

Particle–particle coating in a cyclomix impact mixer

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A B S T R A C T

Dry particle coating can be used to create new-generation materials by combining different powders exhibiting different physical and/or chemical properties. In such processes relatively large particles (host particles) are mechanically coated with fine particles (guest particles), without using solvents and subsequent drying, to create new functionalities or to improve initial characteristics. The coating of a host powder by guest particles can be performed in many different ways ranging from simple stirring of two components, to high energy impact coating.

The purpose here is to examine the use of a Cyclomix high shear mixer granulator for the surface modification of silica particles by dry coating with magnesium stearate, and of cornstarch dry coated with hydrophilic or hydrophobic fume silica. Several powder characterisation methods have been used to study the physico-chemical properties of the coated particles. These include: Observation by environmental scanning electron microscopy (ESEM), Characterization of cohesion and flowability by tap density measurements, Determination of wettability by measurements of the contact angle between the coated particles and water. Measurement of moisture adsorption-desorption isotherms of uncoated and coated particles, and Tribo-electrification characteristics of the powders. The results show that the surface properties of the two host particles are changed, and that the Cyclomix can be used for dry particle coating to modify the properties of silica gel and cornstarch powders.

Keywords:

Dry coating
Starch
Silica gel
Magnesium stearate
Fume silica
Cyclomix
Surface morphology
Moisture adsorption
Flowability
Tribo-charging

1. Introduction

Dry particle coating can find application in many industries for changing the surface properties and/or functionality of powders. For example it may be used in the pharmaceutical, cosmetic, food or ceramics industries for modification of flowability, wettability (hydrophobic/hydrophilic properties), dispersability and rate of dissolution, flavour, particle shape, electrostatic, optical, electric, magnetic, particle properties. As indicated in Fig. 1, in such coating processes, powders with relatively large particle sizes (host particles: 1–500 μm) are mechanically coated with fine particles (guest particles: 0.1–50 μm) in order to create a new functionality or to improve their initial characteristics [1]. The success of the process depends on the “affinity” of the guest particle for the host particles and the mechanical forces brought to bring them together. Since the guest particles are very small, Van der Waals interactions are strong enough to keep them firmly attached to the large host particles [2]. Thus, either a discrete or continuous coating of guest particles can be achieved depending on the operating conditions such as mechanical force used, processing time, weight fraction of guest to host particles and particle properties [3]. Multiple layering is possible when using different coating materials and processing them

one after other. Kangwantrakol et al. (2001) [4] have described a method to make multilayer coating of guest particles.

Dry particle coating is very closely related to ordered mixing (a term coined by Hersey, 1975), where a mixture is formed by covering the surface of larger particles with smaller particles. However in dry particle coating, the aim is to make the surface covering more permanent with stronger physical (or chemical) bonding [3]. Initial work on ordered mixing was done by Hersey [5]. The concept of ordered mixing was also taken one step further (to dry coating) by using dry impact blending, as described in a series of papers by a Japanese group [6–9]. Several different machines have been developed for dry coating allowing the creation of new types of materials. Most of the devices which have been used for dry powder coating are rather specialised laboratory type equipment and not readily available for processing amounts greater than several grams of powder. Pfeffer [3] gives descriptions of these different dry coating systems. Mujumbar et al. [10] have studied dry coating to enhance the moisture resistance of ground magnesium powder by coating its surface with carnuba wax using Magnetically Assisted Impaction Coating (MAIC) and two high-speed impaction devices, the Hybridizer and Mechanofusion device. Yang et al. [11] showed that it is possible to improve the flowability of cornstarch by coating with nanosized silica, using dry coating devices such as MAIC and Hybridizer.

Here we describe an experimental investigation of dry powder coating using a “Cyclomix” high shear mixer. This apparatus is a vertical cone shaped high shear mixer made by Hosokawa Micron and is

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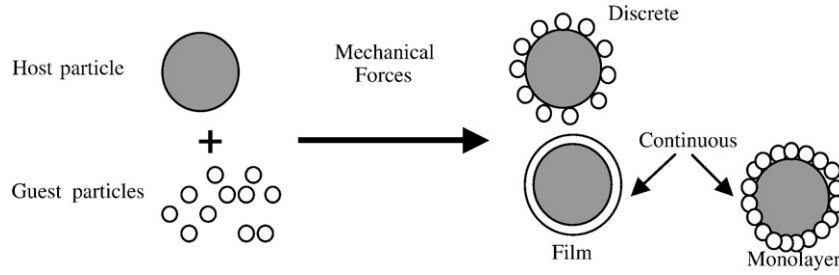


Fig. 1. Dry powder coating.

available in a range of sizes from 1 L to 500 L. Ng et al. [12] presented an experimental study on the solids motion in a Cyclomix by the positron emission particle tracking technique. Kwan et al [13] studied the granulation of hollow glass beads in a range of sizes of Cyclomix for scale-up. Here we report experiments which have been performed using a Cyclomix for mechanical dry coating of silica gel particles with magnesium stearate, and for coating starch particles with hydrophilic and hydrophobic fume silica. The effects of these surface coatings on the flowability, the wettability and tribo-charging are determined.

