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EVALUATION OF BATCH-TO-BATCH AND MANUFACTURER-TO-MANUFACTURER VARIABILITY IN THE PHYSICAL PROPERTIES OF TALC AND STEARIC ACID

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ABSTRACT

Magnesium stearate, talc and stearic acid are commonly used as lubricants in tablet formulations. Many studies on the batch-to-batch and manufacturer-to-manufacturer variability in the physical properties and lubricity of magnesium stearate have been reported in the literature. However, very few similar studies have been reported on talc or stearic acid. In this study, physical properties such as particle size, specific surface area, thermal behavior, moisture content, density, and particle shape and morphology of talc and stearic acid batches obtained from several manufacturers were examined. There was little batch-to-batch variability observed in the talc and stearic acid batches obtained from various manufacturers; however, differences in the particle size and specific surface area were seen in the two types of talc, USP samples obtained from one manufacturer. The scanning electron microscope photomicrographs of the stearic acid samples obtained from various manufacturers also showed some differences in the amount of flakes attached to the spherical particles.

INTRODUCTION

The physical properties of lubricants such as magnesium stearate, stearic acid and talc can affect their performance in tablet and capsule formulations. Many studies have been reported on the batch-to-batch and manufacturer-to-manufacturer variability in the physical properties of magnesium stearate(1,2,3). These studies showed that magnesium

stearate obtained from different manufacturers varied greatly in its physical properties and lubricant efficacy. However, very few such studies have been reported on stearic acid or talc(4). Therefore, the objective of this study was to evaluate the batch-to-batch and manufacturer-to-manufacturer variability in the physical properties of talc and stearic acid batches obtained from various manufacturers and suppliers. The physical properties tested in this study include density, particle size, specific surface area, thermal behavior, moisture content, and particle shape and morphology.

MATERIALS

Talc, USP samples from three batches each were obtained from Penta Manufacturing Co., Fairfield, NJ, and Whittaker, Clark & Daniels Inc., South Plainfield, NJ. PurTalc 6030 BC-USP grade and Sugarloaf Talc USP samples from two batches each were obtained from Charles B. Crystal Co., New York, NY. Samples of stearic acid, NF were obtained from J.T. Baker Inc., Phillipsburg, NJ; Amend Chemical Co., Irvington, NJ; and Witco Corp., Memphis, TN.

METHODS

The bulk density was measured by filling a known weight of sample into a 10 mL graduate and observing the volume occupied by the sample. Using a tap density apparatus(5), the graduate was then tapped 1000 times for stearic acid and 2000 times for talc. The bulk and tapped densities were calculated from the weight and volume values. One measurement per batch was done.

The true density was determined using a helium pycnometer(6). All samples were run in duplicate.

The particle size of the samples was analyzed using a light scattering particle size analyzer(7). The talc samples were dispersed in 0.1% W/W polysorbate 80 in water. A 1% W/W solution of polysorbate in water was used as the dispersion medium for the stearic acid samples. The talc and stearic acid samples were analyzed using lenses with focal lengths of 100 mm and 600 mm, respectively. All samples were measured in duplicate.

The specific surface area was determined using a BET surface area analyzer(8). The talc samples were degassed at 40°C for 1 hour and the stearic acid samples were degassed at room temperature for 1 hour. Single point BET measurements were made using a mixture of 30% nitrogen and 70% helium. Duplicate measurements were made.

TABLE 1 - Data on Physical Properties of Talc, USP

Source	Batch No.	SSA (m^2/g)	GMD (μ)	Density(g/mL) Bulk	Tapped	True
WCD, Inc.	X1550-001	7.4	9.4	0.48	0.78	2.81
	W0162-002	8.4	9.9	0.43	0.77	2.84
	X0639-001	8.7	11.3	0.44	0.74	2.82
Penta	27110	7.9	9.5	0.43	0.74	2.81
	23252	7.9	9.5	0.42	0.71	2.82
	26441	7.9	9.8	0.42	0.73	2.84
CBCrystal PurTalc	BO-122	10.0	10.6	0.45	0.74	2.81
	BO-534	10.0	11.1	0.45	0.74	2.80
Sugarloaf	3622	3.5	18.3	0.42	0.78	2.80
	3818	3.7	17.7	0.41	0.79	2.84

Thermal gravimetric analysis(9) was performed on one sample from each manufacturer. Using a scan rate of 10°C per minute, the talc samples were heated over a range of 30 - 300°C. The stearic acid samples were heated at 10°C per minute from 20°C to 90°C.

