

## ONLINE MEASUREMENT OF THE PARTICLE SIZE DISTRIBUTION

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**Abstract:** New principle of the particle size distribution measurement is described. We analyze acoustic emission signal generated by a flow of particles impacting on a fixed obstacle. Changes of the power spectral density of the signal are well correlated with the particle size distribution. Our method was successfully used for measurement of particle size distribution in the range from 1mm to 10 mm of the ferrous sulphate monohydrate in real chemistry process. Particles were measured at the output of the dry kiln (temperature 150-200 °C, mass flow 4 tons/hour), where it is not possible to use any of other known principles such as particle vision, laser diffraction analyzers, ultrasonic analyzers, focused-beam reflectance measurement etc.

**Keywords:** Particle size distribution, acoustic emission.

### 1. INTRODUCTION

Ferrous sulphate monohydrate ( $\text{FeSO}_4 \cdot \text{H}_2\text{O}$ ) is an intermediate product in process of iron oxide pigment production by calcination. It is manufactured in rotary kiln from mixture of ferrous sulphate heptahydrate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ) and recycled monohydrate and it has quite broad particle size distribution from several micron dust to several mm particles (see Figure 1).



Figure.1 Particles of ferrous sulphate monohydrate

Particle size of monohydrate is important for subsequent production step as for calcination process it is necessary to remove fines from monohydrate. And therefore it is quite important to know how can be the produced monohydrate influenced by controllable variables as are temperature, feed rate, moisture, etc.

Usual way how to evaluate particle size distribution is to take a sample, perform sieve analysis and compute some characteristics of the material e.g.  $D_m$  and  $\gamma$  according to Rosin-Rammler function. The sieve analysis is time and labour consuming, the dust is not very pleasant and sampling a 4 tons/hour stream accurately and reproducibly is not a simple task. Therefore it is practically impossible to gain enough information about the influence of technological variables onto particle size distribution through the sieve analysis.

Other known principles of the particle size distribution measurement as particle vision, laser diffraction analyzers, ultrasonic analyzers, focused-beam reflectance measurement etc. [1], [2] are not suitable for our technology. Main limitations contain high temperature of the monohydrate, the size of the granules is beyond the range of accessible devices, the measurement systems are not constructed for extremely dusty and aggressive atmosphere and also very high price derived from too big — precision of particle size distribution analysis for our needs.

For all those reasons we have decided to design and check up a new method of measurement based on our previous research [3] that should be able to give — with enough of precision — online information about the change of particle size distribution on the output of the dry kiln. From experience we knew that the sound of monohydrate falling from kiln through discharge hopper differs when there is excess fine or excess coarse material. Therefore we decided to evaluate acoustic properties of falling monohydrate and compare the results with values found by sieve analysis of samples taken at the same time. We assume that by further long-term analyzing of specific acoustic parameters it would be possible to find the connection between the technological variables (temperature and velocity of the flow, humidity, etc.) and the changes in particle size distribution.

## 2. METHOD OF OPERATION

Collision of a particle with the surface of an obstacle generates a pressure wave. The reciprocal contact generates local deformation that partly spreads through the obstacle (waveguide) in the form of surface pressure waves – acoustic emission – and partly is the source of acoustic signal spreading over the surroundings. Pressure waves spreading through the space – the air – can be detected by a sensitive microphone and pressure waves spreading through the waveguide can be detected by a piezoelectric sensor.

Theoretical description of the problem is rather complicated. For an approximate description we can use the Hertz theory of impact which truly defines the quasistatic character of impact at a relative velocity of a body before the impact duration of which is much shorter than the time needed by the deformation wave to penetrate through the body and return to the place of impact. As shown in our previous work [3] we may use following approximate equations for the rough estimation of the time of impact  $T$  and the impact force  $F$

$$F \approx \rho^{0.6} v^{1.2} r^2 \quad T \approx \rho^{0.4} v^{-0.2} r \quad (1)$$

where

- $\rho$  – is the specific mass of the sphere material
- $v$  – velocity of particle
- $r$  – radius of the sphere

Out of the equation it is clear that for the impact duration has crucial influence the size of the particle, not the velocity. Also for the amplitude of the impact force (and via this on the amplitude of the pressure wave) is crucial the radius of the particle.

Although the model of the sphere impact onto the plane panel is rather simplifying in comparison with the reality at the moment of monohydrate output, the above explained relations and reasoning lead us to the following hypothesis:

- a) the overall energy of the emitted pressure waves is proportional to the mass flow
- b) emitted signal character is defined mainly by the particle size, less by its velocity
- c) „big“ particles emit long pulses with great amplitude, „small“ particles emit short pulses with small amplitude
- d) Ratio increase of the big particles is in time domain related to the increase of the impulse character of the signal (signal type „burst noise“), to ratio increase of the small particles than relates signal type „continuous noise“.
- e) Ratio increase of the big particles is in frequency domain related to the increase of low frequency spectrum components, ratio increase of the small particles than relates to the increase of higher frequency spectrum components.