In a previous paper on dry particle coating [14] we presented an experimental technique based on dry powder particle size analysis for determining the force required to remove the coating layer. This allowed us to compare the mechanical resistance of coatings made using different techniques such as manual shaking, a Turbula mixer, and a Hybridizer. The advantage of the Cyclomix is that it can treat large quantities of powder and is available for industrial processing, and one of the aims of this paper is to evaluate the resistance of a Cyclomix coating with that made by a Hybridizer.

2. Experimental

2.1. Powders

Silica gel powder supplied by Merck and usually used for chromatography column has been chosen as host particles for dry coating. This silica gel has a hydrophilic porous structure which readily absorbs moisture and as shown in Fig. 3(a) the particles are irregularly shaped and their surface is rough. Hydrophobic magnesium stearate (MgSt) supplied by Chimiray is used as guest particles. The MgSt is a fine, white, greasy and cohesive powder widely used in pharmaceutical formulation as a lubricant. Observation by ESEM showed a wide distribution of size and shape including needle and plate like particles. The starch was Corn Starch B from Roquette Freres. The fumed silica was from Degussa; Aerosil R200 hydrophilic, Aerosil R974 hydrophobic. The properties of host and guest particles used in the experiments are summarized in Table 1.

Table 1
Properties of host and guest particles

Particles	Size d_{50} μm mastersizer	Solid density g/cm^3 HeliumPycnometer	Wetting characteristics	Specific surface (m^2/g) (ASAP 2010)
Silica gel	55	2.07	Hydrophilic	510
Cornstarch	13	1.47	Hydrophilic	–
Aerosil R200	0.12	2.27	Hydrophilic	200
Aerosil R974	0.12	2.27	Hydrophobic	174
MgSt	4.6	1.04	Hydrophobic	6

2.2. Coating processes

The mass percentage of guest particles required in a coating experiment is calculated based on the assumption of 100% surface coverage of the host particles with a monolayer of guest particles. It is assumed that both host and guest particles are spherical (respective sizes D_{host} , D_{guest} , respective densities ρ_{host} , ρ_{guest}), and do not change shape during the coating process. Based on these assumptions, the mass percentage (W) of guest particles for 100% coverage can be written as [10]:

$$W(\%) = \frac{(ND_{\text{guest}}^3\rho_{\text{guest}})}{(D_{\text{host}}^3\rho_{\text{host}}) + (ND_{\text{guest}}^3\rho_{\text{guest}})} \times 100 \quad (1)$$

For $D_{\text{host}} \gg D_{\text{guest}}$ (here, $D_{\text{host}}/D_{\text{guest}} \approx 10$), the number N of guest particles per host particle is given by the expression:

$$N = \frac{4(D_{\text{host}} + D_{\text{guest}})^2}{D_{\text{guest}}^2} \quad (2)$$

From Eq. (1), the percentage of guest particles needed to coat silica gel particles is determined to be 15%. In addition, coating experiments have been carried out with 1% and 5% of magnesium stearate in order to compare what happens mass ratios of guest to host particles lower than 15%. The percentage of fume silica guest particles needed to coat starch particles is determined to be 1%.

A 1 L capacity Cyclomix (Hosokawa Micron B.V.) high impact mixer is used for the coating experiments. The mixer has four pairs of flat-bladed impellers located in series on a centrally located high-speed top driven rotating shaft. This eliminates seals and bearings from the product zone. The shaft is fitted with paddle-shaped mixing elements, which rotate close to the inner vessel wall [12]. Fig. 2 shows a schematic diagram of the action of a Cyclomix mixer. The working principle of the Cyclomix differs from existing mixing techniques and from other devices used for dry

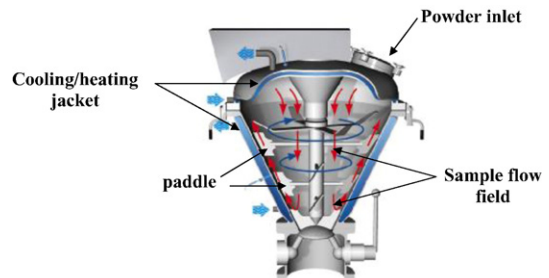


Fig. 2. Schematic diagram of a Cyclomix high shear mixer.

powder coating by the specific interaction between mixing element and vessel wall. The powder (host and guest particles) is loaded into the conical mixing vessel from the top; the degree of filling can range between 30 and 100%. The high-speed of rotation (up to 2500 rpm) of the paddles and the conical shape of the vessel force the product from the bottom to the upper zone of the vessel. Upon reaching the top, the product flows downwards into the centre of the vessel. This flow pattern results in fast macromixing. During the upward motion, the particles are accelerated by the paddles and intensively mixed by friction with vessel walls. The operating conditions used in all experiments presented here are 80% filling and 1500 rpm for 5 min.

