

## CHARACTERIZATION OF TALC SAMPLES FROM DIFFERENT SOURCES

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### ABSTRACT :

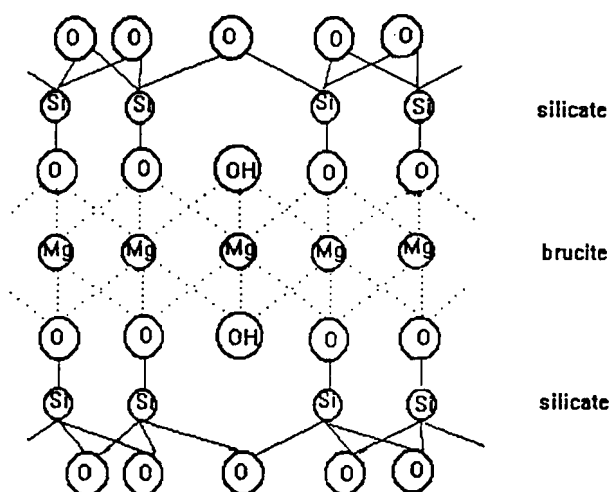
Nine different kinds of U.S.P. grade talc samples were examined for variations in density, particle size, surface area, tendency of preferred orientation, and maximum ejection force. It was found that U.S.P. grade talcs varied in physical properties. The relationships between various physical properties of talc were analyzed using the Pearson's correlation of coefficient. The results suggested that the talc sample with a higher tendency of preferred orientation has closer bulk packing and talc with lower bulk density and higher surface area requires less peak force to break the tablet/die-wall adhesion.

### INTRODUCTION :

Talc is a hydrated magnesium silicate with the empirical formula :  $\text{Mg}_6(\text{Si}_2\text{O}_5)_4(\text{OH})_4$  <sup>(1)</sup>. The structure of talc consists of layers of unit composition. Each layer contains three planes of arrangement :silicate-brucite-

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**FIGURE 1.** Cross section of the molecular structure of talc.

silicate (2) (Figure 1). The upper and lower planes are both sheets of indefinitely repeating six-membered ring silicate tetrahedra. The center plane consists of magnesium hydroxide to form the covalent bond with the oxygen atoms of upper and lower silicate planes (2). Each layer is electrically neutral and the adjacent layers are held together by van der Waals force. Talc exhibits perfect basal cleavage and has a characteristic slippery feel caused by the silicate layers sliding over one another. Since both sides of the silicate-brucite-silicate structure groups are closely packed siloxane surface, talc is known for its chemical inertness.

Natural occurrences of pure talc are relatively rare. Most deposits contain varying percentages of other minerals such as chlorite ( $5\text{MgO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2 \cdot 4\text{H}_2\text{O}$ ), magnetite ( $\text{Fe}_3\text{O}_4$ ), quartz ( $\text{SiO}_2$ ), and dolomite ( $\text{CaMg}(\text{CO}_3)_2$ ) (3). The processing procedure for talc is shown in Figure 2. The physical properties of talc may vary depending on the geographical source of the deposit, grinding process, and method of upgrading. The specific aim of this study was to examine the variations in physical characteristics of U.S.P. grade talcs.

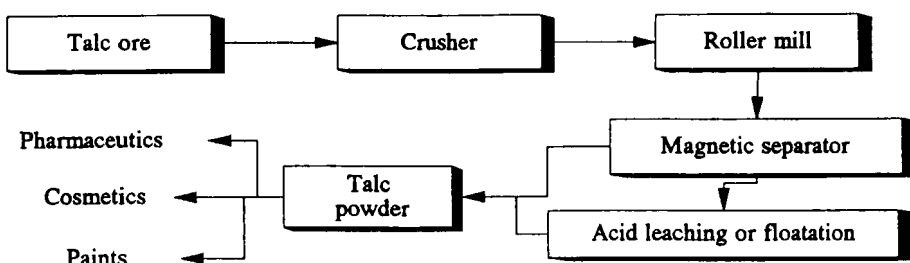


FIGURE 2. The processing flow chart for talc.

