METHOD OF MAKING A COMPRESSED TABLET

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METHOD OF MAKING A COMPRESSED TABLET

CONSIDERATION OF TABLET MATERIALS

- Carrier material
- Non-lubricated particulate product material
- Tablet die lubricant
- Lubricated particulate carrier material

PRODUCTION CYCLE

1. Granulate carrier material
2. Introduce into tablet die
3. Compress into "lubricating tablet"
4. Begin die cycle
5. Eject "lubricating tablet"
6. Compress into "production tablet"
7. Eject "production tablet"
8. Tablet die operates in cycle of alternating "lubricating tablet" and "production tablet"
9. Break up
10. Sell
11. Discard
12. Introduce into lubricated tablet die
13. Compress into "production tablet"
14. Eject "production tablet"
15. Ship, store or convey without danger of breakage

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METHOD OF MAKING A COMPRESSED TABLET

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This invention relates to an improved process for making compressed tablets. In fact, particularly, this invention involves a new process of forming firm, smooth, and otherwise pharmaceutically elegant compressed tablets from particulate tableting compositions devoid of a tablet-die lubricant. This invention further relates to the products made by this new process.

This application is a continuation-in-part of our application Serial No. 842,580, filed September 28, 1959, now abandoned.

In the practice of making compressed tablets for pharmaceutical and other uses, the conventional procedure is to mix the various ingredients and lubricating agents, then lubricate these granules with a tablet-die lubricant, and compress the lubricated granules into tablet form. For many purposes, this procedure is very satisfactory and enables tablets of good quality to be produced quickly and cheaply from the raw materials employed. However, there are many other occasions when the presence of a tablet-die lubricant is deleterious to the finished product. The food-canning industry, for instance, often dispenses flavoring material in tablet form, so that a precise amount is added to each container of food. For example, sugar is commonly added in tablet form when canning peas, corn, tomatoes, and many other foods. Sugar is well known as one of the more difficult materials to tablet, and without a die-lubricant tablets of pure sugar or sugar-containing compositions stick to the die, cap, or easily fracture. However, when the tablet containing lubricant dissolves in the canned-food product, the lubricant contained in the tablet is released; sometimes it coalesces into a globule (as when a steak or other fatty material is used), sometimes it reacts with the can lining, and sometimes it even precipitates out and forms a highly objectionable contaminant throughout the can. Many attempts have been made to overcome this substantial problem, but none of them have been completely satisfactory.

Citrated acid is another material which is very difficult to tablet in adequate amounts. When compositions containing 25 percent or more of citric acid are subjected to normal compression pressure, they stick to the die-face. When the pressure is reduced, the compositions fail to form into a tablet, or form one which is extremely soft and crumbly.

This die-lubricant problem presents itself in yet another manner; namely, when a lubricant is admixed with a tableting composition, only the very small portion of the lubricant in contact with the die-cavity performs its intended function. The rest of the lubricant does no lubrication; it is wasted and only present because, heretofore, no one knew how to get the desired lubrication without distributing the lubricant evenly in the tableting composition.

In large-scale tableting operations, much lubricant material is used, involving substantial expense. Therefore, it means could be devised to place the lubricant only at the granule-die interface, thus allowing the lubricant to be concentrated in the process and a considerable saving would be achieved. Furthermore, the resulting tablet would contain less inert material and therefore a greater percentage of active agents.

An important object of this invention, therefore, is to provide a process for making compressed tablets without incorporating a tablet-die lubricant in the composition to be tableted.

A further object of this invention is to provide a process of forming a pharmaceutically elegant compressed tablet from powdered or granular tableting compositions containing no die-lubricant, even though the compositions have a normal tendency to stick in the die-cavity, cap, split, or otherwise result in deformed tablets.

A still further object of this invention is the provision of a means of lubricating the die-cavity in a compressed-tablet-making machine other than by including a die-lubricant in the tableting compositions to be compressed therein.

A further object of this invention is to achieve a major reduction in the amount of die-lubricant used in the manufacture of compressed tablets.

