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The rheology of wet powders: a measuring instrument, the compresso-rheometer

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Abstract

In order to control the mechanical properties and granulation processes of wet powders, steps were taken to design and develop a measuring instrument, the compresso-rheometer. Initial experiments were carried out on binary associations between microcrystalline cellulose powder and varying quantities of water.

Keywords: Wet powders; Rheology; Granulation; Extrusion; Compresso-rheometer

This work is based on the granulation processes which are required to optimise the physical characteristics of a material and its applications to pellets, developed by our team (Bataille et al., 1990; Sonaglio et al., 1995). Granulation processes consist of two steps. In the first step, one, or several, pulverulent raw materials are associated with a liquid in preset quantities (Malinowski and Smith, 1975). The second step of the process generates pressure forces that macroscopically bring the material into a more homogenous state, so as to shape it during material flow through a screen (standard granulation) or a perforated plate (extrusion). The addition of a liquid to individual powder particles gives unity to the mixture through evidence of interparticle cohesive properties, which lead to the production of a new material, which is able to change in form and able to flow. In this case, this new material falls under the definition of a fluid (Comolet, 1990).

This three-phase state is composed of a base, which is solid (powder particles), a liquid, corresponding to the liquid added during the wetting of the powders, and lastly a gaseous fluid, corresponding to air imprisoned at the time of mixing. This material is defined as an unsaturated porous state, as compared to the fluid reference phase being the added liquid (Coussy, 1991). Due to the effect of shaping stresses, the different fluids, which saturate interstitial space, can be moved. The gaseous content decreases due the combined effects of shear and compressive forces. This in

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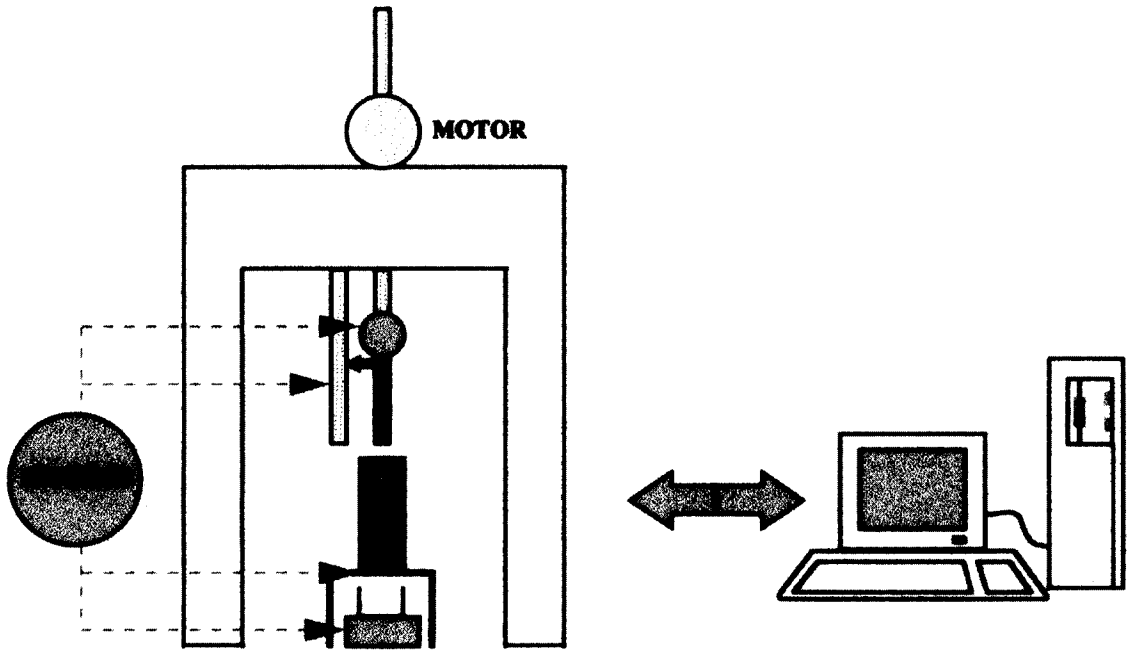

Fig. 1. Schematic representation of the compresso-rheometer.

turn leads to density modification. The complex character of these materials, which must show cohesive, deformation, flow, and even rupture characteristics, explains our interest in developing an instrument to study them.

Relying on the model proposed by Harrison et al., 1985, and also on the theory of capillary rheometers (Couarraze and Grossiord, 1991), the compresso-rheometer was conceived in order to understand the reactions of humid powders when they are submitted to variable and well-defined mechanical stresses, bearing in mind that rheometers on the existing market do not all present the characteristics needed to study these materials.

