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Effect of operating conditions on dry particle coating in a high shear mixer

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ABSTRACT

An investigation into the effects of the operating conditions on a dry particle coating process has been performed with mixtures of sugar grains (Suglet®) and magnesium stearate (MgSt) as the host and invited particles, respectively. Dry coating has been carried out in a high shear mixer (Cyclomix®) at different processing times t, rotational speeds ω, and filling ratios J, of the mixer. In order to determine the effect of the operating conditions on the properties of the products, the changes in flowability and wettability of the mixtures as a function of ω, J and the operating time t have been analyzed.
The flowability is found to be improved by longer processing times. Higher speeds of rotations can improve the flowability more rapidly. However, the flowability does not seem to be sensitive to the filling ratio and it may be concluded that operating with higher filling ratios gives higher production. A method is proposed to measure the mass fraction of invited particles on the surfaces of the host particles. This fraction is linked to the product properties as the flowability and wettability. This result can be predicted by a model using a coating rate constant k_c which can be used to optimize dry coating processes to give desired properties of coated particles. This coating rate constant can define and represent the efficiency of the dry coating.

Keywords:
Dry coating
Wettability
Flowability
Mixture composition
Coating rate constant
High shear mixer

1. Introduction

Surface modification of particles is widely used for many products such as paints, ceramics, cosmetics and pharmaceutics in order to give new functionalities or improvement of particle properties. Dry coating has recently attracted considerable attention as an alternative coating method because of its simplicity and environmental friendliness. In such processes, the fine particles called 'invited particles' and relatively large size particles called 'host particles' are mixed together by mechanical action such as shear or impact forces without any binder so that invited particles become firmly attached on the surface of host particles and change their properties [1,2]. There have been many reports on successful research projects dry particle coating [3–8], but less work has been done on understanding the factors controlling the process of dry coating [9].

To obtain good products by dry coating it is important to control the process. On one hand, poor products can be obtained with weak bonds between host and invited particles due to using inadequate mechanical energy. In this case the coating of invited particles may then be detached in subsequent processing such as pneumatic transport or compression. On the other hand, an excess energy used in the coating process can also lead to poor product properties because of breakage, attrition or erosion of host particles.

Since the coating process is generally done by simply mixing the dry component powders, it may be imagined that the operating conditions of the mixer, such as rotational speed ω, filling ratio J and processing time t, will strongly affect the overall performance of dry coating [10]. This work considers this problem by examining the effects of operating conditions on a dry coating process using a Cyclomix high shear mixer supplied by the firm Hosokawa micron. The properties of the coated products such as flowability, wettability, surface morphology and the mass fraction of invited particles fixed on the host particles are determined so as to understand the phenomena involved in dry coating, and to obtain a general method to estimate the efficiency of a dry coating process .

2. Experimental

2.1. Sample powders

The sample powders chosen are sugar particles (Suglets®) and magnesium stearate (MgSt) for host and invited particles respectively. The properties of these powders are shown in Table 1. Suglets, produced by Colorcon Inc., are spherical cores mainly composed of sucrose and maize starch with a highly hydrophilic character. This material is used for creating sustained or extended release drug layered dosage forms. A SEM image of this material is shown in Fig. 1

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Table 1
Particle properties.

Material	D_{50} [µm]	Pycnometry density [kg/m3]	Water interaction (contact angle [°])
Suglets	250	1580	Hydrophilic (0)
MgSt	5.0	1120	Hydrophobic (132)

(a) which indicates that Suglets seem to be well crystallized and have a relatively spherical shape. Magnesium stearate MgSt supplied by Chimiray, was used as the invited particles. This is a fine, white, cohesive and hydrophobic powder commonly used as a powder lubricant in the pharmaceutical industry. Fig. 1 (b) shows an MgSt SEM image which indicates that grains have irregular shapes. Fig. 2 shows the volume particle size distribution of Suglets and MgSt respectively measured by a Malvern dry feed system (Mastersizer 2000) using a dispersing air pressure of 0.5 bar. The Suglets particles are seen to have a mono-modal size distribution with median diameter (D_{50}) of about 250 µm, which indicates that this powder has a very homogeneous size. In contrast the MgSt has a wide particle size distribution ranging from 0.1 µm to 50 µm with a D_{50} of about 5 µm.