These and other objects have been achieved by applying a conventional tablet-die lubricant directly to the walls of the die-cavity immediately preceding the introduction into the die-cavity of the material to be compressed into a tablet. By this process, a pharmaceutically elegant compressed tablet may be formed from any tableting composition, no matter how difficult to tablet, without previously incorporating a die-lubricant.

This process, in fact, eliminates the presence of practically all the die lubricant in the finished tablet, for no lubricant exists inside the tablet but rather only on its surface, and then in an extremely small amount. This means a new product is provided, at a considerable saving in the amount of lubricant formerly needed.

One of the more valuable advantages in the use of this process is that pharmaceutically elegant tablets, of materials heretofore extremely difficult to compress into tablets, may now be formulated without inclusion of an objectionable amount of die-lubricant. When tablets made by this process are used in flavoring canned goods, no objectionable precipitation or coalescing of the lubricant material occurs, no harmful reacting with the lining of the container takes place, and no other undesirable conditions associated with lubricants arise.

Other objects and advantages of the invention will appear from the following description of some preferred embodiments thereof.

The drawing is a flow-sheet depicting the processes of the invention.

In general, our process comprises applying a minute amount of die-lubricant to all the tablet-contacting surfaces of the die (that is, the die-cavity and the heads of the upper and lower punches), immediately preceding the introduction into this space of the non-lubricated material for forming the tablet. Coating the die, as all these surfaces will be termed henceforth, is accomplished by compressing therein a lubricated carrier material and then ejecting the so-formed "lubricating" tablet. Next, the product composition devoid of lubricant is introduced into this same die and compressed into a "production" tablet. The compression of the lubricated carrier material results in depositing a minute amount of the lubricant on the surfaces of the die. When this lubricating tablet is ejected, enough lubricant is left behind to lubricate the die for compression of the product composition which is devoid of lubricant. This amount of residual lubricant on the die after ejection of the lubricating tablet, but before introduction of the product composition, is sufficient for production of a pharmaceutically elegant tablet from any tableting composition, without danger of capping, splitting, or adhesion thereof to the die surfaces. Although the production tablets made by this process do contain a very slight amount of lubricant, it is only upon their surfaces and, as such, constitutes a most minute proportion of the total tablet.

Our invention is graphically illustrated in the accom-
panying drawing. Starting with the carrier material, which may or may not require granulation, depending on the nature of the carrier (i.e., salt does not need to be granulated), it is first lubricated by admixing with a tablet-die lubricant. This lubricated carrier material is then introduced into a die-cavity of a compressed-tablet-making machine and compressed into a so-called "lubricating tablet." The lubricating tablet is then ejected from the die and may be discarded or broken up (as by crushing or, where the tablet is soft, dropped onto a hard surface) and used again to form another lubricating tablet. If the latter procedure is chosen, it is necessary periodically to replenish within the broken lubricating-tablet material sufficient lubricant to maintain a proper carrier-material-lubricant ratio. Also, if some broken material, or whole lubricating tablets, are lost or spilt, fresh carrier-material may be added to rebuild the supply.

Following ejection of the lubricating tablet, the non-lubricated product material, in particular (i.e., powder, granular, etc.) form, is introduced into the die and compressed into a so-called "production tablet." This production tablet then is ejected from the die, coming out easily without capping or otherwise breaking, to complete one cycle of the machine and place the die in position for another portion of the carrier material. The cycle is therefore "lubricating tablet-then production tablet," and it is repeated for as many tablets as need to be made. This cycle, of course, is the same for each die in the machine, whether it be one or a great number, such as found in the larger rotary-head machines capable of producing thousands of tablets per minute. In every case, the process works to complete satisfaction, providing pharmaceutically elegant tablets from any tabletting composition.

As a carrier-material for the lubricant, any composition may be used which is adaptable for compression into tablet form, and which will not contaminate, in an objectionable manner, the production tablet. For instance, since the canning industry makes use of large amounts of salt in flavorings, and since sodium chloride itself is quite easy to tablet, granular sodium chloride is a satisfactory carrier for the lubricant. If any of the chloride material is carried over by chance into the production tablet, it will not destroy the utility of the tablet, especially if the contamination is very small. A host of other materials, such as potassium chloride, calcium chloride, sodium borate, sugar, vegetable meals, grains, wetting agents, etc., may also be used.

