

# CAVELIER

ABOGADOS

EDIFICIO SISKI

CARRERA 4 No. 72 - 35

BOGOTÁ 8, COLOMBIA

WEB SITE: www.cavelier.com

E-MAIL: cavelier@cavelier.com

TELEFONO: (57) (1) 347-3611

TELEFAX: (57) (1) 217-9211 (57) (1) 310-4995

Señora Jefe de la  
DIVISION DE NUEVAS CREACIONES  
Superintendencia de Industria y Comercio  
E. S. D.

SUPERINDUSTRIA Y COMERCIO

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Dependencia: 2020 DIVISION DE NUEVAS CREACIONES

TRÁMITE : 002

EVENTO : 001

ACTUACIÓN : 538

Referencia : Expediente No. 00-051.041

Asunto : Solicitud de patente para: REVESTIMIENTOS  
DE PELÍCULA Y COMPOSICIONES PARA  
REVESTIMIENTOS DE PELÍCULA BASADAS EN  
ALCOHOL POLIVINILICO

Solicitante : BERWIND PHARMACEUTICAL SERVICES, INC.

Fecha de solicitud : 07 de julio de 2000

Publicada en : la Gaceta No. 519 del 29 de agosto de 2002

1. Yo, Emilio FERRERO, identificado como aparece al pie de mi firma, actuando como apoderado en el asunto de la referencia, atentamente, solicito se efectúe el examen de patentabilidad de la solicitud en referencia según lo establecido en el artículo 44 de la Decisión 486 de la Comisión de la Comunidad Andina. Para tal efecto presento junto con este escrito el recibo No. 03-13,515 por valor de \$335.000.00, que corresponde a la tasa fijada según lo dispuesto en la resolución No. 41687 del 24 de diciembre de 2002.

2. Ruego atentamente a la Señora Jefe se sirva ordenar que se anexe este recibo, para que se efectúe dicho examen y se continúe con el trámite de la solicitud.

Bogotá, 25 FEB 2003  
Señora Jefe,



EMILIO FERRERO  
T.P.A. No. 31.929

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MRE/JFR/ID-000016/WPC12023 NIT. 860.041.367-3



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**Industria y Comercio****SUPERINTENDENCIA**

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NIT : 800176089-2

RECIBO OFICIAL DE CAJA : 03 - 13,515  
FECHA : FEBRERO 24 DE 2003

## \*\*\*\*\* CONSIGNACION \*\*\*\*\*

| DEPOSITANTE       | TIPO PAGO    | BANCO         | CUENTA      | No. PAGO | FECHA PAGO | Vr. PAGO      |
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| CAVELIER ABOGADOS | CONSIGNACION | BANCO POPULAR | 050-00110-6 | 3136963  | 24/02/2003 | 25,680,000.00 |

## \*\*\*\*\* CONCEPTO \*\*\*\*\*

| CANT.   | RENTISTICO              | CONCEPTO                              | TOTAL CONCEPTO |
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SON: TRESCIENTOS TREINTA Y CINCO MIL PESOS

RESPONSABLE : 

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Carrera 13 No. 27-00 Pisos 5, 7 y 10  
Conmutador 382 0840  
Fax 350 5220  
E-mail: info@sic.gov.co  
www.sic.gov.co  
Bogotá, D.C., Colombia

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**United States Patent** [19]  
**Jordan**

[11] **Patent Number:** **5,885,617**  
[45] **Date of Patent:** **Mar. 23, 1999**

[54] **MOISTURE BARRIER FILM COATING  
COMPOSITION, METHOD, AND COATED  
FORM**

|           |        |           |         |
|-----------|--------|-----------|---------|
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[75] **Inventor:** **Martin Philip Jordan**, Orpington,  
England

[73] **Assignee:** **BPSI Holdings, Inc.**, Wilmington, Del.

[21] **Appl. No.:** **466,939**

[22] **Filed:** **Jun. 6, 1995**

[30] **Foreign Application Priority Data**

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A61K 9/36

[52] **U.S. Cl.** ..... **424/474; 424/461; 424/462;**  
424/463; 424/476; 424/480; 424/481; 424/482;  
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514/781; 514/782; 514/786; 106/210.1;  
106/215.1; 106/243

[58] **Field of Search** ..... 424/474, 482,  
424/497, 498, 461, 462, 479, 480, 481,  
493, 494, 496, 476

[56] **References Cited**

**U.S. PATENT DOCUMENTS**

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*Primary Examiner*—Thurman K. Page

*Assistant Examiner*—James M. Spear

*Attorney, Agent, or Firm*—John F. A. Earley; John F. A.  
Earley, III; Harding, Earley, Follmer & Frailey

[57] **ABSTRACT**

A dry moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets and the like comprises polyvinyl alcohol, soya lecithin, and optionally a flow aid, a colorant, and/or a suspending agent. A liquid coating solution or dispersion for forming a moisture barrier film coating for pharmaceutical tablets and the like comprises polyvinyl alcohol, soya lecithin, water, and optionally a flow aid, a colorant, and/or a suspending agent. A method of coating pharmaceutical tablets and the like with a moisture barrier film coating comprises forming a liquid coating solution or dispersion for forming a moisture barrier film coating for pharmaceutical tablets and the like comprising polyvinyl alcohol, soya lecithin, water, and optionally a flow aid, a colorant, and/or a suspending agent, applying the coating solution or dispersion onto the tablets to form a film coating on the tablets, and drying the film coating on the tablets.

PVA  
talco  
lecithina

**33 Claims, No Drawings**

# MOISTURE BARRIER FILM COATING COMPOSITION, METHOD, AND COATED FORM

## BACKGROUND OF THE INVENTION

### 1. Field of the Invention

This invention is in the field of coating of substrates like pharmaceutical tablets, and it is specifically concerned with coating such substrates with a coating that acts as a moisture barrier.

### 2. Description of the Prior Art

Many medicinal substances and the dosage forms into which they are made undergo degradation during storage due to contact with moisture which has been absorbed from the atmosphere. To combat this degradation, it is frequently necessary for such moisture sensitive dosage forms to be packed in special containers to limit the exposure of the dosage forms to the atmosphere.

In territories where the climate is very humid, specialized packaging does not provide a totally satisfactory answer to moisture degradation.

Elimination of the need for the special packaging, or augmentation of the moisture protection provided by such packaging, may be achieved in the case of solid dosage forms, such as tablets, capsules and granules, by coating with materials which reduce the rate at which the dosage form absorbs atmospheric moisture. Moisture barrier coatings of this type have been employed previously. The traditional means of providing the moisture barrier characteristics required for moisture barrier coatings has been by the use of polymers which are soluble in organic solvents or are relatively insoluble in water. The disadvantages of this approach are 1) the need to use either organic solvents or difficult aqueous polymer dispersion techniques to apply these water-insoluble polymer materials, and 2) the reduced Aqueous solubility of the coating leading to prolonged disintegration time of the dosage form in the body when ingested, with consequent delayed or reduced activity of the medicinal substance in the tablet.

The use of the polymer polyvinyl alcohol, PVA, as a moisture barrier coating has been previously suggested. However, practical usage has been inhibited by the stickiness of grades of the polymer which have a fast enough rate of going into solution in water to make a dispersion to render them economical to use in making the coating. A further problem with the use of PVA is in identifying or selecting a plasticizer which does not compromise the moisture barrier properties of the final coating.

