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ACOUSTIC CHARACTERIZATION OF A MICROCRYSTALLINE CELLULOSE POWDER DURING AND AFTER ITS COMPRESSION

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ABSTRACT

Acoustic emissions were detected, both during the roller compaction of the microcrystalline cellulose powder and from single tablets after compaction by a single-punch tablet machine, via air using a microphone with a flat frequency response up to 20 kHz. Both of the compaction units were instrumented for the measurement of applied compressive force. The microcrystalline cellulose roller compacted using compressive forces below 30 kN showed a quite normal compaction behaviour but the product compacted at this force split into two and turned to yellow by its edges. This "capping" phenomenon was indicated by an enhancement of acoustic emission in the region of about 17-23 kHz. Acoustic emissions from single tablets after compaction by a single-punch tablet machine seemed to appear as wave packets consisting in very many frequency components that may, in addition, be time-varying. However, some small peaks were found probably being characteristic of these transient sounds.

INTRODUCTION

Acoustic emission techniques have been developed for the prediction of failure in engineering structures and in geologic materials [1], based on the fact that strain release gives a raise to acoustic emission as well as to plastic deformation in solid state materials.

These techniques have been applied for the monitoring of the compaction of pharmaceutical powders, i. e. drugs and excipients. Rue et al. [2] fitted a piezo-electric transducer to the take off chute of a single-punch tablet machine which was instrumented for the measurement of applied compressive force during compaction, and took the acoustic emission signals of a spray-dried paracetamol powder through a band-pass filter of 70–500 kHz. They pointed out the capping phenomenon of the tablets, i. e. the tablets had split into two, three or multiple parts, correlating with one, two or multiple peaks in the time domain graphs of the acoustic emission signals immidiately after the compression load was removed.

Rue et al. [3] also monitored the acoustic emissions of compressed and ejected tablets of a sodium chloride powder on aforementioned transducer, via a thin layer of an acoustic coupling agent on one face of the tablet. They found out that the acoustic activity of the tablets decreased exponentially during one hour after compression while the strength of the tablets was observed to increase exponentially, and suggested that the mechanism of strength increase was related to the deformation process which produced the acoustic emissions.

The time domain acoustic emission graphs of pharmaceutical materials during compression were also recorded by Waring et al. [4]. They used a piezo-electric transducer with a wave guide which was spring loaded against the side wall of the die of an force-gauge-instrumented single-punch tablet machine using acoustic coupling gel to enhance the acoustic signal from the die to the wave guide. The band of the portable activity monitor they used was set at 95–600 kHz. Similarly to Rue et al. they plotted the acoustic emissions as a function of time together with the applied compressive force. The difference between the results of these authors was that according to Waring et al. the "post-compression peak" appeared quite material-independently every time when the compression load was removed. No clear correlation between the acoustic emission signal and the capping phenomenon was finded out by their work although the tendency to form capped tablets of paracetamol and some other pharmaceutical powders was observed. On the basis of the time domain acoustic emission graphs they divided the compression cycle of the single-punch tablet machine into three stages: a "particle reorganization stage" characterized by the highest emissions, a relatively quiet period called a "particle consolidation stage", and a "post-compression stage" immidiately after the compression load was removed characterized by a sharp peak of emission. They also carried out material dependent effects like the effect of particle size: experiments with sodium chloride and lactose showed that by increasing the particle size both the average signal level during the "particle reorganization stage" and the total emissive counts integrated over the whole compression cycle were increased. Compression cycles both without powder and with a microcrystalline cellulose powder seemed to be quiet and therefore the authors suggested that the brittleness of the material would be the condition of the acoustic emission signal with this type of instrumentation. Ductile materials would not produce detectable signals.

There is a problem with using of these material-transmitting acoustic emission techniques for the monitoring of powder compaction. It is well-known that plastic deformation of crystalline materials like metals gives raise to an acoustic emission. The two basic sources of acoustic emission are (i) nonstationary dislocation motion and (ii) dislocation annihilation process mainly related to the operation of a Frank–Read source during plastic deformation of crystals [5]. Waring et al. [6] studied the acoustic emission during the deformation of lactose monocrystals and observed a clear correlation between the acoustic emission signal and the force–displacement (i. e. work) profile measured simultaneously using a ceramic–head differential type piezo–electric transducer. It is obvious that a great deal of acoustic emission measured through the die wall during powder compaction is caused by the metalwork of the tablet press and so it is also in the case of the post–compression peak because the strain release of the tablet press structures as well as of the compacted material gives a rise to an acoustic emission. Spectral analysis would be required to separate the acoustic signal emitted by the powder from the disturbation signal caused by the metalwork of the tablet press.

