METHOD OF PREPARING PHARMACEUTICAL TABLETS

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Int. Cl. B44A 1/16
3,438,797

13 Claims

ABSTRACT OF THE DISCLOSURE

A method of preparing wax-coated pharmaceutical tablets is provided wherein sugar syrup coatings are applied to the tablets and while the coatings are in a tacky state a wax emulsion is applied. The tablets are then dried to obtain an unpolished wax coating and imprinted with conventional shellac-based ink indicia, if desired.

This invention relates to imprinted pharmaceutical tablets and to a method for the preparation thereof.

The importance of imprinting pharmaceutical tablets with a clearly visible identifying marking to prevent spurious imitations and substitutions is well illustrated by the significant volume of business which has been lost by some manufacturers because of such imitations and substitutions.

For purposes of this invention the pharmaceutical tablet usually includes a pharmaceutical tablet core having a cylindrical edge surface and convex opposite end surfaces. The core is covered with successive undercoats, smoothing coats and suitably colored finish coats consisting primarily of grinding powders and coating syrups which form a sugar coating providing the tablet with a smoothly rounded outer surface.

In accordance with one well-known method, the printing of ink indicia on the sugar-coated tablet is accomplished by applying a confectioner's shellac coating over the sugar coating and then imprinting on the shellac coating with an ink having a shellac base. Due to the presence of shellac in both the ink and the coat, the ink adheres tenaciously to the coat to produce an acceptable product. However, the nature of the shellac surface is considered undesirable by many pharmaceutical manufacturers in view of its lack of polish or luster. In addition, because of the tackiness of the shellac coating it tends to stick to the wall of the coating pan during the coating process and to pull the sugar coating off the tablet core. Also, the tacky character of shellac-coated tablets presents a handling problem. For example, such tablets do not readily flow into and do not readily seat properly in accommodating recesses in a tablet printing machine.

In order to avoid the aforementioned difficulties some manufacturers have considered it desirable to apply a wax coating, for example, of carnauba wax, to each tablet, such coating being capable of taking on a high polish. In this manner the desired pharmaceutical elegance is imparted to the tablet and the tablet is also provided with a very desirable free-sliding or flowing character. However, the wax coating has not proved entirely satisfactory in that conventional shellac-based inks will not readily adhere to the polished wax surface and the markings have a tendency to lift off even after drying.

With regard to imprinting of a shellac-based ink on a wax-coated tablet, the closest prior art of which I am aware is that described in U.S. patent application Ser. No. 312,523 to H. W. Bennett, filed Sept. 30, 1963 and now abandoned, copending herewith and assigned to the assignee of the present invention. That patent specification teaches printing with a shellac-based ink on a pharmaceutical tablet having an unpolished outer coating of paraffin wax applied over the final color coat by tumbling the tablets briefly in contact with a solution of paraffin wax dissolved in a volatile petroleum solvent. A disadvantage of the foregoing concept, however, is that the petroleum solvents used in preparing the wax coating solution make it necessary to provide explosion-proof equipment with provision for adequate ventilation and exhaust for carrying out the tablet coating operation.

Accordingly, a primary object of the present invention is to provide a method for imprinting on wax-coated pharmaceutical tablets, which method does not involve the use of hazardous materials and which does not require special equipment.

A more specific object of the present invention is to provide a method for preparing an imprinted pharmaceutical tablet which is simpler and less time consuming than the known methods. For example, in the prior method mentioned above the wax coating is applied to the tablet following application and drying of the final color coat, whereas in the practice of this invention the sugar coat applied prior to the wax coat is preferably not dried beyond a tacky state and the wax emulsion, which may include a coloring material, is applied to the tacky coat. Application of the wax emulsion to the tacky sugar coat forms a good bond between the resulting wax coating and the underlying sugar coating. When the wax emulsion also includes a coloring material the wax coating produced serves as the final color coat.

Broadly, the present invention resides in a method of preparing imprinted tablets which comprises the steps of tumbling sugar-coated tablets in contact with a wax emulsion for a period of time of sufficient duration to cover the tablets with a thin, uniform, unpolished coating of wax, drying the unpolished wax coating, imprinting ink indicia upon a portion of the dry unpolished coating, drying the imprinted indicia, and then applying a protective transparent outer coating of a suitable material over the imprinted wax coating.