2.2. Coating process

The mass fraction w of invited particles added to the host particles in the experiments was calculated with reference to an ideal 100% surface coverage of host particles. Supposing that all particles are homogeneous and spherical and do not deform during the coating treatment, the mass fraction w can be obtained by the following equation with the size ratio of host/invited particle k_H [10] for perfectly ordered systems:

$$w = \frac{4C_{2D}(k_H+1)^2}{4C_{2D}(k_H+1)^2 + \frac{\rho H}{\rho I} k_H{}^3} \tag{1}$$

$$k_H = \frac{R_H}{R_I}. \tag{2}$$

C_{2D} is the surface packing fraction in 2 dimensions of the invited particles on the surface of host particle.

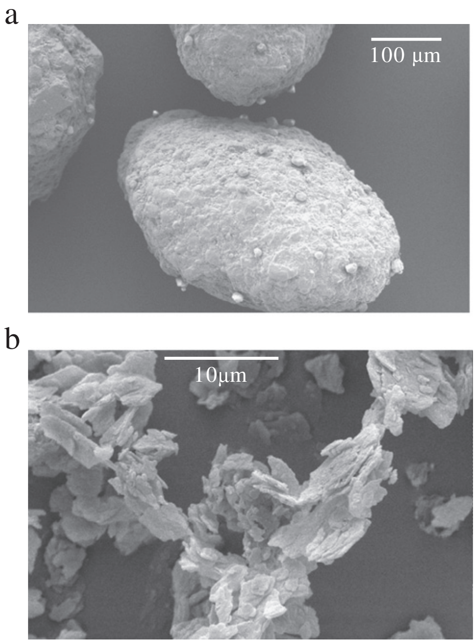

Fig. 1. SEM images of sugar particles (Suglets) and magnesium stearate (MgSt).

ρ_H and ρ_I densities of host and invited particle.
R_H and R_I the radius of host and invited particles, respectively.

Supposing that C_{2D} is that of a hexagonal compact structure with $C_{2D} = 0.906$, the mass fraction of invited particles for 100% coverage is estimated to be about 4.8%. This theoretical value may be subject to variations seconding the type of particles (densities, radius ratio…). In real experiments, a part of the materials may also be stick on the walls of the mixer, and it would be better to choose a higher value like 5% minimum: the loss of particles on the walls is limited anyways because the area of particles is much larger than the one of the walls. The loss can reach some percents of MgSt in the example of MgSt 15%-silica mixtures [14]. In the contrary, a large particle size distribution of fine particles would rather induce lower values for the critical mass fraction w by increasing the mean size ratio k_H.

In the following, the mass fraction of invited particles introduced in the chamber at the beginning of the process w_0 will be fixed at 5% to see clearly the effects of process conditions, even if a smaller value could be a little bit more realistic and efficient enough for modifying surface properties.

A 1 liter high shear Cyclomix supplied by Hosokawa Micron B.V. is used as the coating device (Fig. 3). As can be seen, this device has a conical chamber with a vertical axis fitted from bottom to top with four pairs of flat-bladed impellers which can be rotated at speeds from 200 rpm to 3000 rpm. The Cyclomix is generally used as a high shear granulator, but has been also successfully used for dry coating [11a,b]. To observe the effect of processing conditions, the rotation speed of the mixer, ω is varied from 250 to 1500 rpm and the processing time t varied from 30 to 600 s. The filling ratio of the mixer chamber J, defined as the ratio of the apparent volume of solid particles divided by the volume of mixer chamber, was varied from 20 to 60% (162–486 g of Suglets in 10^{-3} m^3). The operating conditions used in this work are summarized in Table 2.

2.3. Analysis

2.3.1. Wettability

The wettability of the coated and uncoated particles has been determined by the sessile drop test [12,13]. A small 10 µL drop of distilled water is deposited on the surface of a bed of the powder product at room temperature, and the contact angle measured 30 s after the moment the drop is deposited. In addition, to avoid breaking the coating or the particles themselves, the powder bed was not compressed but just patted down with a spatula to give as homogeneous a

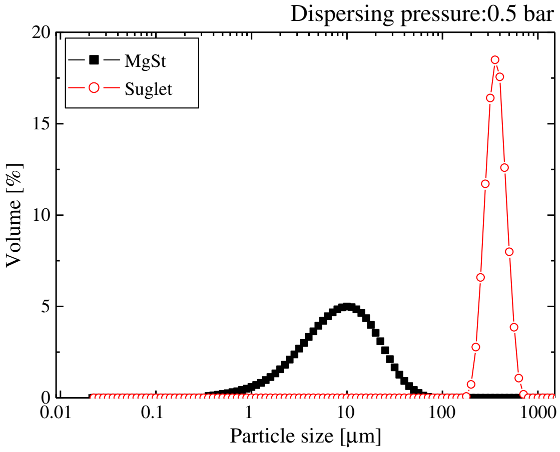

Fig. 2. Volume particle size distribution of Suglets and MgSt.