3. Characterization

The uncoated and coated particles were examined by means of an environmental scanning electron microscopy (ESEM) to study the surface morphology and particle shapes before and after coating. A Volumeter (or Tap-tap) tapped density tester was used to examine the flowability of the uncoated and coated samples. The tapped density was obtained by taking volume readings of a known weight (m) of powder sample contained in a measuring cylinder and subjected mechanical tapping. To reduce variability due the action of filling the cylinder the "initial" volume is taken as that after 10 taps (V_{10}) the final volume is that

when no change in volume takes place (V_{∞}). The flow properties and cohesion of the uncoated and coated powders were evaluated by the Carr's flowability index (IC) and Hausner's ratio (HR).

$$\rho_0 = m/V_{10} \quad \rho_{\infty} = m/V_{\infty}$$

$$\text{Carr index IC} = \frac{\rho_{\infty} - \rho_0}{\rho_{\infty}} \%$$
(3)

$$\text{Hausner ratio HR} = \frac{\rho_{\infty}}{\rho_0}$$
(4)

Flowability is classed as a function of IC as follows:

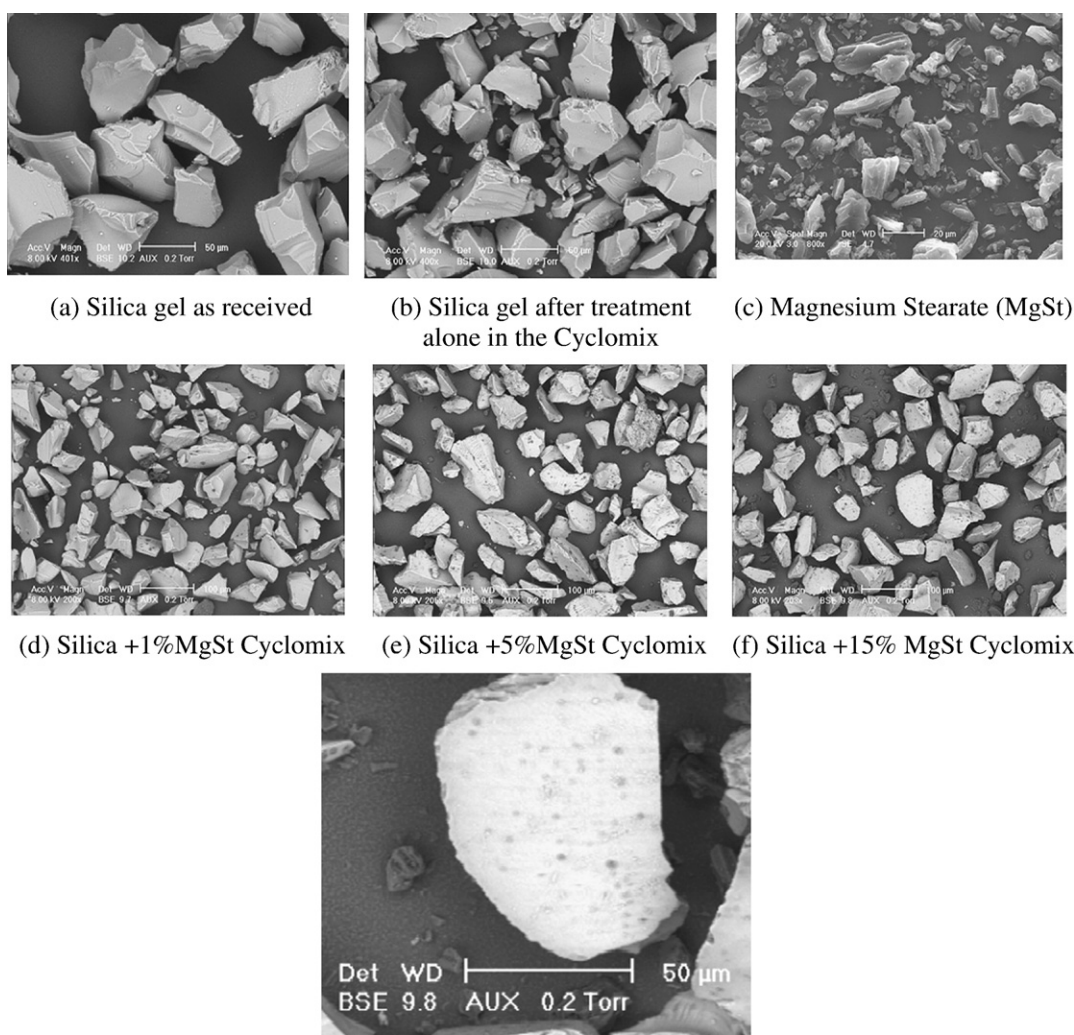
<15% good 15 to 25% poor >25% bad

Cohesivity is classed as a function of HR as follows:

1.0 to 1.2 low cohesion, 1.2 to 1.4 medium cohesion, >1.4 high cohesion.

The wettability was studied by the sessile drop method using an ILSM instrument (GBX S.A.) [15,16]. A small water drop of 10 μ l is deposited on the surface of a powder bed prepared for each sample. The shape of the drop profile is observed and used to determine the contact angle.

The moisture adsorption-desorption isotherms of uncoated and coated particles were obtained by using a Dynamic Vapour Sorption (DVS) analyzer (Surface Measurement Systems Ltd. UK).



