ELSEVIER

International Journal of Pharmaceutics 220 (2001) 85–90

international journal of pharmaceutics

www.elsevier.com/locate/ijpharm

The stimulated acoustic relaxation emission of maize starch tablets

V. Mellin, J. Salonen, E. Laine *

University of Turku, Department of Physics, FIN-20014 Turku, Finland

Received 18 August 2000; accepted 12 March 2001

Abstract

Stimulated acoustic relaxation emissions from maize starch tablets after compression were detected and recorded in the audible region. Stimulation was found to enhance the detected acoustic emission and to maintain recordable acoustic emission level longer. Based on the fact, that the stimulation by a halogen lamp and a preheated chamber produced similar results, the effect of stimulation on acoustic relaxation emission is proposed to be connected to the temperature rather than the visible emission. A mathematical model of acoustic activity and its dependence on stimulation temperature is also introduced.

Keywords: Acoustic emission; Maize starch; Relaxation; Stimulation

1. Introduction

In a compaction process of pharmaceutical materials, elastic energy is stored in the compact, causing stress. If the stress is greater than the shear strength of the compact, a crack will be initiated and, unless rapid local plastic flow occurs, the crack may propagate across the compact, resulting in capping or lamination (Rue et al., 1979). Deformation and fracture processes release energy by elastic waves, or acoustic emission, and it has been found that the stages of compression cycle exhibit different types of acoustic activity in the ultrasound region (Waring et al., 1987a,b). Previous studies, made with a roller compactor and a single-punch tablet machine, have shown that stress relaxation phenomena may be detected by transient broad frequency band acoustic emissions also in the audible region (Hakanen et al., 1993; Hakanen and Laine, 1995; Salonen et al., 1997).

Elastic energy may be released by time-dependent relaxation resulting in increase of tablet crushing strength. The increase in crushing strength of sodium chloride tablets has been found to correlate with the acoustic emission monitored after compression (Rue and Barkworth, 1980). This suggests that the mechanism of strength increase and acoustic emission have a common origin in deformation processes.

* Corresponding author. Tel.: +358-2-3335673; fax: +358-2-3335993.

E-mail address: ensio.laineutu.fi (E. Laine).

0378-5173/01/$ - see front matter
PII: S0378-5173(01)00648-2

Energy relaxation is difficult to control, making the storage behaviour of compacts virtually unpredictable using present methods. Because time-dependent deformation decreases exponentially by time it is essential that acoustic emission measurements be done as quickly as possible after compression. In present work we will show that stimulation can be used to enhance acoustic relaxation emission (ARE) and to maintain recordable ARE level longer. In order to do this, maize starch tablets have been stimulated by halogen tungsten lamp or by a preheated measurement chamber and ARE-levels have been recorded. The effect of stimulation power on ARE has been studied by using halogen lamps of various nominal powers for the stimulation.

2. Materials and methods

The tablets were compressed of maize starch powder with mean particle diameter of 10 μm (standard deviation 2.3 μm), by a hydraulic compression apparatus with a single 20 mm diameter punch or by an eccentric tablet machine (DIAF T.M.D. 30) with 13 mm flat-faced punches. The ARE signal from the hydraulically compressed tablets was recorded in a chamber consisting of acoustically separated sample and reference parts and a halogen lamp for the stimulation (Fig. 1(a)). The tablets were placed in the chamber along with the die after compression. The tablets compressed in the tablet machine were placed in a measurement set-up with a preheated measurement chamber (Fig. 1(b)) after compression. To distinguish between the ARE signal and the outside noise, signals from both sample and reference parts were recorded.

The ARE signal was detected by a Brüel and Kjær 4134 condenser microphone with a flat frequency response up to 20 kHz, amplified by a Brüel and Kjær 2639 preamplifier and then recorded by a Teac DA-P20 digital audio tape (DAT) recorder. The signal was analysed using digital audio editor software.

3. Results and discussion

We assume the elastic energy relaxation, and consequently the ARE to obey the simple linear differential equation:

$$\frac{dA}{dt} = -kA$$

where $A(t)$ is the acoustic activity and k is the specific rate constant. This equation has a solution:

$$A(t) = A_0 e^{-kt} \quad (1)$$

where $A_0 = A(0)$. Thus, the cumulative acoustic relaxation emission (CARE) P is ($P(0) = 0$):