It is possible to combine the manufacture of two different marketable tablets into one cycle by forming the lubricating tablets from materials which are saleable. For instance, where there is a market for tablets containing a lubricant, the above-mentioned sodium chloride lubricating tablet takes the added status of a second "production tablet" while retaining its "lubricating tablet" status. This, of course, can be done with other compositions as well.

Carrier material of a wide range of particle sizes may be utilized in this process, depending on the size of the tablet to be made, the machine, and other factors. Where sodium chloride is used, excellent results are obtained if the "fines" are eliminated. Medium, kiln-dried, granular sodium chloride has been found highly satisfactory. This is a mixture of granules as set forth in the following Table I.

### Table I

**Medium, Kiln-Dried, Granular, Sodium Chloride**

<table>
<thead>
<tr>
<th>Size of Screen</th>
<th>Approximate percent of total tablet-making process</th>
<th>percent of total tablet-making process</th>
</tr>
</thead>
<tbody>
<tr>
<td>6-mesh</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>8-mesh</td>
<td>98</td>
<td>98</td>
</tr>
<tr>
<td>10-mesh</td>
<td>56</td>
<td>56</td>
</tr>
<tr>
<td>14-mesh</td>
<td>15</td>
<td>15</td>
</tr>
<tr>
<td>20-mesh</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>

All meshes U.S. Tyler.

Any conventional tablet-die lubricant may be used in this new process. For instance, white mineral oil (U.S.P.) containing a very small amount of stearic acid is quite satisfactory. Although in some instances it may be more desirable to eliminate all of the stearate, it has been found that the mineral oil-sodium stearate mixture generally gives better results than mineral oil alone. The amount of sodium stearate incorporated in the mineral oil is widely variable, depending upon the composition to be compressed into the production tablet.

One satisfactory lubricating tablet comprises the above-mentioned kiln-dried salt granules lubricated with approximately 2.0 percent, based on the final weight of the lubricating tablet, of a mineral stearate mixture, with the sodium stearate constituting about 0.08 percent of the final weight of the lubricating tablet. This lubricating tablet leaves behind on the die surfaces sufficient lubricant to insure proper lubrication of at least one production tablet, no matter what the composition of the production tablet may be. The amount of mineral oil-sodium stearate retained by these production tablets is negligible. Tests on these production tablets showed no detectable precipitation of this lubricant in various media, and reactions between foods contained in their linings and of lubricated or un lubricated tablet. Satisfactory lubricating tablets may be formed, also, from the aforementioned carrier granules, and such lubricants as stearates (e.g., sodium, calcium, and magnesium) alone, Sterotex (hydrogenated vegetable oil), various vegetable oils, polyethylene glycols, lecithin, hydrated calcium silicate, and many others.

The particle size of the material used in making the production tablets is not critical. Substantially all particle sizes within the realm of conventional tabletting techniques, that is, from the finest powder to the coarsest granule, are operable, and mixtures of different particle sizes also are feasible. Not only may production tablets be made with this process from solid ingredients, but liquid components, such as spice extracts, also may be tableted if admixed with sufficient solid material.

The pressures used in the tabletting operations also are variable, with all of the standard tabletting pressures giving satisfactory results. Rather than having to adjust the pressure to a point intermediate between that producing capping and that producing a soft tablet, this process enables tablets of a wide range of hardness or softness to be produced without objectionable fracturing or other damage.

The invention also is operable with all types of compressed-tablet-making machines. Furthermore, the composition of the dies and punches is not critical, since plain steel for solid tablets containing a lubricant, and punches carry out the method satisfactorily. Carbide dies and punches have given, in general, the best results.

Where the lubricating tablets are not marketable, and their primary use is to impart a thin, film-like coating of lubricant to the die, it is entirely practical to recover them, break them, granulate the broken material if necessary, and use this recovered material to form more lubricating tablets. Naturally, as this is done repeatedly the proportion of lubricant will be diminished and replenishment thereof from time to time is necessary. Once a workable amount of carrier material is established, and properly lubricated, the only significant additional cost is for the slight amount of lubricant needed to maintain the material-lubricant ratio. As is apparent from the minute amount of lubricant retained by the production tablet, this solution passes a very small figure. Of course where economics are suitable, it is entirely feasible to discard the lubricating tablet.

