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Effect of particle morphology on the hiding power of talc powder

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Summary

Two samples of talc powder USP—from different sources—showed remarkable difference, over a wide range of concentrations, in their hiding capacities. Particle size, size distribution and surface area were not adequate to account for the difference in physical behavior of the two samples. Morphological characterization based on Fourier shape descriptors of the particle boundary can better explain the difference. The study suggests that in addition to size parameters, the particle shape is equally important in the quality assurance monographs of pharmaceutical solid excipients.

Introduction

The behavior of bulk solids is strongly influenced by the shape of the particles (Meloy 1977). Powders with identical size and size distribution, density and chemical composition, may behave quite differently as a result of the particle morphology (Luerkens, 1982; Meloy, 1984).

In recent years, an increasing awareness of the need of a better characterization of solid excipients in drug formulation started to emerge (Cooper, 1976). Small changes in crystallization and in size reduction processes can produce variation in particle shape (Kaye and Luerkens, 1980; Durney, 1983). This in turn can lead often to production and quality assurance problems in the drug product formulations (Jones, 1977). Talc powder U.S.P. is used as flow regulator, anti-adhesive and lubricant in tablet marking (Shotton, 1976). It is used also in film coating suspensions as an opacifier (Porter, 1981).

There is no experimental evidence to our knowledge, showing the effect of particle shape on the physical behavior of talc. The present study was designed to determine whether the shape of the particles corrolate with the hiding power of talc powder used as a pharmaceutical excipient.

Experimental

Determination of the hiding power of talc

Talc powder U.S.P. (very fine) was obtained from two different sources [1,2]. A suspension 0.2%

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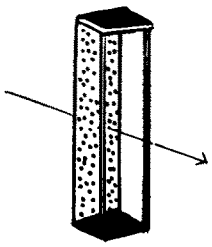

Fig. 1. Open rectangular cuvette used for measuring the opacity of talc.

w/v in bidistilled water was prepared containing 0.2% w/v of sodium lauryl sulfate as wetting agent. To study the hiding capacity, different dilutions (containing 0.01–0.2% w/v talc) were made from stock suspension. 1 ml of the diluted suspension was placed in a cuvette [3] (see Fig. 1) and left to sediment and evaporate to dryness at room temperature. The transmittance of the deposited film of talc particles was measured spectrophotometrically at 450 nm. The hiding power of talc is an indirect measurement of the capability of talc particles of rendering the coating suspension used in film coating opaque. The light transmission method was previously reported by various investigators to study the factors controlling the opacity of white pigments (Jacobsen, 1949; Stutz and Pfund, 1927; Goeb, 1951).

Particle size determination

The particle size distribution of talc was determined using the Coulter counter (aperture size 100 μm) [4]. Scanning electron microscope based automatic image analysis (SEM-AIA) [5] was used to measure the size distribution and the projected surface of the talc particles in the two samples.

Surface area (cm^2/g) for each powder was determined from the mean particle diameter.

Morphological analysis of talc particles

(a) Shape factor determination

Computer control of the electron beam of the SEM coupled with image analysis capabilities (Fritz, 1982) were used to determine the shape factor of the talc particles of each sample. The determination of the shape factor was based on the measurement of 8 diameters passing by the center of gravity of the particle. From the surface area produced and the perimeter of each particle, the shape factor S was calculated as:

$$S = \frac{P^2}{4\pi A}$$

where A is the surface area and P the perimeter. At least 300 particles were used to determine the shape factor distribution for each sample of talc used in this study.

(b) Fourier descriptors

The use of Fourier descriptor for shape analysis of solid particles has been previously described (Beddow, 1977, 1980; Luerkens, 1982). It is based on the digitalization of the particle image by obtaining (x, y) coordinates of the particle boundary. These coordinates are used to calculate a set of invariant shape descriptors. In this study, Fourier descriptors were determined by a method reported recently for cervical cell classification (Nguyen et al., 1983).

