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Evaluation and Formulation of Detergent Tablets

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Abstract

Tests were developed to evaluate the following tableting characteristics of detergent tablets: compressive strength, impact strength, and break-up rate. The reproducibility of these tests was determined. Criteria and requirements for satisfactory commercial tablets were established.

The three tests were applied to evaluate the following variables: manufacturing conditions, formulations, and tablet shapes. The tests were sufficiently sensitive to determine the effects of these variables quantitatively. Details on the test methods and examples of their use in determining the effects of some of these variables are given.

Introduction

DETERGENT TABLETS are now in national distribution, and their number and popularity are increasing. Despite the growth of this product form, and the accompanying research and development activities, nothing has been published on the engineering, formulation, or methods of testing the physical properties of these tablets. The purpose of this paper is to describe several techniques which were devised to measure physical parameters of detergent tablets. Methods required to evaluate performance characteristics of the tablets, such as foaming and soil removal, are the same as those used for other forms of detergents, and will not be considered. These new tests were designed to help establish tableting characteristics and determine whether a detergent tablet will be strong enough to resist shocks encountered in normal handling and shipping, yet will disintegrate rapidly in use when added to water.

Experimental

Preparation of Tablets

Equipment. Carver Press equipped with 0–10,000 psi gauge. The die diameter is 1⅞ in. Two punches, one ½ in. deep and the other 6 in. deep are used to form tablet. Both edges are machined to a 45 degree bevel, ⅛ in. in width.

Preparation. Punches are lubricated with calcium stearate and the powdered detergent is compressed using the Carver Press. The pressed tablet is released by forcing the bottom punch from the die with a steel rod.

Compressive Strength Test

Equipment. Carver Press equipped with 0–600 psi gauge. A steel rod 6 in. long, ½ in. in diameter is used to crush the tablet.

Procedure. The steel rod is placed vertically in the center of the tablet which is resting on the middle of the press platform. The press is pumped gently so that the vertical rod presses against the tablet and the top of the press. Pumping is continued cautiously until the tablet ruptures. At the rupture point the pressure remains constant or starts to go lower. Results are recorded in pounds per square inch (psi); the average of three determinations is used.

Impact Strength Test

Equipment. Ring stand and flat metal plate; 36 in. ruler.

Procedure. The tablet is placed on the edge of the plate on the ring stand at a measured height. The tablet is gently rolled off the plate so that it falls on its edge. The test is repeated at 1 in. increments until tablet breaks or cracks. The height in inches, i.e., 1 in. below the height at which it breaks is a measure of the impact strength. Test is started at between 12–16 in., and it is attempted to bracket the breaking height to get a rough estimate of the endpoint at the beginning of the test. No tablet is used more than three times; if the endpoint is not found after three attempts, a new tablet is used. Results are reported as the average of three determinations.

Break-up Test

Equipment. Tergotometer, U.S. Testing Co., stop watch.

Procedure. 1000 ml of tap water at 120F is added to the metal beaker. Tablet is added to water, and the machine (100 rpm) and timing are started simultaneously. Observations are made visually for break-up into small particles, about the size of normal spray-dried detergents. Time required for this amount of break-up is recorded to the nearest ½ min.

If suds prevent observation, the Tergotometer is stopped at ½ min intervals and the solution poured over a 20-mesh screen and amount of break-up is observed. If large particles of the tablet remain, these are returned to beaker with more water and the test is continued. Results are reported as the average of three determinations, rounded to the nearest ½ min.

Results and Discussion

A series of tests has been devised to give precise information on the physical characteristics of detergent tablets. Detergents in tablet form must have

TABLE I

Performance Characteristics of Commercial Detergent Tablets

	A	B	C	D
Impact Strength—Inches	30	18	18	18
Compressive Strength—psi	80	90	90	90
Break-up—Seconds	60	60	120	120

[1] Presented at the AOCS meeting, Toronto, Canada, 1962.