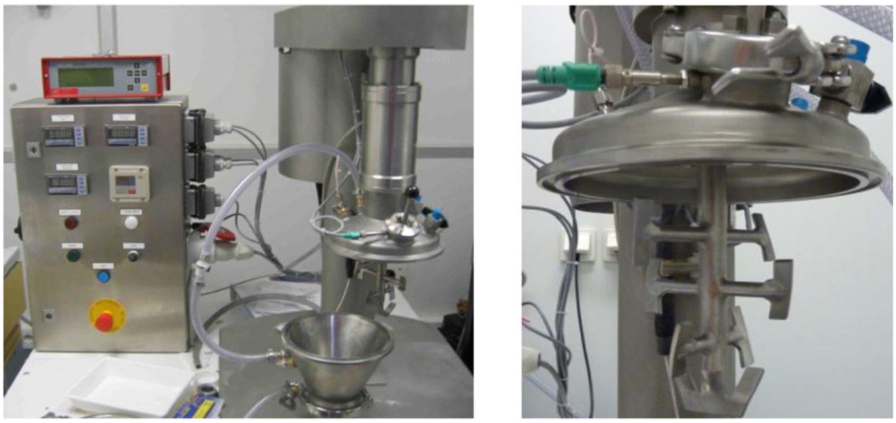

Fig. 3. Schematic diagram of the high shear mixer "Cyclomix".

flat powder surface as possible. The contact angle measurement was carried out 3 times on each sample and the average value taken.

2.3.2. Flowability

An FT-4 Powder Rheometer (Freeman Technology) is used to analyze the flowability of the powder. This device has a specially profiled propeller type blade that can be simultaneously rotated and moved axially into the powder cell to measure the axial force and the rotational force to obtain various rheological characteristics of the powder. In this work, the Flow Rate Index (FRI) has been chosen as an index of the flowability. The higher the FRI is, the lower the flowability of the powder.

2.3.3. Measurement of the mass fraction of the coating of invited particles

Fixed quantities of free MgSt (mass m_0) and Suglets (mass M_0) are introduced in the mixer, with a mass fraction $w_0 = m_0/(M_0 + m_0) = 5\%$. After a mixing time t, in given operating conditions some of the grains of Suglets are coated, with a mass $M_0 - M(t)$, and the others, with a mass $M(t)$, are not coated. Thus some of the invited particles remain as free particles, with a mass $m(t)$, and a mass $m_0 - m(t)$ of invited grains are stuck on the surface of host particles to form a coating. We suppose that only a very small quantity of Suglets or MgSt remains attached to the wall or the blades of the mixer, and the powder mixture removed from the mixer is representative of the total volume of the particles (invited and host) introduced in the mixer.

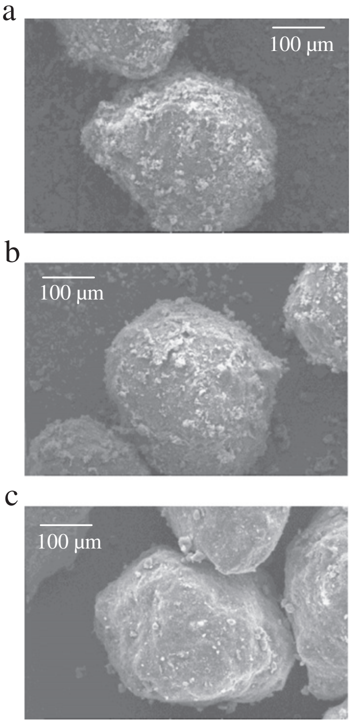

Fig. 4. SEM images of the coated particles at 500 rpm, filling ratio $J = 60\%$ and at operating time t (a) 1 min, (b) 3 min, (c) 10 min.

To estimate the effective mass fraction of invited particles coating the host particles, the total volume of the mixture was sieved at 160 µm for 10 min. Neglecting the possibility that invited particles could be liberated by shear or impact forces occurring during the sieving, and neglecting the possibility that host particles could be broken during "mixing", the mass $m(t)$ of particles passing through the sieve can be considered to be the amount of invited particles that did not stick to the host particles during the process.