In this method, the (x, y) coordinates of the particle boundary are represented in complex form as:

$$\mu(1) = x(1) + iy(1)$$

(Granlund, 1972) and are calculated in terms of Fourier series as:

$$u(1) = \sum_{n=-p}^{p} C_n e^{i2\pi nl/L}$$

where

$$C_n = \frac{1}{L}\int_0^L \mu(1) e^{-i2\pi nl/L}\, dl$$

C_n = the n^{th} Fourier coefficient; n = harmonic number; l = the path length along the particle boundary; L = the perimeter of the particle; i = the

[3] Fisher Brand disposable Styrene cuvets, Fisher Sci. Co. Pittsburgh PA15219.

[4] Coulter-Counter Model TA, Coulter Electronics Inc. Halaleah, FL.

[5] SEM-Jeol, ISM 840, Jeol Ltd. Tokyo, Japan.

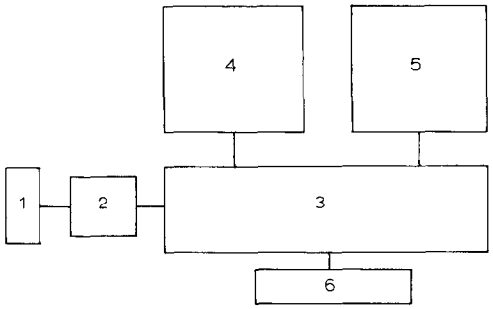

Fig. 2. Schematic diagram of the components of the system used for the digital processing of the particle image. 1 = Hitachi camera; 2 = Video Vangoch digitizer; 3 = Computer (IBM PCXT); 4 = Monitor Amdex for text and graphic display; 5 = Monitor Hitachi for image display; 6 = Printer.

complex value equal $\sqrt{-1}$; p = maximum harmonic number considered.

To obtain magnification invariance, we compute the Fourier coefficients normalized by the amplitude of the first coefficient.

$$a_n = C_n/(|C_1| + |C_{-1}|)$$

Thus, the normalized amplitude of each coefficient is $|a_n| + |a_{-n}|$

Fig. 2 shows schematic diagram of the components of the system selected for use in digitizing and extracting features from the image data (Jordan et al., 1985). Fourier descriptors were obtained using a special computer program written in Fortran and assembler that can determine the amplitude of the Fourier coefficients at different harmonics and represent graphically the computed data.

Results and Discussion

Fig. 3 shows the hiding power of the two talc samples used in this study (at different concentrations). In both cases talc particles were allowed to deposit on a solid surface to form a closely packed powder film. The difference between the two samples appears at a lower concentration and becomes more evident at higher concentration. At 0.2% w/v the hiding power of talc one power is nearly 10 times that of talc no. 2.

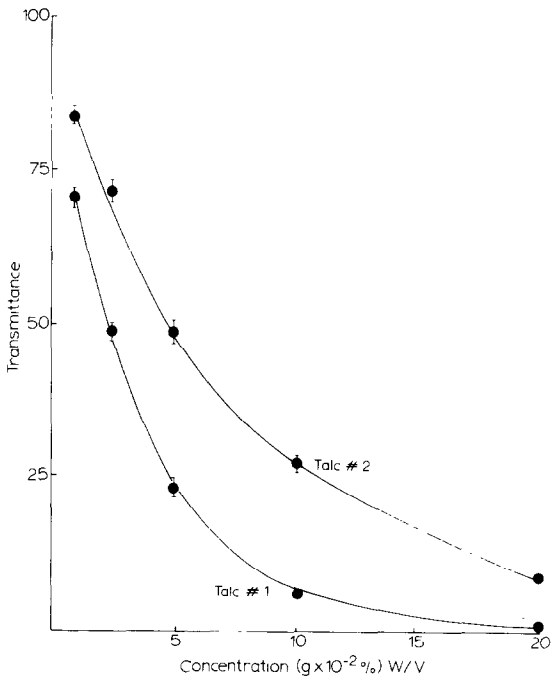

Fig. 3. Hiding power of two samples of talc powder as a function of the concentration.

