

Resolving tablet defects

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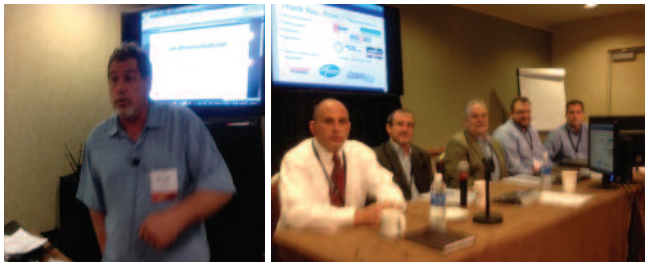
Editor

Tablet defects bedevil many manufacturers. This article outlines some strategies that tablet production experts use to resolve the most common ones, including weight variation, black spots, capping, and sticking and picking.

In May, about 90 people gathered in Cleveland to gain insight on tablet defects from a panel of experts. Led by Mike Tousey of Techceuticals, the panel included Jim Mossop, project manager at Fette Compacting America; Ed Godek, manager of process technology at Glatt Air Techniques; Fabrianno Ferrini, product manager at IMA North America; Fred Murray, president of Korsch America; and Dale Natoli, president of Natoli Engineering.

The most common defects include variations in weight and potency, black spots, capping, and sticking and picking. Others include lamination, mottling, excessive friability, chipping, flashing, and variation in content uniformity.

Accurate weight control is the cornerstone of tablet production, and common causes of weight variation include the condition of the tablet press (settings, worn components) and changes to raw materials (particle size and shape, segregation, moisture). Changes in the environmental temperature and humidity can also have an effect. At the press, weight variation can stem from a number of causes, including improper feeder or compression settings; worn punches, die pockets, or punch guides; and temperature changes during a run.



The symposium, led by Mike Tousey, took place May 21-22 in Cleveland, OH. Panelists included Dale Natoli, Fabriano Ferrini, Jim Mossop, Ed Godek, and Fred Murray.

Tablet weight and the feeder assembly

Weights can vary if the paddle feeder overworks the powder and changes its characteristics, Mossop said. His rule of thumb is to operate the feeder and tail-over-die so that 80 percent of the powder remains in the die, and 20 percent is pushed back. "So with 10-millimeter fill cam, you'd fill 8 millimeters of powder, or something to that effect. If the fill is too deep, you're pushing back too much, and it gets overworked." He added that the condition of the scraper blade and tail-over-die are critical but often overlooked. "I had someone ask me, 'Oh, we need to change them?' These are important components and they are not expensive." He said that he prefers scraper blades with a knife edge over those with a square edge for products that have a tendency to stick to the die table surface.

Blend-sample data don't always correlate to what happens on the press.

Scrapers that wear too quickly may be due to die-table run-out, Natoli said. "The table is not always true, and it's not always a mirror finish. You need to check those horizontal surfaces and identify the high spot. Set the feeder scraper to that." In most cases, the clearance should be 0.002 inch, he said, and the scraper's springs should apply light, even pressure to the die table. Murray said a 0.002- or 0.003-inch clearance is a good place to start. "But if the product is prone to extrude under the feeder, it may have to be higher." He also recommended verifying that the dies are set to the proper depth. "High dies can destroy the scraper in the first 5 minutes of the batch." Conversely, dies set too low or a scraper set too high will allow powder to bypass the scraper and cause other problems, including weight variation, he said.

Tablet weight: The link to potency and blend characteristics

Tousey asked participants whether tablet weight and tablet potency were intertwined, because in a uniform blend or granulation, any under- or over-weight tablets would be inherently sub- and super-potent. Yet only a few participants linked the two issues. One was an attendee whose company makes dietary supplements. "We have to go with weight to meet label claim," she said. "We don't test all those [ingredients] for content uniformity, but we do test weights so we can say that we meet it."

That led to a discussion of blending and how segregation affects content uniformity and/or potency. "You could have a blend meet spec at plus or minus 0.5 percent and not get the required potency," said Godek, who often works with fluid bed processors. "A lot of APIs and granulations are not under control, and many fines are super-potent. It comes down to how the process is run. Fluid beds are also good air classifiers if you're not careful."