The following examples are set forth to better illustrate the invention, with the understanding that they are not to be construed as a limitation in any manner upon the broad concept disclosed herein.
EXAMPLE 1

Tablets containing 67.3 percent sugar, 30.7 percent salt, and 2.0 percent of a liquid spice extract were prepared following the process of this invention. Lubricating-tablet granules of medium kiln-dried sodium chloride were lubricated with approximately 2.0 percent, based on the final weight of the lubricating tablet, of a composition comprising white mineral oil (U.S.P.) and sodium stearate, the sodium stearate constituting approximately 0.08 percent of the final weight of the lubricating tablet. A conventional rotary-head compressed-tablet making machine, with a plurality of dies arranged to compress and ejection tablets per die per revolution of the head, was used. One granulation hopper was filled with the lubricated salt granules and the other hopper filled with the granulated sugar-salt-spice extract production-tablet composition. The machine was set for 1500 tablets per minute, and a cycle of "lubricating tablet-then production tablet" was established in each die. Over 10,000 production-tablets were made by this procedure, all of which were perfectly formed, whole, and none of which clogged or stuck to the tablet-dies. This is in contrast to prior attempts of tablets the same sugar-salt-spice extract compositions using the techniques of the prior art, which resulted in sticking and capping after three or four revolutions of the die-head.

The production-tablets so formed ultimately were shipped to a canner who used them in canning vegetables, and tests showed no trace of stearate on their precipitates and no evidence of a deleterious reaction with the containers or their linings. The production-tablets were quick to dissolve in the canned medium, to give a pleasant and satisfactory taste without any undesirable foreign mater.

EXAMPLE 2

In response to a request for pure sugar tablets, 100 percent granulated sugar was used to form the production-tablet. The lubricating-tablet composition was the same as that of Example 1. The great difficulty in compressing 100 percent sugar into tablet form is well known, especially when no lubricant is applied to the sugar granules, and it was expected that problems would be encountered. However, a tablet-machine of the same type as that described in Example 1 was set at a rate of 1500 tablets per minute, the production-tablet hopper filled with the 100 percent sugar granules and the lubricating-tablet hopper filled with the lubricated salt granules. The same lubricating-tablet-then production tablet cycle of Example 1 was performed without any tablets capping, splitting, or sticking; and the production-tablets, as well as the lubricating-tablets, were pharmacologically elegant.

EXAMPLE 3

The inventive process also has been carried out on a composition comprising approximately 30.0 percent salt, 65.7 percent sugar, 2.4 percent calcium chloride and 1.9 percent spice extract, in conjunction with the lubricating-tablet mixture of Examples 1 and 2. The same tablet-machine of Examples 1 and 2 was used, the production-tablet hopper filled with the salt-sugar-calcium chloride-spice-extract composition and the lubricating-tablet hopper filled with the lubricated salt granules. Following the "lubricating tablet-then production tablet" cycle of Examples 1 and 2, the machine produced pharmaceutically perfect tablets without jamming, without tablets sticking in the die-cavity, and with no evidence of capping or splitting.

EXAMPLE 4

Production-tablets containing approximately 34.5 percent salt, 64.0 percent sugar, and 1.5 percent granular garlic were made following the process of this invention. The lubricating-tablet composition of Examples 1–3 was used. A double-punch tablet-making machine like that of the preceding examples was employed to form tablets from these compositions on a repeating lubricating tablet-then production tablet cycle. All the tablets so formed were structurally sound, pharmacologically elegant in appearance, with no evidence of imperfection. Furthermore, the machine did not jam or malfunction once during the run.

EXAMPLE 5

A composition consisting essentially of 65.2 percent sugar, 32.8 percent salt, 1.9 percent granular garlic, and 0.1 percent granular Cayenne pepper was compressed into production-tablets with the process of this invention. The lubricating-tablet, the tablet-making machine, and the procedural steps were the same as in the preceding examples. These flavoring tablets were uniformly excellent in structure and appearance, and no difficulties in the operation of the machine were encountered.

