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RESEARCH PAPER

Powder Compaction Properties of Sodium Starch Glycolate Disintegrants

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ABSTRACT

The compaction behavior of three "as supplied" commercially available grades of sodium starch glycolate (SSG), Explotab, Primojel, and Vivastar P, was investigated at compression speeds of 0.17 and 30 mm/sec. The results suggested that the three "as supplied" materials exhibit different compression and compaction behavior. Primojel and Explotab exhibited similar compactibility, whereas Vivastar P produced compacts of poor integrity. This behavior was not mirrored in the compressibility of the powders, where Vivastar P and Explotab exhibited similar performance. The materials were studied using x-ray diffraction, scanning electron microscopy, Carr's compressibility index, and swelling volume. In terms of material characteristics, all the products exhibited similar swelling in water. Primojel and Explotab retained most of the crystallographic order from the parent potato starch and exhibited comparable particle surface topographies. Vivastar P contained the lowest moisture level. However, it is not clear if the poor compactibility of Vivastar P is due to differences in moisture content, the reduced surface topography, or subtle differences in the SSG polymer structures (substitution, cross-linking, and crystallinity). Overall, even though the three commercial grades of sodium starch glycolate are successfully used as disintegrants, they do exhibit differences in their "as supplied" powder mechanical properties: Primojel and Explotab exhibit similar compactibility, whereas Vivastar P is poorly compactable but exhibits similar compressibility to

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Explotab. These observations may have implications when formulating poorly compactable or moisture-sensitive drugs.

Key Words: *Disintegrant; Mechanical properties; Sodium starch glycolate*

INTRODUCTION

Pharmaceutical tablets are typically manufactured by the rapid compression of a multi-component powder column containing a drug and excipients. The tablets must exhibit certain specifications such as strength, friability, disintegration, dissolution, and content uniformity. The requirement of a dosage form to adhere to such specifications has led to the development of a wide range of excipients which can impart particular tablet properties, for example, binders such as microcrystalline cellulose can be used to improve strength. The dissolution profiles of pharmaceutical tablets can be modified by the addition of a so-called "super-disintegrant." These polymeric-based materials, such as sodium starch glycolate (SSG), croscarmellose sodium, and crospovidone undergo volume expansion when in contact with water, resulting in rapid disintegration of the tablet matrix.

Sodium starch glycolate is a cross-linked substituted potato starch.[1] The material is available from various manufacturers under several different trade names, such as Explotab, Primojel, and Vivastar. The commercial grades of Explotab and Primojel have been reported to exhibit subtle differences in their chemical compositions and physical characteristics.[2] This study also concluded that, in terms of disintegrant functionality in tablet formulations, the products were pharmaceutically equivalent. A more recent study of several batches of three SSGs (Explotab, Primojel, and Explosol) reported that there were inter-brand and inter-batch differences in the physical properties of the pure products, however, again, in terms of disintegration time and compactibility in a model formulation, the products were essentially equivalent.[3] Interestingly, this study also reported differences in the disintegration times for the formulations containing various croscarmellose sodium brands.

Typically, SSGs are present in 1–5% concentrations in tablet formulations. The mechanical powder properties of the pure SSG materials are not considered, since tablet strength is usually a consequence of the functionality of filler–binders such as microcrystalline cellulose and dicalcium phosphate dihydrate. However, the compactibility of a SSG will become more important when the disintegrant is present in higher proportions. In these cases, the behavior of SSGs in powder blends will have a significant effect on tablet strength, since these materials would be expected to have a detrimental effect on tablet strength due to their elastic properties.

When considering the properties of polymers it is vital that the chemical structure is understood. This is particularly true for SSGs, where cross-linking and substitution are performed on the starch polymer chains. In the case of Explotab and Primojel, it is unclear from their product literatures whether the potato starch is cross-linked before or after reaction with Na chloroacetate. However, early investigations which compared the properties of experimental grades of SSG with Explotab reported that the degree of cross-linking was determined prior to carboxymethylation, suggesting that Explotab may be cross-linked before reaction with Na chloroacetate.[4] In contrast, a similar early study of the properties of experimental grades of SSG with Primojel suggested that the cross-linking was achieved after carboxymethylation in these samples.[5] The manufacturing process for Vivastar is reported to involve the substitution of potato starch with Na chloroacetate followed by the reaction of the Na carboxylate groups with starch primary alcohol groups to produce ester cross-links.[6] Even though it is unclear as to the manufacturing processes for Explotab and Primojel, this is an obvious potential source of any apparent differences in material and formulation properties. Chemical cross-linking of starches using, for example, sodium trimetaphosphate, phosphorus oxychloride, and epichlorohydrin, is thought to occur via the starch alcohol groups,[7] i.e., cross-links are present as phosphate esters or ethers as opposed to the side-chain ester cross-links in Vivastar. Sodium starch glycolates based on Primojel and Explotab are described as having low levels of cross-linking[2] with degrees of substitution of 0.23–0.30.[2,4,5] The effects of cross-linking,

