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A comparative study of the flow enhancing properties of bentonite, magnesium stearate, talc and microcrystalline cellulose

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Abstract

A comparative study of granule flow enhancing property of bentonite, magnesium stearate, talc and microcrystalline cellulose (MCC) was undertaken. Bentonite was processed into fine powder. A 10 %w/w of starch granules was prepared and separated into different sizes (<180, 180-500, 500-710 and 710-850 µm). Each granule size was divided into four parts. The four parts were treated with four different glidants (bentonite, magnesium stearate, talc and MCC) at different concentrations (0, 0.5, 1, 1.5 and 2 %w/w). The resulting granules were subjected to the following analyses: bulk and tapped densities, Hausner's ratio, angle of repose and flow through an orifice. Results show that all the glidants were comparable at each of the concentrations and granule sizes, except at 710-850 µm granule size with magnesium stearate exhibiting superior flow properties to talc, followed by MCC and lastly bentonite, in that order. Bentonite powder was comparable as a glidant to magnesium stearate, talc and microcrystalline cellulose in the concentrations used in this study except at granule size of 710-850 µm. It can therefore be recommended as a suitable glidant in granule formulations where particle size is below 710 µm.

Keywords: Bentonite; Flow property; Glidant; Lubricant

INTRODUCTION

The preparation of essentially many dosage forms involves the handling of solid materials. Among all finished products, solid dosage forms are the most predominant in terms of volume and value. The importance of solid-handling properties, especially flow properties, cannot be overemphasized. The flow properties of solids have great impact on the tableting and encapsulation processes since these dosage form manufacturing processes require the flow of powder materials from storage container to filling stations such as tablet dies or capsule filler. Some in-process steps in the manufacturing industry depend on rapid powder flow; these include mixing and de-mixing of powders, uniformity of die fill, etc. The speed of production which depends on these processes will be influenced by the formulation's flow characteristics. In the final analysis, weight and content uniformity, hardness and disintegration/ dissolution of the final product are affected (VonBehren, 1996).

Some of the major factors that affect the flow properties of powders include particle size and size distributions, particle shape and surface characteristics, moisture

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content, surface forces, porosity and density (Fitzpatrick, *et al*., 2004; Fitzpatrick and Ahrne, 2005; Kaye, 1997; Heng and Staniforth, 1987; York and Pilpel, 1972; Pilpel and Britten, 1979). Different flow properties are required at different stages of processing and these factors should be carefully taken into consideration during formulation and process validation. The use of flow enhancers in the form of glidants or lubricants to ameliorate some of the factors against powder flow has been a long practice in the pharmaceutical industries. Commonly used glidants includes colloidal silicone dioxide, magnesium stearate, talc and starch and several postulates have been proposed of their mechanisms of action (Peleg and Mannheim, 1972; Neumann, 1965; Jones, 1969).

Characterization of powders is essential to quality control of raw materials, active ingredients or excipients, in order to maintain product uniformity (Pilicheva, *et al*., 2015). Flow property studies of powder materials need to be carried out as part of quality control test. Commonly measured indices that are indicative of quality of powder flow are contact angle, bulk and tapped densities (Abdullah and Geldart, 1999), Hausner's ratio (Wong, 2000), Carr's index (Carr, 1965), angle of repose (Erica, *et al*., 2009) and flow through an orifice (Nalluri and Kuentz, 2010). Although some of these tests are not direct measurement of flow, they have been found to be predictive of the flow characteristics of powder materials.

The aim of this study is to compare the flow enhancing properties of bentonite with some conventional glidants (flow enhancers) and to determine the effect of the concentration of glidant on the flowability of granules of different sizes.

EXPERIMENTAL

Materials. Maize starch BP (Ganone GmbH Schonfeld Straβe, Rosenheim, Germany), magnesium stearate and talc (International Co. Ltd. Anhui, China) and microcrystalline cellulose (Edo Pharmaceuticals, Benin City, Nigeria) were used as received. Bentonite was purchased locally and refined in our laboratory. All sieves were BSS (Endecotts Ltd., London, England).