EXAMPLE 6

To determine the operability of several lubricating tablet compositions, production tablets were formed from a mixture of 75 percent sugar and 25 percent salt, using the following compositions in Table II for lubricating the die. In each instance, both the lubricating-tablet and the production-tablet were well-formed, did not stick or bind in the machine, and otherwise very satisfactory.

**TABLE II**

<table>
<thead>
<tr>
<th>Composition</th>
<th>Component</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>salt</td>
<td>32.5 lbs</td>
</tr>
<tr>
<td>2</td>
<td>mineral oil</td>
<td>9.4 oz</td>
</tr>
<tr>
<td>3</td>
<td>salt</td>
<td>32.0 lbs</td>
</tr>
<tr>
<td>4</td>
<td>peanut oil</td>
<td>6.1 oz</td>
</tr>
<tr>
<td>5</td>
<td>salt</td>
<td>32.5 lbs</td>
</tr>
<tr>
<td>6</td>
<td>olive oil</td>
<td>5.8 oz</td>
</tr>
<tr>
<td>7</td>
<td>salt</td>
<td>32.5 lbs</td>
</tr>
<tr>
<td>8</td>
<td>salt</td>
<td>32.5 lbs</td>
</tr>
<tr>
<td>9</td>
<td>salt</td>
<td>32.5 lbs</td>
</tr>
<tr>
<td>10</td>
<td>cornstarch wax</td>
<td>1.5 lbs</td>
</tr>
<tr>
<td>11</td>
<td>salt</td>
<td>32.5 lbs</td>
</tr>
<tr>
<td>12</td>
<td>polyethylene glycol (average molecular weight = 4,000)</td>
<td>1.5 lbs</td>
</tr>
<tr>
<td>13</td>
<td>salt</td>
<td>32.5 lbs</td>
</tr>
<tr>
<td>14</td>
<td>mineral oil</td>
<td>0.6 oz</td>
</tr>
<tr>
<td>15</td>
<td>salt</td>
<td>32.5 lbs</td>
</tr>
<tr>
<td>16</td>
<td>salt</td>
<td>32.5 lbs</td>
</tr>
<tr>
<td>17</td>
<td>polyethylene glycol (average molecular weight = 4,000)</td>
<td>2.0 lbs</td>
</tr>
<tr>
<td>18</td>
<td>salt</td>
<td>32.5 lbs</td>
</tr>
<tr>
<td>19</td>
<td>salt</td>
<td>32.5 lbs</td>
</tr>
<tr>
<td>20</td>
<td>salt</td>
<td>32.5 lbs</td>
</tr>
<tr>
<td>21</td>
<td>salt</td>
<td>32.5 lbs</td>
</tr>
</tbody>
</table>

EXAMPLE 7

Using the procedure of the previous examples, production-tablets were formed from 50 percent salt and 50 percent ascorbic acid. The lubrication-tablets were made from a mixture of 32.5 lbs, salt and 0.5 quart mineral oil. No sticking of either lubrication or production tablets was encountered and the machine performed smoothly and freely.

EXAMPLE 8

In an attempt to make tablets of pure citric acid, a composition comprising 32.5 lbs, salt and 2.0 oz. sodium stearate was utilized as the material for lubricating the die. The cyclic procedure of lubricating tablet-then production tablet was followed. The production-tablets
did not stick in the die or otherwise interfere with the operation of the machine, but because of the nature of pure citric acid (hydrous) these tablets were quite soft. The lubricating-tablets were well-fomed and also presented no problem to the operation of the machine.

**EXAMPLE 9**

Tablets of pure sugar have been made by the process of this invention, using a mixture of 52.5 lbs. of sugar and 0.5 qt. of mineral oil as the lubricating-tablet composition. Both the pure sugar and the sugar-mineral oil composition compressed into pharmaceutically elegant tablets without any sticking in the die or malfunctioning of the machine.

The preceding examples illustrate the versatility of the new process herein disclosed. It may be used with great success with various tableting speeds and tablet dies. It lends itself equally well to compositions easy to tablet and those difficult to tablet, and produces excellent results in either case. Because of the absence of sticking and jamming of the machine, wear and tear on the machine is considerably reduced when using this process, and the speed of the machine may be increased appreciably because of this consistently smooth operation.

