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ACOUSTIC EMISSION DURING POWDER COMPACTION AND ITS FREQUENCY SPECTRAL ANALYSIS

Arvi Hakanen, Ensio Laine
University of Turku, Dept. of Physics, Laboratory of Industrial Physics,
SF-20500 Turku, Finland

Harry Jalonen, Kari Linsaari, Juha Jokinen
Orion Corporation Farmos
P.O.Box 425, SF-20101 Turku, Finland

ABSTRACT

Acoustic emission was detected during compaction of three different pharmaceutical materials: lactose monohydrate, microcrystalline cellulose and maize starch varying compressive forces between 0 and 60 kN. Acoustic emission signals were recorded on magnetic tape with a microphone and transformed to frequency spectra by using FFT-analysis.

After rough identification of frequency components each spectrum was divided into three bands. By using integrated band powers the acoustic activity on these bands could be compared quantitatively. Many frequency peaks were observed, too, and many of them could be identified.

INTRODUCTION

The word "powder compaction" means compressing powder to compressed products that can be crushed into granules of desired shape and size. Powder compaction is an important part of dry granulation in pharmaceutical technology. The dry granulation makes possible to granulate moisture sensitive materials.

Organic powder compaction is based on the fact that the van der Waals' forces are able to cause binding between powder particles the distances of which are smaller than 1000 Å. In addition the binding improves if the number of the connection points between powder particles increases. So the vicinity and the large number of the connection points are the two presumptions of organic powder binding.

Friction work together with possible powder particle fractures give rise to acoustic emission when powder is compressed. The sounds are produced mainly on audible region. Thus acoustic emission signals carry information on the compression behaviour of pharmaceutical materials or on the compaction behaviour if the shape, the strength and the size distribution of the primary powder particles are appropriate concerning the binding presumptions. We can prove that there are general properties (e.g. the van der Waals' forces) but also specific properties (e.g. the shape, the strength and the size distribution) of materials which have an effect on organic powder compaction.

When a sufficient amount of powder is compressed continuously the acoustic emission signal can be recorded with a microphone and transformed to a frequency spectrum by using FFT-analysis. A continuous compressing has been made possible by a special machine called a "roller compactor".

MATERIALS AND PROCEDURE

Three different materials: lactose monohydrate (De Melkindustrie Veghel B.V., Veghel, Holland), microcrystalline cellulose (Edward Mendell Finland Oy, Nastola, Finland) and maize starch (Cerestar Scandinavia A/S, Holte, Denmark) were compacted with forces between 0 and 60 kN. Microcrystalline cellulose had a certain powder particle size of 50 μm and it was the only material of the three that really became "compacted". The value 0 kN means the situation in which the compactor is rolling without any compressive force. When compacting lactose monohydrate the compressive force could not be increased over 35 kN because the material became sticky.

The materials were compacted with Bepex Pharmapaktor 200/50 P roller compactor (see fig. 1). Corrugate profiled rolls were used during every measurement. In this type of machine both roll axles are rigidly supported. This means that they are practically radially inelastic during the compaction process. The gap between the rolls is kept constant. On the other hand the small elasticity of the axles is used for measuring the compressive force.

The compressive force is regulated by changing the feeding flow of the compacting material. The feeding flow can be regulated by changing the rotating speed of the "feeder–compression screw" (also called "force-feeder") of the compactor while the rotating speed of the rolls is kept constant (see fig. 1). This is important when considering the acoustic emission measurements. The sounds produced by the rotating rolls keep constant and therefore it is easier to separate them from the sounds produced by the compacting material. The speed of the rolls was set at 6 rpm.

The rotating speed of the feeder–compression screw is adjusted automatically by an adjustment device which is connected to the force measuring gauge. The force signal is controlled continuously and there is a feedback to the feeder–compression screw. The compressive force can be

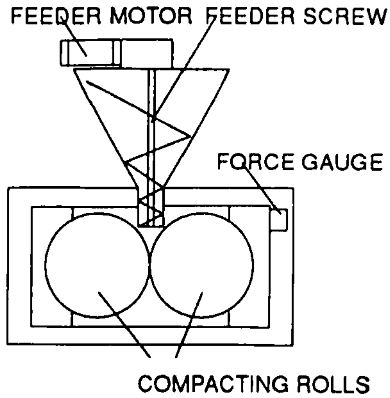


FIGURE 1

A ROLLER COMPACTOR

read in a digital display. Every time the compressive force was expected to become constant before the measurement was started. The force signal could also be recorded during the compaction for analyzing its stability.

Acoustic emission signals were recorded on magnetic tape using an omnidirectional electret condenser microphone with a frequency range of about 50 Hz – 12 kHz. The distance between the microphone and the compacting powder was held about 10 cm. The recording time was two minutes except when maize starch was compacted with compressive force of 60 kN when the material run out.

Acoustic emission signals were transformed to frequency spectra using acoustic analyzer with an operational frequency range of 0 – 100 kHz (see fig. 3, 4 and 5). In the spectra, on X–axis lies the frequency from 50 Hz to 12.85 kHz and on Y–axis the intensity level

$$L[dBV_{rms}] = 20\log(U/U_0)\ , \quad \text{where}$$

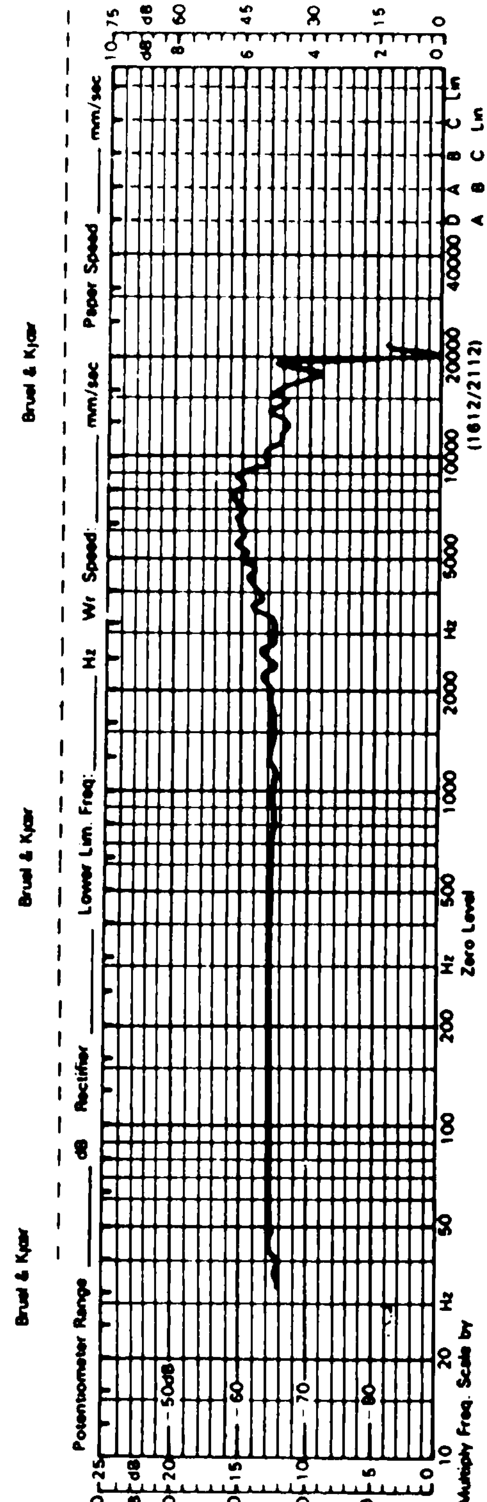
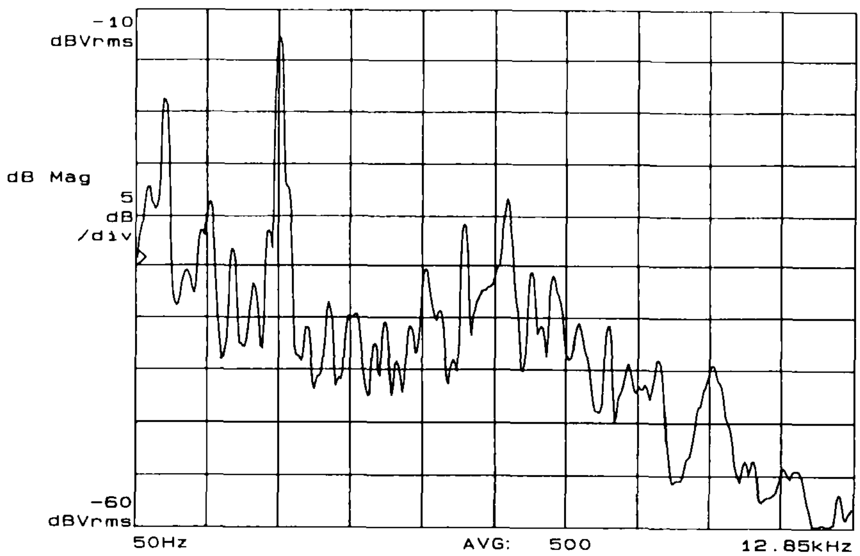