To avoid this problem present authors measured the acoustic emission signals of materials compacted continuously by means of a roller compactor [7]. The acoustic emissions were detected via air using a microphone. Both crystalline (and brittle) materials like lactose, and more or less amorphous (and ductile) materials like maize starch and microcrystalline cellulose were studied. Spectral analysis showed e. g. that the tightening of maize starch powder produces sounds mainly below 2 kHz. The observed capping phenomenon of microcrystalline cellulose was not clearly indicated in the spectra and hence further studies has been conducted using a microphone with a flat frequency response up to 20 kHz. In addition, present contribution contains a study of the acoustic emissions of single microcrystalline cellulose tablets compacted by means of a single–punch tablet machine.

MATERIALS AND METHODS

Microcrystalline cellulose with a particle size of about 50 µm was obtained by Edward Mendell Finland Oy, Nastola, Finland.

The roller compaction was made by a Bepex Pharmapaktor 200/50 P roller compactor. The operation of the roller compactor was described in our earlier contribution [7]. The entry compaction or tabletting was made by a single–punch tablet machine. Both of the compaction units were instrumented for the measurement of applied compressive force during compaction.

Acoustic emissions were detected via air by a Brüel & Kjaer 4134 condenser microphone with a flat frequency response up to 20 kHz (Fig.1). The acoustic emissions from the compacted and ejected tablets were detected in a special silent chamber of brass to maximize the signal–to–noise ratio. The chamber itself and the microphone in the chamber were isolated from surrounding structures by the aid of foamy plastics. The hole in the wall of the chamber for the signal cable was tightened using a packing ring of rubber. The tablet was held in a hole of foamy plastics immidiately in front of the microphone.

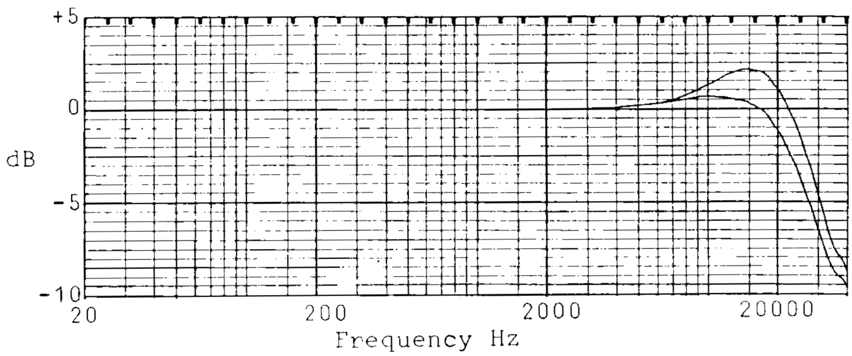

FIGURE 1
Frequency response curve of the microphone used (from the calibration chart of the Brüel & Kjaer 4134 condenser microphone).

Acoustic emission signals were amplified by a Brüel & Kjaer 2639 preamplifier and recorded by a Teac DA–P20 Digital Audio Tape (DAT) recorder with an effective sampling frequency of 48 kHz. The acoustic emission signals were analyzed using a Hewlett–Packard 35665 A Dynamic Signal Analyzer which is a two–channel FFT spectrum analyzer with a frequency range that extends to just over 100 kHz. It takes 1024 samples of time data to produce 512 points of frequency domain data. The effective sampling frequency of the analyzer is set at about 2.56 times the analyzed frequency span. So, when using e. g. a span of 25.6 kHz the length of a time record is about 15.6 ms being simply 1024 divided by the effective sampling frequency which is now about 65.5 kHz. The resolution (in Hz) is the inverse value of the time record length (in seconds) and is now about 64 Hz.

