Optimisation of process parameters in high energy mixing as a method of cohesive powder flowability improvement

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Flowability of fine, highly cohesive calcium carbonate powder was improved using high energy mixing (dry coating) method consisting in coating of $CaCO_3$ particles with a small amount of Aerosil nanoparticles in a planetary ball mill. As measures of flowability the angle of repose and compressibility index were used. As process variables the mixing speed, mixing time, and the amount of Aerosil and amount of isopropanol were chosen. To obtain optimal values of the process variables, a Response Surface Methodology (RSM) based on Central Composite Rotatable Design (CCRD) was applied. To match the RSM requirements it was necessary to perform a total of 31 experimental tests needed to complete mathematical model equations. The equations that are second-order response functions representing the angle of repose and compressibility index were expressed as functions of all the process variables. Predicted values of the responses were found to be in a good agreement with experimental values. The models were presented as 3-D response surface plots from which the optimal values of the process variables could be correctly assigned. The proposed, mechanochemical method of powder treatment coupled with response surface methodology is a new, effective approach to flowability of cohesive powder improvement and powder processing optimisation.

Keywords: flowability, cohesive powder, dry coating, planetary ball mill, response surface methodology

1. INTRODUCTION

Flowability is an important property of powders composed of fine particles and the particle diameter of 100 μm or below is supposed to be the value that limits the flow significantly. In many process industries operations including cohesive materials are common practice particularly in those centered on food, chemicals and pharmaceuticals. Powder flow behaviour is critical where the design and operation of industrial equipment is concerned, as well as the avoidance of such problems as developing arching in silos, segregation in solids and other problems often resulting in process stoppage or poor-quality product (Jürgen and Kleinschmidt, 2009; Schulze, 2008; Zhou et al., 2010). To avoid these difficulties, vibration, aeration and often an addition of lubricant or glidant are used. Although glidants reduce interparticle forces of the bed particles and improve flowability, there is a problem with dispersing them into the bed due to small sizes (d_p < 1μm) and cohesive properties resulting in powder agglomeration. Flow additives have to be mixed with a cohesive powder but conventional mixing is often not sufficient to break up the agglomerates of both powder and admixture particles. As a result mixtures with flow additives may be not uniform and segregation due to differences in size, shape and density may appear (Zhou et al., 2011). Another method that is widely applied for flowability improvement of cohesive powders is granulation. This method is a common practice due to low investment and operating costs and

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advantageous properties of the final product for example better flowability or dust reduction. However, the main drawback of granulation is not uniform particle size distribution, so particle classification and even comminuting are often needed. This requires the use of additional devices and increasing energy input (Gluba, 2012).

A method that has recently proved to be an effective way of cohesive bulk solid flow improvement is high energy mixing. The general idea of the method is to deliver to the agglomerated powder bed some mechanical energy which is needed for development of processes in the bed which are of mechanochemical nature and result in changing the physical or even chemical properties of bed components. If the amount of energy is large enough, the agglomerates are breaking apart and individual particles are released within the whole powder bed. The released (host) particles can be then covered by smaller (guest) particles intentionally introduced to the bed in the form of a small admixture. In this way host particles are separated by guest particles and there is no agglomeration of original, host particles in the bed any longer. This is called interactive or ordered mixing, dry coating or mechanofusion (Alonso and Alguacil, 1999; Pfeffer et al., 2001; Saharan et al., 2008).

The method of dry coating is an effective way of flow enhancement provided that the forces between host and guest particles are stronger than host-host and guest-guest forces. This is usually the case if the dimensions of the guest particles are much smaller as compared to the host ones and, as is believed, to obtain this effect, the difference of two or three orders of magnitude in particle sizes is required. This allows that the van der Waals host-guest interactions are much stronger that other interparticle forces existing in the powder bed and the resulting mixture is therefore permanent. This method is used to design host particles with new surface properties and functionality, for example particulates with better flowability (Mullarney et al., 2011; Yang et al., 2005; Zhou et al., 2010; Zhou et al., 2011), dispersibility, wettability and dissolution (Tay et al., 2012), some new materials of improved electrostatic, electric, magnetic and optical properties, as well as particles of better sphericity and solid reactivity (Pfeffer et al., 2001).

There are many methods and devices used for particle dry coating, especially those manufactured in Japan, e.g. *Mechanofusion* by Hosokawa Micron, *Hybridizer* by Nara Machinery and *Theta Composer* by Tokuju Company. Similar equipment is manufactured in the Unites States, e.g. *Magnetically Assisted Impaction Coater* (*MAIC*) by AVEKA or *Rotating Fluidized Bed Coater* (*RFBC*) invented by Particle Technology Center, New Jersey Institute of Technology (Pfeffer et al., 2001). In the present work a planetary ball mill was used and it seems to be a promising tool for dry coating as was shown by Sonoda et al. (2008) who applied it to coat starch particles with poorly water-soluble drug – flurbiprofen in order to achieve better solubility of the drug in water.