FIGURE 2

THE SENSITIVITY CURVE OF THE MICROPHONE

A **LACTOSE MONOHYDRATE 0 kN**



B **LACTOSE MONOHYDRATE 15 kN**

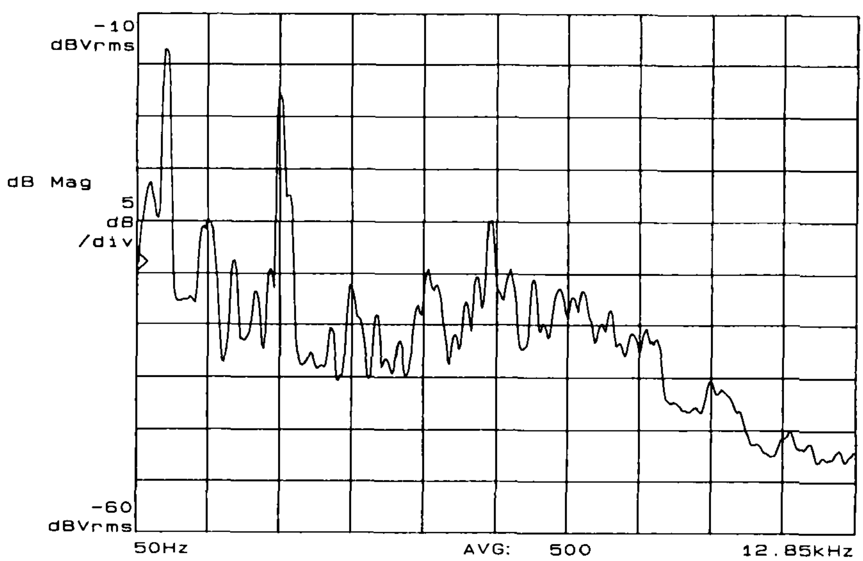
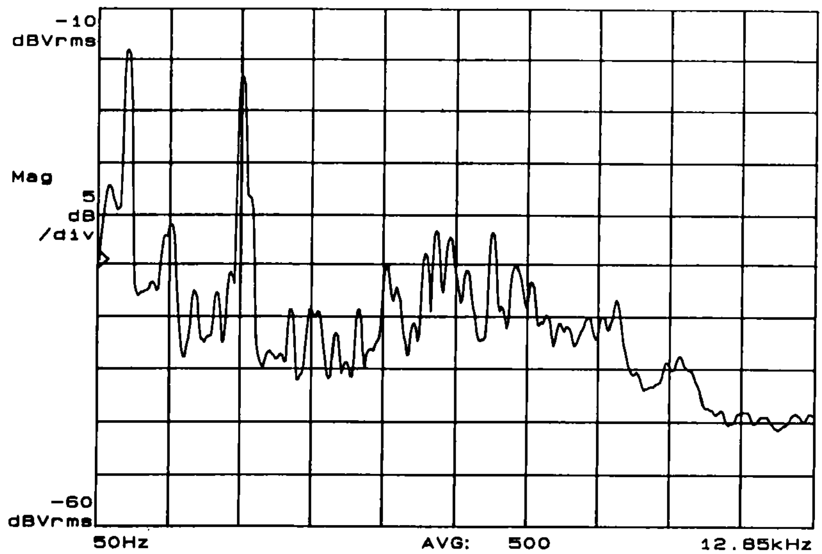


FIGURE 3 (A, B, C, D, E, F)