RESULTS AND DISCUSSION

Roller compaction

Figures 2, 3 and 4 show the acoustic emission spectra of the microcrystalline cellulose powder during roller compaction using compressive forces of 10, 20 and 30 kN. A recording time of about one minute made possible an averaging of 300 records of data. The microcrystalline cellulose compacted using compressive forces below 30 kN showed a quite normal compaction behaviour but the product compacted at this force split into two and turned to yellow by its edges. These results correlate with our previous results [7]. The acoustic emission spectra in the Figures 2 and 3 are almost identical but the spectrum in the Fig. 4 is essentially different. The difference between the spectra in the Figures 2 and 4 is seen in the subtraction curve of the spectra (Fig. 5). A significant difference between the

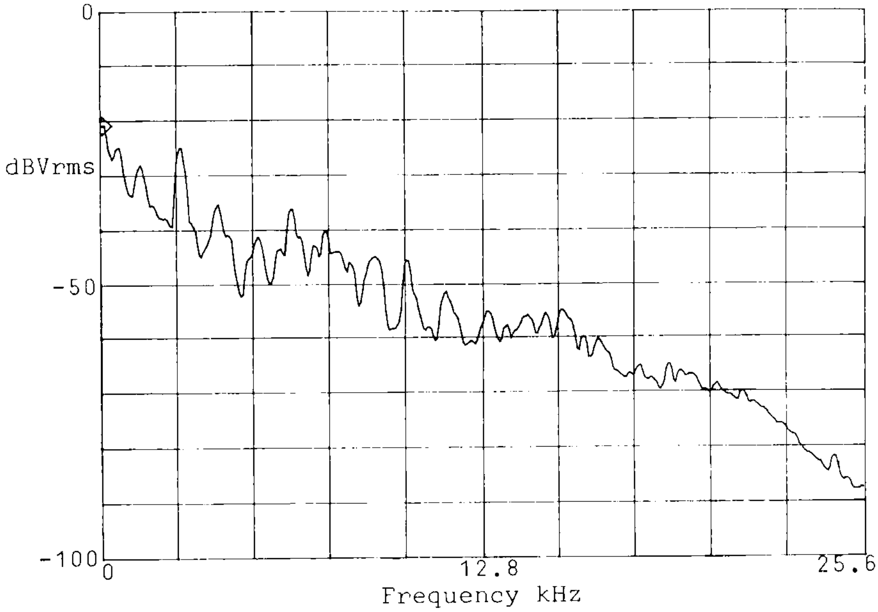

FIGURE 2
Acoustic emission spectrum of a microcrystalline cellulose powder during roller compaction using a compressive force of 10 kN.

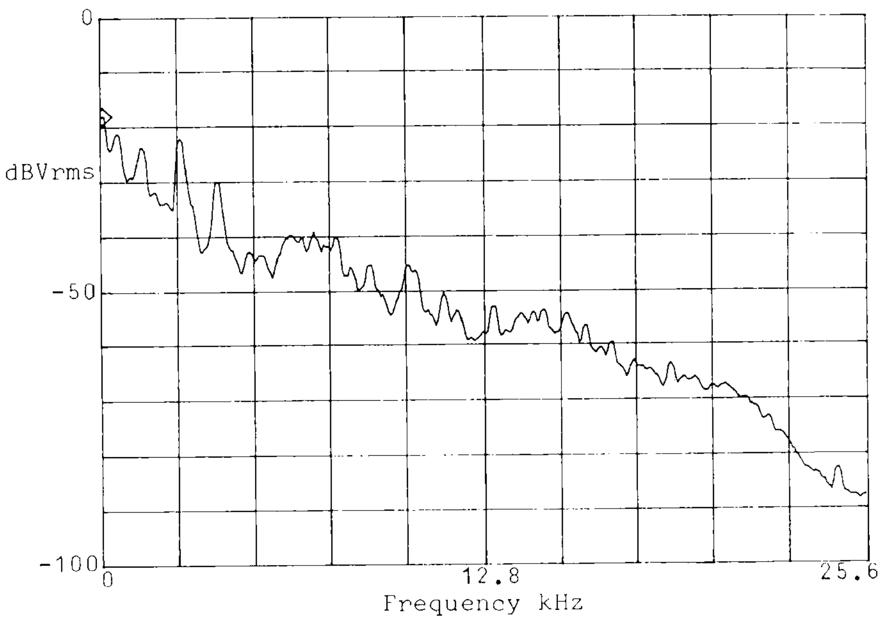

FIGURE 3
Acoustic emission spectrum of a microcrystalline cellulose powder during roller compaction using a compressive force of 20 kN.