The packing density of the powder film is certainly an important limiting factor behind the hiding power of talc powder in these experiments. Packing is a function of particle size–shape characteristics, density and mode of deposition (Gray, 1968). In this study, the absolute density and method of deposition (by gravity sedimentation) are the same for the two samples. Therefore looking into size–shape characteristics might explain the difference in the physical behavior of the two talc powders.

Fig. 4 shows the differential and cumulative distribution of the two powders using the Coulter counter. The difference between the two samples is significant. Using the SEM-AIA, the size distribution based on the projected surface area of talc particles is given in Fig. 5. The difference between the two samples is present but apparently not sufficient to explain the marked difference in the hiding power between the two samples of talc.

Table 1 summarizes the mean diameters and the surface area in cm^2/g determined by the two methods. The small difference in surface area (14%)

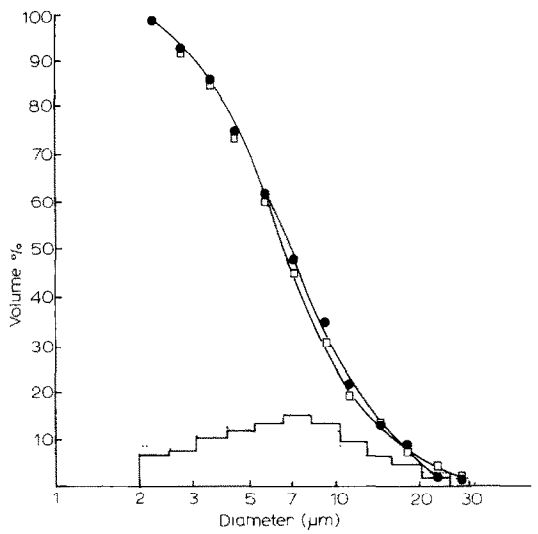

Fig. 4. Particle size distribution of two samples of talc expressed on the basis of volume. ·····, □ = talc no. 1 ———, ● = talc no. 2.

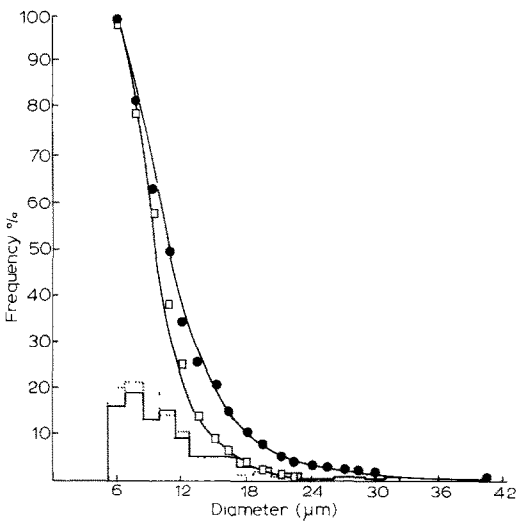

Fig. 5. Particle size distribution of two samples of talc expressed on the basis of surface. ·····, □, = talc no. 1; ———, ● = talc no. 2.

using SEM-AIA mean surface diameters data cannot account for the higher hiding capacity of talc no. 1.

The study of the shape factor using SEM-AIA (Fig. 6) shows clearly the difference in the shape factor frequency distribution in the two talc powders. The mean shape factor is 1.29 ± 0.4 and 1.39 ± 0.05 for talc no. 1 and talc no. 2, respectively. The shape factor calculated by this method, shows clearly that talc no. 1 consists of particles which are more spherical in shape than talc no. 2. The relatively higher irregularity of talc no. 2 might be responsible for the resistance to a more close packing arrangement as compared with talc no. 1.

