Compressed Tablets and Lozenges

7

7.1 Introduction

The pharmaceutical industry first produced lozenges and compressed tablets in the mid to late nineteenth century as a way to deliver a specific amount of a drug. Because drug quantities are often measured in milligrams or less, another powder was needed to provide the necessary bulk as a carrier of the active ingredient. The properties of this other (nonactive) powder define tableting ability in pharmaceutical applications.

The earliest such products used the lozenge process, where the drug was mixed with a paste (often containing starch, but also may contain gelatin or gums) to give a dough-like consistency. The dough was rolled into sheets and cut into individual lozenges, which were then dried in an oven. One problem with this approach was the interaction of the drug with water, which had potential negative effects. The manual nature of this process also led to variations in piece size, shape and form.

In a later development, compressed tablets were developed where the powdered raw materials were mixed together, poured into a mold and then compacted with a die punch. The high pressure caused the individual particles in the powders to bind together in a tablet. This process eliminated many of the problems with the lozenge process.

The development of compressed tablet candies followed tablet developments in the pharmaceutical industry, with the introduction of several confectionery brands in the early 1900s. Since then, numerous compressed tablet and lozenge-type candy products have been developed for the market place. These candies typically use either sucrose or glucose (dextrose) crystals as the primary particles, although tablet and lozenge candies can be found with sugar polyols (e.g., sorbitol) as well. Binders, the materials that hold the particles/granules together, typically include corn syrup and a hydrocolloid, such as gelatin or gum arabic.

Some materials, like glucose (dextrose), sorbitol, maltitol and isomalt are suitable for direct compaction as long as the particle properties (size, distribution, density, water content, etc.) are appropriately chosen. Other materials, particularly sucrose, generally need prior treatment, wet or dry granulation, to develop an acceptable powder for compaction. Wet granulation is the process of aggregating individual particles or crystals into granules that can be more easily compacted. In this case, the binder is applied to the powder to make a paste, which is then dried and ground back into a powder. The binder holds the granules or aggregates together and the applied pressure to form the tablet causes the aggregated granules to bond together to form a hard tablet. Alternatively, dry granulation (or dry

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slugging) can be used to prepare a powder for the tablet press. The primary particle is first pulverized and then mixed with the lubricant before being compacted into pellets. These pellets are ground and sized to produce a powder suitable for compressing into tablets.

The microstructural characteristics of tablets are dependent on the nature of the granulation (base particles, binder used, water content, etc.), the type of press, and the compaction conditions (compaction pressure, compaction speed, etc.). Even environmental conditions (temperature, relative humidity) affect granulation flow properties and tablet formation. Sufficient pressure is applied so that the powder particles physically rearrange and/or chemically bond together to form a hard tablet. Under pressure, particles rearrange in such a way that smaller ones fit more closely within the interstitial spaces between the larger ones. Certain types of particles may also deform (either reversibly or irreversibly depending on the nature of the particle) to allow them to move more easily within the matrix. Fragmentation of particles under the applied pressures may also promote rearrangement. Bonding between particles can occur due to electrostatic forces, capillary forces, or the particles could even melt under high pressure, flow together, and then solidify with solid bridges when the pressure is released.

Because very little water is used to make the powder for compaction (or that water is dried off after wet granulation), the water content of a tabletted candy is typically very low, less than 0.5%. The exception to this is when dextrose monohydrate crystals are used to make the tablets. For each molecule of dextrose, one molecule of water is incorporated into the lattice. This 1 to 1 ratio on a molar basis turns out be about 9% moisture on a mass basis (as measured for example by Karl Fischer titration). However, because this water is all bound with the crystal lattice, it is not free to associate at particle surfaces and does not induce stickiness problems.

The hardness of a tablet depends on the nature of the powdered material, which of the above mechanisms holds the particles together, and the applied conditions. Time under pressure, the pressure exerted and temperature of compaction all influence hardness, and therefore the dissolution ability, of a tablet candy.

Lozenges, on the other hand, are not formed under pressure so particle rearrangements do not occur. Lozenge or wafer-type products are formed by mixing dry powder (e.g., confectioner's sugar) with liquid binder to form a paste. When the liquid binder is added to the powder, some of the sugar dissolves as liquid and solid components of the paste equilibrate. This paste or dough is then formed (i.e., sheeting and cutting, extrusion, etc.) to make individual pieces, which are then dried into hard lozenge candies. When water is removed during drying, sugar crystallization forms bridges between particles, contributing to a hard texture. The physical characteristics of lozenges (e.g., hardness) are influenced by numerous parameters, including the type of base particle used, the nature of the binder (how much used and its composition), the water content of the dough, and the final water content of the dried product.

Compressed tablets are, in general, harder than lozenges, although a wide range of textural characteristics can be found for both candies. Some tablets break apart readily in the mouth, whereas other tablets are extremely hard. Certain types of hard tablets are often called "hard" candy even though they are quite distinct from the glassy candies typically called hard candy (see Chapter 8). The physical characteristics of compressed tablets are influenced by both the ingredients and the process conditions used to make the candy.

Tablets and lozenges are typically quite low in water content and therefore, have low water activity (<0.5) and fairly long shelf life. They are also comprised primarily of crystals so they are not very hygroscopic unless stored at humidity above the deliquescent point of the particle (about 80% for sucrose). This is particularly true for sucrose and dextrose-based candies. Some polyol-based tablets (i.e., sorbitol, xylitol) can be quite hygroscopic, so a lower RH environment is required. The main issues with shelf life of tablets and lozenges are loss of flavors or rancidity. Over time, volatile flavors can diffuse out of the candy leaving a product with reduced flavor impact. Flavors such as mints can oxidize during storage, developing an off-flavor. At times, tablets or lozenges may also pick up off-flavors from the surroundings and thereby, lose quality.

7.2 Formulations and Ingredients

Some typical formulations for tablet and lozenge candies are given in Table 7.1. The bulk of a compressed tablet or lozenge candy is the base material. This is the powdered sweetener that is either compressed into a tablet or held together by a binder to make a lozenge candy. Depending on the nature of the sweetener used, a tablet formulation may also include a binder to hold the particles together prior to compression. Tabletted candies contain a lubricant, which keeps the powder from clumping prior to compression and the tablet from sticking to the die once it has been formed. A disintegrant may be used to help in breaking down the tablet during consumption or use. Flavors, high intensity sweeteners, colors and acids are also added to taste. Active ingredients, like vitamins or other functional ingredients, may be added.

7.2.1 Base

The tablet base, which was initially the vehicle for delivering an active compound in the first pharmaceutical tabletted products, makes up the bulk (90–95%) of a candy tablet. In confections, the most common base materials are sucrose, glucose (dextrose), and sorbitol. Glucose can be used either in the monohydrate crystalline form or as a blend with maltodextrins. Recently, sugar-free tablets have been made from isomalt, mannitol or xylitol powders as well as from microcrystalline cellulose. Powdered gum base can also be used for tableting. Choice of base depends on the desired characteristics of the final product, its compression characteristics, and the cost. If an active component is to be used in the compressed tablet, the capacity of the base (amount of active ingredient that the base can hold), and potential reactions with the base must also be considered. Particle size of the active ingredient must be close to that of the binder to prevent stratification and separation.

Table 7.1 Typical formulations (%) for tablets and lozenges

Ingredient	Direct compaction tablet	Wet granulation tablet	Lozenge
Base	98	90–95	90–95
Binder syrup	–	2–8	2–8
Glycerin	–	–	0–3
Acid	0–1.0	0–1.0	0–1.0
Lubricant	0.5–1.0	0.5–1.0	–
Flavor	0.5–1.0	0.5–1.0	0.5–1.0
Color	0.1	0.1	0.1

The type of base used determines the conditions required for tablet processing and the final characteristics of the tablet. Some types of bases (i.e., glucose and sorbitol) can be compressed directly as long as particle size is correct, whereas other bases (sucrose, isomalt, etc.) generally must be granulated prior to compression. Sucrose typically undergoes a granulation process to ensure proper compression into tablets, although a direct compaction version has been developed (Vink, personal communication). The nature of the granulation fed into the tablet press is determined by the compression properties of the base.

In the pharmaceutical industry, there is growing interest in mixing base materials, often called coprocessed excipients for direct compaction. The intent is to mix a ductile material (cellulose or amorphous sugar) with a brittle material (sucrose) to help hold the tablet together. For example, a directly compressible sucrose powder is available that contains sucrose crystals co-processed with a small amount of a polyol. The polyol moderates the compression mechanisms such that sucrose crystal can be directly compacted into a tablet with acceptable properties.

7.2.2 Binder

In a tablet made by the granulation method, a binder is needed to ensure proper press operation. The binder holds the individual granules of base together for compression. In a lozenge, the binder is the glue that holds individual particles together after drying. Common binders in confections include glucose syrup, maltodextrin, gelatin, natural gums (i.e., gum arabic, tragacanth, etc.) or some combination of these. Binders are typically added at levels between 2% and 8% of the final product. Choice of binder depends on the type of base used and the desired texture/strength of the product, whether a tablet or lozenge.