Its originality is based on the following; its dual functioning mode, flow measurement, measurement of compressibility and relaxation, the continuous monitoring of temperature at capillary level, and the recording of extruded mass weight.

The compresso-rheometer is made of three parts (Fig. 1). The motorized frame structure accomodates the measuring-cell in its centre and serves as a support to a motoreductor, which is capable of exerting a tractive or compressive effort at varying speeds (0.5–5.5 mm s^{-1}).

The measuring-cell is made of a piston which enters a barrel with a corresponding diameter. At the base of the barrel, it is possible to place either a plate with a small diameter orifice (die), or an unperforated plate. This ensures that the base of the barrel can be shut so that compressibility/relaxation tests can be carried out. The measuring-cell uses two strain-gauged force sensors. One measures the resistance exerted by the material on the piston. The second allows extrusion rate determination by continuous weighing of a timed extruded mass. A potentiometric displacement sensor measures, at any time, the position of the piston, and so the precise volume of sample in the barrel. The material density can therefore be calculated. A temperature sensor continuously records the temperature at capillary level.

A data acquisition program using an E/S analogical-digital card and Labview (©1992–1993 National Instruments Corporation) software have allowed the development of a database program and the piloting of the motoreductor. Excel (V.4.0 ©1985–1992 Microsoft Corporation) software was used to process the data. The majority of this

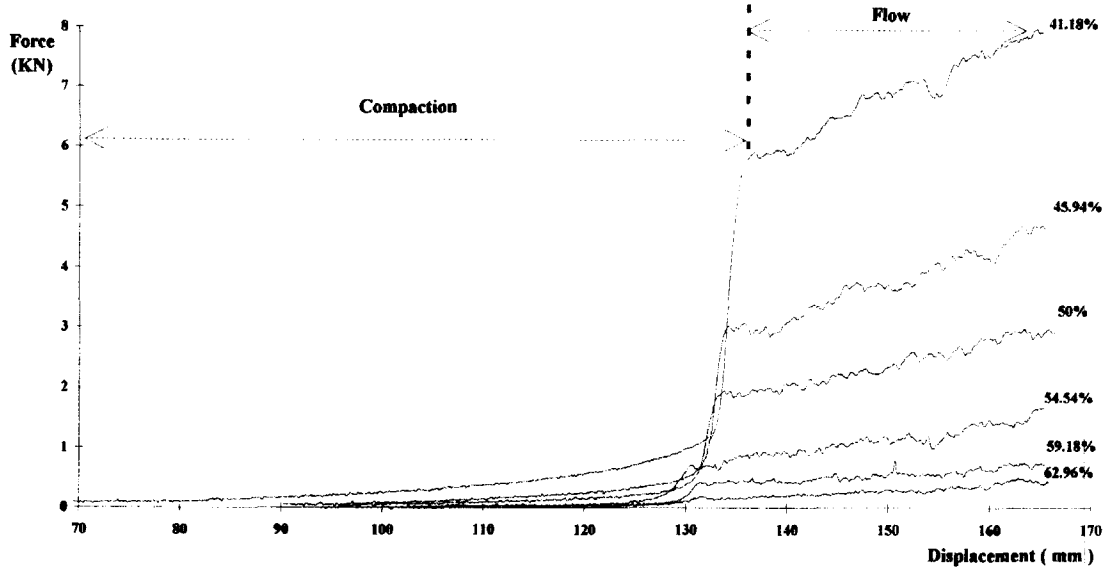

Fig. 2. Flow measurements of Avicel with varying quantities of water.

work was carried out with a microcomputer compatible PC486/66 MHz.

The first experiments used microcrystalline cellulose (Avicel PH 101, FMC Corporation, Philadelphia, USA), which is commonly used in extrusion/spheronisation processes. It was mixed with varying quantities of demineralized water (41–62% w/w). These values were arbitrarily chosen to cover a wide range of behaviour, including a mixture presenting a visually dry aspect, i.e., dusty, but whose flow is possible as an overwetted mass. Mixtures (powder—water) were prepared by mixing for 5 min in a planetary mixer, after which 35 g were taken and placed in the barrel of the compresso-rheometer.

– *Flow measurement*: The die had a diameter of 2 mm and a length of 5 mm. The force exerted by the wet mass on the piston was recorded relative to its displacement in the barrel. The curves obtained (Fig. 2)) show an approximately equivalent profile for the different moisture contents studied. On the other hand, the amplitude of force recorded at the time of flow, as well as the manner in which this force evolved during flow, allowed discrimination between the different mixtures, and also between mixtures containing only small differences in water percentages; this shows just how precisely measurements can be made. As far as curve profiles are concerned, three stages can be observed. In the first stage, weak forces were recorded; this corresponded to the phase of compaction during which the porous volume decreased. This phenomenon offered little resistance to piston advance. One then sees a very rapid change of slope between 130 and 135 mm, according to the cellulose moisture content. This period, during which stress increased far more rapidly than sinking did, corresponded to a phase where the material strongly resisted compaction (critical density). This phase is very rapidly followed by a flow-induced change of slope. The curves then obtained are, for raised moisture levels, parallel to the axis of abscissas, while for weaker moisture contents, a rising curve was recorded during flow.