By measuring the mass $m(t)$ and the mass $M_S(t)$ of the powder that remained on the sieve, the effective mass fraction of MgSt, w_e in the coated product can be estimated, after a time t of mixing by Eq. (3). The parameters w_e and m_0 are the mass fraction of the product and the mass of the invited particles introduced into the high shear mixer before mixing.

$$w_e(t) = \frac{m_o - m(t)}{M_S(t) + m(t)}; w_e(t) \sim \frac{m_o - m(t)}{M_o + m_o} \tag{3}$$

The comparison of the values w_e and w_0 gives information on the degree of efficiency of the coating process. More precisely, the ratio λ can be regarded as a coating ratio (Eq. (4)).

$$\lambda(t) = \frac{w_e}{w_o}; \lambda(t) = 1 - \frac{m(t)}{m_o} \tag{4}$$

3. Results and discussion

3.1. Surface morphology

Fig. 4 shows SEM images of particles coated for different processing times at 500 rpm and for a filling ratio $J = 60\%$. At the start of the operation (after 60 and 180 s mixing), it was observed that invited particles are distributed as discrete particles on the surface of host

Table 2
Operation condition.

Coating device	Rotational speed [rpm]	Mixing time [s]	Filling ratio [%]	Mass fraction of invited particles [%]
Cyclomix	250–1500	60–600	20–60	5

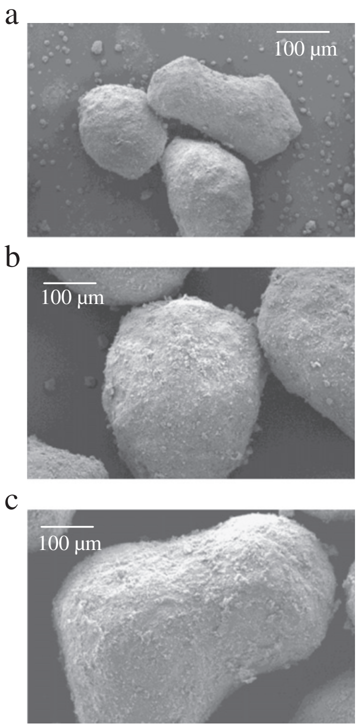

Fig. 5. SEM images of the coated particles at 1000 rpm, filling ratio $J=60\%$ and operating time t (a) 1 min, (b) 3 min, (c) 10 min.

particles. In addition, a number of smaller particles were found. These could be invited particles that have remained free, or fragments of the host particles broken by attrition or erosion. However, at the end of the process (600 s), the surface of particles seems smooth and there are few free small particles observed. These phenomena can be explained as follows. At first, the invited particles stick to host particles by shear forces induced by the mixing, and then those invited particles fixed on the surface become deformed and smeared into a thin film on the host by interaction forces.

Fig. 5 shows SEM images of particles coated at 1000 rpm. As compared to those treated at 500 rpm, these particles have a smooth surface even after only 180 s, which indicates that the thin film has been created faster. At 1500 rpm, there is the same tendency as at 1000 rpm. It may be said that, at speeds above 1000 rpm, enough energy is given to the system to change from discrete to film coating even in a short processing time.

3.2. Wettability

The sessile water drop test has been carried out to analyze the wettability of the products. Images of the water drop test for uncoated Suglets, and for the coated product after 60 s at 1000 rpm and a filling ratio $J=60\%$ have been done. The water drop is seen to be absorbed as soon as it is placed on the powder bed of uncoated Suglets due to its high hydrophilic property whereas the water drop remains on the coated product even after 30 s. Concerning wettability, it may be said that a 60 s coating operation at 1000 rpm is enough to change the surface property from hydrophilic to hydrophobic.

Fig. 6 (a)–(b) shows the contact angle measured 30 s after the water drop is placed on the powder surface for different processing times t, different speeds of rotational ω, and at filling ratio of $J=20\%$ (a), 60% (b). The initial value (0 s operating time) in these figures is calculated by assuming that the contact angle of the product after a certain processing time $\theta\ (t)$ is proportional to the free surface fraction s_H of invited particles (Eq. (5)), and that the host and invited particles are fully mixed at the start of the operation.

$$\theta(t) = s_H\theta_H + (1-s_H)\theta_I \tag{5}$$

This surface fraction of the host particle in the initial state, s_{H0} is given by Eq. (6), as proposed in literature [10]:

$$s_{H0} = \frac{{k_H}^2}{4C_{2D}(k_H+1)^2 + {k_H}^2}. \tag{6}$$

This expression can be obtained by writing that the surface fraction of host particles in the initial state is the ratio of the area of the sphere passing through the points of contact between host and invited particles (radius $\mathrm{R_I}+\mathrm{R_H}$) and the effective area occupied by an invited particle at the surface of host's one ($\pi \mathrm{R_I^2}/\mathrm{C_{2D}}$).