Enlargement of the central particle in figure 3(f)

Fig. 3. ESEM photos of the silica gel magnesium stearate system.

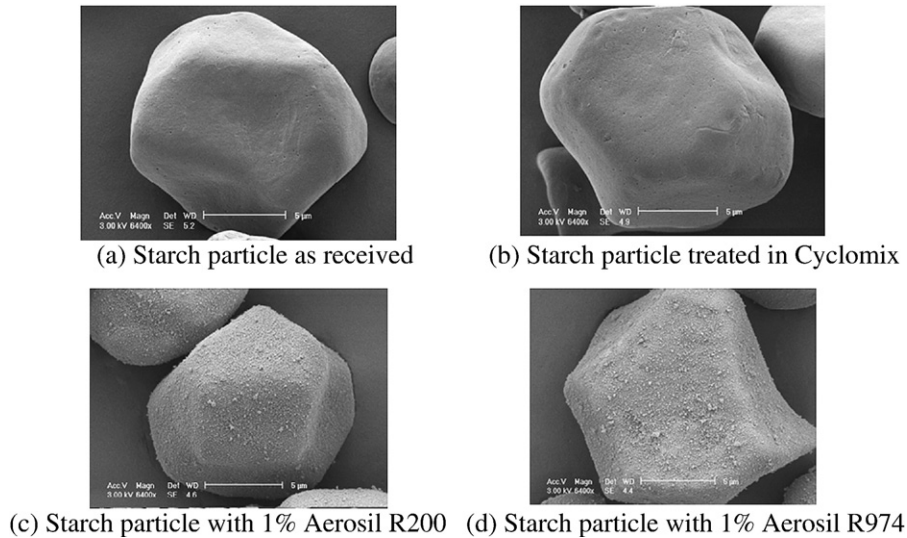


Fig. 4. ESEM photos of starch particles Fumed silica system.

Tribo-electric charging experiments were made with approximately 8 g samples of the powders rotated in a 250 ml stainless steel beaker held at an angle of 45°, and then pouring the powder into a Valitec Faraday cage connected to a Keithley 6514 electrometer [17].

4. Results and discussion

4.1. Surface morphology

4.1.1. Silica gel magnesium stearate experiments

As shown on Fig. 3(a), the silica gel before treatment has irregularly shaped particles. After processing in the Cyclomix, the mean size of the silica gel particles was found to be slightly smaller probably because of some grinding action by the high shear in the mixer (Fig. 3(b)). The magnesium stearate Fig. 3(c) has a wide size distribution and a large variety of particle shapes including needles and platelets. The surface morphology of particles coated with 1%, 5% and 15% (w/w) of MgSt in the Cyclomix is shown in Fig. 3 (d to f). The guest particles are individually distributed on the surface of silica gel particles (see Fig. 3(f) and the enlargement in Fig. 3(g)). The coverage increases as the percentage of MgSt is increased from 1% to 5% to 15% w/w. Coating seems to be more uniform for the particles coated with 15% of MgSt which seems to be more smeared over the surface.

4.1.2. Starch fumed silica experiments

Fig. 4(a and b) shows that the starch particles are rounded and smooth. There was no significant breakage due to processing in the Cyclomix. The coating with fumed silica is seen (Fig. 6 a and b) to be as individual particles set in the surface.

4.2. Coating resistance tests

The resistance of the coating was determined using the technique based on dry powder particle size analysis technique presented elsewhere [14]. Figs. 5 and 6 show the particle size distributions of the silica MgSt systems obtained with a Malvern Mastersizer dry feed system with low 0.5 bar (Fig. 5) and high 3.5 (Fig. 6) dispersing pressure. The volume distributions show very little difference. The number distributions show that at 3.5 bars pressure there is a small release of fine MgSt particles but that the coating is substantially still in place. Similar results were obtained for the Starch Fume silica system.

These results, and comparison with results presented elsewhere [14] for the Nara Hybridiser, indicate that the Cyclomix does give mechanically resistant coatings with these two sets of powders. The number distribution curve for the 15% MgSt coating at 3.5 bar dispersing pressure (Fig. 6(b)) indicates a certain degree of deagglomeration, but the fact that the curve does not fully correspond