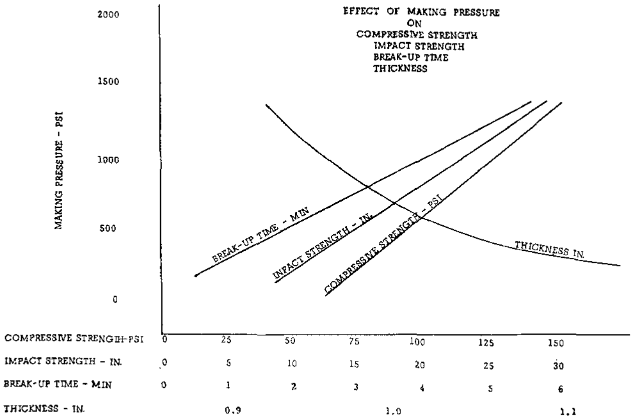

FIG. 1. Effect of making pressure on strength and break-up characteristics of detergent tablets. Also shown is relationship between making pressure and tablet thickness.

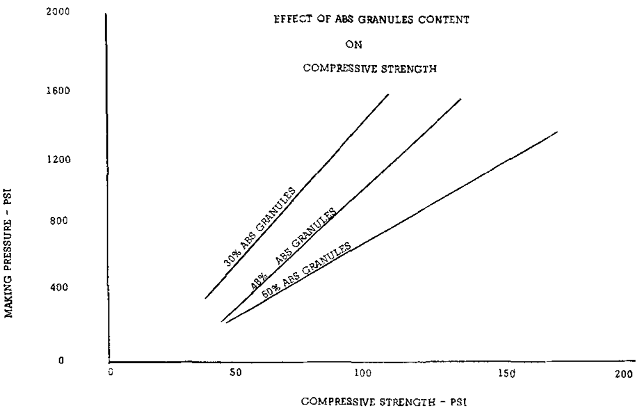

FIG. 2. Effect of ABS granules content on compressive strength of detergent tablets.

solubility characteristics similar to spray-dried detergents in order to be effective in the limited wash cycle time of the modern washing machine. These solubility requirements limit the possibility of making strong, hard, relatively impermeable tablets. Thus, a detergent tablet must be strong enough to survive handling and shipping, but not so hard that it will not disintegrate rapidly in water. New laboratory testing techniques are required to evaluate this combination of strength and weakness in a single product type.

Three tests were developed for this purpose: 1) Compressive Strength Test; 2) Impact Strength Test; and 3) Break-up Test.

The first two, the Compressive Strength Test and the Impact Strength Test, are measures of a product's ability to endure handling, while the Break-up Test defines the disintegration time in water.

Compressive Strength Test. The Compressive Strength Test was designed to measure the resistance of a detergent to "crushing" forces. Such forces could be applied in normal storage and shipping by piling boxes of tablets on top of each other. If a tablet tends to disintegrate readily under a superimposed load, it is unsatisfactory. In the Comprehensive Strength Test the force applied to the tablet by the ½ in. ram of the hydraulic press simulates a cumulative increase in compressive forces, finally resulting in tablet rupture. Higher terminal pressures indicate greater compression resistance.

Results of 24 individual Compressive Strength Determinations ranged from 50–62 psi, having a standard deviation of 3.95 psi. Reproducibility of ± 4.7 psi (95% Confidence Limit) was found for the average of triplicate determinations.

Impact Strength Test. Detergent tablets will be manipulated both individually and as packages during use. If either the individual tablet or a conglomeration of tablets in a package is dropped, there is a possibility of fracture across the face of the tablet. This is particularly true if the tablet is dropped on edge. Resistance to this type of mechanical shock is defined by the Impact Strength Test. In this test, the height of fall before fracture is a measure of impact strength.

Twelve individual Impact Strength Determinations on identical tablets were made. They ranged from 26–28 in., with a standard deviation of 0.6 in. Reproducibility of ± 0.8 in. (95% Confidence Limit) was found for the average of three determinations.