The study of the forces which developed at different levels of a curve, whether or not a permanent flow plateau was obtained, and the

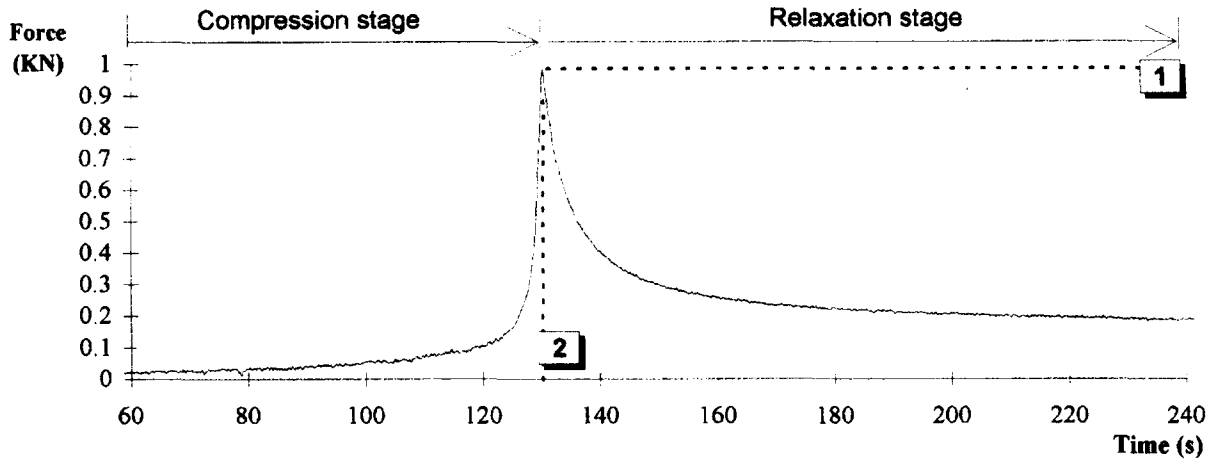

Fig. 3. Compressibility and relaxation measurements of wet Avicel at 54.54%: (1) ideal elastic body behaviour; and (2) pure viscous liquid behaviour.

establishment of relationships between extrusion rate and recorded force for a die of variable geometry, are all values which allow the establishment of a behaviour index for a given material.

– *Compressibility and relaxation tests*: These tests were carried out on the 54.54% w/w hydrated mixture (120 g of water to 100 g of dry product), which corresponded to an experimentally observed extrusion/spheronization optimum (O'Connor, 1983). The force was recorded with respect to time (Fig. 3)). The first part of this curve (compression phase) provides us with information on the density variation in relation to pressure, and allows us to determine the threshold beyond which the material can be looked upon as homogenous (Le Roux, 1993). The phenomenon of stress relaxation was studied in the second part of the curve (relaxation phase), during which the piston was in contact with the mixture, until one obtained an identical compaction to that observed in initial flow during the preceeding experiment (132mm for 54.54% w/w). The recorded force decreased, as time passed, under constant strain. This measurement allows the study of the viscoelastic properties of materials, and so enables mixture assessment. The behaviour of wet Avicel PH 101 at 54.54% w/w is halfway between the ideal elastic body and the pure viscous liquid states. This mixture undergoes flow in addition to recoverable elastic deformation.

Elastic materials convert all mechanical work into potential energy. Elasticity is reversible deformation and, in this case, the recorded force did not decrease during the period of relaxation (Michaud, 1987; Launay, 1991). On the other hand, for a pure viscous material, all the work provided was dissipated in the form of internal viscous rubbing (Rime and Doelker, 1993). This means that mechanical work was converted to heat and the recorded force fell instantly through imposed strain.

Viscoelastic materials exhibit both irreversible flow and elasticity (Schoff, 1990). They were studied by recording the areas under the relaxation curves with respect to time. These measurements were then used to express the time-dependent behaviour of a wet powder mass.

The objective of the compresso-rheometer is to establish correlations between the physical and chemical characteristics of raw materials, the observed rheological behaviour, and the quality of the finished product. It allows the control of the mechanical characteristics of wet powders.

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