The objective of this work was to improve flowability of cohesive calcium carbonate powder using dry coating method. Calcium carbonate is a material often used in polymer composites as a filler, in production of paper and pigments, and also in pharmaceutical industry as an excipient. The advantage of using smaller particles is better quality of the final product, but cohesion problems often of unpredictable nature are usually the reason for difficulties in realisation of operations with fine powders (Jeong et al., 2009; Zhang and Huiren, 2014). To preclude problems related to cohesion and to improve flow properties of calcium carbonate, a mechanochemical process of dry coating is proposed. Larger host particles of calcium carbonate were coated with smaller guest particles of fumed silica using a planetary ball mill to accomplish the dry coating process in a powder bed. In order to obtain optimal process conditions for dry coating in a planetary ball mill, a statistical procedure called *Design of Experiment and Response Surface Methodology* was used.

2. MATERIALS AND METHOD

2.1. Materials and equipment

Finely ground calcium carbonate powder (CHEMPUR, pure) was used in this study for flowability investigation. The basic physical properties and PSD for $CaCO_3$ are presented in Table 1 and Fig. 1, respectively. Before treatment in a planetary ball mill, calcium carbonate powder was dried to obtain water content of a constant level (approximately 0.5%). As an admixture of guest particles, hydrophilic fumed silica (Aerosil®200, EVONIK) was used. The specific surface area of Aerosil was 200 m^2/g and its average particle size was 12 nm.

Table 1. Particle size distribution and physical properties of calcium carbonate

material	d_{50} [μm]	d_{10} [μm]	d_{90} [μm]	$d_n = \dfrac{d_{90} - d_{10}}{d_{50}}$	angle of repose [deg]	aerated bulk density [kg/m^3]	packed bulk density [kg/m^3]	compressibility index [%]
calcium carbonate	2.147	0.775	4.453	1.173	55	433	827	47.6

Particle size distribution of original and processed calcium carbonate powder samples was measured with laser diffraction using a Malvern *Masterizer 2000E* particle size analyser. Measurements were preceded by sample ultrasonication for 60 seconds to break up powder agglomerates. Particle size was determined at the assumption that the reference refractive index was equal to 1.520 for calcium carbonate and 1.390 for isopropanol.
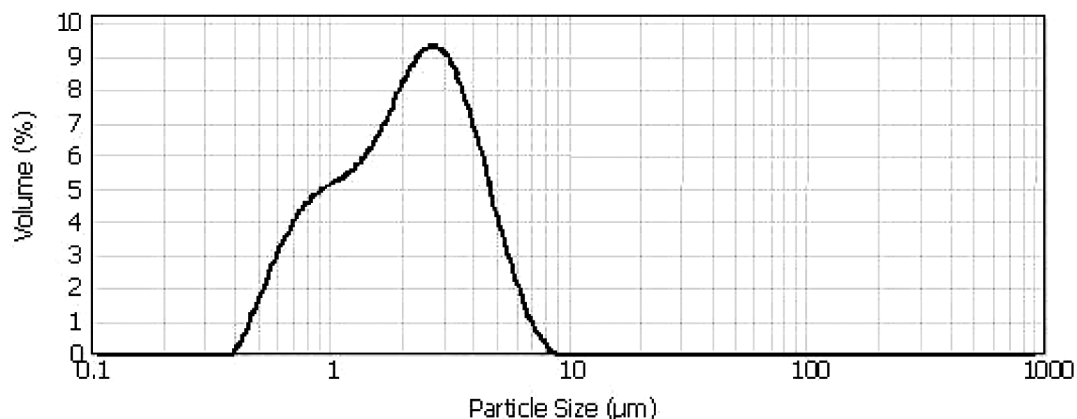

Fig. 1. Particle size distribution for calcium carbonate

The morphology of calcium carbonate samples was investigated using a scanning electron microscope (SEM) (Institute of Catalysis and Surface Chemistry Polish Academy of Sciences). Samples were sputter coated with gold using K575x Turbo Sputter Coater. SEM-EDS measurements data were collected by Scanning Electron Microscope Jeol JSM-7500F coupled with EDS-INCA PentaFETx3 EDX Spectrometer.

As a process control agent (PCA), isopropanol (STANLAB, analytical grade) was used. Using PCA is recommended by the manufacturer of milling/mixing devices (FRITSCH) to avoid caking and powder sticking to the operating device as well as to preclude secondary particle agglomeration (Suryanarayana, 2001; Zhang et al., 2004). PCAs, which mostly are ordinary, organic compounds, act as surfactants and are adsorbed on the surface of particles. It allows to avoid close contact of the particles and to prevent their agglomeration (Balaz, 2008). It is recommended in apparatus manuals when working with ultrafine powders of particle size under 10 μm.
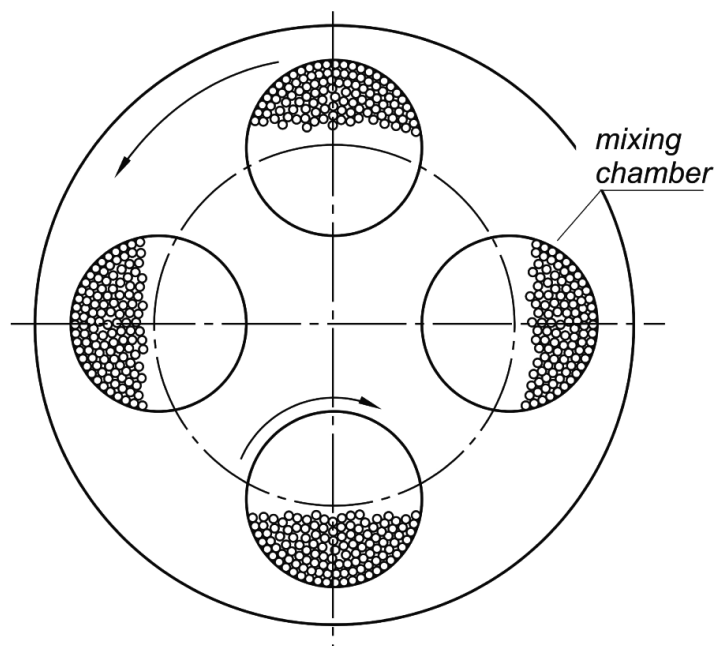
Fig. 2. Schematic representation of planetary ball mill