Break-up Test. The purpose of detergent tablets is to deliver a standard amount of detergent into solution during a washing cycle in order to accomplish a cleaning objective. To do so the tablet must disintegrate quickly in water under mild agitation into particles small enough to dissolve rapidly. The Break-up Test employs a Tergotometer to simulate washing machine agitation under controlled conditions. Rapid disintegration in the Break-up Test is a desirable tablet characteristic.

The reproducibility of this test was also investigated. Twelve determinations were run. Whereas the test is normally carried out by examining tablets at ½ min intervals, continuous hand contact with the tablets was maintained during the reproducibility experiments. This was done to obtain more precise Break-up Test endpoints than are usually required. Reproducibility was found to be ± 10 sec (95% Confidence Limit, triplicate determinations). Range was from 45–70 sec, with a standard deviation of 8.0 sec.

TABLE II

Formula of Test Detergent

	%
Sodium alkyl benzene sulfonate (granules)[1]	48
Stearic acid	2
Sodium tripolyphosphate	39
Sodium metasilicate	10
Sodium carboxymethyl cellulose	1

[1] Granules are spray-dried beads containing 35% sodium dodecylbenzene sulfonate and 65% sodium sulfate.

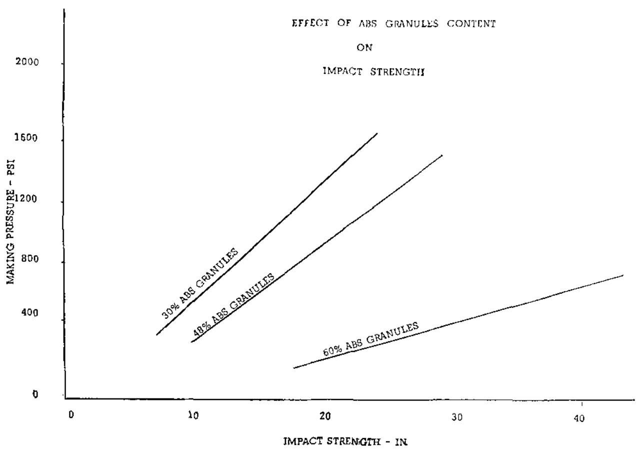

FIG. 3. Effect of ABS granules content on tablet impact strength.

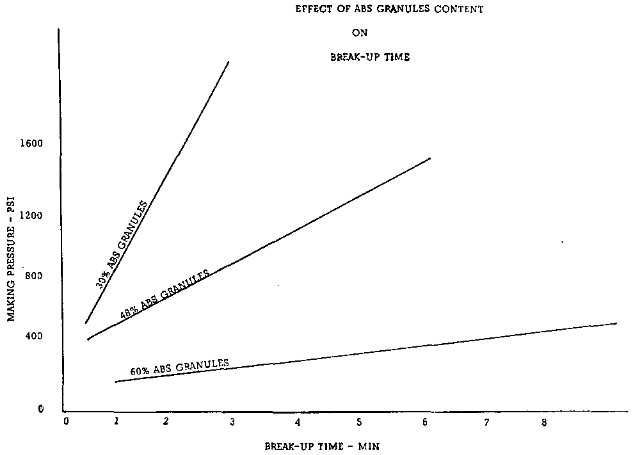

FIG. 4. Effect of ABS granules content on break-up time of detergent tablets.