Another participant agreed that data gathered from the blend don't always correlate to what happens on the press. He described taking samples from a blending bin that failed uniformity testing, but ran "100 percent good" when put on the press. To save the batch, the company demonstrated to the FDA its satisfactory performance on a single-station hydraulic press. "They bought into it," he said.

In truth, Tousey said, there is little tablet press operators can do to change a blend's characteristics, be it segregation, excessive fines, moisture content, or some other property. "You do what you can to save the batch" by adjusting the press. "There's no such thing as a perfect blend. Think of it like shuffling cards." He also said blends can perform differently depending on how long they've sat.

Mossop predicted that tablet press operators won't have to worry about tablet weights thanks to better technology. "Weight adjustment will go by the wayside in the years to come. You'll be able to detect API and make changes on the fly. It's a few years out but it's going to come."

Tablet weight and tooling

Natoli said accurate weights require that the punches, especially the lower punches, are the correct working length. He defined that as the distance from the lowest point of the punch-face cup to the head flat and said it should be within 0.001 inch of specification. Thus, within a set of punches, the difference between the working lengths of the longest and shortest punches should be no more than 0.002 inch. He also recommended separating the upper and lower punches and verifying the working length of new punches added to an old set. As long as it's within tolerance, "a new tool will be fine 95 percent of the time," he said.

For tablet diameters of 5 millimeters or less, Natoli recommended generating a tool-matching report and pairing the longest upper punch with the shortest lower punch. "That's the best scenario for hardness, thickness, and weight control." The dies, he said, don't contribute to weight variation.

Murray also addressed the importance of the punch working length. "You have to understand how the punch working length can impact the press force control system, which provides automatic tablet weight control. Poor tool tolerances will affect the measured compression forces and cause the control system to react to tool length variation instead of actual tablet weight variation."

Black spots

The term black spots (photo, page 14) covers many different defects in tablet appearance. It could be a smudge not a spot, gray not black, or within the tablet not on the surface. A better term, one attendee suggested, is dark visual defects (DVDs), and people used both terms. The causes of DVDs are legion, Tousey said, but if they appear on the tablet surface, they likely came from the press, not the formulation. Indeed, the design and condition of the tooling and press dominated the discussion of DVDs.

Natoli said it's normal for powders, over time, to abrade the tooling and other areas they contact, but only rarely—usually because of a poor choice of steel—is the tooling itself the source of DVDs. More often, black spots result when abrasive wear expands the clearance between the punch and die, entrapping particles. Those particles accumulate on the die wall and turn dark from the heat of friction. At some point, the punch scrapes off a portion of the burnt material and it's deposited on the tablet.

Aggressive cleaning and over-polishing the tooling—by drag finisher or by hand—can produce a similar result, Natoli said. "You can round off corners, the lower relief, and the lower punch tip itself," he said, which allows particles to wedge between the punch and die and creates black spots. When black spots appear, check the condition of the punches and dies, he said.

To prevent heat-induced DVDs, some companies install vortex coolers under the die table, Mossop said. Cooling the entire room is another option, but success with that approach depends on the operators following proper procedure. "It's only good when the doors are closed and you're not stopping and starting the machine," he said.

Worn punch-guide seals are another source of black spots, and testing them for wear is easy, Tousey said. "If the punch falls out under its own weight, the punch seal is worn out. It should hold the punch by itself and act as a squeegee during operation." While the seals can be cleaned, take care, Natoli said. "The brushes for punch guides are not healthy for cleaning punch seals. I wouldn't use them." Mossop agreed that the seals deserve attention. "Some companies replace them at every cleaning, and others have told me they never knew they were there after 5 years" of operating the press. "For chewable tablets especially, you should replace them with new ones often," he said. Tousey said high ejection force may also darken some tablets, particularly at the tablet band. He added that mottling stems from the blend or formulation more often than it does the press.

Operating the tablet press without punch-guide seals guarantees black spots on tablets.

Other sources of DVDs are the take-off blade, felt oilers, poor environmental conditions in the press area, and over- or under-lubrication. Upstream from the press, high-shear equipment is more likely to cause DVDs than fluid beds, Godek said. Downstream from the press, coating pans can leave gray streaks or scuffs. "Even after cleaning with isopropyl alcohol, they can still be there," he said. One participant suggested pre-heating the pan or coating its surface to resolve the problem.

