DRUG DEVELOPMENT AND INDUSTRIAL PHARMACY®
Vol. 29, No. 10, pp. 1127–1135, 2003

RESEARCH PAPER

Talc Functionality as Lubricant: Texture, Mean Diameter, and Specific Surface Area Influence

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ABSTRACT

Talc is widely used as a glidant (flow regulator) for powders. This study highlights the characteristics that confer to talcs new end use properties in improving the lubrication function during compression. We studied the contribution of texture, mean diameter (D50), and specific surface area on the residual die pressure, the ejection pressure, the lubrication index, and the tablet hardness. Different textures were studied: microcrystalline, macrocrystalline, and moderately macrocrystalline talc grade. The compression parameters were improved according to the texture. D50 varies from 0.62 to 15 µm. As D50 decreases, the lubrication performance is improved. Finally, the specific surface area of talcs was studied. This last characteristic of talcs was shown as the most relevant parameter in determining lubrication ability.

Key Words: Talcs; Lubrication; Texture; Specific surface area; Compression parameters.

INTRODUCTION

The pharmaceutical forms available for oral drug administration are numerous and are under constant development. Among the most commonly used solid forms, and representing more than 50% of the drugs in pharmacy, tablets are prevalent because of their many advantages, which include ease of use, unit dosage, good conservation properties, and low production cost.

If their advantages are multiple, their development remains awkward and requires a good

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DOI: 10.1081/DDC-120025870
0363-9045 (Print); 1520-5762 (Online)
www.dekker.com

knowledge of the physical and mechanical properties of the powders used. Physical and functional properties of the powders to be compressed are partly defined by tests registered in the European Pharmacopeia.[1] Among these properties, flow cohesion and lubrication are generally the most critical factors for industrial production.

The mix of powders from which a tablet is made includes an active ingredient and several excipients; the latter are included to improve the compression parameters of the tablet.[2] Magnesium stearate is the reference antiblocking agent.[3] Talc is also typically added to regulate flow.[4] Recently, a study showed the possible relation(s) between talc anti-sticking power and the basal dimension and thickness of talcs particles.[5]

Our study, realized in collaboration with a talc supplier (Talcs de LUZENAC-2 place Edouard Bouillières-31100 Toulouse), compared different grades of talcs: 3 of them (I1, F2, and F3) are commercially available and meet the requirements of the European Pharmacopeia[1]; 12 are new experimental grades of talc, especially developed for our study.

The objective of this study is to investigate the influence of the talc specific parameters on the lubrication function during the manufacture of tablets and also on their mechanical properties. This study was based on a formula for direct compression[6,7] to show the role of the talc-specific characteristics (texture, D50, and specific surface area) on the parameters of compression (lubrication index, residual die pressure, and maximum ejection pressure) and on the tablet hardness.

Figure 1. The SEM observation of microcrystalline talc.

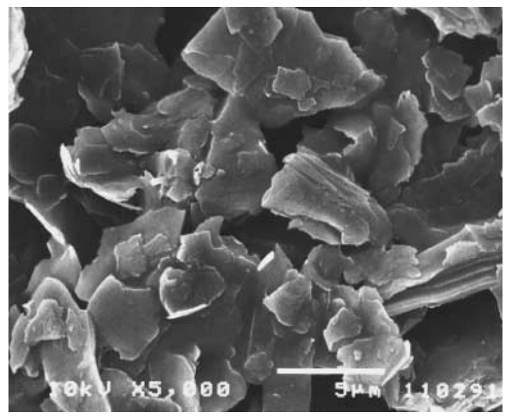

Figure 2. The SEM observation of macrocrystalline talc.

MATERIALS AND METHODS

Talcs

Talcs can be distinguished between their textural types: microcrystalline and macrocrystalline talcs.[8]

Microcrystalline talc is characterized by spheroid particles that are composed of a mass of small particles clustered together with no preferred orientation (Fig. 1). Macrocrystalline talc (Fig. 2) is characterized by sliver-like particles made of elementary macro particles. Intermediate textures also exist.

It is also possible to distinguish microcrystalline and macrocrystalline talcs between their shape coefficient. The shape coefficient is the mean diameter of a disc, which has an area equivalent to the particle area.

Various talcs have been studied. They all come from Luzenac (France). They differ in terms of texture, shape coefficient, mean diameter (D50), and specific surface area and are listed in Table 1.