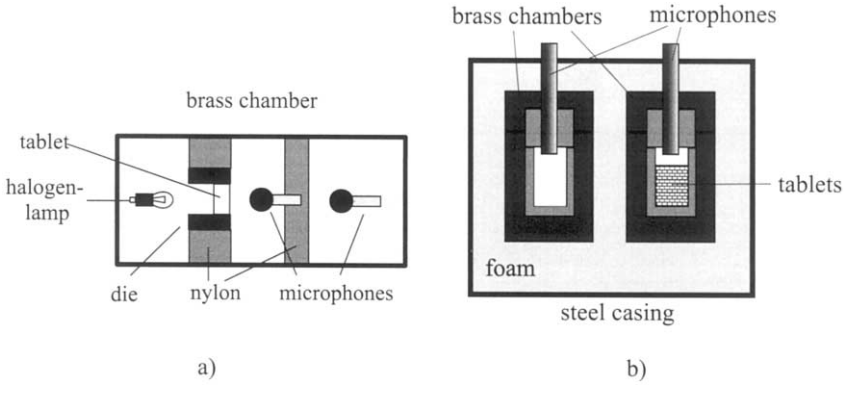

Fig. 1. The measurement set-ups of the hydraulically compressed tablets (a) and the tablets manufactured with the eccentric tablet machine (b).

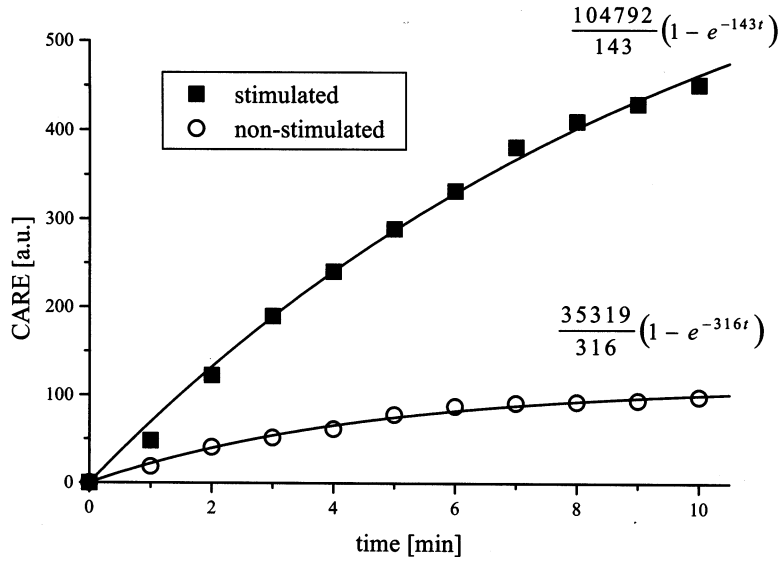

Fig. 2. The cumulative acoustic relaxation emission (CARE) of the stimulated and the non-stimulated maize starch tablets and solid fitting curves. The tablets are compressed hydraulically at 300 MPa and the stimulation is started at $t = 0$.

$$P(t) = \int A(t)\mathrm{d}t = \int A_0 e^{-kt}\mathrm{d}t = \frac{A_0}{k}(1 - e^{-kt}) \qquad (2)$$

Fig. 2 presents CARE measured from maize starch tablets compressed by the hydraulic compression apparatus at 300 MPa and stimulated by a 35 W halogen lamp. The solid lines in Fig. 2 represent fitting curves. We find the acoustic activity to increase nearly threefold and the rate constant to reduce to less than a half as a result of the stimulation.

To study the effect of stimulation on acoustic activity maize starch tablets were compressed by the hydraulic apparatus and stimulated by halogen lamps of 10 W, 20 and 35 W nominal powers. The temperature T in the measurement chamber is:

$$T(t) = \left\{ \begin{array}{ll} T_0, & t \le t_0 \\ T_0 + \dfrac{C}{b}(1 - e^{-b(t-t_0)}), & t > t_0 \end{array} \right\}, \qquad (3)$$

where T_0 is the room temperature, t_0 is the time at which the stimulation is started, C is a stimulation-power-dependent constant and b is dependent on the measurement chamber's thermal conductivity. Using the exponential dependence of CARE at $t = 10$ min ($\mathrm{CARE}_{10\ \mathrm{min}}$) on the temperature in the chamber at the same time (T_{10} min) (Fig. 3) we assumed that both A_0 and k depend exponentially on temperature. Therefore, we replaced A_0 and k in equation 2 with:

$$A_{\mathrm{T}}(T) = A_0 \exp\left(\frac{T - T_0}{d}\right) \qquad (4)$$

$$k_{\mathrm{T}}(T) = k \exp\left(1 - \frac{T}{T_0}\right),$$

where d is a fitting parameter. Using Equations 2–4, we get:

$$A(t) = \left\{ \begin{array}{ll} A_0 \exp[-k_0 t], & t \le t_0 \\ A_0 \exp\left[\dfrac{C}{bd}(1 - e^{-b(t-t_0)}) - kt \exp\left(-\dfrac{C}{bT_0}(1 - e^{-b(t-t_0)})\right)\right], & t > t_0 \end{array} \right\}. \qquad (5)$$