The wax emulsions of the present invention are oil-in-water emulsions of waxes such as paraffin wax or carnauba wax, which are obtained by emulsification of the wax in distilled water with a suitable surfactant or a mixture or blend of surfactants. Although the preferred waxes are paraffin wax and carnauba wax, any of the commercially available, pharmaceutically acceptable waxes, such as for example beeswax, may be used in the practice of this invention.

The preferred surfactants are non-ionic in character. It is contemplated, however, that surfactants having anionic, cationic and amphotytic properties may also be used in the practice of this invention. Surfactants which have been found to be particularly useful in preparing the wax emulsions of this invention are a series of commercially available non-ionic surfactants comprising sorbitan fatty acid esters and polyoxyethylene sorbitan fatty acid esters. Among other commercially available non-ionic surfactants which may be used in the practice of this invention are mono and diglycerides of fat-forming fatty acids.

In preparing the wax emulsion the type and amount of the surfactant (emulsifier) used to obtain a satisfactorily stable emulsion is determined according to the Atlas HLB System, as described in the publication "The Atlas HLB System," Atlas Chemical Industries, Inc., 3rd Edition (1963). In the HLB System each emulsifier is assigned a numerical value, which is designated as its HLB. The HLB of an emulsifier is an expression of its hydrophilic-lipophilic balance, that is, the balance of the size and strength of the hydrophilic (water-loving or polar) and the lipophilic (oil-loving or non-polar) groups of the emulsifier. Under the Atlas system it has also been found that oils, waxes and other materials likely to be incor-
porated into emulsions have an individual “Required HLB.” For example, the required HLB of paraffin wax is 10 and that of carnauba wax is 12. In the case of paraffin wax, therefore, this means that any emulsifier, or a blend of emulsifiers, having a combined HLB of 10, will make a more stable fluid oil-in-water paraffin emulsion than emulsifiers of any other HLB value. Likewise, any emulsifier or blend of emulsifiers having a combined HLB of 12 would be best suited for making a stable emulsion of carnauba wax. From long experience in using the HLB system it is generally accepted in the art that the emulsifiers which are best, that is, it gives the most stable emulsion. In the practice of this invention, therefore, the preferred emulsifying agents used in making up the wax emulsions are blends or mixtures of various non-ionic surfactants having the required HLB values.

The color coating solution of this invention comprises a conventional syrup solution, as an aqueous suspending medium, in which is suspended a sufficient amount of an appropriate coloring material to evenly coat the tablets with the desired color. The preferred syrup solution is an aqueous solution of sucrose known in the art as Simple Syrup, USP (85% sucrose in purified water, specific gravity 1.313). Suitable coloring materials which may be used are any of the various non-toxic, water-soluble dyes and water-insoluble lakes which have been certified for food and/or pharmaceutical use. For example, FD & C Red No. 2, Red No. 3, Yellow No. 5 and Yellow No. 6, with Titanium Dioxide, N.F.

In the practice of the present invention successive smoothing and finishing coats, consisting primarily of glossing powders and coating syrups are applied to the tablets in conventional rotary coating pans to give the tablets a smooth outer surface, this procedure being that generally designated in the art as sugar coating. After the last of several coats of syrup, each of which may contain various amounts of color, has been applied to the tablets, the tablets are allowed to tumble to the point where the coating is still slightly moist, or in other words, tacky. At this point the wax emulsion is then applied to the tablets in a conventional manner. Only enough solution to coat all of the tablet is used, and all of the tablets are sufficiently covered with the wax solution, as determined by visual observation, the coating pan is shut down. As previously stated, the wax emulsion may also include a coloring material, in which case the tablet is provided with both a final color coat and an unpolished wax coating in a one-step operation. In the practice of this invention it is preferred to include the coloring material in the wax emulsion in the interest of saving time and thus providing a more economical coating operation.