Talcs F1, F2, F3, F4, and F5 present an intermediate texture. Because of their shape coefficient close to the shape coefficient of the macrocrystalline talcs, we called them moderately macrocrystalline.

The mean diameter D50 was determined by measurement of the sedimentation rate (Stockes law) using Sedigraph® apparatus. The specific surface area was determined by the BET method (nitrogen adsorption) using Nova 1000 Quantachrome® apparatus. The shape coefficient was determined by using a Malvern® laser diffraction granulometer. These apparatus are traditionnally used by Talcs de Luzenac to characterize their talcs.

The particle size distribution is monodispersed and Gaussian so the D50 really represent the mean

Table 1. Studied talcs.

Name	Texture	Shape coefficient	D50 (μm)	Specific surface area (m^2/g)
S1	Microcrystalline	2.2	12.00	7.0
S2			8.00	8.5
S3			3.80	11.5
S4			1.70	13.5
S5			0.62	18.0
I1	Macrocrystalline	4.6	15.00	3.0
I2			6.00	4.5
I3			3.50	8.0
I4			2.10	9.5
I5			0.89	13.5
F1	Moderately	4.0	14.00	1.7
F2	macrocrystalline		11.00	3.0
F3			5.00	6.3
F4			1.60	9.5
F5			0.90	13.0

Table 2. Base formula.

Products	(%)
Microcrystalline cellulose ≪Avicel PH 101≫	40.0
Lactose ≪Fast flo≫	45.0
Starch ≪Sepistab ST200≫	15.0

diameter of our particles, for each texture. We used talcs with D50 from 0.62 to 15 μm.

Tablet Manufacture

The study was conducted by using the base formula presented in Table 2.

This base formula was selected because of its poor lubrication properties (cf. Table 3).

Tablets were manufactured by using this base formula to which 3% of talc was added; 3% is the classical usage rate of talc in pharmaceutical formulations. This rate is also the discrimination rate to use to characterize talcs influence; 200-g batches of the various mixes were blended in a TURBULA mixer (40 rpm) for 10 min.

For each formula with talc, the flow time was measured in accordance with the European Pharmacopeia 4th Edition (§ II-9-16).[1] In each case, the result was less than 7 sec, which confirm the glidant (flow regulator) property of talcs.

The tablets were manufactured with an alternative Korsch EKO tabletting machine, which was instrumented for punch pressure and displacement and fitted with a set of flat cylindrical punches with a surface area of 1 cm^2.[9]

The volume of the compression chamber was calibrated to obtain tablets with an approximate average mass of 350 mg and about 3-mm thick. This volume was kept constant for all formulas.

The compression pressure, determined by the upper punch setting, was kept constant for all formulations at about 150 MPa.

The lubrication performance was determined by measurement of the lubrication index, the residual die pressure, and the maximum ejection pressure.[10] The lubrication index is the ratio of the lower punch transmitted pressure to the upper punch applied pressure. The closer this index is to 1, the more satisfactory the lubrication. The residual die pressure is the pressure exerted on the lower punch by the tablet when the upper punch is no longer in contact with it. The ejection pressure is the maximum pressure exerted by the lower punch to eject the tablet. This is equal to the sum of the residual die pressure and the pressure required to dislodge the tablet from the die walls.

The rupture resistance of the tablets, also called hardness, was measured in accordance with the European Pharmacopeia 4th Edition (§ II-9-8).[1]

RESULTS AND DISCUSSION

The results obtained are listed in Table 3. They represent the average of 10 measurements.

Role of Talc Texture

Figures 3 to 6 compare the residual die pressure, the ejection pressure, the lubrication index, and the tablet hardness according to D50 for the three different textures and show a relationship between talc texture and lubrication performance.

Remember that the lower the residual and ejection pressure are, the better is the lubrication and higher are the lubrication index and the tablets hardness.

The results show that the behavior of moderately macrocrystalline talcs approaches that of macrocrystalline talcs. We point out that moderately macrocrystalline talcs and macrocrystalline talcs belong to the same family: the lamellar talcs. Therefore, for the further discussion we will combine them with macrocrystalline talcs.

Table 3. Results.

Talc	Residual die pressure (MPa)	Ejection pressure (MPa)	Lubrication index	Hardness (N)
Base formula	11.4 ± 0.6	18.1 ± 0.9	0.64 ± 0.006	107.0 ± 5.3
S1	6.4 ± 0.3	7.0 ± 0.4	0.72 ± 0.007	118.0 ± 5.9
S2	5.0 ± 0.2	5.7 ± 0.3	0.74 ± 0.007	125.0 ± 6.2
S3	4.0 ± 0.2	5.0 ± 0.2	0.76 ± 0.008	114.0 ± 5.7
S4	4.0 ± 0.2	5.4 ± 0.2	0.80 ± 0.008	87.0 ± 4.3
S5	4.0 ± 0.2	5.1 ± 0.2	0.79 ± 0.008	75.0 ± 3.7
I1	8.0 ± 0.4	8.7 ± 0.4	0.69 ± 0.007	109.0 ± 5.4
I2	6.0 ± 0.3	6.7 ± 0.4	0.72 ± 0.007	104.0 ± 5.2
I3	5.4 ± 0.3	5.7 ± 0.3	0.75 ± 0.007	109.0 ± 5.4
I4	5.3 ± 0.3	4.3 ± 0.2	0.79 ± 0.008	82.0 ± 4.1
I5	5.1 ± 0.2	4.0 ± 0.2	0.79 ± 0.008	77.0 ± 3.8
F1	8.0 ± 0.4	8.7 ± 0.4	0.71 ± 0.007	112.0 ± 5.6
F2	7.7 ± 0.4	8.7 ± 0.4	0.70 ± 0.007	109.0 ± 5.4
F3	5.0 ± 0.2	5.4 ± 0.2	0.74 ± 0.007	111.0 ± 5.5
F4	5.4 ± 0.2	4.6 ± 0.2	0.79 ± 0.008	88.7 ± 4.4
F5	5.1 ± 0.2	4.0 ± 0.2	0.80 ± 0.008	78.5 ± 3.9

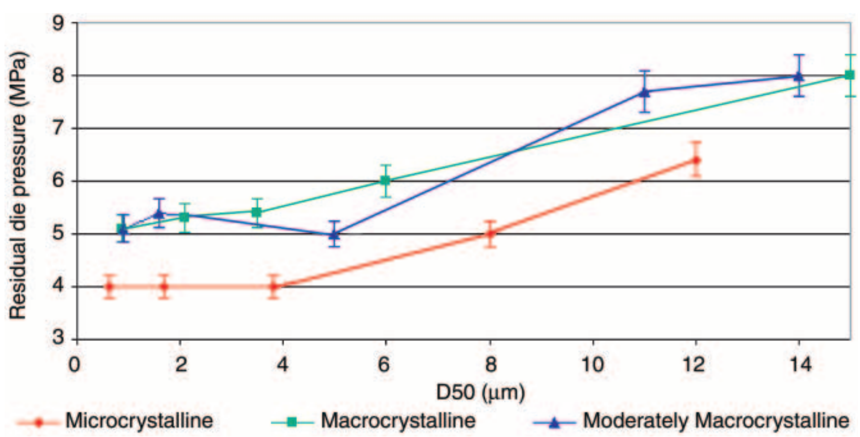

Figure 3. Residual die pressure = f (D50).

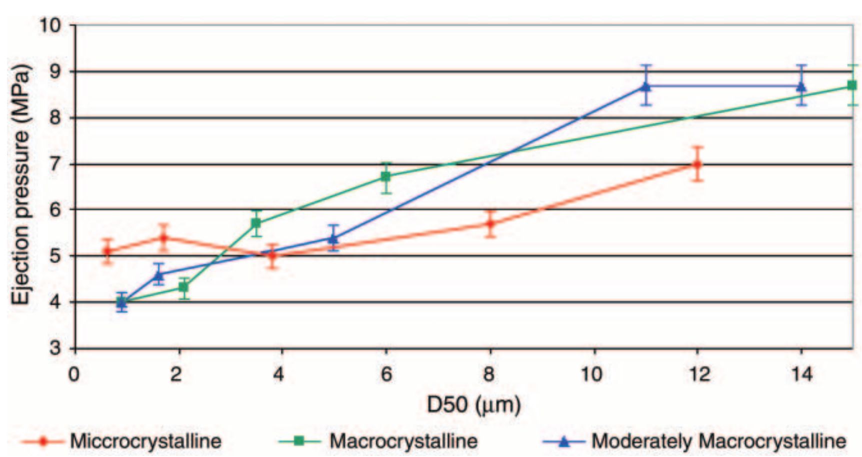

Figure 4. Ejection pressure = f (D50).