### MATERIAL :

Nine different kinds of U.S.P. grade talcs were obtained from Cyprus Industrial Mineral Company.

### EXPERIMENT :

The samples were labeled with S1 to S9. The following physical properties of the samples were determined.

#### (1) Bulk and tap density

The bulk density was determined by pouring a 30 gram sample into a 100 ml-cylindrical graduate and then measuring the volume occupied by the weight of the sample. The tapped density was determined using a Vanderkamp Tap Density Tester. The tabulated value is the average of four determinations.

#### (2) Surface area measurement :

Surface area measurements were conducted using the Autosorb-1 (Quantachrome Corp.) sorption system. The volume of nitrogen adsorbed by a degassed sample (1g) was determined at five pressures. The surface areas were then calculated using the standard BET equation<sup>(4)</sup>.

#### (3) Loss on drying :

One gram of accurately weighed talc was dried in a 105°C oven for three hours.

$$\text{Loss on drying (\%)} = \frac{\text{weight loss} \times 100}{\text{sample weight}}$$

(4) Scanning electron microscope (SEM) morphology :

The samples were sputtered with aluminum and were examined at 15 kV, 2200 magnification using a JEOL Model JSM-6300 scanning electron microscope.

(5) Particle size determination :

Particle size distribution of various samples was determined using a Microtrac Full Range Particle Size Analyzer (Leeds & Northrup 9200 series). 4 mg of talc powder were dispersed in 80 ml of methanol and the size of the fifty percentile particle was determined. Results were the average of three runs.

(6) Tendency of preferred orientation :

The X-ray diffraction (XRD) pattern of various talc samples was measured on a Siemens Diffractometer using Cu K $\alpha$  radiation and a 0.15 receiving slit. A step scan (from 17.5° to 20.5°) was made for each sample. The step size was  $2\theta = 0.02^\circ$  and the count time was 4 seconds.

XRD samples were prepared by placing approximately 20 ml of talc in a small beaker. Acetone was added (with stirring) to bring the volume of the resulting slurry to a total of 40 ml. The slurry was then poured into a 10 cm diameter culture dish and stirred again. A quartz plate was dipped horizontally into the dish and pulled out. The excess acetone was allowed to drain by holding the plate vertically and gently rocking it from end to end. The samples were then air dried. The ratio of peak intensity at  $d=4.6\text{\AA}$  (004) to the peak intensity at  $d=4.5\text{\AA}$  (020) was obtained. Results were the average of four runs.

(7) Maximum ejection force :

Powder mixtures consisting of 10% talc and 90% direct compression lactose were mixed for 5 minutes.. Tablets made from the powder mixture were compressed on a Carver press (Fred. S. Carver Inc.) at a 5000 lbs force, using a half inch punch set. The tablet weight was maintained at  $700 \pm 5$  mg. Prior to each compression, the die wall and the punches were cleaned with water and acetone and then dried to equalize the experimental condition. The maximum forces for ejecting the tablet out of the die were recorded. The tabulated value was the average of six determinations.

## **RESULTS AND DISCUSSION :**

The variation in the bulk density, tap density, median particle size, surface area, tendency of preferred orientation and loss on drying value of talc powder and the variation in maximum ejection force of the talc-lactose compact are shown in Table 1. The loss on drying value for all samples implies the nonhygroscopic nature of talc. The nitrogen adsorption isotherm of talc is a typical type II isotherm. That indicates that talc is a nonporous or macroporous adsorbent.

As shown in Table 1, sample S3 has the largest bulk density, tap density, median particle size, tendency of preferred orientation, and the lowest value for BET surface area. SEM photomicrograph (Figure 3) confirmed that the particle size distribution of sample S3 is more heterogeneous than the other samples. The smaller particles of sample S3 can fill the void space among large particles resulting in the largest bulk and tap density. The largest particle size of sample S3 may be responsible for the lower surface area.

