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Roller compaction of moist pharmaceutical powders

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ABSTRACT

The compression behaviour of powders during roller compaction is dominated by a number of factors, such as process conditions (roll speed, roll gap, feeding mechanisms and feeding speed) and powder properties (particle size, shape, moisture content). The moisture content affects the powder properties, such as the flowability and cohesion, but it is not clear how the moisture content will influence the powder compression behaviour during roller compaction. In this study, the effect of moisture contents on roller compaction behaviour of microcrystalline cellulose (MCC, Avicel PH102) was investigated experimentally. MCC samples of different moisture contents were prepared by mixing as-received MCC powder with different amount of water that was sprayed onto the powder bed being agitated in a rotary mixer. The flowability of these samples were evaluated in terms of the poured angle of repose and flow functions. The moist powders were then compacted using the instrumented roller compactor developed at the University of Birmingham. The flow and compression behaviour during roller compaction and the properties of produced ribbons were examined. It has been found that, as the moisture content increases, the flowability of moist MCC powders decreases and the powder becomes more cohesive. As a consequence of non-uniform flow of powder into the compaction zone induced by the friction between powder and side cheek plates, all produced ribbons have a higher density in the middle and lower densities at the edges. For the ribbons made of powders with high moisture contents, different hydration states across the ribbon width were also identified from SEM images. Moreover, it was interesting to find that these ribbons were split into two halves. This is attributed to the reduction in the mechanical strength of moist powder compacts with high moisture contents produced at high compression pressures.

1. Introduction

Roller compaction is a dry granulation process for producing (large) granules with fine powders in order to improve the flowability and homogeneity of powder mixtures, so that they can be handled readily. It is an automated, continuous and efficient process that has been widely utilized in pharmaceutical, chemical, foundry and mineral industries. In addition, it is also an effective process to increase the bulk density of powders and minimises the dust problem during the manufacturing processes. Furthermore, it is a preferable process for the materials that are sensitive to heat or moistures as neither additional heat nor liquid are needed in this process (Bindhumadhavan et al., 2005; Kleinebudde, 2004; Mansa et al., 2008).

During roller compaction, powders are fed into the compaction zone that is formed by two counter-rotating rollers and side cheek (sealing) plates or the rim on one of the rollers. Depending upon the flowability of powders, different feeding mechanisms can be used to supply the powder into the compaction zone in the roller compaction, such as screw-feeding, in which the powder is fed with a rotating screw, and gravity feeding, in which no feeding device is used to assist and the powder is allowed to freely flow into the compaction zone. The powder is then squeezed into a narrow region between the rollers (nip region) as a result of the pressure induced by the screw feeder (or gravitational force) and the shear forces generated by the friction between powders and the rotating rollers. The powder is gripped between the rollers and compacted into ribbons or flakes with fluted (knurled) or smooth rolls, or into briquettes with pocket rolls (Guigon and Simon, 2003) which are subsequently milled into granules of desirable sizes.

The compression behaviour of powders during roller compaction is determined by a number of factors, such as process conditions (e.g., roll speed, roll gap, feeding mechanism and feeding speed) and powder properties (particle size, shape, moisture content, etc.) and has been investigated by many researchers (Bindhumadhavan et al., 2005; Kleinebudde, 2004; Mansa et al., 2008; Guigon and Simon, 2003; Miller, 1997; Shlieout et al., 2000;

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Simon and Guigon, 2003; Weyenberg et al., 2005; Guigon et al., 2007; Miguelez-Moran et al., 2008, 2009). Bindhumadhavan et al. (2005) examined the effects of roll gap, roll speed, and interparticle and wall frictions on the compression behaviour of microcrystalline cellulose and compared with the predictions of Johansson theory (Johanson, 1965). They found that Johanson theory can predict the effect of material properties on the nip angle, which defines the size of nip region and the compression duration and the peak pressure, which is related to the maximum degree of densification, but is unable to take the influence of roll speed into account. It is well recognized that rough rolls are able to drag the compacting material into compaction zone with stronger shear force than smooth rolls. Therefore, the peak pressure and the nip angle are determined by flow properties of powders, roll surface, roll gap, roll speed and feeding pressure (Bindhumadhavan et al., 2005; Kleinebudde, 2004; Mansa et al., 2008; Guigon and Simon, 2003; Miller, 1997; Shlieout et al., 2000; Simon and Guigon, 2003; Weyenberg et al., 2005; Guigon et al., 2007). Miguelez-Moran et al. (2008, 2009) explored the effect of friction conditions on the properties of roller compacted ribbons and revealed that the heterogeneity of ribbon density distribution is attributed to the non-uniform feeding of powder into the compaction zone. A new concept, the drag angle, was introduced to quantify the powder feeding behaviour.

