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Acoustic emission of pharmaceutical powders during compaction

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ABSTRACT: acoustic emission measurements aim at understanding and distinguishing all the phenomena that occur during powder compaction of pharmaceutical products. Those phenomena are granular rearrangement, fragmentation and visco-plastic deformation of grains or granules. Other considerations such as specific area and porosity evolutions versus compaction pressure provide valuable data on domains where whether fragmentation or visco-plastic deformation could be predominant. Relationships between acoustic emissions and such phenomena occurring during compaction of pharmaceutical powders are proposed. For instance, compaction of brittle products like aspirin or saccharose produces strong acoustic emission whereas compaction of starch exhibiting a high visco-plastic behaviour produces a low acoustic emission.

1 INTRODUCTION

This work aims at describing an apparatus detecting accurately waves emitted during powder compaction in order to establish relationships between this acoustic emission (A.E.) and phenomena occurring during compaction.

Several studies had been already driven on A.E. during pharmaceutical powder "shaping" with industrial devices [1] or during compaction [2,3]. None of these studies allows to clearly distinguish between the different phenomena that occur during compaction itself. For that purpose, it appears that complementary methods, like specific surface area and porosity measurements, would be helpful.

In order to understand and propose mechanisms involved in powder compaction, an experimental A.E study has been developed and results compared with those of complementary methods.

2 EXPERIMENTAL DEVICE AND PROCEDURE

2.1 *A.E. detection system on an uniaxial die*

A piezoelectic sensor has been placed on the outer wall of the die in order to record A.E. during compaction. This device is shown in Figure 1.

An oscilloscope placed in parallel of the counter

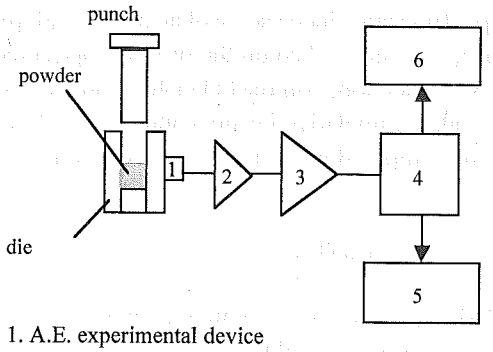

Figure 1. A.E. experimental device

1 : piezoelectric sensor (µ30 Euro physical acoustic)
2 : preamplifier (gain 40 db) +band-pass filter 200 à 300 kHz(Brüel et Krael model 2637)
3 : Amplifier (gain 30 db) + high-band filter (0.1 à 2MHz) (Brüel et Krael model 2638)
4 : Events counter
5 : verification by oscilloscope
6 : Data acquisition

the bursts or the augmentation of background noise or even the lack of true A.E. In this study, oscilloscope data are not interpreted at all.

The instrumented press is linked to a computer, the brand is LLOYD and the model LR 50 K. The maximal strength applied in this press is 50 kN. The cylindrical die presents an inner diameter of 8 mm and an outer diameter of 54 mm. The inner wall of the die is always lubricated with an ethanol solution

of magnesium salt of stearic acid at a concentration of $5 g.L^{-1}$.

2.2 *Experimental procedure*

We always follow the same experimental procedure. After putting the powder inside the die (0.25 ± 0.05 g of powders settled by small hurts on the die wall), the sensor is placed on the die. A grease guarantees a material continuum between the plane sensor and the cylindrical die. We have observed that the results depend on the position of the sensor on the die. That's why the sensor is always placed on the same position, at half way-up of the initial settled powder.

The sensor signal is registered on a counter analysing different frequencies. After preliminary tests, we choose to study signals of frequencies between 0.1 et 2 MHz. In fact, whereas low frequency signals (under 0.1 MHz) are not workable, the high-frequencies ones selected are suitable for most of the pharmaceutical products used.

The compaction and A.E. registration procedures are simultaneously started. During all the compaction steps (pressure increase, isobar stage, and pressure drop), A.E. is checked on the oscilloscope screen.

After ejection, compacts height is about 4 mm. At the end, a cumulative events count versus time curve can be compared with axial stress versus time.

3 EXPERIMENTAL RESULTS

All these tests allow a count of cumulative events, responsible for A.E. during powder compaction.

During powder compaction, several phenomena can generate A.E:

- punch / die wall, grain / wall, or grain / grain frictions
- grains or granules fragmentation
- plastic deformation of grains
- press deformation

The die used is always lubricated, which minimises A.E. of frictions onto the wall in comparison with A.E. of grain/grain friction. Furthermore, before starting the tests, we check that the press external environment does not generate A.E. So only grain/grain frictions, grain fragmentation and plastic deformation can constitute the A.E. events.

Now, let us try to see if all these phenomena can be distinguished with A.E. during compaction of pharmaceutical powders.

3.1 *Work on pure products*

3.1.1 *Work on aspirin AC 360*

Aspirin AC 360 is a crystallised aspirin given by Rhône Poulenc Rorer. The grain size is 360 µm ± 100 µm and the grains look like sticks.