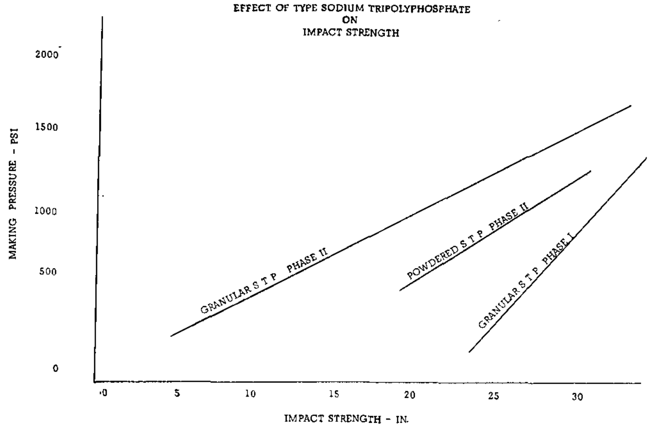

FIG. 6. Effect of form of sodium tripolyphosphate on tablet impact strength.

Application of Test Methods

The potential uses of these three tablet testing methods are extremely varied. Nearly every aspect of tablet development and use can be evaluated by these simple methods. The most important applications are: 1) Examination of Commercial Tableted Products; 2) Evaluation of Manufacturing Variables; 3) Appraisal of Formulation Changes; and 4) Determination of Optimum Tablet Shape. Some typical applications of the three tests will be demonstrated.

Evaluation of Commercial Detergent Tablets

Four commercial detergent products in tablet form were tested by the three methods. The results are summarized in Table I. With the exception of Tablet "A" which had greater impact strength, all behaved very similarly. No tablet required more than 2 min to disintegrate in the Break-up Test, and all demonstrated compressive strength of 80–90 lb. The fracture resistance of three of the products was 18 in. as measured by the Impact Test. These results provide benchmarks for experimental work on tablet formulations.

Evaluation of Manufacturing Variables

The most important possible variation in tablet manufacture is the pressure applied during tablet formation. In Figure 1 the effect of making pressure on the tablet characteristics measured by the Impact, Compressive Strength, and Break-up Tests are shown. The type formula used is shown in Table II. Impact strength increased from 10 in. at 250 psi to 25 in. at 1500 psi in nearly a straight-line fashion. This indicates that higher tableting pressure gives a more impact-resistant tablet. Similarly compressive strength increased from 60–130 psi over the same range of manufacturing pressures. Break-up time also increased with higher making pressures. Thus at 500 psi the break-up time was found to be an acceptable 2 min, while higher making pressure led to excessively high break-up times. Applying the criteria of satisfactory tablet performance obtained by testing commercial tablets, it can be seen that the test composition makes a poor tablet. When sufficiently high making pressure was applied to give a strong tablet, the break-up time was much too long. Conversely, when a low making pressure was employed to assure fast break-up, the tablet failed the strength tests. Another measure of applied pressure is tablet thickness, and thickness vs. making pressure results are also shown in Figure 1.

Appraisal of Formulation Variables

Two changes in the basic test formula were studied by these tests for their effect on tablet characteristics. Those variables tested were a) Variations in the amount of sodium alkylbenzene sulfonate (ABS); and b) The effect of crystal type of sodium tripolyphosphate (Phase I vs. Phase II).

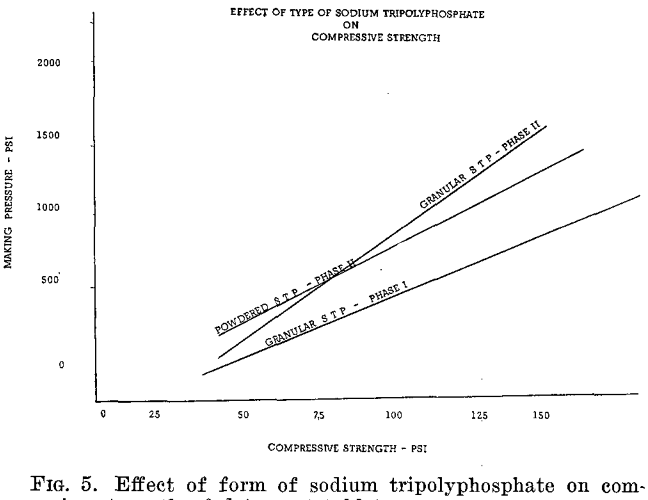

FIG. 5. Effect of form of sodium tripolyphosphate on compressive strength of detergent tablets.

