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Effect of Stearic Acid Particle Size on Surface Characteristics of Film-Coated Tablets

N. H. Shah,* C. I. Patel, M. H. Infeld, R. J. Margolis, A. M. Railkar, and A. W. Malick

Pharmaceutical Research and Development, Hoffmann-La Roche, Inc., Nutley, NJ 07110

ABSTRACT

Raw material specifications are vital for many excipients used in the manufacture of tablets. Stearic acid powder is widely used in the pharmaceutical industry as a tablet lubricant. This study shows that the variation in particle size of stearic acid not only affects die wall lubrication properties (ejection force) but also affects surface characteristics of film-coated tablets. A coarser grade of stearic acid can dislodge from tablet surfaces during the film-coating process leaving pit marks, whereas a finer grade of stearic acid (less than 100 mesh) results in film-coated tablets having very smooth surfaces. The mechanism of pitting on the tablet surface is described. A specification for stearic acid particle size to overcome this problem is suggested.

*To whom correspondence should be addressed.

INTRODUCTION

The industrial production of tablets has been part of pharmaceutical practice for more than 100 years. The formulation and production of tablets has yet remained complex. Selection of appropriate raw materials in the tablet formulation is vital to maintain its efficiency, identity, potency, purity, quality, stability, and safety of the patients. Variations in raw material can occur due to variations: (i) among suppliers of the same material, (ii) in batches from the same supplier, and (iii) within a batch. The importance of standardizing excipient tests, methods, and specifications to minimize the effect of excipients on the desired properties of the dosage forms is well recognized, and significant progress has been made by corporations (1), pharmacopeial authorities (2–4), and International Pharmaceutical Excipients Council (IPEC) (5). Raw material characterization often takes place in a problem-solving situation. When this happens, developmental projects or production schedules are delayed until the problem is identified and resolved (6,7). The variability in the physical properties of talc and stearic acid from batch to batch and manufacturer to manufacturer is reported by Phadke et al. (8). This report describes the effect of stearic acid particle size not only on the lubrication properties of compressed tablets but also on the surface characteristics of film-coated tablets.

EXPERIMENTAL

Raw Materials

Lactose anhydrous DTG (DTG = direct tableting grade—Sheffield Products, Division of Quest international Inc. (Norwich, NY)
Microcrystalline cellulose, Avicel PH-102 (FMC Corporation, Philadelphia, PA)
Stearic acid (Humko Chemical, Division of Witco Corporation, Memphis, TN)
Stearic acid (Mallinckrodt, St. Louis, MO)
Stearic acid (Emery Industries, Inc., Cincinnati, OH)
Opadry Red and Starch 1500 (Colorcon Inc., West Point, PA)

Equipment

Accela-cota 24 in. (Thomas Engineering Inc., Hoffmann Estates, IL)
Spray system (Graco Inc., West Caldwell, NJ)
Optical microscope (Carl Zeiss, Germany)
Instrumented single-station tablet press (Hoffmann-La Roche, Inc., Nutley, NJ)
Sieve analyzer (Alpine Engineering, Augsburg, Germany).

Particle Size Determination of Various Grades of Stearic Acid

The particle size of each grade of stearic acid was determined using an Alpine Air Jet Sieve A200LS; 10 g of sample was used in the analysis.

MANUFACTURE OF TABLETS

Tablet Formulation

Lactose anhydrous DTG, microcrystalline cellulose, and Starch 1500 were mixed in a planetary mixer for 10 min. Stearic acid was added to this powder mixture and mixed for 5 min. The tablets were compressed on a single-punch instrumented tablet press (F Press), and the compression versus ejection profile for each grade of stearic acid was determined. The tablet formulation is shown in Table 1.

Table 1

Tablet Formulation

Material	% (w/w)
Lactose anhydrous DTG	65
Avicel PH-102	19
Starch 1500	15
Stearic acid	1
	100

Film Coating of Tablets

The tablets were film coated using the film-coating formulation shown in Table 2.

The following in-process conditions were used during the film-coating process:

Inlet temperature	85°C
Bed temperature	45°C
Spray nozzle	1.2 mm
Pump pressure	60–80 psig
Fluid pressure	40 psi
Atomizing air pressure	12–15 psi
Rotation of pan	Continuous
Spray rate	50–60 ml/min
Amount of coating applied	~3% of tablet weight

Table 2

Film-Coating Formulation

Material	% (w/w)
Opadry Red	15
Water	85
	100

Evaluation of Tablet Surfaces

Tablet surfaces were evaluated for pit marks using an optical microscope at 125 magnification.