Different binders deliver different properties, particularly in lozenges where the binder is the primary mechanism of holding particles together. Gum arabic typically produces a brittle lozenge, tragacanth gives a hard and smooth lozenge, and gelatin gives a more elastic texture. Combinations of binders give properties intermediate of the individual binder and are often used to reduce costs.

7.2.3 Lubricant

In compressed tablet confections, a lubricant is also needed to ensure proper press operation. The lubricant serves several important purposes. Initially, the lubricant must act as a glidant, to aid flow of the powder material into the press and ensure uniformity of the final product. The lubricant must also act as an anti-adhesive, to prevent the base from sticking to the punch (for compression). Finally, the lubricant helps to ensure that the tablet releases easily from the press without sticking to the die walls.

In confections, the most common lubricants are magnesium stearate, calcium stearate, and stearic acid. In some tablet candies, hardened vegetable fats and mono- and diglycerides serve as lubricant. Choice of lubricant is based on the characteristics of the final product, efficiency of the lubricant with the chosen base and binder, and reactivity with the other components in the pressed tablet. In some systems, higher levels of calcium stearate are needed to give the same lubrication compared to magnesium stearate or stearic acid. However, magnesium stearate and stearic acid tend to be more reactive than calcium stearate, particularly in tablets containing flavors that are prone to oxidation. For example, magnesium stearate can lead to a soapy off-flavor, particularly when made from an animal fat.

Lubricants are added at levels between about 0.5% and 1% of the final product, at a sufficient level to coat the surface of the granulation. Particle size and density of the lubricant, in addition to mixing time and intensity, are factors that affect the ability of the lubricant to coat the granulation. Although it is important that the lubricant coat the granulation, this lubricant coating interferes with bonding between the particles and thereby, decreases hardness of the tablet. For this reason, the lowest level possible for lubrication purposes should be used.

7.2.4 Disintegrant

In some cases, it is desirable to have a product that disintegrates quickly, either upon consumption or, for example, when dropped in a glass of water. Certain types of starch or a soda/acid mix (citric acid and sodium bicarbonate) may be used to help break down candy tablets upon contact with water or saliva. For example, pre-gelatinized (cold swelling) starch swells when it comes in contact with water, breaking the bonds between the particles of the granulation and leading to disruption of the tablet. The combination of citric acid and sodium bicarbonate provide another disintegrant mechanism. They react upon contact of the tablet with water, causing release of carbon dioxide gas and disintegration of the tablet. Tablets that fizz when dropped in water (Fizzies or antacid products) disintegrate rapidly based on the soda/acid system. However, the acid/bicarbonate system is prone to moisture uptake, so their use is somewhat limited.

7.2.5 Acids

A variety of acidulants can be used in pressed tablets, although the main organic acids used are citric and malic acids because they are available in powder form. When using citric acid, tableting must take place at very low relative humidity since the powder is quite hygroscopic. Acid is usually added at 1% of the product mass, although some tablet products contain higher acid content to provide an intensely sour effect.

Acids must be used with some care since they can cause problems. For one, the candy will become more hygroscopic, with a reduced shelf life. Also, the acid may "break" the lake, leading to spotted or mottled effects, particularly for some red and blue lake colorings.

7.2.6 Flavors

The choice of flavor type and level in tablets and lozenges depends on numerous factors. The type of base (whether sucrose, glucose or polyol) has a significant impact on flavor sensation. The sweetness (or lack thereof) and cooling sensation that comes from glucose and most polyols are certain to impact flavor sensation. Furthermore, the compaction density and thus, the rate of dissolution in the mouth, influence the rate of flavor release and consumer perception. Mints and spice flavors were most common for tablets in the past due to their more potent flavoring, although sour fruits with a potent acidulant are now also quite common.

Both liquid and spray dried flavors are used in compressed tablets. However, the effects of the flavor solvent must be considered when using liquid flavors. Oil-based flavors (mints, spices, etc.) can be added in liquid form with no problem. However, solvents like propylene glycol would impede lubricant functionality and result in excessive stickiness of the granulation; thus, spray-dried forms of these flavors are preferred despite their higher cost and higher use levels. Either liquid or solid flavors can be used in the lozenge process.

Flavor levels are generally quite high (0.5–1%) in compressed tablet confections since the slow dissolution of the tablet in the mouth retards flavor release. The exception to this is mint and sour fruit flavors, which give more rapid release. To prevent flavor losses during blending and storage, a problem compounded by the porous nature of the tablet, encapsulated flavors are often used. Spray dried flavors generally lose some potency when the flavor particles are crushed during pressing, but retain flavor better during storage.

Flavor interactions with other tablet components, primarily the lubricant, can lead to problems with compaction. Interactions between flavors, particularly liquid flavors, and lubricant decrease the ability of the lubricant to promote uniform flow and may lead to problems during compaction. Specifically, water or glycol-based flavors can cause the granulation to become sticky and impede flow into the press. In severe cases, the granulation can stick to the punch faces, causing a problem called picking, or misshapen surfaces. High levels of oil-soluble flavors can also cause a reduction in lubricant efficiency, leading to scoring of the side of the tablet as it is ejected from the die. Liquid flavors (nonmint) that contain triacetin or medium-chain triglycerides as their solvent system will provide less reduction of lubricant efficiency than oil-based flavors at a high level.

Fairly high levels of flavors are also used in lozenge candies because of the volatile loss during the drying process. Significant flavor loss, particularly with volatile flavors (e.g., peppermint), occurs during drying. Encapsulated flavors may reduce flavor loss during drying.

7.2.7 Colors

Either dyes or lakes can be used, depending on the process for making tablets or lozenges. In lozenges and wet granulations for tablets, soluble dyes can be used because there is sufficient liquid added initially to disperse the color. In lozenges, color solution is added prior to making the wet granulation. However, tableting by dry granulation or color addition in direct compaction generally requires the use of lakes, in dry form. Very fine lake colors are used to coat the granules. If the granulation is too large, however, the large granules may get crushed and the lake may not be fully dispersed. This leaves white, colorless regions and a speckled or white spotted appearance.

Many exempt or "natural" colors act as dyes and can be used in place of dyes. Carmine and some of the new matrix colors (natural pigments) can replace lakes in direct compression. Color specks and even edible glitter can be incorporated in dry granulation or direct compression for a speckled effect and visual differentiation.

The actual hue for a color depends to some extent on processing conditions, particularly compression pressure. Since the bulk density of the piece changes with applied pressure, the specific hue of given color may vary somewhat.

7.2.8 Actives

Compressed tablet candies can contain up to 25% of active ingredients, although this depends to some extent on interaction effects in compression. Common active ingredients include vitamins, minerals or other functional components like breath fresheners. The advantage of delivery of some of these compounds in tablet form is that there is minimal heating and thus, minimal thermal degradation of the active ingredient. The lack of water is also beneficial to stability of many active ingredients. Some actives are easier to incorporate into the compressed tablet matrix without causing problems, such as excessive softening.

7.3 Processing

Lozenges or wafer candies are made by combining a powder together with a binder, without the use of high pressure to fuse particles (Figure 7.1). In this case, the binder (sometimes called a mucilage) is used to simply "glue" the solid particles together in the wafer form. Once a paste or dough has been formed, lozenges or wafers can be shaped by sheeting and cutting, or they can be extruded and cut to the desired size. Once shaped, the lozenges are dried to a final moisture content less than 1%. The lozenge process is essentially the same process used in making a wet granulation for a tablet press.

Tableted confections are made by compression of a powder in a tablet press. The powdered or granulated raw material is filled into a cavity or die, compressed between punches, and then released from the mold, as shown schematically in Figure 7.2. Controlling the size and shape of the cavity as well as the applied pressure governs the nature of the candy product. In general, higher applied pressure leads to harder tablets, although different powdered materials have their own

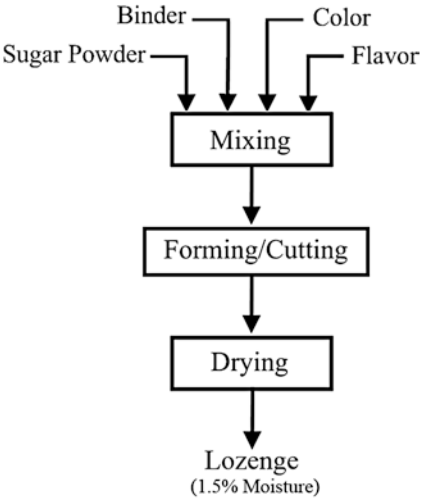


Figure 7.1 General steps for lozenge process

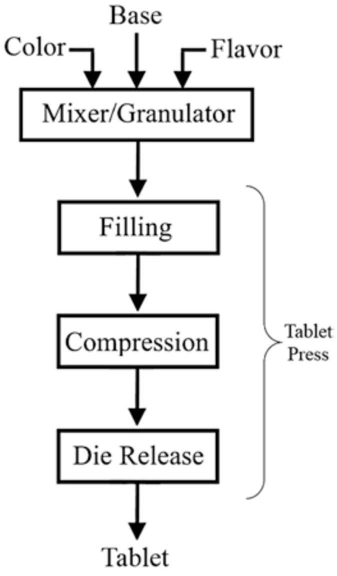


Figure 7.2 General steps in compressing a candy tablet

unique response to compression, particularly when mixed with the various ingredients (flavors, lubricants, etc.) that go into a tablet candy. Particulate characteristics like density, particle size, and shape also influence tablet characteristics. Thus, careful preparation of the powdered materials prior to compaction is essential for control of the desired tablet properties.

