

## **MICRO PULSE MIXING- AN EFFICIENT MIXING METHOD OF SMALL QUANTITIES OF POWDERS**

**A. M. AKSENCZUK, A. CALKA  
and D. WEXLER**

Faculty of Engineering  
University of Wollongong  
Northfields Avenue  
Wollongong, NSW 2522  
Australia

e-mail: [ama757@uowmail.edu.au](mailto:ama757@uowmail.edu.au)

[andrzej.aksenczuk@gmail.com](mailto:andrzej.aksenczuk@gmail.com)

### **Abstract**

This paper describes a novel mixing method together with mixing device. The Micro Pulse Mixer ( $\mu$ PM) has been designed, constructed and tested using a new mixing concept based on damped harmonic oscillator motion. Advantages of the technique include simplicity of the device, high rates of mixing and high uniformity of product. Furthermore, the device design, with discrete powder containers, allows rapid loading and unloading of samples ( $\sim$  30-60s) enabling powder sampling and/or complex blending operations. The mixer does not require frequent cleaning. It is designed to mix relatively small volumes of powders (up to several grams) for use in either laboratory work or for small commercial samples of high value.

In this study we compared the  $\mu$ PM method with rotating tumbling mixing. Mixing products were evaluated by analysis of scanning electron microscope (SEM) images while X-ray diffraction (XRD) was used to monitor phase evolution

---

Keywords and phrases: powder mixing, pulse mixer, mixing mechanisms, deagglomeration, effective mixing coefficient.

Received March 3, 2017

during mixing. Powder particle distributions were obtained based on image analysis techniques with mixing efficiency parameters determined based on the calculation of the percentage compounds content within sampled powder is proposed.

We believe that the more efficient approach of impulse mixing could be beneficial for laboratory purposes and as a simple and cost-effective powder mixing and blending method.

## 1. Introduction

Mixing and blending of granular materials, can greatly influence material preparation in the pharmaceutical, metallurgy, ceramics or many other industries [1]. Mixing can be defined as a process of combining particles of solids, liquids or solutions, to obtain a homogeneous product.

Fine and ultrafine particles are particularly difficult to mix due to both feeding and handling problems during processing [2]. Fine particles with large surface areas often are accompanied by high chemical reactivity. Furthermore, fine powders have a tendency to form particle agglomerations. Fine powder dissolution within a liquid is one of the methods which helps to obtain uniform particle distributions [3-5]. However, such solutions are not always desirable because liquids can react with mixing powders or the container material. Additionally, the liquid needs to be removed after the mixing, which is an additional complication, therefore, often dry powder mixing is preferred. Despite the issues related to mixing, fine powders have potential benefits for commercial application in industrial and military products [5]. In the pharmaceutical industry, particle homogeneity may influence both the mechanical strength and drug release rate of tablets [6-8]. In this case, a preliminary study performed on small powder quantities represents a fast and cost efficient method in new products examination. For these reasons, research on mixing technologies of a small volume of fine particles is of great importance.

For powders to be mixed, the individual particles must be distributed most uniformly over the substrate's volume. Mixing of the solids can be achieved by the following mechanisms: diffusion, convection and/or shear [9]. Mixing by diffusion can be defined as random distribution of the particles over freshly developed interface. An example of diffusion mixing device can be double v-cone tumbler. Mixing by convection can be characterized by rapid movement or flow of the group of particles due to force of rotating agitators. Bending is done by removing the force of attraction between particles, for example, by using a high speed chopper blades / mixer with tulip and knife blades. Mixing by share is achieved by removing the force of attraction between particles, for example via split shear waves or mechanical shearing using sliding steel balls (during ball milling).

Tumbling ball mixing (or ball milling) is common method for dry powder mixing. The tumbler is a rotating container partially filled with a powder. Different powder flow regimes inside the vessel include: avalanching, slumping, rolling, cascading, raining and centrifuging. The flow is dependent on the rotational speed and container shape [10]. Efficiency in tumbler mixers significantly depends on the container fill level. When filling level increases, efficiency decreases. This phenomenon is related to powder kinetics inside the container [11].

High energy is applied to the substrate by moving the container or mixer tools [12] to break down agglomerates and mix fine particles. Additional balls inside the rotating container apply extra energy to the process. One of the tumbling methods using this phenomenon is the ball-mill/mix technique. Ball milling (BM) was developed by International Nickel Company in late 1960 [14]. In the BM technique, balls, together with the substrates, are arranged within the rotating chamber. The energy from balls collisions, impact the materials (which most often have the form of a powder), by mixing, crushing and grinding them. Powder trapped between colliding balls can be subjected to localized temperature

increase. Depending on the process parameters, we can obtain various energies of the milling. With low-energy BM, it is possible to obtain different end-products, such as homogeneous mixing and deagglomeration [16] of the powders, or to change particle sizes [15]. Mechanical ball-milling is recognized as a simple and cost-effective method to produce fine and homogeneous powders [13]. Disadvantages of this method are firstly the contact with the vessel and balls that can result in contamination of the final product; and secondly, during the mixing, some amount of the powder remains on the container walls and is hard to remove after the process is finished. Chamber cleaning and preparation for mixing is time-consuming.

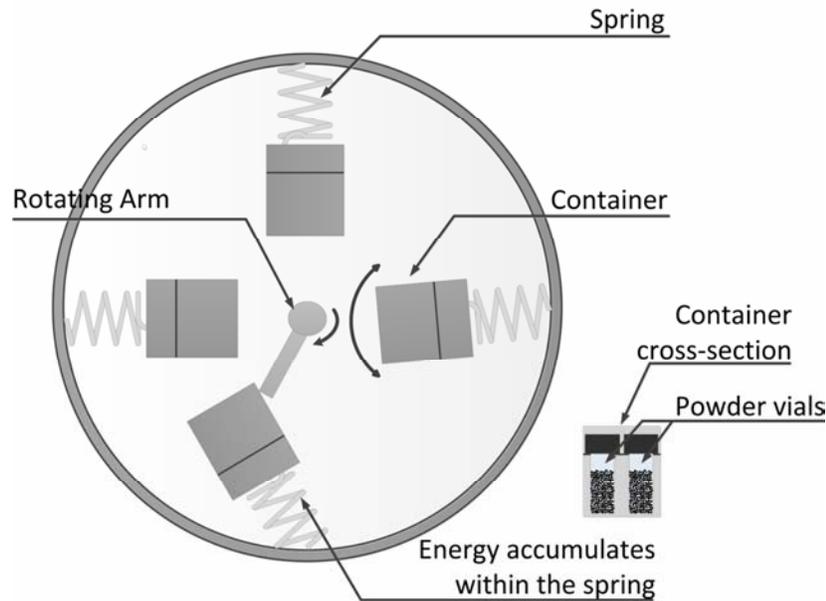
Taking into account above the mentioned disadvantages of conventional mixing a novel alternative method has been developed. The micro pulse mixer ( $\mu$ PM) is a device that uses damped harmonic oscillator motion to produce fine and coarse powder mixing on a laboratory scale. Advantages of the technique include simplicity of the device, high rates of mixing and high uniformity of product. Furthermore, the device design, with discrete powder containers, allows rapid loading and unloading of samples (which takes approx. 30-60s) enabling powder sampling and/or complex blending operations. The mixer does not require frequent cleaning. It is designed to mix relatively small volumes of powders (up to several grams) for use in either laboratory work or for small commercial samples of high value.