Planetary ball mill (FRITSCH Mono PULVERISETTE 6) that was used in the present work is schematically shown in Fig. 2. The device is usually used for high energy milling to obtain microsized particles. In this work, however, the energy produced in the mill was used for mixing different materials rather than for size reduction of the same material. The planetary ball mill consists of a mixing chamber (500 ml volume) filled with mixing balls (5 mm diameter) and placed on rotating basis plate. Both the chamber and the balls are made of zirconium oxide. The working chamber is attached to the basis plate which rotates around the central axis, while the chamber rotates around its own axis in an opposite direction. This planet-like movement of the chamber generates large impact energies of milling balls inside the chamber as a result of centrifugal forces created due to the mill parts rotational movement. The ratio of velocity of the basis plate and the chamber is constant and in this series of mills (Pulverisette 6) is 1: –1.82. The "minus" sign indicates that the directions of plate rotation and the revolution of chamber are opposite. Balls to powder weight ratio was like 8:1.

In the experiments pre-mixed samples (100 g) of calcium carbonate and Aerosil powders were fed into the working chamber and a proper amount of isopropanol was added. During the process of mixing the material is trapped between colliding balls and between the balls and the walls of the chamber which causes breaking agglomerates apart and deposition of guest particles on the surface of host particles (Burmeister and Kwade, 2013; Suryanarayana, 2001).

2.2. Flowability indicators

Angle of repose and compressibility index are typical, simple measures of powder flow. They were determined using Hosokawa *Powder Tester PT-S*. Each measurement was replicated five times, and the results were averaged. Measurement of angle of repose is the easiest and the most popular test for powder flowability. Powders with a smaller angle of repose exhibit better flow properties. Compressibility index was calculated from aerated bulk density (ρ_a) and tapped density (ρ_t), after 180 taps, according to formula:

$$CI = \frac{\rho_t - \rho_a}{\rho_t} \cdot 100\% \tag{1}$$

Smaller compressibility index indicates better powder flowability (Jallo et al., 2012).

2.3. Design of Experiment and Response Surface Methodology

Response Surface Methodology (RSM) is a convenient method to model, analyse and optimise engineering processes using statistical approach. RSM is efficient tool to evaluate the influence of process conditions (input parameters) on the response of the model (output parameters) (Aslan, 2008; Bezzera et al., 2008; Dora et al., 2013). The first step in RSM is performing a series of experiments according to Design of Experiments (DOE) approach, next the mathematical model for the response data is fitted and optimised. The last step in RSM is representing the results in 3-D plots (Aslan, 2008).

Design of Experiment tool allows for minimising the number of experiments compared to full factorial design for multiparametric processes, as it is the case for dry coating process. In this study it was decided to take advantage of the Central Composite Rotatable Design (CCRD) to perform all the intended experiments as the most proper design tool for fitting second-order models due to its flexibility, sequential nature and rotatability (Myers et al., 2009). CCRD requires $N=2^k+2k+n$ tests, where k is number of variables (factors), 2^k is the number of standard factorial tests, $2k$ is the number of tests at star (axial) points needed to generate quadratic terms and n is the number of replicate tests at the center of experiment. Star points are the points fitted at a distance α from center to allow rotatability. It is calculated as follows: $\alpha=2^{k/4}$. In CCRD all factors are examined on five coded levels: ±1, 0, $\pm\alpha$ (Aslan, 2008; Bezzera et al., 2008; Polański, 1984).

The first step in this work was running pre-experiments to select the most important process variables and the range of their changes required for flowability improvement.

Planetary ball mixing is a multi-parametric process, therefore it was necessary to select the most important process parameters. The main parameters influencing the process – the process factors – are mixing speed, mixing time, the amount of isopropanol and the amount of Aerosil admixture. Other process parameters including the working volume of mixing chamber, size of the balls and balls to powder weight ratio were chosen on the basis of literature data and were kept constant.