The moisture content affects the flowability of powders, which will inevitably influence the powder compression behaviour during roller compaction. For moist powders, it is very likely that liquid bridges exist between particles, even solid bridges (bonds) between particles can be established. As the number of liquid and solid bridges increases, cohesion, that represents the forces of particles attracted to each other and friction that is related to the resistant forces exerted by one particle against the motion of its contacting particles, will increase (Nokhodchi, 2005; Emery et al., 2009). If the moisture content of a powder is very high, caking may occur (Dawoodbahai and Rhodes, 1989), which will dramatically reduce the flowability of the powder. On the other hand, the flowability of powders can also be improved with the addition of water, as identified by Staniforth et al. (1988) who examined the effect of the addition of water on the rheological and mechanical properties of microcrystalline cellulose (Avicel PH-101 and Emcocel) and suggested that, if there were enough water content in a powder to cover the particle surfaces, the water would act as advantageous lubrication so that the flowability of moist powder was improved due to the reduction of friction (see also Nokhodchi, 2005). Furthermore, the flowability of moist powders can be improved with the formation of large, round agglomerates (Emery et al., 2009).

The effect of moisture content on the compaction behaviour during uniaxial compaction has been explored by many researchers. Water in the moist powder can act as a lubricant along the die wall during compaction, which facilitates the force transmission between two punches and consequently reduces the density variation in tablets, especially at low compression pressures (Nokhodchi, 2005; Rees et al., 1970). The presence of water can also facilitate the formation of liquid bridges, especially at low moisture contents, which typically enable strange compacts to be produced (Khan and Pilpel, 1986, 1987). However, as the moisture content increases further, a large amount of free water may be present at the particle–particle interfaces, this will either induce hydrostatic resistance to consolidation or reduce intermolecular attraction forces, consequently, the tensile strength of moist powder compacts decreases. Hence there is an optimal moisture content to produce the strongest powder compacts (Pilpel and Ingham, 1988; Khan et al., 1981; Ahlneck and Alderborn, 1989; Armstrong and Patel, 1986; Garr and Rubinstein, 1992; Bangudu and Pilpel, 1985).

The influence of moisture content on the compaction behaviour during roller compaction was investigated by Gupta et al. (Gupta et al., 2005a,b), who focused on the effect of moisture content on the properties of roller compacted ribbons (relative density, tensile strength and Young's modulus). They argued that the moisture facilitated the powder compaction since it promoted the rearrangement and the deformation of particles. In addition, they showed that, for the mixture of MCC and crystalline acetaminophen (APAP), the tensile strength of the roller compacted ribbons first increased and then decreased with increasing moisture content (Gupta et al., 2005a). For the ribbons produced with MCC (Avicel, PH-200) at constant roller compactor settings and feed mass, the density kept unchanged but the tensile strength decreased as the moisture content increased (Gupta et al., 2005b).

However, it is still unclear how the moisture content affects the powder behaviour during roller compaction, in particular, the nip angle, pressure distribution and peak pressure. This is explored in this study using an instrumented roller compactor. A widely used pharmaceutical excipient, MCC (Avicel PH-102), is used and the effect of moisture content on the flowability of MCC, the flow and compression behavior during roller compaction and ribbon properties is investigated. In addition, the effect of roll speed and the characteristics of the produced ribbons are also examined.

2. Materials and methods

A widely used pharmaceutical excipient, microcrystalline cellulose (MCC; Avicel PH102; FMC Biopolymer, USA) was used for all experiments reported here. MCC blends with different moisture contents were prepared by slowly spraying a certain amount of water (2.5–15%, w/w) onto the powder bed when it was agitated in a rotary mixer (Lab-Mixer, Pascall, UK). In order to distribute the water uniformly in the powder, the rotation speed of the mixer was set at 6 rpm for low water content samples (2.5%, w/w and 5%, w/w), while it was set at 18 rpm for higher water content samples (10%, w/w and 15%, w/w). After adding the water into the powder bed, the powder was stirred in the mixer for 20 min at 60 rpm in order to achieve a better distribution of water. The moisture contents of the prepared moist powders and the as-received powder were measured using a moisture analyzer (Sartorius, MA30 Goettingen, Germany). The measurements were repeated three times and the average moisture content and the corresponding standard deviation were listed in Table 1.

The flowability of the moist powders were evaluated in terms of angle of repose and flow function. The angle of repose was determined using the Geldart angle of repose tester (Geldart et al., 2009). The average value and standard deviation of the angle of repose obtained from three measurements were given in Table 1. The flow function was determined using a ring shear tester (Schulze RST-XS, Wolfenbüttel, Germany) with normal loads at pre-shearing of 4, 6, and 8 kPa. No significant change in the flow function value was observed for the different normal loads. From the shear test, cohesion was also obtained and was found to vary with the normal load applied during the pre-shear.

The moist powders were then roller compacted using the instrumented roller compactor developed at the University of Birmingham (see Fig. 1). In this roller compactor, the two smooth rolls are 45 mm wide and 200 mm in diameter. The roll gap was set at 1 mm for all the experiments reported here, but two roll speeds were used (i.e., 1, and 2 rpm). The powder was gravity fed into the compaction zone through a feeding hopper. For all blends, the volume of the power used in each run was kept constant and was fed into the hopper in the same manner as follows: the powder was fed into the hopper until a small heap was formed over the top of the hopper wall, then it was leveled off. Thereafter, the roller compaction began and the evolution of the compression pressure was recorded with the pressure transducer that was fit in the middle of the roll surface. Meanwhile, in order to understand the flow

Table 1
Moisture content and flow properties of the moist powders considered.