The mixing problem is complex, and no precise mathematical model has been found yet. Because of this complexity, most models are based on experimental work. The performance of the mixer can be predicted by using different powders with the general theory of powder mixing.

## **2. Device Description**

The micro pulse mixer ( $\mu$ PM) was designed for fine and coarse laboratory scale dry powder mixing. It comprises two major components:

a bottom body, where the mixer driver is located, and an exchangeable upper body, where the powder is mixed. The powder container (Figure 1) consists of two part called powder vials, and outer sample holders. The powders to be mixed are placed inside the powder vials.

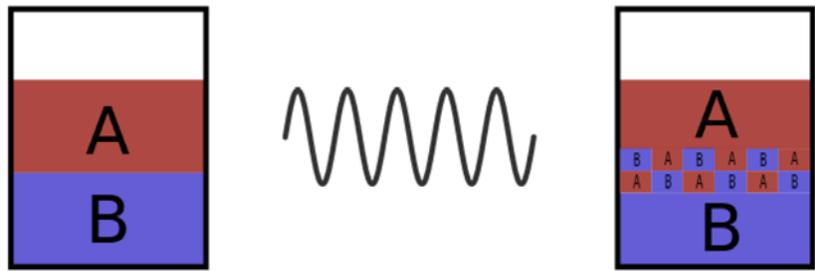


**Figure 1.** Micro pulse mixer ( $\mu$ PM) device overall scheme. Mixer container consists of powder vials and outer sample holder.

After being filled with powder, the powder vials are placed into the tightly fitted outer sample holder. Use of a system with powder vials and outer sample holders significantly reduces the time necessary to exchange samples between mixing sessions, to within 30-60 seconds. This approach also facilitates the transport of powders. The outer sample holder is mounted on a spring connected to the housing of the upper body of the device (Figure 1). In a recent version of the device, 2ml bottles were used as the powder vials. Containers can be made of metal, plastic or any other solid material. Up to 16 samples can be mixed at once. In a scaled up version bigger powder vials, up to 20ml can be used.

During operation a rotating arm transfers energy to the spring (flat, tension, compression spring or just a piece of spring still) and displaces the spring out of its balance position. Depending on the rotational speed and the length of the arm, different amounts of energy can be transferred to freely moving powder inside the powder vials. Importantly, the energy is transferred to the powder via a short duration pulse/shock manner. To improve mixing efficiency additional balls can be placed inside powder vials.

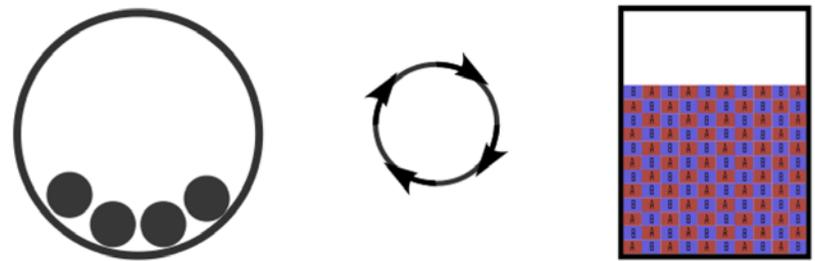
The mixing phenomenon within the  $\mu$ PM originates from damped oscillation movement of sample holder, which differ significantly from constant vibrational movement. Upon vibration of the powder, the smaller particles will gradually to regions between the bigger ones and thus lead to the separation of the differently sized particles [2, 9]. In the presented experiments, different sizes and weights of the particles were used; nonetheless, particles separation was avoided. Figure 2 shows schematics of three different state of powders mixing via; constant amplitude vibration, micro pulse mixing, and ball milling.



(a) Vibrations



(b) Micro Pulse Mixing



(c) Ball Milling

**Figure 2.** Schemes present different states of powders mixing by: (a) constant amplitude vibrations; (b) micro pulse mixing damped harmonic oscillation motion (variable amplitude mode); and (c) ball milling.

### 3. Mixing Mechanism Study

Mixing of two or more powders is an energy and time consuming process that, in case of perfect mixing, should produce equal and uniform distributions of particles. This process requires application of external energy, which can be provided by the motion of a container, by the motion of agitator/blade passing through the bulk, by gravity or by a combination of the above factors. In fact, the case of perfect mixing is almost impossible to achieve and most mixers tend to randomise particle distributions. Therefore, mixing of solids can be achieved within one of the three main mixing modes [19]:

(1) Mixing by diffusion – which can be characterized as mixing by random motion of solid particles, often movement of individual particles.

(2) Mixing by convection – where particles groups are rapidly moving from position A to position B most often due to the action of a rotating agitator.

(3) Mixing by shear – where mixing is achieved due to the removal of the force of attraction between particles. Shear blending generally involves the development of slip planes and strains within a bed of material, which can be followed by diffusion and convection mechanisms.

The micro pulse mixer can operate in both diffusion and/or convection mixing modes, depending on the additional mixing medium (e.g., plastic balls).

When the rotating arm (Figure 1) is passing the sample holder, energy is accumulated within the spring by displacing it out of balance position to a distance  $A$ . When the arm passes the holder, the stored energy is released. Some portion of this energy is transferred into powders in a form of a pulse/shock and causes powders mixing. According to the  $\mu$ PM construction, motion of the sample container can be approximated by the damped harmonic oscillator equation:

$$m \frac{d^2x(t)}{dt^2} + b \frac{dx(t)}{dt} + kx(t) = 0 \quad \text{or} \quad \frac{d^2x(t)}{dt^2} + \frac{b}{m} \frac{dx(t)}{dt} + \frac{k}{m} x(t) = 0, \quad (1) \quad [17]$$

where

$k$  – is the spring constant;

$b$  – is a damping coefficient (resistive force);

$m$  – is the mass.