## SUMMARY OF THE INVENTION

A dry powder moisture barrier coating composition is made to form a moisture barrier film coating for pharmaceutical tablets and the like, which comprises polyvinyl alcohol in combination with soya lecithin, together with, none, one or more of the following components which enhances the desirable coating characteristics of the resulting film coating: a flow aid, a colorant, and a suspending agent.

A method of making the moisture barrier film coating powder composition of the invention comprises the steps of mixing polyvinyl alcohol with soya lecithin and optionally with one or more of the following components, until a dry homogenous powder mixture is produced: a flow aid, a colorant, and a suspending agent. Preferably, the mixture of polyvinyl alcohol and soya lecithin, or the mixture of

polyvinyl alcohol and soya lecithin and any of the following components, if any of the following components is to be included in the coating composition, is milled: a flow aid, a colorant, and a suspending agent. The resulting dry powder coating composition is readily soluble or dispersible in cold water to form a liquid coating solution or dispersion that is ready to use within 45 minutes.

Another method of making a dry edible film coating composition of the invention comprises producing a mixture of the ingredients processed into a granular form to form a granular coating composition by using any of the following methods: wet massing, fluid bed granulation, spray granulation and dry compaction, roller compaction or slugging.

The invention also includes a liquid coating solution or dispersion for forming a moisture barrier film coating which comprises polyvinyl alcohol, soya lecithin, an optional flow aid, an optional colorant, and an optional suspending agent mixed into water.

The method of making the liquid coating dispersion of the invention comprises dispersing the dry powder coating composition, the granular coating composition, or the individual ingredients of the coating composition separately, into hot or cold water, and/or milling and/or stirring until a homogenous mixture of a desired correct viscosity is produced.

The film former of the coating is the polyvinyl alcohol, which may be GOHSENL polyvinyl alcohol made by NIPPON GOHSEI, for example.

Excellent moisture barrier properties are obtained when hot water soluble grades of PVA are used in the inventive coating, but use of such grades requires that the water of the liquid coating dispersion be heated. The coatings of the invention may use cold water soluble grades of PVA to provide moisture barrier protection, but they provide less moisture barrier protection than that provided by the inventive coatings that use hot water soluble grades of PVA. A preferred grade of PVA for use in the inventive coating is a grade in the medium range (i.e., the grades of PVA between the grades that dissolve only in hot water and the grades that dissolve very easily in cold water) of PVA because the step of heating the water of the liquid coating dispersion may not be necessary, while still maintaining excellent moisture barrier properties in the inventive coating.

The flow aid may be talc, fumed silica, lactose or starch.

The colorant may be any food approved colors, opacifiers, or dyes. For example, these may be aluminum lakes, iron oxides, titanium dioxide, or natural colors.

The soya lecithin, which may be ALCOLEC soya lecithin made by American Lecithin Company, for example, acts as an anti-tack agent, and surprisingly, and unexpectedly, acts as a plasticizer by locking moisture in the coating so the coating stays flexible and not brittle. Surprisingly and unexpectedly, this locked-in moisture in the coating, while contributing to plasticization, does not compromise the moisture barrier properties of the overall coating.

The suspending agent acts as a viscosity modifier stabilizing the coating dispersion. A preferred suspending agent is xanthan gum. Examples of alternative suitable suspending agents include hydroxypropylmethlycellulose (HPMC), alginates, hydroxypropyl cellulose (HPC), natural gums, and carboxymethylcellulose.

The ranges for each component of the dry coating compositions of the invention are as follows, by weight:

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PVA

|                   | Acceptable<br>Ranges | Preferred<br>Ranges |
|-------------------|----------------------|---------------------|
| Polyvinyl Alcohol | 20.0% to 99.8%       | 30 to 99.0%         |
| Flow Aid          | 0.0% to 40.0%        | 14.0 to 25.0%       |
| Colorant          | 0.0% to 60.0%        | 25.0 to 40.0%       |
| Soya Lecithin     | 0.2% to 10.0%        | 1.0 to 6.0%         |
| Suspending Agent  | 0.0% to 2.0%         | 0.15 to 1.0%        |

The ranges for each component of the liquid coating dispersions of the invention are as follows, by weight:

|                   | Acceptable<br>Ranges | Preferred<br>Ranges |
|-------------------|----------------------|---------------------|
| Polyvinyl Alcohol | 5.0 to 40.0%         | 7.0 to 12.0%        |
| Flow Aid          | 0.0 to 16.0%         | 3.0 to 5.0%         |
| Colorant          | 0.0 to 24.0%         | 4.8 to 8.0%         |
| Soya Lecithin     | 0.04 to 4.0%         | 0.3 to 0.5%         |
| Suspending Agent  | 0.0 to 0.8%          | 0.07 to 0.12%       |
| Water             | 60.0 to 95.0%        | 75.0 to 85.0%       |

#### DETAILED DESCRIPTION

We now turn to the examples of the invention, all ingredients being by weight.

#### EXAMPLE 1

455.2 grams of polyvinyl alcohol, 200.0 grams of talc, 320.0 grams of titanium dioxide, 4.8 grams of xanthan gum, and 20.0 grams of soya lecithin are loaded into a dry powder mill, such as a PK blender, and vigorously mixed therein for 25 minutes or until a homogenous mix is achieved, resulting in one formulation of the inventive dry powder moisture barrier film coating composition.

480 grams of this formulation of the inventive dry powder moisture barrier film coating composition is dispersed in 1701.8 grams of purified water to form an aqueous suspension of the invention. Specifically, 1701.8 grams of purified water at ambient temperature is loaded into a vessel having a diameter that is approximately equal to the depth of the final suspension. A low shear mixer, preferably having a mixing head that has a diameter that is approximately  $\frac{1}{3}$  the diameter of the mixing vessel, is lowered into the water and turned on to create a vortex from the edge of the vessel down to just above the mixing head without any air being drawn into the water. Then, the 480.0 grams of the moisture barrier film coating powder composition is added to the vortex at a rate where there is no excessive build up of dry powder. The speed or depth of the mixing head is adjusted to avoid air being drawn into the suspension so as to avoid foaming. The suspension is stirred for 45 minutes and is then ready for spraying onto substrates like pharmaceutical tablets.

In this Example 1, 10 Kg of tablets are coated with the coating of the invention so as to obtain about a 4.0% weight gain. The extra 80 grams of the dry powder moisture barrier film coating composition above the 400 grams of the dry powder moisture barrier film coating composition needed to obtain a 4.0% weight gain on the tablets is included to allow for losses of coating composition during the coating procedure. 10 Kg of tablets which have logos embossed therein are loaded into a Manesty Model 10 Accela-Cota coater, and the tablets are rotated in the coater at a rotation speed of 12 rpm. The tablets are sprayed with the coating suspension of this Example 1 while the tablets are being rotated. Hot air at 60° C. is used to evaporate the water in sprayed coating suspension and to maintain the tablets at 38° C.

Upon completion of the coating process, the tablets show a smooth surface with excellent logo definition.

In the following Examples 2 to 14, one Kg batches of coating suspension are produced by mixing 220 grams of inventive dry powder moisture barrier coating composition into 780 grams of purified water. These coating suspensions are then sprayed onto tablets. The procedures set out in Example 1 for making the dry powder moisture barrier coating composition, for making the coating suspension, and for spraying the coating suspension onto tablets are used in Examples 2 to 14. The coated tablets of the Examples 2 to 14 show a smooth surface with excellent logo definition.