## 3. EXPERIMENT

Experimental verification was done two times at production technology of Precheza company, Ltd, Prerov. The experiment setup is clear from the schematic drawing (see Figure 2) and photographs (see Figure 3).

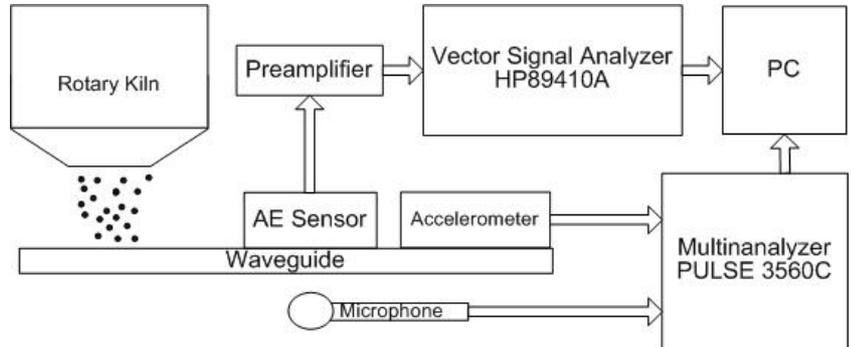


Figure 2. Basic structure of experimental setup

### 3.1 Measured quantities and used sensors and equipment

- vibrations measured on the rod where the granules fall, sensor: piezoelectric accelerometer Brüel&Kjaer 4393 (4507) and charge amplifier Brüel & Kjaer 2647A
- vibrations measured on the rod where the granules fall, sensor: piezoelectric accelerometer RFT KD91 (Brüel & Kjaer 4507) and charge amplifier Brüel & Kjaer 2647A
- noise caused by granules fall onto the testing rod, sensor: condenser microphone Brüel&Kjaer 4135
- acoustic emission signal generated by the granules' fall onto the rod, sensor: contact sensor AE Aura Milevsko SV416



Figure 3. Microphone position and position of the waveguide with AE sensor

Signals from the vibration sensors and the microphone were recorded directly by PULSE of Brüel&Kjaer company type 2827-002 apparatus (sampling frequency 25 kHz, 16

bit) with software PULSE LabShop 6.1.4.4. Signal from piezoelectric sensor of acoustic emission was at first amplified by 30 dB by Technická akustika comp. amplifier in the frequency range of 30 kHz to 1 MHz. The amplified signal was during the first measurement measured by low-frequency millivoltmeter, during the second measurement was conducted by two-wired unshielded twisted cable at about 100 meters length onto Vector Signal Analyzer HP89410A. The recorded quantity is the estimate of the frequency spectrum gathered by average from 50 realisations.

### 3.2 Measurement procedure

In the defined interval was take up a sample of granules and started recording of measured signals. After the end of recording (approx. 20 sec.) was again take up a sample of granules for further comparative sieve granulometry.

### 3.3 Processing of the data acquired by PULSE device

From the recorded time sequence of the signal were calculated the following signal parameters:

#### Time domain:

- effective value of the signal (RMS), absolute parameter
- statistical parameter “kurtosis” (4<sup>th</sup> order moment), ratio parameter
- frequency of the signal passes through the effective value of the signal to the number of passes of the signal through mean value, ratio parameter, relates to kurtosis, the advantage is the easy technical realisation
- number of passes of the signal through effective value of the signal to the overall number of the signal samples, ratio parameter

#### Frequency domain:

- ratio of octave spectral power to overall power, ratio parameter

### 3.4 Processing of the data acquired by HP 89410A analyser

Spectrum estimate was normalised because of rejection of the actual mass flow fluctuation influence. Normalisation is done by division of measured power density on individual frequencies by the overall mean value of the power density in the whole frequency range. The acquired spectra were further smoothed out by the moving average method and decimated.

## 4. DISCUSSION

Acquired characteristics of the acoustic signal were correlated with parameters describing granulometry of the removed samples gathered by sieve analysis – percentile parts of individual monohydrate fractions and coefficients of Rosin-Rammler distribution function.

The Rosin-Rammler function is represented by two parameters: mean particle size ( $D_m$ ) and  $\gamma$  value (width of distribution) and a goodness-of-fit factor:

$$R = 100 \text{Exp} \left[ -\frac{D}{D_m} \right]^\gamma \quad (2)$$

R is the retained weight fraction (%), D is the particle size ( $\mu\text{m}$ ),  $D_m$  is the mean particle size ( $\mu\text{m}$ ), and  $\gamma$  is a measure of the spread of particle sizes.

During our tests we found that the best achievable precision in sieve analysis is in this case approximately 1.6 (at  $\alpha$  0.05) for one measurement. So we can not distinguish between two measurements of  $D_m$  unless there are at least 1.6 apart. It is 40% change from estimated average  $D_m$  as seen from the Table 1 below.