Fig. 4 shows cumulative acoustic relaxation (CARE) measured and solid fitting curves, which represent Riemann sum of equation 5 in 1 s intervals. The difference between fitting curves and measured values of CARE may be due to the

effect of temperature gradient in the tablet during stimulation on relaxation. Also, the temperature profiles in the chamber during stimulation were measured in the chamber, not in the tablet, and energy absorption to the tablet and to the chamber may differ.

Fig. 5 presents cumulative acoustic events measured from maize starch tablets that were compressed by the eccentric tablet machine and after compression placed in the measurement chamber preheated to 60°C. In this case the temperature in the measurement chamber by time is:

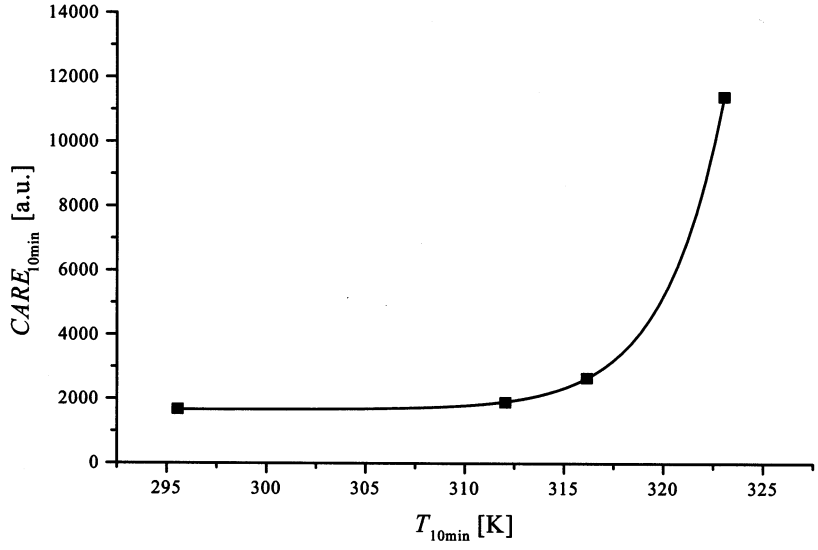

Fig. 3. The cumulative acoustic relaxation emission (CARE) of maize starch tablets stimulated by halogen lamps of 10, 20 and 35 nominal powers, respectively. Datasets are normalized at $t = 2$ min at which time the stimulation is started and solid lines are fitting curves.

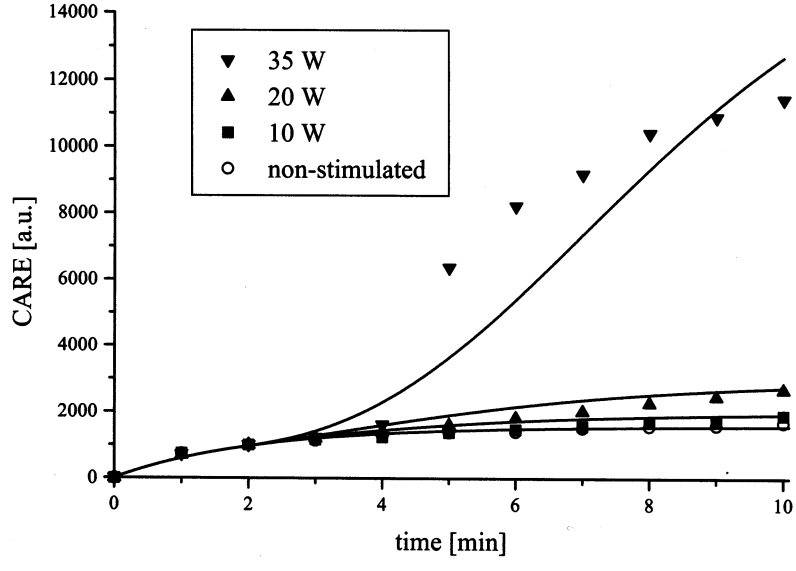

Fig. 4. Cumulative acoustic relaxation emission at $t = 10$ min ($CARE_{10\ min}$) by temperature at the same time ($T_{10\ min}$).

