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On the difficulty of assessing the specific surface area of magnesium stearate

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Abstract

The water content of as-received commercial magnesium stearate batches from animal and vegetable sources have been modified by ageing in humid air at room temperature or by vacuum treatment. The complete adsorption–desorption isotherms of nitrogen and krypton vapours by samples of these as received and modified materials have been measured at liquid nitrogen temperature after standardised vacuum degassing. They are greatly affected by the initial water content of the material. In particular: (a) the BET surface area values computed from the adsorption branch vary widely and is increasing with increasing water content; (b) anomalous hysteresis of varying amplitude is observed in all cases except adsorption of krypton on the material with the lowest water content; (c) the hysteresis loops extend down to very low desorption pressure values and cannot be accounted for by capillary condensation. Lastly, the surface area value of a given material computed from nitrogen and krypton adsorption may differ by a factor as high as six. Accordingly, the very significance of BET surface area values obtained from routine adsorption experiments should be regarded as questionable, at least until the mechanisms of adsorption are fully clarified. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Magnesium stearate; Surface area; Adsorption isotherm; Density; Water content

1. Introduction

The performance of any particular pharmaceutical form is dependent on the proper control of the quality and more particularly of the functionality of the raw materials entering its fabrication process. Magnesium stearate, a major lubricating excipient, has been widely used for a long time in the formulation of oral solid pharmaceutical forms. It is currently regarded as a critical excipient due, mainly, to its highly hydrophobic nature. Indeed, the later may negatively influence the disaggregation of the form, the dissolution kinetics and even the stability of the active drug. The lubricating capacity of magnesium stearate taken

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from different batches or from different origin may not always prove identical, which brings about problems of reproducibility at the development or production steps. Furthermore, following the development of the bovine spongiform encephalopathy, raw products from vegetable origin have been progressively substituted in the industrial production processes for products from animal origin. All this provides renewed impetus for improving the present state of knowledge of the material physical properties, particularly at the stage of its preformulation.

Several authors already have investigated the main physical–chemical properties of magnesium stearate, among which should be mentioned the palmitate–stearate ratio, crystallinity, phase composition, water content and granularity including surface area (Miller and York, 1985; Brittain, 1989). The striking feature emerging from the related literature is certainly the wide diversity of observed structural and granular characteristics and the supposedly related variations of lubricating capacity. Such a diversity appears to be linked to various factors ranging from the material origin and fabrication process or to its drying and storing conditions, as revealed by its varying purity, crystallisation state and hydration rate. Measurement of the specific surface area has been particularly utilised despite its restricted significance as demonstrated by the present paper. Indeed, it was and remains currently admitted that the lubricating character of the material may be regarded as a surface property the intensity of which would be in some way related to the extent of its surface area. Various attempts have been carried out to find correlation between values of the material surface area and values of various estimators of its functionality. For example, the die ejection force of tablets or force transmission to die and punches is currently regarded as an estimator of the material lubricating capacity. Most of these attempts were based on alleged measurements of the material surface area by nitrogen vapour adsorption at liquid nitrogen temperature. In such experiments, the stearate samples are routinely degassed beforehand under vacuum at temperature ranging from room temperature to as much as 80°C without ever questioning explicitly the material stability in such conditions. Also, it is striking to realise that the type of adsorption isotherm has never been determined while some of the reported surface area measurements have been carried out using the so called single-point BET procedure. That procedure is only approximate and its utilisation requires prior knowledge of the initial part, at least, of the adsorption isotherm. A well-characterised type II or IV isotherm with a high value of the C constant[1] in the BET formula is highly desirable. Otherwise, more or less erroneous conclusions may be drawn. Based on these various considerations, one should not be surprised to finally observe that only weak correlation or no correlation at all have been found between values of the specific surface area and lubricating capacity of magnesium stearate (Ertel and Cartensen, 1988; Leinonen et al., 1992).

In contrast, the only example (Hölzer, 1983) known to the authors of a well-characterised correlation between these very same parameters was observed in experiments where surface area was determined by permeametry. That technique essentially measures the envelope surface area of the particles and may thus be supposed to be quite insensitive to microstructural modifications of the material such as the development of internal porosity or cracks that may result from dehydration induced by thermal and/or vacuum treatments.