This process is operable for making any conventional compressed table where it is desired to eliminate all or practically all of the lubricant from the final product. It is not sensitive to any particular tableting composition, so long as the composition is reasonably susceptible to compression into tablet form.

To those skilled in the art to which this invention relates, many changes in construction and widely differing embodiments and applications of the invention will suggest themselves without departing from the spirit and scope of the invention. The disclosures and the description herein are purely illustrative and are not intended to be in any sense limiting.

We claim:

1. A process for manufacturing compressed tablets, comprising: introducing into a tablet-die a first composition comprising a tableting material lubricated with a pharmaceutical tablet-die lubricant; compacting this said first composition into a lubricating tablet; ejecting this said lubricating tablet; introducing into the same tablet-die a second composition comprising a non-lubricating tableting material; compacting this said second composition into a production tablet; ejecting this said production tablet; and repeating the cycle of "lubricating tablet-then production tablet" until a desired amount of tablets is formed.

2. A process as defined in claim 1, wherein the lubricating tablets are formed from a marketable composition.

3. A process as defined in claim 1, wherein the lubricating tablets are broken and the broken material is granulated.

4. A process as defined in claim 3, wherein the desired ratio of lubricant in the lubricating-tablets is maintained by periodic addition of fresh lubricant to the broken material.

5. A process as defined in claim 1, wherein the die-lubricant is selected from the group consisting of mineral oil, vegetable oils, waxes, hydrogenated vegetable oils, alkali metal stearates, alkali earth metal stearates, alkali metal benzoates, and mixtures thereof.

6. A process as defined in claim 1, wherein the die-lubricant is a mixture of white mineral oil and sodium stearate.

7. A process as defined in claim 1, wherein the die-lubricant is sodium stearate.

8. A process as defined in claim 1, wherein the particular material is selected from the group consisting of salt, sugar, wheat, barley, linseed meal, soya bean meal, and ground alfalfa.

9. A process for manufacturing compressed sugar tablets, comprising: introducing into a tablet-die a first composition comprising granular sodium chloride lubricated with a pharmaceutical tablet-die lubricant selected from the group consisting of mineral oil, vegetable oils, waxes, hydrogenated vegetable oils, alkali metal stearates, alkali earth metal stearates, alkali metal benzoates, and mixtures thereof; compacting this said first composition into a lubricating-tablet; ejecting this said lubricating-tablet; introducing into the same tablet-die a second composition comprising non-lubricated sugar particles; compacting this said second composition into a sugar tablet; ejecting this said sugar tablet; and repeating the cycle of "lubricating tablet-then sugar tablet" until a desired amount of tablets are formed.

10. A process as defined in claim 10, wherein the lubricating-tablets are broken and the broken material used to form new lubricating tablets.

11. A process as defined in claim 11, wherein the desired ratio of lubricant in the lubricating-tablets is maintained by periodic addition of fresh lubricant to the broken material.

12. A process as defined in claim 11, wherein the desired ratio of lubricant in the lubricating-tablets is maintained by periodic addition of fresh lubricant to the broken material.

13. The process of claim 10, wherein the lubricating-tablet comprises about 98.0 percent sodium chloride and 2.0 percent lubricant.

14. The process of claim 13, wherein the lubricant comprises white mineral oil (U.S.P.) and sodium stearate.

15. A method of tablet production wherein two different tableting compositions are compressed, alternately, in the same tablet-die, one of the said compositions containing a tablet-die lubricant and the other said composition being devoid of tablet-die lubricant.

**References Cited in the file of this patent**

**UNITED STATES PATENTS**

1,289,570 Stokes  December 31, 1918
1,046,833 Meyer  July 7, 1936
1,444,282 Creve  June 29, 1948
1,733,380 Bowes  Feb. 21, 1956
UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,042,531

Bernardine J. Leal et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 7, line 4, for "well-fomed" read -- well-formed

--; line 39, for "tablet-diet" read -- tablet-die --.

Signed and sealed this 30th day of October 1962.

(Seal)

Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents