Tableting of Detergents

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TABLETING OFFERS A NEW APPROACH to the marketing of synthetic detergents, and so has stirred up a great deal of interest for various consumer and industrial products. Tableting is not a new process. It has been used for many years by various industries—particularly by the pharmaceutical people. In their case, it was necessary to assure, among other things, that an accurate measured dosage was taken by the consumer to obtain the desired effect. The tablet accomplished this.

Recently, marketing people in the chemical specialty industry realized that the tablet would also give them this feature. Not only does the tablet provide for accurate measurements, but it offers other sales features, too. It is very convenient to use; it is economical, since material is not wasted due to splitting; and it is easy to handle. Marketing people found that the tablet had benefits not only for the consumer, but also for their customer—the storekeeper. They found that the tablet required less space than its equivalent amount of loose powder, and space is vitally important and expensive in this age of supermarkets. In addition, shipping space required in trucks and rail cars was greatly reduced by compacting the powder into a tablet form.

The Aspects

There are three main aspects to be considered in the tableting of detergents—size, shape, and density. The diameter of the tablet will, no doubt, be established by the department responsible for marketing. At the present time, the three leading detergent manufacturers are offering three different diameters, namely: 1¾″, 2¼″, and 2½″. To produce a specific diameter is the easiest phase of the tabletting operation. You merely size the die cavity to the required diameter is the easiest phase of the tableting operatablets are pressed at very low pressures, there is very little expansion of the tablet after it is ejected from the die bore. Normal expansion on most detergent formulations is less than 0.001. Therefore, to produce a tablet to a specific diameter, the bore of the die is machined to 0.001 less than the desired size. In other words, to produce a tablet 2.250 in diam, the tools would be machined to 2.249.

The thickness is governed by the depth of fill, compression ratio and the pressure applied to the feed material. For the most part, detergent formulations have a compression ratio of from 1.4:1–1.7:1. This means that to produce a tablet 1 in. thick, a 1.7:1 compression ratio would require a fill of 1¾ in.

Most detergent tablets are pressed at relatively low pressures in order to keep the density of the tablet to a minimum. The average range is 700–800 psi.

On a tableting press, the thickness is set by the distance between the upper and lower punches and their supporting pressures at compression. This distance can be made constant and thickness can be controlled very closely. However, due to changes in the bulk density of the feed material, which will be discussed later, it is necessary to design press features that make compensating adjustments, insuring that the material is not overpressed. However, if the formulation is prepared properly, and possesses a close particle size range as well as good flow characteristics, the thickness and density will be controlled.

The laundry detergent tablets currently being marketed range from 1¾ in. to 2½ in. in diam. Most of them are very similar in shape—round and flat on opposing surfaces with deep beveled edges. This is not only an attractive feature, but functional as well, as it results in stronger edges, which is very important in handling.

Practically any shape can be made on a tableting machine and we are sure that in the near future more and more functional and novelty shapes will be used. Car shapes for a carwash product or a dish shaped tablet for a dishwasher product are only a few examples of what can be done to increase tablet saleability. Another shape, not yet explored, is the donut type, similar to a lifesaver. This shape is both functional and attractive. It allows a larger surface to contact the water and thereby, increases the disintegration rate of the tablet. For these different shapes it is necessary to make only minor design provisions in the tableting press.

The density of a tablet depends on the bulk density of the feed material, the thickness, and the required weight. It is extremely important that all three factors be maintained since density directly affects the disintegration rate of the tablet. In regard to the feed material use of a light bulk density will improve the disintegration. The average laundry detergent bulk density is approximately 0.6–0.7 g per cc.

The weight required in a tablet will depend on the individual formula being used. At the present time, most laundry detergent tablets on the market are in the one-ounce and two-ounce size. It is important that the weight be controlled for a number of reasons. One is that it is necessary that the amount being shown on the box is actually contained. Supplying excessive weight over the amount shown on the box is actually a give-away of the product. The weight of the tablet is dependent on the bulk density of the feed material. Since all tablet machines are fed on a volumetric basis, it is easy to see how the weight is affected by variation in the feed material density. Additionally, this affects the disintegration rate and therefore, it is extremely important that density be held constant. The average two-ounce tablet with a density of one g per cc, should disintegrate within approximately 100 sec using 120F agitated water. If bulk density is varied from batch to batch, or if the granulation has uneven flow properties, the density and weight will be affected. Devices such as pressure equalizers can be designed into equipment. Again, for a successful tableting operation, it is extremely important that the bulk density of the feed material be controlled.

As far as the tableting operation goes, we are not too concerned with the actual constituents of a formulation; that is, whether it is nonionic or an anionic type. From the various formulae we have tested, we feel design changes can be made in the press to compensate for any variables. We are concerned chiefly with the condition of the feed material. As pointed out, the bulk density of the feed material affects both the weight and density of the tablet. In addition, the condition of the feed material will affect the flow characteristics and the amount of pressure required. We have found that very fine particle granulations have poor tableting characteristics. They usually do not flow well and require higher pressures to compact. Naturally, with higher pressures, the tablet has poor disintegration properties. We have found that a good tableting formula consists essentially of "agglomerated particles." These agglomerates should be very soft and held lightly together by some type of binder. Binders can be either organic or inorganic of the H_2O or solvent dispersible type. The material should have a bulk density of 0.6–0.7 g per cc. A typical screen analysis of what we feel is a good granulation is:

Mesh	%	
+8	2.0	
+16	28.5	95%
+30	40.5	
+60	26.0	
+100	3.0	
−100	0.0	

This type granulation has good flow properties and requires little pressure to form a tablet which has good disintegrating properties. To produce such a granulation, great care must be exerted in the blending stage. First of all, the type of mixer used is extremely important. Mixers that tend to densify material, such as the ribbon type are not recommended. We have found that the best types are the planetary or rotary cone types which blend the material very gently without densifying.

If care is exerted in this step and a uniform granulation is produced, the tableting problems will be greatly reduced. We feel strongly that the preparation of the feed material is one of the most important steps in tablet making.

Tableting Operation

Now that there has been a brief discussion of the various aspects of a tablet, I would like to cover types and operation of compacting presses. I will discuss only the mechanical type since it is felt that they are better suited for detergent tableting due to capacity requirements. Basically, there are two types—the single punch and the rotary press.

The single punch press is used for laboratory work and small production requirements. Information gained from the single punch press can be extrapolated to high speed production equipment. The rotary press, of course, is used for high production requirements.

The single punch compacting press consists of an upper punch, a lower punch, and die. One tablet is produced during every cycle of the press. The presses can run up to a speed of 60 tablets per min, depending upon the flow characteristics of the feed material and the size of the tablet. These presses can be obtained to exert pressure from above only, or from both above and below. Since pressure requirements are so low in detergent applications, we have found that pressure from above is sufficient. The operation of the press is as follows:

The material is fed into the die cavity volumetrically by means of gravity through a feed shoe. The feed shoe passes across on the die tablet and is oscillated over the open die, wherein the material flows into the cavity. As the feeder retracts, it scrapes the powder from the top of the die to insure an even fill. The amount of fill is controlled by the adjustable position of the lower punch in the die. Since there is no overfill arrangement, that is the depositioning of more material than needed into a die cavity and then scraping off the excess, it is extremely important to have a good flowing granulation on a single punch press.

After the feed shoe retracts to its starting position, the upper punch enters the die and compacts the material against the stationary lower punch. The motion on most single punch presses used in a mechanical press originates from an eccentric shaft. The amount of pressure being exerted depends on the position of the upper plunger which is set by adjusting the eccentric. The upper punch then is retracted from the die and the tablet is ready for ejection.

On mechanical presses it is impossible to determine the amount of pressure being exerted unless some pneumatic or hydraulic means is inserted into the compression mechanism. Since detergents require such low pressures, hydraulic units are not as effective as a pneumatic type.

A pneumatic cylinder is inserted in the lower plunger level mechanism which supports the lower punch. The inert gas pressure in the cylinder is used to support the lower punch unit. The mechanical advantage of the lever system is high so that a one ton pressure at the punch requires only 20 lb in the air cylinder. If there is an overload due to excessive material being fed into the die cavity, or for any other reason, the lower punch will release when the support pressure is exceeded. This results in variations of thickness but insures that the tablets are pressed at the same pressure so that the density and hardness will be uniform from one tablet to another. Another advantage of the pneumatic equalizer is that it protects the tools and press from excessive overload.

After the material has been compressed and the tablet formed, the upper punch continues in motion and starts to rise out of the die cavity. The lower punch is then lifted by means of a level mechanism working off a cam which is on the main shaft. The lower punch pushes the tablet up to the die table. The feed shoe then comes across and pushes the tablet down the ejection chute. The cycle is then repeated.

The single punch press is a valuable laboratory tool. It can provide all of the basic information required for a successful tableting operation. Flowability, disintegrating properties, binding abilities, and stickiness all can be determined. In addition, the single punch press can be used for pilot plant productions.

The rotary press is a mechanical compacting press for high speed production. It consists of multiple sets of tools located around the head of the press. The head is rotated by means of a drive worm which is engaged in a gear in the lower side of the head. As the head rotates the punches are guided up and down by fixed cam tracks which control the proper sequence of filling, compression, and ejection. The head can have any number of stations, depending on the diameter of the tablet. On the Model 533 press, for instance, we have supplied 19 station heads for making pieces up to 2½ in. in diam and 33 station heads for pieces up to 1¾ in. in diam.

Each tool station consists of an upper punch, lower punch, and die. The press can have either one or two fixed pressing stations. For high capacity, of course, the press would have two pressing stations so that for every head revolution, two tablets would be produced from each tool station. The pressing station consists of an upper and lower pressure roll. Punches pass between the rolls and the distance that they are apart, represents the final thickness of the tablet. To guide the punches as they revolve around in the head, the upper and lower stationary cam tracks are provided.

The upper cam track consists of a lowering and raising cam at the pressing station and an upper cam body for the punch to ride during the feeding operation.

The lower cam track consists of an ejection cam, pulldown cam and weight adjusting cam. The ejection cam is directly after the pressure roll and raises the lower punch to eject the tablet. The punch is then pulled down for fill by the pulldown cam. The punch passes over the weight adjusting cam for final fill. The punch then passes between the pressure rolls.

The feeder operation is much better on a rotary press than on a single punch press. It employs both an underfill and an overfill arrangement. The material is fed by gravity into a stationary feed frame that is positioned over the die cavities. As the lower punch moves under the frame, it is pulled down to fill by the pulldown cam. The punch is brought down to full drop provided in the press. It then passes over the weight adjusting cam which pushes out excess material. At this point, the punch passes under the scrape off bar, which is positioned at the end of the feed frame. This feeding action is termed "overfilling." It is very desirable since it compensates for poor flowing material. The excess material scraped off is recirculated back to the feed frame. After passing under the scrape off bar, the lower punch drops off the weight adjuster and the material is dropped below the die table before the upper punch enters the die. It also assures that the die bore is sealed before going under the pressure roll. This is termed "underfill." The two punches then go under the pressure rolls.

Pressure is exerted on the material by means of the upper and lower punches moving between the upper and lower pressure rolls. The tablet thickness is determined by the set distance between the two punches. On some straight mechanical presses this is a fixed dimension and if there is any variation in the feed material, it will result in a softer or harder tablet. To compensate for this, it is possible to equip the press with a pneumatic equalizer similar to the type used on the single punch press. If there is overfeeding, the upper or lower roll, depending on the design of the press, will release so that the same amount of pressure is exerted on the material at all times. This assures a uniform density but results in a difference in thickness. Again, it is important that the feed material be kept as uniform as possible to get these variables, namely, thickness and density, within specifications.

After compaction, the upper punch is lifted out of the die by the upper raising cam. The lower punch then rides up the ejection cam and pushes the tablets from the die. The tablet sits on the die table and is knocked off by the front of the feed frame and goes down a take-off chute. The lower punch is then pulled down and goes through the next cycle.

The production rates of the rotary press will depend on many factors such as diameter, depth of fill, and flow properties of the feed material. The presses can be run at extremely high speeds. The 33 station press can produce over 2000 TPM depending on the factors. From what we have seen, we would estimate an average speed of approximately 1200 TPM for a 2 in. diam tablet with a free flowing granulation.

Specific Problems

Now that you are familiar with the tableting presses in general, I would like to cover some of the specific problems detergents cause on high speed rotary presses. Problems encountered on the single punch will not be discussed because most of these presses are used in the laboratory and problems are not too severe. In production, however, these problems can cause serious downtime and be very costly.