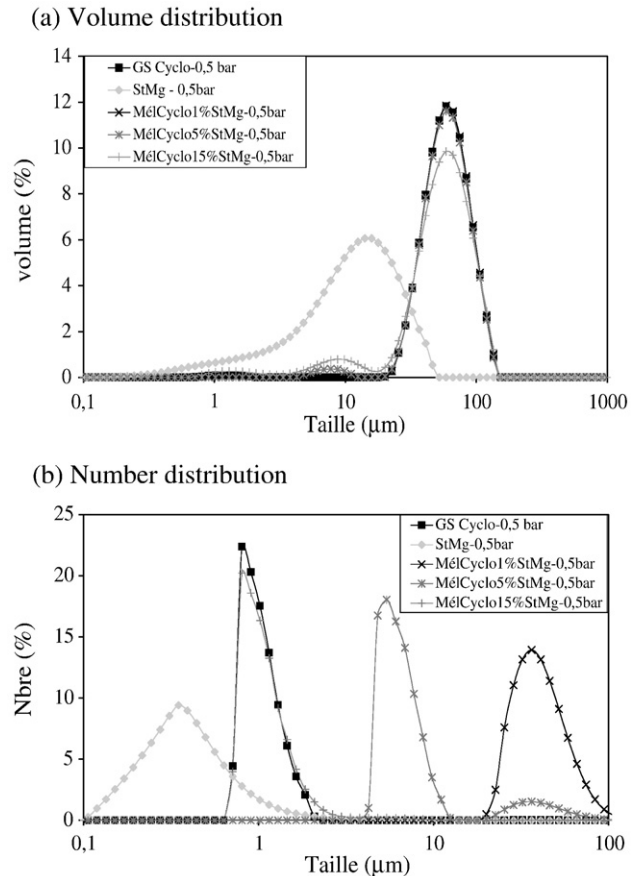
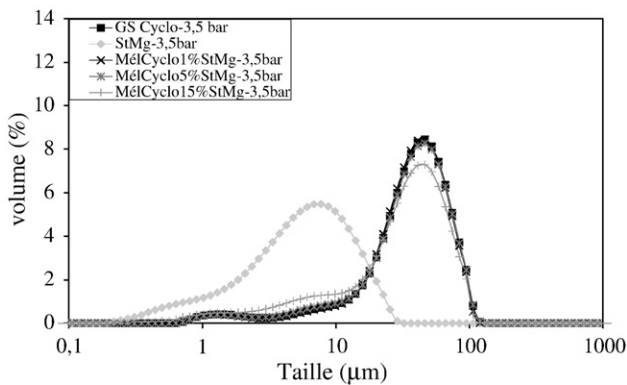


Fig. 5. Particle size distributions silica + MgSt at 0.5 bar dispersing pressure.

(a) Volume distribution



(b) Number distribution

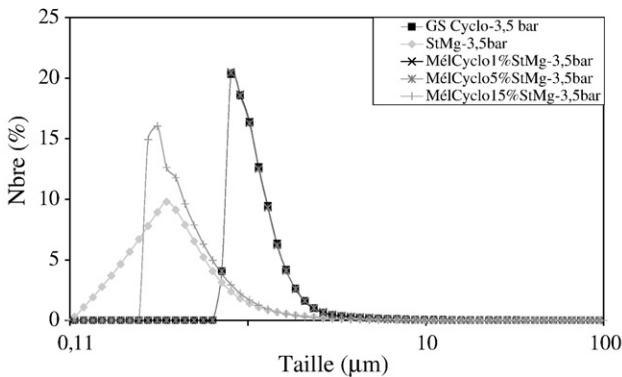


Fig. 6. Particle size distributions silica + MgSt at 3.5 bar dispersing pressure.

to that of free MgSt indicates there is not full liberation of the fine silica particles.

4.3. Flowability tests

4.3.1. Silica gel and magnesium stearate

The values of the Carr's flowability index (IC) and Hausner's ratio (HR) of uncoated and coated silica gel particles and MgSt as determined from tap-tap settling tests are given in Table 2. The as-received silica gel powder presents good flow properties (IC=15.8; HR=1.25). This is slightly changed by processing with the Cyclomix probably due to slight breakage. The MgSt powder has bad flow properties because of its high cohesivity (IC=34.1; HR=1.5). The coated particles have characteristics intermediate between those of the initial powders, i.e. poor to bad flowability.

4.3.2. Starch and fume silica

Fig. 7 shows the variation of the tapped density measured for the starch coated with 1%, of both types of Silica in the Cyclomix. The

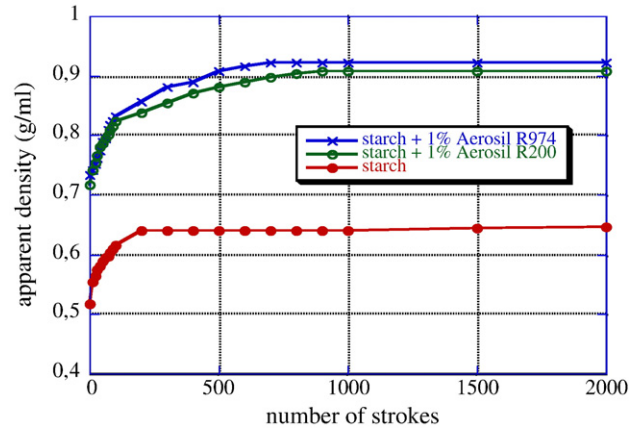


Fig. 7. Tap density kinetics for binary mixtures of starch fume silica.

results are compared to those obtained for untreated starch particles. It can be seen that the particles treated with Aerosil have higher bulk densities (0.91 to 0.92 g/ml) than the starch alone (0.65 g/ml) indicating that the silica coating improves packing. Furthermore it can be seen that the settling for the starch alone is essentially ended after 300 taps whereas the curves for the treated samples requires up to 1000 taps. The Carr and Hausner indices indicate that the silica coating brings only slight changes but that the hydrophobic silica has a slightly greater effect (Table 3).