No.	Water added (w/w, %)	Moisture content (w/w, %)	Angle of repose (°)	Flow function
1	0	4.07 ± 0.12	36.93 ± 0.91	7.10 ± 0.61
2	2.5	6.18 ± 0.08	37.15 ± 0.85	6.67 ± 0.12
3	5	9.72 ± 0.03	36.27 ± 0.93	6.42 ± 0.27
4	10	11.44 ± 0.32	39.53 ± 0.58	6.13 ± 0.24
5	15	15.20 ± 0.14	41.87 ± 0.30	4.85 ± 0.17

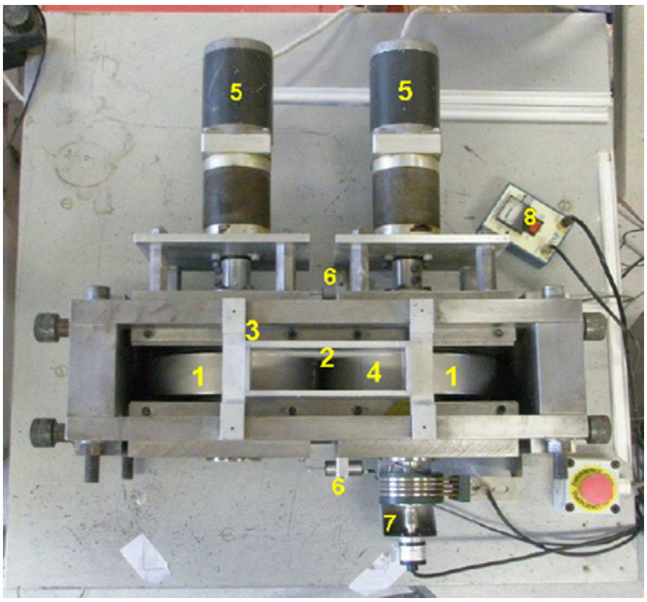

Fig. 1. The instrumented roller compactor utilized in this study. (1) Rollers, (2) cheek side plates, (3) hopper, (4) pressure transducer, (5) stepper motors, (6) displacement transducers, (7) encoder, (8) signal amplifier (Miguelez-Moran et al., 2008, 2009).

behaviour of the powders during roller compaction, a digital camera was used to record the powder flow patterns. From the digital images, the drag angle that is defined as the angle formed by the powder at the interface between the powder and the roll surface was determined.

3. Result and discussion

3.1. The effect of moisture content on powder flow properties

Fig. 2 shows the variation of angle of repose (AOR) and flow function (FFC) with the moisture content. It is clear that the AOR is essentially unchanged at low moisture contents (e.g. <10%), while it increases with the increasing moisture contents when the moisture contents are relatively high. This indicates that the AOR is not sensitive to moisture at low moisture contents, but a noticeable change in the AOR can be observed at high moisture content. Since a lower AOR is generally associated with a powder having better flowability (Geldart et al., 2009), it is clear from Fig. 2 that at lower moisture contents the moisture contents has a limited effect on the flowability, but as the moisture content increases, the powder flowability decreases. This is further confirmed with the flow functions obtained for the powders of different moisture content using the shear cell tester. It can be seen from Fig. 2 that the flow function decreases as the moisture content increases, indicating that the powder flowability decreases, although all the moist powders considered are classified as easy flowing powder with a flow function in the range between 4 and 10 (Herting and Kleinebudde, 2007; Ganesan et al., 2008). Furthermore, a close examination reveals that the flow function decreases with the increasing moisture content at a slightly increased rate when the moisture content is higher than 10%, which is consistent with the observation from the AOR measurements.

Using the ring shear cell tester, the slope angles of the linearised yield locus, $\varphi_{\rm lin}$, and the effective angles of internal friction, $\varphi_{\rm e}$, for the powders of different moisture contents were also determined and presented in Fig. 3. The effective angle of internal friction $\varphi_{\rm e}$ is regarded as a measure of the internal friction at steady-state flow (Teunou et al., 1999; Schulze, 2008). It is clear from Fig. 3 that both $\varphi_{\rm e}$ and $\varphi_{\rm lin}$ keep essentially constant when the moisture content is less than 10%, but smaller values were obtained at higher moisture contents. This indicates that the internal friction is reduced at high moisture contents, implying that water between particles acts as a lubricant when the powder undergoes shear deformation. Nevertheless, the cohesion obtained from shear cell tests (Fig. 4) increases as the moisture content increases, and the cohesion increases at a higher rate when the moisture content is higher than 10%. This implies that the powder becomes more cohesive as the moisture content increases. The data for cohesion presented in Fig. 4 have a much larger scattering when compared to Figs. 2 and 3, this is because the cohesion is more sensitive to the normal loads (4, 6, and 8 kPa) applied than the frictional angle (Fig. 3) and flow function (Fig. 2). It can be concluded from Figs. 2–4 that the moisture

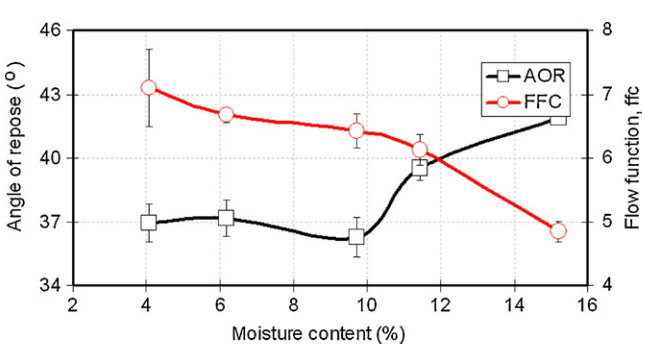

Fig. 2. The variation of the angle of repose (AOR) and flow function (ffc) with moisture content.