THE ACOUSTIC EMISSION SPECTRA ON COMPACTING LACTOSE MONOHYDRATE WITH VARIOUS COMPRESSIVE FORCES

1. See fig. E (30 kN). The increasing jump in the third band (7.7 – 12.85 kHz) power is also seen in table 1.

C **LACTOSE MONOHYDRATE 20 kN**



D **LACTOSE MONOHYDRATE 25 kN**

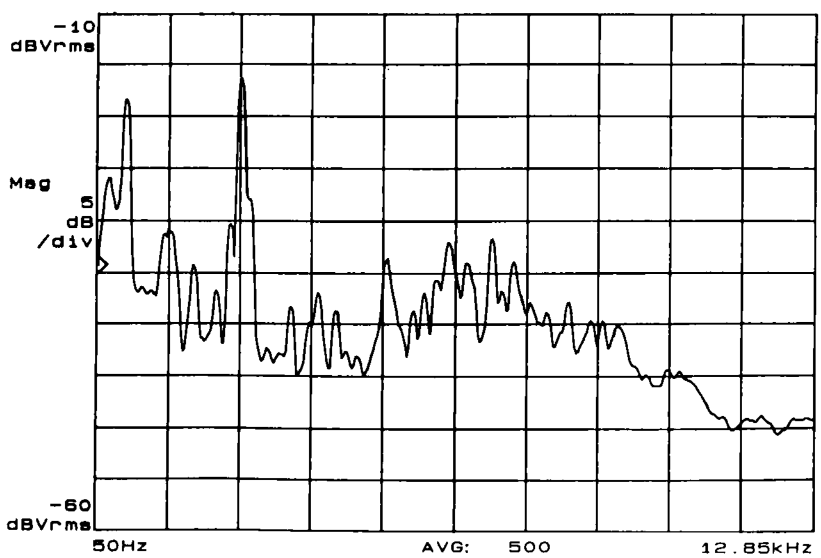


FIGURE 3. Continued

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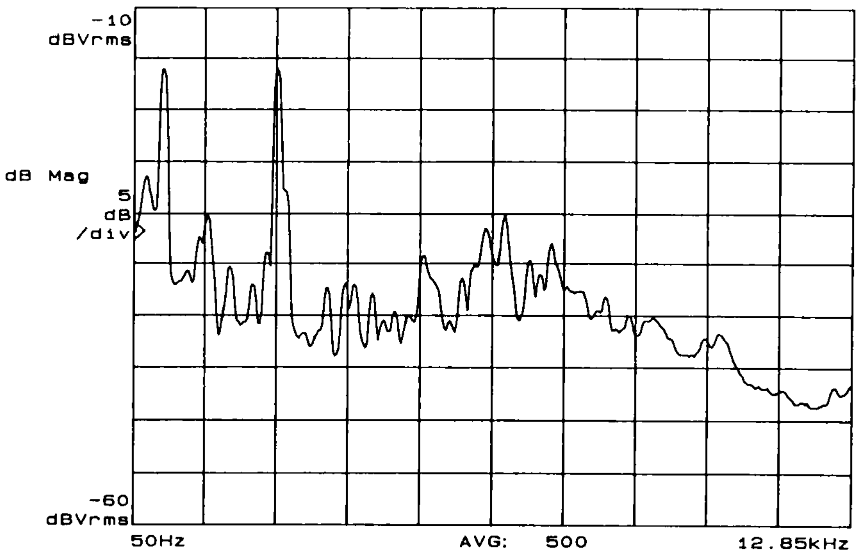
E **LACTOSE MONOHYDRATE 30 kN**



F **LACTOSE MONOHYDRATE 35 kN**

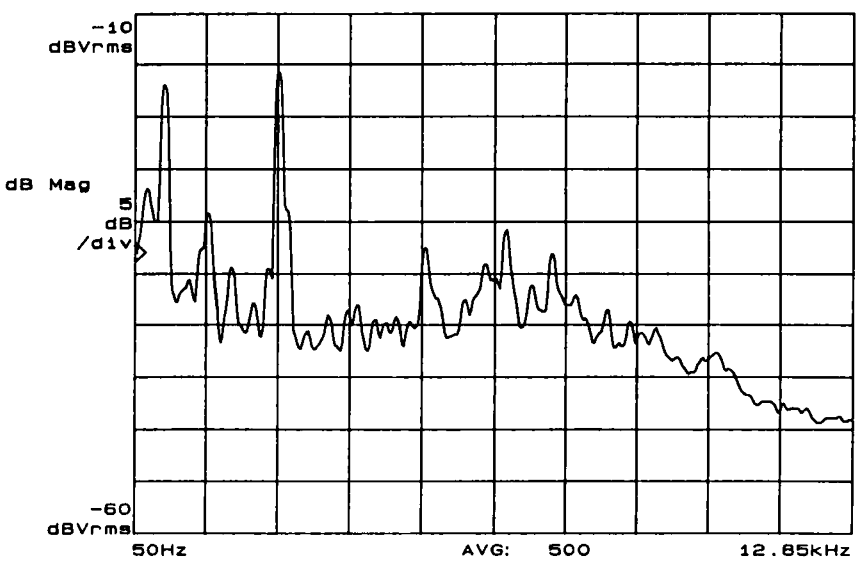
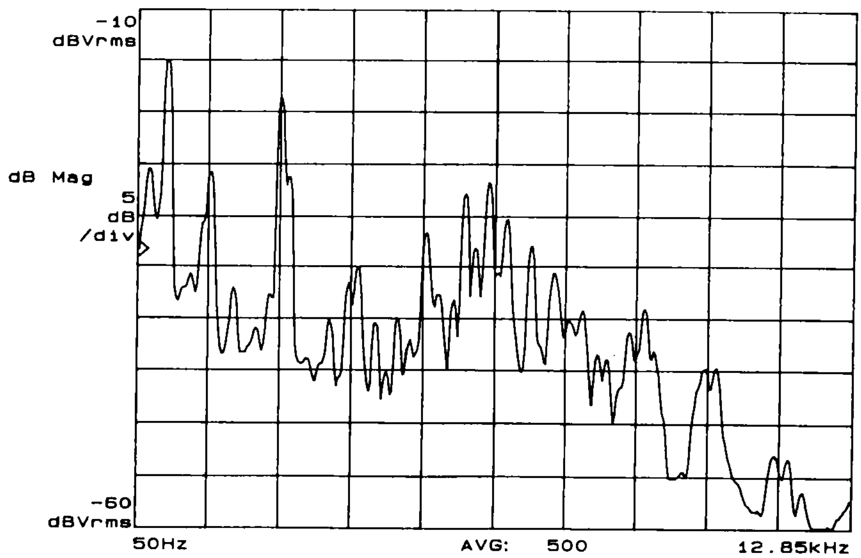


FIGURE 3. Continued

A **MICROCRYSTALLINE CELLULOSE 0 kN**



B **MICROCRYSTALLINE CELLULOSE 5 kN**

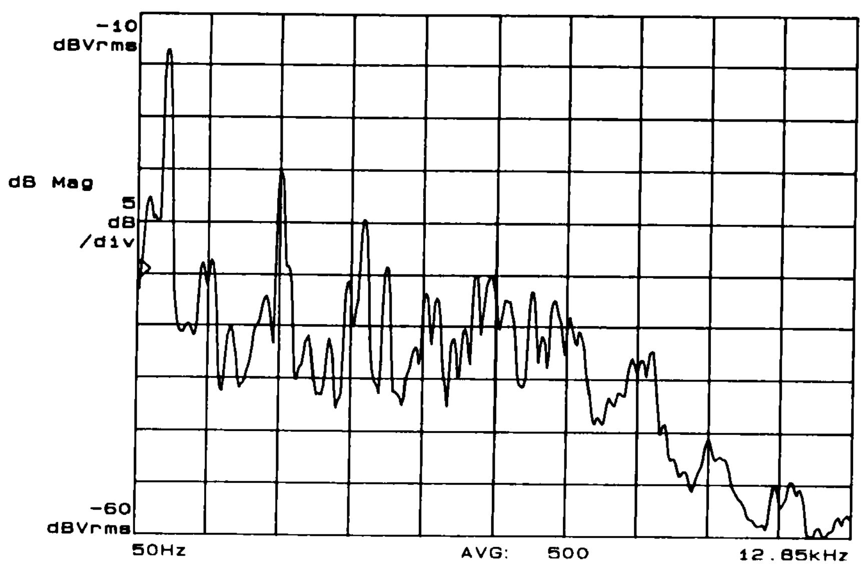


FIGURE 4 (A, B, C, D, E, F, G, H, I)

THE ACOUSTIC EMISSION SPECTRA ON COMPACTING MICROCRYSTALLINE CELLULOSE WITH VARIOUS COMPRESSIVE FORCES

1. See fig. B, C, D, E and F. The feeder–compression screw scratched the cylinder wall and gave rise to a pair of peaks in the beginning of the second band (3.8 – 7.7 kHz).