To attain the desired unpolished coating of wax on the tablets, those skilled in the art will appreciate that the amount of coating solution to be used, the concentration of wax in the coating solution and the time the tablets are allowed to tumble will vary according to such factors as the number of tablets to be coated in a single operation, the size of the tablets, and the like. According to the practice of this invention sugar-coated tablets measuring approximately 0.235 inch thick by 0.360 inch in diameter were wax coated in batches of about 160,000 tablets. For coating a batch of tablets of this size it was found that an operable concentration of wax in the coating solution is from about 0.5 to about 4.6 weight percent. More specifically, in the case of paraffin wax, an operable concentration is from about 0.5 to 4.6 weight percent and preferably, about 1.0 weight percent. For a carnauba wax concentration of wax in the coating solution should be from 0.1 to about 1.0 weight percent, with the preferred concentration being about 0.5 weight percent. As noted hereinabove, the time the tablets are allowed to tumble in the coating solution, that is, the coating time, is not narrowly critical; it will vary according to such factors as the number of tablets to be coated in a single operation, size of the tablets, etc. In the practice of this invention best results are obtained by allowing the tablets to tumble in the coating solution for from about 2 minutes to about 3 minutes.

After application of the wax coating the tablets are removed from the coating pan and placed on drying racks where they are allowed to remain until the wax coating is dry. Once the coating is thoroughly dry the wax-coated tablets may be embossed with the indicia engraved therein or embossed thereon and supplied with an edible ink. For imprinting of the ink indicia on the tablets, it is preferred to use any of the various commercially available shellac-based edible ink compositions.

After the tablets are imprinted they are allowed to dry at room temperature until the ink indicia have set, which usually requires a period of about 8 hours. Once the indicia have set the tablets are given a protective outer coating of a suitable transparent material, such as, for example, carnauba wax, beeswax, or a combination thereof. The application of the protective coating may be carried out in a conventional rotary polishing pan. The indicia being of a different color than the underlying coating color, will be clearly visible through the transparent outer coating which functions to protect the tablet against chipping and flaking under normal handling conditions. Preferably, the outer coating is then polished to impart a pharmaceutically elegant appearance to the tablet.

The following examples are illustrative of formulations of wax coating solutions found to be satisfactory in the practice of this invention. It is understood that these examples are not to be construed in a limitative sense:

**EXAMPLE 1**

<table>
<thead>
<tr>
<th>Grams</th>
<th>Weight percent</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Paraffin wax 1</td>
</tr>
<tr>
<td></td>
<td>Sorbitan monostearate 2</td>
</tr>
<tr>
<td></td>
<td>Polyoxyethylene sorbitan monostearate 3</td>
</tr>
<tr>
<td></td>
<td>Distilled water</td>
</tr>
<tr>
<td>Total</td>
<td>100.000</td>
</tr>
</tbody>
</table>

1. PABOWAX (Standard Oil Company, Indiana).
2. SPAN 60 (Atlas Chemical Industries).
3. TWEEEN 60 (Colidex).

The distilled water is charged to a beaker and heated to about 130°F on a steam heated water bath. The paraffin wax and the surfactant mixture are each charged to separate beakers and heated on a steam-heated water bath to about 130°F. The surfactant mixture is then added to the melted paraffin and the mixture is mechanically stirred. The distilled water is then added to the aforementioned mixture slowly and with stirring, while maintaining the temperature at about 130°F. The heat is then turned off and with continued agitation the paraffin wax emulsion is allowed to cool to room temperature.

In the meantime the syrup solution is charged to a beaker and heated to about 120°F. While maintaining the temperature of the syrup solution at about 120°F the paraffin wax emulsion is added, with stirring, to give the final coating solution.

This wax coating solution was then used in the preparation of coated tablets following the procedure described hereinabove.

The following examples are illustrative of wax coating
solutions containing coloring material which may be used in the practice of this invention:

<table>
<thead>
<tr>
<th>EXAMPLE 2</th>
<th>Grams</th>
<th>Weight percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paraffin wax</td>
<td>2.470</td>
<td>1.0</td>
</tr>
<tr>
<td>Sorbitan monostearate</td>
<td>2.470</td>
<td>1.0</td>
</tr>
<tr>
<td>Polyoxyethylene sorbitan monostearate</td>
<td>2.470</td>
<td>1.0</td>
</tr>
<tr>
<td>Distilled water</td>
<td>2.470</td>
<td>1.0</td>
</tr>
<tr>
<td>Color (Simple Syrup, U.S.P., 288.7 g.) (FD&amp;C Red No. 4, 3.44 g.)</td>
<td>227.100</td>
<td>96.0</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>247.000</strong></td>
<td><strong>100.0</strong></td>
</tr>
</tbody>
</table>

1. **PAROWAX**.  
2. **SPAN 60**.  
3. **TWEEN 60**.