Preparation of bentonite (edible clay). About 10 kg baked blocks of bentonite were broken into smaller pieces using a mortar and pestle and subsequently reduced into fine powder using a dry mill. Sufficient water was added to the powder to form a suspension and the slurry blended using an electric blender. The blended slurry was soaked in a dilute solution of sodium hypochlorite, stirred and left overnight to sediment. The supernatant layer was decanted and more hypochlorite solution added and stirred. This process was repeated for four days in order to whiten the clay and also remove all water soluble contaminants.

The resulting sediment on the fifth day was washed five times with water. Each washing consisted of soaking the sediment in about thrice its volume of water, stirring continuously for about a minute, allowing for sedimentation and decanting the supernatant layer of water. After the last washing, more water was added to the sediment and transferred into a muslin bag. The bag was suspended over a container and allowed to drain for five days. The content of the muslin bag was discarded and the content of the container was left to sediment over time. The clear upper water layer of the container was decanted and the sediment air dried for several days. The dried mass was milled and passed through a 180 μm sieve to obtain the test bentonite powder.

Microscopy. The glidant powders were thinly spread over a glass slide and viewed under a light microscope (Labo Microsystems GmbH, Germany) via a calibrated eyepiece and the particle size range was recorded.

Preparation of starch granules. The wet granulation method of massing and screening was used in preparing the starch granules. A 10 % starch mucilage was prepared by dispersing 100 g of starch powder in 50 ml distilled water and stirred to form a homogenous mixture. Boiling water was immediately poured into the mixture to form a paste and stirred vigorously. The boiling water was used to make the mucilage up to 1L volume (Karr, *et al*., 1990; Odeku and Itiola, 2002). A sufficient quantity of the starch mucilage (10 %w/v) was added to 1.5 kg of maize starch powder in a mixer to form a wet mass. The wet mass was passed through a 2.0 mm sieve mesh screen and the resulting granules dried at 60 ºC for 30 min in a hot air oven (Gallenkamp, UK). The granules were rescreened through a nest of sieves (850, 710, 500 and 180 μm) arranged in descending order on a sieve shaker (Gallenkamp, UK) and the resulting size fractions further dried for another 30 min. The dry granules collected under the 850 μm sieve (710-850 μm size fraction) was divided into four parts of 50 g each and each part further subdivided into five parts of 10 g each. The five parts were each mixed with 0, 0.5, 1, 1.5 and 2 % w/w of bentonite powder. The same concentrations of magnesium stearate, talc and microcrystalline cellulose were applied to the other five parts from the other three batches. The same procedures were applied to the other size fraction granules and then stored in air tight containers until further analysis.

Granule analysis

Bulk density: A 10 g quantity of the granules was poured gently into a 100 mL graduated measure. The volume of the granules was read and the bulk density calculated.

Tapped density: The measure containing the 10 g of the granules was tapped 100 times on a wooden platform to constant volume. The volume was noted and used in calculating the tapped density.

Hausner's ratio: The ratio of the tapped density over the bulk density of the granules was calculated as the Hausner's quotient.

Angle of repose: The hollow tube method was used. A short hollow tube of 3 cm in internal diameter sitting on a circular horizontal surface of same diameter was filled with granules. The tube was withdrawn vertically and excess granules allowed to fall off the edge of the circular horizontal surface. The height of the heap was measured. The angle of repose, θ, was calculated using Equation 1.

$$\theta = \tan^{-1}(h/r) \quad \text{- - - - - - - - - - - - - - - - 1}$$

where h = height of the heap of granules and
r = radius of the circular base

Flow rate: An Erweka flow tester was used. The time taken for 10 g of the granules to pass through its orifice was recorded. This was carried out in triplicate and the mean values recorded.

RESULTS AND DISCUSSION

The particle size distribution of the glidants/lubricant is shown in Table 1. Magnesium stearate powders showed the largest particle size and a wide particle size distribution while the processed bentonite powder exhibited the smallest size and a narrow particle size distribution. Results from the analysis of the granules show that the particle size and glidants/lubricant concentration influenced the flow properties of the granules. Figure 1a,b,c and d shows the Hausner's ratios of the granules studied at different granule sizes and glidant concentrations. Powders with low antiparticle friction have Hausner's ratio values of about 1.2, whereas more cohesive powders have values greater than 1.6 (Hausner, 1967). At the concentrations studied, no glidant generated Hausner's value of up to 1.6. However, only granules lubricated with magnesium stearate generated Hausner's ratio below 1.2. The lubricant/glidant effects of the test samples were comparable at various concentrations and granule sizes except at 710-850 μm size of the granules. Magnesium

stearate was superior to talc followed by MCC and lastly bentonite in that order as lubricant/glidant.