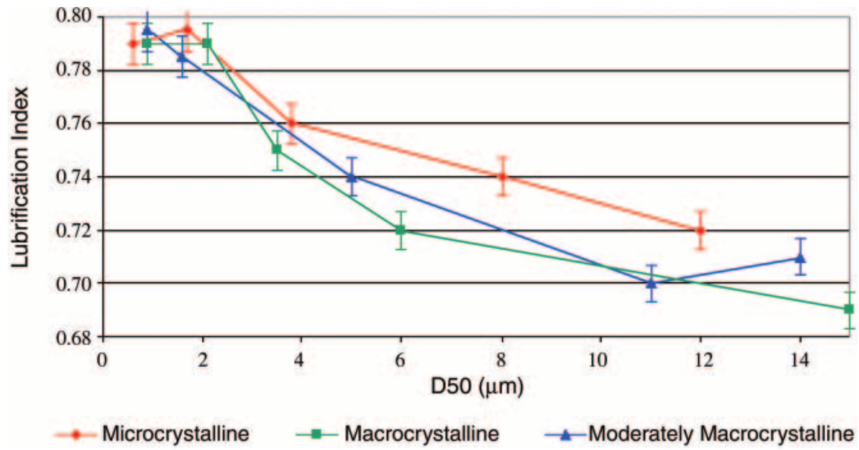

Figure 5. Lubrication index = f (D50).

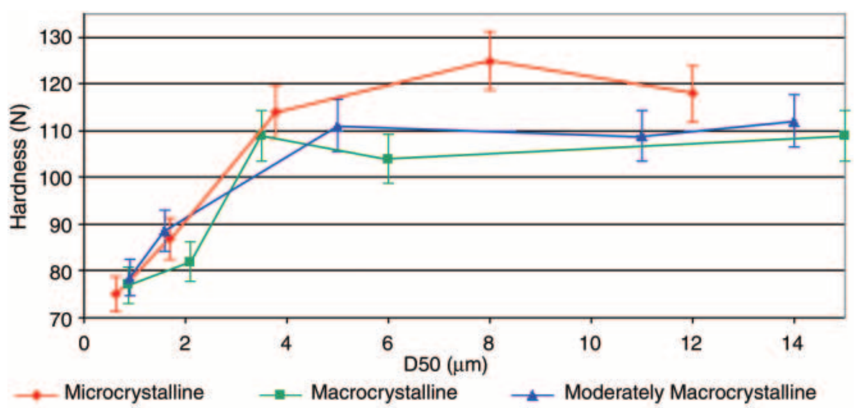

Figure 6. Hardness = f (D50).

Talcs with D50 > 5 μm are obtained from a classical manufacturing process, whereas talcs with D50 < 5 μm require a micronization process that is unprofitable industrially. To discuss the influence of the talc texture, we propose to divide the results in two parts: D50 > 5 μm or D50 < 5 μm.

When D50 > 5 μm, we notice the influence of talc texture on the compression parameters and also on the tablet hardness. Indeed, the reduction in residual die pressure for a formula using microcrystalline talc when compared to the base formula (11.4 MPa) is at least 44%; the reduction for a formula with a macrocrystalline talc is still 14%.

We also observe a reduction in ejection pressures. The difference between formulas with microcrystalline talc and the base formula (18.1 MPa) is at least 61% and the difference between formulas with microcrystalline talc and those with macrocrystalline talc is still up to 13%.

It is the same for the lubrication index. The use of microcrystalline talc allows an increase of at least 19% compared to the base formulas (0.64) and 2% compared to formulas with macrocrystalline talc.

Lastly, the tablet hardness is less strongly influenced by talc use. With a microcrystalline talc, an increase of 6.5% compared to the base formula (107 N) is observed; there is also an increase of 5.4% compared to formulas with a macrocrystalline talc.

When D50 < 5 μm, we note that the results converge. Indeed, crushing results in the convergence of the textures and thus the performances become equivalent.

Thus, whatever their texture, the inclusion of talcs positively influence compression characteristics as well as tablet hardness. However, microcrystalline talcs have a greater influence on the parameters studied for D50 > 5 μm. The performances are equivalent for D50 < 5 μm.

Afterward, we highlight the correlation between the compression parameters and the D50 of talcs.

The largest particles are those of talc I1 (macrocrystalline, D50 = 15 μm). For this talc, we observe a reduction of 47.4% in the residual die pressure, a reduction of 63% in the ejection pressure and an increase of 12.5% in the lubrication index compared to the base formula.

