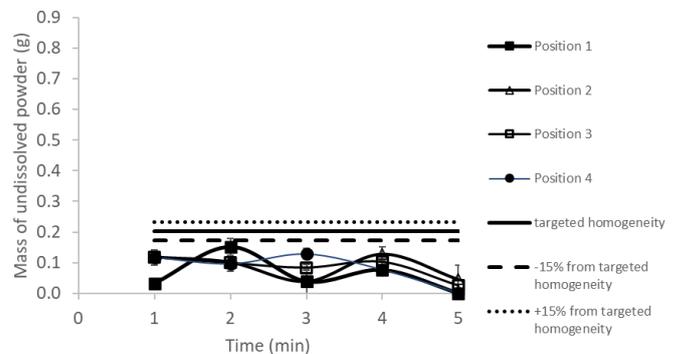


Figure 9. Homogeneity of 95% mannitol: 5% cocoa mixture at 40 rpm mixing speed.

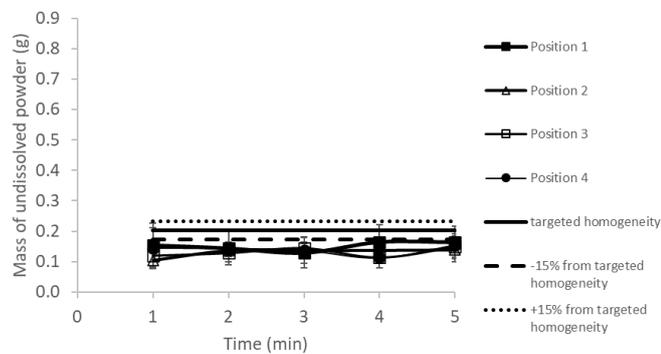


Figure 10. Homogeneity of 95% mannitol: 5% cocoa mixture at 20 rpm mixing speed.

although the homogeneity of the powder bed surface was found to be unacceptable based upon the required homogeneity of the mixture. This was because the undissolved mass of the samples obtained at the different locations on the powder bed surface were becoming more similar as depicted by lower variations between the positions as illustrated in Figures 2-10. Similarly, slowing the rotational speed also led to lesser variations in the homogeneity between the different positions on the powder bed surface, which were located near the wall, middle as well as between the wall and the middle of the powder bed indicating increased uniformity at the powder bed surface.

4. Conclusion

Poor mixing performance was achieved during the mixing of the cohesive cocoa and flowable mannitol powders at the rotational speed and composition combinations used in this work. This work showed that the acceptable homogeneity was only achieved at the two extremes of the powder mass ratios; 95%: 5% and 5%: 95% of mannitol: cocoa for some of the locations on the powder bed surface, especially near the wall of the mixer. This highlights the importance of the proper selection of mixing procedures involving cohesive powders during powder mixture preparation in laboratories especially for academic teaching and research purposes. Preparation of the sample mixture individually to eliminate inhomogeneity due to sampling thus ensuring each sample has the required composition as well as optimizing the mixing parameters before proceeding to subsequent processes such as tableting can be implemented to ensure the final product achieved the acceptable uniformity.

Acknowledgement

The authors gratefully acknowledged the funding received from Universiti Putra Malaysia IPS grant vot no: 9660700 and from UPM RMC.

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