The solution of the above equation can be written as

$$x(t) = Ae^{-\frac{bt}{2m}} \cos(\omega't + \varphi), \quad (2) [18]$$

where

$Ae^{-\frac{bt}{2m}}$  – represents the amplitude of damped oscillations;

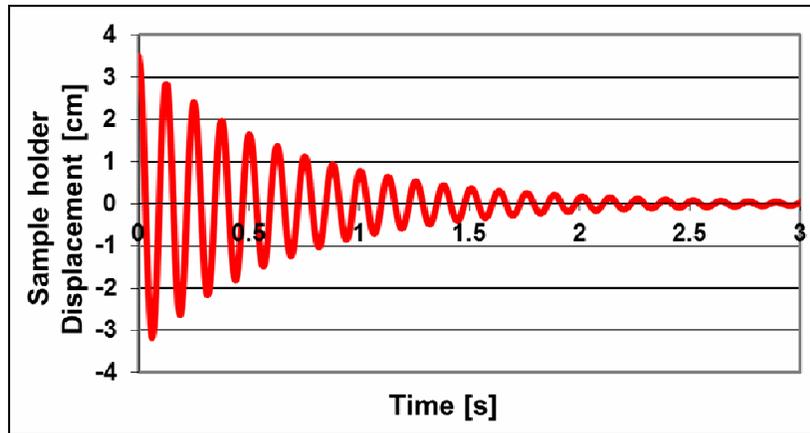
$\omega' = \sqrt{\frac{k}{m} - \frac{b^2}{4m^2}}$  – is circular frequency of damped oscillations.

If we now replace the maximum displacement ( $x$ ) in the equation for the total energy in a classic oscillator ( $E = \frac{1}{2} kx^2$ ) with the amplitude of the damped oscillations, the oscillation energy can be approximated as:

$$E(t) = \frac{1}{2} k(Ae^{-\frac{bt}{2m}})^2. \quad (3)$$

It was noticed that powder container movement is in three-dimensional motion. Most of the harmonic oscillation is visible within the X-Y plane. By placing easily deformable foam below the outer sample holder during the motion  $\sim 1\text{mm}$  dents in the foam were noticed. This suggest that an additional, low intensity damped harmonic motion within Z axis occurs. This motion comes from the use of extension springs and is considered as an additional factor increasing the mixing properties. To avoid this, flat spring should be used.

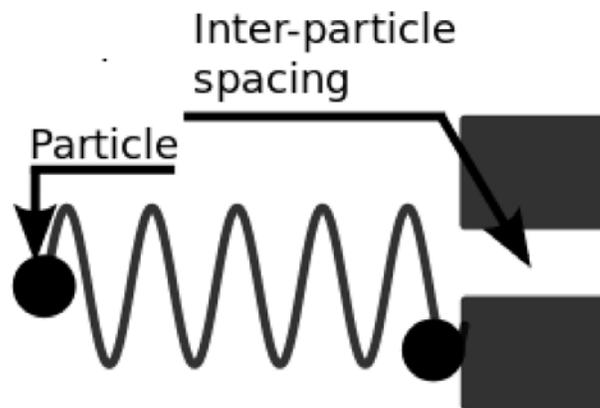
In the micro pulse mixer the energy accumulated within the spring, and is released when the arm passes the sample holder. The powder vials are rigidly fixed to the outer sample holder. As the connection between the outer sample holder and the spring is also rigid, when spring starts moving and releases the stored energy, some portion of that energy is applied to unconstrained powder particles inside the powder vials, which causes their mixing. Figure 3 shows the relationship of the sample holder position to time in the presented  $\mu$ PM design.



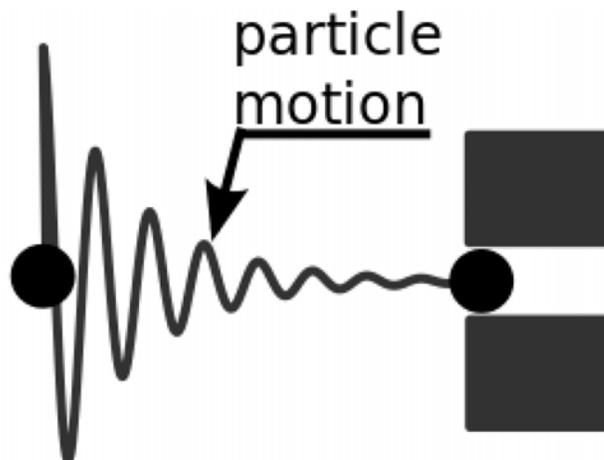
**Figure 3.** Damped harmonic oscillator motion approximation of the  $\mu$ PM sample holder calculated for the used device design. The starting amplitude  $A = 3.5[\text{cm}]$ ; circular frequency of damped oscillation  $\omega' \sim 50$  [rad/s]; and the damping coefficient  $b \sim 1.5[1/\text{s}]$ .

Several springs with various elasticity coefficients have been used. Experiments confirmed that springs with relatively low elasticity coefficient result in improved mixing. During observation, it was also noticed that the best mixing effect is achieved within a single shock of the holder (motion tending to critical damping). It is believed that a low elasticity coefficient in the springs transfers more energy directly to mixed powder.

The reason why damped harmonic oscillator motion is believed to increase the movement of powder particles through inter-particle spacings is illustrated in Figures 4(a) and 4(b).



(a) Constant amplitude motion



(b) Variable amplitude motion

**Figure 4.** Movement of powder particles through inter-particle spacings for: (a) constant vibrations; (b) damped harmonic oscillations.

#### 4. Experimental Trails

A set of the samples was prepared using an in-house micro pulse mixer with and without additional balls inside the mixing containers.

X-ray diffraction pattern was performed on the samples, to confirm that no chemical reaction had a place during the mixing. X-ray diffraction analysis of starting and milled powders was performed using GBC Mini-Material Analyser (MMA) diffractometer with Cu K $\alpha$  radiation and graphite monochromator. Diffractometer output was analyzed using Traces Version 5.1.0 software. Phase identification was carried out using the International Centre for Diffraction Data (JCPDS-ICDD 2000) Powder Diffraction Files (PDF).