The melting point was determined by differential scanning calorimetry(10) using aluminum pans. One batch of talc from each manufacturer was analyzed at a scan rate of 20°C per minute over a range of 30 - 300°C. All stearic acid samples were heated at a rate of 10°C per minute over a range of 30 - 70°C. One batch of stearic acid frrom each manufacturer was also tested at a scan rate of 2°C per minute.

The particle shape and appearance were observed using a scanning electron microscope(11). Pictures of samples were taken at x1000 magnification for the talc samples and x750 magnification for the stearic acid samples.

RESULTS AND DISCUSSION

The specific surface area(SSA), geometric mean diameter(GMD), and bulk, tapped and true density data for the various talc and stearic acid batches are summarized in Tables 1 and 2 respectively.

TABLE 2 - Data on Physical Properties of Stearic Acid, NF

Source	Batch No.	SSA (m^2/g)	GMD (μ)	Density(g/mL) Bulk	Tapped	True
Baker	A49347	1.8	47.6	0.39	0.48	0.98
	C22339	1.6	44.4	0.39	0.47	0.98
	D05334	1.9	43.8	0.41	0.48	0.98
Amend	H20431B25	1.3	44.7	0.38	0.53	0.99
	H20431B10	1.3	45.8	0.46	0.54	0.98
	H18042A25	1.2	49.3	0.43	0.54	0.98
Witco	8V4681	1.3	56.8	0.38	0.52	0.98
	OU4230	1.1	54.2	0.45	0.53	0.99
	7X6776	1.7	49.8	0.39	0.48	0.97

TABLE 3 - Melting Point and Heat of Fusion Data for Stearic Acid, NF

Source	Batch No.	Onset Temp. (°C)	Max. Temp. (°C)	Heat of Fusion (J/g)
Baker	A49347	54.7	57.0	194.7
	C22339	54.4	56.7	197.6
	D05334	54.2	56.2	196.5
Amend	H20431B25	52.8	55.6	191.9
	H20431B10	52.2	55.4	193.2
	H18042A25	52.2	55.2	189.2
Witco	8V4681	53.9	56.6	193.0
	OU4230	53.3	56.1	192.5
	7X6776	54.7	56.9	198.6

(Data obtained from DSC runs at a scan rate of 10°C per minute)

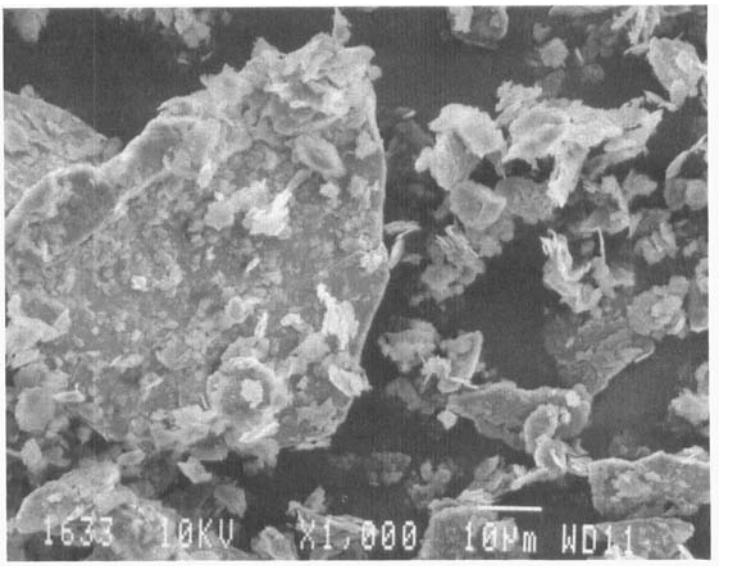
The melting point and heat of fusion values for the stearic acid samples are shown in Table 3.

The data on talc, as shown in Table 1, indicate that there was only a small batch-to-batch variability in the physical properties of batches from the same manufacturer. Also, the data are very similar for the batches obtained from Whittaker, Clark & Daniels Inc. and Penta manufacturing Co. However, the specific surface area and geometric mean diameter values for the Sugarloaf talc, USP batches obtained from Charles B. Crystal Co. were significantly different from those obtained for the PurTalc, USP batches from the same manufacturer. The specific surface area values for the Sugarloaf batches were significantly smaller than those for the PurTalc batches. Consistent with these data, the geometric mean diameter values for the Sugarloaf batches were higher than those for the PurTalc batches.

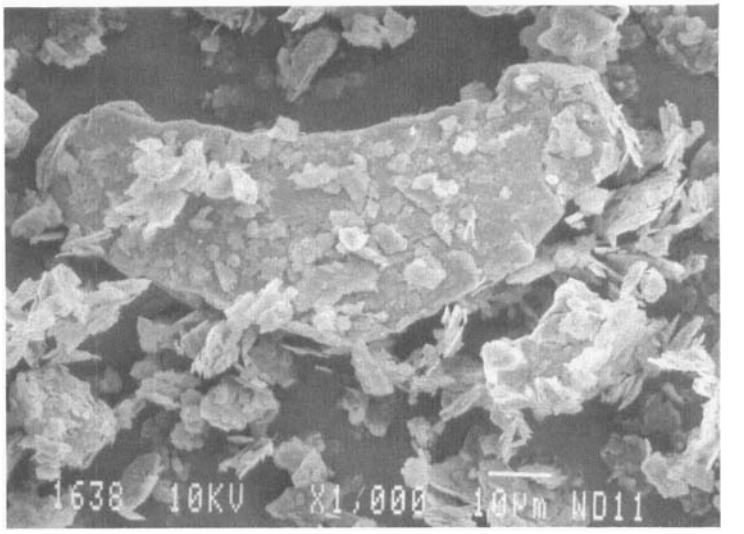
Thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) runs were performed on one batch of talc from each manufacturer. The TGA thermograms did not show any weight loss for any of these batches. Similarly, the DSC runs showed no endotherms for these batches.

The SEM photomicrographs for one batch of talc from each manufacturer are shown in Figures 1 and 2. All talc samples except the Sugarloaf talc appear similar and can be characterized as a mixture of various sized, laminated flakes and aggregates of flakes. The range of sizes (widths) for the flakes was approximately 2 - 5 microns for the individual flakes and 50 -150 microns for the aggregates. No discernible difference between manufacturers or batches was obvious among these samples. The only unique sample was the Sugarloaf talc batches with flakes that were consistently larger in size. Most of the individual flakes were approximately 40 - 60 microns wide, and these batches also showed fewer aggregates. Based on these observations and the specific surface area and geometric mean diameter data, it appears that the Sugarloaf talc batches were not as finely ground as the other samples. It is also likely that the larger flakes of the Sugarloaf talc resisted aggregation due to their size.

The data on stearic acid as shown in Tables 2 and 3 show very little batch-to-batch or manufacturer-to-manufacturer variability. The TGA thermograms showed no weight loss for batches from the three manufacturers. The DSC thermograms at a scan rate of 10°C per minute for the Baker and Witco samples were similar, however, the Amend batches showed a small shoulder at the beginning of the endotherm. To investigate this further, samples from one batch each from the three manufacturers were run at a scan rate of 2°C per minute. These thermograms are shown in Figure 3. The slower scan rate gave a little better resolution of the shoulder observed in the endotherm for the Amend sample. The significance of this difference in the endotherms is difficult to interpret at this time.



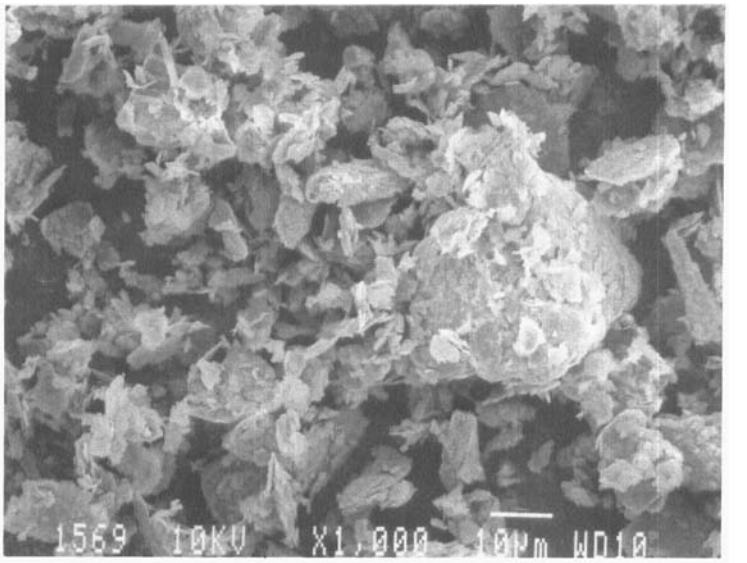
Whittaker, Clark & Daniels, Batch No. X1550-001