The morphological difference between the two

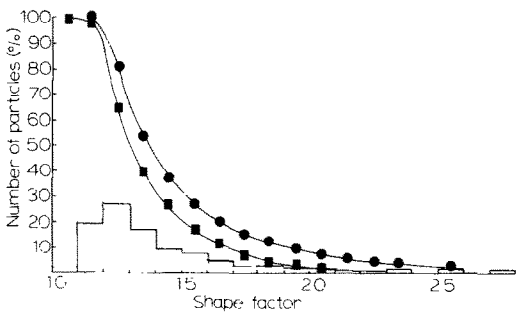

Fig. 6. Shape factor distribution of the two samples of talc, determined by SEM-AIA. ·····, □ = talc no. 1; ———, ● = talc no. 2.

TABLE 1

SIZE PARAMETERS OF THE TWO TALC POWDERS

Method	Talc	Mean diameter (μm)	Surface (cm^2/g)
Coulter counter	1	8.38	2898
	2	8.43	2881
S.E.M.	1	8.92	2723
	2	10.39	2337

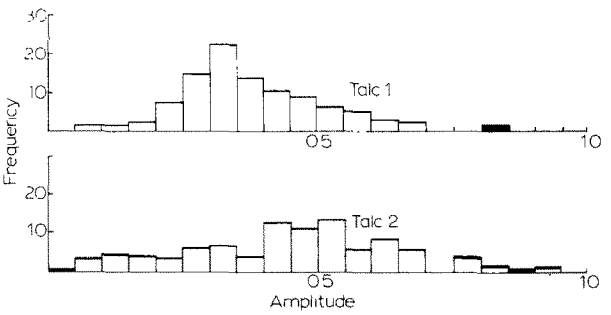

Fig. 7. Shape frequency distribution. Harmonic 3.

samples was better characterized and quantified by the use of Fourier descriptors as seen in Figs. 7, 8 and 9. More specifically in Fig. 8, the average normalized Fourier amplitudes, at harmonic number 2–8 of talc no. 1 are 30–50% less than talc no. 2. These amplitudes show strongly that talc no. 1 consists of more particles of elliptical shape (less of multimodal shape) and of smoother boundaries than talc no. 2. The morphological analysis of talc powders using this method gave a more precise and definitive quantification of particle shape. Fig. 7 is an example of the frequency of the amplitude at the 3rd harmonic. Fig. 9 shows log amplitude vs the harmonic number illustrating the 'particle signature' of the two talc powders used in this study.

From the data obtained, it appears that morphological characteristics of the samples of talc can better explain the difference in the behavior between the two samples of talc. The study suggests that, when using talc powder U.S.P. as an opacifier in pharmaceutical formulations, one has to realize that size and size distribution are not sufficient specifications for the performance of this pharmaceutical raw material. The morphological characteristics of talc which depend on the size reduction process employed can significantly affect the physical behavior of this excipient as an opacifier and account for unexplained examples of batch to batch variation.

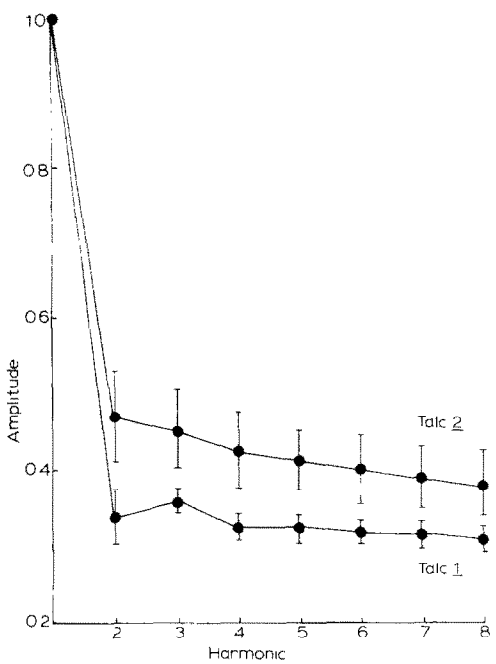

Fig. 8. Amplitude spectra. The amplitude vs harmonic number for the two samples of talc.