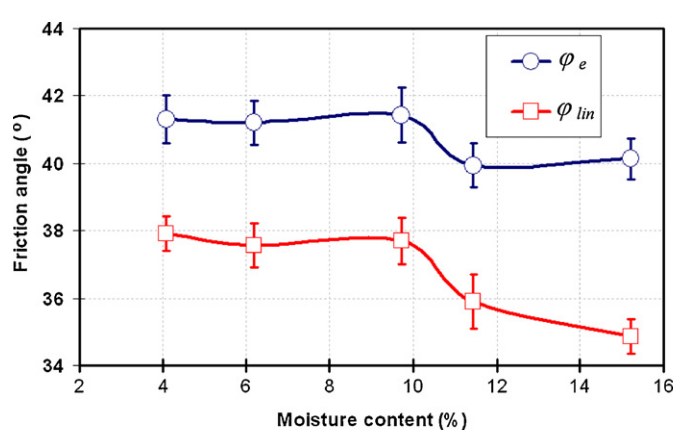

Fig. 3. The variation of effective and internal friction angles with moisture content.

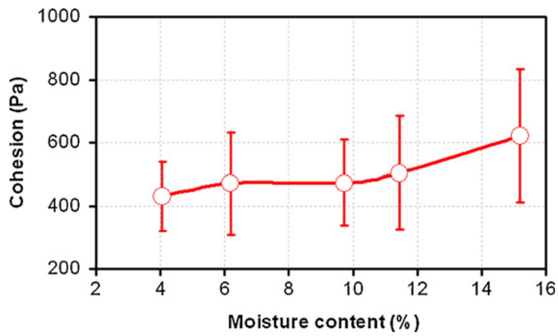

Fig. 4. The variation of cohesion with moisture content.

influences the flow properties of MCC powders in two ways: on one hand, the moisture will make the powder more cohesive and reduce the powder flowability. This is attributed to the formation of liquid bridges, even solid bridges, between particles (Dawoodbahai and Rhodes, 1989). On the other hand, the internal friction of the powders is reduced when the moisture content is sufficiently high. This is believed to be due to liquid films formed at particle surfaces that will act as a lubricant when the powder is sheared (Staniforth et al., 1988).

3.2. The effect of moisture content on powder flow behaviour during roller compaction

Miguelez-Moran et al. (2008, 2009) revealed that the friction between the powder and the fixed side cheek plates (FSCP) reduced the flow rate of the powder close to the FSCP into the compaction zone during the roller compaction with gravity feeding, consequently a non-uniform powder flow in the feeding hopper was observed. This caused the heterogeneity of density distributions in the produced ribbon. They also found that lubricating MCC powder with magnesium stearate could promote uniform powder feeding and subsequently the homogeneity of density distribution in the ribbon. A new concept, drag angle, was introduced to quantify the non-uniform powder feeding and it was found that the drag angle decreased (i.e., more uneven powder feeding) with the increasing roll speed. This is further confirmed by the present study, in which roller compaction was performed with the same powder but at lower roller speeds (1 and 2 rpm, instead of 3 and 5 rpm used by Miguelez-Moran et al., 2008). The powder profiles at various instants for roller compaction of as-received MCC at a roll speed of 2 rpm are shown in Fig. 5. It can be seen that, as the two rolls rotate, the powder in the middle of the roll width is gripped into the compaction zone at a higher rate than that at the edges (Fig. 5b–d), although the initial powder bed has a flat top surface (Fig. 5a). Compared to the flow patterns at a higher roll speed (see Fig. 4 in Miguelez-Moran et al., 2009), a larger drag angle (Fig. 5c) is obtained for the roller compaction at 2 rpm, indicating that the powder is gripped into the compaction zone more uniformly.

The typical flow patterns during roller compaction with powders of different moisture contents at a roll speed of 2 rpm are shown in Fig. 6. It was noticed that, as the moisture content increases, the powder becomes more cohesive and it starts to cascade intermittently onto the rotating rolls as large chunks of agglomerated powder, especially for high moisture contents (see Fig. 6d and e). This flow pattern is similar to the intermittent flow patterns observed during die filling with cohesive pharmaceutical powders (Sinka et al., 2004). It can be seen from Fig. 6 that, for all the moist powders considered, the powder in the middle of the roll width is gripped into the compaction zone at a slightly higher rate than those close to the FSCP.

3.3. The effect of moisture content on roller compaction behaviour

It has been identified that two key parameters can be used to characterize the compaction behaviour of powders during roller compaction: (i) the maximum pressure (P_{max}) and (ii) the nip angle. The nip angle defines the size of the nip region and the compression duration for a given roll speed, while the maximum pressure (P_{max}) indicates the maximum degree of densification for a given powder. These two parameters are dependent on the inherent powder properties (cohesion, internal friction and the friction between the powder and the tooling, etc.) and the processing conditions, such as roll speed and roll gap (Bindhumadhavan et al., 2005; Kleinebudde, 2004; Mansa et al., 2008; Guigon and Simon, 2003; Miller, 1997; Shlieout et al., 2000; Simon and Guigon, 2003; Weyenberg et al., 2005; Guigon et al., 2007). The pressure distributions during the roll compaction with the powders of various moisture contents at roll speeds of 1 and 2 rpm were obtained using the instrumented roller compactor. The representative pressure distributions for all cases considered are presented in Fig. 7. It can be seen that the pressure distributions for low moistures contents are similar, while become significantly different when the moisture content is relatively high (say >10%). It is also clear that the decompression is much faster than compression.

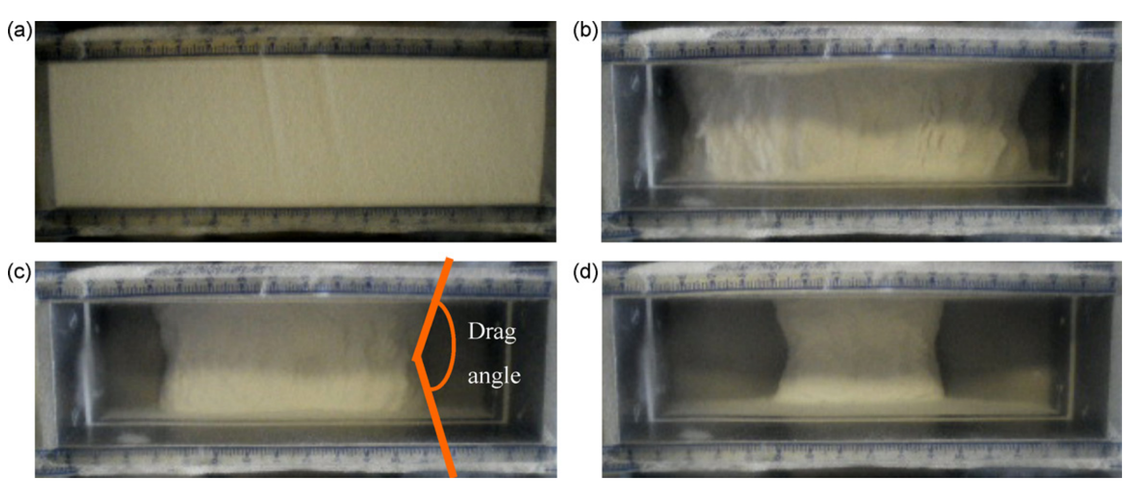

Fig. 5. Powder profiles at various instants (a–d) during roller compaction with as-received MCC at 2 rpm.

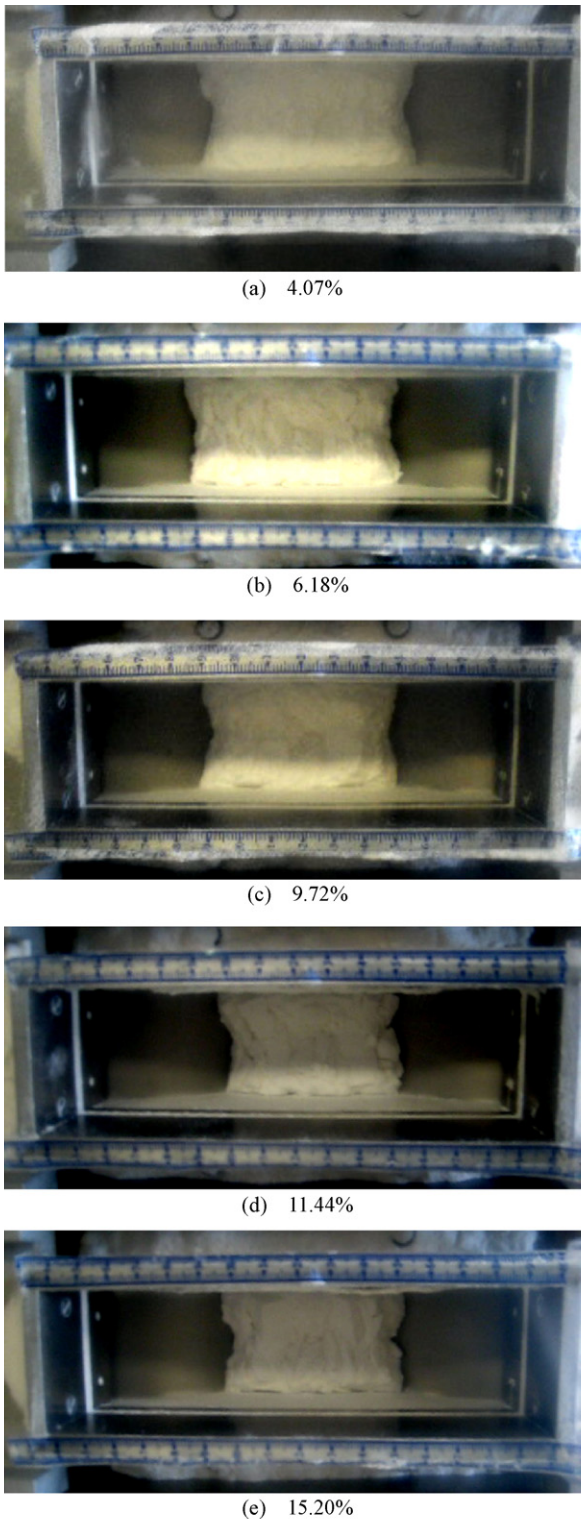

Fig. 6. Comparison of powder feeding patterns during roller compaction with different moisture contents at 2 rpm.

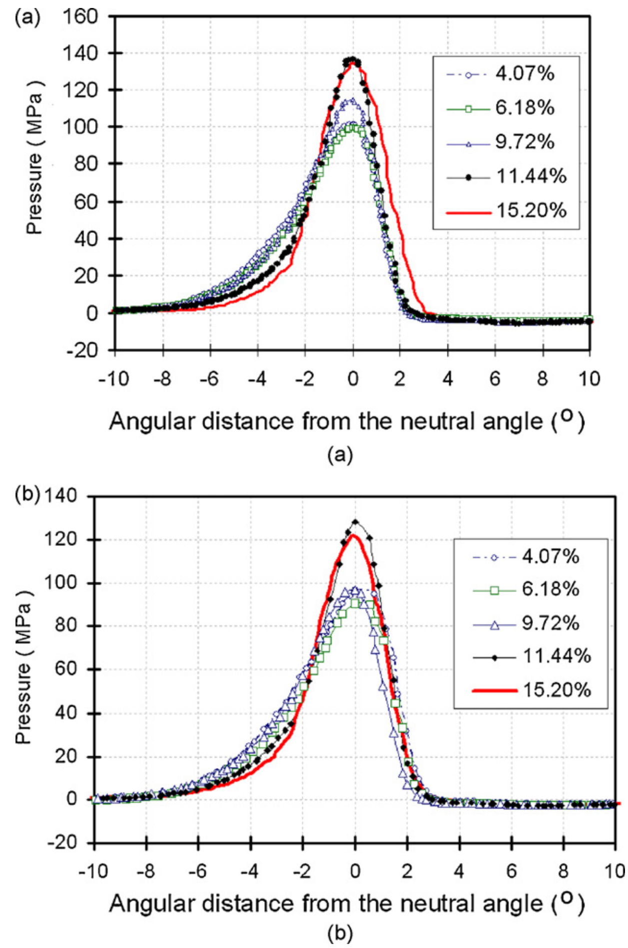

Fig. 7. Compression pressure profiles for the roller compaction with different moisture contents at (a) 1 and (b) 2 rpm.

The variation of the average value of the maximum pressure P_{max}, which was determined by averaging the maximum pressures over 3–5 revolutions of roller compaction operations, with the moisture content is shown in Fig. 8. It is clear that P_{max} increases as the roll speed decreases. This is consistent with the results published in the literature (Bindhumadhavan et al., 2005; Mansa et al., 2008; Miguelez-Moran et al., 2008, 2009). This is attributed to an increasing amount of powder being gripped into the compaction zone when the roll speed is decreased. It is interesting to note that, when the moisture content is relatively low (say <10%), P_{max} is insensitive to the moisture content, however, there is a sharp increase in P_{max} when the moisture content is higher than 10%. A maximum value of P_{max} is obtained for the roller compaction with a powder of 11.44% moisture content. The sharp increase in P_{max} is attributed to the increase in cohesion (Fig. 4) and the decrease in the flowability (Fig. 2), so that a larger amount of powder mass was gripped into the compaction zone and compressed. In other words, if the powder is less cohesive and has a good flowability, it may pass through the roll gap more easily and a lower amount of powder mass will be compressed in the compaction zone, as

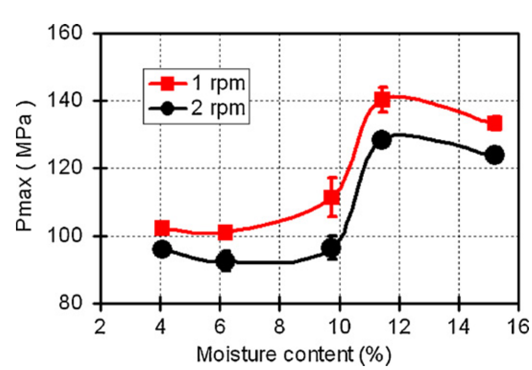

Fig. 8. The variation of the peak pressure (P_{max}) with moisture content.

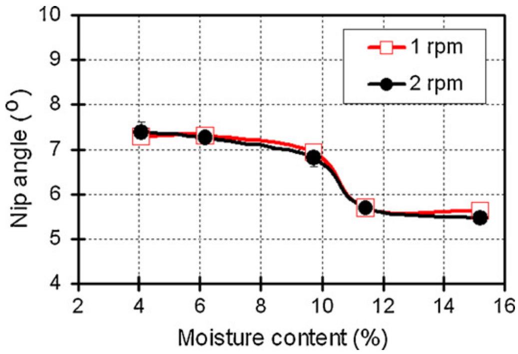

Fig. 9. The variation of the nip angle with moisture content.

observed for most free flowing powders. The decrease in the effective angle of internal friction at high moisture contents (Fig. 3) also promotes more powder being gripped into the nip zone and compressed. Therefore, it is suggested that P_{max} is directly related to the powder flow properties (cohesion, flowability, and effective angle of internal friction). A slight drop in P_{max} at the highest moisture content (15.2%) is believed to be due to the increasing lubrication effect when the moisture content is sufficiently high so that the powder behave like a paste as it is squeezed through the roll gap and the friction between powder and roller surfaces is reduced.