A.E. tests are realised with the same following compaction procedure. The maximum pressure (300 Mpa) is reached with a punch velocity of $1 mm.min^{-1}$, this pressure is hold during 1 min, then pressure drop is carried out with a punch velocity $10 mm.min^{-1}$.

The curves of cumulative event count and axial stress as a function of time are shown in Figure 2.

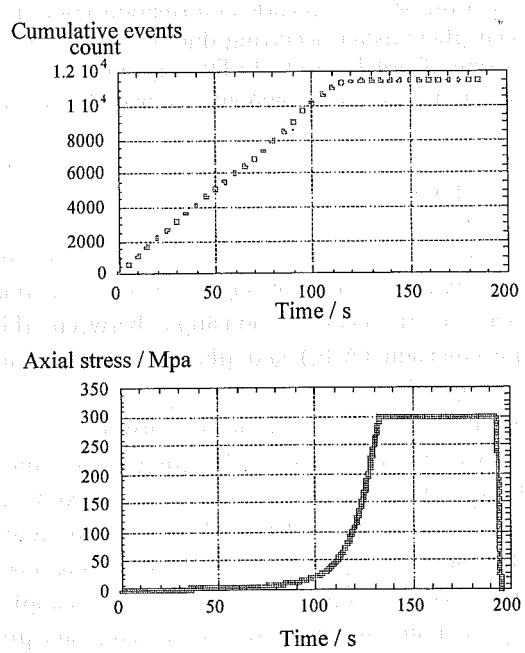

Figure 2. cumulative events counts and axial stress versus time for aspirin AC 360

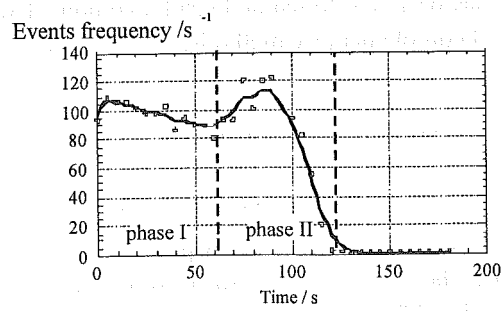

Figure 3. Events frequency versus time

To strengthen these results we study the cumulative event counts versus time derivative. The curve obtained (event frequency versus time) allows us to precise A.E. evolution. According to this curve represented in Figure 3, the A.E. can be divided into two parts.

In the first part (phase I, 0<t<60 s, 0<P<5 MPa), the number of bursts seen is much greater than in the second one (phase II, 60<t<120 s, 5<P<80 MPa).

Moreover, the bursts look like different. In the first part, each burst includes a great number of events, with signals of high intensity. The events 'seen' by the counter are certainly underestimated and an energy sensitive detector would give better information. The event counting in the second part is much satisfactorily evaluated because the signal intensity is smaller and the event frequency more representative.

In the first part, as the axial stress is lower than 5 MPa, the signal probably corresponds to grain rearrangements, and may be some few grain fragmentation. This can be explained by the fact that when the punch moves, rearrangements of grains may easily take place in a high porosity packing, emphasised by some fragmentation. In the same time, a lot of big bursts are seen on the screen of the oscilloscope.

In the second part, A.E. is attributed mainly to grain fragmentation and the subsequent small local rearrangements of grain pieces .

On Figure 4, are reported the curves of specific area and porosity as a function of pressure, for the aspirin AC.

The specific surface area curve presents a maximum located at 60 MPa. The two phenomena that could increase the specific area of compacts is grain crack during tablet compaction or cracks generated during the ejection. We have considered that inter-granular or intra granular cracks might occur during compact

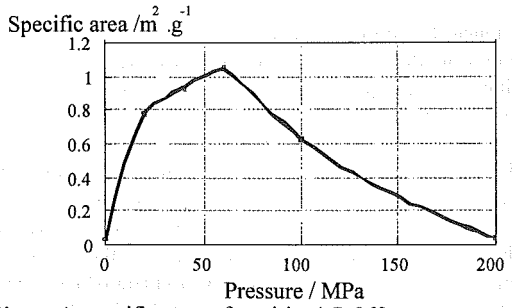

Figure 4. specific area of aspirin AC 360 versus compaction pressure

ejection. Nevertheless, it seems that these phenomena do not occur for the pharmaceutical products used in this work (normal evolution of mechanical properties with pressure, no elastic recovery during ejection).

So, we can say that fragmentation during compaction mainly occurs until 60 Mpa, and is responsible for the A.E. registered in the same period.

Furthermore, let us consider the relaxed porosity evolution shown in Figure 5: from 100 MPa to 500 Mpa, the porosity, as specific area, is always decreasing.