7.3.1 Lozenges/Wafers

The mixing of ingredients for lozenge production is relatively simple compared to the steps necessary for tablet production. To make a lozenge candy, the binder ingredients are first blended together, with the binder slowly added to the powdered base until a dough-like consistency has been attained. After the dough is formed and cut, the lozenge pieces are dried in an oven (Figure 7.1).

7.3.1.1 Dough Mixing

A lozenge binder is made by mixing corn syrup, water, and a hydrocolloid (i.e., gelatin, gum arabic, or combination). The solids content of the binder may be about 40–50%. To make a dough consistency, about 15–20% binder is added to the lozenge base (e.g., powdered sugar). This gives a dough with a water content of about 5–10% and a water activity above 0.75. The dough produced in this way is malleable and easily formed into the desired shapes, yet holds its shape during drying. If too much binder is added, the dough becomes sticky and is hard to shape, whereas too little binder results in dough that is crumbly and too firm to manipulate.

Dough mixers for lozenge processing are typically large batch mixers, with powerful motors to completely integrate the binder into powdered base material. Z-blade mixers can be used for mixing dough, although other mixer elements may be used as well.

7.3.1.2 Forming and Shaping

Once the dough is developed, it must be quickly formed and cut to the desired shapes before it loses too much moisture to the environment and becomes hard. A common method of forming lozenges starts by forming a sheet with the desired thickness by forcing it between two rollers, a process sometimes called calendaring. The sheet of dough is collected on a conveyor that removes the sheet at the same speed as it is formed from the rollers. Cutter heads (cookie cutters, drop frame rollers or rolling and scoring cutters) then cut the dough into the desired shapes. In some cases, the cutters have spring release mechanisms to ensure that the cut pieces do not get stuck in the cutters. The webbing material from between the candy pieces is separated by off-set conveyors and recycled back into the mixing device for reuse. Careful control of ingredients and processing conditions are needed to ensure that the dough does not stick to cutting or processing equipment (knives, rollers, conveyors, etc.).

Alternatively, the sugar dough can be extruded into the desired shape and cut to the desired size as it exits the die. The dough is fed to a forming extruder where it is forced through a die with the desired candy shape. As the dough exits the die of the extruder, a knife cuts the stream of dough into a candy piece with the desired thickness. The pieces are collected in shallow trays or on a conveyor and taken to the oven for drying. Often products like sprinkles, confetti and nonpareils are processed in this method.

Products like Conversation Hearts and other wafer candies often have a logo or saying imprinted on one side, sometimes in a color different from the candy color. Such lettering may either be embossed (raised lettering) or, more commonly, debossed (indented lettering) with the appropriate punch design as the pieces are cut out of the dough sheet. Lettering can be printed in edible ink on the surface of a candy in a flexographic rubber roller process, where the lettering is applied to the sheeted candy in a sequenced operation prior to stamping out the piece. The ink is made of edible colors and shellac in a solvent (ethanol is the most common solvent, but some water-based inks are now available).

7.3.1.3 Drying

The formed candy pieces then go through a drier to remove water. Drying must start slowly, with air temperature initially only 24 °C (75 °F), to reduce shrinkage and cracking. Final drying (or stoving) is done with air at 35–40 °C (95–104 °F) and relative humidity less than about 50% to promote drying. Complete drying may take up to 12–15 h to attain the final moisture content. The candy pieces are often turned once during drying to expose both surfaces equally. Once the lozenge candies are dried to about 1.5% moisture, they are cooled and then ready for packaging.

7.3.2 Tablets

Compressed tablet candies are made by forming a compressible powdered granulation and then compressing the powdered material under high pressure in a tablet press (Figure 7.2). Careful attention to the properties of the powder fed to the press is necessary to efficiently produce candy tablets with minimal defects.

In most cases, some preparation of the base and other ingredients is necessary to ensure that the powder flows easily through the press and that the desired compaction takes place. For example, if a granulated sucrose powder (table sugar) was compressed in a tablet press without prior treatment, it would not form a suitable tablet confection. The large (≈400 μm) crystals in granulated sugar are not sufficiently compactable and do not form a cohesive tablet with the desired strength. Even finer powdered sucrose does not compact directly due to the response of the sugar crystals to compaction pressure. To prepare sucrose for tableting, it must first be pulverized to smaller size and then processed with other ingredients before compression. The most common method for preparing sucrose for tableting is the wet granulation method, where a dough is made from binder and sucrose (similar to lozenge processing) and pressed or extruded through a screen. The dough is dried, ground and sieved to proper particle size before being blended with other ingredients (color, flavor, lubricant, etc.) and then fed to the tablet press. However, each base material has different requirements dependent on its properties and compression characteristics.

7.3.2.1 Ingredient Preparation

To help ensure that the powder is suitable for compaction, certain preparation steps are typically needed. Powder preparation may be done in the manufacturing facility or may be done by an ingredient supplier. The three options available to the tablet maker are wet granulation, dry granulation, or direct compaction. The steps involved with each process are outlined in Figure 7.3.

7.3.2.1.1 Wet Granulation

In wet granulation, table sugar must first be pulverized, typically by grinding in a mill (Fitzmill, Tornado mill, Micropulverizer, etc.), to reduce particle size. Grinding should continue until the powder is sufficiently fine. To this ground powder, the wet ingredients like binder (e.g., glucose syrup) and any liquid colors would be added. Since the amount of powder far outweighs the amount of binder, high torque mixers are required to ensure good mixing and a thick dough-like

Figure 7.3 Process steps for ingredient preparation for tablet production (Adapted from Vink 1996)

Wet Granulation	Dry Granulation	Direct Compaction
Pulverizing	Pulverizing	Dry Mixing
Wet Mixing	Dry Mixing	Tableting
Granulation	Compaction	
Drying	Grinding	
Grinding	Sizing	
Sizing	Mixing	
Mixing	Tableting	
Tableting		

product is the result. Often, dough mixers or sigma-blade mixers would be used for this purpose. The dough would then be squeezed through a screen to form a granule. A paddle or bar forces the dough through the screen, which can be either rotary or oscillating type. In batch processing, each of the above steps would be performed in a separate piece of equipment. The candy mass would be moved either manually or by conveyor from one piece of equipment to the next.

The wet granulated product may contain anywhere between 3% and 5% moisture and should be dried to 0.2–0.8% moisture. Static bed driers, fluidized bed driers or a continuous rotary (kiln) drier can be used to remove moisture. The dried product must then be ground and sieved to the correct size for compaction. Temperature control during drying is important to minimize negative changes in the granulation. For example, if gelatin is used as the binder, temperatures above the point where degradation occurs must be avoided in the drying stage of wet granulation, so air temperatures should be less than about 93 °C (200 °F).

At this point, it is necessary to ensure uniform particle size in order to prevent problems with control of the compaction step and the final product characteristics. The dried product is ground in a mill until it reaches the desired particle size distribution. The optimal size range for tableting depends on the powdered material being compacted, among other factors. The nature of the other ingredients used in the formulation also influences the optimal size range. No single standard size distribution recommendations cover all types of granulations and base materials. For a sucrose powder based on wet granulation, a standard recommendation might be that 100% of the particles pass through a 20-mesh sieve (840 μm) and at least 90% is retained on a 60-mesh sieve (250 μm). For a sorbitol product, a distribution of sizes between a 60 and 100 mesh size (150–250 μm) gives good compaction results. In all cases, it is important to have somewhere around 10% fine particles that fit into the interstitial spaces between the larger particles, helping to hold them together and give strength to the tablet.

Once the granulation has been ground and sized, all other ingredients (flavor, dry color, acid, any active ingredients and the lubricant) are mixed in with the granulated base material. Typically, liquid flavors are added first to provide for uniform distribution. Dry colors and acids are added separate from liquids to avoid spotting. The lubricant is added last to ensure complete coating of all particles. This mixture now forms the free-flowing base for the tableting step.

Several processes are available for what might be called “all-in-one” wet granulation, where all the steps leading to formation of a granule for grinding are done in a single unit. For example, a fluidized bed granulator can be used to prepare a wet granulation for compaction. An air stream fluidizes the individual particles and the wet binder is sprayed onto them. Agglomeration of individual particles forms a granule, which is then dried by the warm air fluidizing the particles. Lubricant and flavor may also be added to the dried granules in the fluidized bed. Another efficient process for wet granulation is extrusion. Extruders that combine wet mixing, granulating, drying and final blending in a single unit are available. For example, a twin screw extruder can be used to blend the powder with the wet ingredients, dry the mass by pulling a vacuum within the extruder, and then blend the dry ingredients to make the final granulation. Subsequent screening may still be necessary prior to feeding the tablet press. The continuous extrusion process is extremely efficient; however, fairly high product throughputs (up to 2,500 kg/h; 5,500 lbs/hr) are needed to make the process economical.