The finest talc particle correspond to S5 (microcrystalline, D50 = 0.62 μm). For this talc, we observe a reduction of 65% in the residual die pressure, a reduction of 72% in the ejection pressure, and an increase of 23.5% in the lubrication index compared to the base formula.

Then the smaller the D50, the greater the lubrication efficiency.

Role of the Talc-Specific Surface Area

We investigated more precisely the role of the talc-specific surface area on the compression parameters and tablet hardness. According to the previous conclusion, only talcs with D50 < 5μm were studied.

First, we notice that specific surface area discriminates talcs (Fig. 7).

We note then that, whatever the nature of talc used, the increase of specific surface area generates a reduction in the hardness of the tablets. The three curves take a similar form. All the three fall exponentially from 110 N hardness, with the increase of specific surface area, until reaching a plateau for 75 N hardness, in the studied range.

It seems that beyond this end point the increase of specific surface area has less influence on the tablet hardness. A 30% decrease of hardness is observed for a 56% specific surface area increase for microcrystalline talcs, 69% for microcrystalline talcs, and 106% for moderately microcrystalline talcs.

Whatever the nature and whatever the specific surface area of talc used, we observe a constancy of the residual die pressure (Fig. 8). The values of the residual die pressure are equivalent for macrocrystalline and moderately macrocrystalline talcs. We note that the residual die pressure is better for microcrystalline talcs (20% decrease compared to macrocrystalline and moderately macrocrystalline talcs).

For macrocrystalline and moderately macrocrystalline talcs, we observe the decrease of the ejection pressure with the increase of specific surface area (Fig. 9) in the studied range. Indeed, between the use of talc F3 (specific surface area = 6.3 m^2/g) and of talc F5 (specific surface area = 13 m^2/g), we observe an improvement of the ejection pressure: 26% reduction in the ejection pressure for a 106% increase of the specific surface area.

For microcrystalline talcs, it seems that the ejection pressure reaches an optimum from 13 m^2/g. The increase of the specific surface area beyond this value does not act on the ejection pressure.

For the lubrication index (Fig. 10), we observe an improvement with the increase of the specific surface area. An optimum is reached (0.8) whatever the texture of talc used, for a 13 m^2/g specific surface area.

Based on the talc nature, an explanation can be stated. The microcrystalline talcs, made of small

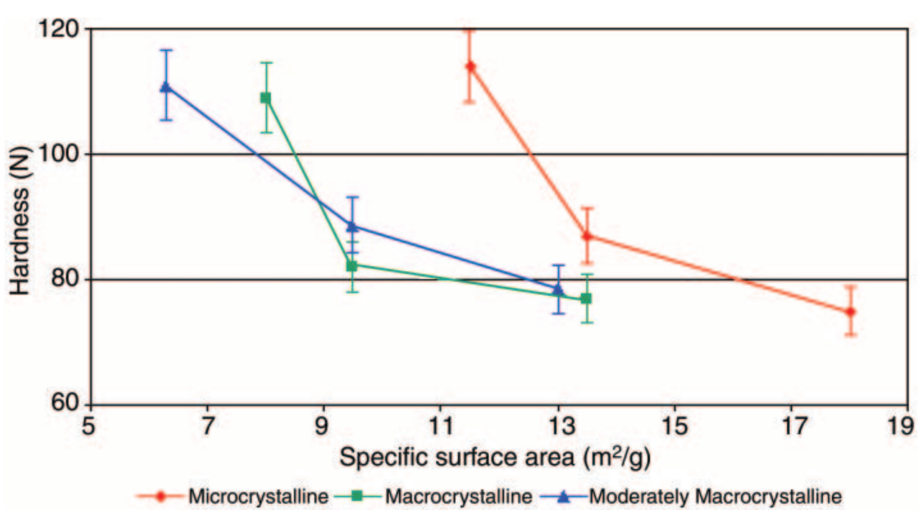

Figure 7. Hardness = f (specific surface area).