It is of interest to analyze if there is any correlation between the primary characteristics and secondary characteristics of talc or between any two properties of talc. The Pearson product moment correlation coefficient can provide a quantitative measure of the strength of the linear relationship between two variables<sup>(5)</sup>. Therefore, a statistical program (SAS version 6) was run to search the correlation between the mean value of any two different physical properties of various talc samples. The Pearson correlation coefficients between different pairs of physical properties were listed in Table 2. It was found that surface area had a significant negative correlation with both bulk and tap density. The median particle size had a significant positive correlation with tap density and had a negative correlation with surface area. This correlation is not statistically significant at  $\alpha = 0.05$  level. From the result in Table 2, tap density is significantly correlated to all other physical properties of talc. Therefore, comparison of the tap density of different talc samples could provide a qualitative judgment for the other characteristics of talc.

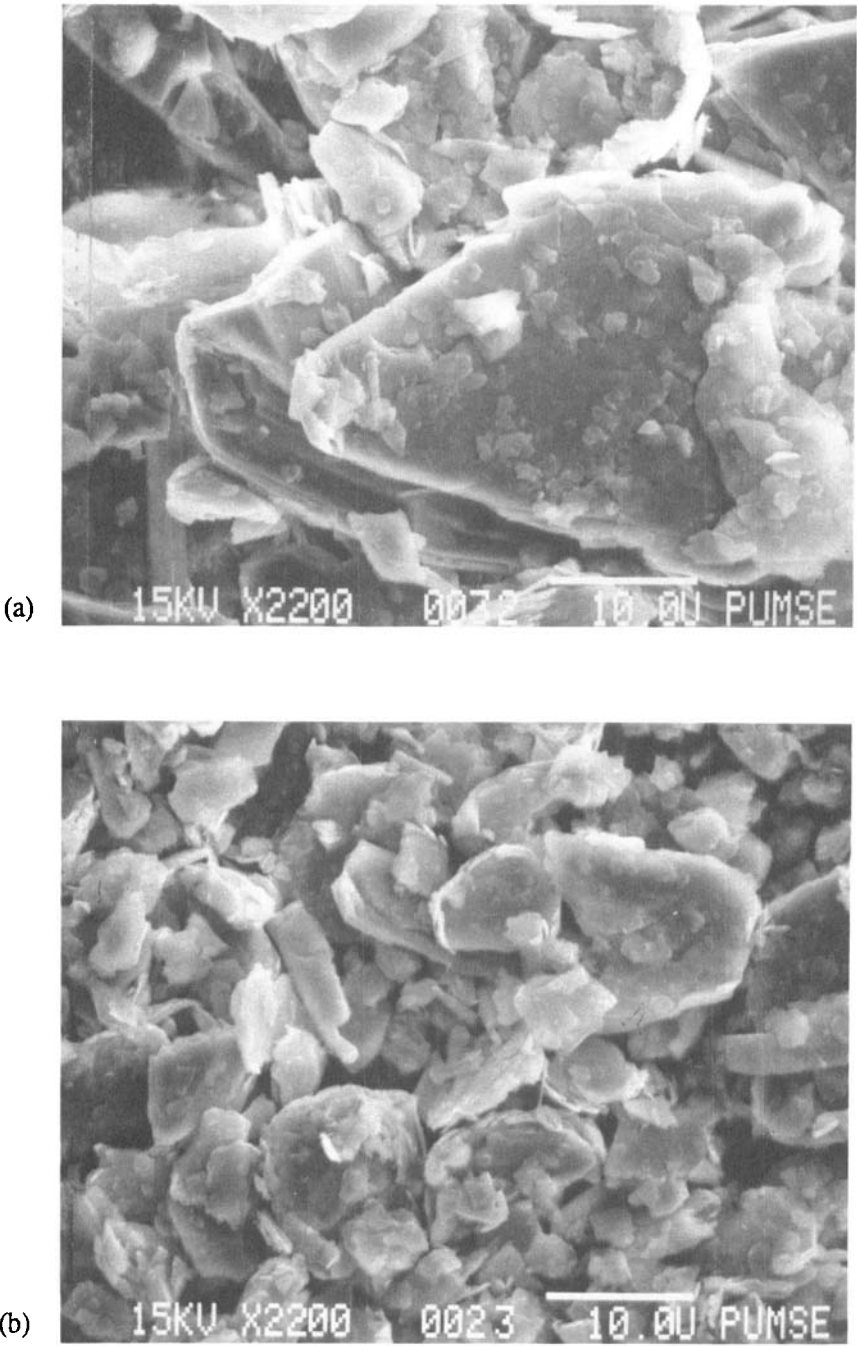
It is well known that talc exhibits different degrees of preferred orientation when examined by XRD, and the preferred orientation is due to the layered

**TABLE 1.** Physical properties of talc samples.

name	Bulk density (g/ml)	Tap density (g/ml)	Median size ( $\mu$ )	Max. ejection force (lbs)	T.P.O.	Surface area (m <sup>2</sup> /g)	Loss on drying (%)
S1	0.413 (0.012)	0.770 (0.003)	8.52 (0.01)	456.0 (11.5)	4.06 (0.25)	6.33 (0.03)	< 0.1
S2	0.347 (0.016)	0.705 (0.008)	11.4 (0.01)	423.0 (12.1)	2.94 (0.20)	7.01 (0.05)	< 0.1
S3	0.502 (0.010)	1.034 (0.010)	14.3 (0.47)	450.0 (10.0)	6.56 (0.22)	3.49 (0.24)	< 0.1
S4	0.325 (0.014)	0.705 (0.013)	11.0 (0.07)	423.0 (9.6)	3.05 (0.22)	7.21 (0.05)	< 0.1
S5	0.363 (0.015)	0.656 (0.015)	6.27 (0.08)	403.0 (5.0)	3.90 (0.22)	7.91 (0.50)	< 0.1
S6	0.395 (0.024)	0.751 (0.015)	7.65 (0.21)	434.0 (10.1)	5.21 (0.62)	5.84 (0.06)	< 0.1
S7	0.357 (0.002)	0.702 (0.004)	7.67 (0.08)	425.0 (5.0)	3.53 (0.12)	8.69 (0.17)	< 0.1
S8	0.337 (0.006)	0.645 (0.010)	7.67 (0.20)	418.0 (11.5)	2.66 (0.20)	8.12 (0.34)	< 0.1
S9	0.375 (0.010)	0.741 (0.013)	6.67 (0.13)	421.0 (11.8)	3.90 (0.70)	5.97 (0.38)	< 0.1

The value in parenthesis is standard deviation.

T.P.O. : Tendency of preferred orientation.



**FIGURE 3.** SEM photomicrograph of (a) talc sample S3, (b) talc sample S1.

**TABLE 2.** Pearson correlation coefficient between the physical properties of different talc samples.

	Bulk density	Tap density	Surface area	Median size	T.P.O.*	Max. ejection force
Bulk density	1	0.94 <u>(0.0002)</u>	-0.86 <u>(0.0027)</u>	0.49 (0.1739)	0.93 <u>(0.0003)</u>	0.73 <u>(0.0264)</u>
Tap density		1	-0.91 <u>(0.0008)</u>	0.72 <u>(0.028)</u>	0.87 <u>(0.0024)</u>	0.72 <u>(0.0291)</u>
Surface area			1	-0.60 (0.0850)	-0.84 <u>(0.0040)</u>	-0.66 <u>(0.0500)</u>
Median size				1	0.39 (0.3011)	0.503 (0.1667)
T.P.O.					1	0.58 (0.1015)
Max. ejection force						1

\* : Tendency of preferred orientation.

The value in the parenthesis was the probability of accepting the hypothesis that the population correlation coefficient ( $\rho$ ) = 0. The underlined value showed that the two variables were linearly related at  $\alpha=0.05$  significance level.



structure in which the basal planes (001) tend to lie parallel to the plane of the sample holder surface. It is the intensity of these planes that is exaggerated. It has been found that preparing the samples in a manner which will maximize the orientation will also maximize this effect. Many methods have been introduced to maximize or minimize preferred orientation in clay specimens (6). However minimizing the orientation is inherently more difficult than maximizing it, and can only be successfully done using spray dried samples (7). The sample preparation method, acetone mount, applied in this experiment can maximize the preferred orientation of talc (8). (001) and (0k0) peaks are necessary for relating the XRD data to the morphology of the talc(8). Therefore the (004) peak ( $d=4.6 \text{ \AA}$ ) and (020) peak ( $d=4.5 \text{ \AA}$ ) were chosen empirically to evaluate the tendency of preferred orientation of the talc. The higher the ratio is, the more preferred oriented packing of the talc bulk may be.

As indicated in Table 2, the tendency of preferred orientation is linearly related to the bulk, tap density, and surface area. A talc sample with a greater tendency to lie parallel to the contact surface would have a closer bulk and tap packing and a talc with a smaller surface area would tend to have a greater preferred orientation tendency.

Talc is a popular lubricant and antiadherent. The lubricating mechanism for talc is attributed to its loosely bound layers sliding over each other when placed between moving surfaces (9). In the compression of a tablet, the lubricant is expected to prevent sticking and binding to the punch faces and die wall and to reduce friction at the tablet-die wall interface during compression and ejection. Maximum ejection force in the compression of a tablet was reported to have a linear relationship with the energy consumption for ejection (10) and was used as an indicator for evaluation of lubricants (11) (12). Lower ejection force of a tablet can be expected for choosing a more efficient lubricant in the formulation. Also as shown in Table 2, the maximum ejection force is linearly proportional to the bulk density and tap density and is inversely proportional to the surface area with a statistical significance level at  $\alpha = 0.05$ . Therefore, it was suggested that a talc sample with lower bulk density and larger surface area would have a better lubricating ability.

### **CONCLUSION :**

The results of this study revealed the variations of the physical characteristics of different U.S.P. grade talc samples. Also by using the Pearson correlation of coefficient, the relationships between any two physical properties of talc were shown. It is suggested that a talc with a larger tap density would have a lower surface area, larger median particle size and tendency of preferred orientation, and have a less lubricating ability.

### **REFERENCES :**

- (1) "Handbook of Pharmaceutical Excipient" American Pharm. Assoc., Washington DC, USA, (1986)
- (2) C.A. Discher, T. Medwick, and L.C. Bailey, "Modern Inorganic Pharmaceutical Chemistry", 2nd Edition, Waveland Press Inc., Illinois, (1985)
- (3) R.W. Grexa, and C.J. Parmentier, *Cosmet. & Toiletries*, 94, 29-33 (1979)
- (4) S. Lowell, and J.E. Shields, "Powder Surface area and Porosity", 2nd Edition, Chapman & Hall, London, New York, 22 (1984)
- (5) W. Mendenhall and T. Sincich, "A Second Course in Business Statistics : Regression Analysis", 3rd Edition, Dellen Publishing Co., New Jersey, (1989)
- (6) G.W. Brindley and G. Brown Edit, "X-ray Diffraction Procedures for Clay Mineral Identification", Mineral Society, London, 310, (1980)
- (7) D.L. Bish and J.E. Post, Edit "Modern Powder Diffraction" The Mineralogical Society of American, Washington DC Vol. 20, (1990)
- (8) M. J. Murtagh and H.J. Holland, Internal report, Corning Incorp. (1989)
- (9) S. Dawoodbhai and C.T. Rhodes, *Drug Dev. & Ind. Pharm.*, 16, 2409-2429, (1990)
- (10) Y. Matsuda, Y. Minamida, and S.I. Hayashi, *J. Pharm. Sci.*, 65, 8, 1155-1160, (1976)

- (11) B. Mechttersheimer and H. Sucker, Pharm. Technol. Feb., 38-50, (1986)
- (12) E. Nelson, S.M. Naqvi, L.W. Busse, and T. Higuchi, J. Am. Pharm. Assoc. Sci. Ed., 43, 596-602, (1954)