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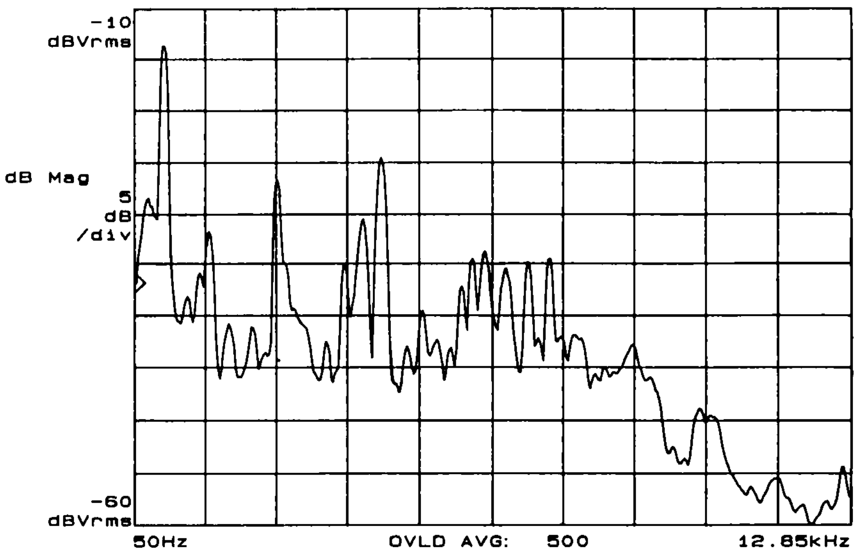
C MICROCRYSTALLINE CELLULOSE 10 kN



D MICROCRYSTALLINE CELLULOSE 15 kN

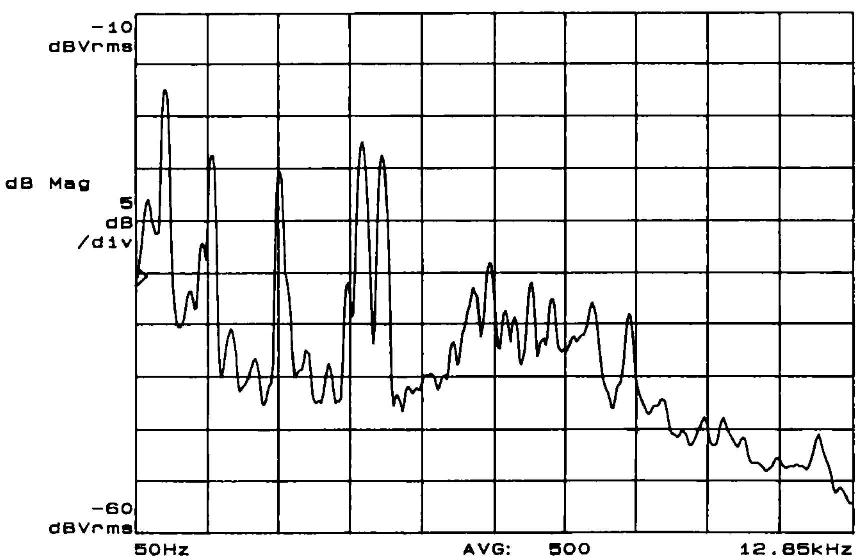
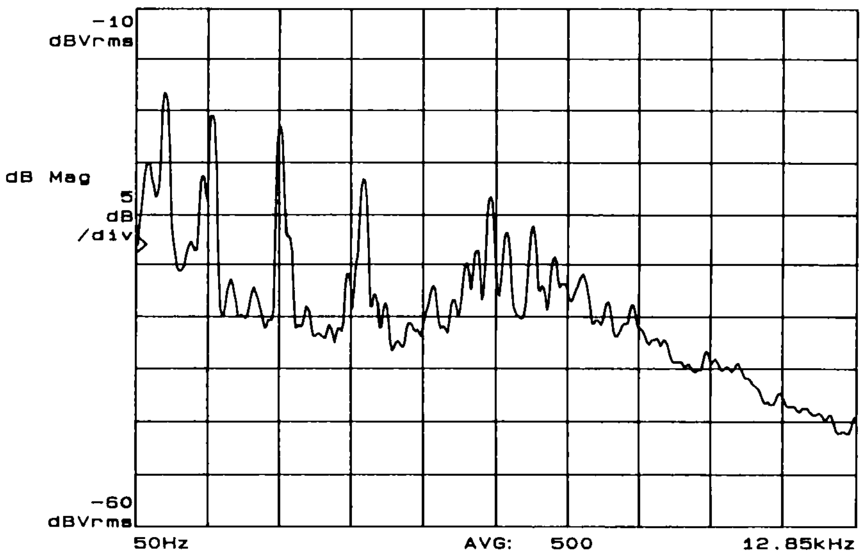


