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LACTOSE TEXTURE MONITORING DURING COMPACTING.

II. DETAILED TEXTURAL ANALYSIS IN PORE GROUPS

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SUMMARY

Study of the texture of lactose compacts under various confinement pressures (100, 300 and 600 bar) was carried out by pore group analysis. Awareness of the groups of large pores, the number of which is reduced in response to pressure to yield more, smaller pores which are therefore increased in number and the change in the pore profile in terms of volume, diameter and length has made it possible to obtain a better understanding of the mechanisms of densification and cohesion acquisition in terms of lactose type and compression parameters.

The authors distinguish between:
-substances which resist rearrangement and acquire cohesion whilst maintaining a high degree of porosity,
- substances which increase in density by rearrangement and then fragmentation which is accompanied by a significant increase in cohesion,
- substances the structure of which collapses, by fragmentation, multiplying contact points, but in which the energy cannot be converted to sufficient cohesion.

INTRODUCTION

In a previous study (1), the texture of lactose compacts was investigated by mercury porosimetry. Distinctions were drawn between the materials investigated in this study on the basis of the interparticular pore volume and the total length of the theoretical pores calculated from the surface area. These porosimetry data were used to analyse the data for the mechanical properties of the lactose obtained by a study of resistance to simple compression. A technological classification was proposed and it was possible to interpret the mechanisms of densification and cohesion acquisition. In order to obtain further information about these interpretations, which are based on global porosity values, an analysis in terms of pore groups has been undertaken.

MATERIALS AND METHODS

1-1 Materials

Eight lactoses were selected, whose physico-chemical, rheological, mechanical and textural (1-2) properties had already been investigated:

- lactose Fast Flo	FF
- lactose DCL 11	DCL 11
- lactose DCL 21	DCL 21
- tablettose	Tab
- lactose Extra Fin Cristaux	EFC (Extra-Fine Crystals)
- lactose Merck	M
- lactose Poudre Fine	PF (Fine Powder)
- lactose Impalpable	PI

1-2- Methods

1-2-1- Simple compression

The compression tests were carried out using a Seditech single-axis press - densification in an appropriate mould followed by crushing the corresponding compacts (1) - in a range of pressures from 100 to 600 bars.

The cohesion values were fitted using a model (2-3) with the following form:

$$R = R_\infty(1-n)^\gamma$$

where:

n = porosity calculated after eliminating the constraint

R = resistance of the compact (bar)

R_∞ = resistance of the compact at zero porosity (bar)

γ = parameter expressing the capacity to acquire cohesion and the quality of the bonds.

The physical significance of γ and R was discussed previously.

1-2-2- Texture analysis

The compacts produced under pressures of 100, 300 and 600 bars were subjected to a study of structure. The porosity was determined using a Micromeretics 9300 mercury porosimeter (1).

Adopting a model with cylindrical pores and assuming that the entire pore volume, V, is distributed as a single long pore of diameter, d_p, it is possible to calculate its length, L.

The interpretation previously given on the basis of global porosity values (1-4) was analysed further by considering seven groups with pore sizes corresponding to the following ranges: 0.1 - 3 µm; 3-6 µm; 6-9 µm; 9-12 µm; 12-15 µm; 15-18 µm and 18-21 µm.

It should be noted first of all that $L = \sum n_i l_j$

where n_i = number of pores with diameter d_{pi}

l_j = length of the various pores with diameter d_{pi}

The pores had an order of greatness $d_{pi} = \frac{\phi}{5}$ where ϕ = particle diameter.

Taking l_i = l = constant, L reflects of n_i which in turn reflects the contact points or close points of the particles.

This is confirmed by the fact that L is great if d_{pi} is very small ($L = k\frac{V}{d_p^2}$), small pores signifying very tight particle/particle confinement.

It is therefore possible to monitor this tight confinement by monitoring L.

It should now be noted that $V_i = \frac{\pi}{4} d_p^2 \sum n_i l_j$ depends mainly on D_i.

If we monitor V_i, we therefore monitor the resistance of the bed to the filling of its large pores.

RESULTS AND INTERPRETATION

2-1- Distribution of the pore length, L_i, as a function of pressure

Figures 1a to 1h show the pore distribution for each lactose expressed in the terms of equivalent pore length as a function of pore pressure.