Table 1. Sieve analysis result

Test	One	Two
average $D_m$	3.73	3.88
SD	0.89	0.74
$\gamma$	2.12	2.33
SD	0.78	0.85
no of measurements	50	45

SD - standard deviation

### 4.1 Evaluation of measurement made by PULSE

By comparing the correlation coefficients acquired from the first and second measurement we found, that each measurement gives completely different results. The most probable cause is wrong choice of sensors during the first measurement, when the used sensors BK 4507 had too low vibration frequency. Another very probable cause of the different results – mainly at the microphone measuring – is a big influence of the background noise, which it is impossible to reduce effectively.

Due to uneven particle flow (modulation via rotational frequency of the spiral conveyor) it is not possible to use for the measurement the absolute parameters of the signal (eg. effective value type), as these parameters are directly connected to the current mass flow.

The results acquired by signal processing in the time domain are not satisfactory. The hypothesis that the kurtosis growth of the probabilistic division of acoustic signal amplitude will correlate to the growth of proportional representation of the big particles was not confirmed.

### 4.2 Evaluation of measurement made by HP 89410A

In Table 2 you can see the correlation coefficient values acquired by comparison of the effective voltage values in three frequency domains with the values gathered from removed samples granulometry.

**Table 2: Figure of the correlation coefficients for overall granulometry parameters and ratio of the effective voltage values in chosen frequency domain.**

	$D_m$	gama	big	medium	small
U1/U2	0,60	0,02	0,62	-0,49	-0,25
U2/U1	-0,72	-0,07	-0,73	0,42	0,46
U3/U1	-0,70	-0,33	-0,73	0,17	0,72
U1	0,72	0,08	0,73	-0,45	-0,42
U2	-0,39	0,12	-0,44	0,52	0,00
U3	-0,55	-0,50	-0,65	0,06	0,74

where the frequency domain of the measured voltage is:

U1: 3 - 25 kHz

U2: 50 - 80 kHz

U3: 100 - 150 kHz

More detailed idea about the interrelation of the frequency spectrum and particle size you can get out of Figure 4.

## 5. CONCLUSION

From the above mentioned it is clear that from the frequency spectrum of the acoustic emission signal you can conclude the proportional representation of the particle size. In agreement with the hypothesis, the proportion growth of the granules with bigger diameter causes the amplitude growth in lower frequencies and the proportion growth of the granules with smaller diameter causes the growth of spectrum components in higher frequencies. But it is necessary to perform the signal normalisation, which reduces the fluctuation influence of the particle mass flow falling onto the measurement rod.

From the performed measurements we can assume the following presumptions for measurement chain proposition:

- the best sensor is acoustic emission sensor (AE)

- particle size information is carried mainly by frequency spectrum of the range from 10 kHz to 250 kHz
- the overall dynamic signal range is approx. 60 dB, in individual frequency domains approx. 30 dB

The above described method of measuring of the particle size distribution represents a supplement to usually used techniques. Our method is low-cost and robust, but it is less exact than classical methods. Measurements can be carried out even at high temperatures and the only restriction is the Curie temperature of the piezoelectric material of the acoustic emission sensor.

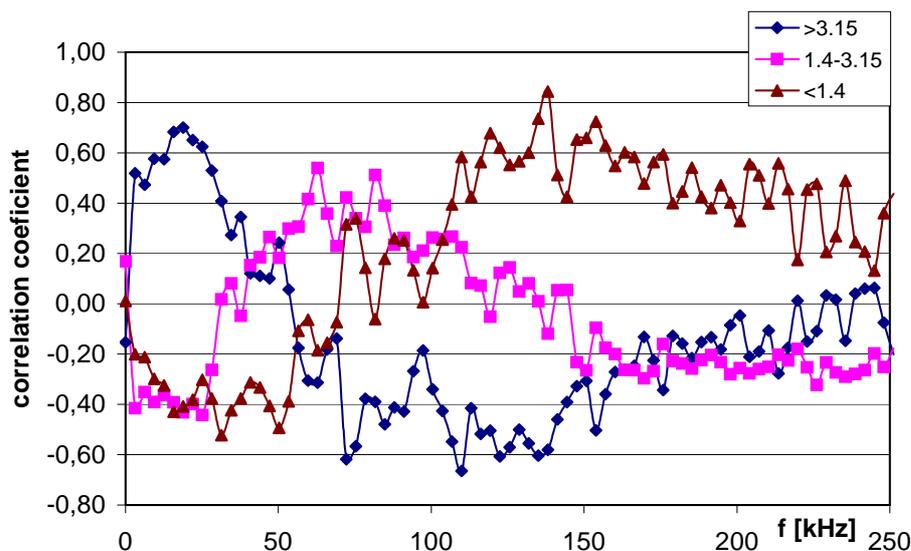
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**Fig. 4. The dependence of correlation coefficients on frequency for particles of small dimension (<1,4 mm), medium dimension, (1,4-3,15 mm) and large dimension (>3,15 mm)**