One can assume that the influence of process factors on the response variables (angle of repose and compressibility index) is non-linear and the factors are not completely independent on one another. Mixing speed (x_1, rpm), mixing time (x_2, min), the amount of Aerosil (x_3, mass fraction, %) and the amount of isopropanol (x_4, ml) as analysed factors were considered on five coded levels according to their actual values. If CCRD procedure includes four factors, the star point is $\alpha=2$ and $n=7$ center runs are required to estimate the experimental error (Polański, 1984). The overall number of tests required to accomplish this CCRD procedure with four variables is then equal to 31.

Table 2. Levels of independent variables

variables	symbol		-α	-1	0	+1	+α
	coded	actual					
mixing speed [rpm]	x_1	z_1	200	240	280	320	360
mixing time [min]	x_2	z_2	2	6	10	14	18
amount of Aerosil, mass fraction [%]	x_3	z_3	1	1.5	2	2.5	3
amount of isopropanol [ml]	x_4	z_4	0.5	0.75	1	1.25	1.5

Table 2 shows the coded and natural values of studied factors. The levels were chosen by considering the operating limits of the planetary ball mill and the properties of calcium carbonate and Aerosil. The main problem was to determine the right range of the mixing speed values. With high speed (400 rpm) the process recovery (the weight of sample after mixing / the weight of sample before mixing × 100%) was lower than 75% but with the low speed (below 200 rpm) after mixing for few seconds the balls were trapped under layer of powder and there was powder caking rather than mixing. With the lowest speed (100 rpm) the

amount of energy transferred to powder was insufficient to de-agglomerate both powders even after three hours of mixing. The time of mixing was limited to achieve process recovery minimum 75%. The longer time of mixing, the recovery is lower due to powder sticking to the balls and to the walls of the working chamber. The amount of isopropanol needed was determined experimentally and the amount of Aerosil at center level was taken as in the literature for dry coating to obtain near 100% surface coverage of the host particle with monolayer of guest particles (Ouabbas et al., 2009).

The *Design of Experiment* (CCRD) was created using *Statistica 10* software (StatSoft) and it is shown in Table 3 together with the experimental values of the responses. *Statistica 10* was also used to obtain regression equations of the responses and to make statistical analysis of the results (ANOVA – *ANalysis Of VAriance*). Response Surface Methodology was employed to analyse the experimental data. Results of the experiments for both responses i.e. angle of repose and compressibility index were fitted to polynomial equation – second-order model with interaction as a function of coded process variables:

$$y = (b_0 + \varepsilon) + \sum_{i=1}^{4} b_i x_i + \sum_{i=1}^{4} b_{ii} x_{ii}^2 + \sum_{i=1}^{4} \sum_{j=i+1}^{4} b_{ij} x_i x_j \qquad (2)$$

where ε is residual associated with the experiment, and b_0 is the constant term.

Then the evaluation of the models was made using ANOVA procedure. The next step was to determine optimal conditions using *MATLAB 7.11.0* software.

Table 3. Experimental design matrix and responses

	test number	coded level of variables				angle of repose	compressibility index		test number	coded level of variables				angle of repose	compressibility index
		x_1	x_2	x_3	x_4	y_1, deg	y_2, %			x_1	x_2	x_3	x_4	y_1, deg	y_2, %
factorial design	1	-1	-1	-1	-1	45.6	49.1	axial points	17	-2	0	0	0	44.7	45.6
	2	-1	-1	-1	1	46.5	49.1		18	2	0	0	0	42.4	43.9
	3	-1	-1	1	-1	42.7	46.4		19	0	-2	0	0	44.2	49.3
	4	-1	-1	1	1	43.7	48.0		20	0	2	0	0	41.6	43.3
	5	-1	1	-1	-1	42.8	46.5		21	0	0	-2	0	46.2	47.8
	6	-1	1	-1	1	46.0	48.4		22	0	0	2	0	41.8	44.2
	7	-1	1	1	-1	41.8	44.6		23	0	0	0	-2	40.9	43.6
	8	-1	1	1	1	42.4	45.2		24	0	0	0	2	42.7	46.4
	9	1	-1	-1	-1	43.6	45.8	center points	25	0	0	0	0	41.9	46.3
	10	1	-1	-1	1	44.1	47.8		26	0	0	0	0	42.1	45.6
	11	1	-1	1	-1	41.1	43.6		27	0	0	0	0	41.4	45.6
	12	1	-1	1	1	42.3	44.6		28	0	0	0	0	42.2	46.0
	13	1	1	-1	-1	41.4	44.1		29	0	0	0	0	42.0	46.1
	14	1	1	-1	1	44.0	45.5		30	0	0	0	0	42.3	45.6
	15	1	1	1	-1	41.9	43.4		31	0	0	0	0	42.0	45.0
	16	1	1	1	1	42.4	42.8								

3. RESULTS

3.1. Statistical analysis

For modelling compressibility index changes, a second-order regression model without interactions can be assumed to be valid, but for angle of repose it was necessary to apply a second-order model with interactions. The models for coded levels of variables after removing coefficients, which are not significant for model solution ($p{>}0.05$), were formulated again and the final empirical model for the angle of repose is:

$$y_1 = 42.0 - 0.637x_1 - 0.504x_2 - 1.02x_3 + 0.587x_4 + 0.427x_1^2 + 0.264x_2^2 + 0.539x_3^2 +$$
$$+0.256x_1x_2 + 0.306x_1x_3 + 0.286x_2x_3 + 0.206x_2x_4 - 0.244x_3x_4$$

(3)

The same model formulated for actual levels of variables is as follows:

$$y_1 = 94.5 - 0.212z_1 - 1.38z_2 - 14.3z_3 + 4.19z_4 + 2.67 \cdot 10^{-4}z_1^2 + 0.0165z_2^2 + 2.16z_3^2 +$$
$$+0.00160z_1z_2 + 0.0153z_1z_3 + 0.134z_2z_3 + 0.206z_2z_4 - 1.95z_3z_4$$

(4)

The models for compressibility index formulated for coded and actual values of variables respectively, are:

$$y_2 = 45.6 - 0.962x_1 - 1.08x_2 - 1.04x_3 + 0.562x_4 + 0.223x_2^2$$

(5)

and

$$y_2 = 58.3 - 0.0241z_1 - 0.549z_2 - 2.07z_3 + 2.25z_4 + 0.0139z_2^2$$

(6)

The ANOVA shows that all factors have a significant influence on the responses in experimental values range and the surface models are significant due to the lack of fit tests ($p{>}0.05$).

The experimental and predicted values of angle of repose and compressibility index calculated from Eqs. 3 and 5 are presented in Fig. 3. Good agreement between predicted and experimental values of both responses can be seen (the value of coefficient of correlation R^2 for angle of repose is 0.94975 and for compressibility index 0.87829).
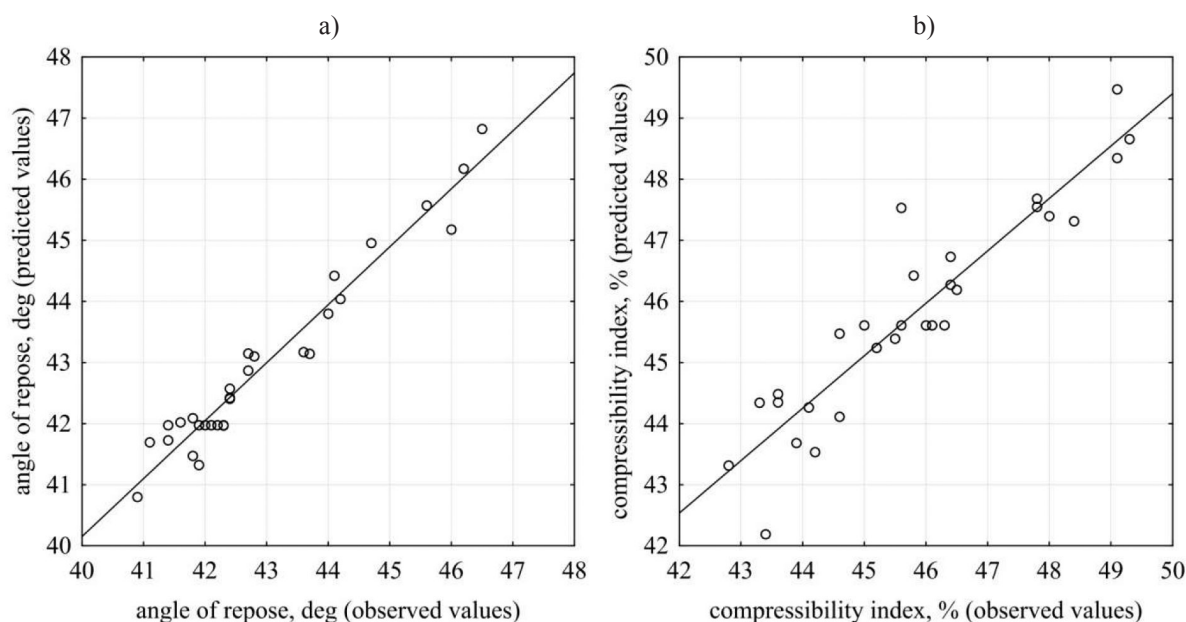

Fig. 3. Relation between experimental and predicted values of: a) angle of repose according to Eq. (3); b) compressibility index according to Eq. (5)

3.2. Effect of variables on angle of repose and compressibility index and process optimisation

The model given by Eq. 4 can be presented on 3-D response surface plots. For example, in Fig. 4a, the effect of actual variables (speed and time of mixing) on angle of repose at the center level of other factors is shown. As can be seen, the surface achieves evident minimum in experimental range of variables. Similar results regarding angle of repose changes (not shown) one can obtain for the effect of time and speed of mixing versus the amount of Aerosil added. A different situation is shown in Fig. 4b, where the effect of the amount of isopropanol and mixing time on angle of repose is presented. In this case the smallest addition of isopropanol is needed to obtain the best flowability of calcium carbonate (the smallest angle of repose).