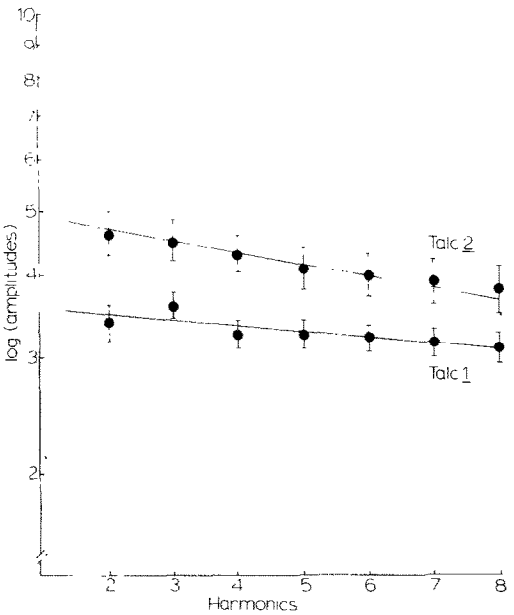

Fig. 9. Plot of Log An vs the harmonic number for the two samples of talc.

Acknowledgement

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References

Beddow, J.K., Philip, G.C. and Vetter, A.F., On relating some particle profile characteristics to the profile Fourier coefficients. Powder Technol., 18 (1977) 19–25.

Beddow, J.K. and Meloy, T.P., Advanced Particulate Morphology, CRC Press, 1980.

Beddow, J.K., Physical characterization of particulates. Powtech '77. Proceedings of the Fourth International Powder Technology and Bulk Solids Conference, International Powder Institute, 1976, p. 99.

Cooper, J., 'INERT' components of pharmaceutical preparations. Austr. J. Pharm. Sci., 7 (1976) 9–20.

Durney, T.E., Particle shape effects due to crushing method and particle shape, M.Sc.thesis, 1983, West Virginia University.

Fritz, G., Particle Recognition and Characterization Program (PRC-TN1912), Tracor Northern (1982).

Goeb, A., Particle size and particle shape of pigments and their significance. Farbe u. Lack, 57 (1951) 14–18.

Granlund, G., Fourier Preprocessing for hand print character recognition. IEEE Trans. Comput., (1972).

Gray, W.A., Packing of Solid Particles, Ch. 9, Chapman and Hall London, 1962, p. 118.

Jacobsen, A.E., Can. Chem. Process. Ind., 33 (1949) 124–130.

Jones, T.M., The influence of physical characteristics of excipients on the design and preparation of tablets and capsules. Pharm. Ind., 39 (1977) 469–476.

Jordan, B. and Nguyen, N.G., Dirt counting with microcomputer. J. Pulp and Paper Sci., 11 (1985) 73–77.

Kaye, B.H., Direct characterization of the fine particles. Proceeding of the 9th Annual Powder and Bulk Solids Conference, International Powder Institute, 1984, pp. 286–293.

Luerkens, D.W. and Beddow, J.K., Morphological Fourier Descriptors. Powder Technol, 31 (1982) 209–215.

Meloy, T.P., Hypothesis for morphological characterization of particle shape and physicochemical properties. Powder Technol., 16 (1977) 233–253.

Meloy, T.P., Systematic size-shape changes in small particles. Proceedings of the 9th annual Powder & Bulk Solids Conference, International Powder Institute, 1984, pp. 272–285.

Nguyen, N.G., Poulsen, R.S. and Louis, C., Some new color features and their application to cervical cell classification. Pattern Recognition, 16 (1983) 401–411.

Porter, S.C., Tablet coating. Drug Cosmet. Ind., 128 (1981) 46–56.

Shotton, E., Hersey, J.H. and Wray, P.E., Compaction and compression. In Lieberman and Kanig (Eds.), The Theory and Practice of Industrial Pharmacy, Lachman, 2nd edn., 1972.

Stutz, G.F.A. and Pfund, A.C., A relative method for determining particle size of pigments. Ind. Eng. Chem., 19 (1927) 51–53.