Morphological properties of the mixed powders were measured using scanning electron microscopy (SEM) backscattered imaging using a JEOL JSM-6490LV instrument equipped with JEOL energy dispersive analysis (EDS) system.

##### 4.1. Starting powder

All samples were prepared using Sigma-Aldrich powders of  $\geq 99\%$  purity. In each experiment, silicon (45 $\mu\text{m}$ ) powder was mixed with one of five elements (see Table 1). Silicon powder was placed in  $\mu\text{PM}$  powder vials together with one of the elements from Table 1, in weight ratio 1:1. The total mass of two powders was 1g. Powder volumes, which will vary for each elements mixture pair, allow us to perform a preliminary study of the influence of container fill level on mixing.

**Table 1.** Powders used for mixing tests

Powder	Size [ $\mu\text{m}$ ]	Bulk density [ $\text{g}/\text{cm}^3$ ]
Si	45	2.33
Al	75	2.4
C	20	2.26
Fe	45	7.86
Mo	5	10.3
Ta	45	16.65

#### 4.2. Experimental protocol

In the first step, samples were placed in the containers and mixed respectively for 20, 40, and 60 minutes. In the second step, a small portion of powder was removed from each sample, from the top of the container and cold pressed under 0.2MPa pressure to form 4.3mm diameter pellets.

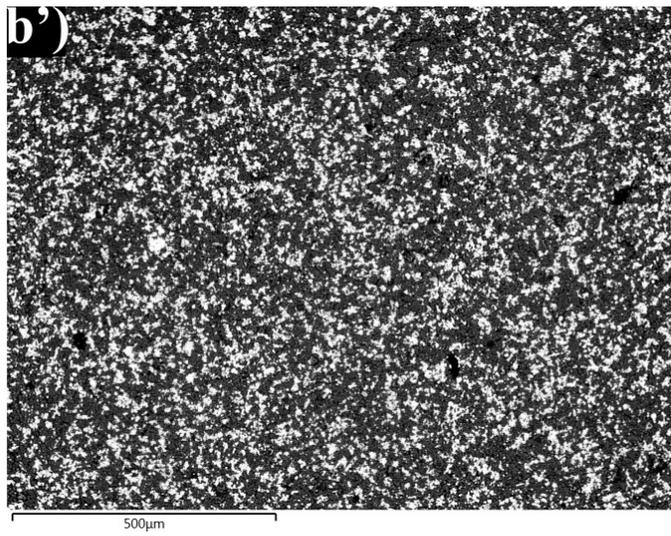
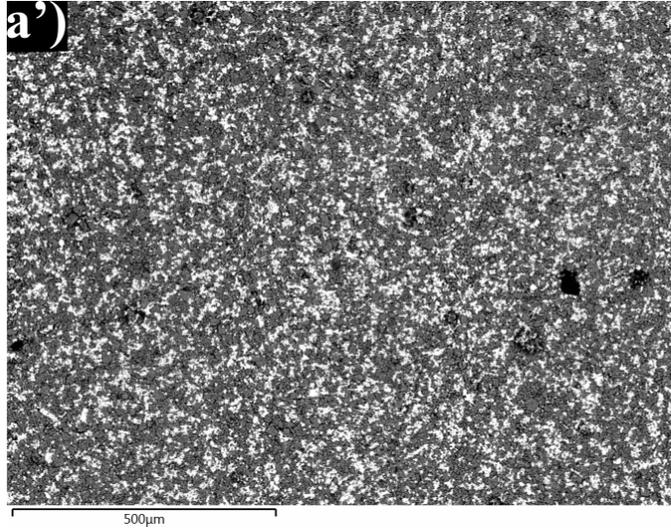
In the next step, pellets were analyzed by SEM backscattered electron imaging (BEI) to acquire images of different phases differentiated by atomic number contrast. Energy dispersive spectroscopy (EDS) was used to confirm the composition of each phase. At least three random places were chosen for each pellet. For the last stage, imageJ software was used to calculate the percentage content of elements from the grey-level calibrated backscattered electron images. The effective mixing coefficients were then calculated.

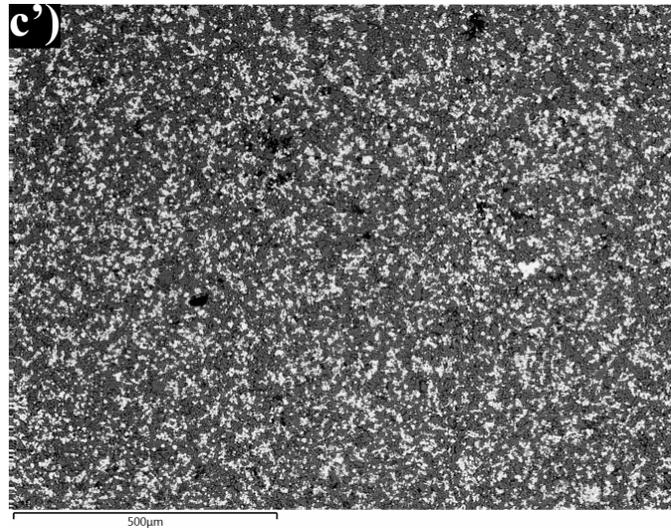
The difference in the brightness of BEI images, which depends on powder elements atomic weight, allows for the particle distribution studies. If the components have similar atomic weight (as in case on Si and Al) EDS map analysis was performed to the check particles distribution.

### 5. Powder Sample Characterization after Mixing

Figure 5 shows Si and Fe powder mixtures prepared using micro pulse mixing for respectively 20 min (Figure 5(a')), 40 min (Figure 5(b')), and 60 min (Figure 5(c')) mixing. Black regions in the images are porosity associated with incomplete densification during cold pressing. The samples were observed to tend to random mixing in both cases after only 20 minutes (Figure 5(a')). The distribution of the silicon and iron particles change within pellet when the mixing time increases, and the powder becomes more and more deagglomerated. Powder deagglomeration can be observed during  $\mu$ PM, although no additional mixing medium (such as balls) was used (Figure 5(c')).

Impact from a mixing agitator, such as a blade or balls, to achieve powder deagglomeration can also decrease particle sizes and result in contamination from the mixing medium. Furthermore, often for small amount of starting powder (e.g., 1g), only a fraction can be recovered due to adherence of the powder to the milling chamber. In comparison, mixing within  $\mu$ PM requires only the powder vials to be replaced. Virtually 100% of the powder was recovered after mixing and the technique was not observed to change the powder particle sizes, the latter representing an additional advantage of pulse mixing technique.