Figure 2 shows the angle of repose values of the granules studied at different granule sizes and glidants concentrations. Powders with smaller angle of repose (< 25º) have very good flow properties, whereas those with larger angles (≥ 50º) have unsatisfactory flow properties (Stanifort, 2002).

Table 1: Particle size range of the glidants

Glidant	Particle Size Range (μm)
Magnesium stearate	10-100
Talc	10-30
Microcrystalline cellulose	10-50
Bentonite	2-10

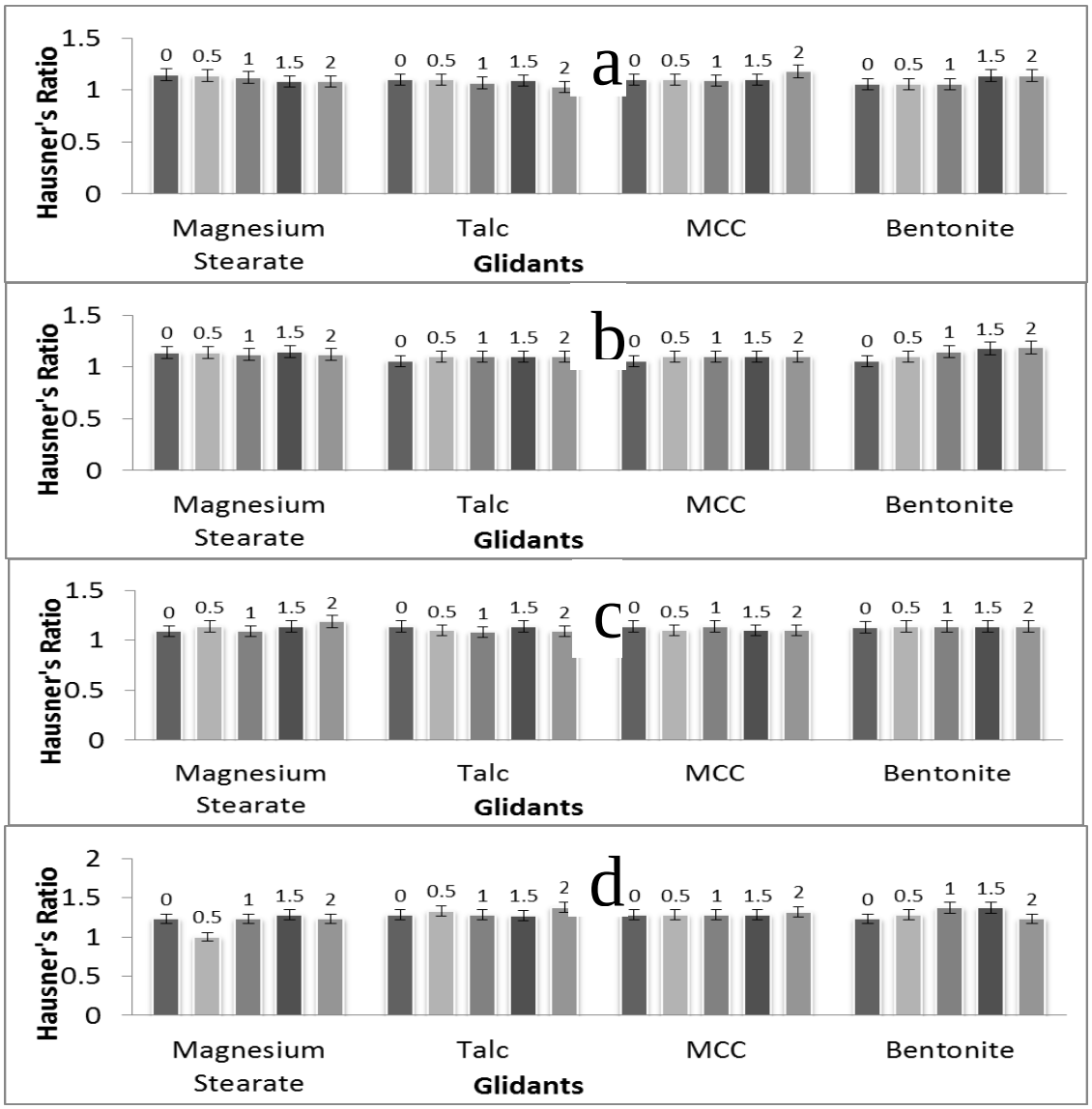

Figure 1: Hausner's ratios of granule size <180 μm (a), 180-500 μm (b), 500-710 μm (c) and 710-850 μm (d) at different glidant concentrations.

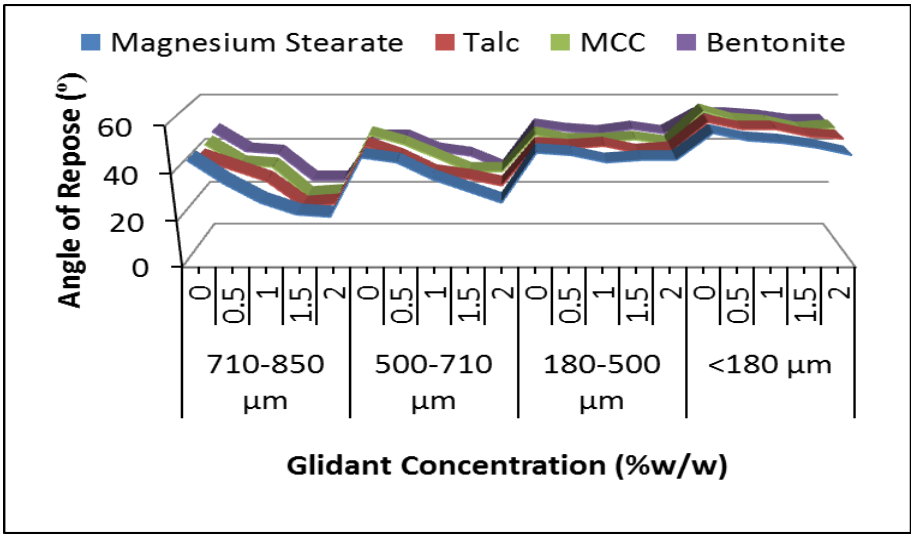

Figure 2: Angle of repose of the granules at different granule sizes and glidant concentrations