RESULTS AND DISCUSSION

The particle size distribution of each grade of stearic acid investigated is shown in Figs. 1 and 2. Humko and Mallinckrodt stearic acid powders were finer than Emery stearic acid powder. In the case of Humko and Mallinckrodt powders, >90% of stearic acid was finer than 100 mesh (U.S. Std.). However, <40% of Emery stearic acid was finer than 100 mesh (U.S. Std.).

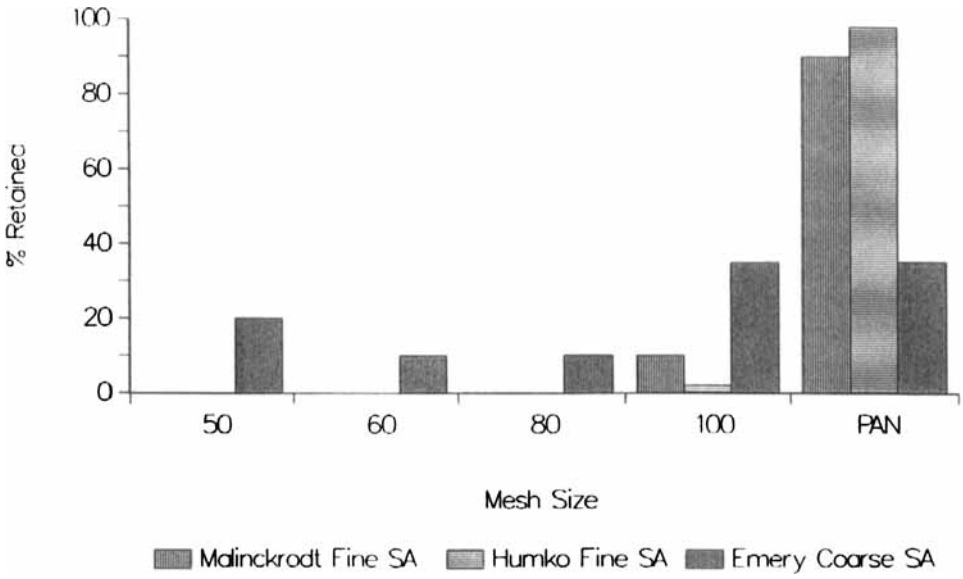

Figure 1. Particle size distributions of various grades of stearic acid. (SA = stearic acid).