Table 4. Optimal values of process variables

Optimum conditions	Values needed to obtain minimum value of angle of repose	Values needed to obtain minimum value of compressibility index	Values needed to obtain minimum values of both responses simultaneously
mixing speed [rpm]	290	360	320
mixing time [min]	16	18	16
amount of Areosil, mass fraction [%]	2	3	2
amount of isopropanol [ml]	0.5	0.5	4
initial value of angle of repose [deg]	55	55	55
angle of repose [deg]	40.0	44.9	40.6
initial value of compressibility index [%]	47.6	47.6	47.6
compressibility index [%]	43.0	39.1	41.5

*) It is worth pointing out that flowability of resulting mixtures – as observed visually – was much better than that following from decreasing the angle of repose and compressibility values shown in the Table. This confirms the opinion of Sonoda et al. (2008) that flowability indices commonly used in research on powders do not necessarily reflect exactly the obtained results.

To obtain optimal conditions for dry coating in planetary ball mill (to achieve the lowest angle of repose), process optimisation was accomplished with MATLAB 7.11.0 The Optimization Tool *fmincon-Constrained nonlinear minimization* solver with *Active set* algorithm for coded levels of variables were used. The resulted optimal values of actual process variables in experimental ranges needed to achieve the lowest angle of repose are given in the Table 4*).
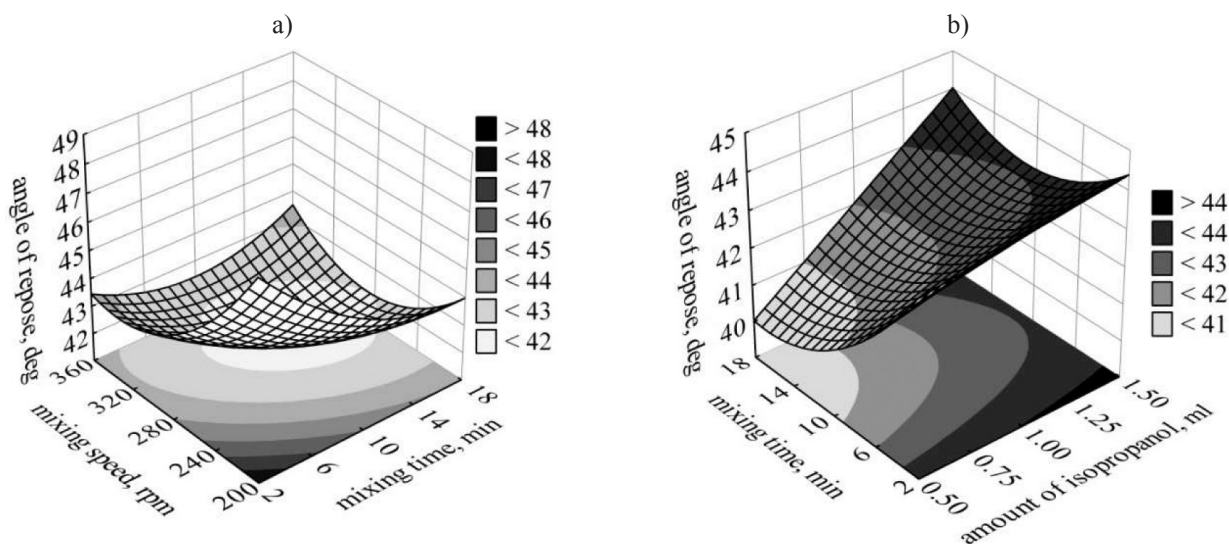

Fig. 4. Effect of process variables on angle of repose (factors not shown on the plots are at the center level)

a)   b)
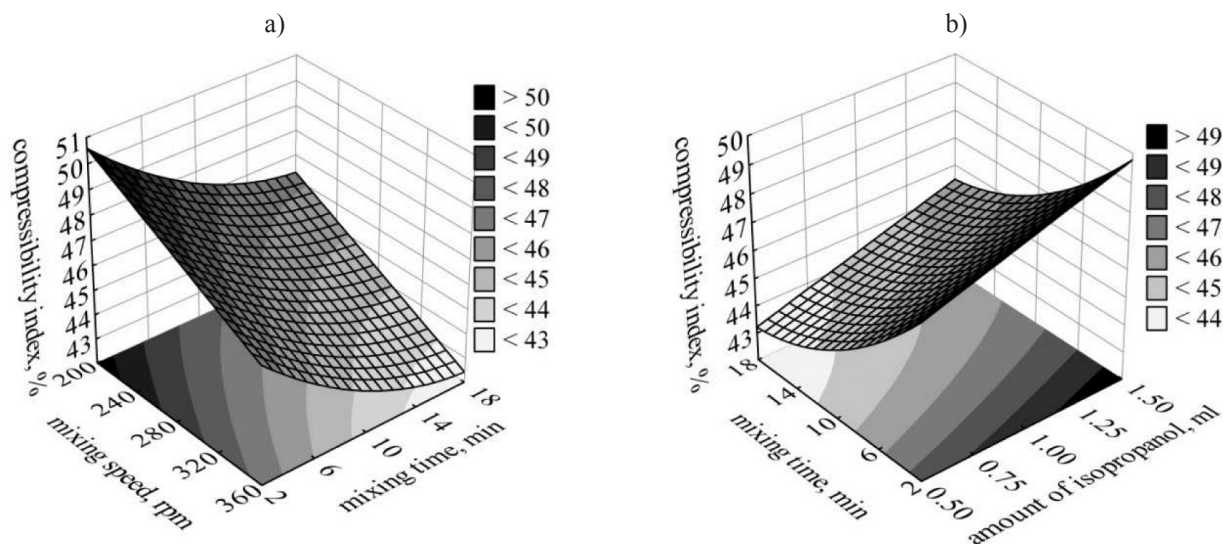

Fig. 5. Effect of process variables on compressibility index (factors not shown
on the plots are at the center level)

The second of the two analysed measures of flowability is powder compressibility index. The 3-D response surface plots show that in order to obtain the smallest value of compressibility index, maximum values of speed of mixing, mixing time and the amount of Aerosil, and minimum value of the amount of isopropanol are required (Fig. 5; in these Figure factors not shown on the plots are all at the center level). These results are different from those obtained earlier for angle of repose except the amount of isopropanol, which is at the minimum level and the optimal time of mixing, which is similar for both responses (Table 4). The major differences concern the case of optimal values of the mixing speed and the amount of Aerosil admixture. It may result from the fact that powder compressibility index is influenced by more regular particle size after longer time of mixing and small particles of silica. Moreover, compressibility index may be a flowability measure not as sensitive as angle of repose for some groups of powders (Santomaso et al., 2003).

Process parameter values needed to obtain optimum values of both responses are not rigid and they may be changed in some range. To obtain possibly the smallest value of angle of repose with the possible smallest value of compressibility index the weighted sum method for multi-objective optimisation with the same weights for both responses equal 0.5 was used. In that case the input factors should be as presented in Table 4.

3.3. Morphology and chemical analysis of calcium carbonate samples

To examine changes in morphology of the calcium carbonate particle surface taking place during mixing, SEM analysis of some samples taken after mixing was performed. Figs. 6a-b show SEM images of original Aerosil and calcium carbonate particles. Figs. 6c-d show that the average size of calcium carbonate particles is similar before and after treatment with Aerosil but the surface of the particles had obviously changed in the course of mixing. The images of particle surface shown in Figs. 6c-d (calcium carbonate with and without Aerosil respectively) are similar to some extent, and a question arises what the nature of the small particles seen on the surface of calcium carbonate actually is. To explain this SEM-EDS analysis was accomplished with the same Scanning Electron Microscope as used for particle morphology investigation. The results of the analysis are given in Fig. 7. They confirm the presence of guest Aerosil (Si) particles deposited on host calcium carbonate surface and this provide evidence that dry coating process has occurred indeed as result of high energy mixing in planetary ball mill.

a)   b)

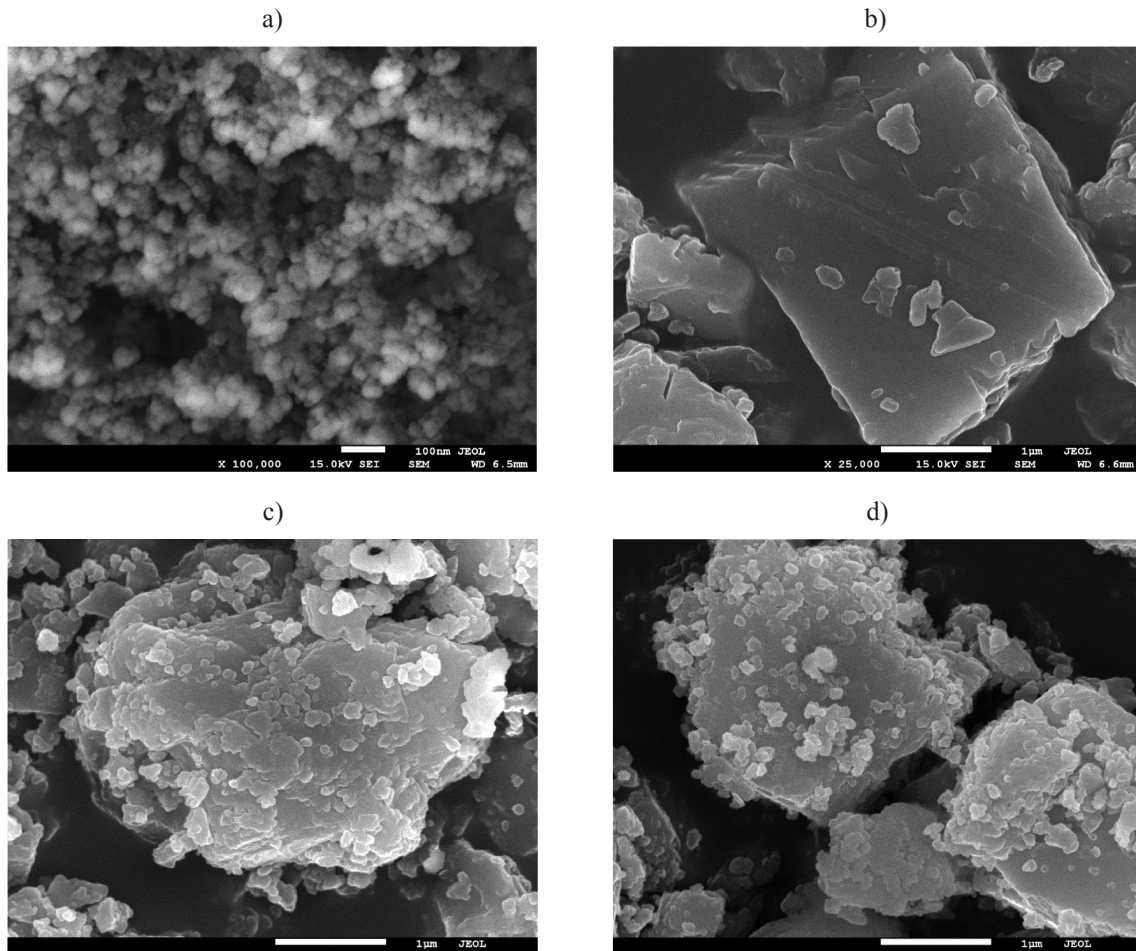c)   d)