Since the contact angle of a host particle θ_H and that of an invited particle θ_I are 0° and 132° respectively, the theoretical value derived from Eq. (5) of the contact angle for the initial mixture θ (0) is found to be 104°. We can notice that the physical meaning for $t=0$ corresponds to a state where the two kinds of grains are present without any coating.

In Fig. 6 (a), at 250 rpm, the contact angle increases exponentially with increase of processing time. From the assumption above, the surface fraction of the coated material will also increase. In other words, the coating process proceeds as a function of the processing time; at 500 rpm, it increases more rapidly than at 250 rpm. At 1000 rpm, $\theta\ (t)$ increases with time and is asymptote to the θ_I of

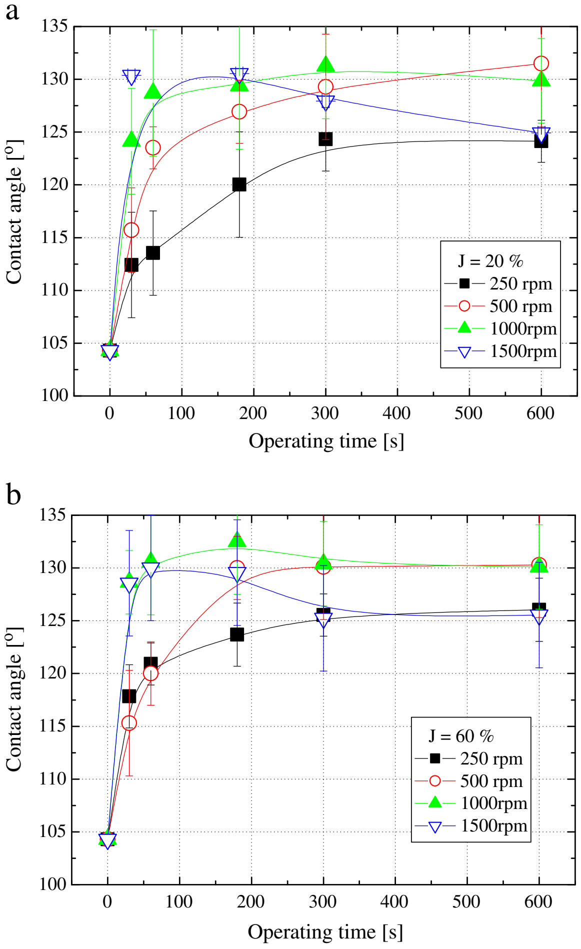

Fig. 6. Dependence of contact angles on the operating time t, and on the different rotational speeds ω for different filling ratios (a) $J=20\%$, (b) 60%.

invited particles. At 1500 rpm, it increases up to 180 s and then starts to gradually decrease. SEM images of the particles coated at 1500 rpm indicate that there is little major breakage of the host particles but it can be said that, once fine fragments of the host particles are generated by attrition or erosion during the process, these fine fragments of host particles stick again on the surface of the host particles. These phenomena could bring about a decrease in contact angle and the surface fraction of coated particles in high speed coating operations. In Fig. 6 (b), it is observed that these curves have almost the same general tendency as that of Fig. 6 (a) thus the product contact angle is not sensitive to the filling ratio of Cyclomix. These results have also been confirmed for $J = 40\%$. To summarize, all the coated particles have a contact angle greater than 90°, and the wettability of the coated particles goes from hydrophilic to hydrophobic even with the smallest processing time and the lowest speed of rotation of the mixer. This indicates that this mixer has a good efficiency for coating particles where the particle size ratio between host and invited particles is big enough so that Van Der Waals forces are high enough to firmly coat the invited particles on the host particles.