7.3.2.1.2 Dry Granulation

In dry granulation, the first step of the process is to pulverize the base material, just as in wet granulation. A small amount of dry binder (e.g., gelatin) is added after pulverizing – the granulation and drying steps found in wet granulations are not needed in this process.

After the pulverized base has been mixed with the binder, the mixture is compacted into pellets. This may be done in a roller compactor, where the powder is fed into a narrow gap between two pressure rollers, or by slugging, where the powder is formed into tablets at slow speed and high pressures. The compacted pellets are ground into smaller size in a communitor (or oscillating granulator) and sized prior to addition of flavor, color, acid, active ingredients, and lubricant. Addition of these components is done in the same order as in wet granulation. The same granulation size distributions as in wet granulation apply here as well. The free-flowing mixture is then ready for tableting. Sucrose, glucose, lactose, microcrystalline cellulose, and sorbitol are examples of materials that can be dry granulated.

7.3.2.1.3 Direct Compaction

In direct compaction, the base material particulates possess desired properties (fracture, deformation, etc.) for making a suitable candy tablet. These bases generally do not require granulation to produce acceptable tablets. However, not all base materials have these properties. Sorbitol powders can be compacted directly without pretreatment due to the nature of sorbitol crystals. The γ-polymorph of sorbitol has the best direct compaction properties and is not as hygroscopic as the other forms. Lactose, lactitol and mannitol powders can also be directly compressed into a suitable tablet. Amorphous corn syrup powders can also be directly compacted. In fact, spray-dried materials such as glucose syrup solids (high DE) and sorbitol mixtures make excellent bases for direct compaction because the granules are spherical and have lubricant only on the outside surface. When compacted, the nonlubricated center of the granule provides good adhesion and results in stronger bonds between particles.

In another form of direct compaction, a prepared base material is purchased from a supplier and the candy manufacturer simply adds the desired colors, flavors, acids, active ingredients, and lubricant prior to tableting. The advantage to the candy manufacturer is the ease of use and simplicity of the process. However, because the supplier has performed the preparation steps (wet or dry granulation), where necessary, the cost of the pre-made base is typically greater than that of the raw materials.

7.3.2.2 Powder Conveyance

In preparation of the base prior to and during tableting, it is important that no changes take place during conveyance to the press. For one, environmental conditions must be carefully controlled. Since many powders are hygroscopic, relative humidity in the air must be sufficiently low that moisture pick up is not a concern. If relative humidity is too high, the powder (particularly for sorbitol and dextrose monohydrate) quickly loses its free-flowing capacity as the surface of the particles become sticky. This can lead to numerous problems during tableting. Temperature may also be a concern for certain base materials. Melting of the lipid components may be a problem at elevated temperatures. Amorphous base materials may also become sticky with increased temperature.

An important property of the granulated powder fed to the tablet press is its particle size distribution. This determines how well the particles squeeze together under pressure to bond into a tablet. In general, a fairly uniform distribution of sizes is best, with sufficient "fines" to fill in the interstitial spaces between larger particles. Air conveying can cause classification of sizes that cause problems with tableting (weights off and thickness differences). Thus, bucket conveyors are generally a better bet for ensuring that the powder is fed uniformly into the hoppers. Screw conveyors or air conveyance may also be used but must be carefully controlled to minimize particle degradation.

Although numerous factors affect the flow characteristics of powders, the Carr's Index (or Carr's Compressibility Index) is often used as a quick check on these properties. The Carr's Index (CI) is defined as:

$$CI = \frac{\left(V_B - V_T\right)}{V_B} 100 \qquad (7.1)$$

where V_B is the volume of the powder allowed to settle freely while V_T is the volume of that powder after controlled tapping to allow it to settle further. The Carr's Index can also be written in terms of bulk densities (mass per unit volume of the powder):

$$CI = \frac{(\rho_T - \rho_B)}{\rho_T}100 = \left(1 - \frac{\rho_B}{\rho_T}\right)100 \quad (7.2)$$

where ρ_T is tapped bulk density and ρ_B is freely settled bulk density.

The CI provides insight into how well a granulation will flow and compact. As a rule of thumb, CI values between 15 and 25–30 give good flow properties and compact well. CI values below 15 will flow well but will not compact very well. CI values above 25–30 will compress well, but will cause flow problems when feeding the press. Factors that affect CI include particle size distribution and mean size, particle shape and moisture content, especially surface moisture. This latter parameter depends to a great extent on the relative humidity of the environment, which should be controlled to ensure proper flow properties.

7.3.2.3 Compression

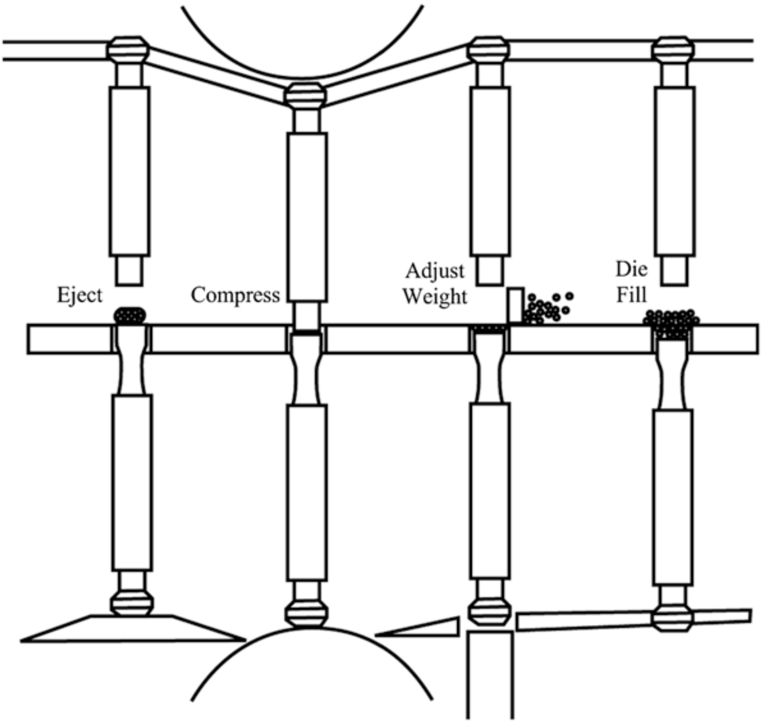
Once an appropriate powder has been prepared, it is ready for feeding to the tablet press. For the best tablet production, both environmental conditions and tablet press operating conditions must be carefully controlled (Vink 1993).

7.3.2.3.1 Tablet Press

Once the dry powder materials are ready, tablets are formed by compression of the prepared base in a die press. A tablet press involves a series of steps, with powder flowing into the unit through a hopper and tablets exiting the chute after compression (Figure 7.4). First, the granulation is fed to the press and flows into the die (Figure 7.5a) where the weight of powder in the cavity is adjusted to give the desired weight of tablet (Figure 7.5b). The powder is then compressed between two punches under high pressure to form the tablet (Figure 7.5c), which is then ejected from the press, usually by raising the bottom punch to push the tablet out of the press (Figure 7.5d). Typically, these operations take place in a continuous series of events in a rotary device, where movement of both bottom and top punches is controlled by a set of cams and tracks (Figure 7.6).

To control product piece weight, it is necessary to control the flow of the granulation into the

Figure 7.4 Operating steps of a rotary tablet press (From Vink 1996)



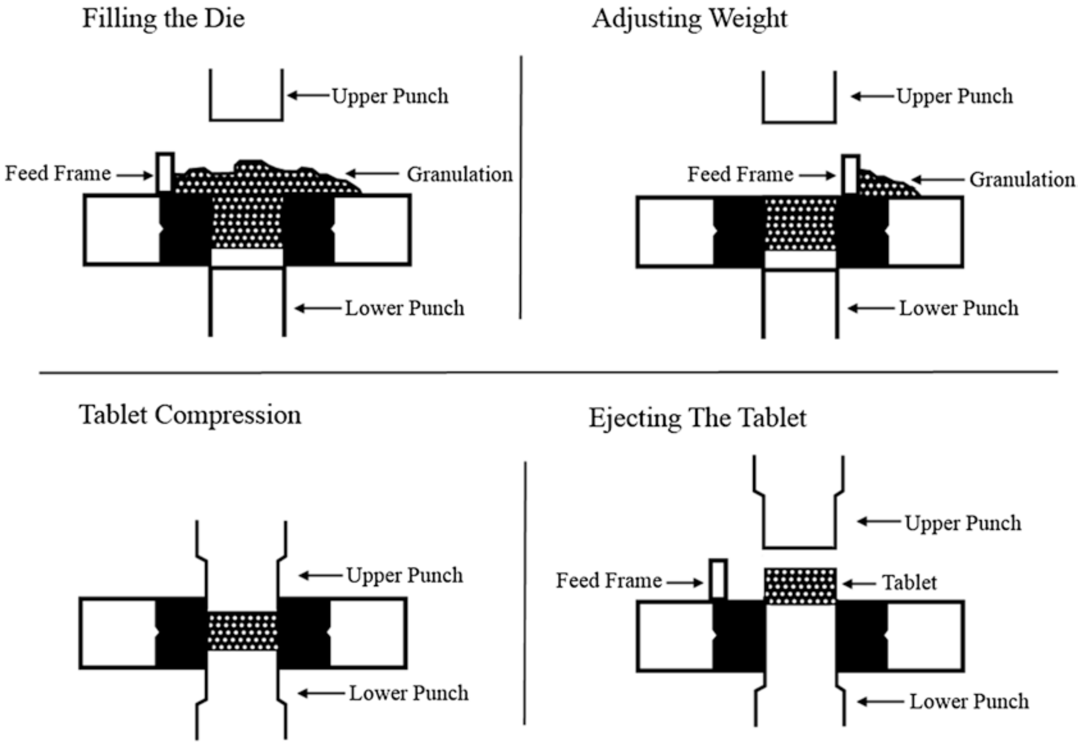


Figure 7.5 Detailed operations of a tablet press (From Vink 1996)

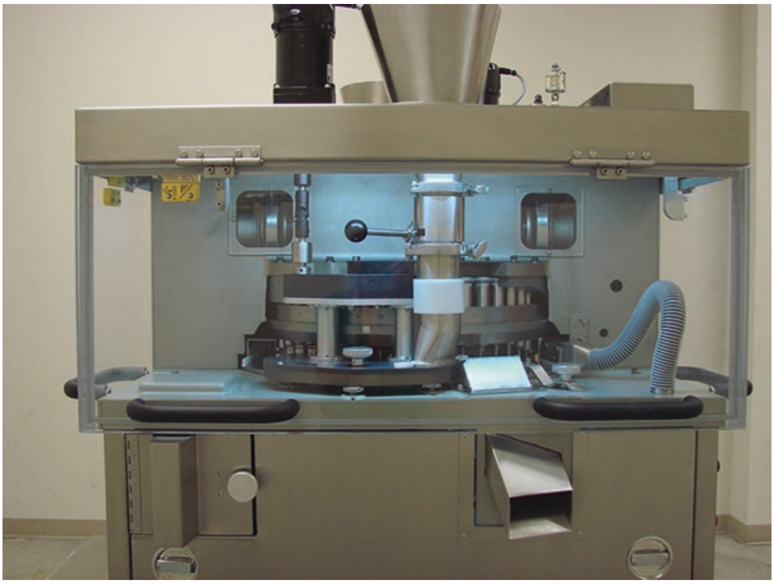


Figure 7.6 Rotary tablet press, model Stokes 328 MFA (Courtesy of Compression Components and Service, LLC)

press (Figure 7.5a). It is absolutely essential for good press operation that the powder be completely free flowing. Further, if the base material has not been mixed properly and does not have uniform bulk density, control of base flow will be difficult and problems in operation of the press will be encountered. Either controlled gravity flow or a forced flow feeder can be used to ensure proper filling of the cavity in the die press.

Once the die has been filled with a base material, the bottom punch moves into the correct position to set the weight of the piece and a scraper clears excess base material off the shelf (Figure 7.5b). This step ensures that the same weight of base material is compressed in each stroke. Once the die has been properly filled, the bottom punch is positioned at the correct level and the top punch enters the die cavity (Figure 7.5c). The tablet is compressed between the two punches with the force controlled by the distance set between the punches (set by the rollers that control the punches), the depth of fill, and the override pressure, which is set on the press. Compression forces are applied in the normal direction of the punch movement (vertically) with a resultant force in the normal direction to punch operation. The dwell time of the tablet under compression must be controlled to ensure proper bonding of the particulates. Typically, dwell time is governed by the operating speed of the press, but usually falls within 50–500 ms duration.

After the tablet has been compressed, the top punch is pulled away and the bottom punch is raised to eject the tablet from the cavity (Figure 7.5d). Candy pieces are then collected from an exit chute, and sent on for packaging.

To set the tablet press, trials are performed at different applied pressures and the tablets are weighed to ensure the proper weight is attained. If adjustments are necessary (weight too high or low), the applied pressure and the cavity fill level are adjusted until product with the correct hardness and set weight are produced consistently. Tablet thickness is governed by the amount of powder fed into the die (fill weight) and the pressure applied during compaction.

Many modern tablet presses have the capability of producing multiple layers or a layer within a layer. Sequential feeding zones and compression steps allow production of these more complex tablets.

The shape of the candy tablet is, of course, dependent on the shape of the die punches used to compress the tablet, with a wide range of sizes, shapes and thickness available. However, some shapes are easier to make because of the nature of the applied forces during compression. Discs are the easiest to compress because the forces applied in the punches are primarily normal to the shape of the tablet and the forces are uniformly applied across the entire piece. As the shape deviates more and more from disc-shaped, the distribution of applied forces leads to different extents of compaction across the tablet. This variation across the piece may lead to nonuniform products.

Text can be applied to the surface of the tablet through special design of the die punches. For example, embossed lettering on the tablet piece is created by making a die with the reverse lettering on the surface. As the punch compresses the powder, the raised lettering on the punch creates the reverse lettering indentation on the tablet. Debossed lettering may be made with a punch that has indented lettering on the surface.

7.3.2.3.2 Compaction Pressure

To compress a tablet, a force is applied through a die punch into the powder to cause compaction of the granulation. Compaction pressure is then defined as the applied force divided by the surface contact area between the die punch and the powder. A larger punch size requires more applied force to attain the same compaction pressure, which effectively limits the size of tablets that can be produced. For this reason, larger tablets tend in general to be softer because of the lower applied pressure (larger surface area).

Compaction pressures in the confectionery industry are typically defined in tons, which is actually a misnomer. The unit “tons” is actually a mass unit, similar to pounds (1 ton = 2,000 lb) or grams. However, just as mass is sometimes used to define a force (as in pounds-force), ton-force (or 2,000 lb-force) is also used to define the force applied to the die punch in tableting. The actual pressure then depends on the contact area between the punch and powder. A more correct unit for applied pressure is the MPa (megaPascal), although the confectionery industry generally does not use these units, preferring the older "ton" of pressure. Regardless of the units, only when the punch size is defined is there a simple conversion from applied force to pressure (force per unit area).

With that understanding of the terminology for pressure and force, confectionery presses are typically operated in the range of 2–10 tons of pressure. Typical presses used for candy manufacturing have maximum over-ride pressures in the range of 3–6.5 tons for "B" machines (maximum tablet diameter of 7/16–5/8 in. or 1.11–1.59 cm) and 10 tons for "D" machines (maximum tablet diameter 1 1/16 in. or 2.7 cm). This is the pressure at which the over-ride spring or hydraulic system will release the punch pressure. Note that presses are not run at these pressures because damage can occur to the press. The over-ride is simply a safety device. Many operators mistakenly think this is the actual tableting pressure. More expensive computer controlled machines, typical of those used in the pharmaceutical industry, can measure actual tableting pressure and tablet release forces.

The actual pressure at the tip of a set of punches is a function of the area of the tip. That means that a quarter inch diameter (0.635 cm) punch running on a thickness setting for a half inch (1.27 cm) tip will exert six times the pressure that the half inch punch will exert. The ratio is calculated as the ratio of the square of the radius of the tips. The quarter inch punch would have to be run at an over-ride of one-sixth of the half inch setting to provide the protection to the press and the tooling.

7.3.2.4 Safety in Manufacturing

Besides the safety and sanitation concerns of any food processing facility, tableting plants have additional concerns related to fine powders in the environment. Given the proper environmental conditions, the dust generated in operations of a tablet facility can ignite, leading to an explosion and subsequent fire. Dry conditions, abundant fine powdered particles and a spark are what it takes to have a powder explosion. Although relatively rare, when such explosions do occur, there is often significant structural damage.

Most facilities that are especially prone to powder explosions have various safety procedures and mechanisms incorporated into the plant operation. Such strategies include adequate cleaning procedures, proper construction design, and explosion containment devices. Most importantly, good housekeeping practices help to minimize dust build-up. Constant cleaning to minimize dust build-up will remove the potential for a dust explosion. Construction practices are also often part of the prevention process. Construction designs that minimize dust build-up help because they prevent accumulation of sufficient dust to foster an explosion. For example, minimizing pipe and ductwork near the ceiling of a plant and enclosing open construction materials (e.g., I-beams) help prevent dust build-up.

One element needed for a powder explosion is a confined space. Thus, plants with high powder explosion risk may be designed with, for example, a blow-out roof. That is, as soon as an explosion occurs and the pressure builds, the roof of the facility blows out, allowing the blast to release itself. This minimizes damage within the plant itself (but of course raises the roof). Fire containment strategies are also used to control fire explosions. These may be designed, for example, to release a chemical retardant at a strategic location if a pressure sensor indicates the onset of an explosion.

