MANUFACTURE AND CHARACTERISATION OF MAGNESIUM STEARATE AND PALMITATE POWDERS OF HIGH PURITY

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Magnesium stearate is used extensively as a lubricant in tablet formulation. Commercial samples vary in both chemical (Pilpel 1971) and physical character (Butcher & Jones 1972) and have an unpredictable effect on formulations (Billany 1981). Here we have made and characterised pure samples of magnesium stearate (M.S.) and its major commercial contaminant, magnesium palmitate (M.P.). This knowledge is required for the identification of a specific lubricant feature in commercial samples of magnesium stearate.

We manufactured two batches each of magnesium stearate (A&B) and magnesium palmitate (C&D) by a precipitation method. The pH was controlled to enable either plate or needle-like crystals to be produced (Müller 1977). The existence of these crystal forms was verified by electron microscopy. The chemical composition of the samples was assessed using a gas chromatography technique and a moisture evolution analyser (Du Pont 903), and powder surface area by nitrogen adsorption.

Table 1. Some physicochemical properties of prepared magnesium stearate and palmitate.

Sample	pH of preparation	crystal form	surface area $m^2 g^{-1}$	chemical composition: Mg salt of fatty acid % w/w	chemical composition: moisture % w/w
M.S. (A)	10.1 - 6.9	needle	4.17	92.56	5.16
M.S. (B)	6.5 - 5.1	plate	1.57	94.48	5.83
M.P. (C)	9.7 - 6.9	needle	2.99	90.81	6.21
M.P. (D)	6.6 - 5.7	plate	1.43	86.61	6.32

All prepared samples have a high degree of purity. The needle-like crystal form has a higher surface area than the plates for both materials. Interestingly, the moisture content figures do not show the proposed discrete hydration for the two crystal forms (Müller 1977; Butcher 1973) which suggests irregular water incorporation into the crystal lattice during manufacture.

Differential scanning calorimetry, (Du Pont 1090/910) at $10^{\circ}C\ min^{-1}$ under a nitrogen gas stream, showed all four untreated samples to have different thermal profiles with the major endotherm attributed primarily to water loss. Profiles were simplified and more reproducible when the powders were dried for 2 h at $104^{\circ}C$ (see Fig. 1). The differential thermogram of the dried needle-like form (A) is discernible from that of dried plate-like crystals (B) by the presence of an additional small endothern at $127^{\circ}C$.

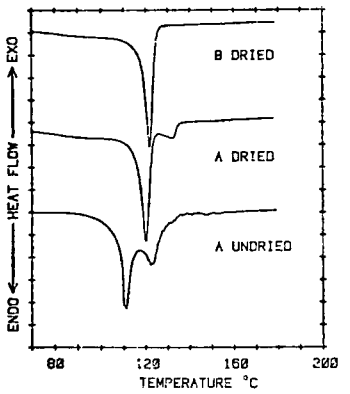

Fig. 1. Differential thermograms for magnesium stearate needle-like crystals (A) and plates (B). Drying, 2 h at $104^{\circ}C$.

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