3.3. Flowability

Flowability of powders can be affected by several physical properties of particles, such as their size, shape, surface roughness and texture. In this work, it is assumed that changes in the surface texture of the particles by coating MgSt could influence flowability by functioning as a lubricant. Fig. 7 (a)–(b) presents the Flow Rate Index (FRI) as a function of operating time t at different speeds of rotation of the mixer ω and at filling ratios $J = 20\%$ (a), 60% (b). The initial value of FRI is that of the uncoated host particles. In Fig. 7 (a), at 250 rpm, the FRI decreases with an increase in the processing time due to coating by MgSt. However, the FRI at 250 rpm decreases very slowly compared to that obtained for other rotational speeds. This could be because the coating process is slower than at higher speeds. At 500 rpm, FRI decreases also with an increase in the processing time. In other words, the flowability of the coated particles improves with increasing processing time. In addition, as seen in Fig. 4, at 500 rpm, the surface of the coated particles becomes smoother with processing time. This could help the particles to have better flowability. At 1000 rpm, the FRI decreases with an increase in the processing time more rapidly than at 500 rpm. At 1500 rpm, the FRI decreases with an increase in the processing time up to 180 s, and then it increases slightly. This could be attributed to an increase in the roughness of the surface of the coated particles, due to fragmentation of the host particles, giving an over-coating with some small fragments sticking to the surface of the host particles. In Fig. 7 (b), at $J = 60\%$, the same behavior is observed as at $J = 20\%$. However, the FRI decreases more slowly than that of $J = 20\%$ even though the difference is rather small perhaps due to the fact that at higher filling ratios less mechanical energy given to each particle. Experiments at 40% gave the same tendencies.

3.4. Conversion ratio

Fig. 8 presents the variation of the conversion ratio $\lambda(t)$ of the coating process as a function of processing time t at different

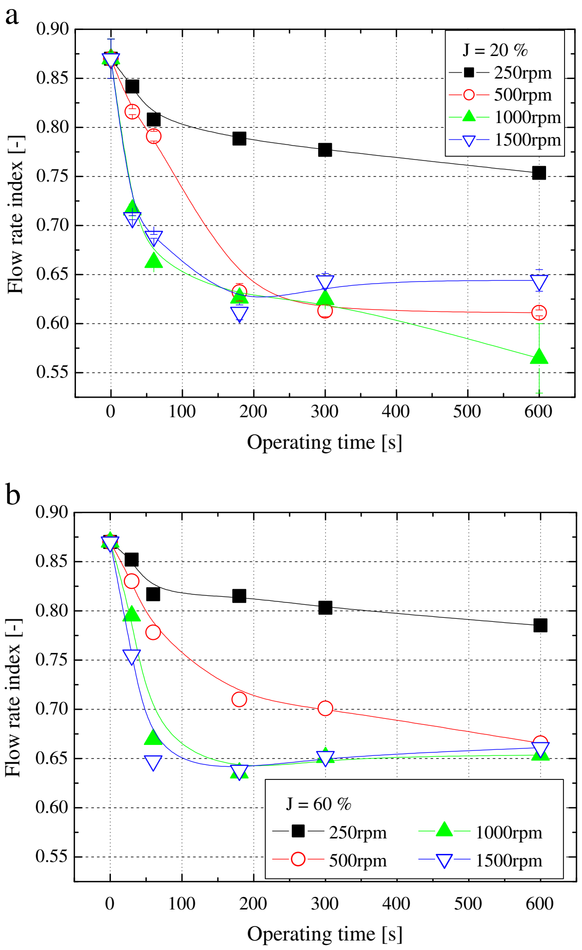

Fig. 7. Dependence of the Flow Rate Index (FRI) on the operating time t and on the different rotational speeds ω for different filling ratios (a) $J = 20\%$, (b) 60%.