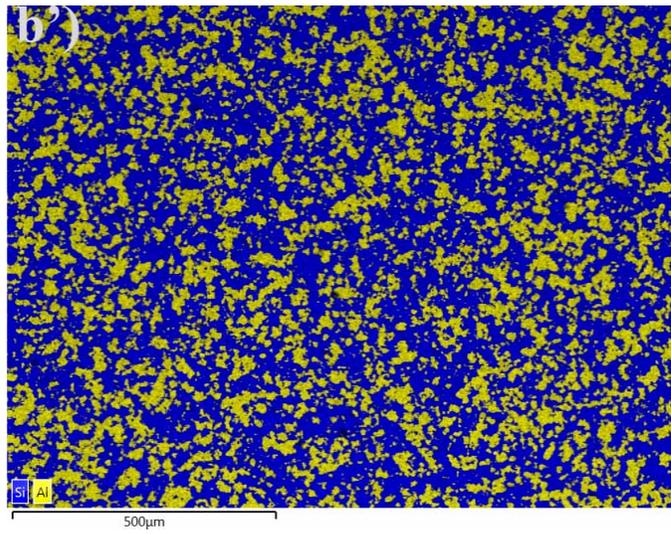
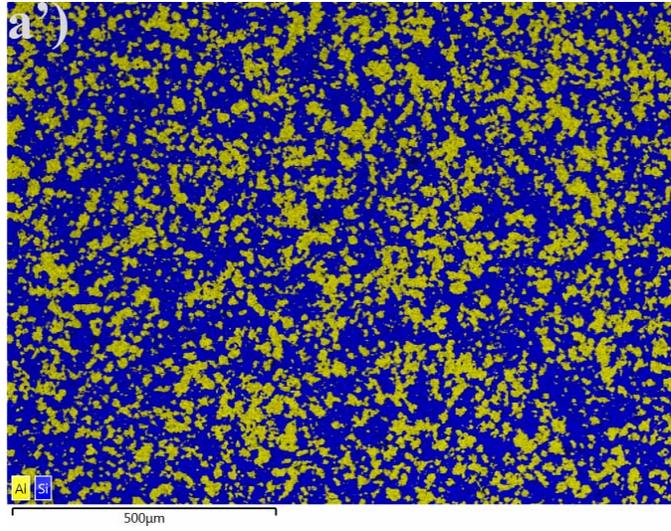


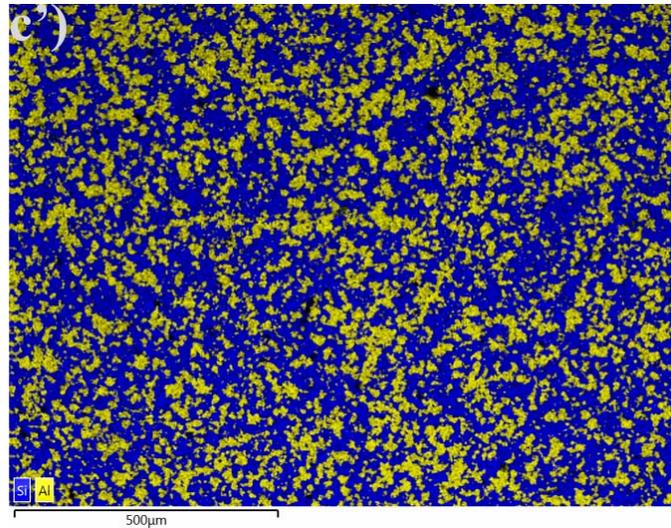


**Figure 5.** Backscattered electron images of Si (dark-gray colour) and Fe (bright colour) powders after mixing: for (a') 20 min; (b') 40 min using; and (c') 60 min using micro pulse mixer.

As the atomic weight of silicon and aluminium are similar, phase distinction based on backscattered electron images was hard to perform. However, the energy-dispersive spectroscopy (EDS) with map analysis tool allowed clear distinguishing of phases and estimation of the powder distribution (Figure 6). BEI is simple and fast method for phase analysis. Analysis based on SEM-EDS mapping could be performed for all samples, however, the BEI analysis, with grey level matching composition is more spatially accurate than EDS mapping, the later method involving larger interaction volumes below the analysed surface.

Similarly to the Si and Fe mixture, samples of Si and Al were randomly mixed in both cases after only 20 minutes (Figure 6(a')). The distribution of the silicon and aluminium particles within the pellet becomes more homogeneous when the mixing time increases.

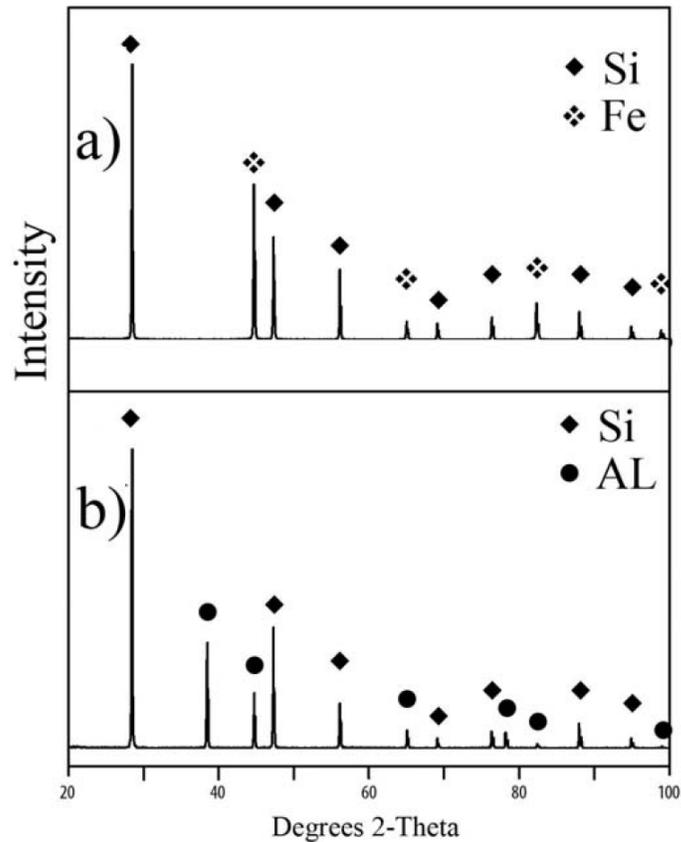




**Figure 6.** Energy-dispersive spectroscopy (EDS) maps of Si (blue) and Al (yellow) powders after mixing: for (a') 20 min using; (b') 40 min; and (c') 60 min using micro pulse mixer.