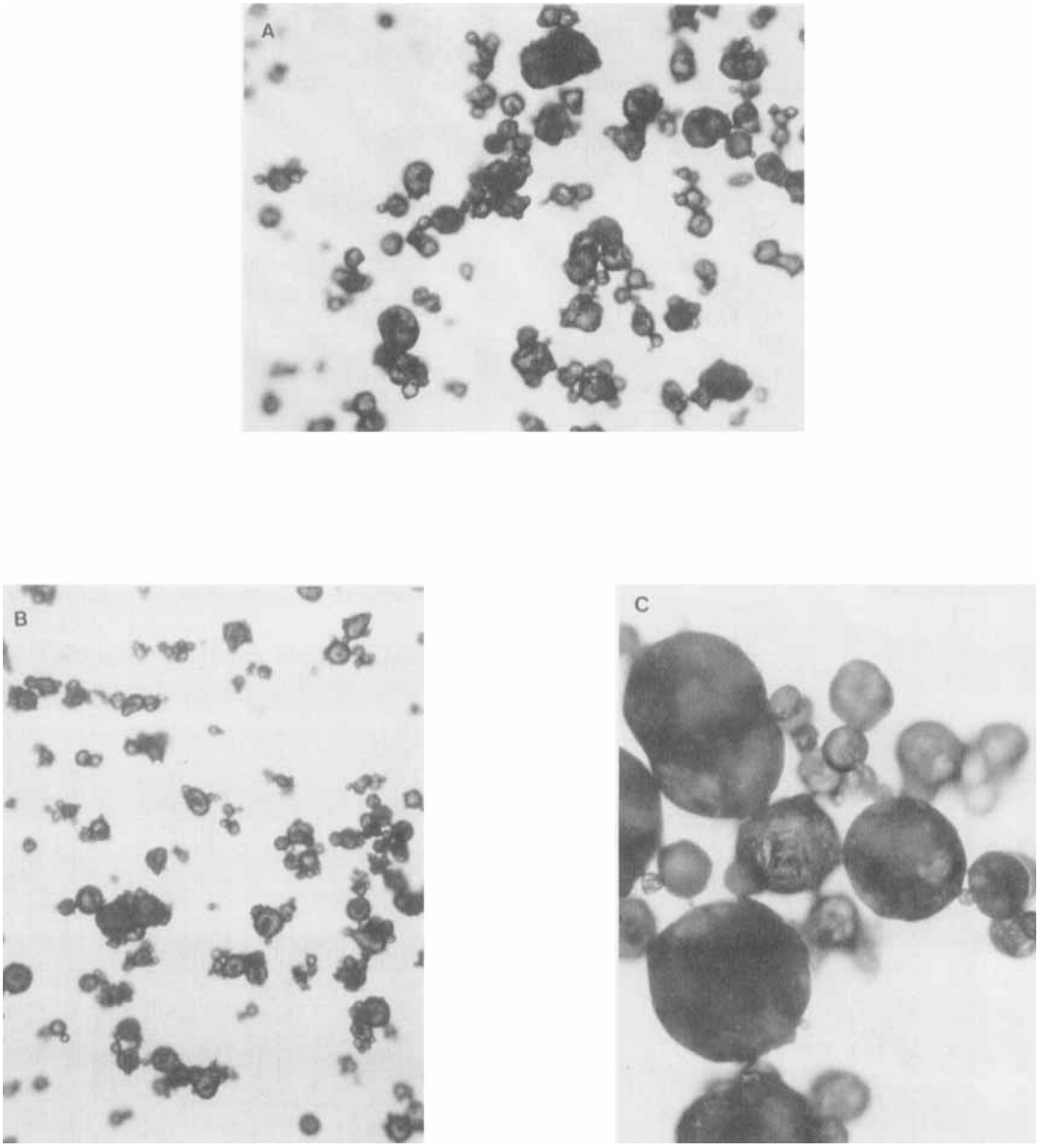

Figure 2. Photomicrographs showing the particle size distribution of various grades of stearic acid. (A) Mallinckrodt fine stearic acid; (B) Humko fine stearic acid; (C) Emery coarse stearic acid.

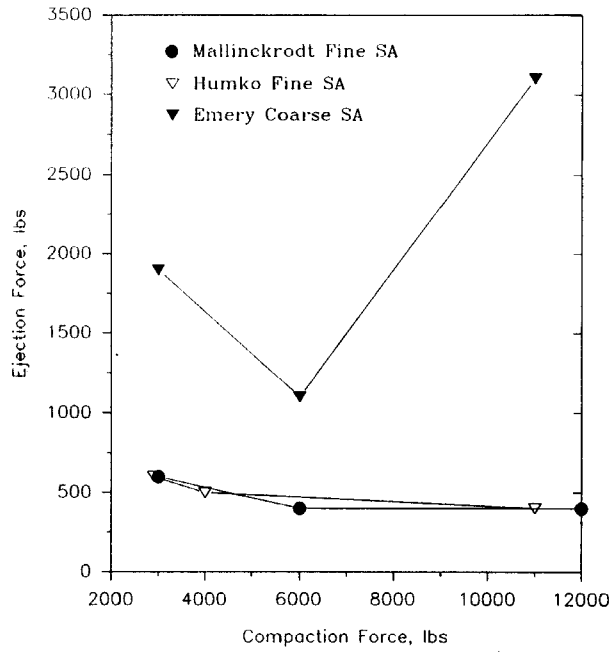

Figure 3. Compaction force versus ejection force profile of tablets containing 1% stearic acid. (SA = stearic acid).

The compression versus ejection profile of placebo tablets prepared using the various grades of stearic acid is shown in Fig. 3. The data clearly indicate that a finer grade of stearic acid (Humko and Mallinckrodt) exhibits superior lubrication properties compared to a coarser grade of stearic acid (Emery). During mixing, the finer grade of stearic acid produces a partial coating on the tablet granulation which reduces friction between the powder granules and improves lubricity. The coarser grade of stearic acid does not provide any significant coating, resulting in poor lubrication.

The photomicrographs of the film-coated tablets were quite interesting (Fig. 4). The tablets produced with the coarser grade of stearic acid resulted in surfaces with pit marks; those produced with a finer grade of stearic acid had very smooth surfaces. When the tablet bed is heated during coating, the tablets undergo relaxation, and the finer grade of stearic acid distributes evenly onto the tablets, producing smooth surfaces. However, the tablets produced with the coarser grade of stearic acid have surfaces with pit marks due to dislodging of the larger stearic acid particles during relaxation. They do not get evenly or homogeneously distributed onto the tablet surface. The particle size specification for stearic acid to overcome pitting problems is presented in Table 3.