Capping

Capping is a defect in which the top of the tablet—the area above the band—cleaves (photo). It can be a stealthy defect, undetected until hardnesss and friability testing or until entering the pre-heat cycle of the coating process. Several sources of the defect were proposed. Ferrini of IMA said it can arise when tablets are over-compressed and entrap air. "You can create a tough area in the tablet that doesn't allow air out," he said. "Then, when heat is applied—in the coating pan for example—the air expands and the tablet caps. It gives a loud popping sound." Since most air is expelled at pre-compression, reducing pre-compression force and allowing more air to escape can mitigate capping, Tousey said.

A capped tablet. Capping can occur when entrapped air is released from the tablet or as a result of the formulation's elastic properties.

The characteristics of the formulation and how the tablet releases pressure when it exits the die can also be factors. "It caps at the top because it relaxes at the top, but not at bottom," Natoli said, and Murray agreed but also offered other possibilities. "The top is not constrained by the die wall as it ejects, so it's more likely to occur there. It also has to do with the elasticity of the formulation. With elastic materials, it could be that force is applied too quickly," he said. "Try slowing the machine to decrease the rate of force application to see if that alleviates the problem."

Tousey asked the panelists whether a domed punch head helped prevent capping better than an angled punch head (Figure 1). Natoli said he generally preferred a domed head in most cases and that it might help with capping. That's because the pressure roll contacts the domed head "lower, further way from the head flat, so it allows more venting before entering full compression. A domed head reduces the shock, so there is less energy to relieve at ejection." Tousey said that on a press in which half the stations

FIGURE 1

Using punches with domed heads instead of angled heads may help reduce capping.

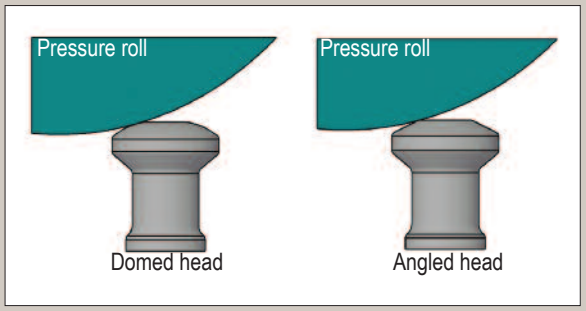
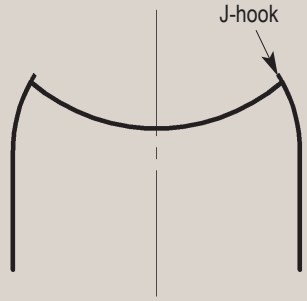


FIGURE 2

A j-hook can form at the punch tip when the land wears off, exacerbating capping and other defects.



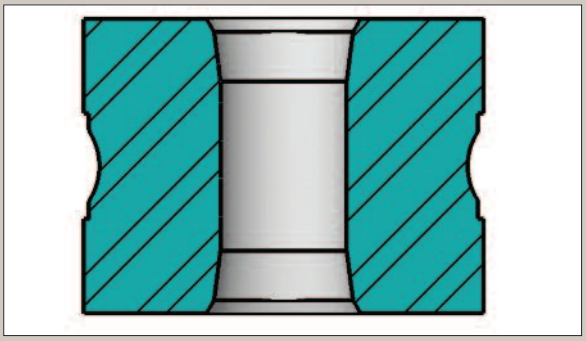
had domed heads and half had angled heads, the tablets made with the domed heads capped less.

In addition to checking weight, thickness, and hardness when capping occurs, the panelists recommended verifying or adjusting punch penetration. Natoli said to check for "j-hooks," a distinctive wear pattern, on the lower punch tip (Figure 2).

Lamination—or delamination, as some preferred to call it—resembles capping but calls for other remedies, such as reducing pressure and increasing press speed. Adjusting pre-compression, dwell time, and feeder speed were other ideas. One attendee said lamination is almost always due to over-compression, while Tousey said that it can result from too much or not enough pressure, adding that it can occur when settings stray from a very narrow range, thus making it difficult to control. Natoli said that punches play little role in lamination, but that tablet configuration can contribute to it, as can dwell time and press speed. "Avoid a deep cup," he said. "The deeper the cup, the more it contributes to lamination. A tapered die might help." (See Figure 3.) The size of the punch—B versus D—will affect how much compressive force can be applied and could also be implicated in lamination, he said.