Fig. 6. SEM micrographs of: a) Aerosil; b) untreated calcium carbonate; c) calcium carbonate treated without Aerosil; d) calcium carbonate treated with 2% of Aerosil; both samples after mixing for 10 min with 300 rpm
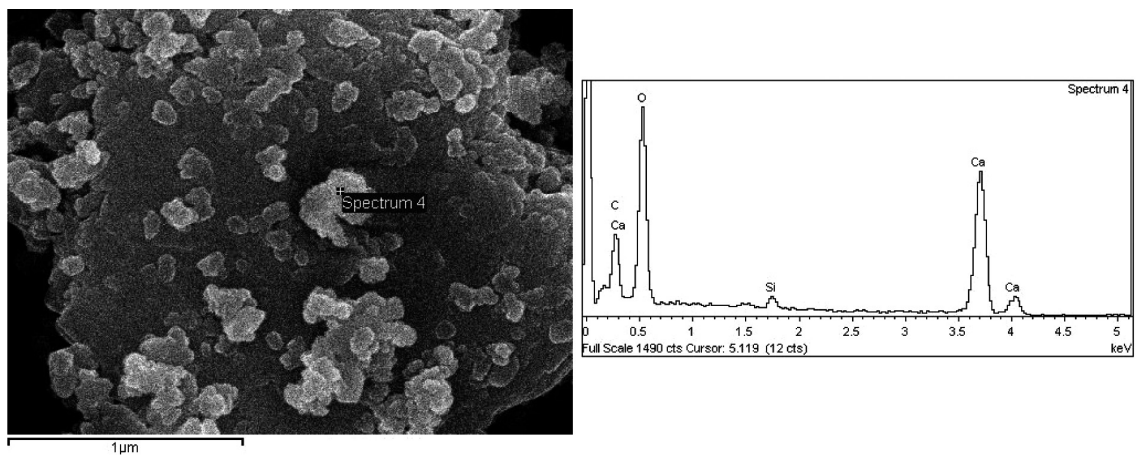

Fig. 7. SEM-EDS analysis of calcium carbonate particle after mixing with Aerosil for 10 min at 300 rpm

4. CONCLUSIONS

- The dry coating method performed with a planetary ball mill proved to be an effective way for improving flow of cohesive powders. Using an admixture of colloidal silica Aerosil in the amount of only 2 wt%, a considerable decrease of both angle of repose (from 55 to about 40 deg) and compressibility index (from about 48 to 39%) of calcium carbonate powder was obtained at moderate mixing time and rate conditions.

- To find the optimal mixing parameters, Response Surface Methodology coupled with Design of Experiment approach was applied. RSM based on Central Composite Rotatable Design was employed to study the influence of four process variables (mixing time and rate, amount of Aerosil and isopropanol) on two responses (angle of repose and compressibility index) used as powder flow indicators. The response model equations showed that all the process variables are significant process factors and should be used for both interpretation and prediction of the mixing process output.

- The values of angle of repose and compressibility index predicted from the model equations are in good agreement with experimental data. Optimisation procedure of the dry coating process was based on the analysis of model equations with MATLAB software. The resulting optimal process parameters providing the best flowability of calcium carbonate powder are: mixing speed 320 rpm, mixing time 16 min, amount of Aerosil admixture 2 wt% and amount of isopropanol addition 0.5 ml.

- Planetary ball mill technique and Response Surface Methodology with Central Composite Rotatable Design procedure can be successfully applied to conduct dry coating process of cohesive powders and to find its optimal process conditions.

SYMBOLS

b_0 constant term
b_i, b_{ii}, b_{ij} regression coefficients
CI compressibility index, %
x_1, x_2, x_3, x_4 coded variables: mixing speed, mixing time, amount of Aerosil and amount of isopropanol, respectively
y_1, y_2 responses: angle of repose, deg, and compressibility index, %
z_1, z_2, z_3, z_4 actual variables: mixing speed, rpm; mixing time, min; amount of Aerosil, %, and amount of isopropanol, ml

Greek symbols
α axial, coded distance from the center of experiment
ε residual associated with the experiment
ρ_a aerated bulk density, kg/m^3
ρ_t tapped density, kg/m^3

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