4.4. Wettability tests

4.4.1. Silica gel and magnesium stearate

Since MgSt is hydrophobic, its use as a coating should make the hydrophilic silica gel surface become hydrophobic. To evaluate the impact of coating on the reduction of the high affinity between silica gel and water, wettability tests were carried out for the uncoated particles and the silica gel particles coated in the Cyclomix. The values of the measured contact angle (at $t=0s$) and the work of adhesion (W_{adh}) describing the interaction between two different phases (the required work to extract the water drop from the surface of powder bed) are summarized in Table 4.

Fig. 8 shows photos taken after two different times of a water drop placed on the uncoated silica gel powder bed. It can be seen that the water drop disappears instantaneously (after 1s) into the powder because of the high affinity between silica gel and water.

As for silica gel powder, the water drop disappears rapidly from the surface of powder bed prepared for particles coated with 1% of MgSt (after only 3 s; Fig. 8(a)) whereas the contact angle increased and the work of adhesion decreased. The mass ratio of 1% of MgSt gives a very poor coating of the host particles and is insufficient to reduce the high affinity between silica gel and water. After coating with 5% and 15% of MgSt (Fig. 8(b), (c)), the water drop is not absorbed and remains on the surface of silica gel powder. The measured contact angle increases when the MgSt percentage increases whereas the work of adhesion decreases (Table 4). These results clearly prove that, on one hand the surface of the silica gel particles is coated by the MgSt particles and on

Table 2

Carr index (IC) and Hausner ratio (HR) Silica gel MgSt system

Samples	ρ_0 g/ml	ρ_∞ g/ml	IC	HR	Cohesion	Flowability
Silica gel (SG)	0.465	0.579	15.8	1.25	Low	Good
Cyclomix SG	0.478	0.568	19.7	1.19	Low	Poor
SG + 1%MgSt	0.456	0.564	19.1	1.24	Low	Poor
SG + 5%MgSt	0.426	0.560	23.9	1.31	Medium	Bad
SG + 15%MgSt	0.406	0.533	23.8	1.31	Medium	Bad
MgSt	0.296	0.450	34.1	1.52	High	Bad

Table 3

Carr's index (IC) and Hausner's ratio (HR) for the Starch fume silica system

Sample	ρ_0 g/ml	ρ_∞ g/ml	IC	HR	Cohesion	Flowability
Starch	0.555	0.647	14.2	1.17	Low	Good
Starch + 1% aerosil R200	0.741	0.907	18.3	1.22	Medium	Poor
Starch + 1% aerosil R974	0.742	0.921	19.4	1.24	Medium	Poor

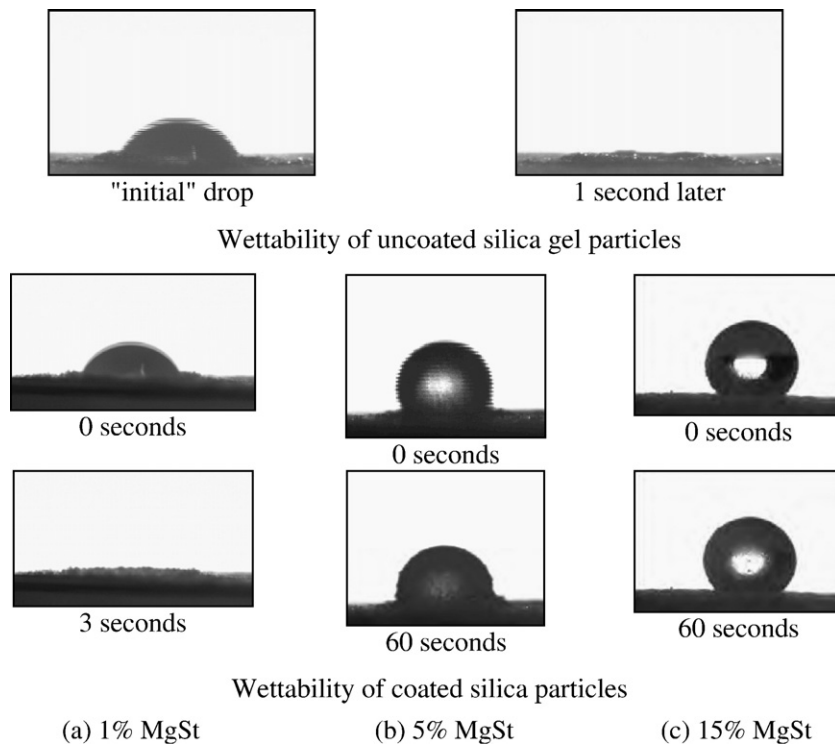


Fig. 8. Wettability of coated silica particles.

the other hand that the affinity of the silica gel powder for water is reduced by dry coating in the Cyclomix.

4.4.2. Starch fumed silica system

Starch system is too hydrophilic for the sessile drop test to be applied. The liquid is immediately taken up by the starch and the drop does not remain on the powder surface. Coating with fume silica does not change this situation.