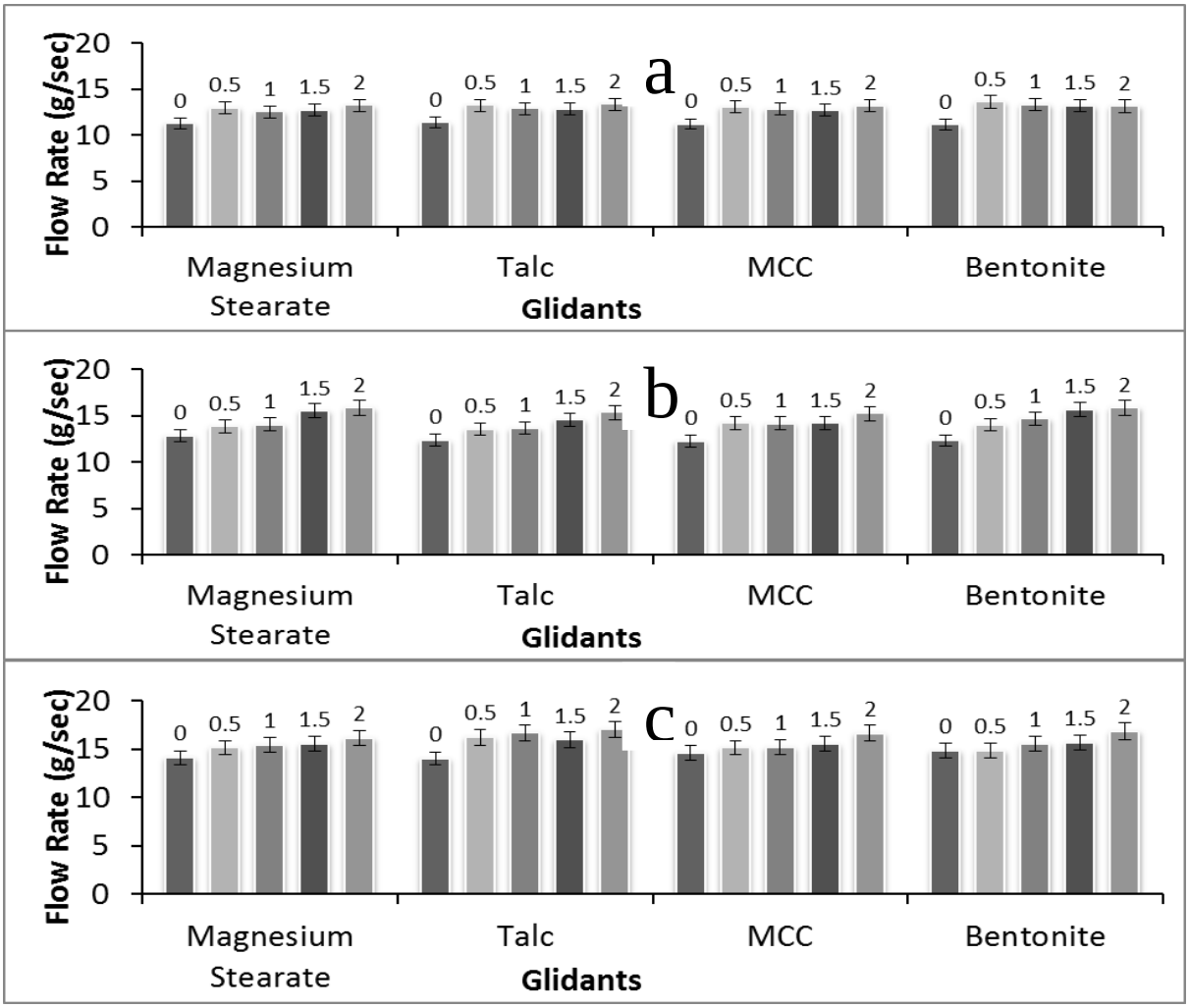

Figure 3: Flow rate of granule size <180 µm (a), 180-500 µm (b), 500-710 µm (c) and 710-850 µm (d) at different glidant concentrations.

Although the results from the measurement of this parameter are sensitive to the methodology used, results obtained show that the angle of repose of the granules have an inverse proportionality with granule sizes and glidant concentrations. There was an increase in the angle of repose with a decrease in granule sizes and a decrease in angle of repose with increase in glidant concentrations. All the glidants showed comparable angles of repose at the various granules sizes studied.

The rates of flow through an orifice are listed in Figure 3. This measurement is a more direct reflection on the flow characteristics of the starch granules. There was no flow from the batches of granule size < 180 μm at all the concentrations of the glidants tested. There was a slight increase in the flow rate of the granules with increasing granule size. There was also increase in the flow rate with increase in the concentration of each glidant. This is consistent firstly with the filling of the pits on the surface of the irregular shaped particles with the fines of the glidant, thereby preventing mechanical interlocking of the particles; and secondly, adherence of the glidant to the surfaces of the particles to reduce interparticulate friction (Wong, 2000). The increase in glidant concentration could lead to aggregation of the glidant fines on the surface of the particles leading to greater interparticulate distance. This increase in interparticulate distance will reduce the forces of attraction between particles, reduce the action of capillary adhesion forces and also prevent the formation of solid bridges between particles. The increase in flow rate resulting from increase in particle sizes of the granules could be attributed to a decrease in surface free energy of the granule particles and decrease in frictional forces between the granules leading to faster flow (Iwuagwu, *et al*., 1986).

Conclusion. Bentonite powder was comparable to magnesium stearate, talc and microcrystalline cellulose in the concentrations used in this study except at granule size of 710-850 μm. It can therefore be recommended as a suitable substitute in formulations where the particle size ranges lies below 710 μm.

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