The contribution to L was essentially that of the pores measuring 0.1 to 3 µm. For FF, DCL 11, EFC and M, this contribution remained low regardless of pressure. It was clearly greater for DCL 21, Tab, PF and Pl; DCL 21 and Tab were the most likely to create 0.1-3 µm pores in response to pressure, whereas the impalpable powder Pl had numerous small pores from low pressure values which were partially filled as the pressure rose. In the case of PF, the biggest pores disappeared from the outset to yield the 0.1-3 µm class.

The participation of the small pores in the total length increased in the order listed below:

EFC < FF = DCL 11 = M << DCL 21 < Tab

but the mechanism differs in the various substances.

In the case of PF and Pl, the pores disappear due to rearrangement of the fine particles; some degree of fragmentation is, however, required to account for the increase in L and therefore in the number of particles.

For DCL 21 and Tab, the fine particles appear as a result of "crushability" in response to pressure.

For the first four substances (EFC, FF, DCL 11, M), the largest particles, which are poorly "crushable", resist pressure.

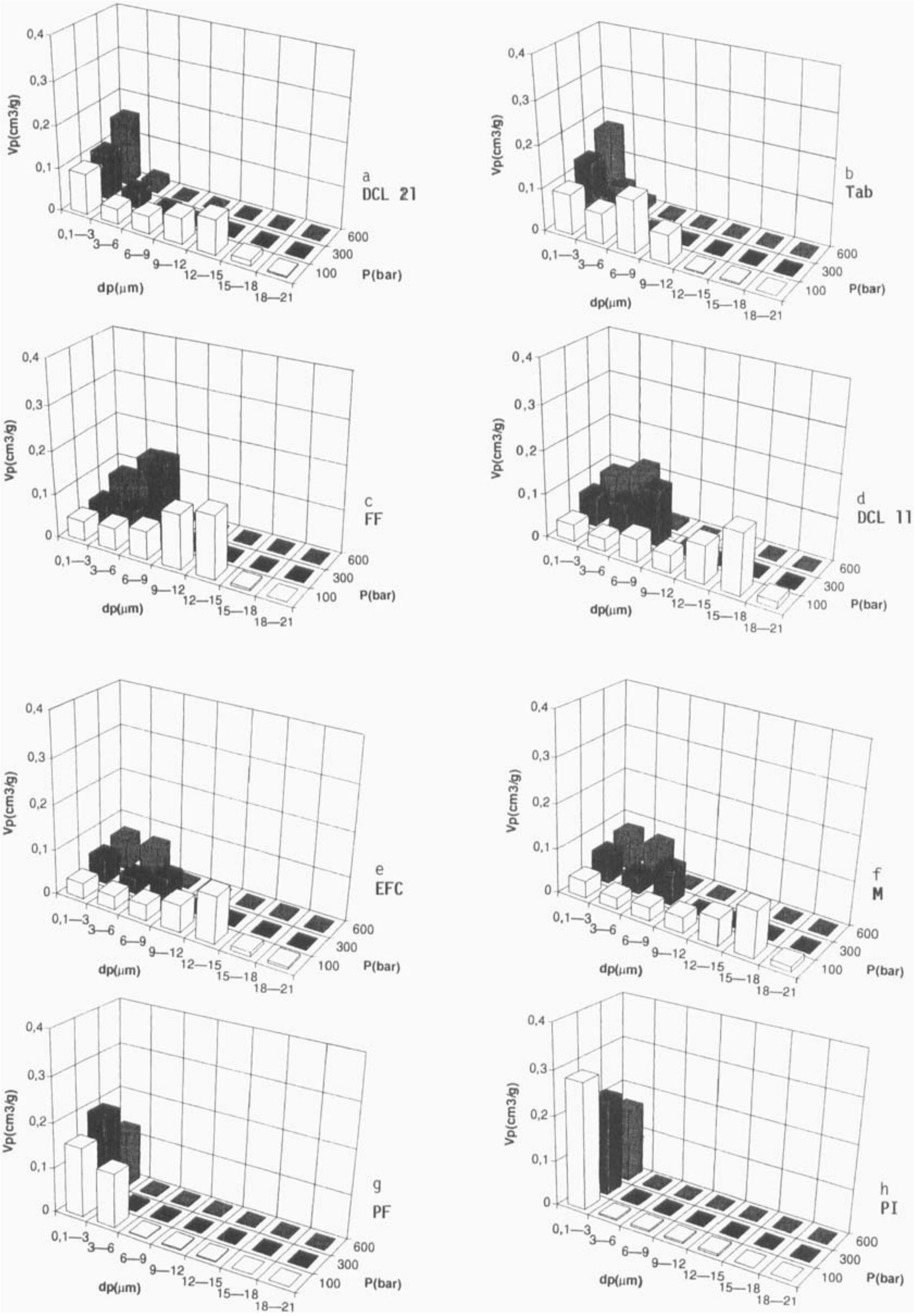
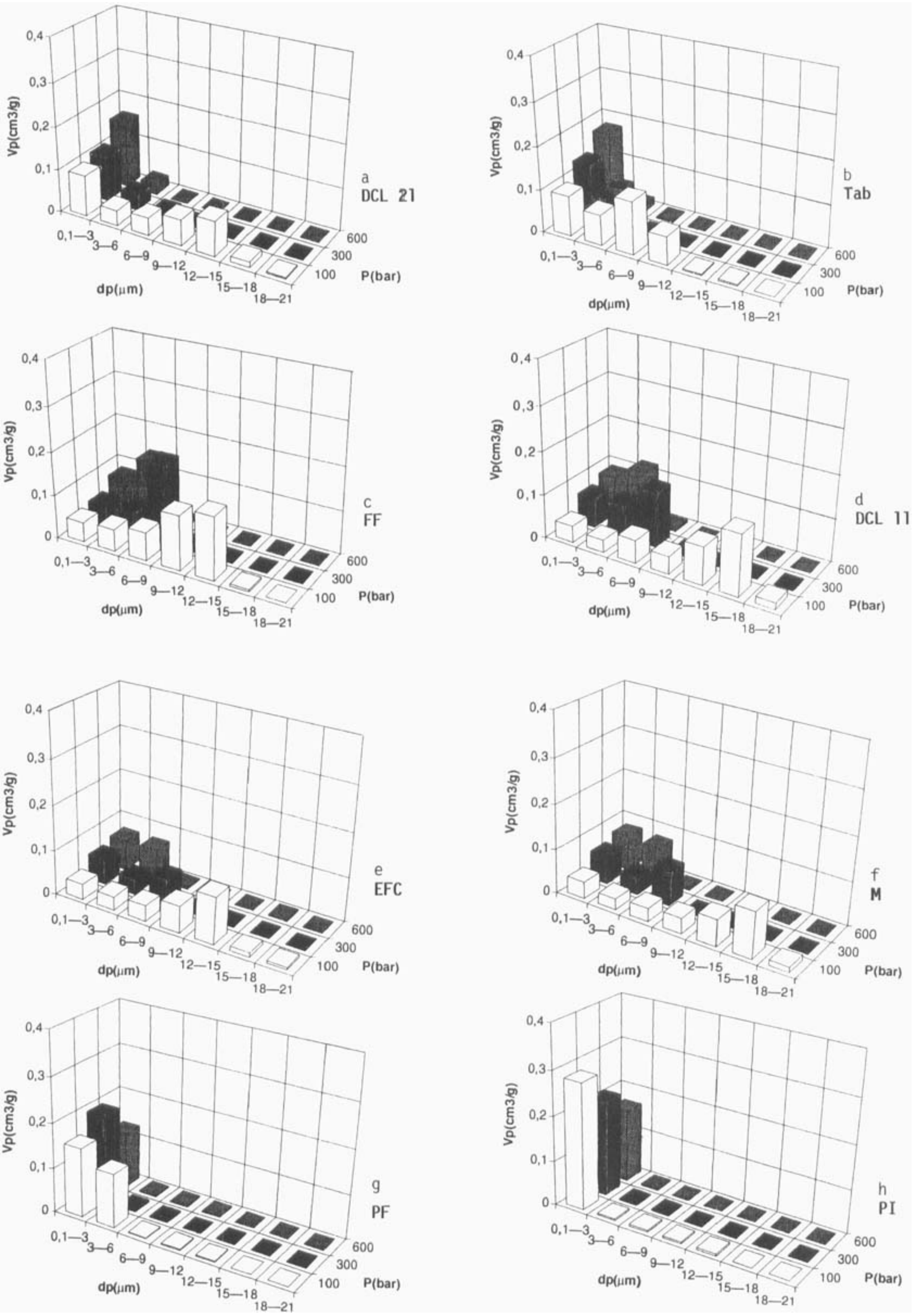


FIGURE 1a - 1h
Distribution of the Pore Length, Li, as a function of the Diameter and Pressure Classes



FIGURES 2a - 2h
Distribution of the Pore Volume, V_i, as a function of the Diameter and Pressure Classes

2-2- Distribution of the pore volume, V_i, as a function of pressure

Figures 2a to 2h show the pore distribution, expressed as the pore volume, $V_{i,}$ for each lactose as a function of pressure.