Through a combination of good manufacturing practices and proper facility design, the danger of powder explosions can be considerably reduced.

7.4 Product Characteristics

Tablets and lozenges are somewhat unique among confections since they are not made by boiling a sugar syrup to specific water contents. Instead, they are prepared by mixing powdered sweeteners with binders, either wet or dry. In the case of tablets, the powders are compressed under high pressures to force interparticulate bonding. The nature of the interparticulate forces determines the properties of the candy tablet. With lozenges, a combination of particulate bridging due to recrystallization (as the binder is dried) and adhesive action of the remaining liquid binder holds particles together and imparts the physical properties of the lozenge candy. Both tablets and lozenges are typically very hard, although hardness can be moderated to some extent through an understanding of the effects of formulation process conditions on the microstructure.

7.4.1 Microstructure

The arrangement of particles is slightly different between tablets and lozenges based on the different process for their manufacture and the forces that hold the piece together. In fact, lozenge formation is generally the starting point for tablet formation through wet granulation. That is, the dough made during wet granulation is either formed and dried to make lozenges or it is dried, ground and sieved to make a powder suitable to feed into the tablet press.

7.4.1.1 Lozenges and Wafers

A typical lozenge binder contains corn syrup, stabilizer (e.g., gelatin and/or gum arabic) and water, with a dissolved solids content around 40%. When this liquid binder is added to the powder base, a small amount of the base crystals dissolve in the water until the liquid phase equilibrates with the solid crystalline phase. Although the binder initially does not have any dissolved base material (sucrose, dextrose, sorbitol, etc.), once it has begun to mix with the dry powder, the base dissolves until the equilibrium solubility concentration for that particular mixture (see Section 2.8) is reached. Because the amount of binder added is relatively small and the crystalline content is still very high (>90–95%) in the mixture, a dough-like paste is formed. With the proper ratio of binder to base, this dough can be easily formed and shaped into the desired pieces.

The consistency of the dough depends primarily on the water content, although the amount and composition of the binder added have an effect as well. High water content gives a dough that is soft and pliable, but that may be prone to stickiness. If the water content (binder level) in the dough is relatively low, the dough is firm and may be difficult to form.

Upon drying to about 1–2% moisture, most of the water in the binder is removed, resulting in subsequent recrystallization of the sugar as the water is removed and a supersaturation is generated. Because the base particles are in such close proximity, it is likely that solid bridges form to hold some of the base particles together. Other particles are held together by the action of the hydrocolloid and corn syrup in the binder through capillary forces.

The water content of the dough has a significant effect on the hardness of the final product. A high water content in the dough means that a relatively large amount of liquid binder solution has been added to the powdered base. Thus, more base crystals dissolve in the water of the binder, meaning that more base must recrystallize when the lozenge is dried to the final water content (1–2%). More bridges are formed between the sugar particles and these structural elements lead to a harder product, one that requires more force to break. In contrast, dough made with low water content (relatively low binder addition level) means less recrystallized sugar, which generally leads to a softer wafer.

A combination of solid bridges, capillary forces, and hydrogen bonding forces are at work to hold the particles together in the dried lozenge. Depending on the relative extent of these forces, lozenge and wafer candies can be found with a wide range of hardness. In general, these candies are quite hard, such that they are either sucked in the mouth or crunched by the teeth with considerable force.

7.4.1.2 Compressed Tablets

During compaction under an applied pressure, a powder transforms from a free flowing mixture of independent particles to a unified tablet held together by various forces. The forces that hold the particles together in a tablet may be chemical (molecular) interactions between the particles or physical forces that prevent movement. Usually, a combination of physical and chemical forces holds a tablet together. The microstructure of a compressed tablet depends on the applied pressure, the dwell time under pressure, and the nature of the bonding forces between particles.

To make a tablet, a free-flowing powder of discrete particles (either individual particles or agglomerated granules) enters the die press where it is compressed at high pressures between two die punches. The initial stages of applied pressure cause rearrangement and, in some cases, fragmentation of the powder particles as the forces applied to any single particle increase. Rearrangement and fragmentation depend on the particle size and shape as well as the fracturability of the individual particles. Under applied pressure, the particles may also deform, either in elastic deformation or plastic deformation. Plastic deformation causes an irreversible change in shape, whereas elastic deformation means the particle returns, at least for the most part, to its original shape. Of course, these deformations result in further rearrangement under the applied pressure. At the same time, interparticle bonds are being formed as influenced by both electrostatic forces and molecular interactions (van der Waal's forces, electrostatic interactions, etc.). Furthermore, if any water at all is involved (for example in water of hydration in a crystal or even residual water in the granulation) there may be capillary forces that influence interparticle bonding. Any type of flow, whether due to the presence of water or deformation (plastic or elastic) of the particles can lead to formation of solid bridges between the particles when the pressure is released. These solid bridges result in formation of a strong, not easily broken, interaction between particles, which leads to a hard tablet. It is the sum of all the forces holding particles together that leads to the physical characteristics (density, hardness, etc.) of the finished candy tablet.

The combination of mechanisms holding any specific tablet together depends, to a large extent, on the type of granulation used to prepare the powder for compaction. In tablet formation following wet granulation, the binding between the aggregates of the granulation will certainly involve capillary forces. In wet granulation, the surfaces of the individual particles of the base (and other materials) are wetted by the binder (and other liquid ingredients). When this wetted powder is compacted and dried, capillary forces are responsible for holding the individual particles together in a granule. Although some aggregates are broken down by subsequent milling, many retain their aggregated nature when they are put into the tablet press. Application of force in the press causes rearrangement and fragmentation of these aggregates in the granulation, resulting in the physical forces holding the aggregates together. In this sense, the binder serves to hold the aggregates within the granulation together (often called binding), but it is primarily the physical forces exerted during compaction that hold the tablet together (often called bonding). Figure 7.7 schematically shows the granular and particulate structure of a tablet pressed from a wet granulation preparation. The two levels of structure, within the granules and within the tablet, are clearly evident.

In dry granulation, particularly in the absence of a significant amount of water, rearrangement and particle fragmentation are the primary mechanisms that lead to formation of a coherent tablet although particle deformation may also have an impact depending on the nature of the particles. A dry binder, like microcrystalline cellulose or maltodextrins, may be added to help

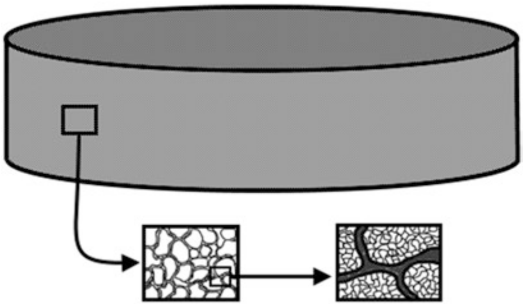


Figure 7.7 Schematic depiction of tablet microstructure when prepared from a wet granulation (After Alderborn and Wikberg 1996)

rearrangement and bonding of the crystals. However, because the dry binder is typically not as uniformly dispersed as the binder in the wet granulation process, more variability in the tableting process and in the final properties of the tablets may be noticed with dry granulation.

Direct compaction, similar to dry granulation, causes particulate rearrangement due simply to the applied forces. However, direct compaction does not work successfully for all tablet bases due to the nature of particulate breakdown. For example, although sucrose crystal powders have adequate flow properties and potentially can be directly compacted, tablets made by direct compaction of sucrose have tablet strength that is generally too low for confectionery products. Extensive fragmentation of sucrose crystals occurs under high applied pressures; however, it is thought that these fragments remain where they form instead of migrating into the interstitial spaces between larger particles to give stronger interparticle forces. Apparently, the large frictional forces between fragments prevent this migration and the result is weak tablets that break up too easily. Because of the poor compactibility of sucrose crystals, wet granulations were developed to make it more efficient for compaction. Dry granulations where sucrose crystals are blended with other materials (i.e., maltodextrins, polyols, etc.) to help compaction are now available.

7.4.2 Porosity and Density

Although it is probably hardness of candy tablets that signifies physical properties to the consumer, it is first necessary to understand the particulate nature of these candies and the powders from which they are made. Porosity and bulk density of a powder are functions of the size, shape, and interparticle forces between individual particles. A powder can be considered a mixture of a solid material (the particles with high specific density) and air (with low density). The amount of air entrapped between the particles of a powder determines the bulk density of the mixture and also the porosity, or the space between particles. The ability for molecules to move within the powder is primarily governed by porosity since it is only in this phase that molecules like flavors can move. Particle shape and size distribution are important factors in porosity. Uniformly large particles cannot pack as tightly as a mixture of large and small particles, nor can irregularly shaped particles pack as tightly as spheres. Thus, both particle size and shape greatly impact bulk density of a powder.