SUMMARY

The effect of stearic acid particle size on the lubrication properties of compressed tablets, as well as on the surface characteristics of film-coated tablets, is presented. This study clearly shows the effect and impor-

Table 3

Particle Size Specifications for Stearic Acid

1. Appearance	1. Fine powder
2. Particle size	2. Minimum 95% through 80 mesh (U.S. Std.) Minimum 90% through 100 mesh (U.S. Std.)

Testing procedure for particle size: Use an Alpine Air Jet Sieve A200LS or equivalent and a 10-g sample for each screen.

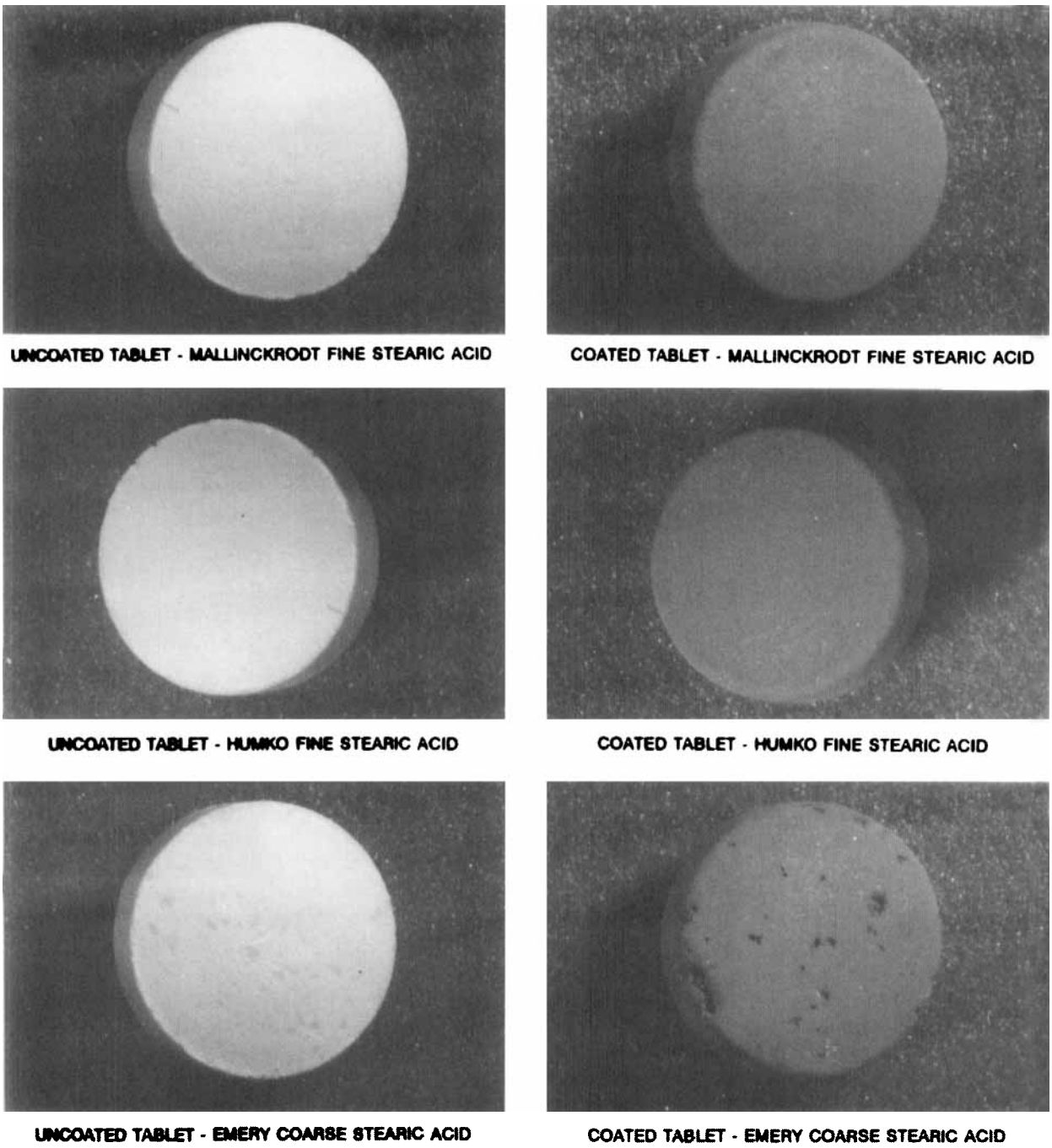

Figure 4. Photomicrographs of uncoated and film-coated tablets produced using stearic acid from Emery, Mallinckrodt, and Humko.

tance of raw material specifications in determining the performance of tablets. The raw material characterization should be a part of routine, planned activity in overall product development.

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