The paraffin wax emulsion of this example is prepared according to the procedure described in Example 1. In preparing the color solution the syrup is charged to a beaker and heated to about 120°F. The dye is then added to the syrup, with stirring, while maintaining the temperature at about 120°F. After the color solution is thoroughly mixed the wax emulsion is added with stirring to give the final coating solution.

This wax coating solution was then used in the preparation of imprinted pharmaceutical tablets following the procedure described above.

<table>
<thead>
<tr>
<th>EXAMPLE 3</th>
<th>Grams</th>
<th>Weight percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paraffin wax</td>
<td>1.258</td>
<td>0.5</td>
</tr>
<tr>
<td>Sorbitan monostearate</td>
<td>1.258</td>
<td>0.5</td>
</tr>
<tr>
<td>Polyoxyethylene sorbitan monostearate</td>
<td>1.258</td>
<td>0.5</td>
</tr>
<tr>
<td>Distilled water</td>
<td>1.258</td>
<td>0.5</td>
</tr>
<tr>
<td>Color (Simple Syrup, U.S.P., 288.5 g.) (FD&amp;C Red No. 4, 3.399 g.)</td>
<td>242.060</td>
<td>98.0</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>247.000</strong></td>
<td><strong>100.0</strong></td>
</tr>
</tbody>
</table>

1. **PAROWAX**.  
2. **SPAN 60**.  
3. **TWEEN 60**.

The procedure for formulating the coating solution of this example is identical to that described in Example 2. This wax coating solution was then used in the preparation of imprinted pharmaceutical tablets following the procedure described above.

<table>
<thead>
<tr>
<th>EXAMPLE 4</th>
<th>Grams</th>
<th>Weight percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paraffin wax</td>
<td>11.963</td>
<td>4.6</td>
</tr>
<tr>
<td>Polyoxyethylene sorbitan monostearate</td>
<td>11.963</td>
<td>4.6</td>
</tr>
<tr>
<td>Distilled water</td>
<td>11.963</td>
<td>4.6</td>
</tr>
<tr>
<td>Color (Simple Syrup, U.S.P., 288.933 g.) (FD&amp;C Red No. 4, 2.881 g.)</td>
<td>222.914</td>
<td>86.2</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>247.000</strong></td>
<td><strong>100.0</strong></td>
</tr>
</tbody>
</table>

1. **PAROWAX**.  
2. **SPAN 60**.  
3. **TWEEN 60**.

The procedure for formulating the coating solution set out above is identical to that described in Example 2. This wax coating solution was then used in the preparation of imprinted pharmaceutical tablets following the procedure described above.

<table>
<thead>
<tr>
<th>EXAMPLE 5</th>
<th>Grams</th>
<th>Weight percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paraffin wax</td>
<td>11.963</td>
<td>4.0</td>
</tr>
<tr>
<td>Sorbitan trioleate</td>
<td>8.705</td>
<td>3.6</td>
</tr>
<tr>
<td>Polyoxyethylene sorbitan trioleate</td>
<td>8.705</td>
<td>3.6</td>
</tr>
<tr>
<td>Distilled water</td>
<td>11.963</td>
<td>4.6</td>
</tr>
<tr>
<td>Color (Simple Syrup, U.S.P., 287.427 g.) (FD&amp;C Red No. 4, 2.881 g.)</td>
<td>190.100</td>
<td>77.0</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>247.000</strong></td>
<td><strong>100.0</strong></td>
</tr>
</tbody>
</table>

1. **PAROWAX**.  
2. **SPAN 60** (Atlas Chemical Industries, Inc.).  
3. **TWEEN 60** (Atlas Chemical Industries, Inc.).

The process for formulating the coating solution set out above is identical to that described in Example 2. This wax coating solution was then used in the preparation of imprinted pharmaceutical tablets following the procedure described above.

