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Notes

Monitoring the acoustic activity of a pharmaceutical powder during roller compaction

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Abstract

Acoustic relaxation emissions (ARE) from microcrystalline cellulose (MC) and maize starch (MS) were detected during roller compaction using a microphone with flat frequency response up to 20 kHz and a tunable high pass filter the limiting frequency of which was set at 15 kHz. The noise from the compactor itself was found to appear mainly below 15 kHz. The ARE intensity of MC was observed to increase as a function of applied compressive force up to 45 kN, while the ARE intensity–force curve of MS had a maximum at 50 kN. A Gaussian-shaped function fitted reasonably to the data in both cases. © 1997 Elsevier Science B.V.

Keywords: Acoustic emission; Gaussian function; Lamination; Pharmaceutical powder; Roller compaction

Acoustic emission measurements are popular in metallurgy as a non-destructive testing method, but are also widely used in various fields of science. In pharmaceutical technology, only few reports have been published, despite the promising results that have been obtained (Rue et al., 1979; Rue and Barkworth, 1980; Waring et al., 1987a,b; Wong et al., 1990). Unfortunately, the experimental set-ups and instrumentations have varied so much that the reported results are not comparable.

After the first acoustic emission measurements with pharmaceutical powders using the roller compactor (Hakanen et al., 1993), hopes of achieving a novel method for monitoring of the roller compaction process have risen. The lamination of microcrystalline cellulose was indicated by an enhancement of acoustic emission in the region of about 17–23 kHz (Hakanen and Laine, 1995). In present work it will be shown that the inte-

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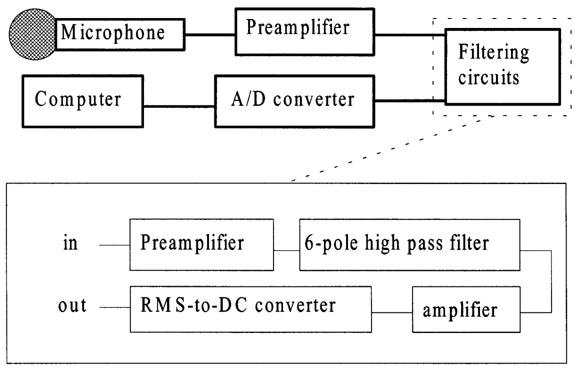

Fig. 1. The schematic experimental set-up. The preamplifier as well as the amplifier after filtering consists of one operational amplifier stage, and the high pass filter of three op-amp stage.

grated acoustic relaxation emission (ARE) as a function of compressive force is characteristic of the powder being compacted.

The compacted materials were microcrystalline cellulose (MC) (Edward Mendell, Finland Oy) and maize starch (MS) (Cerestar, Germany). MC had a particle size of about 50 μm.

The ARE signal was detected via air by a Brüel and Kjaer 4134 condenser microphone with a flat frequency response up to 20 kHz. The microphone was placed under the rollers of the compactor (Bepex Pharmapaktor 200/50P) (Hakanen et al., 1993) and the signal was amplified by a Brüel and Kjaer 2639 preamplifier and recorded by a Teac DA-P20 digital audio tape (DAT) recorder with effective sampling frequency of 48 kHz. The signal from the DAT recorder was analyzed using electronic circuit and ADC-100 A/D-converter as a data logger (100 kHz sampling).

The electronic circuit consists of a tunable preamplifier, an active high pass filter (six-pole, three op-amp) and a true RMS-to-DC converter (MAXIM AD536A). The filter was tunable and the RMS-to-DC converter was used in standard connection with external capacitor (Fig. 1).

In studies of noise produced by the roller compactor itself, it was found that the machine noise appears complete at values less than 15 kHz. Therefore, the acoustic relaxation emission of compacting powder was detected in the range 15–20 kHz using a microphone (Hakanen and Laine, 1995) with frequency response, which extends to 21 kHz (− 3 dB) and a tunable high pass filter.

It was observed that the ARE signal from compacting powder consists of short acoustic pulses with varying intensities (Fig. 2).