The corresponding nip angles for various moisture contents considered are shown in Fig. 9. It is clear that the nip angle generally decreases as the moisture content increases. In addition, a sharp decrease in nip angle occurs when the moisture content is higher than 10%. Again, this is due to the changes in the powder flow properties as shown in Figs. 2–4. In addition, moisture reduces the friction between the bulk and the roller surfaces due to the lubricating effect of water when added to MCC (Khan et al., 1981). As a consequence, the angular position at which the shear stress necessary to drag the powder to the roller gap is lower. As a consequence, the nip angle value is reduced. The nip angles obtained at two different roll speeds are very close (essentially identical), however, a close examination revealed that the nip angles obtained at 1 rpm are slightly larger than those with a roll speed of 2 rpm. This is consistent with previous publications showing that the nip angle decreased as the roll speed increased (Mansa et al., 2008; Miguelez-Moran et al., 2008, 2009).

3.4. The effect of moisture content on ribbon properties

For the roller compaction with the powders of low moisture contents (say <10%) at both roll speed employed, the width of the produced ribbons are essentially the same as the roll width (i.e. 45 mm, see Fig. 10a). In addition, at medium and high moisture contents (say >9.72%), there is a noticeably change in the color in the middle of the ribbon, which become brown rather than white as observed in the loose powder and in the ribbons produced with relative low moisture contents. This is believed to be due to hydrolysis and caramelization of the glucose monomers as a result of the high pressure and hence high temperature induced in the middle of the roller width. Furthermore, it is interesting to note that the ribbons produced with powders of high moisture contents (>10%) split into two halves during the roller compaction (Fig. 10b). Scanning electron micrography (SEM) was then employed to examine the ribbons in order to identify the mechanisms for the color change and splitting. Particular attention has been paid onto the packing structures at the areas close to the edge of the ribbons (i.e., spot A, C, D, F in Fig. 10) and in the middle of the ribbon (i.e., spots B and E in Fig. 10).

The representative SEM images for the spots A–F indicated in Fig. 10 are presented in Fig. 11. It can be seen that, for the ribbon produced with powders of low moisture contents (say 4.07%, Fig. 11a and b), individual particles can be identified even though they are compressed to bond together. In addition, it appears that the powder was compressed to a denser state in the middle of the ribbon when compared to those at the edges (see Fig. 11a and c), as there are less pores in the middle of the ribbon and the pore sizes are much smaller (see Fig. 11b). This is consistent with the analysis of Miguelez-Moran et al. (2008, 2009). For the ribbons produced with powders of high moisture contents (Fig. 11d–f), the edges of the ribbon are apparently occupied by clusters of dense compacted agglomerates that are surrounded by relatively large pores. While in the middle, a paste-like structure can be clearly observed. This is attributed to the high water concentration in the middle of the ribbon when the powder of high moisture content was compressed to a much denser state as a result of non-uniform powder feeding (see Figs. 5 and 6). As it has been recognized that the tensile strength of moist MCC compacts will reach maximum at a moisture content of ca. 10% (Pilpel and Ingham, 1988; Khan et al., 1981; Ahlneck and Alderborn, 1989; Armstrong and Patel, 1986; Garr and Rubinstein, 1992; Bangudu and Pilpel, 1985), further increase in moisture contents will result in a decrease in the tensile strength. Therefore, it is anticipated that the tensile strength in the middle of the ribbon width is smaller and the particle bonding in this region is weak. This is why the ribbon splits into two halves at high moisture contents (see Fig. 10).

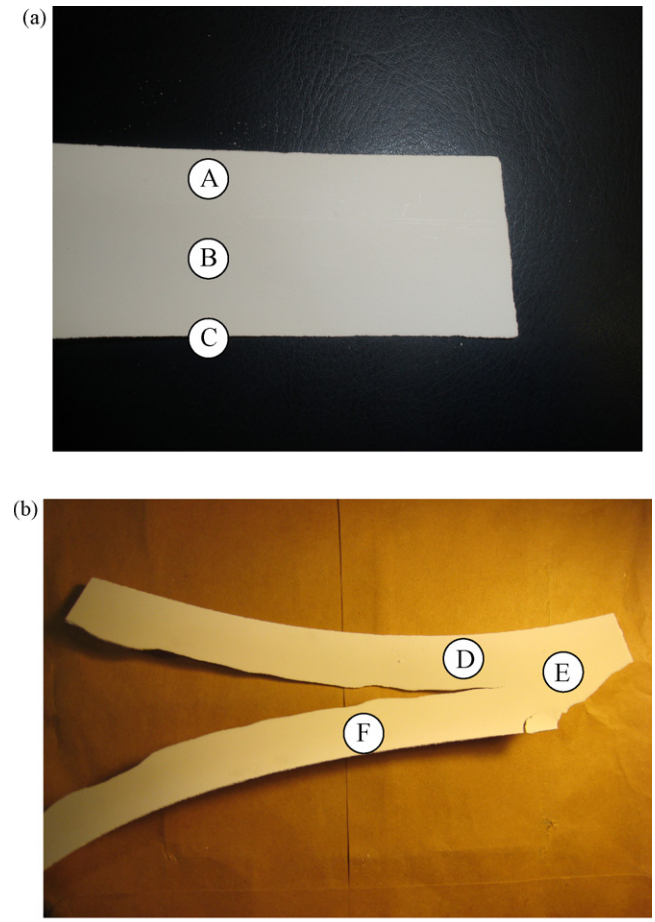

Fig. 10. Photograph of the produced ribbons at a roll speed of 1 rpm with powders of different moisture contents (a) 4.07% and (b) 11.44%.