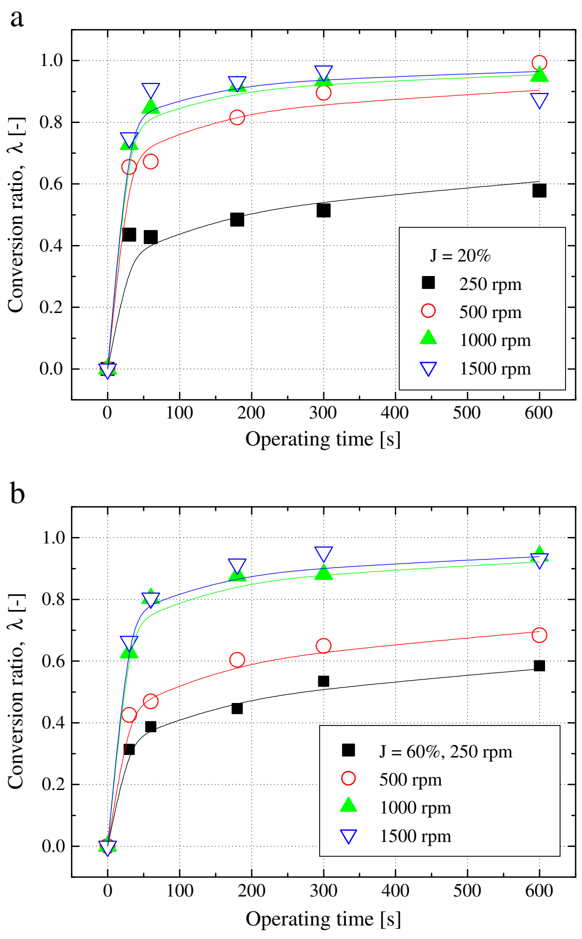

Fig. 8. Dependence of the conversion ratio on the operating time t and on the rotational speed ω for different filling ratios (a) $J = 20\%$, (b) 60%. The curves are those fitted by the exponential empirical model.

rotational speeds ω, and for two filling ratios $J=20\%$ (a), 60% (b). In general, the conversion ratio increases with an increase of processing time. The higher the speed of rotation the faster the conversion ratio increases. Higher rotation speeds of the mixer provide more energy to coat the host particles. The influence of the filling ratio is not observed clearly, however at smaller filling ratios the conversion ratio increases more rapidly. For $J=40\%$, analogous curves were observed.

All the data plotted in Fig. 8 indicate that the conversion ratio increases exponentially with an increase in processing time up to the asymptotic value of 1.0, the coating obtained when almost all the invited particles stick to the host particles. Hence the conversion ratio obtained at certain processing time λ(t) can be expressed by an empirical exponential equation with one parameter k_c given in Eq. (7).

$$\lambda(t) = 1 - e^{-k_c t^{0.25}} \tag{7}$$

The parameter k_c was obtained by the nonlinear least square fitting of the curves as shown in Fig. 8. The curves calculated by Eq. (7) present a fairly good agreement with the experimental results with correlation coefficients over 0.98. It can be concluded that this empirical equation is able to express the mass fraction of invited particle forming the coating with processing time. The parameter k_c represents the ability of the mixer to make a coating, and we shall call it the coating rate constant.

The k_c value obtained by comparing experiments and modeling has been plotted in Fig. 9. k_c increases with an increase in rotational speed, however it does not seem to vary linearly. In particular, at higher speeds from 1000 to 1500 rpm, the coating rate constant k_c does not increase very much. This indicates that there is an optimum rotational speed for coating the host particles and if an excess energy is given, it does not contribute to improving the coating but leads to heat dissipation, attrition and erosion due to too rapid particle flow. In general the coating rate constant does not seem to depend on the filling ratio but k_c does seem to increase at smaller filling ratios.

3.5. Relation between conversion ratio λ and particle properties

It is very interesting to study the relation between the mixture composition and the particle properties. If there is a relation between the mass fraction of invited particles forming a coating and the coated product properties, then product properties can be predicted. Furthermore, determining the mass fraction of the coating does not require special equipment and is simply determined by measuring masses after sieving.

Fig. 10 shows the relation between flowability and the conversion ratio λ as shown in Fig. 8. It is observed that even though the plotted points are widely spread, the FRI is linearly dependent of the conversion ratio. The flowability depends not only on the conversion ratio but also on the shape and angularity of the particles, which explains why the initial point is located below the line.

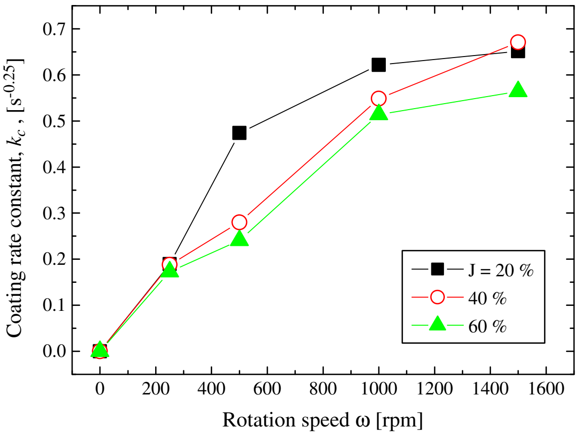

Fig. 9. Coating rate constant k_c as a function of rotational speed.