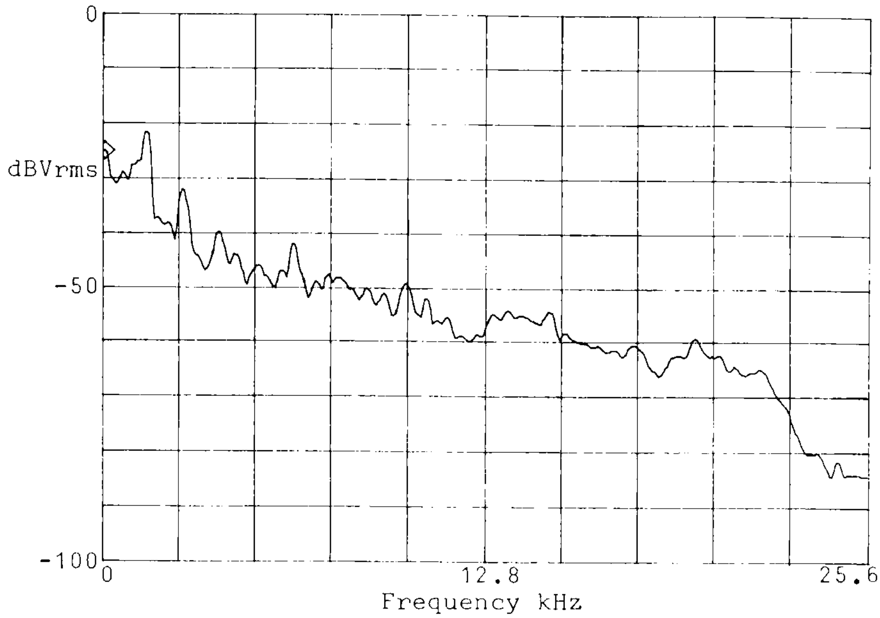

FIGURE 4
Acoustic emission spectrum of a microcrystalline cellulose powder during roller compaction using a compressive force of 30 kN.

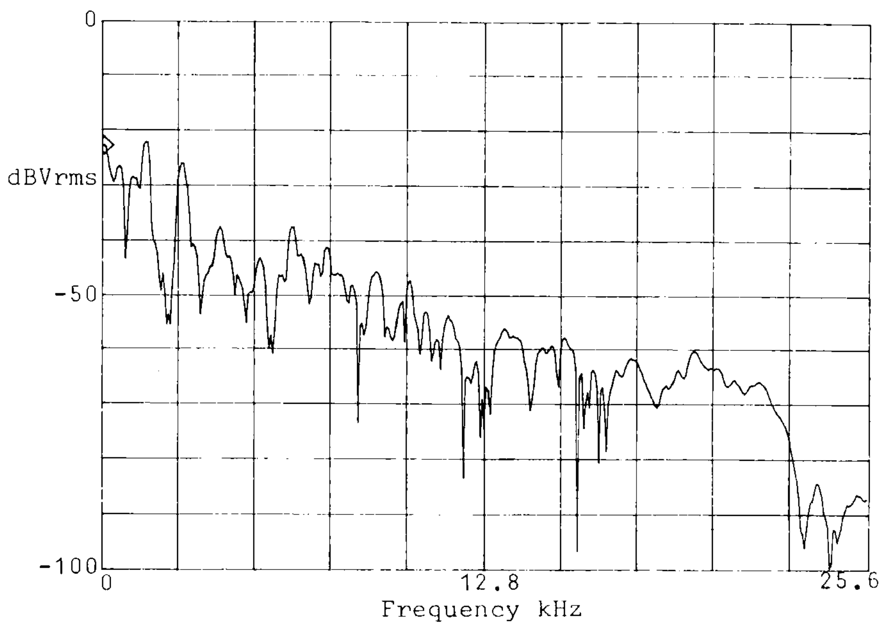

FIGURE 5
The subtraction of the spectra in the Figures 2 and 4.