As discussed, in order to operate satisfactorily, it is extremely important to have a good free flowing granulation. Most of the materials we have seen possess good flowing properties. Most of these have a moisture content of approximately 10% or less. However, as more moisture is added, the granulations become less free flowing and more difficult to feed into the die cavities.

To obtain good flow of material over the entire die, the feed frame has a number of baffles that direct the granulation over and into the cavities. The openings in the baffles are adjustable and the amount they are opened will depend on the flowability of the material.

Since the diameter is usually large, and each tablet requires a substantial amount of material, it is important to use special wide throat hoppers and feed frames (opening of 4 x 2). However, some material will not flow through this type of feeder and it is necessary to move the powder over the die bore by means of a mechanical feeder. This device consists of two paddle wheels running in separate chambers. The material is fed into the first chamber and transferred over the die cavities by means of a paddle. In the front section of the frame, the lower punch is pulled down for fill. While still under the feeder, it passes over the weight adjusting cam so that excessive material is pushed out of the cavity. The second chamber assures that the die cavity is completely filled. The two wheels are driven by separate varidrive units so that they can be operated at different speeds. The speed should be set so that the feeder transfers from the hopper the amount of material being used in the die cavities. It is extremely important with this type of feeder that the material is not reduced by the bars.

The term "scrape-off" refers to the amount of material that is removed or scraped off by the scrape off bar as the lower punch passes over the weight adjusting cam. The amount is equal to the full fill of the press minus the fill required to produce a given tablet. Material is directed against the spindle of the press and recirculated around to the next feed station. On most tableting operations, this excessive material presents no problems. However, in a detergent tableting operation, due to the large diameter and consequently, more volume of material, excess scrape off can be quite a problem. If it is too great it will build up in the frame, since it cannot be carried away fast enough, and bridge over the scrape off bar back into the die cavity. This will cause a variation in the weight. To counteract this, we have been supplying fill cams at approximately ⅛ in. deeper than the required fill. In other words, if a 1-in. fill is required to produce a piece, we would supply 1⅛ in. fill cams.

The term "sticking" refers to the build up of detergent material on the punch faces. Several years ago this was the problem on all detergent application. However, in the last year we have seen a great many formulae that do not stick to the punch faces. However, in many detergent tableting operations this still is a problem. Normally, in a tableting operation, tool steel is used as the punch and die material. In detergent applications usually it is necessary to use a different material, since material build up is excessive with tool steel.

On most of the presses we have sold, we have recommended Teflon tooling. The tools are made up of a tool holder, metal insert, and teflon insert. As the Teflon insert wears, it can be readily replaced. The tool holder and metal insert wear very little and consequently, replacement is very seldom. We are able to use Teflon in this application, due to the light pressure requirements, 700–800 psi.

We have tried other materials in our laboratory, including silicon spray, different types of resins, etc., but it still appears that Teflon is the best material, In conjunction with the Teflon insert, we have been using a brushing and silicone spray system. After the tablet is ejected, the upper and lower punches are held in position and pass over and under a nylon brush. A silicone emulsion has been sprayed on the brush so that the Teflon face is wiped clean by the brush and at the same time, given a light coating of silicone. With this approach, we have been able to eliminate the sticking problems.

Due to the gummy nature of the detergent materials they tend to build up in various areas on the tablet press. Some materials we have seen will build up on any metallic surface. In this case, it is necessary to eliminate any material to metal contact. To do so, it is necessary to use Teflon in all contact areas. In addition, it is necessary to coat the feed frames and other members with Teflon that comes in contact with the material.

Most detergent materials tend to build up in the die bore. To eliminate this, it is necessary to use an undercut on the lower punches. This undercut serves as a scraper that cleans the die bore as the lower punch is dropped to its fill position. The material is scrapped off and drops through the punch clearances where it is picked up by the vacuum system of the press. You will notice that we use as small an area as possible on the diameter to keep the contact area to a minimum.

Another area of build up is the lower punch socket. When running the tools it is necessary to have clearance between the punch and the die. This clearance enables the fine material to sift down to the punch sockets. To prevent this it is recommended to have an excellent vacuum system at the position underneath the die bore and just above the punch socket.

This has been a brief coverage of detergent tableting. To conclude, we again point out the most important factors in a successful tableting operation are the preparation of feed material and the design of the press.

Phosphate Builders for Detergents

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CRYSTALLINE sodium tripolyphosphate was first made by Schwartz (1) in 1895, by melting together the proper proportions of $Na_4P_2O_7$ and $NaPO_3$, and slowly cooling the melt.

At the present time, two crystalline anhydrous polymorphic forms of sodium tripolyphosphate are known: the low temperature form—Type II, and the high temperature form—Type I. There is also an hydrated form—$Na_5P_3O_{10}\cdot 6H_2O$ which results when either anhydrous form is crystallized from water.

Sodium tripolyphosphate is made commercially by the thermal intermolecular dehydration of a mixture of NaH_2PO_4 and Na_2HPO_4, such that Na/P is 1.67. When such a mixture is heated above 200C, $Na_5P_3O_{10}$ starts to form at an appreciable rate—the Na_2HPO_4 portion of the mix passing through $Na_4P_2O_7$. At 300C, the rate of conversion is sufficiently fast to form $Na_5P_3O_{10}$ in a few minutes. When the orthophosphate mixture is heated to a final temperature of 350–400C, the product resulting is mostly $Na_5P_3O_{10}$-II.

In the temperature range of 450–500C, there is a rapid transformation of $Na_5P_3O_{10}$-II to $Na_5P_3O_{10}$-I. This conversion is usually not complete and the commercial mixed type product contains 25–35% $Na_5P_3O_{10}$-I. With continued rise in temperature, $Na_5P_3O_{10}$ melts incongruently at 622C, forming $NaPO_3$ and $Na_4P_2O_7$.

The formation, decomposition and hydration of $Na_5P_3O_{10}$ are summarized in the following equations:

$$NaH_2PO_4 + 2Na_2HPO_4 \xrightarrow{300\text{–}500C} Na_5P_3O_{10} + 2H_2O \quad [1]$$

$$Na_5P_3O_{10} \xrightarrow{>650C} Na_4P_2O_7 + NaPO_3 \quad [2]$$

$$Na_5P_3O_{10} + 6H_2O \longrightarrow Na_5P_3O_{10}\cdot 6H_2O \quad [3]$$

The thermal intermolecular dehydration of Na_2HPO_4 to $Na_4P_2O_7$ is much more simple and straightforward than the case of $Na_5P_3O_{10}$. Heating Na_2HPO_4 in the range 175–250C causes a disappearance of the orthophosphate and a simultaneous buildup of pyrophosphate. No further change appears to take place until the melting point of $Na_4P_2O_7$ is reached at 985C.

Commercially, a solution of sodium orthophosphate, having a Na/P of 2, is evaporated and thermally converted to $Na_4P_2O_7$. The formation, hydration and dehydration of $Na_4P_2O_7$ are shown here:

$$2Na_2HPO_4 \xrightarrow{200\text{–}400C} Na_4P_2O_7 + H_2O \quad [4]$$

$$Na_4P_2O_7 + 10H_2O \longrightarrow Na_4P_2O_7\cdot 10H_2O \quad [5]$$

$$Na_4P_2O_7\cdot 10H_2O \xrightarrow{30\text{–}40C} Na_4P_2O_7 + 10H_2O \quad [6]$$

The preparation of $K_4P_2O_7$ is quite analogous to that of $Na_4P_2O_7$. K_2HPO_4 begins to dehydrate at temperatures near 250C, the dehydration becoming rapid at 350C. A complication in this process is the ease with which KH_2PO_4 is converted to insoluble KPO_3. It is therefore necessary to adjust the K/P ratio carefully to 2 and convert rapidly, so that the disproportionation of K_2HPO_4 into KH_2PO_4 is minimized, in order to produce a completely soluble product. The preparation of $K_4P_2O_7$ is shown in equation 7. The formation of KPO_3 is given in equation 8.

$$2K_2HPO_4 \xrightarrow{350\text{–}400C} K_4P_2O_7 + H_2O \quad [7]$$

$$KH_2PO_4 \xrightarrow{\Delta} KPO_3 + H_2O \quad [8]$$

The preparation of the glassy sodium polyphosphates is a relatively simple matter. Sodium orthophosphate of the desired Na/P ratio is heated above its melting point of 628C, e.g., 700C, and then quenched by cooling rapidly to room temperature. Graham's salt, which has the theoretical Na/P of 1, contains 69.6% P_2O_5. The glassy polyphosphate of