When a granulated powder is compressed, the particles are forced closer together, the volume of the powder bed decreases, its bulk density increases as more air is excluded, and thus, its porosity decreases. The void spaces between the particles or aggregates decrease as compaction occurs. The extent of compression, which is related to the applied force, determines the extent of decrease in volume and porosity, and consequent increase in bulk density. Bulk density can be defined as the apparent density of the powder or compaction and is given by the weight per bulk volume of the powder. Measuring bulk density requires accurate measurement of the height and diameter of the tablet (or powder column for the granulation prior to compaction). The relationship between particle density (ρ_s), bulk density (ρ_b), and porosity (ε) is given as:

$$\rho_b = \rho_s (1-\varepsilon) \tag{7.3}$$

According to Equation 7.3, as the void fraction of air in the tablet decreases with compaction, the bulk density of the tablet increases. Equation 7.3 can be rewritten for porosity:

$$\varepsilon = 1 - \frac{\rho_b}{\rho_s} \tag{7.4}$$

Thus, porosity decreases as the bulk density of the tablet increases. As bulk density approaches particle density, the amount of entrapped air decreases and porosity decreases. This is exactly what happens during tableting, where a powder with high porosity is compressed into a tablet with higher bulk density (more particles are packed into the same volume). Porosity decreases during tableting, depending on the applied forces in the press and the nature of the granulation.

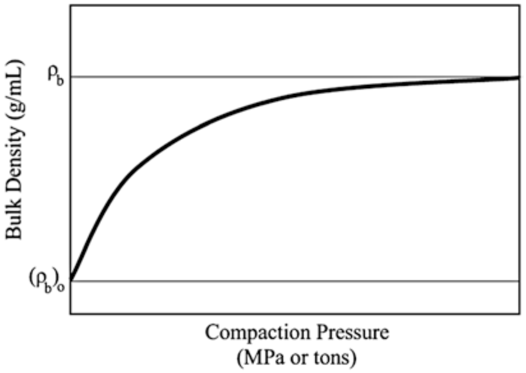


Figure 7.8 Typical increase in bulk density, ρ_b, with compaction pressure (After Shinohara 1984)

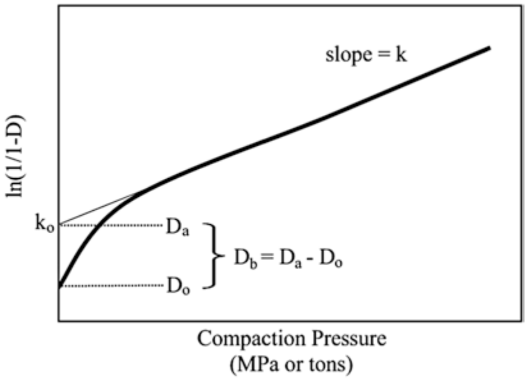


Figure 7.9 Typical Heckel plot showing change in relative density (D) with compaction pressure (After Paronen and Ilkka 1996)

In general, higher compression forces lead to thinner tablets with higher bulk density and lower porosity, although there is typically some upper limit where no further changes are observed with greater applied pressures. That is, particle fragmentation and flow reach some upper limit, beyond which no further rearrangements of individual particles are possible even with higher compression forces. The general trend of bulk density increasing with applied pressure during compaction is shown in Figure 7.8. At relatively low applied pressures is where the most significant changes in bulk density occur. At higher applied pressures, subsequent changes in bulk density are smaller since further changes in the particle distribution and orientation are limited.

The change in porosity, or volume, as a function of compression force describes the processes (and mechanisms) of powder compaction. Various models have been used to characterize these changes, with the parameters giving useful information on the general mechanisms of compaction. One of the most common compression models used in the pharmaceutical field is the Heckel model (Paronen and Ilkka 1996), given as:

$$\ln\left[\frac{1}{1-D}\right] = k_1 P + k_o \tag{7.5}$$

where D is the relative density (ρ_b/ρ_s), P is the applied pressure during compaction, and k_o and k_1 are empirical constants. The nature of the relationship between the inverse of the porosity (1-D) and applied pressure gives some indication of the mechanisms at work during compaction. Figure 7.9 shows a schematic Heckel plot, with the y-axis representing the Heckel parameter and the x-axis indicating compaction pressure. As compaction pressure increases, the Heckel parameter increases, eventually reaching a plateau at higher compaction pressures. The linear portion of the curve indicates where the Heckel model (Equation 7.5) appropriately fits compaction data.

The inverse slope of the Heckel plot, $1/k_1$, is often quoted to represent a mean yield pressure, which can be physically related to the mechanisms of compaction. Materials with a high value of the mean yield pressure (low values of k_1) are typically materials that are easily fragmented. Materials with a low value of mean yield pressure (high values of k_1) compact through either plastic or elastic deformation.

The parameter, k_o, represents the value of the Heckel parameter if the linear portion of the curve is extrapolated to zero compaction pressure. It is associated with an apparent initial relative density, D_a, of the granulation. The difference between D_a and the true initial relative density of the granulation, D_o, is a measure of how much rearrangement of the granulation occurs during the initial stages of compaction.

Numerous parameters can affect the response of a powder to compaction under pressure. Figure 7.10 shows typical Heckel plots for

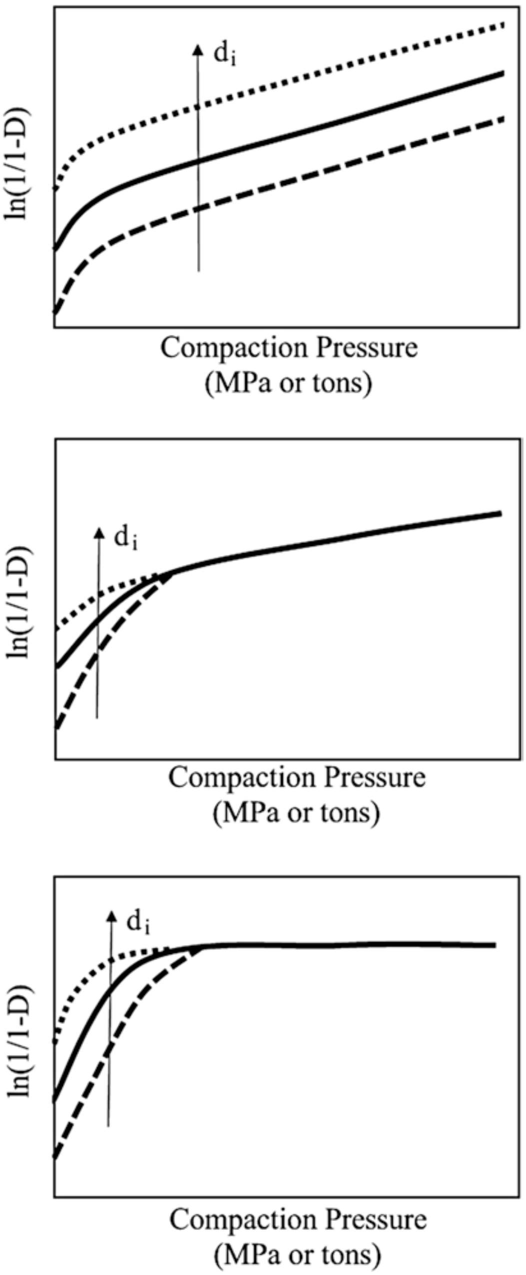


Figure 7.10 Heckel plots (D is relative density) showing different responses for initial particle size, d_i, based on prevalent mechanism of compaction. (**a**) Compaction attributed to particle rearrangement followed by plastic flow. (**b**) Compaction attributed to particle fragmentation. (**c**) Compaction attributed to plastic flow (no fragmentation) (After Paronen and Ilkka 1996)

different particle size fractions according to the dominant mechanism of compaction. In Figure 7.10a, the different particle size fractions started with different bulk density and retained that difference at all compression pressures. This behavior is attributed to compaction first by particle rearrangement followed by plastic flow. This behavior might be observed for amorphous materials that are easily broken and that exhibit plastic flow. In Figure 7.10b, the different particle size fractions again start out with different bulk density, but quickly come together in the same curve with applied pressure. In this case, compaction occurs due primarily to particle fragmentation. Once the particles are broken into fragments, they pack in the same manner regardless of their starting size. Sucrose granulations typically fit in this category. Finally, in Figure 7.10c, the material exhibits a sharp initial increase in bulk density followed by a leveling off where very little additional compaction occurs despite increasing pressure. In this case, different initial particle sizes do not result in different bulk densities in the plateau region. This is attributed to compaction by plastic flow with no particle fragmentation.

7.4.3 Mechanical Properties

These changes in structural attributes during compression lead to concomitant changes in the mechanical properties of the tablet. Of the mechanical properties of tablets, hardness is probably the most important in confectionery tablets since it correlates directly to a sensory attribute – the ability to break the confection in the teeth. Furthermore, an inverse relationship between hardness and dissolution rate typically applies for compressed tablets. That is, tablets made under higher compressive pressure are harder and will be more likely to dissolve slower, releasing flavors at a slower rate.