#### EXAMPLE 2

| COMPONENT        | PERCENTAGE | GRAMS   |
|------------------|------------|---------|
| PVA              | 45.85      | 458.50  |
| TALC             | 20.00      | 200.00  |
| TITANIUM DIOXIDE | 32.00      | 320.00  |
| XANTHAN GUM      | 0.15       | 1.50    |
| SOYA LECITHIN    | 2.00       | 20.00   |
|                  | 100.00     | 1000.00 |

#### EXAMPLE 3

| COMPONENT        | PERCENTAGE | GRAMS   |
|------------------|------------|---------|
| PVA              | 44.80      | 448.00  |
| TALC             | 20.00      | 200.00  |
| TITANIUM DIOXIDE | 32.00      | 320.00  |
| XANTHAN GUM      | 1.20       | 12.00   |
| SOYA LECITHIN    | 2.00       | 20.00   |
|                  | 100.00     | 1000.00 |

#### EXAMPLE 4

| COMPONENT        | PERCENTAGE | GRAMS   |
|------------------|------------|---------|
| PVA              | 46.52      | 465.20  |
| TALC             | 20.00      | 200.00  |
| TITANIUM DIOXIDE | 32.00      | 320.00  |
| XANTHAN GUM      | 0.48       | 4.80    |
| SOYA LECITHIN    | 1.00       | 10.00   |
|                  | 100.00     | 1000.00 |

#### EXAMPLE 5

| COMPONENT         | PERCENTAGE | GRAMS   |
|-------------------|------------|---------|
| PVA               | 41.52      | 415.20  |
| TALC              | 20.00      | 200.00  |
| TITANIUM DIOXIDE  | 18.00      | 180.00  |
| YELLOW IRON OXIDE | 14.00      | 140.00  |
| XANTHAN GUM       | 0.48       | 4.80    |
| SOYA LECITHIN     | 6.00       | 60.00   |
|                   | 100.00     | 1000.00 |

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EXAMPLE 6

| COMPONENT        | PERCENTAGE | GRAMS   |
|------------------|------------|---------|
| PVA              | 31.64      | 316.40  |
| TALC             | 34.09      | 340.90  |
| TITANIUM DIOXIDE | 32.00      | 320.00  |
| XANTHAN GUM      | 1.13       | 11.30   |
| SOYA LECITHIN    | 1.14       | 11.40   |
|                  | 100.00     | 1000.00 |

## EXAMPLE 7

| COMPONENT        | PERCENTAGE | GRAMS   |
|------------------|------------|---------|
| PVA              | 50.40      | 504.00  |
| TALC             | 14.40      | 144.00  |
| TITANIUM DIOXIDE | 32.00      | 320.00  |
| XANTHAN GUM      | 1.20       | 10.00   |
| SOYA LECITHIN    | 2.00       | 20.00   |
|                  | 100.00     | 1000.00 |

## EXAMPLE 8

| COMPONENT     | PERCENTAGE | GRAMS   |
|---------------|------------|---------|
| PVA           | 99.00      | 990.00  |
| SOYA LECITHIN | 1.00       | 10.00   |
|               | 100.00     | 1000.00 |

## EXAMPLE 9

| COMPONENT     | PERCENTAGE | GRAMS   |
|---------------|------------|---------|
| PVA           | 99.80      | 998.00  |
| SOYA LECITHIN | 0.20       | 2.00    |
|               | 100.00     | 1000.00 |

## EXAMPLE 10

| COMPONENT                | PERCENTAGE | GRAMS   |
|--------------------------|------------|---------|
| PVA                      | 44.00      | 440.00  |
| TALC                     | 20.00      | 200.00  |
| TITANIUM DIOXIDE         | 18.00      | 180.00  |
| TARTRAZINE ALUMINUM LAKE | 14.00      | 140.00  |
| XANTHAN GUM              | 2.00       | 20.00   |
| SOYA LECITHIN            | 2.00       | 20.00   |
|                          | 100.00     | 1000.00 |

## EXAMPLE 11

| COMPONENT        | PERCENTAGE | GRAMS  |
|------------------|------------|--------|
| PVA              | 50.80      | 508.00 |
| TALC             | 14.00      | 140.00 |
| TITANIUM DIOXIDE | 32.00      | 320.00 |

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-continued

| COMPONENT     | PERCENTAGE | GRAMS   |
|---------------|------------|---------|
| XANTHAN GUM   | 1.20       | 12.00   |
| SOYA LECITHIN | 2.00       | 20.00   |
|               | 100.00     | 1000.00 |

## EXAMPLE 12

| COMPONENT        | PERCENTAGE | GRAMS   |
|------------------|------------|---------|
| PVA              | 40.52      | 405.20  |
| TALC             | 25.00      | 250.00  |
| TITANIUM DIOXIDE | 32.00      | 320.00  |
| XANTHAN GUM      | 0.48       | 4.80    |
| SOYA LECITHIN    | 2.00       | 20.00   |
|                  | 100.00     | 1000.00 |

## EXAMPLE 13

| COMPONENT        | PERCENTAGE | GRAMS   |
|------------------|------------|---------|
| PVA              | 25.52      | 255.20  |
| TALC             | 40.00      | 400.00  |
| TITANIUM DIOXIDE | 32.00      | 320.00  |
| XANTHAN GUM      | 0.48       | 4.80    |
| SOYA LECITHIN    | 2.00       | 20.00   |
|                  | 100.00     | 1000.00 |

## EXAMPLE 14

| COMPONENT        | PERCENTAGE | GRAMS   |
|------------------|------------|---------|
| PVA              | 45.52      | 455.20  |
| TALC             | 20.00      | 200.00  |
| TITANIUM DIOXIDE | 32.00      | 320.00  |
| XANTHAN GUM      | 0.48       | 4.80    |
| SOYA LECITHIN    | 2.00       | 20.00   |
|                  | 100.00     | 1000.00 |

## EXAMPLE 15

| COMPONENT        | GRAMS   |
|------------------|---------|
| WATER            | 1170.00 |
| PVA              | 150.00  |
| TALC             | 66.00   |
| TITANIUM DIOXIDE | 105.75  |
| XANTHAN GUM      | 1.65    |
| SOYA LECITHIN    | 6.60    |
|                  | 1500.00 |

The coating dispersion of this Example 15 is made and used using the procedures set out in Example 1.

It was unexpectedly found that soya lecithin, included for its tack reduction properties, acts as a plasticizer by locking moisture in the coating so as to keep the coating flexible and not brittle. It is also considered unexpected that this moisture while contributing to plasticization, does not compromise the moisture barrier properties of the overall coating.

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This invention provides the means of preparing a moisture barrier coating for application to pharmaceutical solid dosage forms in which the coating composition material is presented as a dry particle powder or granular powder which is readily dispersible in cold water. The resultant coating solution or dispersion is ready to use by applying it to tablets within 45 minutes. The coating solution or dispersion may also be presented in the form of a ready-to-use liquid, or liquid concentrate which is diluted prior to use.

Compared with existing moisture barrier coatings using water insoluble polymers, there is negligible effect on the disintegration time of the coated dosage forms coated with the inventive coating.

The dry form of the invention is manufactured using rotary mixers or mills. The ingredients are loaded and then milled until a homogenous mix is produced. In the case of liquid dispersions, the ingredients are dispersed into hot or cold water and stirred or milled until a homogenous dispersion or solution of the desired correct viscosity is produced.