FIGURE 4. Continued

2. See fig. E (20 kN). The increasing jump in the third band (7.7 – 12.85 kHz) power is also seen in table 1.

3. See fig. G, H and I. A peak in the middle of the third band (7.7 – 12.85 kHz) may be caused by the capping phenomen.

E **MICROCRYSTALLINE CELLULOSE 20 kN**



F **MICROCRYSTALLINE CELLULOSE 25 kN**

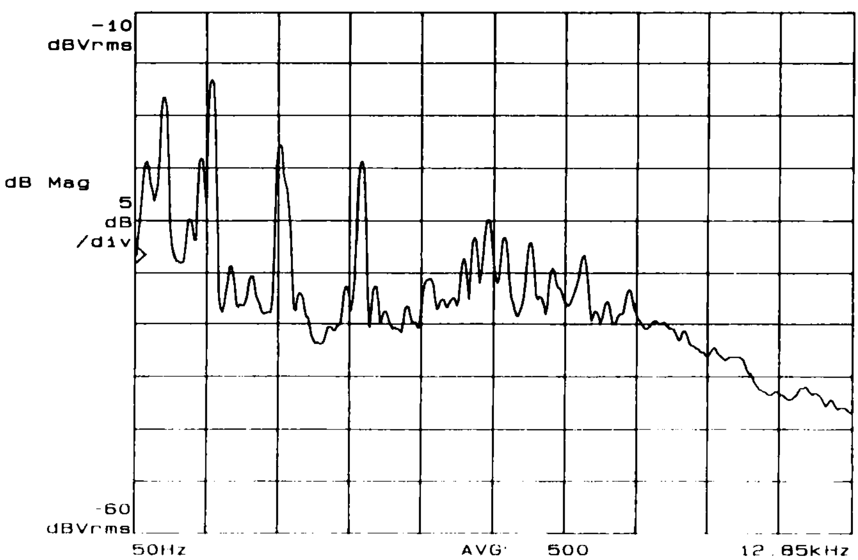


FIGURE 4. Continued

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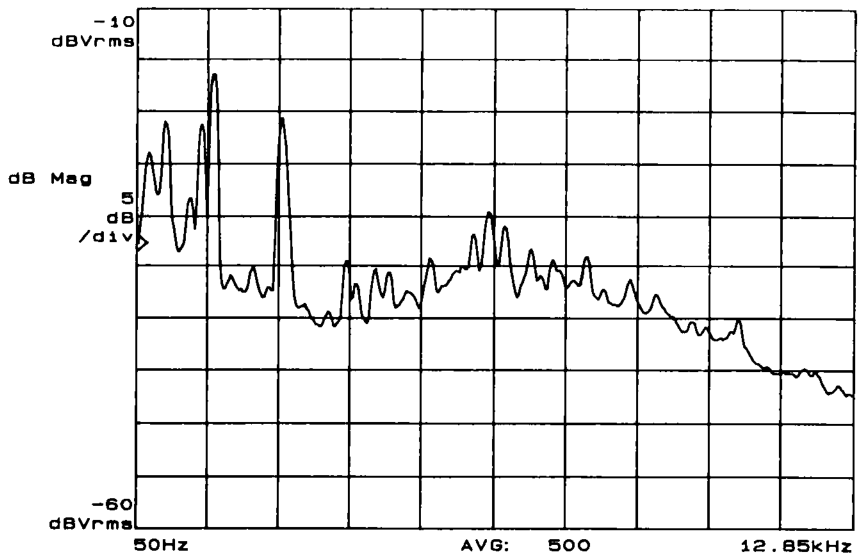
G **MICROCRYSTALLINE CELLULOSE 30 kN**



H **MICROCRYSTALLINE CELLULOSE 35 kN**

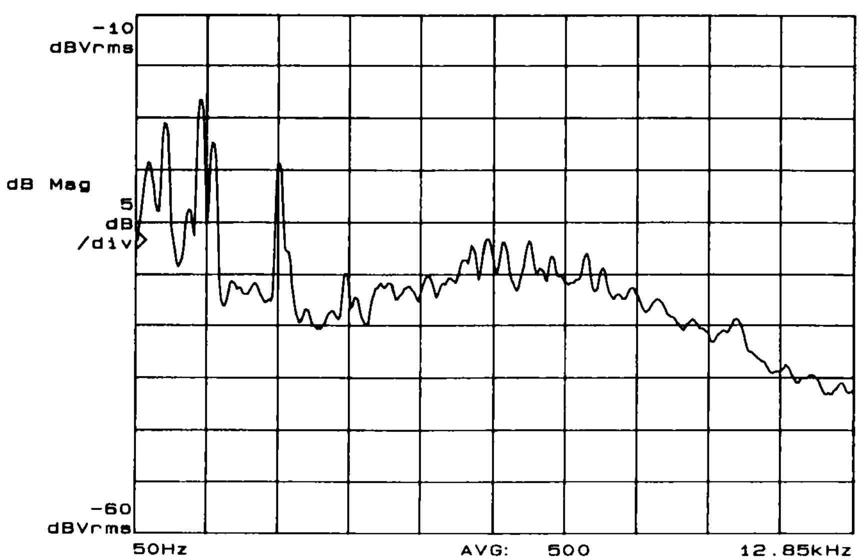


FIGURE 4. Continued

I MICROCRYSTALLINE CELLULOSE 40 kN

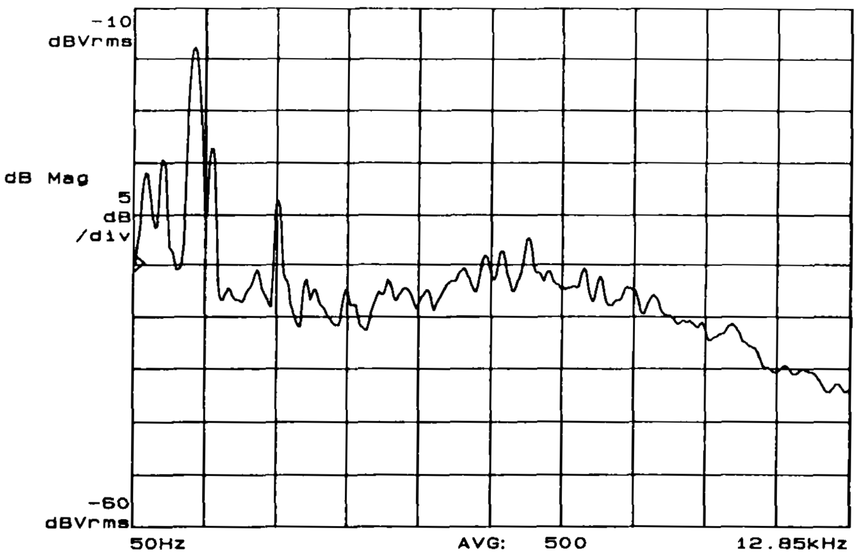


FIGURE 4. Continued

$U[V_{rms}]$ = signal voltage

U_0 = reference voltage = 1 V_{rms} = maximum signal voltage,

from –60 dBV_{rms} to –10 dBV_{rms}. The sensitivity of the microphone was –66 dBV_{rms} ± 3 dBV_{rms}. The shape of the sensitivity curve of the microphone had an effect on the general shape of the spectra.

RESULTS AND DISCUSSION

It was found out that the sounds (and their harmonics) produced by the roller compactor were discrete frequency peaks whereas the sounds produced by the compacting powder appeared on wider frequency bands. In

A MAIZE STARCH 0 kN

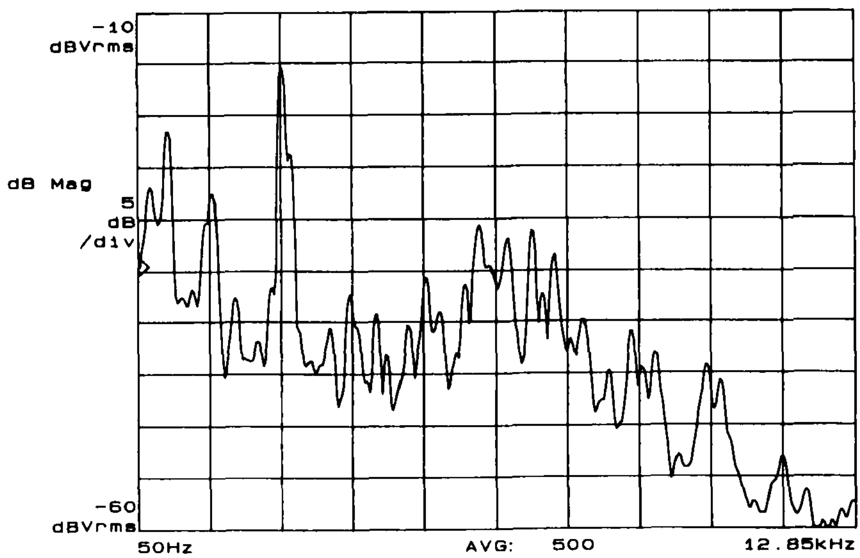


FIGURE 5 (A, B, C, D, E, F, G, H, I, J, K)

THE ACOUSTIC EMISSION SPECTRA ON COMPACTING MAIZE STARCH WITH VARIOUS COMPRESSIVE FORCES

1. See fig. B (5 kN). A maximum in the first band (50 Hz – 3.8 kHz) power caused by tightening of the powder is also seen in table 1.

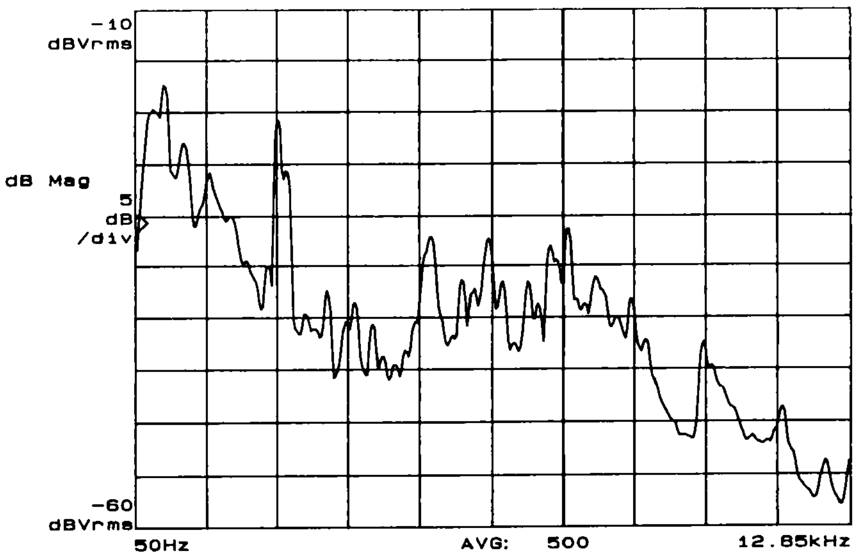
2. See fig. F (25 kN). The increasing jump in the third band (7.7 – 12.85 kHz) power is also seen in table 1.

addition the "machine-frequencies" held their positions in the spectra relatively well when the compressive force was varied.

The total spectrum or the total band (50 Hz – 12.85 kHz) was divided into three bands by means of the changes observed in the spectra with various compressive forces. The band power [dBV_{rms}] was integrated over each band (see table 1).

The first band (50 Hz – 3.8 kHz) included the fundamental "machine-frequencies" and its band power was near the total band power. When

B MAIZE STARCH 5 kN



C MAIZE STARCH 10 kN

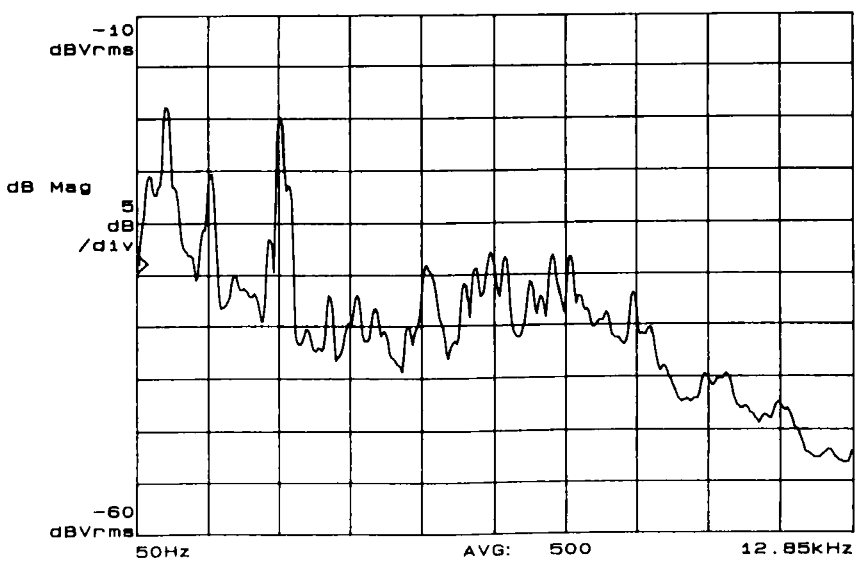
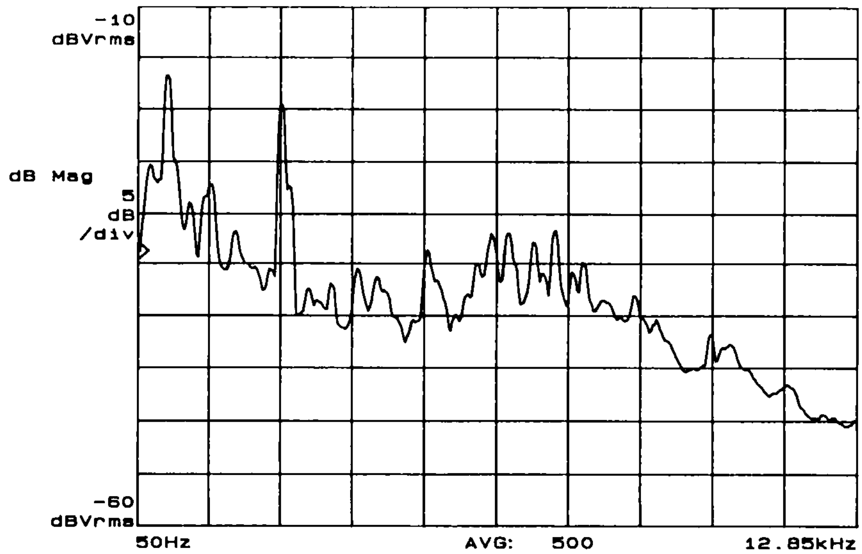


FIGURE 5. Continued

(continued)

D **MAIZE STARCH 15 kN**



E **MAIZE STARCH 20 kN**

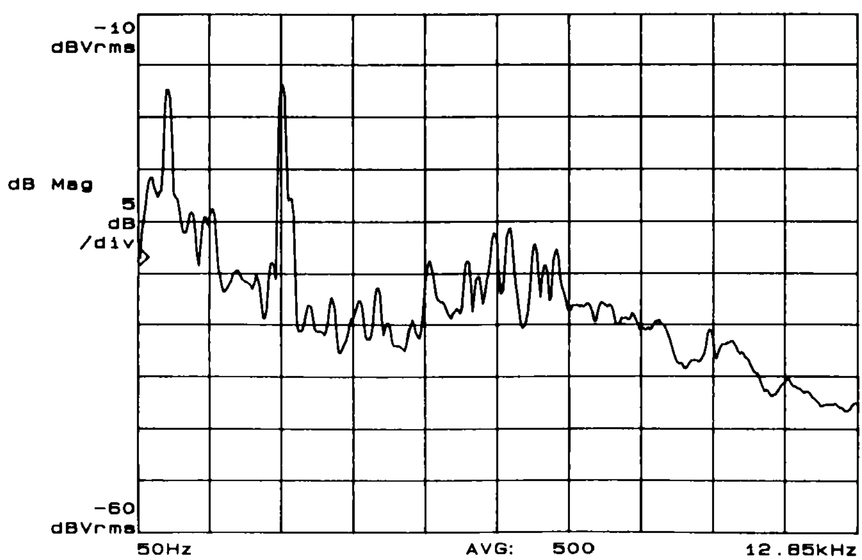
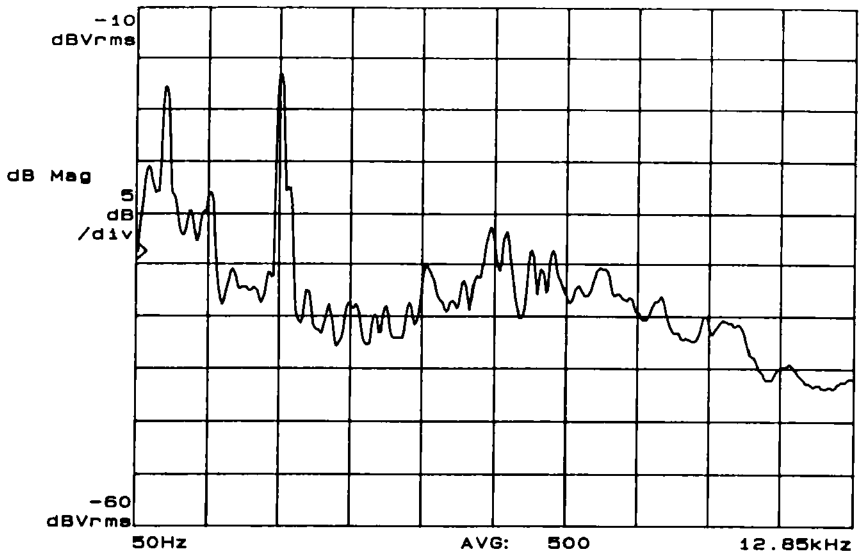


FIGURE 5. Continued

F **MAIZE STARCH 25 kN**



G **MAIZE STARCH 30 kN**

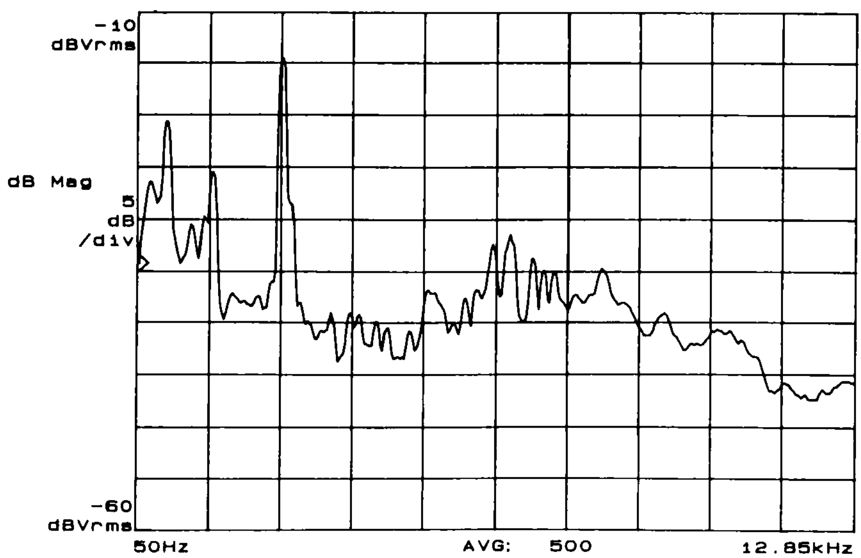
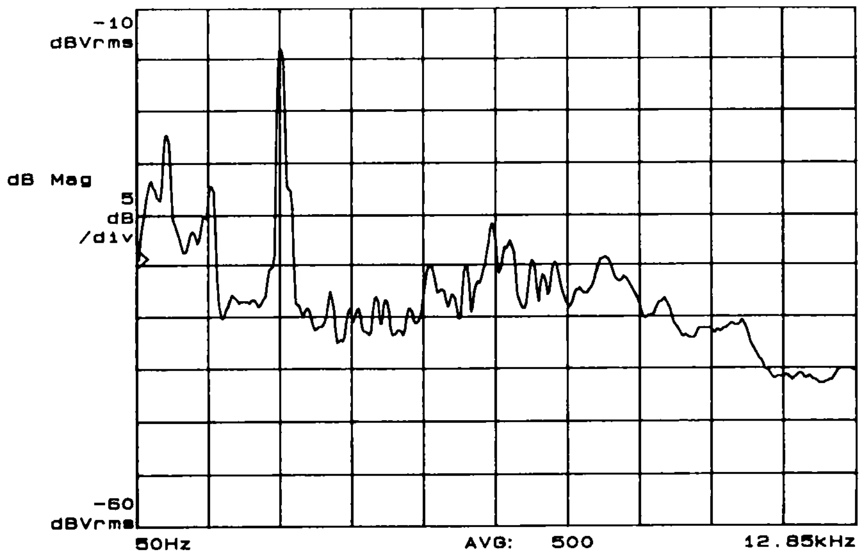


FIGURE 5. Continued

(continued)

H MAIZE STARCH 35 kN



I MAIZE STARCH 40 kN

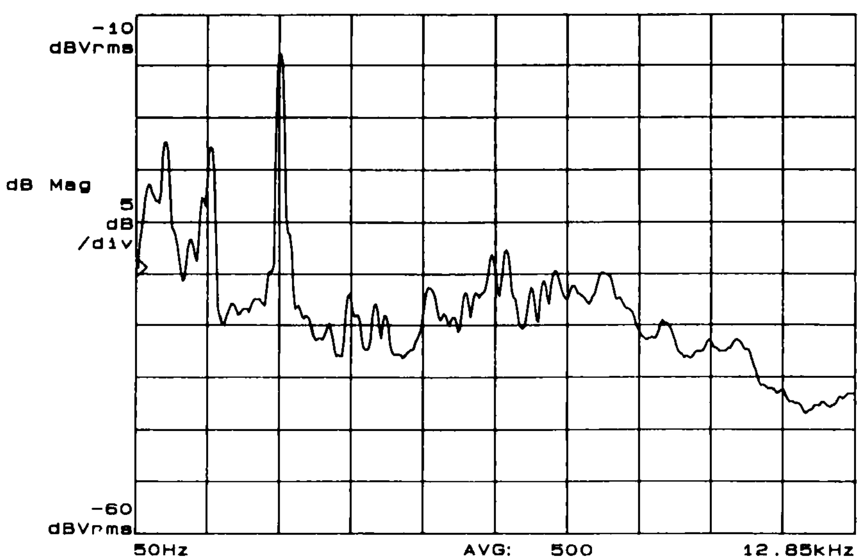
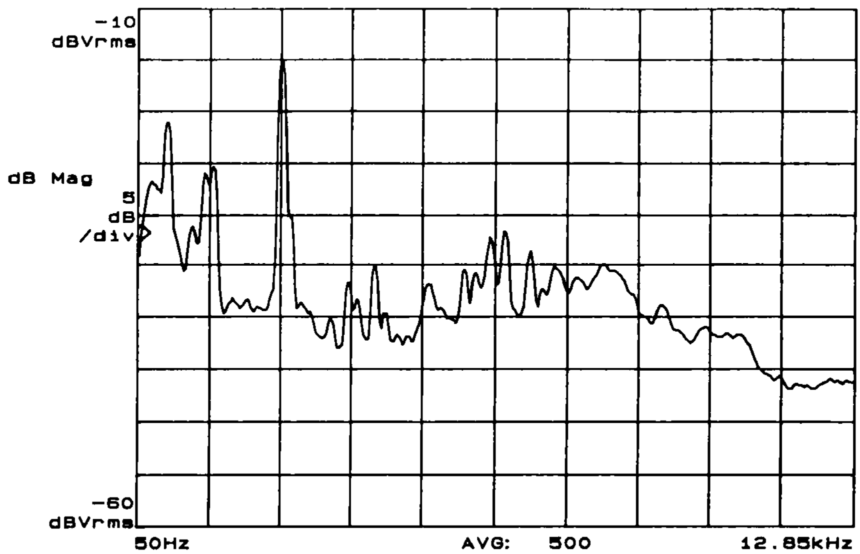


FIGURE 5. Continued

J MAIZE STARCH 50 kN



K MAIZE STARCH 60 kN

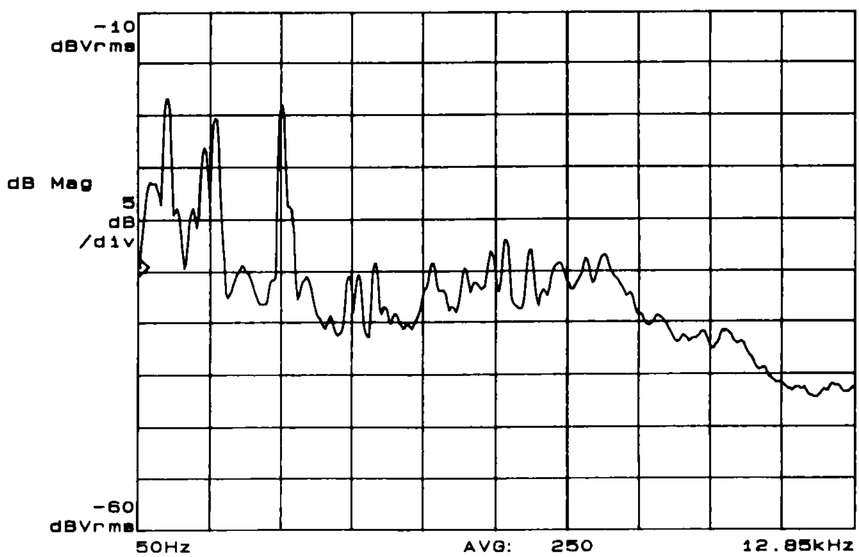


FIGURE 5. Continued

TABLE 1

INTEGRATED BAND POWERS [dBV_{RMS}]

COMPACTED MATERIAL AND THE COMPRESSIVE FORCE	TOTAL BAND 50Hz-12.85kHz	BAND I 50Hz-3.8kHz	BAND II 3.8-7.7kHz	BAND III 7.7-12.85kHz
LACTOSE MONOHYDRATE 0 kN	-10.5	-10.8	-22.4	-31.6
LACTOSE MONOHYDRATE 15 kN	-10.8	-11.2	-22.6	-27.5
LACTOSE MONOHYDRATE 20 kN	-10.9	-11.3	-22.5	-27.2
LACTOSE MONOHYDRATE 25 kN	-12.2	-12.8	-22.2	-27.0
LACTOSE MONOHYDRATE 30 kN	-11.3	-11.9	-21.3	-25.9
LACTOSE MONOHYDRATE 35 kN	-11.6	-12.2	-22.0	-26.5
MICROCRYST. CELLULOSE 0 kN	-11.4	-12.1	-20.3	-30.0
MICROCRYST. CELLULOSE 5 kN	-12.1	-12.5	-23.2	-31.6
MICROCRYST. CELLULOSE 10 kN	-12.0	-12.7	-20.5	-31.9
MICROCRYST. CELLULOSE 15 kN	-13.3	-15.0	-18.5	-30.1
MICROCRYST. CELLULOSE 20 kN	-12.3	-13.3	-20.1	-26.4
MICROCRYST. CELLULOSE 25 kN	-11.0	-11.9	-19.2	-24.5
MICROCRYST. CELLULOSE 30 kN	-11.0	-11.8	-20.1	-23.8
MICROCRYST. CELLULOSE 35 kN	-11.8	-13.0	-19.9	-22.7
MICROCRYST. CELLULOSE 40 kN	-10.5	-11.0	-21.5	-23.9
MAIZE STARCH 0 kN	-12.3	-12.9	-22.2	-31.3
MAIZE STARCH 5 kN	-10.6	-11.0	-22.9	-25.9
MAIZE STARCH 10 kN	-12.7	-13.5	-22.3	-26.1
MAIZE STARCH 15 kN	-11.7	-12.4	-21.2	-25.6
MAIZE STARCH 20 kN	-11.5	-12.3	-21.1	-25.4
MAIZE STARCH 25 kN	-11.6	-12.3	-21.7	-24.1
MAIZE STARCH 30 kN	-11.4	-12.0	-22.3	-24.3
MAIZE STARCH 35 kN	-11.3	-12.0	-21.7	-23.6
MAIZE STARCH 40 kN	-11.1	-11.7	-22.6	-24.3
MAIZE STARCH 50 kN	-11.7	-12.4	-22.1	-23.8
MAIZE STARCH 60 kN	-11.5	-12.3	-21.3	-23.2

compressing maize starch with a force of 5 kN there was an exceptional maximum in the first band power because of tightening of the powder (see table 1 and figure 5B). The sound was clearly audible and appeared every time when the machine was started, too.

Generally there were few changes on the second band (3.8 – 7.7 kHz) when the compressive force was varied. However, when compacting microcrystalline cellulose the compactor screw scratched the cylinder wall giving rise to a pair of peaks seen in figures 4B, C, D, E and F. The sounds were clearly audible, too.

The third band (7.7 – 12.85 kHz) was the most interesting band concerning the compaction. Special increasing jumps were observed in the third band power indicating possible changes in compression or compaction mechanisms. These appeared in the cases of lactose monohydrate, microcrystalline cellulose and maize starch with forces of 30, 20 and 25 kN. Lactose monohydrate and microcrystalline cellulose were better compacted with these forces but maize starch was not. The reason may be the shape and the strength of the primary powder particles. The number of the connection points between the powder particles did not become large enough under the compression. The binding could be improved by increasing the compression time so that the number of the connection points had time to increase during the plastic deformation.

It is possible to separate discrete peaks on the third band, too, carrying information on certain phenomena. For example in the case of microcrystalline cellulose there appeared a peak with forces 30, 35 and 40 kN possibly indicating the capping phenomen (the compacted product was split in two) that was observed (see fig. 4G, H and I). Discrete peaks with exact identification would make quantitative analysis of such kind of phenomena possible.

The signal-to-noise ratio could be improved by using digital recording particularly on higher frequencies. Furthermore more sensitive microphones could be probed. Peak overlapping is another problem concerning quantitative analysis. The wide frequency range of the sounds on the third band could be explained by large powder particle size distribution. Fractures of different size powder particles would produce sounds with different frequencies. Hence further acoustic emission studies could be made together with powder particle size distribution analysis. There are also harmonics of the fundamental "machine-frequencies" causing peak overlapping on the third band. The third problem concerning quantitative analysis is the fact that when increasing the compressive force the material flow through the gap between the rolls increases, too. This makes possible sound effects stronger with higher compressive forces.

ACKNOWLEDGMENTS

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