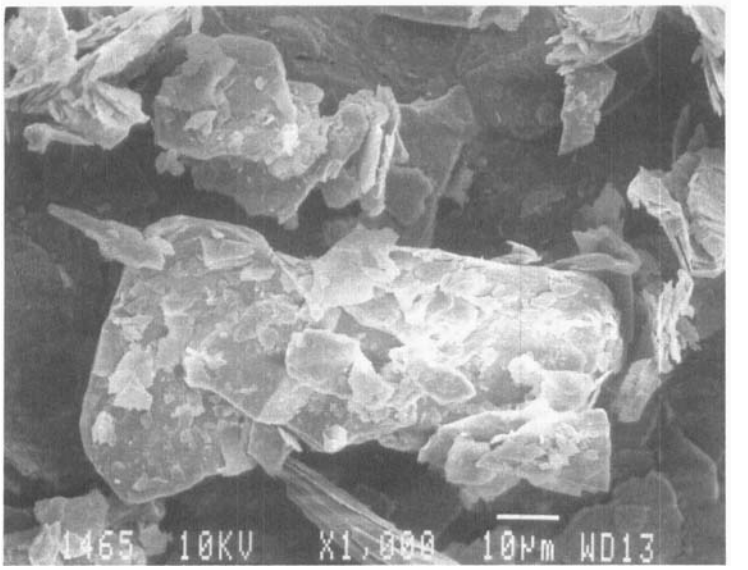
Penta Manufacturing Co., Batch No. 27110

FIGURE 1

SEM Photomicrographs for Talc, USP Samples from Whittaker, Clark & Daniels, Batch No. X1550-001 and Penta Manufacturing Co., Batch No. 27110



PurTalc, USP, Batch No. BO-122



Sugarloaf Talc, USP, Batch No. 3622

FIGURE 2

SEM Photomicrographs for Talc, USP samples from Charles B. Crystal Co.; PurTalc, USP, Batch No. BO-122 and Sugarloaf Talc, USP, Batch No. 3622.