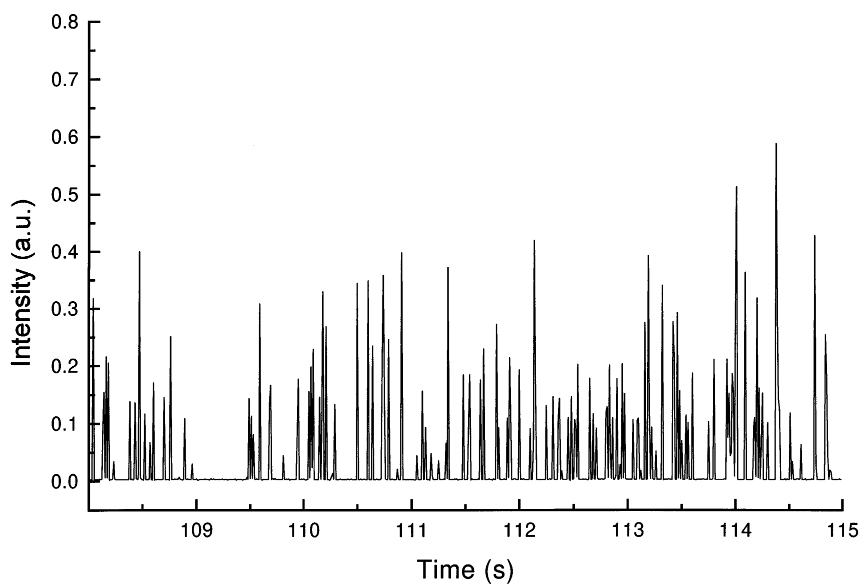

Fig. 2. A 7 s period of the ARE from maize starch during roller compaction when the compressive force is set at 50 kN.

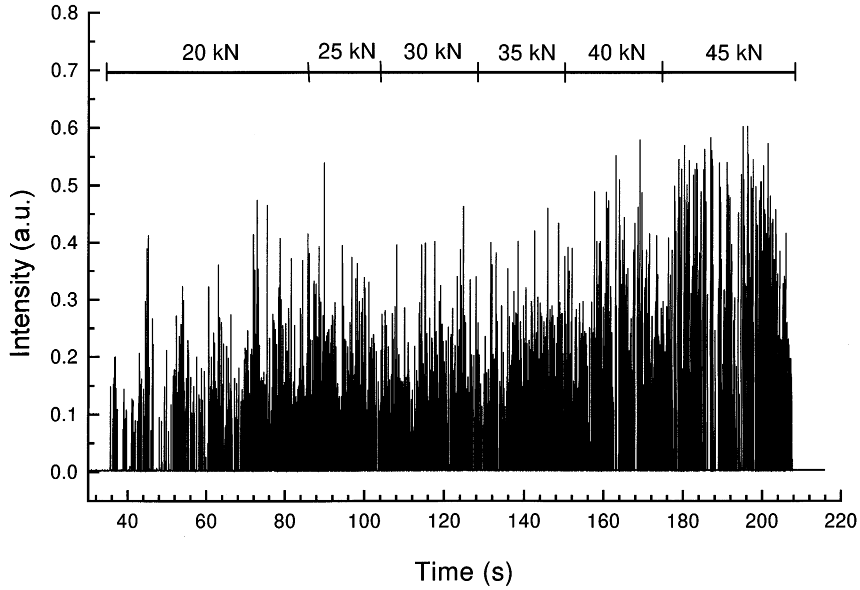

Fig. 3. The ARE from microcrystalline cellulose, when applied compressive force is altered from 15 to 45 kN. Below 15 kN, the ARE cannot be resolved from the background noise.

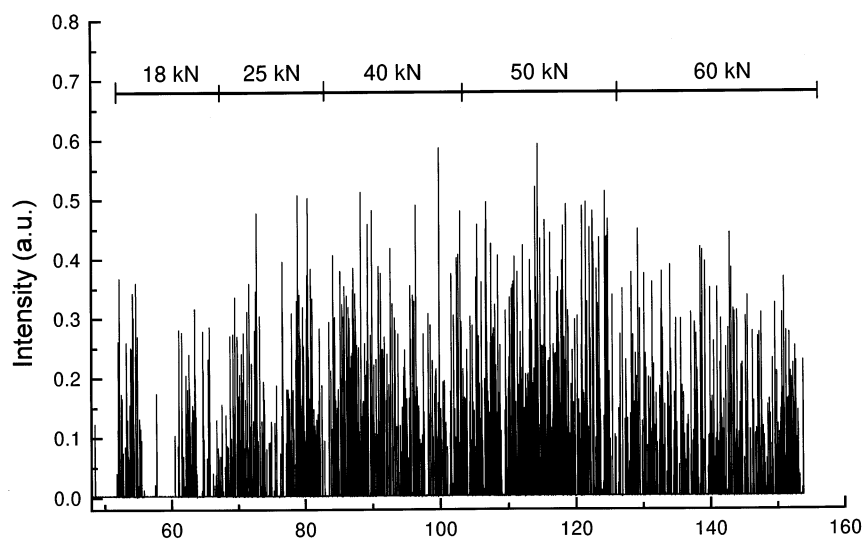

Fig. 4. The ARE from maize starch, when applied compressive force is altered from 18 to 60 kN. Below 18 kN, the ARE cannot be resolved from the background noise.