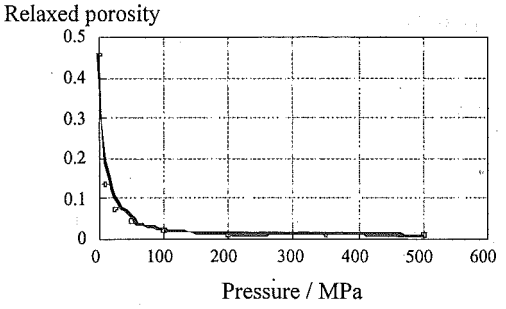

Figure 5. relaxed porosity of aspirin AC 360 versus compaction pressure

The main basic phenomena are plastic or viscoplastic deformations in this region. These deformations are responsible for interface grain welding leading to specific area fall after a compaction pressure of 60 MPa. So for the aspirin under study, this kind of deformations are real, but not detected by A.E.

For this first work on aspirin AC 360 A.E. during compaction, it can be concluded that grain fragmentation and the consecutive grain rearrangements are characterised by A.E. emitted during compaction. This A.E. is represented by several bursts on the oscilloscope screen and a creasing curve of cumulative events counts versus time. Furthermore, viscoplastic deformations of aspirin grains are accompanied of a very weak A.E..

3.1.2 *Work on corn starch*

Now we will study A.E. of another pharmaceutical material: the corn starch, is an excipient which is generally considered as not brittle. Two kinds of starch are used, both provided by "Roquette frères" a standard starch (CS) and a pregelatinised one (PGS).

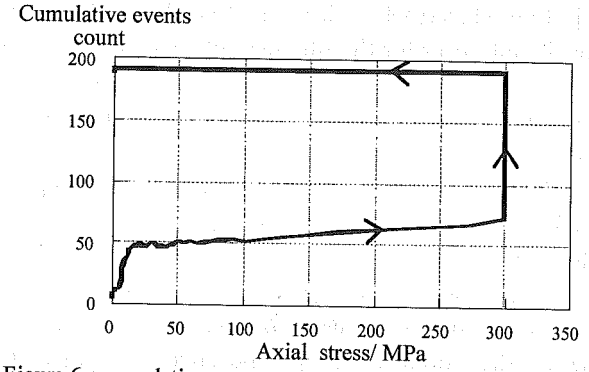

Figure 6 : cumulative event counts versus axial stress for Corn Starch

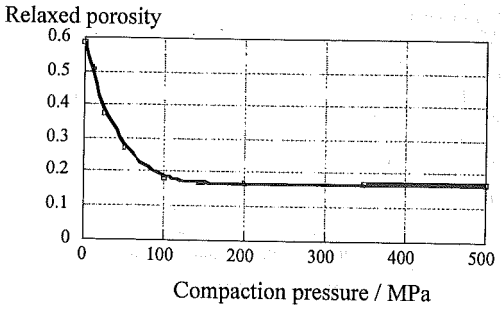

Figure 7. Corn Starch relaxed porosity versus compaction pressure

Compaction procedure is the same as for aspirin. The results are shown on the curves in Figure 6. Only are shown the results for CS, because those for PGS are nearly the same, notwithstanding important feature differences in the solid structure.

First of all, the main difference observed between A.E. of aspirin and starch is that the number of events is far greater for aspirin (hundred time greater for aspirin). Furthermore no burst can be seen on the oscilloscope screen for starch.

Another difference must be also emphasised: for starch, A.E. is detected after an axial stress equal to 150 Mpa, and also during the isobar stage, whereas A.E. of aspirin is registered until 100 MPa. This small continuous A.E. without burst may be due to the visco-plasticity or elasticity of corn starch. These starch characteristics can be deduced from the curve shown in Figure 7 and 8.

First, the relaxed porosity of starch compacts is constant for compaction pressure upper than 150 MPa while the specific area is always decreasing. These results are the consequence of the viscous, plastic and elastic character of starch compacts.

So either elasticity or visco-plasticity of starch are attributed to the small A.E., seen as a background noise augmentation.

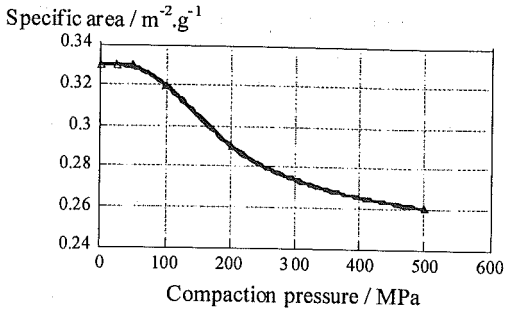

Figure 8. Corn Starch specific surface area versus compaction pressure

4. CONCLUSION

Either grain rearrangement or grain fragmentation are responsible for important A.E., accompanied with numerous bursts on the oscilloscope screen, and an increasing curve of cumulative event counts as a function of time. Furthermore, visco-plastic deformations or elasticity generate a weak A.E. (small augmentation of background noise). A.E. analysis study can be used to distinguish the physical phenomena that occur during pharmaceutical powder compaction. To deepen this work, an analyse of the bursts observed on the oscilloscope screen, and the study of the answers at different frequencies might be interesting. Such inquiries would be useful to characterise the phenomena occurring during powder compaction by giving an A.E. signature for each of them.

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