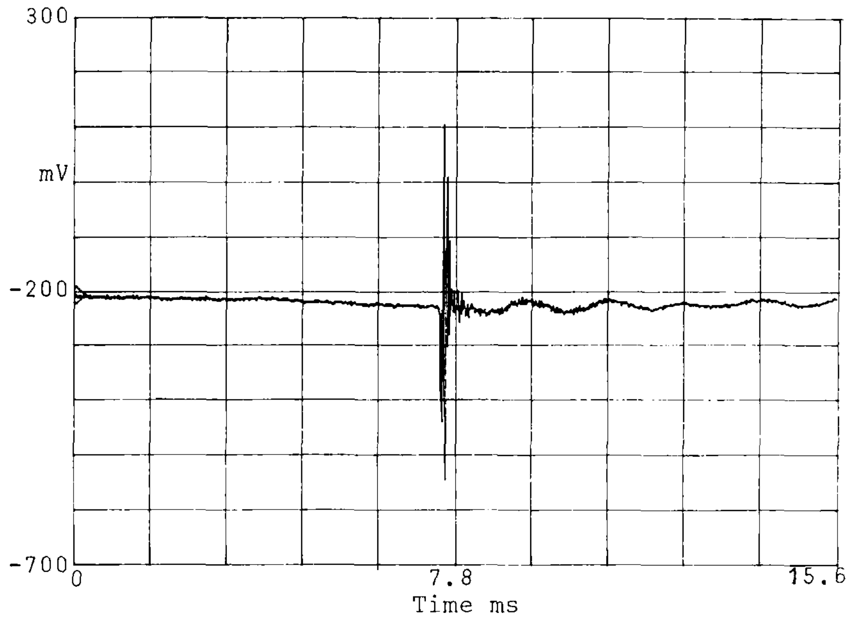

FIGURE 6
A time capture of the acoustic emission signal of a single tablet compacted by a single-punch tablet machine using a compressive force of 10 kN, including a wave packet of acoustic emission.

spectra in the region of about 17–23 kHz is related to an enhancement of emission due to aforementioned "capping" phenomenon (see also Figures 2 and 4). The integration of the acoustic power over this frequency span would make possible the quantitative analysis of the "capping" phenomenon as a function of applied compressive force, and the integral signal could be used as a feedback signal to adjust the compressive force to avoid the unwanted phenomenon.

Tablets

Acoustic emissions from single tablets compacted by a single-punch tablet machine using a compressive force of 10 kN were detected in a special silent chamber of brass during one minute period after compaction. A few distinct wave packets were observed and they were clearly audible, too. Fig. 6 shows a time capture of about 15.6 ms including one wave packet in question. This wave packet could be "multiplied" by taking a time capture several times over the period which included the wave packet (Fig. 7). By comparing the spectra of the single and the multiple wave packets (Figures 8 and 9) it became clear that such a wave packet consist in very many frequency components that may, in addition,

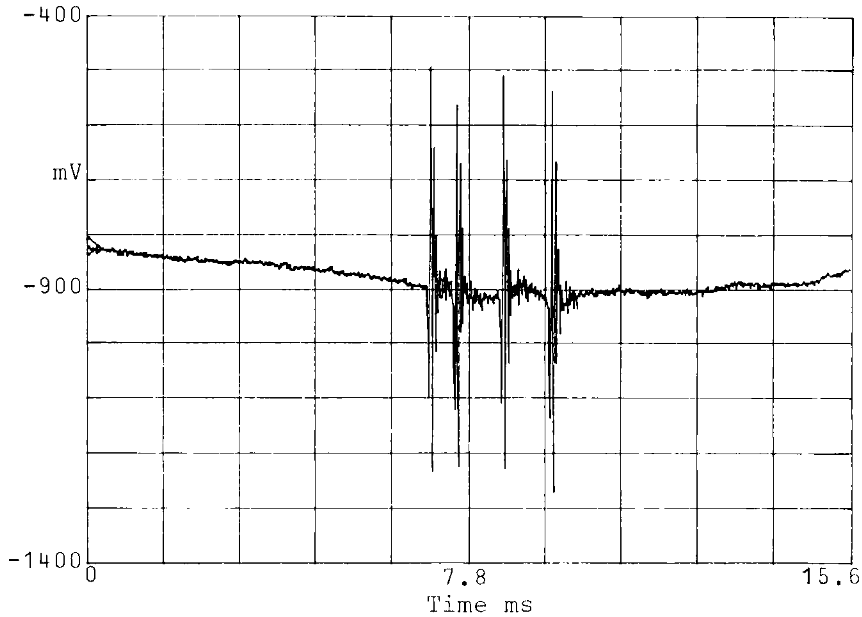

FIGURE 7
A multiple time capture over the wave packet in the Fig. 6.