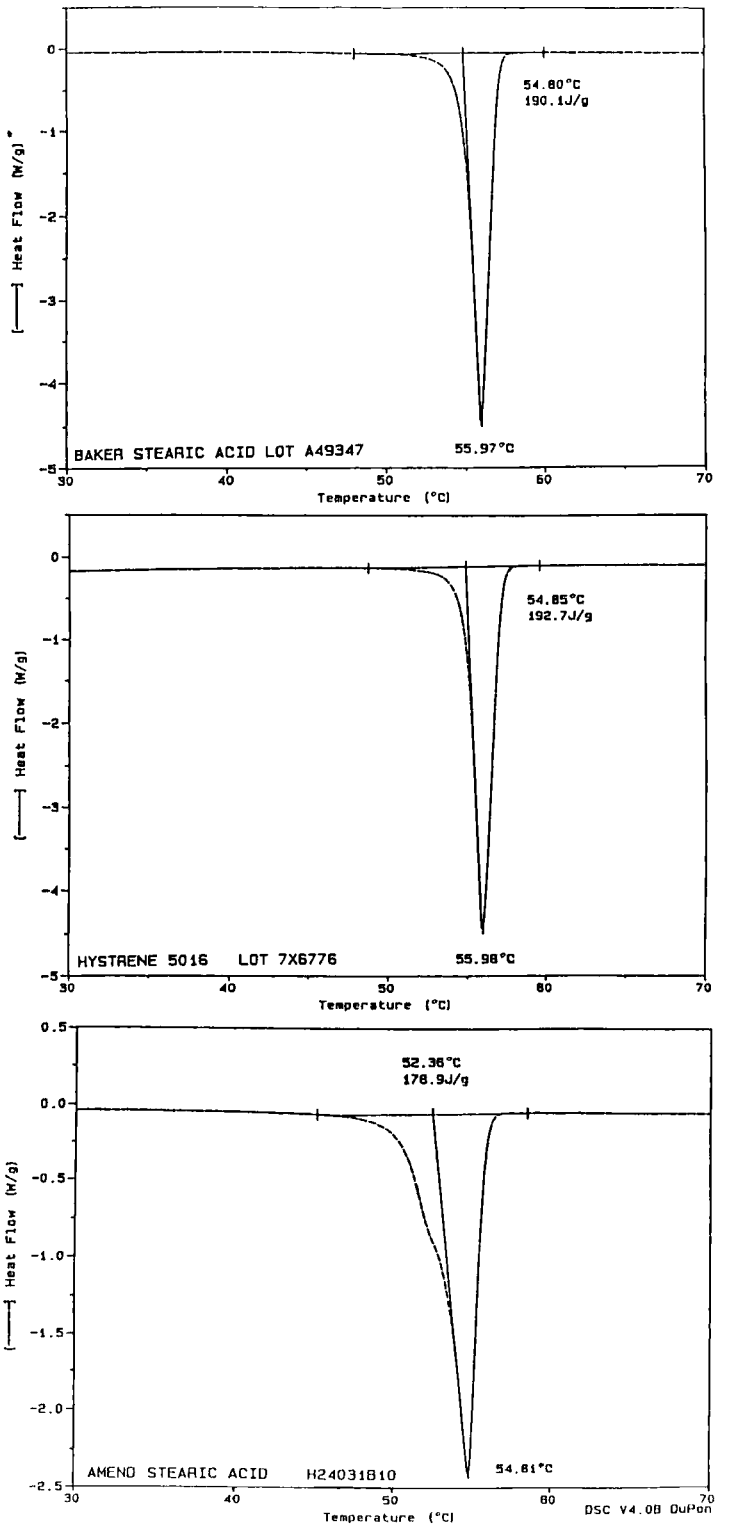
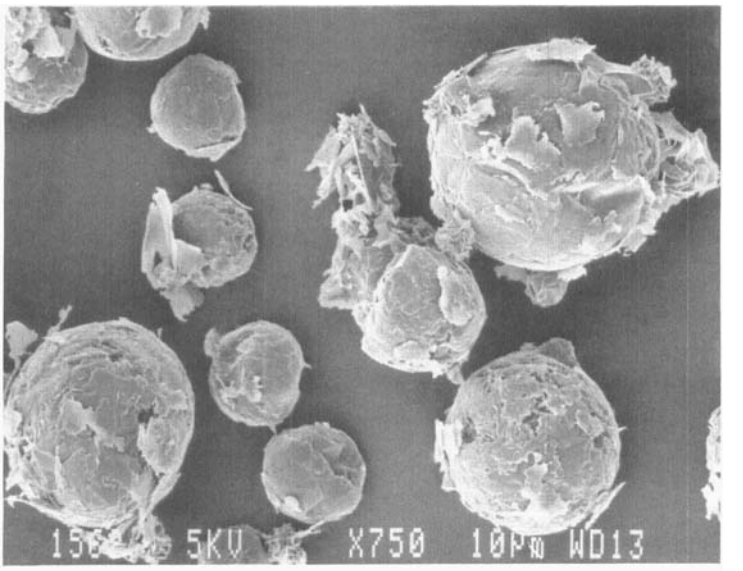
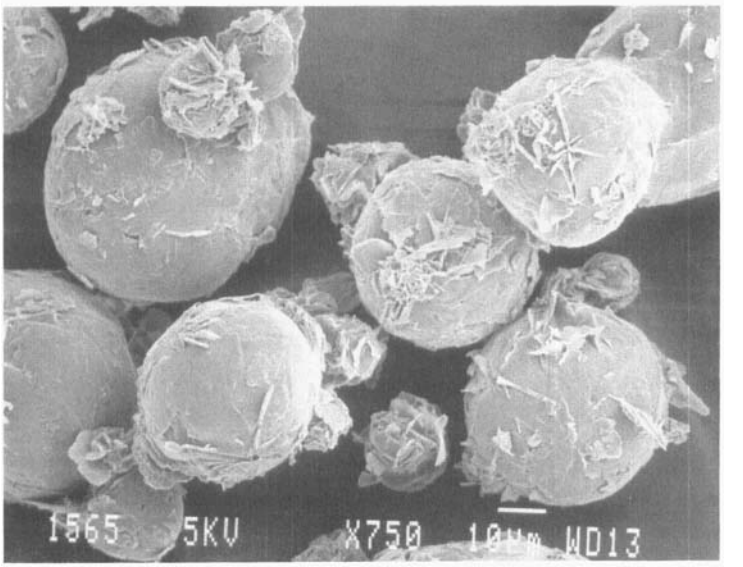