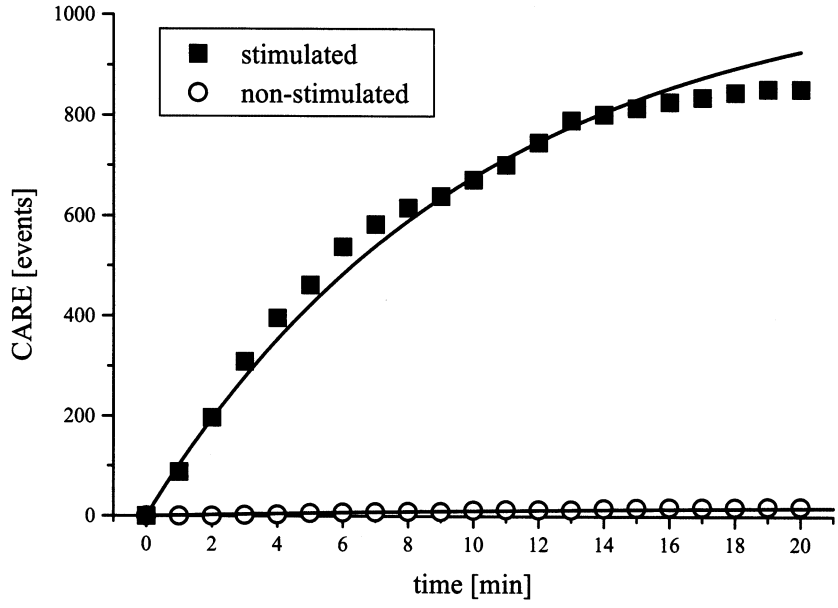

Fig. 5. Cumulative acoustic relaxation emission (CARE) of maize starch tablets compressed by eccentric tablet machine. Stimulation by a preheated measurement chamber.

$$T(t) = T_0 + T_1 e^{-bt}, \quad (6)$$

where temperature in the chamber at $t = 0$ is $T_0 + T_1$. Thus, the acoustic activity is:

$$A(t) = A_0 \exp\left[\frac{T_1}{d} e^{-bt} - kt \exp\left(-\frac{T_1}{T_0} e^{-bt}\right)\right] \quad (7)$$

The solid lines in Fig. 5 represent fitting curves calculated from equation 7, again by Riemann sum method in 1 s intervals. The non-stimulated tablets show little acoustic activity whereas the stimulated tablets show considerable activity. This demonstrates the importance of stimulation in acoustic relaxation studies.

4. Conclusions

While studying acoustic relaxation emission (ARE) of maize starch tablets, we found that stimulation by halogen lamps and by a preheated chamber can be used to enhance acoustic activity and to maintain recordable ARE level longer. The fact that stimulation by a halogen lamp and by a preheated chamber produce similar results indicates that the effect of stimulation on ARE is connected to the temperature rather than the visible emission of halogen lamps. We also introduce a mathematical model of the effect of stimulation temperature on acoustic activity and on the rate constant of ARE.

References

Hakanen, A., Laine, E., 1995. Acoustic characterization of a microcrystalline cellulose powder during and after its compression. Drug Dev. Ind. Pharm. 21, 1573–1582.

Hakanen, A., Laine, E., Jalonen, H., Linsaari, K., Jokinen, J., 1993. Acoustic emission during powder compaction and its frequency spectral analysis. Drug Dev. Ind. Pharm. 19, 2539–2560.

Rue, P.J., Barkworth, P.M.R., 1980. The mechanism of time-dependent strength increase of sodium chloride tablets. Int. J. Pharm. Tech. Prod. Mfr 1, 2–3.

Rue, P.J., Barkworth, P.M.R., Ridgway-Watt, P., Rough, P., Sharland, D.C., Seager, H., Fisher, H., 1979. Analysis of tablet fracture during tabletting by acoustic emission techniques. Int. J. Pharm. Tech. Prod. Mfr 1, 2–5.

Salonen, J., Salmi, K., Hakanen, A., Laine, E., Linsaari, K., 1997. Monitoring the acoustic activity of a pharmaceutical powder during roller compaction. Int. J. Pharm. 153, 257–261.

Waring, M.J., Rubinstein, M.H., Howard, J.R., 1987a. Acous

tic emission of pharmaceutical materials during compression. Int. J. Pharm. 36, 29–36.
Waring, M.J., Rubinstein, M.H., Howard, J.R., 1987b. Acoustic emission of pharmaceutical materials: the effect of compression speed, ejection, lubrication and tablet weight. Int. J. Pharm. 40, 15–22.