The 100 bar pressure distinguished between PF and Pl and the other substances. The porosity was due to the 0.1-3 μm pores in the case of Pl and the 0.1-6 μm pores for PF. The porosity was due to a wider scatter of pores from 0.1-20 μm for the other lactoses. DCL 11 still had numerous 15-18 μm pores.

The 300 bar pressure distinguished even more clearly between the substances: PF and Pl had pores predominantly measuring 0.1-3 μm. Most of the pore volume of FF and DCL 11 was attributable to 6-9 μm pores with a total range of 0.1-9 μm. Between 100 and 300 bars, FF "resisted" more than DCL 11.

DCL 21 was similar to Tab and EFC and resembles M.

At 600 bar, the 0.1-3 μm class was greater than at 300 bar for all substances except PF and Pl, for which the volume of these pores tended to diminish due to filling following the disappearance of the 3-6 μm pores. In the case of FF and DCL 11, it was the 3-6 μm pores which made the greatest contribution to the pore volume.

DCL 21 and Tab converged towards 0.1-3 μm and showed only very few 3-6 μm pores, unlike EFC and M which retained more of these pores.

The decaking or crushing of particles accounted for these behaviour patterns: FF and DCL 11 were not finely "crushed" by pressure: they rearranged and retained their large pores with a low L. PF and Pl, which were fine from the outset, rearranged mainly at about 0.1-3 μm. The other substances were finely "crushed" (DCL 21 and Tab) or moderately "crushed" (EFC and M).

The preservation of large pores decreased in the order listed below:

FF ≈ DCL 11 > M ≈ EFC > DCL 21 = Tab = PF = Pl

CONCLUSION

This study demonstrated the role of crystallographic and textural parameters in compressibility. Using first order models with $R = f(n)$, $P = g(n)$ and investigating behaviour by porosimetry of the compacts, we have highlighted the role of the characteristic γ, related to the quality of interparticular bonds (1). The lactoses can be distinguished on the basis of R_∞ (1) and the texture of the bed (a property which depends on the arrangement and therefore the size, shape and distribution). This texture, which is monitored here from V_i, D_i and L_i shows that:

- FF and DCL 11 showed greater resistance to "fine" rearrangement; the number of contacts was not increased. FF may have been slightly more efficient in maintaining a high value of V. These were the most effective substances.
- DCL 21, initially fragmented, had high intrinsic resistance, but it rearranged so that the resistance rose much more slowly (600 bar, low V).

the other substances were much less effective:

- Tab was similar to DCL 21, but had much lower cohesion; its capacity to develop cohesion, related to its α monohydrate structure, accounted for the fact that it responded less well than DCL 21, which is β anhydrous compound.

- EFC and M increased in density, but did not acquire sufficient cohesion at 600 bar. EFC, which can be confined, increased the number of contacts, but these remain weak due to the dilution of pressure.

- PF and Pl, which were fine from the outset, were crushed, which increased the number of contacts; at 600 bar the state of confinement achieved did not allow them to convert the energy input into cohesion. Pl in particular underwent very fine rearrangement with no bridging forces.

- γ increased from M-PF-Tab-EFC-Pl, indicating that the texture had less and less capacity to acquire cohesion for similar structure.

Some authors accept that fragmentation promoted the acquisition of cohesion (4), but from this study, it would appear that the multiplication of contact points due to fragmentation has a negative impact on the effectiveness of Pl and PF and also of EFC and M. In the cases of DCL 21 and Tab, this effect is compensated for by the unusual structure and texture respectively of these two substances and they both showed significant cohesion. Cohesion involving Van der Waals forces calls for small interparticular distances and consequently, FF and DCL 11 which resist rearrangement and maintain greater porosity when subjected to pressure, owe their high cohesion to high local pressures.

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