4.5. Water vapour sorption and desorption isotherms

4.5.1. Silica gel magnesium stearate system

Moisture sorption isotherms were obtained by measurement of moisture uptake using a DVS apparatus. Prior to being exposed to water vapour, the samples (100 mg) are equilibrated at 0% relative humidity (RH) to establish a dry mass baseline. Experiments were performed with samples of untreated silica gel and silica gel coated with MgSt. The kinetics of adsorption was determined by following moisture uptake from 0% to 60% relative humidity with time. This is shown in Fig. 9. Equilibrium adsorption isotherms were then measured at 25 °C from 0% to 90% RH in increments of 10% RH. These are shown on Fig. 10. It can be seen in Fig. 10 that the kinetics of moisture uptake is slowed by the presence of the 5% and 15% MgSt coating. In addition the final plateau reached shows that amount of moisture taken up is also changed. This equilibrium effect is found on Fig. 10 of the overall moisture adsorption isotherm. The 1% MgSt coating has little effect as it can be seen that the kinetics curve and the

adsorption isotherm are very similar to the results for silica gel alone. As for wettability tests results, the quantity of MgSt (1%) used was not sufficient to reduce the uptake of water on silica gel after coating.

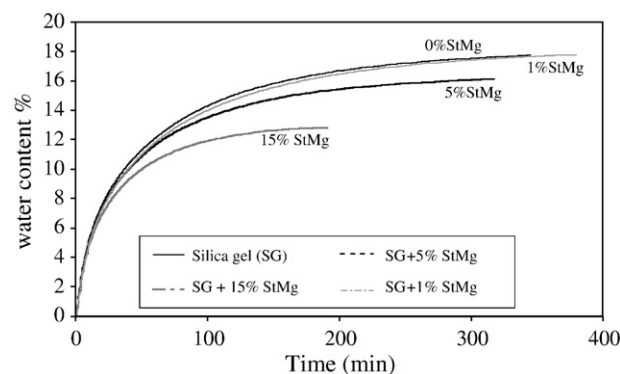


Fig. 9. Kinetics of moisture adsorption at 25 °C, silica gel and silica gel coated with MgSt.

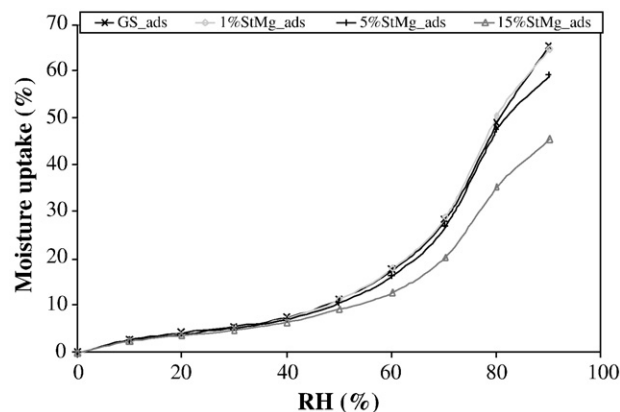


Fig. 10. Moisture adsorption isotherm 25 °C, silica gel and silica gel coated with MgSt.

Table 4
Results of the measured contact angles and the work of adhesion

Samples	Average contact angle (θ°)	W_{adh} (mN m ⁻¹)
Silica gel	15.2	112
1% MgSt Cyclomix	80	86
5% MgSt Cyclomix	98	62
15% MgSt Cyclomix	127	29
MgSt	125	32

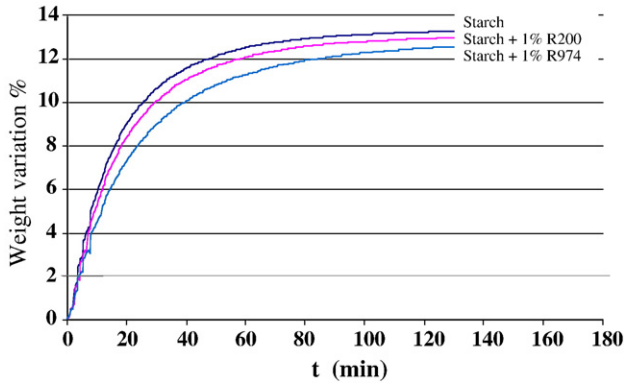


Fig. 11. Moisture adsorption kinetics for a step change from 0 to 60% relative humidity.

The total amount of the water sorbed decreased by almost 10% for silica gel particles coated with 5% of MgSt and 30% for particles coated with 15% of MgSt. This suggests that the 5% and 15% MgSt coating impermeabilise the silica gel surface and may be in the form of a continuous film.

4.5.2. Starch, fume silica system

Fig. 11 shows the kinetics of moisture absorption of starch and starch coated with R200 (hydrophilic) and R974 (hydrophobic) fume silica. It can be seen that the silica coatings have a slight slowing down effect of moisture uptake and that the hydrophobic coating slows uptake more than the hydrophilic coating. However the final plateau value reached is practically the same in each case indicating that overall amount of moisture taken up is the same. This means that the absorption isotherm is unchanged by the silica coatings. This is in contrast to the results with MgSt coatings given above, and could suggest that the fume silica is in the form of isolated individual particles and does not prevent moisture reaching the starch.