Preparation of the paraffin wax emulsion of the above coating solution is the same as that described in Example 1. In preparing the color solution the syrup is charged to a beaker and heated to about 120°F. The lake color is then added to the syrup while maintaining the temperature at about 120°F and the resulting solution is put into a homogenizer or a ball mill and thoroughly mixed. While maintaining the color solution at about 120°F the wax emulsion is added with stirring, to give the final coating solution. This wax coating solution was then used in the preparation of imprinted pharmaceutical tablets following the procedure described above.

In summary, the present invention is directed to an imprinted pharmaceutical tablet and to a method of preparing the same which includes applying an unpolished wax coating to tablets to provide a surface which is particularly deceptive to impriming of conventional shellac-based ink indicia thereon. Application of the wax coating is accomplished by tumbling tacky sugar-coated tablets in contact with an emulsion of wax, such as paraffin wax or carnauba wax, which may also include a coloring material.

What is claimed is:

1. A method of preparing a wax-coated pharmaceutical tablet which comprises the steps of (a) applying a sugar syrup coating to pharmaceutical tablets, and (b) while said coating is in a tacky state applying to said tablets a coating solution containing a wax emulsion.
2. A method according to claim 1, wherein said wax emulsion consists of (a) paraffin wax or carnauba wax, (b) a surfactant or mixtures thereof, and (c) distilled water.

3. A method according to claim 1, wherein said wax emulsion consists of (a) paraffin wax or carnauba wax, (b) a non-ionic surfactant selected from a member of the group consisting of sorbitan fatty acid esters, polyoxyethylene sorbitan fatty acid esters, mono and diglycerides of fat-forming fatty acids, or mixture of said surfactants, and (c) distilled water.

4. A method according to claim 1, wherein the concentration of wax in said coating solution is from about 0.5 to about 4.6 weight percent.

5. A method according to claim 1, wherein the wax is a paraffin wax having a concentration in said coating solution of from about 0.5 to about 4.6 weight percent.

6. A method according to claim 1, wherein the wax is a paraffin wax having a concentration in said coating solution of about 1.0 weight percent.

7. A method according to claim 1, wherein the wax is a carnauba wax having a concentration in said coating solution of from about 1.0 to about 4.0 weight percent.

8. A method according to claim 1, wherein the wax is a carnauba wax having a concentration in said coating solution of about 1.0 weight percent.

9. A method according to claim 1, wherein the tablets are tumbled in said coating solution for a period of from about 2 minutes to about 3 minutes.

10. A method according to claim 1 wherein the resulting wax coating is dried and then imprinted with ink indicia.

11. A method according to claim 10 wherein a transparent protective coating is thereafter applied to the imprinted tablets.

12. A method of preparing imprinted pharmaceutical tablets, which comprises the steps of (a) applying a sugar syrup coating to pharmaceutical tablets, and (b) while said coating is in a tacky state, tumbling said tablets in contact with a coating solution containing a wax emulsion for a period of time sufficient only to completely coat said tablets with an unpolished coating of said wax, (c) drying the unpolished wax coating, and (d) then imprinting ink indicia on a portion of said unpolished wax coating.

13. A method according to claim 8 wherein said wax emulsion is (a) an oil-in-water emulsion of paraffin wax or carnauba wax, and (b) said ink indicia is a shellac-based ink indicia.

References Cited

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3,000,753 9/1961 Rockland 117—92 X
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ALFRED L. LEAVITT, Primary Examiner.
C. R. WILSON, Assistant Examiner.

U.S. Cl. X.R.

117—84, 92, 109; 424—6, 35, 38
UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,438,797

Jerry Allen Biddle, Sr.

It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

Column 4, line 18, "varous" should read -- various --; line 28, "underlyng" should read -- underlying --; in the table, first column, line 5 thereof, "syrup" should read -- Syrup --. Column 6, line 49, "I" should read -- 1 --. Column 8, line 13, claim reference numeral "8" should read -- 12 --.

Signed and sealed this 7th day of April 1970.

(SEAL)
Attest:
Edward M. Fletcher, Jr.
Attesting Officer

WILLIAM E. SCHUYLER, JR.
Commissioner of Patents