The dry form of the invention, which comprises a powder of PVA and soya lecithin, and optionally a flow aid, a colorant, and a suspending agent, is dispersed in water and stirred until the PVA is hydrated and a homogenous mixture is produced. The liquid solution or dispersion formed, which comprises PVA, soya lecithin, and any optional components mixed into water, may be ready-to-use or require simple dilution with water prior to use. The resulting coating dispersion is sprayed onto the dosage forms using airless or airborne methods. The movement of air around or through the tablet bed facilitates drying of the film on the tablet surface.

I claim:

1. A liquid moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising

polyvinyl alcohol,  
soya lecithin,  
water, and

a viscosity modifier,

the viscosity modifier being xanthan gum, hydroxypropyl-methylcellulose, alginate, hydroxypropyl cellulose, natural gums, carboxymethylcellulose, or combinations thereof.

2. A liquid moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising

polyvinyl alcohol,  
soya lecithin, and  
water,

the water being in a range of about 60.0% to about 95.0% by weight of the composition.

3. The liquid composition of claim 2,

the water being in a range of about 75.0% to about 85.0% by weight of the composition.

4. A pharmaceutical tablet coated with a moisture barrier film coating made using a dry moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising polyvinyl alcohol, a flow aid, a colorant, a viscosity modifier, and soya lecithin.

5. A dry moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising

polyvinyl alcohol, and  
soya lecithin

the polyvinyl alcohol being in a range of about 20.0% to about 99.8% by weight of the composition, and  
the soya lecithin being in a range of about 0.2% to about 10.0% by weight of the composition.

6. The coating composition of claim 5,

the polyvinyl alcohol being in a range of about 30.0% to about 99.0% by weight of the composition, and  
the soya lecithin being in a range of about 1.0% to about 6.0% by weight of the composition.

7. A dry moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising

polyvinyl alcohol,  
soya lecithin, and  
a flow aid

the flow aid being in a range of greater than 0.0% to about 40.0% by weight of the composition.

8. The composition of claim 7,

the flow aid being in a range of about 14.0% to about 25% by weight of the composition.

9. A dry moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising

polyvinyl alcohol,  
soya lecithin, and  
a flow aid,

the flow aid being talc, fumed silica, lactose, or starch.

10. A dry moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising

polyvinyl alcohol,  
soya lecithin, and  
a colorant,

the colorant being in a range of greater than 0.0% to about 60.0% by weight of the composition.

11. The coating composition of claim 10,

the colorant being in a range of about 25.0% to about 40.0% by weight of the composition.

12. A dry moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising

polyvinyl alcohol,  
soya lecithin, and  
a colorant,

the colorant being any food approved colors, opacifiers or dyes.

13. A dry moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising

polyvinyl alcohol,  
soya lecithin, and  
a viscosity modifier,

the viscosity modifier being in a range of greater than 0.0% to about 2.0% by weight of the composition.

14. The coating composition of claim 13,

the viscosity modifier being in a range of about 0.15% to about 1.0% by weight of the composition.

15. A dry moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising

polyvinyl alcohol,  
soya lecithin, and  
a viscosity modifier,  
the viscosity modifier being a xanthan gum.

16. A dry moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising  
polyvinyl alcohol,  
soya lecithin, and  
a viscosity modifier,  
the viscosity modifier being hydroxypropyl-methylcellulose, alginate, hydroxypropyl cellulose, natural gums, carboxymethylcellulose, or combinations thereof.

17. The coating composition of claim 6,  
the soya lecithin being in a range of about 1.0% further including  
a flow aid,  
the flow aid being in a range of about 14% to about 25% by weight of the composition,  
the flow aid being talc, fumed silica, lactose, or starch,  
a colorant,  
the colorant being in a range of about 25.0% to about 40.0% by weight of the composition,  
the colorant being any food approved colors, opacifiers, or dyes,  
a viscosity modifier,  
the viscosity modifier being in a range of about 0.15% to about 1.0% by weight of the composition,  
the viscosity modifier being xanthan gum, hydroxypropyl-methylcellulose, alginate, hydroxypropyl cellulose, natural gums, carboxymethylcellulose, or combinations thereof.

18. A method of coating substrates with a moisture barrier film coating, comprising  
mixing the coating composition according to claim 5 into water to form an aqueous coating solution/dispersion,  
applying the coating solution/suspension onto the substrates to form a film coating on the substrates, and  
drying the film coating on said substrates.

19. A pharmaceutical tablet coated with a moisture barrier film coating made using the coating composition according to claim 17.

20. A method of coating substrates with a moisture barrier film coating, comprising  
mixing the coating composition according to claim 17 into water to form an aqueous coating solution/dispersion,  
applying the coating solution/suspension onto the substrates to form a film coating on the substrates, and  
drying the film coating on said substrates.

21. A dry moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising  
polyvinyl alcohol, and  
soya lecithin,  
the polyvinyl alcohol being in a range of about 20.0% to about 99.8% by weight of the composition.

22. A dry moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising  
polyvinyl alcohol, and

soya lecithin,  
the soya lecithin being in a range of about 0.2% to about 10.0% by weight of the composition.

23. A coated substrate made in accordance with the method of claim 18.

24. A liquid moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising  
polyvinyl alcohol,  
soya lecithin, and  
water,  
the polyvinyl alcohol being in a range of about 5.0% to about 40.0% by weight of the composition, and  
the soya lecithin being in a range of about 0.04% to about 4.0% by weight of the composition.

25. The liquid composition of claim 24,  
the polyvinyl alcohol being in a range of about 7.0% to about 12.0% by weight of the composition, and  
the soya lecithin being in a range of about 0.3% to about 0.5% by weight of the composition.

26. A liquid moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising  
polyvinyl alcohol,  
soya lecithin,  
water, and  
a flow aid,  
the flow aid being in a range of greater than 0.0% to about 16.0% by weight of the composition.

27. The liquid composition of claim 26,  
the flow aid being in a range of about 3.0% to about 5.0% by weight of the composition.

28. A liquid moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising  
polyvinyl alcohol,  
soya lecithin,  
water, and  
a flow aid,  
the flow aid being talc, fumed silica, lactose, or starch.

29. A liquid moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising  
polyvinyl alcohol,  
soya lecithin,  
water, and  
a colorant,  
the colorant being in a range of greater than 0.0% to about 24.0% by weight of the composition.

30. The liquid composition of claim 29,  
the colorant being in a range of about 4.8% to about 8.0% by weight of the composition.

31. A liquid moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising  
polyvinyl alcohol,  
soya lecithin,  
water, and  
a colorant,  
the colorant being any food approved colors, opacifiers or dyes.

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32. A liquid moisture barrier film coating composition for forming a moisture barrier film coating for pharmaceutical tablets comprising

polyvinyl alcohol,  
soya lecithin,  
water, and  
a viscosity modifier,

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the viscosity modifier being in a range of greater than 0.0% to about 0.8% by weight of the composition.

33. The liquid composition of claim 32,

the viscosity modifier being in a range of about 0.07% to about 0.12% by weight of the composition.

\* \* \* \* \*

# United States Patent [19]

Keith et al.

[11]

4,432,965

[45]

Feb. 21, 1984

## [54] QUINIDINE SUSTAINED RELEASE DOSAGE FORMULATION

[75] Inventors: Alec D. Keith, Miami; Charles Hsiao,  
Cooper City, both of Fla.