FIGURE 3

DSC Thermograms (2°C/min) for Stearic Acid, NF from J.T. Baker, Batch No. A49347, Witco Corp., Batch No. 7X6776 and Amend Chemical Co. Batch No. H24031B10



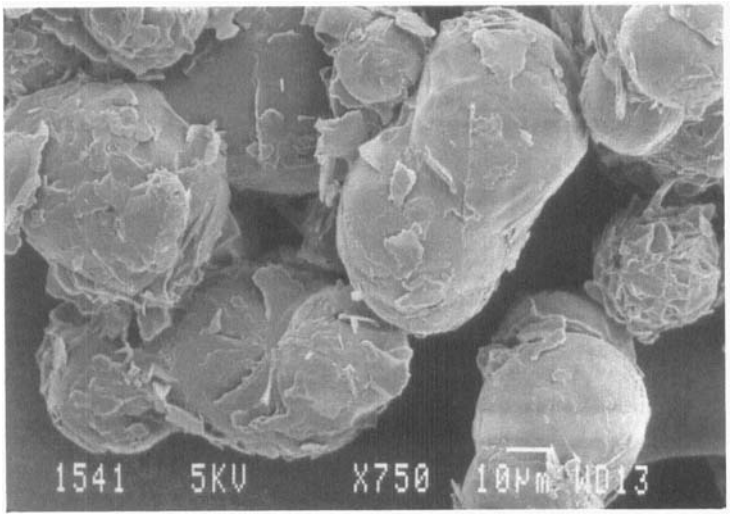
Stearic Acid, NF, J.T. Baker, Batch No. D05334



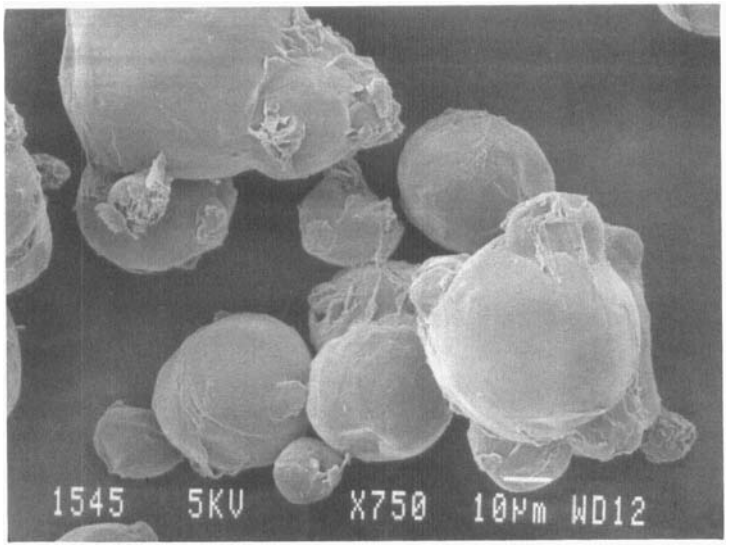
Stearic Acid, NF, J.T.Baker, Batch No. C22339

FIGURE 4

SEM Photomicrographs for Stearic Acid, NF from J.T. Baker, Batch Nos. D05334 and C22339



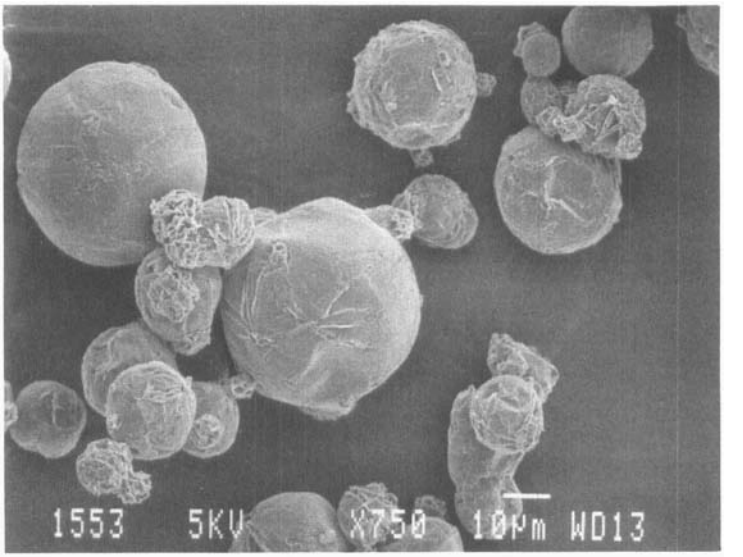
Stearic Acid, NF, Witco Corp., Batch No. 7X6776



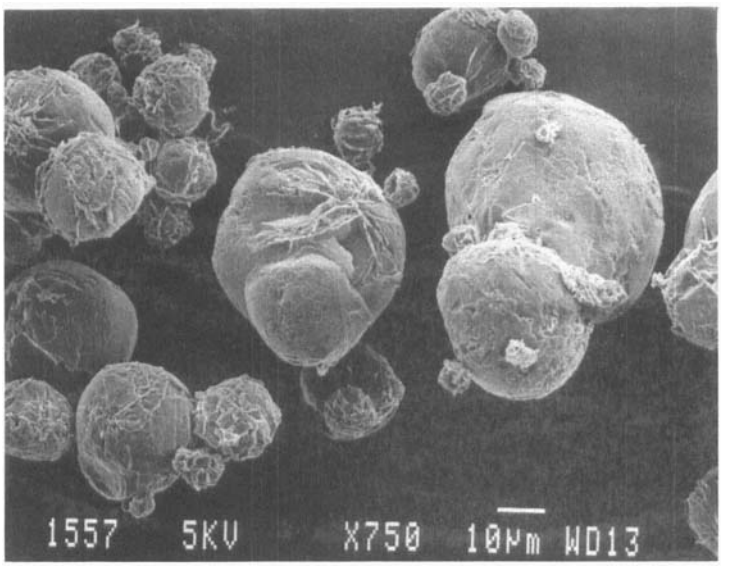
Stearic Acid, NF, Witco Corp., Batch No. 8V4681

FIGURE 5

SEM Photomicrographs for Stearic Acid, NF from Witco Corp. Batch Nos. 7X6776 and 8V4681



Stearic Acid, NF, Amend Chem. Co., Batch No. H20431B25



Stearic Acid, NF, Amend Chem. Co., Batch No. H20431B10

FIGURE 6

SEM Photomicrographs for Stearic Acid, NF from Amend Chemical Co., Batch Nos. H20431B25 and H20431B10

The SEM photomicrographs for the stearic acid samples (two batches from each manufacturer) are shown in Figures 4, 5, and 6, respectively. Although all samples were composed of various sized spheres, there were differences in the textures or amount of flakes present on the surface of the spherical particles. The Witco batch 7X6776 showed a moderate amount of flakes attached to the surface of the stearic acid spheres, whereas Witco batch 8V4681 exhibited much smoother spheres with fewer flakes attached. The surface of the spheres from the Amend batches was more finely textured than those from the other two manufacturers. Also, no surface flakes were observed on these samples. No obvious differences were observed among the three Amend batches. The most surface flakes were observed in the batches from J.T. Baker, Inc. with batch D05334 spheres showing more flakes attempting to peel off from the spheres, giving the spheres a rougher texture than those from batch C22338. Brittain et al.(12) in a recent publication reported that the rounded stearic acid particles were superior to the angular flakes in their lubricant property. The significance of these differences in the amount of flakes present on the stearic acid spheres and their effect on the lubricating efficacy is difficult to interpret at this time, but will be examined in a future study in our laboratory.

CONCLUSION

Overall, only a small batch-to-batch variability was observed for the various talc and stearic acid samples investigated in this study. However, the Sugarloaf talc, USP batches exhibited significantly larger particle size as compared to the other talc, USP batches. This difference could have an effect on its glidant properties. The stearic acid samples, although generally similar in their physical properties, showed differences in the amount of flakes on the surface of the spheres which could affect their lubricant efficacy.

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