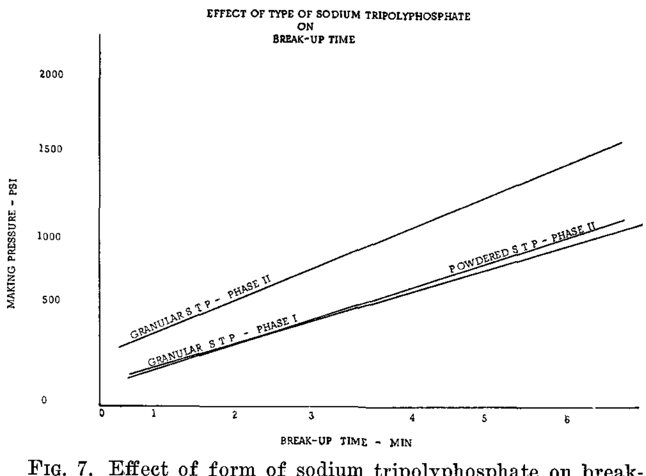

FIG. 7. Effect of form of sodium tripolyphosphate on break-up time of detergent tablets.

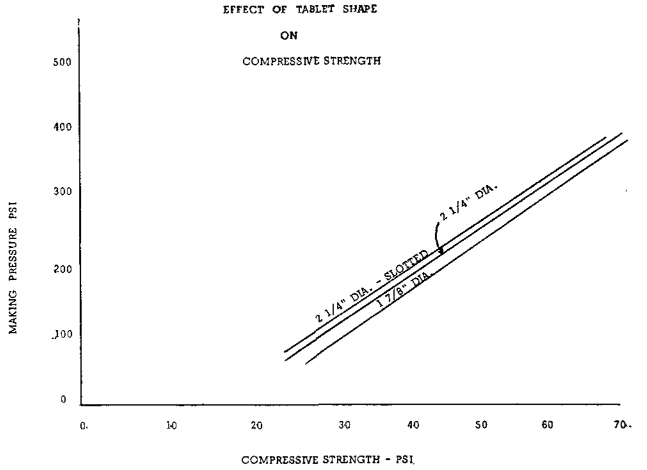

FIG. 8. Effect of tablet diameter and design on its compressive strength.

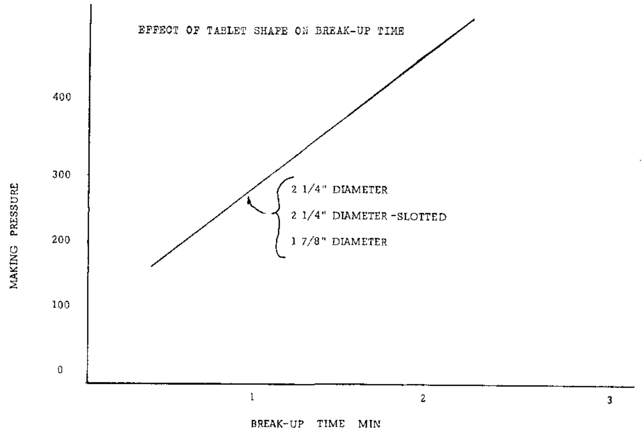

FIG. 10. Effect of tablet diameter and design on its break-up time.

Since the alkylbenzene sulfonate granules act as the tablet binder, changes in the amount of this ingredient would be expected to exert a major effect on tablet properties. In Figure 2, the effect of increasing amounts of ABS granules (35% Active) on compressive strength at several making pressures is shown. At any given pressure, the higher ABS formula has a higher compressive strength. In the test formulas used, ABS granules replace sodium tripolyphosphate to obtain the percentage shown. In Figure 3, a similar trend is shown by the Impact Strength Test. Higher ABS content results in a stronger tablet. An even more exaggerated effect on break-up time is shown by increased ABS percentages. It can be seen from Figure 4 that a tablet containing 30% ABS granules pressed at 500 psi breaks up in 30 sec and a 48% ABS formula in 1 min. When the ABS level was increased to 60%, the tablet made at 500 psi required more than 8 min to disintegrate. Thus a level below 60% ABS granules must be used to assure satisfactory break-up.