[73] Assignee: Key Pharmaceuticals, Inc., Miami,  
Fla.

[21] Appl. No.: 396,668

[22] Filed: Jul. 9, 1982

[51] Int. Cl.<sup>3</sup> ..... A61K 9/22; A61K 9/24;  
A61K 9/32

[52] U.S. Cl. .... 424/19; 424/21;  
424/32; 424/33

[58] Field of Search ..... 424/19, 22, 32, 33

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Primary Examiner—Shep K. Rose

Attorney, Agent, or Firm—Wegner & Bretschneider

[57]

## ABSTRACT

A sustained release oral dosage form is provided comprising a tablet core containing a pharmaceutically effective amount of quinidine. The tablet core is coated with a sustained release polymeric coating which contains about 5 to about 20 percent by weight polyethylene glycol component having a molecular weight of from about 500 to about 200, and from about 80 to 95 percent by weight polyvinylalcohol component. The polyvinylalcohol component comprises from about one to about ten parts by weight of a partially hydrolyzed polyvinylalcohol subcomponent having a degree of hydrolysis of from about 75 to about 92 percent and about one part by weight of a substantially completely hydrolyzed polyvinylalcohol subcomponent having a degree of hydrolysis in excess of 95%.

4 Claims, No Drawings

- PVA  
- PEG

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in the preceding Examples. There were thusly obtained pharmacologically satisfactory, pink colored tablet coatings which had a good gloss.

Coatings with a different, deeper red color, as well as somewhat shorter breakdown times, were obtained by admixture of the same synthetic resin dispersion with an aqueous suspension of 25 parts of talc, 5 parts of saccharose, 5 parts of polyethylene glycol (average molecular weight 20,000), 5 parts of ferric oxide pigment and 2 parts of titanium dioxide in 80 parts of demineralized water.

#### EXAMPLE 8

Example 7 is duplicated using in place of ACRONAL 14 D, EUDRAGIT L 30D and EUDRAGIT E 30D aqueous polyacrylate dispersions. Good results are obtained.

#### EXAMPLE 9

20 parts of talc, 30 parts of chalk, 7 parts of saccharose, 2 parts of polyvinyl pyrrolidone ("Kollidon 25"), 10 parts of ferric oxide pigment were suspended in 120 parts of demineralized water and mixed with 200 parts of an approximately 50% aqueous dispersion of a butadiene-styrene copolymer ("Litex SB 40").

When this mixture was applied to tablet cores by the method described in Examples 1 or 2, there were obtained pharmacologically satisfactory tablet coatings of good appearance which broke down about as quickly as good dragees.

What is claimed is:

1. Process for preparing coated tablets which comprises applying to a core of active material at least one layer of a coating composition comprising a film forming aqueous synthetic resin dispersion with a resin content between 30 and 60% by weight, from 2 to 50% by

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weight of a material which is soluble in either the gastric or alkaline juices of the small intestine and up to 50% by weight of one or more insoluble fillers the amounts of said soluble material and said fillers being based on the amount of said aqueous synthetic resin dispersion, the combined amount of said water soluble material and said fillers being at least 5-6%, and permitting said coating composition layer to dry.

2. Process of claim 1 which comprises first applying to the tablet core a coating of a member selected from the group consisting of polyvinyl alcohol, polyvinyl pyrrolidone and solid polyethylene glycol.

3. Process of claim 1 which comprises first applying to the tablet cores a coating of a composition resistant to gastric juice.

4. Process of claim 1 wherein said material which is soluble in either the gastric or alkaline juices is selected from the group of salt, sugar, polyethylene glycol, polyvinyl pyrrolidone and starch syrup.

5. Process of claim 1 wherein said material which is soluble in either the gastric or alkaline juices is a higher fatty acid.

6. Process of claim 1 wherein said aqueous dispersion contains a synthetic resin selected from the group of polyvinyl ester, polyvinyl acetal, polyvinyl chloride, butadiene-styrene copolymer and polyacrylic acid esters.

7. Process of claim 1 wherein said aqueous dispersion contains a polyacrylate.

8. Process of claim 1 wherein said cores are introduced into a tumbling zone and said aqueous dispersion is sprayed into said zone and the resultant coated cores are dried by introducing dry air into said tumbling zone and withdrawing water containing air therefrom.

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# United States Patent [19]

Branco et al.

[11] Patent Number: 4,665,648

[45] Date of Patent: May 19, 1987

[54] FILM-FORMING COMPOSITIONS FOR  
ENVELOPING GRAINS AND SEEDS

[75] Inventors: Bernard Branco, Le Chesnay; Michel  
Malandain, Marly Le Roe, both of  
France

[73] Assignee: Seppic SA, Paris, France

[21] Appl. No.: 823,458

[22] Filed: Jan. 28, 1986

## Related U.S. Application Data

[60] Continuation-in-part of Ser. No. 666,762, Oct. 31, 1984,  
Pat. No. 4,576,646, which is a division of Ser. No.  
627,665, Jul. 3, 1984, Pat. No. 4,513,019.

## [30] Foreign Application Priority Data

Jul. 6, 1983 [FR] France ..... 83 11276

[51] Int. Cl.<sup>4</sup> ..... A61K 9/36; C08L 1/00;  
A01C 1/06

[52] U.S. Cl. .... 47/57.6; 47/56;  
47/74; 106/163.1

[58] Field of Search ..... 47/57.6, 56, 74

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Primary Examiner—Theodore Morris

Attorney, Agent, or Firm—Dennison, Meserole, Pollack  
& Scheiner

## [57] ABSTRACT

The present invention relates to film-forming composi-  
tions for enveloping grains and seeds, comprising by  
weight:

15 to 85% of a cellulosic film-forming substance,  
10 to 70% of at least one alpha cellulose,  
1 to 30% of at least one plasticizer suitable for com-  
sumption.

8 Claims, No Drawings

D4 152  
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## FILM-FORMING COMPOSITIONS FOR ENVELOPING GRAINS AND SEEDS

This application is a continuation-in-part of Ser. No. 666,762, now U.S. Pat. No. 4,576,646 filed Oct. 31, 1984, which is a divisional of Ser. No. 627,665, filed July 3, 1984, now U.S. Pat. No. 4,513,019. These applications are hereby incorporated by reference.

The present invention relates to film-forming compositions for enveloping solid forms, particularly grains and seeds; it also relates to a process for enveloping with said compositions and to products obtained, coated with these compositions.

In the field of grains and seeds, coating is the operation in which seeds of various shapes are treated to obtain seeds of a spherical and regular shape. This operation is generally carried out with fillers such as silica, kaolin, peat and cork and synthetic or natural polymer binders such as gelatin, polyvinyl alcohol, and cellulosic derivatives.

This coating operation is distinguished from the film-forming operation, in which coated or uncoated seeds are enveloped with a fine and even film. Film-forming has been carried out most often by powdering at the surface of the seeds, mixtures of pigments and phytosanitary treatment products. Film-forming can also be performed with polymer binders of different types such as acrylic and vinyl. These binders should be used cautiously since they tend to delay or prevent germination of seeds. Powdering on the other hand, has the disadvantages of poor adherence of the powder to the seed surface, and involving dust and treatment irregularities.

### SUMMARY OF THE INVENTION

It is an object of the present invention to provide an improved composition for film-forming on coated and uncoated seeds.

It is a further object of the present invention to provide a composition for film-forming on grains and seeds which can be applied at a low temperature without damaging the grains and seeds and without affecting the germination thereof.

It is a further object of the present invention to provide a film-forming composition with high binding power which enables large quantities of phytosanitary products, organic and inorganic pigments, and mineral fillers to adhere to the surface of the grain.

To achieve these and other objects, the present invention provides a method for film-forming on grains and seeds comprising spraying on to the grains or seeds an aqueous solution or dispersion of a composition comprising, by weight, 15 to 85% of a cellulosic film-forming substance, 10 to 70% of at least one alpha cellulose, and 1 to 30% of at least one plasticizer.

### DETAILED DESCRIPTION OF THE INVENTION

The cellulosic film-forming substances used according to the present invention are known materials which have previously been used in the coating art. Generally these materials may be selected from the alkylethers of cellulose, the hydroxyalkylethers of cellulose, the monocarboxylic esters of cellulose and the mixed ether esters of cellulose. The preferred celluloses are hydroxypropylmethylcelluloses, hydroxypropylethylcelluloses, hydroxypropylcelluloses, and methylcelluloses, having a

viscosity in a 2% by weight aqueous solution at ambient temperature of less than 60 cps.

The film-forming compositions of the present invention should contain at least about 15% by weight of the cellulosic film-forming substance. Contents of the film-forming substance less than about 15% by weight are insufficient to form a continuous film. In that case, the envelope, as seen by examination by microscope, is a simple juxtaposition of particles. On the other hand, with contents of film-forming substances greater than 85%, the envelope formed is similar to that of the prior art, presenting the problems known to the art.

The plasticizer used according to the present invention will preferably be one which is suitable for consumption. While the use of plasticizers with cellulosic film-forming substances is known, the essential function of the plasticizers in the present case is not to lower the melting point of the celluloses, but rather to modify the suppleness and strength of the films made with these celluloses. The plasticizer utilized may be a hydrophilic or hydrophobic product suitably selected to improve the suppleness of the film made.

Hydrophilic plasticizers make it possible to obtain films which are rapidly disintegrated in an acid medium or in water. Hydrophilic plasticizers which are suitable include glycerine, propylene glycol, polyethyleneglycol, sorbitol and saccharose.

Hydrophobic plasticizers make it possible to delay disintegration of the film. Suitable hydrophobic plasticizers include conventional fatty substances, such as alcohols, acids, fatty esters and derivatives thereof, and ethyl and butyl esters of phthalic, citric and sebacic acid.

The preferred plasticizers for coating grains and seeds are polyethylene glycols having a molecular weight of 400 to 6000, esters of fatty acids and polyethyleneglycol, and polyethoxylated fatty acids. One such preferred plasticizer is the stearate ester of polyoxyethylene 8, which is hydrodispersible and facilitates dispersion of the film-forming compositions of the present invention.

The film-forming compositions of the present invention will preferably contain at least 1% by weight (with respect to the dry film-forming substance) of the plasticizer. The plasticizer amount utilized for film coating of grains and seeds can be less than the amount which would be required in coating, for example, pharmaceutical tablets, due to the higher surface hardness of grain and seeds, and the lesser tendency for deformation. Moreover, when germination occurs, the film formed at the seed surface must crack easily to allow the plant to sprout through. A plasticizer content of more than 30% modifies the mechanical properties of the envelope formed, tending to make it too fragile.

The alpha celluloses which may be used in the compositions according to the present invention are preferably microcrystalline or fibrous alpha celluloses which are known to the art, and which have a mean granulometry of less than 100 microns, and preferably less than 60 microns. It is known that alpha celluloses are capable of performing the role of "binding agent" in products with OH functions and in particular, cellulosic derivatives, due to their propensity to create hydrogen bonds with other materials. In the present film-forming composition, the alpha cellulose also performs an important role in effecting good adherence of the coating film on the core seed or grain, which contains materials having OH functions.

The alpha cellulose content of the present film-forming compositions should be at least about 10%, since lower amounts do not give a binding power sufficient to modify the film-forming substance, and the film does not adhere well to the core. An alpha cellulose content greater than about 70% by weight, however, produces a film of insufficient pliability.

The compositions according to the invention may further comprise known additives conventionally used for modifying the properties of the coating material, such as may affect color or speed of dissolution, and additives for protecting the coating material, such as anti-oxidants and anti-ultra-violet additives.

The preferred process for utilizing the film-forming compositions according to the present invention comprises dissolving or suspending the various components of the composition in a suitable solvent such as an aqueous medium, and then spraying the solution or suspension obtained onto the previously prepared core grain or seeds. The aqueous solutions or dispersions may contain concentrations up to about 25% by weight of the film-forming composition, which enables coatings to be made in a short period of time, for example 15 minutes. The films obtained are very covering and adhere strongly to the solid substrate. Consequently, they present an excellent resistance to abrasion and peeling. Moreover, the hardness of the cores coated with these film-forming compositions is especially increased, and the films obtained are regular and may provide attractive colors with small quantities sprayed.

The film-forming compositions according to the present invention have a number of purposes other than simply increasing the hardness of the seeds and grains. These film-formers can be used to adhere phytosanitary, insecticidal, fungicidal and pigmented coatings to the grain and seeds. The pigmented products are especially useful in identifying the type of seed or grain and in checking the efficiency and regularity of the sowings. The coating followed by film-forming can also be used to diminish unevenness in the seeds to make the outflow from the sowing drills easier.

The quantity of fillers, phytosanitary products, and pigments which can be bound to the grains or seeds with the present film-forming compositions, can be as much as twelve times the weight of the film-forming composition itself without any tendency for powdering. By utilizing these fillers and other treatment products directly as coatings on the seeds, it is possible to modify the gloss of the color of the seeds and the sliding properties of the seeds. Coloration may also be added by adding a colorant adhering to the cellulosic film-former.

Another reason for the use of the film-forming composition is to maintain an effective protection against dampness while maintaining or improving the germinative properties of the seeds.

The film-forming compositions used in conjunction with seeds and grains will preferably be low temperature coating compositions.

The following examples will serve to illustrate the invention.

#### EXAMPLE 1

##### Film-forming on Onion Seeds

A film-forming composition containing 35 parts by weight hydroxypropylmethylcellulose (15 cps), 55 parts alpha cellulose (average particle size 20 microns), and 10 parts by weight polyethyleneglycol 6000 (PEG 6000) is prepared. The hydroxypropylmethylcellulose

and the powdered alpha cellulose are first mixed for 4 minutes in a Lodge granulator. Then, a 20% solution of PEG 6000 in water is slowly added to the powder mixture to produce a composition having a granulometry of about 0.5 mm, which is homogeneous enough to avoid calibration. The granulate is removed from the granulator and is dried over a plate oven to produce the film-forming composition.

A pigmentary composition is prepared from 20 parts by weight blue phthalocyanine pigment, 40 parts titanium dioxide, and 40 parts talc. The powder mixture is introduced in a mixture of 190 parts water with 10 parts polyethoxylated nonylphenol. This mixture is then ground in a horizontal ball crusher to produce a blue fluid paste having about 35% solids.

A coating composition is prepared by introducing 10 parts by weight of the above formed film-forming composition into 78 parts water with stirring. After 20 minutes, a homogeneous suspension is obtained to which is added 12 parts by weight of the pigmentary composition.

300 grams of onion seeds are placed in suspension in a tepid air stream in a Aeromatic fluidized bed system provided with a spraying system. The air intake temperature of the fluidized bed is 55° C. while the exiting air temperature is 30° C. The coating composition is sprayed onto the onion seeds at a rate of 16 grams per minute at a spraying pressure of 2 Kg/cm<sup>2</sup>. Coating takes place over a period of 12 minutes during which a quantity of coating composition equal to 8% of the starting weight of the onion seeds is sprayed. The resultant coated seeds are uniformly and homogeneously blue in color. Germination tests demonstrate that the seeds are not affected by the coating treatment.

#### EXAMPLE 2

##### Film-forming on Coated Lettuce Seeds

A film-forming composition is prepared by mixing together 30 parts by weight hydroxypropylmethylcellulose (15 cps), 60 parts by weight microcrystalline alpha cellulose, and 50 parts by weight talc in a granulator. 10 parts by weight polyethyleneglycol 6000 in a 20% aqueous solution is added to the granulator along with 30 parts by weight of a yellow pigmentary dispersion comprising 50% yellow iron oxide. The granulate produced is dried as in Example 1, and is dispersed in 700 parts by weight water by stirring for 20 minutes. 100 parts by weight of a phytosanitary paste (50% actives) is then added to the dispersion, the paste comprising a mixture of insecticide and fungicide. This produces a coating composition containing about 3% hydroxypropylmethylcellulose, 1% polyethyleneglycol 6000, 6% microcrystalline cellulose, 2.5% pigment, 5% talc, and 5% phytosanitary products, for a total of about 22.5% solids.

Lettuce seeds are initially rendered spherical by coating with a mixture of clay, kaolin and polyvinyl binder. The coated seeds weigh 1.5 kg. The film-forming composition is applied to the seeds in a conventional 40" diameter turbine rotating at 15 rpm. The lettuce seeds are maintained at 30° C. Spraying of the film-forming composition takes place with a Binks 460 "Airspray" spraying gun at a rate of 12 grams per minute and a pressure of 2 kg/cm<sup>2</sup>. Coating takes place for 30 minutes, during which time an amount of film-forming composition equal to 8% of the weight of the seeds is applied. The seeds are dried with a hot air generator

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with an adjustable power of 200 to 800 watts. A yield of about 85% seeds with film formed is obtained, with the seeds being yellow in color, slightly glossy and homogeneous. The germination of these seeds is identical to that of seeds without the film.

### EXAMPLE 3

#### Film-Forming on Maize Seeds

A film-forming composition is prepared from:

- 20 parts of methylcellulose, 15 cps
- 60 parts of cellulose powder, average particle size 60 microns
- 2 parts of polyethylene glycol 400
- 14 parts of talcum
- 4 parts of iron oxide yellow.

by the following process: 5 kg of methylcellulose, 15 cps, is mixed with 15 kg of cellulose powder, average particle size is 60 microns, and 3.5 kg of talcum in a DIOSNA granulator having a volume of 100 l.

An aqueous dispersion is prepared in a high-speed disperser with 500 g of PEG 400 and 1 kg of iron oxide yellow in 10 liters of water. This dispersion is added to the powder in the DIOSNA granulator.

Granulation of the mixture is then carried out with 1.5 liters of water, using the high-speed to uniformize the particle size of granules which are formed. The granules are then dried in an oven for 9 hours at 80° C.

The resultant granulates are yellow, and have a particle size between 0.2 and 3 mm.

150 g of these granules are easily dispersed in 850 g of water, under moderate stirring for 20 minutes. A homogeneous suspension of 350 cps viscosity is obtained. 150 g of phytosanitary product (based on captane/anthraquinone) are added to the suspension to obtain a coating composition.

1.5 kg of maize seeds are put in a conventional pan of 40 cm diameter, and 44 g of the coating composition are sprayed, using a Binks 460 air-spray gun, on the 1.5 kg of maize in rotation in the pan over a period of 4 min-

utes. A uniform and slightly glossy coating is obtained on the seeds.

The total deposit on the seeds is 0.4% by weight of the phytosanitary product and 0.4% by weight of the dry substance of the film forming product, relative to the weight of the coated seeds.

What is claimed is:

1. Seeds or grains having a coating thereon, said coating comprising, by weight, 15 to 85% of a cellulosic film-forming substance, 10 to 70% of at least one alpha cellulose, and 1 to 30% of at least one plasticizer.

2. Seeds or grains according to claim 1, wherein said cellulosic film-forming substance is selected from the group consisting of alkylethers of cellulose, hydroxyalkylethers of cellulose, monocarboxylic esters of cellulose, and mixed ether esters of cellulose.

3. Seeds or grains according to claim 2, wherein said cellulosic film-forming substance is a hyroxpropylmethylcellulose having a viscosity of less than 60 cps at ambient temperature in a 2% by weight aqueous solution.

4. Seeds or grains according to claim 1, wherein said alpha cellulose is a microcrystalline or fibrous alpha cellulose having an average granulometry less than 60 microns.

5. Seeds or grains according to claim 1, wherein said plasticizer is selected from the group consisting of polyethyleneglycols of molecular weight 400 to 6000, esters of fatty acids and polyethyleneglycol, and polyethoxylated fatty acids.

6. Seeds or grains according to claim 1, wherein said coating additionally comprises at least one additional substance selected from the group consisting of fillers, pigments, and phytosanitary products.

7. Seeds or grains according to claim 6, wherein said phytosanitary products are selected from the group consisting of insecticides, fungicides, and mixtures thereof.

8. Seeds or grains according to claim 1 which are rendered spherical by an initial coating thereon.

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**UNITED STATES PATENT AND TRADEMARK OFFICE  
CERTIFICATE OF CORRECTION**

PATENT NO. : 4,665,648

DATED : May 19, 1987

INVENTOR(S) : Bernard Brancq; Michel Malandain

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

On the Title Page, Item [75] "Branco" should read -- Brancq --.

**Signed and Sealed this  
Eleventh Day of August, 1987**

*Attest:*

DONALD J. QUIGG

*Attesting Officer*

*Commissioner of Patents and Trademarks*

# United States Patent Office

D5 157 153  
3,554,767

Patented Jan. 12, 1971

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3,554,767

## COATED CONFECTIONERY

Wilhelm Daum, deceased, late of Darmstadt, Germany, by Martin Daum, Elisabeth Daum, Wilfried Daum, and Irmgard Daum, heirs, all of Darmstadt, Germany, and Eberhard Nürnberg and Hans Joachim Sippel, Darmstadt, Germany, assignors to E. Merck A.G., Patentabteilung, Darmstadt, Germany

No Drawing. Continuation-in-part of application Ser. No. 835,823, Jan. 6, 1969, which is a division of application Ser. No. 449,637, Apr. 20, 1965, now Patent No. 3,420,931. This application Feb. 24, 1969, Ser. No. 802,759

Claims priority, application Germany, Apr. 23, 1964, M 60,752

Int. Cl. A23g 3/00; A23l 1/27; B44d 1/00  
U.S. Cl. 99—134

16 Claims

## ABSTRACT OF THE DISCLOSURE

An improved method for producing coated confectioneries wherein the confectionery coating solution comprises about 35–60% by weight of sugar and about 0.5–10% by weight of a vinyl polymer, such as polyvinyl pyrrolidone, this method permitting automatic operation, and markedly reducing the time conventionally employed for producing sugar coated candies. The resultant confectioneries are also claimed.