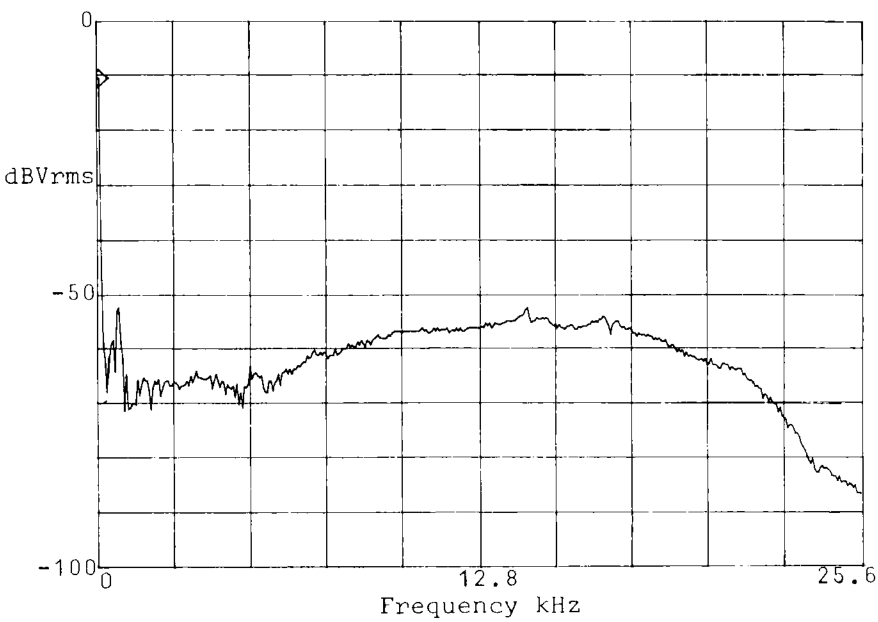

FIGURE 8
The spectrum of the wave packet in the Fig. 6.

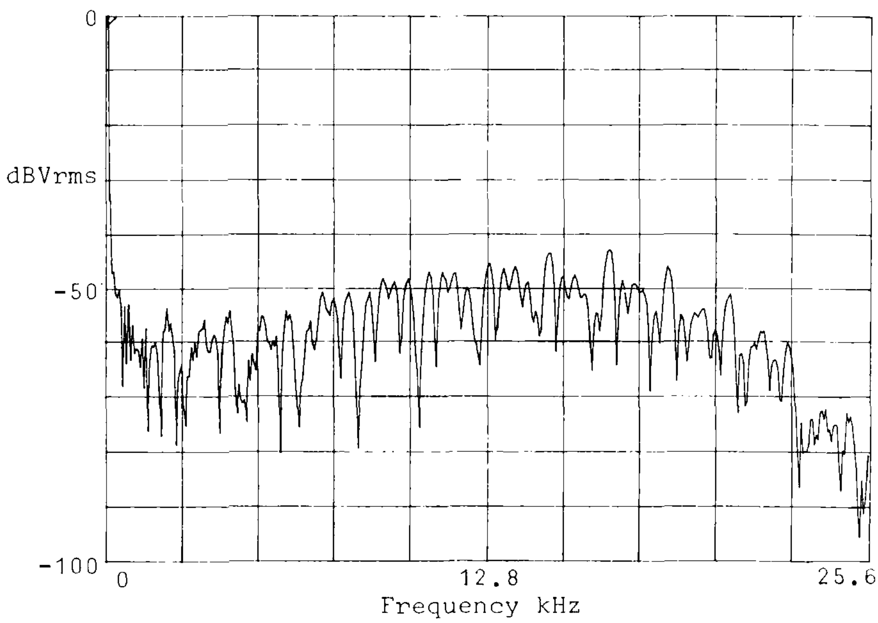

FIGURE 9
The spectrum of the multiple wave packet in the Fig. 7.

be time–varying resulting in an additional problem within the Fourier analysis of the wave packet. However, the spectrum of the multiple wave packet (Fig. 9) showed small peaks at 14.8, 16.9, 18.9 and 21.0 kHz weakly correlating with the spectrum of the single wave packet (Fig. 8) and thus, it can be stated that these peaks might be characteristic of these transient sounds.

CONCLUSION

Acoustic emission signal of a powder both during and after its compression can be detected via air using microphones, and to some extend it is possible to separate the frequency components of the signal to catch information on physical phenomena related to the compaction behaviour of the powder, and to use it for adjusting and/or controlling the compaction process.

Of course, a lot of the acoustic energy emitted by a powder during and after its compression appear at higher frequencies and cannot be detected via air. The high frequency part of the acoustic signal emitted by a finished tablet can be turned to a voltage signal using a piezo–electric transducer on one face of the tablet. Similarly the high frequency part of the acoustic signal emitted by a powder which is being compacted by a tablet machine can be detected through the side wall of the die of the tablet machine but it may be very difficult to separate the signal from the disturbation signal caused by the metalwork of the tablet machine.

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