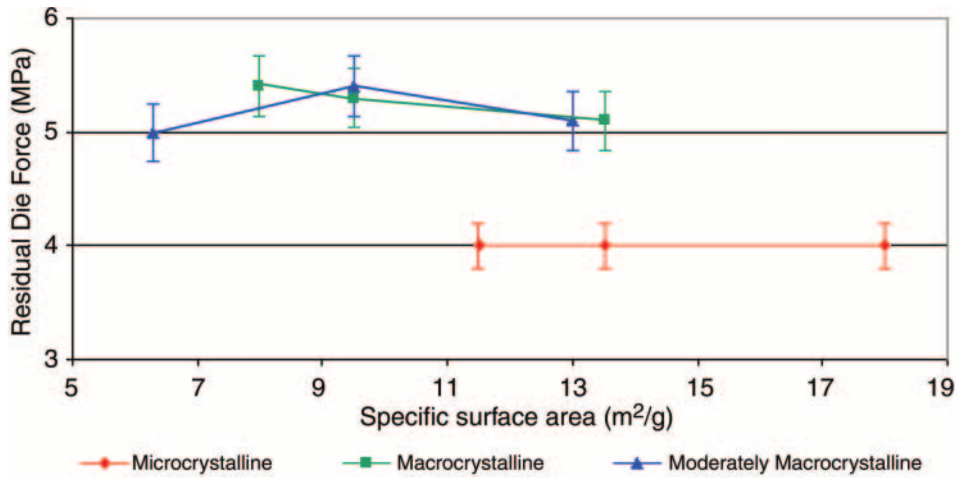

Figure 8. Residual die pressure = f (specific surface area).

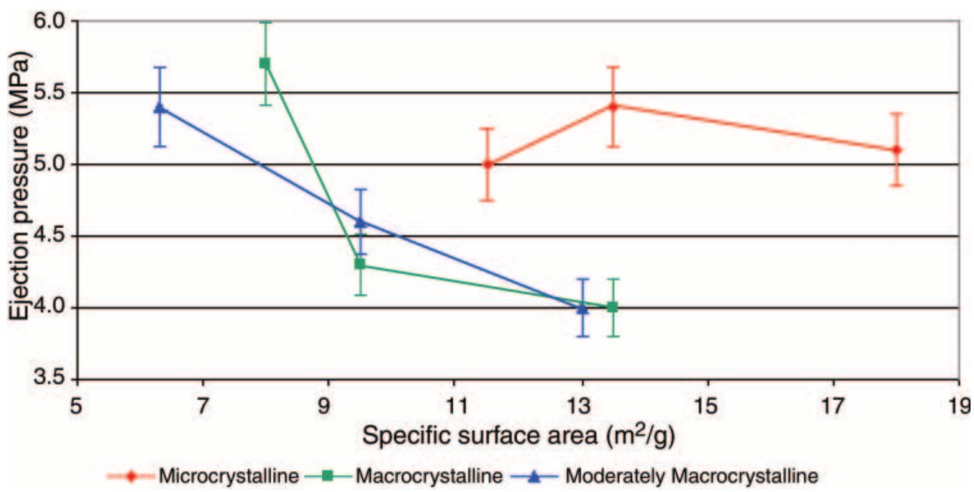

Figure 9. Ejection pressure = f (specific surface area).

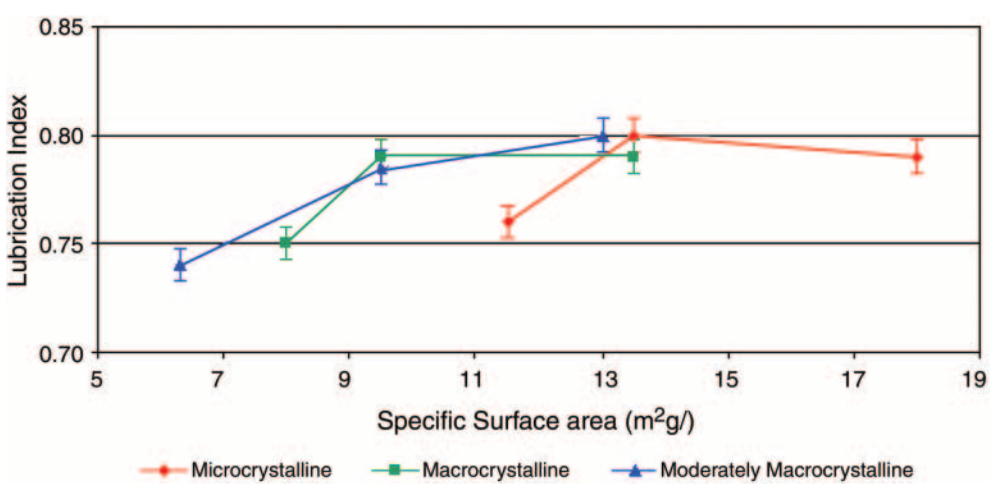

Figure 10. Lubrication index = f (specific surface area).