The type of sodium tripolyphosphate (STP) used also has an appreciable effect on tablet characteristics. The three types evaluated in this study with the test formula (Table II) were: a) Granular sodium tripolyphosphate. Phase 1, 95% thru 30 mesh, 30% thru 100 mesh; b) Powdered sodium tripolyphosphate, Phase II, 90% thru 100 mesh; and c) Granular, sodium tripolyphosphate, Phase II, 100% thru 20 mesh, 25% thru 100 mesh.

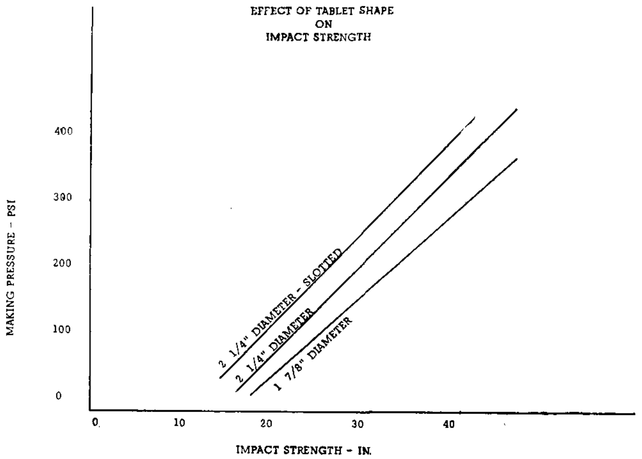

FIG. 9. Effect of tablet diameter and design on its impact strength.

Figure 5 shows that the Phase I sodium tripolyphosphate gave the greatest compressive strength at a given making pressure. It also gave the greatest impact strength (Fig. 6). Both the powdered Phase II material and the granular Phase I STP give longer break-up times at a given making pressure. However, the Phase I sodium tripolyphosphate gave greater strength at lower making pressure. Tablets made at the lower pressure from the Phase I STP had sufficient strength and gave good break-up performance. For instance, at a making pressure of 300 psi, the formula made with the Phase I STP had a break-up time under 2 min, a compressive strength of 80 psi, and an impact strength of 25 in., placing it in the tablet characteristic range of the commercial tablets examined. It is the most suitable of all of the sodium tripolyphosphate types for use in the test formula (Table II).

Determination of Optimum Tablet Diameter

For a given tablet weight, there is a limit to the number of reasonable shapes which are possible. The properties of three of these selected shapes for a two-ounce tablet of the test formula were measured. In Figure 8, the compressive strength of a $2\frac{1}{4}$ in. diameter slotted and unslotted, and a $1\frac{7}{8}$ in. diameter tablet are shown. Although differences are small, the narrower $1\frac{7}{8}$ in. tablet is stronger than the two wider tablets at a given making pressure. Also the slot weakens the tablet somewhat. In Figure 9, the differences in impact strength of tablets of these three designs are shown. Larger changes in stability with corresponding changes in dimension are observed using the Impact Test. For instance, at a making pressure of 200 psi, the slotted $2\frac{1}{4}$ in. tablet has an impact strength of 27 in., while the unslotted tablet of the same size has a 30 in. strength, and the smaller $1\frac{7}{8}$ in. unslotted tablet resists drops up to 35 in.

In Figure 10, the break-up time of all three shapes is shown to be equal. This indicates that a limited variation in tablet shape is not detected by this test. The conclusion on the basis of the three tests is that the $1\frac{7}{8}$ in. tablet is the optimum diameter tested as far as strength is concerned. The final choice of tablet diameter may be dictated by esthetic considerations, not measurable by physical tests.

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