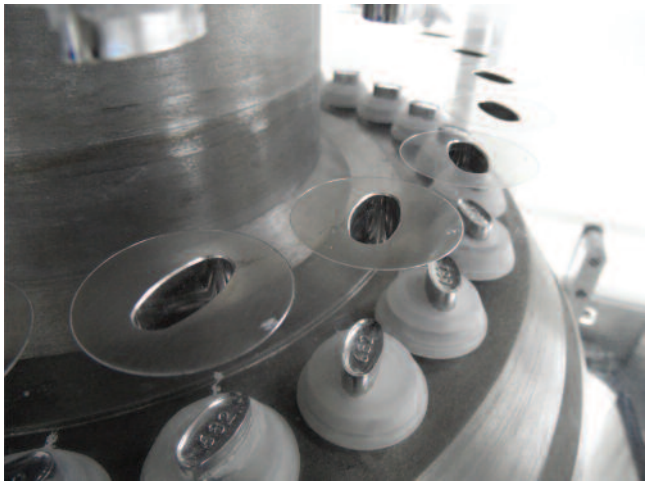
FIGURE 3

Typical tapered die



Sticking and picking

The root causes of sticking and picking—when the formulation sticks to the punch face and/or the punch face picks material from the tablet surface—is difficult to pin down. It often occurs at startup, perhaps because the equipment is "too clean," Tousey said. "The magnesium stearate is not dispersed evenly. It's like a cast iron pan: It needs seasoning so the product doesn't stick." He said operators sometimes scatter a handful of magnesium stearate on the press at startup to avoid these defects, a practice he doesn't endorse and one that he said would make QA people "shudder."



Embossed numbers on the punch face can pick the formulation from the tablet.

Tousey said many operators make the mistake of removing the punches and cleaning or polishing them. "If it sticks to punches, it likely also sticks to other surfaces, like the die table," Tousey said. "Plus, if you have to stop and polish, then you don't have an optimized process." In cases where removing the punches solves the problem, Natoli said, the temperature of the punches could be an issue, since they get warm during a run and cool when removed.

Most sticking occurs at the top of the tablet, and that's an easier issue to resolve, Tousey said. "When I troubleshoot sticking on the upper punch, I check weight and thickness. Next, I check the dwell time and air release. But when it sticks on the bottom, it's harder to eliminate. There's not much to do on the machine except adjust the dwell time." He said the source of sticking and picking at the bottom of the tablet and on the lower punches is more likely a formulation or tooling issue.

Natoli said that when sticking appears on the lower punch after about an hour of runtime, the issue could be, again, related to temperature, which can be checked using a non-contacting infrared thermometer. It's also

possible that recirculation of the powder has changed its characteristics and caused the problem.

"We don't know enough about picking and we need scientific research," Natoli said. "Coating the punch may work, but that's less than 50 percent effective. It's a variable that no one really understands, and working with the tool design and the steel type is a better approach." He said a punch made from steel with a high chromium content releases the tablet better, and Tousey said he considers type S7 steel (3 percent chromium) the "go-to" steel. Other steels contain as much 18 or 19 percent chromium, but Natoli cautioned that high chromium content reduces the amount of compression force the punch can handle.

Mossop agreed that coating the punch faces has limited success and that changing the type of steel is a better option when the formula itself cannot be changed. He recommended conducting a design of experiment in which tools made of several types of steel are installed on the tablet press to see which performs best.

Tablet design is also known to affect sticking and picking, Mossop said. "If you know that, then you should address it during tooling design." That includes selecting letters, numbers, and logos for the punch face that minimize enclosed or semi-enclosed areas, known as islands and peninsulas. Islands appear, for example in "B" and "8," and peninsulas appear in "H" and "2." Natoli said that tapering the embossing or making it shallower often decreases sticking and picking. *T&C*

For further reading

Find more information on the operation and troubleshooting of tablet presses and tablet press tooling in articles listed under "Tablet presses" and "Tooling" in *Tablets & Capsules'* article index in the November 2012 issue or at T&C's website, www.tabletscapsules.com.