The intensity of the ARE from MC was found to increase when applied compressive force was increased (Fig. 3). The intensity of the ARE and the interval between acoustic events varied despite the constant compressive force. However, the maximum of the interval was found to be less than 1 s.

The lamination phenomenon was obtained when the applied force was increased from 35 to 40 kN. This could not be observed from the

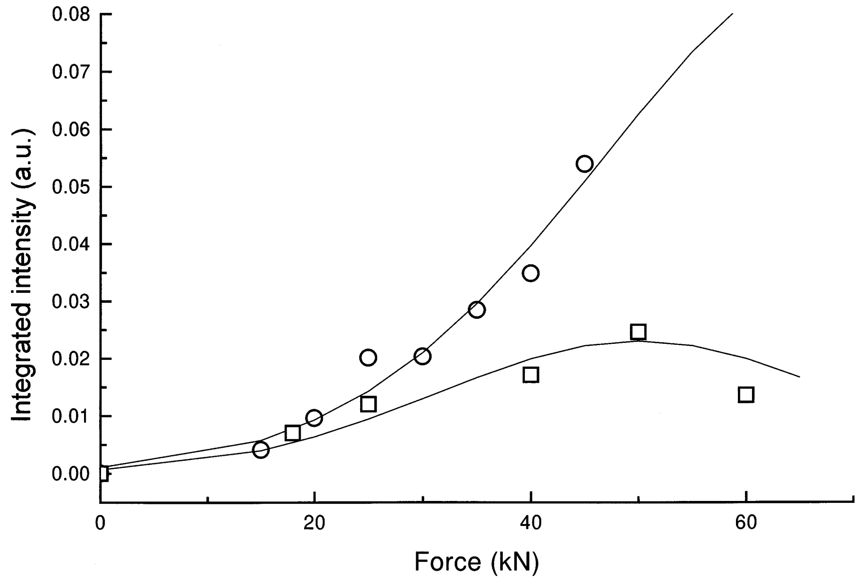

Fig. 5. The integrated ARE intensities of microcrystalline cellulose (circles) and maize starch (squares) as a function of the applied compressive force, together with fitted curves (solid lines) calculated from Eq. (1), using fitting parameters presented in Table 1.

intensity of the ARE. Despite the lamination, MC was easily compacted and its compressibility was good; it does not stick in rollers causing variation in applied force as, for example, lactose does.

The ARE intensity from MS was also found to increase when applied force was increased, but the intensity was lower than in the case of MC. Also MS was quite easily compacted, but it broke up into pieces without lamination, immediately after compacting. The maximum ARE intensity of the MS was obtained at 50 kN and after that the intensity started to decrease (Fig. 4).

To obtain a value for ARE intensity, a 7 s period of the signal was integrated (see Fig. 2). Using these values, the ARE intensities of MC and MS were plotted as a function of the compressive force (Fig. 5).

Table 1
Fitting parameters of the curves in Fig. 5

	A	α	w
MC	0.09	70	−1100
MS	0.023	50	−700

We introduce a Gaussian-shaped function for the distribution of integrated intensity I, which fits reasonably to the data (solid lines in Fig. 5):

$$I = A \exp \frac{(F-\alpha)^2}{w} \qquad (1)$$

where F = applied force and A, α, w are fitting parameters, which are presented in Table 1. The parameter A describes the maximum value of integrated ARE and $\alpha = F$ when the maximum integrated ARE are achieved. The parameter w is associated with the width of the curve.

The observed acoustic characteristics could be due to different strength and elasticity properties of the powder particles in question. While MC has a Young's modulus value of $E = 7.1-10.3$ GPa, the value for MS is only $E = 3.7$ GPa, which is more than 2 times lower. The direct relationship between the cohesive energy density (CED) and Young's modulus has been reported by (Rowe and Roberts, 1996). On the other hand, CED might be related to ARE and clarify a certain parallel between ARE and Young's modulus. However, this requires further investigation to ensure elucidation.

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