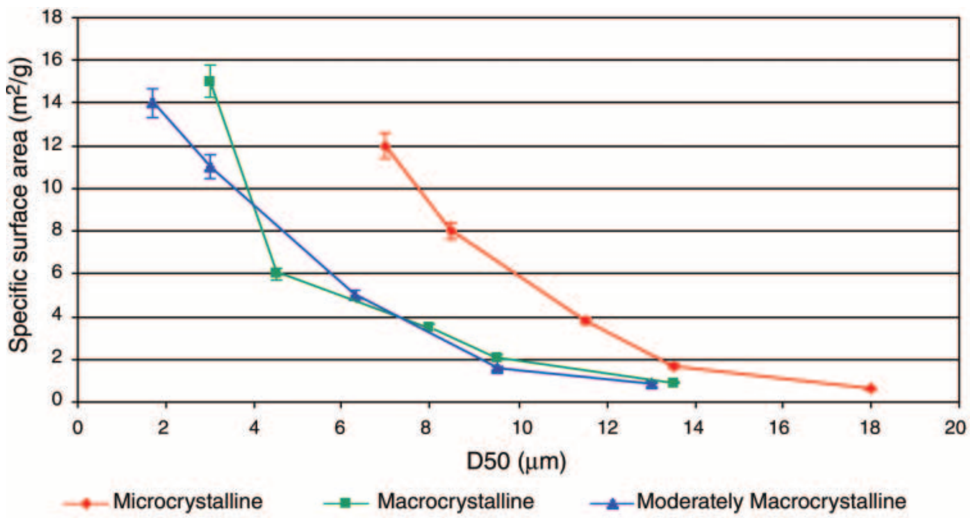

Figure 11. D50 = f (specific surface area).

particles clustered together, offer a rough surface, which could perform as fixing points for all the other formulation ingredients. This ‘‘binder action’’ generates a better cohesion or compaction in the powders, which allows a decrease of the residual die pressure. However, this rough surface will also increase the ejection pressure of the tablet by increasing the friction between the tablet and die walls. For macrocrystalline talcs, made of coarse lamellar particles, an opposite mechanism maybe involved. They offer a smooth and flat surface with no fixing points; therefore, the cohesion or compaction of the tablet is reduced and the residual die pressure increased. However, this smooth and flat surface, combined with the intrinsic lubricant properties of talc, decreases the ejection pressure.

According to the talc nature, the surface specific area increase allows the improvement of the residual die pressure for microcrystalline talcs or the ejection pressure for macrocrystalline and moderately macrocrystalline talcs. The surface specific area increase improves the lubrication index.

Correlation Between Specific Surface Area and D50

In Fig. 11, we show results from our investigation of the correlation between talcs specific surface area and talcs D50 for the the three different textures.

The results show that the behavior of moderately macrocrystalline talcs approaches that of macrocrystalline talcs. That finding confirms that moderately macrocrystalline talcs could be connected with macrocrystalline talcs.

CONCLUSION

Talc is typically added to regulate flow in direct compression. This study highlighted the characteristics that confer to talcs new functinalities in improving the lubrication ability.

First, we showed that for $D50 > 5\,\mu m$, the talc texture influences the compression parameters as well as tablet hardness: microcrystalline talcs are more efficient than macrocrystalline talcs. Next, the D50 of talcs also influences the lubrication properties: a low D50 induces better lubrication. When $D50 < 5\mu m$, the performances become equivalent because of the crushing, which makes the textures converge. Then the finer the D50, the greater the lubrication efficiency. Consequently, when microcrystalline talcs are used, every granulometry can be recommended. For fine particles ($D50 < 5\mu m$), either microcrystalline (S5), macrocrystalline (I5), or moderately macrocrystalline (F5) talcs can be used to increase lubricant parameters.

Finally, the specific surface area of a talc seems to be the most relevant parameter in determining lubrication ability: its increase improves the pressure transmission in the compacted mass and results in an improvement of the lubrication index. The increase of specific surface area also creates a reduction of particle/wall friction, which confers an antiblocking property to these talcs.

Texture, D50, and specific surface area are three talc characteristics, that govern lubricating properties. Therefore, it seems to be of interest to control and optimize these characteristics so that talc could be used not only as a flow regulator but also to provide additional lubricating properties.

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