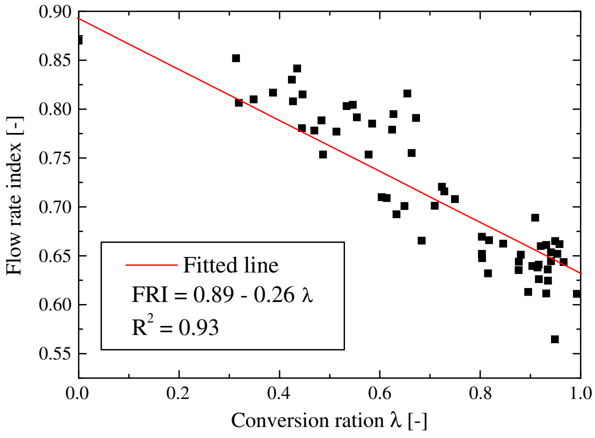

Fig. 10. Relationship between the conversion ratio and the FRI-fitted line.

Fig. 11 presents a linear relation between the contact angle and the conversion ratio λ. The plotted points are seen to be more widely scattered than that of flowability data. One possible reason is the powder bed used to determine the contact angle was not compressed and thus the surface was not perfectly uniform, thus inducing experimental errors. To alleviate this problem the contact angle was measured at 3 different locations on each sample. The physical state of the surface, characterized for example by the porosity of the surface, is a significant factor in determining the contact angle.

To summarize, there seems to be a linear relationship the physical properties of the coated particles and the conversion ratio, which indicates that the physical properties of the coated particles can be estimated by the conversion ratio which gives the information on the degree of efficiency of the coating process.

4. Conclusion

Investigations on the effects of the operating parameters when using Cyclomix mixer to carry on dry particle-particle coating has been carried out using model powders: Suglets as a host particles and MgSt as invited particles. The coated particles produced were characterized for flowability, wettability and mass fraction of

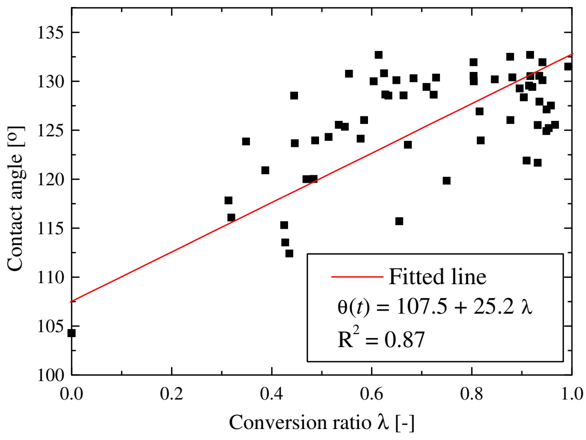

Fig. 11. Relationship between the conversion ratio and the contact angle θ-fitted line.

invited particles forming the coating. The results are summarized below.

1) The surface morphology of the coated particles indicates that, at first, the invited particles are distributed as discrete particles on the surface of the host particles. Then, as processing time increases, the invited particles fixed on the host particles are deformed by mechanical action such as shear and impact forces, and the coating changes from being in the form of discrete particles to become a film-like coating. At high rotational speeds, these phenomena proceed rapidly, rearrangements of the superficial grains occur so that a somewhat continuous film-like coating is obtained, even at short processing times.
2) Determination of the wettability of the coated particles shows that it can change dramatically from hydrophilic to hydrophobic even for short processing times and at low speeds of rotational. The contact angle determined by the sessile drop test increases with an increase in the processing time and also with the speed of the rotation. The wettability is not sensitive to the filling ratio.
3) The flowability of the coated particles improves with processing time. The improvement in the flowability proceeds faster at higher speeds of rotation. The filling ratio is not a significant factor in coating performance.
4) The conversion ratio of the invited particle to form a coating increases exponentially to reach an asymptotic value around the value for an ideal coating. This behavior has been fitted by an exponential model with one variable parameter defined as k_c. The fitted curved represents the mass fraction of invited particles forming a coating with processing time.
5) A linear relationship is found between the properties of the coated product and the conversion ratio which gives the information on the degree of efficiency of the coating process, even though there is some variability.

In conclusion, it is found that the properties of the coated particles can be predicted by the conversion ratio at a given time in the coating process. The linear relation between the mass fraction of invited particles on the surfaces of the host particles and the product properties can be quantified by a model involving a coating rate constant k_c, which can be used to optimize dry coating processes to give desired properties of coated particles.

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