4. Conclusions

In this study, the effect of moisture content on flow properties and roller compaction behaviour of MCC powder was investigated.

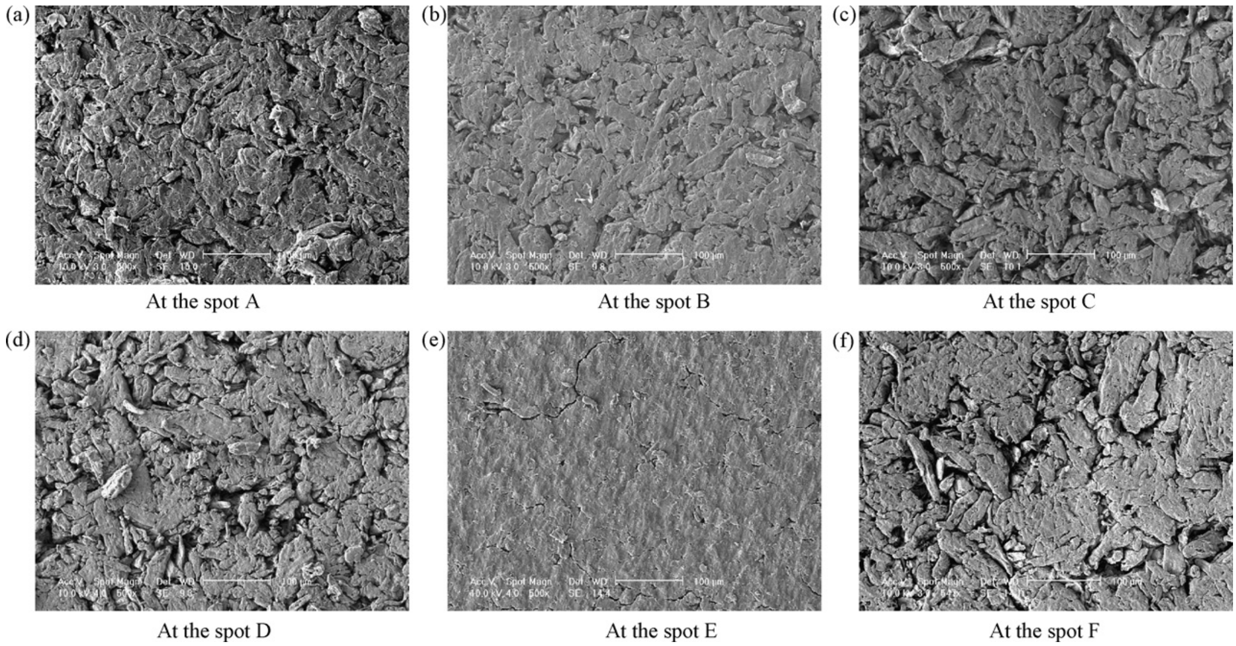

Fig. 11. SEM images of ribbon produced with the powder of 4.07% moisture content (a–c) and 11.44% moisture content (d–f) at various spots indicated in Fig. 10.

MCC samples with various moisture contents were prepared by mixing as-received MCC powder with different amount of water (0–15%, w/w) that were sprayed onto the powder bed as it was being agitated. The flow properties of the moist powders were determined using an AOR tester and a ring shear cell tester. The powder flowability was then assessed in terms of angle of repose and flow function. In addition, cohesion and internal friction angles (effective and linearised) were also determined. It has been shown that the powder flowability is not sensitive to moisture contents below 10% moisture. However, there are significant changes in flowability, cohesion and internal friction angle when the moisture content is over 10%. As the moisture content increases further, the flowability and internal friction angles decreases and the cohesion increases.

The flow behaviour of the powder during roller compaction with various moisture contents was also investigated. It has been found that the powder in the middle of the roll width is gripped into the compaction zone at a higher rate than those close to the roll edges. Intermittent flow, in which powder cascades onto the roll surfaces as chunks of agglomerated powder, takes place when the moisture content is relatively high. It has also been found that, for the roller compaction of a given powder with certain moisture content, the P_{max} increases as the roller speed decreases. When the moisture content is relative low, P_{max} is not sensitive to the moisture content. A sharp increase in P_{max} is observed when the flow properties start to change with the moisture content, indicating that powder flow properties have a dominant role in roller compaction. This is also true for the effect of moisture content on the nip angle. An interesting phenomenon observed in this study is that, at high moisture contents (>10%), the produced ribbon split into two halves. This is caused by strong hydrostatic resistance to consolidation induced as a large amount of free water present in the middle of the ribbon and consequently a reduction in the bonding strength.

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