4.6. Tribo-electric charging

4.6.1. Silica gel magnesium stearate system

Fig. 12 shows the tribo-charging characteristics for the silica gel alone and coated with 1%, 5%, and 15% MgSt. It can be seen that the uncoated silica gel has an initial negative charge of about $1.5 \mu\text{C}$ per gram and which remains over the test period of 30 min rotation in the stainless steel beaker. The silica gel samples coated with MgSt have a positive charge of around $0.5 \mu\text{C}$ per gram and this remains constant over the experiment. There is no substantial difference in the results for the

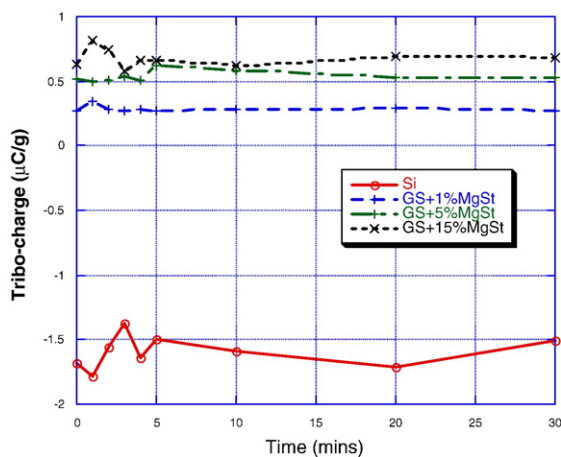


Fig. 12. Tribo-charging characteristics for silica gel and silica gel coated with MgSt.

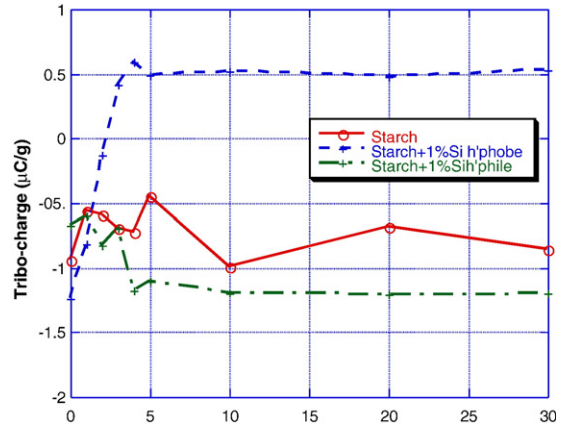


Fig. 13. Tribo-charging characteristics for starch and starch coated with fume silica.

different coatings except that for the 1% coating is lower than the other two, and the 15% seems to be slightly higher than the results for the 5% coating.

4.6.2. Starch, fume silica system

Fig. 13 shows the tribo-charging characteristics of the untreated starch and starch coated with R200 (hydrophilic) and R974 (hydrophobic) fume silica. It can be seen that the starch by itself has a negative charge of around $0.8 \mu\text{C}$ per gram, which remains stable over the duration of the experiment. The results for the starch coated with hydrophilic fume silica are similar with a negative charge of about $1 \mu\text{C}$ per gram. The results for the starch with the hydrophobic coating are very different. The initial charge is similar to the others at a value of about $-1 \mu\text{C}$ per gram. This rises rapidly over the first 5 min to become positive at a value of about $0.5 \mu\text{C}$ per gram.

5. Conclusions

This study indicates the Cyclomix can be used for dry powder coating of particles and that the mechanical resistance of the coatings is good, as determined by a laser diffraction particle size dispersion test. Two powder-powder systems have been tested: silica gel particles coated with hydrophobic magnesium stearate and starch particles coated with either hydrophilic or hydrophobic fume silica. The silica gel treated alone in the Cyclomix was slightly fragmented and the starch was relatively undamaged. The ESEM images of the uncoated and coated particles showed that MgSt seemed to be smeared over host particles and this effect increased as the MgSt mass ratio increased to 15%. The two types of fume silica were fixed on the surface as individual particles.

The changes in overall powder characteristics have been determined by tap-tap test for cohesion and flowability using the Carr and Hausner indices, the wettability by sessile drop and DVS measurements and by tribo-charging tests. The flowability of the silica gel powder was significantly decreased after treatment the Cyclomix mixer with 15% of MgSt. It is also found that the coating by hydrophobic MgSt in the Cyclomix can also reduce the high affinity between silica gel and water after treatment with 5% and 15% of MgSt. The tribo-charging tests show that coating the Silica gel with MgSt changes them from negative to positively charged particles. The settling characteristics of the starch particles was significantly changed by coating with fume silicas giving high bulk densities. However the Carr and Hausner indices were found not to be able to discriminate. The overall moisture adsorption isotherm was little changed by the coating but the kinetics of moisture uptake was found to be significantly slower for the coated starch particles. Finally the tribo-charging tests show a significant different in the case of the hydrophobic coating. To summarize, it has been demonstrated that the high shear

mixer "Cyclomix" can be used as dry coating device to change the properties of host powders.

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