Figure 7(a)-(b) shows X-ray diffraction results obtained from silicon-Iron and silicon-aluminium powders after mixing for 60 minutes using the micro pulse mixing equipment. A set of X-ray peaks in Figure 7(a) can be indexed to the powder diffraction files of phase Si published in JCPDS-PDF 05-0665 and phase Fe published in JCPDS-PDF 06-0696. After 60 min of Si and Fe  $\mu$ PM, only patterns that belong to starting powders can be noticed.

Similarly, no compositional changes were noticed in the second powder X-ray peaks shown in Figure 7(b), which can be indexed to the powder diffraction files of phase Si published in JCPDS-PDF 05-0665 and phase Al published in JCPDS-PDF 01-1176. The diffraction pattern shows that, within the detectability limits of the XRD method, no chemical reactions appear during powder mixing.



**Figure 7.** X-ray diffraction patterns of after 60 min mixing of: (a) Si and Fe powders; (b) Si and Al powders in  $\mu$ PM.

For each binary powder mixture, the imageJ *Analyze Particles* tool was used to estimate the percentage area occupied by the given powder within the pellet. The percentage content was calculated as the arithmetic average of at least three randomly chosen pellet regions (Table 2). The theoretical volume ratio of those binary powders compounds: first  $V_1$  and the second  $V_2$ , can be calculated according to equation:

$$\frac{V_1}{V_2} = \frac{\frac{m_1}{\rho_1}}{\frac{m_2}{\rho_2}} = \frac{m_1 \rho_2}{m_2 \rho_1}, \quad (4)$$

where

$\rho$  – (rho) is a density;

$m$  – is the mass; and

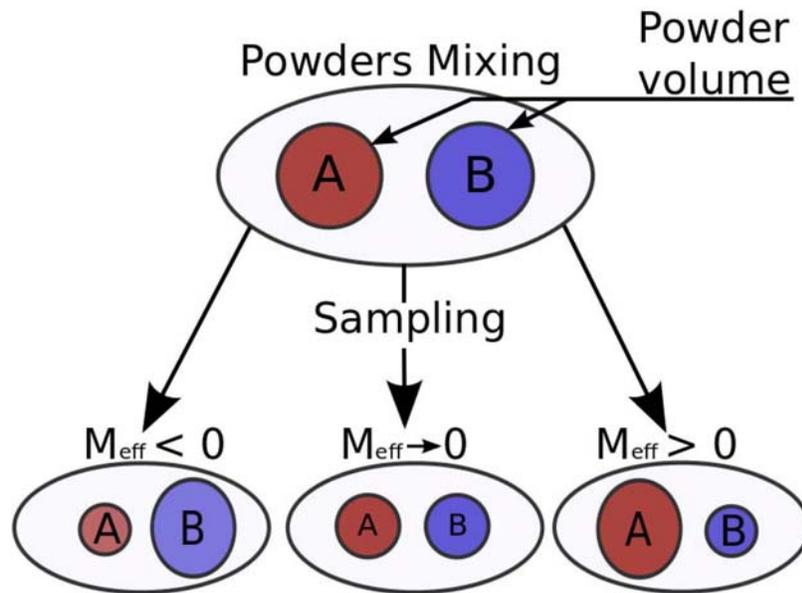
$V$  – is the volume.

Now, if we firstly mix those powders, secondly split the product into infinite numbers of small portions and the volume ratio of those small portions is equal to the theoretical volume ratio ( $V'_1/V'_2 = V_1/V_2$ ) then we can presume that those powders are uniformly mixed.

As the densities and the weights of the used powders were known, and the percentage area ( $S\%$ ) occupied by the given powder within the sampled pellet was calculated, we can calculate the effective mixing coefficient ( $M_{eff}$ ), which can be considered as an approximation of the actual state of mixing (Table 2), by the following formula:

$$M_{eff} \approx \frac{S_{1\%}}{S_{2\%}} = \frac{V_1}{V_2} = \frac{S_{1\%}}{S_{2\%}} = \frac{m_1 \rho_2}{m_2 \rho_1}, \quad (5)$$

where  $S_{1\%}$  and  $S_{2\%}$  are, respectively, calculated percentage areas of the first and the second powder.



**Figure 8.** Effective mixing coefficient graphical representation.

Let us consider two powders of equal weight. Let the first powder be silicon and the second tantalum. The density  $\rho_{\text{Si}} \sim 2.33\text{g/cm}^3$  and  $\rho_{\text{Ta}} \sim 16.65\text{g/cm}^3$  (Figure 8, powders **A** and **B**, respectively). The theoretical volume ratio  $V_1/V_2 \sim 7.15$ . If the calculated percentage area ratio for these two powders  $S_{1\%}/S_{2\%} = 9.12$  (which indicates that there is 9.12 time more Si than Ta within sampled powder) the effective mixing coefficient  $M_{\text{eff}} \approx 2.81$ .

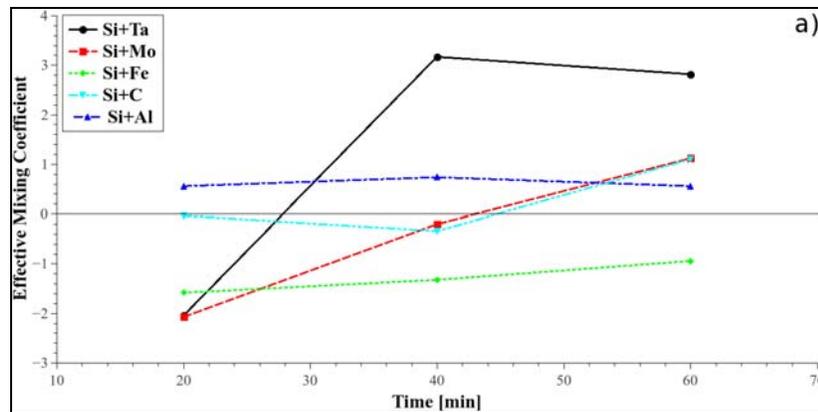
The effective mixing coefficient should tend to zero for the ideally mixed powders. To increase the accuracy of the measurement, more than one powder pellet can be analyzed. The limitation of this method can be appearance of large particle agglomerations, which are not taken into account using this mixing criteria. Experimentally, it was found that the influence of this problem can be decreased by increasing the sample pellets number.

**Table 2.** Calculated powder percentage area and effective volume ratio ( $V_{1E}/V_{2E}$ ) within cold press pellets after mixing in the micro pulse mixer

Micro Pulse Mixer						
Time [min]	1st	Area [%]	2nd	Area [%]	$V_1/V_2$	$M_{eff}$
20	Si	61.46	Al	38.54	1.03	0.56
20	Si	48.52	C	51.48	0.97	-0.03
20	Si	64.18	Fe	35.82	3.37	-1.58
20	Si	70.16	Mo	29.84	4.42	-2.07
20	Si	83.63	Ta	16.37	7.15	-2.04
40	Si	63.90	Al	36.10	1.03	0.74
40	Si	38.62	C	61.38	0.97	-0.34
40	Si	67.27	Fe	32.73	3.37	-1.32
40	Si	80.84	Mo	19.16	4.42	-0.20
40	Si	91.16	Ta	8.84	7.15	3.17
60	Si	61.43	Al	38.57	1.03	0.56
60	Si	67.45	C	32.55	0.97	1.10
60	Si	70.87	Fe	29.13	3.37	-0.94
60	Si	84.72	Mo	15.28	4.42	1.12
60	Si	90.88	Ta	9.12	7.15	2.82

The mixing depends significantly on differences in mixed element masses as well as powder particles sizes and volumes (Table 2). For the Fe and Ta experiments in  $\mu$ PM, mixing efficiency of iron was greater than for Ta, although both have same particles size, which suggests that lower density powders mix better. The effective mixing coefficient ( $M_{eff}$ ) varied with time for all samples (Figure 9). In this blending experiment no additional mixing media was used.

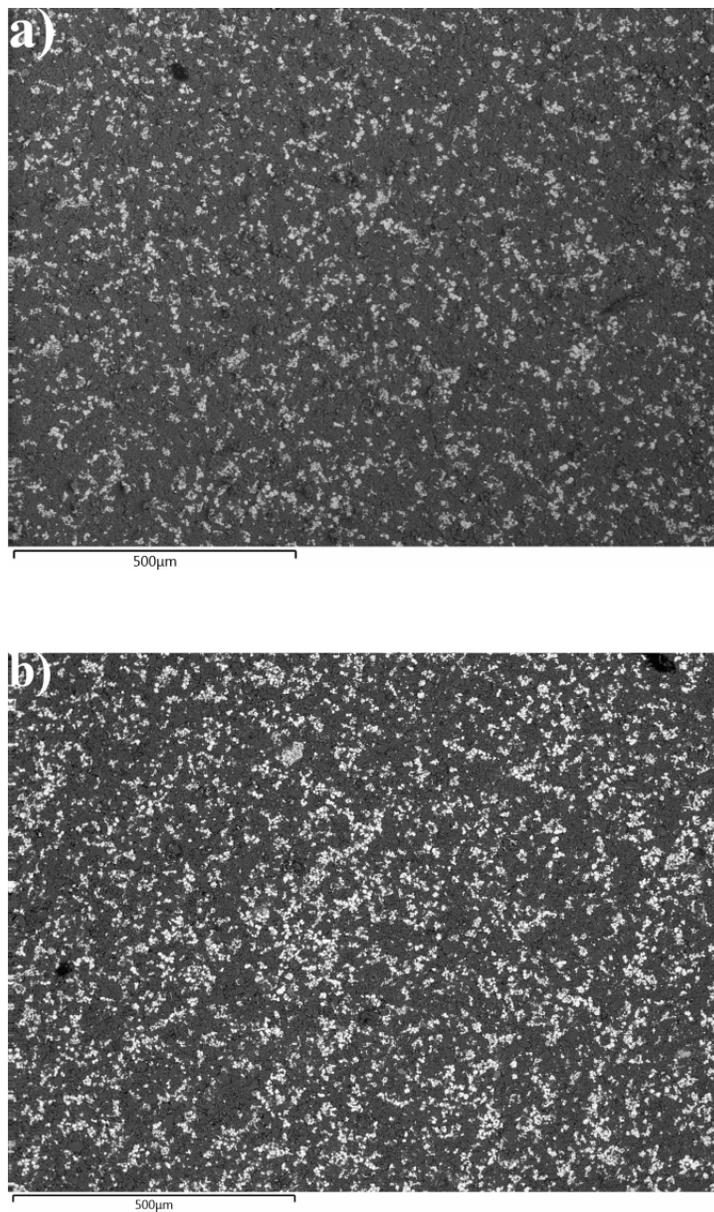
The percentage of a compound within a binary powder mixture varies and depends on the starting powder particle size and mass, which can be verified by BEI and EDS analysis. It also depends on the effective mixing coefficient.



**Figure 9.** The effective mixing coefficient ( $M_{eff}$ ) for  $\mu$ PM mixing as a function of mixing time for different elemental powder combinations.

The container fill level for micro pulse mixing was in the range 30-90%. Use of exactly the same weight instead of volume of various elemental powders caused variations in mixing container fill level. Nevertheless, mixing still took place, which can suggest that, in contrast to tumbling, mixing using the  $\mu$ PM technique depends less on the container's fill level than mixing using other methods. This result is preliminary and requires further investigation.

To experiment with increases in mixing speed and deagglomeration level, additional acrylonitrile butadiene styrene balls of 3mm diameter and total weight  $\sim 0.4g$  were placed inside powder vials, together with powders. Figure 10 shows backscattered electron images of silicon ( $45\mu m$ ) and molybdenum ( $5\mu m$ ) after 60 minutes of pulse mixing with and without additional balls. The increases in deagglomeration level are clearly visible.



**Figure 10.** Backscattered electron images of Si (dark-gray colour) and Mo (bright colour) powders after mixing: for 60 min using micro pulse mixer (a) without and (b) with two 3mm diameter acrylonitrile butadiene styrene balls of weight < 0.3g.

According to the results of EDS analysis, the use of additional mixing media for micro pulse mixing did not cause (or caused negligible) increases in impurities during mixing. This result can be attributed to several factors: the very low ball weights, small ball sizes, and small mixing container. The authors believe that increases of ball weight can significantly improve the powder distribution. However, increases of milling media size or weight can also result in increased impurities within a sample.

**Table 3.** Micro pulse mixer mixing properties summarized [2]

	Micro Pulse Mixer
Material handling	Powder
Dust-tight	Yes
Require low maintenance and energy	Yes
Can be change and clean easily	Yes
Does the entire amount of powder can be recovered after agitation	Yes
Does the mixer have flexibility to cope with a variable batch size?	Yes
Can the mixing vessel can be transported between the operations like loading, mixing, packaging?	Yes
Does it have easy access for sampling?	Yes
How well can the mixer separate the process materials?	Well
Does the mixer require frequent cleaning? If so what are the standards?	No
Process limitation	Relatively small amount of the powder
What should be the nature of the mixing surface?	Fine and coarse powder

Table 3 shows the ball milling and micro pulse mixer mixing properties summarized. The  $\mu$ PM was design for small powder quantity and currently uses glass bottles of volume in range of 2 ÷ 20ml.

## 6. Conclusions

The pulse mixing technique and the micro pulse mixing technique can be applied as an alternative approach to small powder volume mixing. The  $\mu$ PM method is based on damped harmonic oscillator motion, and is an efficient powder mixing method. Significant advantages over methods such as ball milling are short time of sample preparation, possibility of mixing small amounts of powder with negligible powder volume losses. Furthermore, no chamber cleaning after mixing is required. The  $\mu$ PM device can be used with additional mixing medium to increase powder deagglomeration. The above method, due to its simplicity and sample exchange speed, could be a practical solution for laboratory and small volume industrial use.

The effective mixing coefficient calculation represents a simple and fast approximation method of calculating powder particle mixing levels.

Although pulse mixing techniques show promising results, further research needs to be performed investigating different mixing properties, energy transfer efficiencies, and the influences of ball diameters and ball masses on the quality of mixing.

## Acknowledgement

This work was supported by funding from the Australian Research Council Discovery Grant No. DP130101390. We would like to thank Justyna Wimonc for the editorial work.

## Highlights

- We developed a new mixing method and a mixing device.
- This micro pulse mixer works on damped harmonic oscillations motion.
- Investigation of powder particles mixing properties in this method and conventional ball milling.
- We propose calculation of effective mixing coefficient as a measure of powder mixing efficiency.

### References

- [1] J. J. McCarthy, Troy Shinbrot, Guy Metcalfe, J. Eduardo Wolf and Julio M. Ottino, Mixing of granular materials in slowly rotated containers, *AIChE Journal* 42 (1996), 3351-3363.
- [2] Vivien Esnault, Abderrahim Michrafy, Daniel Heitzmann, Mohamed Michrafy and Driss Oulahna, Processing fine powders by roll press, *Powder Technology* 270 (2015), 484-489.
- [3] Tao Peng and Isaac Chang, Uniformly dispersion of carbon nanotube in aluminum powders by wet shake-mixing approach, *Powder Technology* 284 (2015), 32-39.
- [4] Yoshio Yamaga, Mitsugu Kanatani and Shuichi Nomura, Usefulness of a rotation-revolution mixer for mixing powder-liquid reline material, *Journal of Prosthodontics Research* 59 (2015), 71-78.
- [5] Dongguang Wei, Rajesh Dave and Robert Pfeffer, Mixing and characterization of nanosized powders: An assessment of different techniques, *Journal of Nanoparticle Research* 4 (2002), 21-41.
- [6] B. Yohannes, M. Gonzalez, A. Abebe, O. Sprockel, F. Nikfar, S. Kang and A. M. Cuitino, The role of fine particles on compaction and tensile strength of pharmaceutical powders, *Powder Technology* 274 (2015), 372-378.
- [7] Viral Shah, Manju Misra and Ekta Bharvada, Exploring mixing uniformity of a pharmaceutical blend in a high shear mixture granulator using enthalpy values obtained from DSC, *Powder Technology* 276 (2015), 103-111.
- [8] Aditya U. Vanarase, Juan G. Osorio and Fernando J. Muzzio, Effects of powder flow properties and shear environment on the performance of continuous mixing of pharmaceutical powders, *Powder Technology* 246 (2013), 63-72.
- [9] R. Deveswaran, S. Bharath, B. V. Basavaraj, Sindhu Abraham, Sharon Furtado and V. Madhavan, Concepts and techniques of pharmaceutical powder mixing process: A current update, *Research J. Pharm. and Tech.* 2(2) (2009), 245-249.
- [10] D. V. Khakhar, J. J. McCarthy, T. Shinbrot and J. M. Ottino, Transverse flow and mixing of granular materials in a rotating cylinder, *Phys. of Fluids* 9(31) (1997).
- [11] E. Wolf, Geometrical Aspects of Granular Solids Mixing, MS Thesis, Northwestern Univ., Evanston, IL, 1995.
- [12] Jens Fruhstorfer, Stefan Schafföner and Christos G. Aneziris, Dry ball mixing and deagglomeration of alumina and zirconia composite fine powders using a bimodal ball size distribution, *Ceramics International* 40 (2014), 15293-15302.
- [13] Sung-Mo Hong, Jin-Ju Park, Eun-Kwang Park, Kyeong-Youl Kim, Jung-Gu Lee, Min-Ku Lee, Chang-Kyu Rhee and Jin Kyu Lee, Fabrication of titanium carbide nano-powders by a very high speed planetary ball milling with a help of process control agents, *Powder Technology* 274 (2015), 393-401.

- [14] John S. Benjamin, Mechanical Alloying – A Perspective, Metal Powder Report 45 (1990), 122-127.
- [15] Ian Krycer and John A. Hersey, Fine powder mixing in a vibratory ball mill, International Journal of Pharmaceutics 6(2) (1980), 119-129.
- [16] H. Ferkel and R. J. Hellmig, Effect of nanopowder deagglomeration on the densities of nanocrystalline ceramic green bodies and their sintering behaviour, Nanostruct. Mater. 11(5) (1999), 617-622.
- [17] Holliday & Resnick, Fundamentals of Physics 1 (2011), 386-405.
- [18] Oscylator harmoniczny tłumiony (Damped harmonic oscillator), 2012.  
[http://efizyka.net.pl/oscylator-harmoniczny-tlumiony\\_8530](http://efizyka.net.pl/oscylator-harmoniczny-tlumiony_8530)
- [19] Mayer-Laigle, C. Gatumel and H. Berthiaux, Mixing dynamics for easy flowing powders in a lab-scale Turbula® mixer, Chemical Engineering Research and Design 95 (2015), 248-261.