degree of substitution, and purity on the strength, disintegration, dissolution, and compaction properties of formulations containing SSGs based on Primojel and Explotab have been reported.[2,4,5,8] These studies suggested that substitution and cross-linking in Primojel and Explotab are optimal in terms of disintegrant properties.

Sodium starch glycolate is a starch derivative. Starch is a complex, naturally occurring material which can be used as a binder, diluent, and disintegrant in tablet and capsule formulations.[9] The compaction and disintegration properties of starches have been reported to be dependent on source.[10–12] Starch, and modified starches, can be considered as partially crystalline materials, however, the properties of starches are further complicated by the presence of other minor components, such as lipids.[13] Starch granules typically consist of two polymers, amylose, which is amorphous, and amylopectin, which is semicrystalline. This has obvious implications for the structure of SSGs, since amylose and amylopectin can be carboxymethylated during the manufacturing process. In terms of the functionality of SSGs, the compactibility will be determined by the number of *inter*-particle interactions and the mechanical properties of the bulk particles. An amorphous material, such as amylose, would be expected to exhibit a relatively low modulus above its glass transition temperature (T_g). It has been reported that the Young's moduli of starches are lower than those of another semicrystalline polymer excipient, microcrystalline cellulose.[14] The presence of these amorphous domains with relatively low Young's modulus would be expected to be responsible for the apparent greater elasticity of starches and would contribute towards their relatively low compactibility. It is generally accepted that high tablet strength is a result of several material characteristics, including low elasticity.[15] In terms of composite materials, the presence of elastic domains in a plastic matrix would be expected to have a significant effect on mechanical properties.

As pure materials, the compressibility and compactibility of SSGs will depend on the mechanical properties of the polymers which comprise the modified starch granules (*inter*- and *intra*-polymer interactions), and the topography and composition of the surfaces of the particles (*inter*-particle interactions). The *intra*-particle interactions in SSGs, would be expected to be primarily amylose, amylopectin, substituted amylopectin, and substituted amylose polymer interactions. However, other components of the granule, such as processing by-products and lipids, may be important.

Another important factor for the interactions between hydrophilic organic-based polymers, such as starches, is the role of solvents, in particular water. Any solvents which are employed during the manufacturing process of SSGs will affect polymer mobility and the solubility of the polymers in multi-component polymer blends. The interaction of water with starch and cellulose-based polymers is complex and it has been reported that water is present in at least three thermodynamic states.[16] The presence of moisture is reported to affect the compactibility of starches.[12] Also, it has been reported that water affects the T_g of Primojel.[17] Additionally, the presence of surface moisture will affect *inter*-particle adhesive and cohesive interactions. When considering SSGs, many questions concerning functionality arise. These are based on two primary functional properties: disintegration and compactibility. As previously stated, the materials show excellent disintegration properties in tablet formulations. Also, Primojel and Explotab are reported to be essentially equivalent in terms of pharmaceutical functionality.[2,3] However, in terms of tabletting functionality, the compactibility of starches and pregelatinized starch are, in general, lower than for other plastically deforming binders, which has been ascribed to the significant elastic properties of starches.[18] In the case of SSGs, the situation is further complicated by the presence of the ionic Na carboxymethylether groups. These functional groups would be expected to affect the modulus of the base starch components and modify the granule water sorption properties. However, other properties such as surface chemical composition, roughness, and structure (crystallinity and cross-linking) may also have an effect.

The complexity of starch chemistry and the relationship of starch structure to material properties would be expected to have a significant effect on the properties of starch derivatives such as SSGs. In order to try to understand the reasons for the compaction functionality of SSGs, the pure materials have been studied using slow and medium-speed powder compression. In order to identify any possible relationships between manufacturer, composition, and functionality, the materials were studied using x-ray diffraction, loss on drying, swellability, and Carr's compressibility index.

EXPERIMENTAL

Three fresh "as supplied" commercially available sodium starch glycolates, Explotab (Penwest, New York. Batch Number: E8114), Primojel (Avebe, Holland. Batch Number: 99281-5254), and Vivastar P (Rettenmaier, Germany. Batch Number: 0005/141-142/73) along with Emcocel 90M (Penwest, New York. Batch Number: 9S6055) and potato starch (Roquette Frères, France. Batch Number: 682631) were used as supplied.

Scanning Electron Microscopy

Scanning electron microscopy was performed after carbon coating (Edwards Model 12E6, BOC Edwards, Crawley, UK) using a Jeol 6310 (Jeol Instruments, Tokyo, Japan) system.

Compressibility and Compactibility

Compressibility was measured as the powder column height reduction on application of an applied load from 0 to 100 kN at a compression rate of 0.17 mm/sec.

Compactibility was measured as the tensile strength of the compacts produced at compression rates of 0.17 and 30 mm/sec. Data are reported for $N=8$ or $N=6$. However, in some cases, the integrity of the Vivastar P samples was insufficient to allow the mechanical testing of all the compacts.

Slow-Speed Compression

Compacts (6 g, 25 mm diameter) were prepared at a compression rate of 0.17 mm/sec (100 kN, ca. 204 MPa, dwell time 1 min) using an Instron 1185 test machine. Diametric compression testing (5 mm/min) was performed 60 min after compaction using an Instron 1125. Compression and compaction of each set of batches was performed on the same day to reduce the effects of ambient humidity and temperature. The tensile strength of the compacts was calculated from the failure load.[19]

Medium Speed Compression

Compacts (0.4 g, 10 mm diameter) were prepared using a compaction simulator (ESH, UK) using a saw tooth profile at 30 mm/sec (16 kN, ca. 204 MPa). Diametric compression testing (6 mm/min) was performed 5 min after compaction using a TA-HDi texture analyzer (Stable Micro Systems, UK). Compression and compaction of each set of batches was performed on the same day to reduce the effects of ambient humidity and temperature. The tensile strength of the compacts was calculated from the failure load.[19]

Even though the number of repeat measurements is small, the standard deviations have been calculated and are given in parentheses as an indication of the spread of the reported data.

Carr's Compressibility Index

Samples of the powders (25 g) were poured into a measuring cylinder (50 mL). The measuring cylinder was stoppered and the bulk density recorded. The cylinder was tapped (2000 times, no change in tapped density was detected for more than 2000 taps) using a jolting volumeter (J. Engelsmann, Ludwigshafen, Germany) and the tapped density recorded. Carr's compressibility index was calculated from the bulk and tapped densities.[20]

Loss on Drying

Loss on drying (LOD) was determined using 2 g of powder at 105°C for 3 hr. The sample (2 g) was accurately weighed into a glass sample vial. The vial was placed in an oven at 105°C for 3 hr. The vial was removed from the oven and stoppered. The weight loss was recorded 30 min after removal from the oven. Blanks employing sample vials without the SSG samples were used to confirm that the vials containing the SSGs were satisfactorily sealed after removal from the oven. Samples of the batches were evaluated before every functionality test.

Swelling Value

Samples of the SSGs (0.5 g) were placed in a measuring cylinder (50 mL). Deionized water (25 cm^3) was added and the mixture was mixed and left to stand at room temperature. After 60 min, the volume of the swollen samples was noted and the swelling value calculated according to:

$$\text{Swelling value} = \text{Swelling volume}/\text{Sample mass}$$

X-ray Diffraction

Diffraction patterns were obtained using an x-ray powder diffraction system (Phillips X-ray Analytical, Cambridge, UK). Each sample was analyzed by a single 2θ sweep at a rate of 0.02° 2θ, step time 13 sec. Samples were also analyzed using a sweep rate of 1° 2θ/min to ensure that any apparent differences in diffraction patterns were not due to prolonged exposure to x-rays or atmospheric moisture.

RESULTS AND DISCUSSION

As part of the development of the understanding of the functionality excipients, it is vital that the structure/property relationships of the materials are elucidated. This allows a real understanding of the scientific reasons for pharmaceutical functionality. This is particularly true for polymer-based materials such as SSGs. A simplified chemical structure of SSG is shown in Fig. 1. Primojel and Explotab are described as having low levels of cross-linking[2] with degrees of substitution of 0.23–0.30.[2,4,5] However, it should be remembered that the cross-linking of Vivastar is reportedly via the Na carboxylate and starch primary alcohol groups rather than simply via starch alcohol groups, which probably occurs in Primojel and Explotab, making direct comparisons difficult.

In terms of tabletting, the compactibility of excipients will depend on many characteristics, such as: surface area, crystallinity, morphology, surface energy, and surface and bulk structure. As previously stated, the effects of cross-linking, degree of substitution, and purity on tablet disintegration have been described in the literature.[4,5] In general, these investigations, using experimental grades of Explotab and Primojel, suggested that the products were close to the optimum functionality. However, it was also clear from these studies that subtle changes in the degree of cross-linking, substitution, and purity can affect powder functionality and tablet properties.

This investigation into the characterization and properties of SSGs has been divided into particle structure, particle properties, particle compressibility, and particle compactibility.

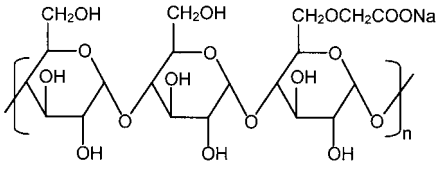

Figure 1. A simplified molecular structure of sodium starch glycolate.

PARTICLE STRUCTURE

Samples of Explotab, Vivastar P, and Primojel were studied using scanning electron microscopy (SEM) to investigate the particle shape and topography of the products. Samples have also been studied using x-ray diffraction in order to investigate the crystallography/order in the materials.

Morphology

Scanning Electron Microscopy

Low magnification examination of the three SSGs and potato starch suggested that, as expected, the disintegrants exhibit particle shapes similar to potato starch.[1] This suggests that the manufacturing processes do not significantly affect particle shape. Typical higher magnification SEM images are shown in Figs. 2–4. The images suggest, as expected,[1,5] that the surfaces of the particles contained small (micron sized) features, presumably of NaCl, and/or Na citrates, and/or Na glycolate: the apparent coverage being similar for Primojel and Explotab, whereas Vivastar P particles contained relatively few such features. These features are a consequence of the manufacturing process, since the surfaces of potato starch particles contained no such features, which is in agreement with a previous study.[9]

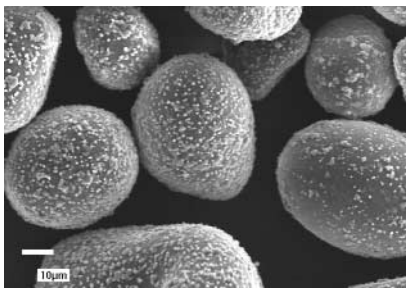

Figure 2. Typical SEM image of Explotab.

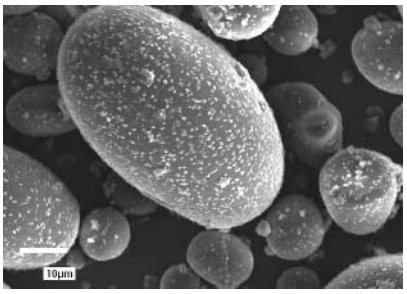

Figure 3. Typical SEM image of Primojel.

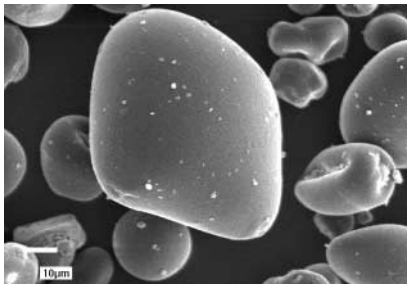

Figure 4. Typical SEM image of Vivastar P.

Batch Impurities

Batches of SSGs have been reported to exhibit batch-to-batch differences in terms of particle properties.[3] One important factor for the compressibility and compactibility of powders is purity. As previously stated, SSGs contain NaCl and Na glycolate and possibly Na citrate salts. However, other impurities, especially those which significantly modify the particle size distribution characteristics, such as agglomerates and manufacturing by-products, would be expected to affect the behavior of the powder column. These impurities will modify the mechanical properties of the composite powder in terms of its response to stress.

When the samples were studied using SEM, examples of anomalous structures were found. Typical images are shown in Figs. 5–7. For example, Figs. 5 and 6 show agglomerates in Explotab and Primojel. The impurity in Vivastar P may be a manufacturing residue, since these types of structures bear no relationship to the shapes of potato starch particles and contained a high level of Na. These observations may be important when considering the mechanical properties of these products and the previously described batch-to-batch variations of SSGs.[3]

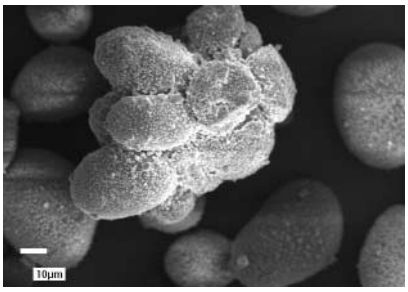

Figure 5. Typical SEM image of agglomerate found in Explotab.

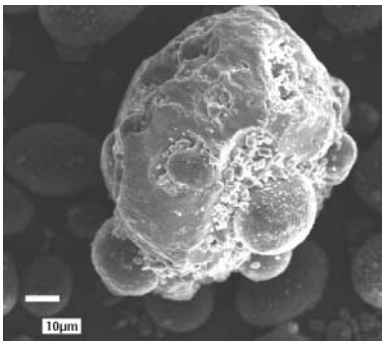

Figure 6. Typical SEM image of agglomerate found in Primojel.

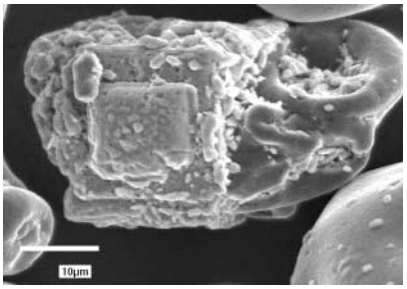

Figure 7. Typical SEM image of agglomerate found in Vivastar P.

Crystallinity

In terms of polymer science, the mechanical properties of semicrystalline materials will be strongly dependent on the crystalline domains. It is widely accepted that starch and starch derivatives are semicrystalline.[13] However, the amorphous components would be expected to have a significant effect on the mechanical properties of starches. Samples of the three SSGs and potato starch were analyzed using x-ray diffraction (XRD). No attempt was made to

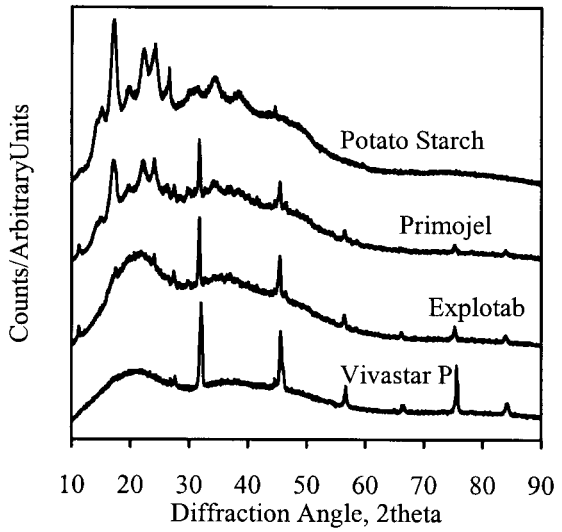

Figure 8. X-ray diffraction patterns of Primojel, Explotab, Vivastar P, and potato starch.

control the humidity during the exposure. Data was collected at a slow step rate in order to increase the signal-to-noise ratio. Shorter scan times were also used to ensure that any apparent diffraction patterns were not a consequence of prolonged exposure to x-rays or atmospheric moisture. The XRD data obtained for Explotab, Primojel, Vivastar P, and potato starch are shown in Fig. 8. The diffraction patterns of the three SSGs contained lines associated with NaCl at ca. 27, 31, 45, 56, 66, 75, 84° 2θ. The polymer diffraction lines of Primojel appear in similar positions to the lines found in potato starch. This suggests that a significant amount of the order in potato starch is retained during the manufacturing process, or that a similar crystallographic order occurs in Primojel and potato starch. The XRD pattern of Explotab did not exhibit the same degree of polymer crystallographic order as observed in Primojel. However, closer examination of the diffraction pattern did reveal that some of the crystallographic order of potato starch was present in Explotab. The XRD pattern of Vivastar P contained few polymer features, suggesting a lack of polymer crystallinity/order in this product. The degree of polymer crystallographic order is Primojel > Explotab > Vivastar P. Additionally, the relative amount of polymer crystallographic order to NaCl content is Primojel > Explotab > Vivastar P. This is an important observation since the data may reflect the destructiveness of the manufacturing process to the integrity of the starch granules. It is also interesting to note that poorly compactable potato starch appears to exhibit a greater degree of crystallinity/order than the SSGs.

PARTICLE PROPERTIES

The core properties of excipients such as SSGs are disintegration, flow and compactibility. The samples were tested for their swelling values as an indication of the swellability of the materials in contact with water. In terms of flow, the average particle size of SSGs is $< 50\,\mu m$, which would be expected to result in relatively poor flow. In order to compare the particle properties, the particle flow and swelling behavior of the SSGs have been investigated.

Swelling Value

The swelling values for the three SSGs are given in Table 1. It can be seen from Table 1 that, as expected, all the samples show considerable swelling in water. All the samples exhibited similar swelling, however, Vivastar P may be more swellable, which may reflect the greater SSG content in this product (higher solid content). The swelling values suggest that the manufacturing processes produce materials with comparable swelling properties. This is in agreement with previous studies which reported similar sedimentation volumes for Primojel and Explotab.[2,3] However, it should be emphasized that these studies did not report the moisture contents in the samples. Moisture levels in SSGs would be expected to affect their apparent particle properties.

Carr's Compressibility Index

The Carr's compressibility index values of the three SSGs are presented in Table 1. It can be seen from Table 1 that the powders exhibit similar Carr's compressibility index values, suggesting that the flow of the powders would be similar. The Carr's compressibility value of Primojel was slightly higher than those of Vivastar P and Explotab, suggesting poorer flow properties.

Table 1

Carr's Compressibility Indices and Swelling Values of SSGs, N=*3 (The Values After ± Are the Range of Data)*

Sample	LOD (%)	Carr's Compressibility Index	Swelling Value (cm^3/g)
Vivastar P	4.0	20 ± 1	24 ± 1
Primojel	8.0	26 ± 1	21 ± 1
Explotab	5.2	19 ± 1	22 ± 1

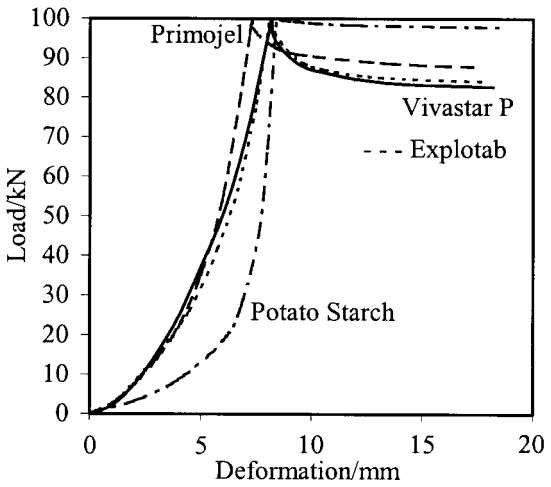

Figure 9. Typical powder compression profiles of SSGs and potato starch. Compression rate 0.17 mm/sec, 6 g, 25 mm diameter.

PARTICLE COMPRESSIBILITY

In order to investigate the behavior of the three SSGs and potato starch under compressive stress, the samples were compressed at a rate of 0.17 mm/sec. Typical compression profiles are shown in Fig. 9. The results suggest that the SSGs and potato starch behave differently under an applied load. The data suggests that, at high stress, Vivastar P and Explotab are more compressible than Primojel (greater reduction in die volume at comparable stress). However, potato starch is a more compressible material under low stress. The compressibility of the SSGs under low stress is similar. The previously described Carr's compressibility indices suggested that Primojel exhibits a higher Carr's compressibility index than Explotab and Vivastar P. The higher Carr's compressibility index suggests that a powder column of Primojel is more compressible than Explotab and Vivastar P. However, it is not possible to compare this apparent increased compressibility with the compressibility of powder columns determined "in-die," since the in-die method employs significantly higher stresses to irreversibly deform the particles. The compression behavior of potato starch, in comparison to starches from other sources, has been explained in terms of its particle shape and consequent relatively high bulk density.[10] However, the compression profiles of SSGs appear to be very different to potato starch. This suggests that even though the shapes of the potato starch granules are retained during the manufacturing processes, the particle packing properties have been modified resulting in powders with different compressibility. This particle modification is also manifested in the apparent reduced lubricant sensitivities of SSG compared to potato starch.[21,22]

All the three SSGs exhibit relaxation during the dwell time, however, potato starch shows a relatively small degree of relaxation. Obviously, the apparent differences in compact elastic recovery and cohesion may reflect the stored energy, since any changes in apparent compressibility may result in the use of different amounts of energy during the compression cycles. However, the difference in compressibility between the three SSGs suggests that these products would be expected to exhibit different in-die powder flow/material properties from pure materials.

PARTICLE COMPACTIBILITY

Samples of the three SSGs were compacted at 0.17 and 30 mm/sec. The mechanical properties of the compacts were evaluated at 5 and 6 mm/min, respectively. Batches of samples were tested on the same day to reduce the effect of ambient

conditions: compacts prepared at 0.17 and 30 mm/sec were tested 60 min and 5 min after ejection, respectively. Additionally, batches of samples were tested on several different days to investigate the effect of powder storage aging (moisture sorption and ambient conditions) on the apparent powder properties. There were no significant changes in compressibility and compactibility when the powders were stored in sealed containers over a relatively short timescale. Typical powder and compact properties of two sets of compression and compaction experiments are shown in Tables 2 and 3. It can be seen from Tables 2 and 3 that there are subtle differences in the powder compaction properties of the three SSGs. In terms of compactibility, Explotab and Primojel form robust compacts with similar compactibilities, whereas Vivastar P was poorly compactable; the compacts invariably lost their integrity during the measurement of their dimensions. Compacts of Vivastar P exhibited poor strength and lower density compared to compacts of Primojel and Explotab. This may be due to moisture sorption and ambient conditions, which suggests that moisture may have a dramatic effect on the apparent properties of pure SSGs. Interestingly, the loss on drying values of the three samples were different, reflecting differing water contents and again suggesting that water may affect the mechanical properties of SSGs. This makes a quantitative comparison between the three samples difficult. The differences in properties between Primojel, Explotab, and Vivastar P are further demonstrated by the compact mass (moisture) gain and toughness (energy of failure) data.

Typical stress/strain curves, which describe the relationship of stress and strain in the test sample during a mechanical property test of the compacts, are shown in Fig. 10. For comparison, a typical stress/strain plot for a compact of microcrystalline cellulose (MCC), Emcocel 90M, prepared under

Table 2

*Powder and Compact Properties of Explotab, Primojel, and Vivastar P (Numbers in Parentheses Represent Standard Deviation, N=8 Except *N = 1, **N = 5. Compacts: 0.17 mm/sec, 6 g, 25 mm Diameter, ca. 204 MPa. Test Rate: 5 mm/min)*

Property	Explotab	Primojel	Vivastar P
Powder LOD (%)*	5.2	7.4	2.1
Compressibility (mm)	8.0 (0.3)	7.3 (0.2)	8.1 (0.4)
Relaxation (%)	17.4 (1.2)	12.6 (0.5)	16.8 (0.3)
Tensile strength (MPa)	2.2 (0.2)	2.6 (0.3)	<0.1*
Compact weight Gain (%)	0.31 (0.02)	0.41 (0.05)	0.95 (0.56)**
Compact density (g/cm^3)	1.30 (0.02)	1.36 (0.01)	ca. 1.1*
Test deformation (mm)	0.33 (0.02)	0.39 (0.03)	0.15*
Energy of failure (J)	0.12 (0.02)	0.16 (0.3)	<0.01*
Norm work of failure (J/m^2)	315 (35)	450 (95)	<10*

Table 3

*Powder and Compact Properties of Explotab, Primojel, and Vivastar P (Numbers in Parentheses Represent Standard Deviation, N = 6 Except *N = 1, **N = 2. Compacts: 30 mm/sec, 0.4 g, 10 mm diameter, ca. 204 MPa. Test Rate: 6 mm/min)*

Property	Explotab	Primojel	Vivastar P
Powder LOD (%)*	5.9	8.1	4.0
Tensile strength (MPa)	0.62 (0.08)	0.29 (0.06)	< 0.1**
Compact density (g/cm^3)	1.18 (0.02)	1.18 (0.01)	1.05**
Energy of failure (mJ)	3.2 (0.7)	1.4 (0.3)	< 0.5**
Norm work of failure (J/m^2)	48 (10)	21 (4)	< 5**

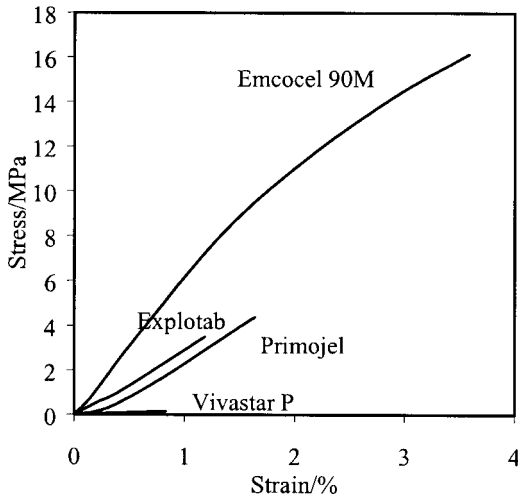

Figure 10. Typical stress/strain data of compacts of SSGs and Emcocel 90M during diametric compression test. Compacts: 0.17 mm/sec, 6 g, 25 mm diameter, ca. 204 MPa. Test rate 5 mm/min.

identical conditions, is superimposed in the figure. It should be remembered that the data do not take into account any differences in compact density. However, it can be seen from Fig. 10 that compacts of Primojel and Explotab exhibit similar stress/strain behavior. Additionally, in terms of binding capability, the mechanical properties of the SSGs are, as expected, considerably poorer than MCC. Compacts of MCC prepared at 0.17 mm/sec exhibited tensile strengths of the order of 10 MPa compared to values of ca. 2 MPa for Explotab and Primojel.

This study suggested that three "as supplied" SSG powders exhibited subtle differences in their behavior under an applied stress during compaction. In terms of compactibility, Explotab and Primojel produce compacts of comparable mechanical properties; Vivastar P exhibits poor compactibility but is more compressible than Primojel. Additionally, Explotab appears to be more compactable at the faster compression rate (the density of the compacts of Primojel and Explotab are comparable at this compression rate). One obvious explanation for this difference in functionality is the moisture content of the materials. However, from the compressibility and compactibility data, particle topography, and XRD studies it is clear that the relationship between SSG source, moisture content (LOD) and mechanical properties is complex. The presence of moisture affects the T_g of polymers and it has been reported that the presence of water affects the T_g of "wetted" Primojel.[17] However, from the compact mechanical property data in Tables 2 and 3, there does not appear to be any dramatic change in the compacted material properties, suggesting that the differences in compressibility between Explotab and Primojel are not a consequence of T_g modification at these moisture contents (a dramatic change in mechanical stiffness would be expected in a polymer which was above and below its T_g).

A possible reason for differences in the mechanical properties of polymers is crystallinity. The XRD data of the three SSGs suggested that Primojel contained the greatest degree of order. This would be expected to result in a powder column with greater stiffness (at higher deformations) than a less crystalline/ordered material. This is observed in the powder compression profiles of Primojel. However, it is still difficult to quantify the relationship between crystallinity and mechanical properties when the materials contain different levels of moisture. An increase in crystallinity of a polymer would also be expected to produce a material with greater strength. This may be a partial explanation for the differences in compactibility of Primojel, Explotab, and Vivastar P.

An important factor for powder compressibility and compactibility is the composition and texture of the particle surfaces. The study of the materials using SEM suggested that the surfaces of Explotab and Primojel particles contained micron-sized features, presumably NaCl, although Na glycolate and Na citrate salts may be present. The size and distribution of these features would be expected to affect powder compression properties; at small deformation the rearrangement (in-die flow) process may be more important, but at higher deformation, mechanical keying may become important. The presence of these impurities further complicates the possible reasons for the differences in compressibility and compactibility of the SSGs. In particular, the effect that moisture has on the adhesive and cohesive behavior between the starch-based polymers and the impurities is unclear, since water can have a dramatic effect on the mechanical properties of starch-based composites.[23]

CONCLUSIONS

Overall, batches of "as supplied" SSGs exhibit subtle differences in compressibility and compactibility: Explotab and Primojel exhibit similar

compactibility, whereas Vivastar P exhibits the poorest compactibility. This behavior was not mirrored in the compressibility of the powders, where Vivastar P and Explotab exhibited similar performance. Characterization studies suggested that Vivastar P is a subtly different material to Primojel and Explotab in terms of moisture content, crystallographic order, and particle topography. However, it is not clear if this may also be due to these differences or subtle variations in the SSG polymer structures.

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