However, hardness is not an easily measured property. Numerous methods have been developed to characterize the physical attributes relating to how easily a tablet can be crushed. The best measurement method of hardness of a confectionery tablet would be one that simulated the action of breakage in the mouth. However, such simulatory methods tend to be qualitative in nature and do not provide information on the fundamental properties of the materials. Furthermore, it is generally impossible to ascertain anything about fracture mechanisms using these simulatory tests. Yet, the tests of fundamental mechanical properties do not necessarily correlate with sensory perceptions during consumption. This problem is typical of most rheological or mechanical property measurements in the food industry, not just the candy industry. Specifically, is it better to measure empirical parameters that correlate reasonably well with sensory properties or is it better to measure fundamental properties that give mechanistic information about the material but do not correlate very well with sensory measurements? Both types of measurements are probably necessary to allow a complete understanding of quality of food products.

The simplest tests for hardness involve application of a compressive force to the tablet. Either the force required to fracture the tablet can be measured or a penetration depth for a given applied compressive force can be used to characterize hardness. These methods give different values since they measure slightly different aspects of the tablet strength. A more fundamental measurement would be to correlate compressive stress (applied force over a unit area) to the measured strain in the tablet. The ratio of applied stress to measured strain (deformation) is a measure of Young's modulus of the material. A Young's modulus measured in this manner is dependent on the porosity of the tablet and is often extrapolated to zero porosity for comparison purposes.

Another method of tablet characterization is the diametral compression test, where two cone probes (point source) apply a force on opposite sides of the tablet. Fracture stress is usually obtained from this technique. Although this test does not exactly mimic consumers pressing a tablet between their teeth, it is often considered a decent simulation of hardness correlated with sensory measurements.

The strength of a tablet is influenced by many factors, including compaction pressure and particle size, as shown schematically in Figure 7.11. In both cases, for sucrose and lactose, an increase in compaction pressure gives an increase in tablet strength (e.g., hardness or force to fracture). However, in the case of sucrose, no effect of initial particle size was observed, whereas with lactose, tablet strength was higher when initial particle size was smaller. The difference in particle size effects between lactose and sucrose may be related to the mechanisms of compaction, due to the extent of particle fragmentation (number of interparticle binding sites), the binding force of interparticle bonds or some other mechanism. Sucrose is known to undergo significant (complete) particle fragmentation and, as a result, the hardness of sucrose tablets should be independent of particle size (Bolhuis and Chowhain 1996).

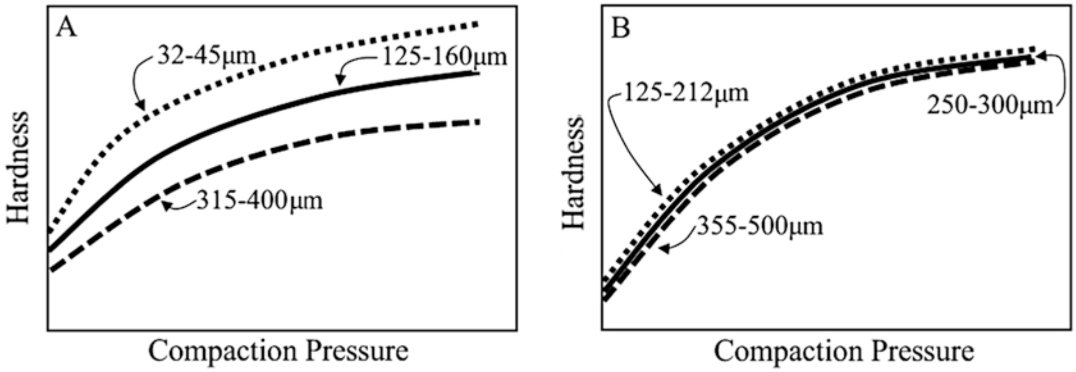


Figure 7.11 Increase in tablet strength with increased compaction pressure and different particle size: (**a**) α lactose monohydrate crystals; (**b**) sucrose crystals (After Alderborn and Wikberg 1996)

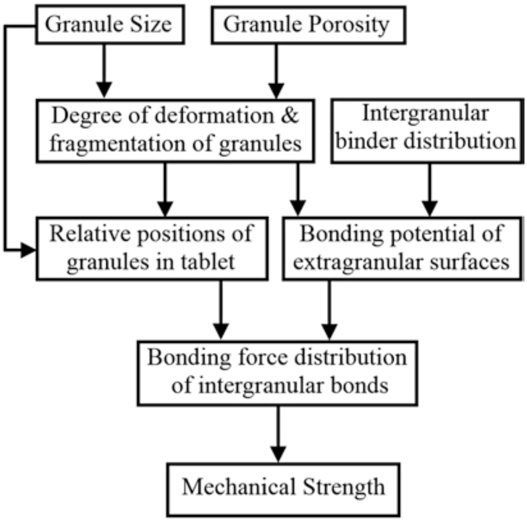


Figure 7.12 Summary of physical and chemical interactions among granules that influence tablet hardness (After Alderborn and Wikberg 1996)

In general, particle size must be within a certain range to produce high-quality tablet candies. The ideal size distribution for granules is 100% through a 20 mesh (841 μm) screen and 100% on a 60 mesh (250 μm). A particle size with 25% on a 100 mesh (149 μm) or 5% on a 325 mesh (44 μm) would be approximate limits. However, almost any size distribution can be handled with a good press.

In summary, Figure 7.12 shows schematically how attributes of the individual granules that make up a tablet influence the mechanical strength of the compacted product. Granule size and porosity, and distribution of the binder affect both physical and chemical bonding forces that hold the tablet together. The magnitude of these binding forces determines the hardness of the product.

7.4.4 Stability

The shelf life of tableted and lozenge candy is generally quite long, in large part due to the nature of the candy itself. Since these candies contain very little water and are primarily crystalline in nature, there are few changes that can occur over time during storage.

Sugar-based tablets and lozenges are resistant to moisture uptake from the air. As long as they are not exposed to water (which can dissolve them), these candies are very stable to moisture-related problems. Sugar-free tablets may be more hygroscopic and absorb moisture from humid air.

Notwithstanding potential moisture sorption issues, the main problems that occur during storage of tableted candies are either rancidity from the lubricant or oxidation of flavors. Proper selection of lubricant and flavors can slow or eliminate rancidity and flavor oxidation so that lozenges and candy tablets can last on the shelf for over a year with little to no change in quality.

7.5 Problems and Trouble Shooting

As with most confections, careful attention to the steps of ingredient preparation and product manufacture result in high-quality, consistent product. However, at times, unacceptable product may be produced due to problems caused either by improper ingredient preparation or problems in the tablet press itself. As a general rule of thumb, if the problem is consistent and repeatable, the tablet press operation is usually the cause of the problem. If the problem is more sporadic or inconsistent, however, the powder preparation or granulation is most likely the place to look. Some specific problems associated with tablet candies include pitting, capping, scoring, and variable tablet properties (thickness, weight, hardness, etc.). Improper environmental conditions (temperature and RH) in the mixing and process areas will also cause problems.

7.5.1 Pitting

Pitting, or sticking, occurs when the tablet sticks to the punch face, usually the upper punch, during tableting. There are several potential causes of this problem, including (1) wet or improperly mixed granulation fed into the tablet press, (2) worn or scratched punch faces, (3) failure of the lubricant to function as an anti-adhesive, or (4) a punch design that is too intricate for the granulation. The granulation tends to stick to designs with too many fine lines and intricate designs, which leads to pitting or sticking. This can be resolved by using a bit more lubricant. If there are wet spots within the powder granulation, the problem can come and go.

7.5.2 Capping

The problem of capping, or laminating, occurs when the tablet splits after it exits the press. Capping may be caused by air entrapment within the granulation, which results in separation of product layers upon ejection from the press. The solution to this problem is primarily with the granulation density. A higher density granulation means there is less air trapped between the particles and less chance of capping. If this is the case, adding some fines to the granulation may resolve the problem. Alternatively, capping may be caused by compression pressures that are too high, which results in loss of bonding by crushing the granulation and splitting upon release from the press. The solution is to reduce the applied pressure until the capping stops.

7.5.3 Scoring

If the lubricant is not functioning properly, there will be scoring on the belly band, or side, of the tablet, accompanied by noise and strain on the press. This problem may be caused by too low a level of lubricant in the formulation or improper addition of the lubricant (order of addition of ingredients is important in tablet making). The lubricant must be added as the last ingredient to ensure complete coating of the base particles and prevent problems during compression. The problem can also be caused by the presence of excessive fines. Alternatively, worn dies give a similar problem of scoring and noisy operation; however, the difference is that a worn die gives a uniform scoring of the tablet whereas problems with the lubricant give random scoring.

7.5.4 Variations in Size or Shape

Variations in tablet properties (hardness or size/shape) may occur due to problems in the press unit or to inconsistency in the granulation fed to the press. Variations in thickness of the tablet may be caused by variations in the length of the upper punch (consistent weight but variable thickness), a variation in the length of the lower punch (thicker tablets weigh more than thinner tablets), or variability in the bulk density of the feed granulation (random variations). Random variations in weight of the individual tablets may be caused by variability in the bulk density of the feed granulation or a problem with the feed frame of the press. Random variations in hardness of the tablets are due either to wearing on the upper compression roller or in the roller bearing itself. Variability in bulk density can also cause this problem.

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