The objective of the present report is to demonstrate how the characteristics of the physical adsorption of nitrogen on magnesium stearate are modified by prior thermal treatment and degassing, and how the determination of surface area may thus be affected to a point that raises serious questions as to the usefulness of a continued utilisation of the technique and as to the significance of the data values already reported in the literature.

Besides, the work emphasises the need for an improved understanding of the chemical mecha-

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[1] $C = A\ \exp(E_0 - E_{\mathrm{Liq}})$ where $E0_0$ is the heat of adsorption of the first monolayer of adsorbate on the solid surface, E_{Liq} the heat of liquefaction and A is a constant. The larger the difference $(E_0 - E_{\mathrm{Liq}})$ the more relevant the BET model.

nism of the degassing of magnesium stearate under vacuum, as a preliminary to the standardisation of the experimental protocol for nitrogen physisorption experiments on this material. Indeed, this would pave the way towards the quantitative assessment of significant surface area values and, finally, the understanding of the supposed relation between its lubricating capacity and granular properties.

2. Materials and methods

The various so-called as received stearate batches have been obtained from two distinct laboratories and originate from either vegetable or animal raw materials as specified in Table 1.

The so-called modified batches have been prepared by ageing in an incubator at controlled temperature and relative humidity, respectively 25 (±0.1) °C and 85 (±1)%; relation humidity during 1, 2, 3 and 4 months. Indeed, according to Sharpe et al. (1997), such conditions allow well-crystallised magnesium stearate trihydrate starting from the anhydrate phase to be obtained.

Density has been measured by helium pycnometry using the Ultrapycnometer from Quantachrome. By following particular experimental and data acquisition and reduction procedures that have been described elsewhere (Andrès et al., 1998) an accuracy of about 1×10^{-4} g cm^{-3} can be reached.

The water content was determined by the Karl-Fisher (KF) method in anhydrous methanol. Magnesium stearate appears to be insoluble in this medium but dissolves totally upon addition of the KF reagent. This is indication that the overall water content can actually be measured. For a sample about 400 mg in weight and a typical water content equal to 5% w/w, the relative uncertainty on measured values of the later can be estimated to about 0.01% w/w. However, five independent measurements were carried out on each material and the 95% confidence interval on the result proves much larger than that, ranging from 0.05 to 0.5% w/w. Two distinct sources of systematic errors can be identified as responsible for that discrepancy: the variability associated with sampling of the particulate materials and the introduction of small amounts of air including water vapour when transferring the sample in the analysis cell.

The granularity of the various batches has been investigated by resorting to scanning electron microscopy (SEM), laser light scattering granulometry and surface area measurement by nitrogen and krypton vapour adsorption.

For SEM examination and in order to avoid electrical charging the JEOL microscope (model 6400F) was operated at a low beam voltage of 5 kV only and the samples were metalised (using either gold, copper or carbon with equally satisfactory results).

Light scattering experiments were carried out on either liquid or air suspensions of the particulate materials using the Coulter LS130 from Coultronics equipped with either the so-called standard liquid module or dry module respectively. In the former case the liquid medium consisted of either a 0.5% w/v solution of sodium laurylsulphate in filtered water or isopropylic alcohol.

In these experiments, use of the polarisation induced differential scattering option (PIDS) appeared unnecessary, and the radial distribution of diffracted intensity was best analysed using the Mie-theory based algorithm with the following values of the real and imaginary parts of the refractive index: $Re(n) = 1.4$–1.5 and $Im(n) = 0.001$–0.01.

Nitrogen and krypton vapour adsorption and desorption isotherms were measured at liquid ni-

Table 1
Producer, origin and designation of investigated magnesium stearate batches

Producer/origin	Vegetable	Animal
FACI[a]	VF	AF
GREVEN[b]	VG	AG

[a] Retailer Marcel Quarré, 10–12 rue Massue BP46, 94302 Vincennes, France.

[b] Retailer Quimdis, 85 Edouard Vaillant, BP 329, 92307 Levallois Perret, France.

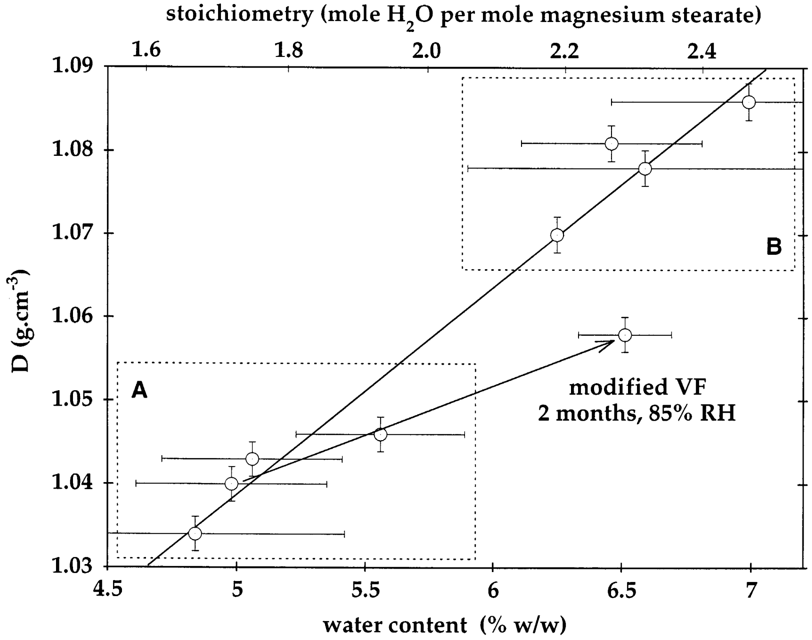

Fig. 1. Variation of the density of magnesium stearate as measured by helium pycnometry with overall water content as measured by the Karl–Fisher technique.

trogen temperature with the following two distinct pieces of equipment: the Autosorb 1C from Quantachrome and the ASAP 2000 from Micromeritics. Prior to adsorption experiments, analysed samples were degassed under vacuum during a length of time ranging from 18 to 40 h at room temperature (25°C) and for one particular VG sample at 50°C. The associated weight loss resulting from desorption and/or structural water release was measured in each case.

3. Results

3.1. Density and water content

In Fig. 1 it can seen that the density of the different as received batches increases slightly with increasing water content. The difference between the highest and lowest observed values is less than 6% of the mean. Ageing in humid atmosphere results in the simultaneous increase of both density and water content. This is shown in Fig. 1 by the arrow linking the points corresponding to as received VF and the same batch after 2 months ageing. The stoichiometric compositions corresponding to the water content values in Fig. 1 range approximately from 1.5 to 2.5 mol H_2O per mole of magnesium stearate as appears on the upper abscissa axis. It could be verified by weighing the samples before and after experiment that the flow of dry helium through the powder sample in the pycnometer cell does not cause the dehydration of even the most hydrated phases (batches as received VG and modified VF).

One may state that a significant correlation is observed between both variables, density and overall water content. Indeed, the data in Fig. 1 group into two subsets, one (noted A) associating low water content and density values (batches VF and AF with less than two H_2O molecules per magnesium stearate molecule) and the other (noted B) associating high water content and density values (batches VG and AG with more than two H_2O molecules per magnesium stearate molecule).

3.2. Granularity

3.2.1. Microscopy

It proves impossible to differentiate the various materials in terms of particle shape based on SEM observations. Indeed, even the very small samples actually examined under the microscope exhibit a very large distribution of different particle shapes including the needle and platelet types described in the literature (Butcher and Jones, 1972; Von Müller, 1977; Miller and York, 1985; Marwaha and Rubinstein, 1988). As for particle size the only noticeable difference comes down to the selective observation of a few larger particles (typically 50–100 μm) in batch VG.

3.2.2. Laser light scattering

The volume based particle size distribution of batch VG exhibits a slight but reproducible broadening towards large particle diameter when compared to that of batch VF. This remains valid even after changing the optical model or varying the values of the real and imaginary parts of the complex refractive index within physically consistent ranges.

This observation qualitatively supports the results of SEM just mentioned but it would be vain to search for any quantitative correlation between particle size distribution and other granular properties in view of the large diversity and complexity of particle shape.

3.2.3. Vapour adsorption

As already mentioned in Section 1 all specific surface area values reported in the literature have been obtained by nitrogen physical adsorption using the standard or single point BET method even though the adsorption isotherm type was unknown. Accordingly, the reversible or irreversible nature of the process (that would be capable, in principle, to reveal mesopores by the occurrence of capillary condensation or capillary evaporation) has never been investigated. Neither has been the adsorption of other adsorbates, including krypton, which could provide one with additional pieces of information.

3.2.3.1. Nitrogen as adsorbate

As received VG and AG; modified VF. The adsorption isotherms observed with the highly hydrated magnesium stearates, i.e. containing typically more than 6% w/w H_2O (group B in Fig. 1), are characterised by an ascending (adsorption) branch that can be classified as a limit type II isotherm (see Fig. 2). Indeed the value of constant C computed from the best fit of the BET linear transform (Sing et al., 1985) to the data points is very low, typically less than 5, except for VF aged 1 month (Table 2). This means that the BET physical adsorption model is not really appropriate for the determination of the specific surface area of these materials. Nevertheless, for the sake of comparison with the literature, it must mentioned that, based on the standard application of this model, using data points in the range $0.1 < P/P_0 < 0.3$, specific surface area values ranging from 8 to 45 $m^2\ g^{-1}$ can be computed (Table 2). At the same time the utilisation of a fundamentally different model based on purely thermodynamical ground (Adolphs and Setzer, 1996) does not provide really different results. In particular the hierarchy of the various materials in terms of estimated surface area remains unchanged. Accordingly, no attempt is made here and in the following to systematically test the many different adsorption models available.

An important observation regarding the modified VF batches appears in Fig. 3. The capacity of adsorption increases with ageing, i.e. with water content as demonstrated in Fig. 1.

All the isotherms in Fig. 2 and Fig. 3 share a few remarkable common features. Firstly, they exhibit hysteresis, but the hysteresis loop does not close at a finite relative pressure, e.g. around 0.3–0.4 as is very often observed when using nitrogen adsorbate. Second, hysteresis extends down to the lowest possible relative pressure value and persists even under vacuum at the same temperature whereas the amount of nitrogen so retained can only be released by returning the sample to room temperature. Third, a small step on the desorption branch of the isotherm is systematically observed in the interval $0.45 < P/P_0 < 0.50$. According to these observations, hysteresis cannot result from capillary condensation and/or capillary evaporation only. Finally, based on the comparison of the reduced isotherms $V_{\mathrm{desorption}}/V_{\mathrm{adsorption}} = f(P/P_0)$ not shown here, it also appears that the shape of the hysteresis loop may be considered as constant at least to a certain extent, independently of the amount absorbed.

Fig. 2 also allows the comparison of the adsorption-desorption isotherms of one material (here VG) measured using two distinct sorptometers. Even though the adsorbed volumes and surface area values are not identical (see Table 2), all the general features of the isotherms are reproducible, in particular as regards the hysteresis loops.

As received VF and VG degassed at 50°C under vacuum. In both cases the adsorption branches of the isotherms are unambiguously of type II with values of C larger than 10 (Table 2). The surface area values computed from the standard BET procedure are smaller than in the previous cases and typically lower than 6 m^2 g^{-1}. As appears in

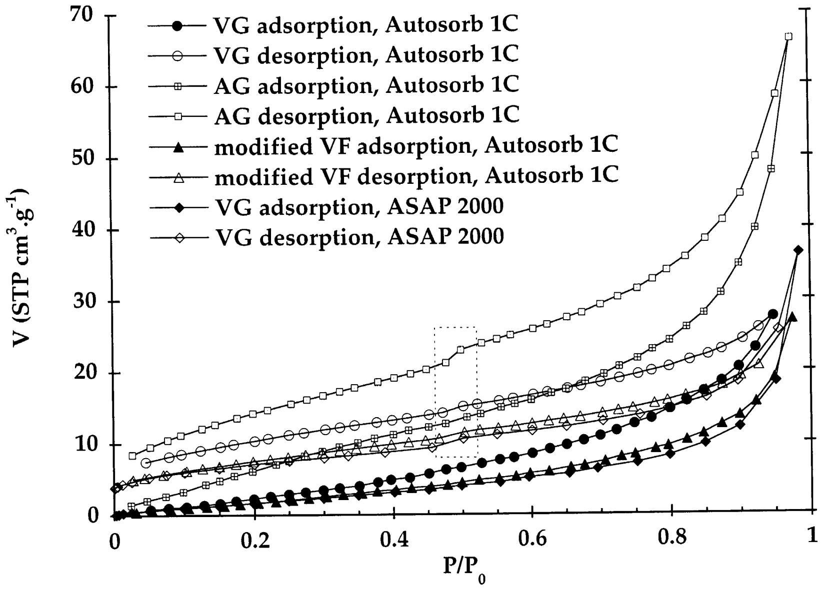

Fig. 2. Nitrogen vapour adsorption–desorption isotherms at liquid nitrogen temperature of as received VG, as received AG and modified VF, all degassed at 25°C under vacuum.

Table 2
BET surface area values measured by physical adsorption of nitrogen vapour at liquid nitrogen temperature[a]

		BET specific surface area (m^2 g^{-1})	*C* (dimensionless)
As received VF		2.9[b]	16.5[b]
Modified VF	aged 1 month	8.1	10.0
(25°C/85% RH)	aged 2 months	13.8	2.9
As received VG		19.1	2.9
		10.5[b]	5[b]
	degassed at 50°C	5.5[b]	20.3[b]
As received AG		44.8	3.7

[a] If not otherwise stated, samples are degassed at room temperature and surface areas are measured using the Autosorb 1C, Quantachrome.

[b] Experiment carried out using the ASAP2000, Micromeritics.

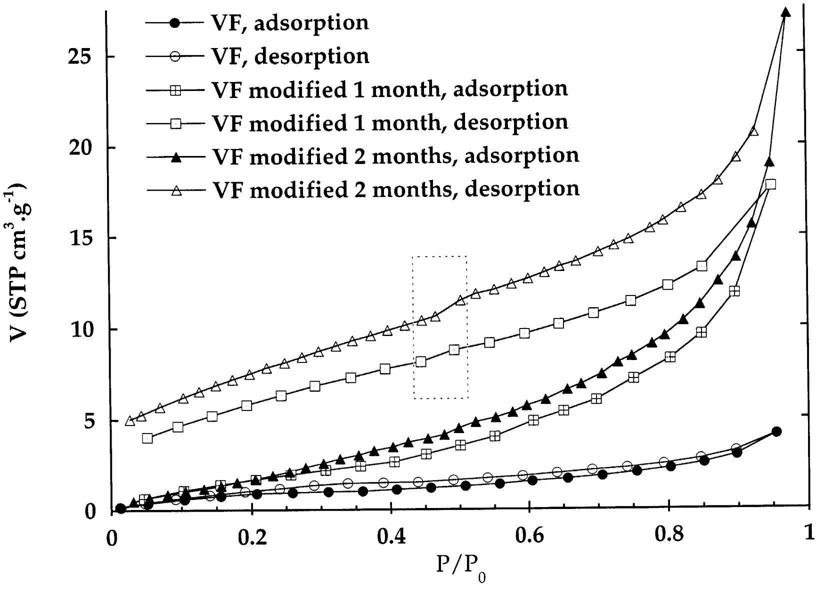

Fig. 3. Evolution on ageing of the nitrogen vapour adsorption–desorption isotherms at liquid nitrogen temperature of VF. Measurements are carried out using the Autosorb 1C, samples are degassed at 25°C under vacuum.

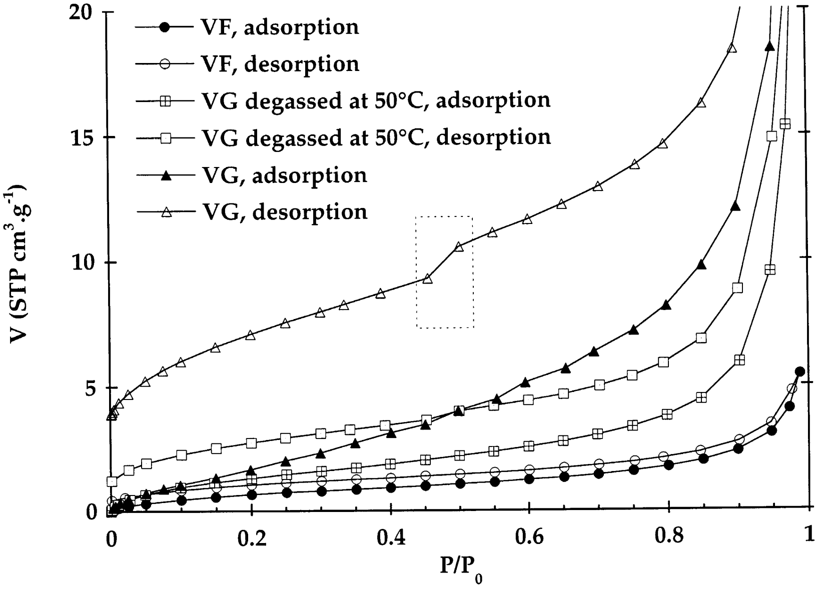

Fig. 4. Nitrogen vapour adsorption–desorption isotherms at liquid nitrogen temperature of a) as received VF and VG both degassed at 25°C under vacuum, and b) VG degassed at 50°C under vacuum. All measurements are carried out using the ASAP 2000.

Fig. 4, hysteresis is still observed on desorption but then its amplitude expressed in terms of adsorbed volume is obviously much smaller. Again it persists down to zero relative pressure but the step on the desorption branch is very small or hardly detectable.

3.2.3.2. Krypton as adsorbate. Usually, krypton adsorption at liquid nitrogen temperature is utilised for the measurement of small or very small specific surface areas so as to take advantage of its very low vapour pressure at that temperature in order to increase accuracy. In the present case it also serves to remove some ambiguity associated with the utilisation of nitrogen.

For all batches, the standard BET procedure allows one to obtain reasonably high C values ranging from 8 to 12 (Table 3) and reliable estimates of the surface area from the well-characterised type II adsorption branch of the isotherm. These are then systematically smaller by a factor 2–6 as compared to those inferred from nitrogen adsorption.

As received VF. The isotherm of krypton adsorption on as received VF (Fig. 5) exhibits no hysteresis and the BET surface area value is only 1.4 $m^2\ g^{-1}$ (Table 3).

As received VG and AG, modified VF. These are the three materials that exhibit the largest differences between the surface area values obtained from nitrogen and krypton adsorption, the former being typically five or six times larger than the latter (Table 3). Hysteresis is still observed in all cases (see Fig. 6) but then its amplitude is drastically reduced compared to what it is with nitrogen

Table 3
BET surface area values measured by physical adsorption of krypton vapour at liquid nitrogen temperature[a]

		BET specific surface area ($m^2\ g^{-1}$)	C (dimensionless)
As received VF		1.4	11.9
Modified VF (25°C/85% relative humidity)	aged 2 months	2.4	8.6
As received VG		3.3	9.3
As received AG		8.8	11.8

[a] Samples are degassed at room temperature and surface areas are measured using the Autosorb 1C, Quantachrome.

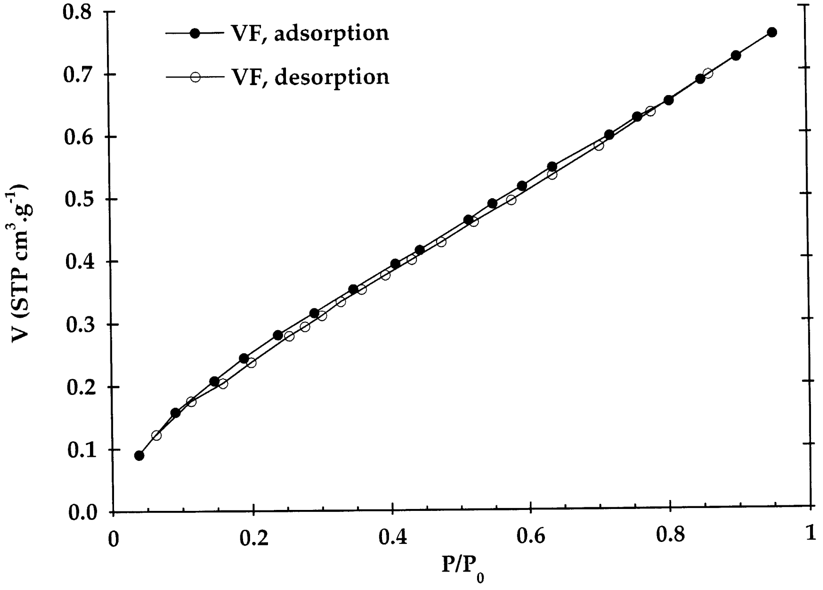

Fig. 5. Krypton vapour adsorption–desorption isotherm at liquid nitrogen temperature of as received VF, degassed at 25°C under vacuum. The measurement is carried out using the Autosorb 1C.

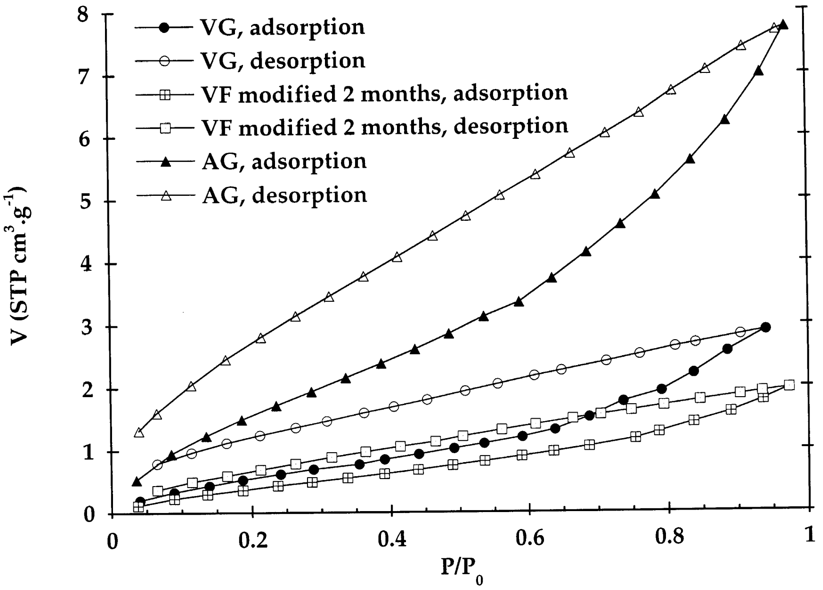

Fig. 6. Krypton vapour adsorption–desorption isotherms at liquid nitrogen temperature of as received VG, as received AG and modified VF, all degassed at 25°C under vacuum. Measurements are carried out using the Autosorb 1C.

adsorption (Fig. 2). Again the hysteresis loop extends down to the smallest relative pressure values here equal to 0.05, but the step on the desorption branch is no longer detected around $P/P_0 = 0.45–0.50$.

A last but important observation must be emphasised here: when the ageing of batch VF is lengthened from one month to two, and its water content consequently increased, the amplitude of the hysteresis on the isotherm (Fig. 3) and of the apparent value of the specific BET surface area (Table 2) are both found to increase.

4. Discussion

The nitrogen adsorption isotherms of the stearate batches with the highest water contents, namely VG, AG and modified VF fit none of the standard isotherm types listed in the relevant IUPAC classification (Sing et al., 1985). Indeed a significant fraction of the adsorbate cannot be desorbed even at the lowest experimentally attainable relative pressure value. Such behaviour can be understood either in microstructural or energetic terms (Sing et al., 1985). A simple explanation could be that one fraction of the adsorbate would be chemisorbed (the first monolayer necessarily) but then the value of C computed from the BET linear transform of the adsorption branch of the isotherm should be very high, which is clearly at variance with the present results. In fact, the problem here is to find how to reconcile a certain initial ‘difficulty’ to adsorb nitrogen at low relative pressure (as reflected in the weak initial curvature of the isotherm and low C value) with a clear final ‘difficulty’ to desorb the adsorbed phase even under vacuum. One alternative explanation could be that one is faced with some sort of intercalation of the nitrogen molecules, more or less comparable with what is known with clay minerals (Barrer and MacLeod, 1954). This would increase the adsorbed volume compared to pure surface adsorption. In such a situation, the apparent BET specific surface area should logically no longer be interpreted in purely geometrical terms. In accordance, it must be emphasised that even though the shape of the hysteresis loop remains approximately unchanged, its amplitude (measured for instance by its area) varies always in the same way as the adsorbed volume at a given P/P_0 value and as the apparent BET surface area.

The mechanisms responsible for these particular characteristics and behaviour are most likely related with specific structural features of the hydrated magnesium stearate phases (and more generally of all metal soaps). Despite their obvious fundamental interest their discussion clearly exceeds the limits of the present paper.

In the context of the modification of the VF batch (which initially exhibits a low water content) the amplitude of the adsorption–desorption hysteresis increases with ageing time. Finally, modified VF materials can reach water contents comparable with that of the as received VG batch and are then characterised by a strong adsorption–desorption hysteresis. Based on the work of Sharpe et al. (1997) and the literature cited herein, such an increasing water content is likely to be accompanied by the crystallisation of the di- and trihydrated phases.

Conversely, the low water content of batch VG once exposed to vacuum at room temperature is very similar to that of the as received VF batch. Though, of these two, only the former exhibits a strong adsorption–desorption hysteresis. A very likely explanation for this apparent contradiction may be stated as follows: it would be the very degassing process carried out under vacuum and necessarily preceding the adsorption experiment that brings about the appearance of hysteresis. One objection here might be that the low hydration state of the as received VF batch is reached, at the latest stage of its fabrication process, as a result of the thermal dehydration of hydrate phases following intensive drying but that anhysteretic adsorption prevails nevertheless. The additional assumption needed to reconcile this with the preceding statement would be that dehydration under vacuum at room temperature or at least at low temperature brings about a particular and thermally unstable microstructure responsible for the adsorption hysteresis. This would be in agreement with the influence of temperature on the microstructure of products of dehydration and even more generally of most decomposition reactions of inorganic compounds. Highly disordered structures obtained at low temperatures are generally associated with high surface areas and porosity at the micro or mesoscale. The effect of increasing the temperature is then to bring about some sort of 'sintering' in the broadest sense of that word, and in particular the closure of the fine porosity.

Independently of what just precedes, the demonstrated inability to correctly estimate the surface area of the investigated materials by resorting to standard nitrogen physical adsorption experiments, allows one to understand the failure of the previous attempts to explain of the variability of the lubricating properties of magnesium stearate based on a search for a correlation between numerical values of the specific surface area, of the water content and of one rheological parameter. The results reported in the present paper clearly suggest that surface area values obtained from routine nitrogen adsorption experiments actually refer to more or less modified materials and that any such search for correlation between values of surface area and functionality should explicitly take this into account. However, it is never the case in the various publications known to the authors and quoted above. Accordingly, their conclusions and tentative explanations should be considered with great caution.

The need to validate alternative techniques for the measurement of the specific surface area of magnesium stearate (and probably of other metal soaps) appears clearly. From this standpoint, laser light scattering may look more reliable at first sight. However, its inherent inability to correctly characterise the size distribution and consequently the surface area of non-spherical powders, and in particular those made of flakes or needle-shaped particles, should not be overlooked. The physical adsorption of krypton and possibly of several other adsorbates that remain to be tested, only partially solves the problem. Indeed the fundamental complication resulting from the partial irreversibility of nitrogen adsorption may be overcome in some cases but the problem of the structural and microstructural modification of the hydrated materials under vacuum during the unavoidable degassing step remains. Accordingly, elucidating the origin of the variability of the lubricating properties of magnesium stearate based on measurements of surface area appears biased. One strategy to circumvent that difficulty and allow to continue taking advantage of the incomparable capacity of physical adsorption to characterise the microstructure of particulate solids at different scales of length is now under investigation. This is based on the simple idea of comparing values of surface area and of one estimator of the lubricating capacity of different magnesium stearates measured on samples degassed in strictly similar conditions.

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