This is a continuation-in-part application of copending application S.N. 835,823, filed Jan. 6, 1969, a divisional of earlier application S.N. 449,637, filed Apr. 20, 1965, now Pat. 3,420,931, the claim of priority of German application M 60,752 filed Apr. 23, 1964, being maintained for this application.

This invention relates to the production of sugar-containing coatings on masses of ingestible solid material.

Whereas this invention is broadly directed to the production of sugar-containing coatings on shaped masses of ingestible solid material, it has particular application to the production of coated confectioneries.

For the purposes of this invention, a mass without a sugar-containing coating will be referred to as a "candy core" including but not limited to such ingestibles as chocolate, gum, raisins, nuts, etc.

Though it may appear to be a simple task, heretofore the production of sugar coated candies has been time-consuming. To build up an aesthetically acceptable coating of sufficient thickness, the practice has been to apply several thin coats with scrupulous care to prevent the candies from adhering to one another, as well as to avoid non-uniformity of the final product.

An object of this invention, therefore, is to provide an improved process for the production of coated shaped masses of solid material, and in particular to an improved process for the production of sugar coated confectioneries.

Upon further study of the specification and claims, other objects and advantages of the present invention will become apparent.

To attain these objects, there is provided a process which basically comprises coating a core with an aqueous dispersion comprising, in percent by weight, 1 to 60, preferably 35 to 40% of a sugar and 0.5 to 10% of a vinyl polymer, preferably a polymer of vinyl pyrrolidone and/or a polymer of vinyl acetate.

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medicament is coated with a solution of these high molecular weight substances alone, the solutions being applied to the tablets or other pressings in any suitable way, for example, by spraying. Varnished or film-coated tablets obtained in this way cannot be regarded as dragées since they have only a thin coating of varnish consisting of the polymer and they do not have a conventional dragée coating made up principally of sugar and the above-mentioned additives. The use of a vinyl polymer in the coloring of dragées, that is, dragées, which already have the actual dragée coating, has also been described in the literature, Kohler et al., Deutsche Apotheker-Zeitung 102(1): 1–8, Jan. 4, 1962. The term "dragée" in its dictionary sense is defined as "anything sugar coated as a pill" (Funk & Wagnalls New Standard Dictionary of the English Language, page 756, 1942 Funk & Wagnalls Co.) (1a): a sugar coated nut, (b): a silver coated candy for decorating cakes, (2): a sugar coated medicated confection (Webster's Seventh New College Dictionary, page 251, 1963, G. & C. Merriam Co.). As used herein, the term dragée does not embrace medicated confections which are covered in the earlier application, now Pat. 3,420,911. The coating pigment suspension used contained 0.325% of polyvinyl pyrrolidone in addition to sugar and other substances and is used to color the dragées by pigment dyes, that is, only for the last stage of the dragées coating process.

If this suspension described in literature is used as the sole coating composition for the coating process and is applied to the uncoated cores, it is found that the coating process is very considerably lengthened unless pulverulent substances, such as talc, are additionally introduced into the rotating vessel. The surface of the dragées obtained tends to be sticky and a large number of application is necessary.

Using the coating composition according to the invention, it has surprisingly been found that the entire coating process can be carried out with this composition alone since it can be applied manually or by suitable apparatus, with or without spraying, without any inconvenient sticking. It has not been found necessary to additionally introduce pulverulent substances, such as talc, into the rotating vessel during the time of the coating process. With rationalized operation, the entire coating process can be carried out in one or two days. It was particularly unexpected that such a substantially improvement can be achieved by increasing the polymer from 0.325% to at least 0.5%, preferably to about 2%.

The preferred polymers include homopolymers of vinyl pyrrolidone, vinyl acetate, and vinyl alcohol, and copolymers of vinyl pyrrolidone-vinyl acetate, and vinyl alcohol-vinyl acetate. These polymers are characterized by their physiological compatibility.

It has been particularly advantageous to apply commercial preparations of polyvinyl pyrrolidone with a dry content of at least 94 percent, a maximal ash content of 0.02 percent and a maximal monomer content of 0.8 percent, molecular weights between 10,000 and 800,000, preferably about 20,000 to 30,000, showing viscosity parameters K of between 17 and 90, preferably about 25.

(The viscosity parameter K is defined by the following equation:

$$\log Z = \left( \frac{75K^2}{1 + 1.5Kc} \right) c$$

and 40 percent vinyl acetate with the following characteristics: dry content: at least 95 percent; maximal monomer content: 0.8 percent; viscosity parameter K:  $34 \pm 6$ ; nitrogen content: about 7.6%.

The molecular weight of the polymer can vary widely. In general, polymers of molecular weights between about 10,000 and 2,000,000 are applied. It is important, however, that the monomer content of the polymer is kept as low as possible because of the possibly toxic side effects of higher concentrations of some of the monomers. In general, monomer contents lower than 1 percent by weight are unobjectionable.

As for the other essential ingredients of the coating, the percent by weight of sugar in the aqueous suspension is 1 to 60, preferably 35 to 40. Various types of sugars can be employed, such as sucrose, glucose, sorbitol, mannitol, etc.; it is preferable, however, to use sucrose.

Aside from water, sugar and polymer, it is possible to incorporate other materials in the dispersion. The effectiveness of the vinyl polymers, particularly of the polyvinyl pyrrolidone, can be increased by adding other high-molecular compounds, such as cellulose derivatives, e.g. methyl cellulose, ethyl cellulose, carboxymethyl cellulose, hydroxyethyl cellulose, hydroxypropyl cellulose, or starches, e.g. potato, corn, wheat, rice starch, or starch gum. It is, for instance, possible to reduce the polymer content to one-half of the original by adding about 0.5 percent by weight of one of the cellulose derivatives mentioned. This is a considerable economical and, in some cases, also a processing improvement.

Moreover, the suspension may contain pigments, such as titanium dioxide or calcium carbonate, lubricants (talc or finely divided silica), fillers such as flour, binders, plasticizers and/or softeners, e.g. glycerine, and preservatives. Usually, in accordance with one advantage of this invention, the addition of preservatives can be avoided because it is possible to work up a batch in a few days and during this time no growth of bacteria or fungi occurs.

The viscosity of the final dispersion can vary widely, depending on the composition of it, furthermore, on temperature, air content, thixotropic effects, previous agitation and even time. In general, the viscosity of the dispersion is between 500 and 15,000 preferably about 2000 centipoises.

By using the dispersion of this invention, it was surprisingly discovered that it is possible to conduct the entire coating process only with one coating composition. This is the case because the dispersion of this invention can be applied either manually or mechanically, with or without spraying, and without the candies sticking to one another. Of most importance is that it is possible to carry out the entire coating process within one to two days. In contrast thereto, the average working time for the manufacture of one batch according to the conventional process is approximately one week. Thus, compared with the usual process, the new process represents a considerable saving of time, resulting in substantial labor savings, as well as in substantially increased plant capacity.

As another distinct advantage, the dispersion of this invention is so stable that it is unnecessary during the course of candy manufacture to use mixing or stirring devices, pumps, or other homogenizing equipment to prevent settling. A still further advantage of the invention consists in the fact that during the course of the whole process, there is no requirement for organic solvents. Moreover, during the course of the process it is unnecessary to employ intermediate drying steps for which purpose, in the usual coating processes, the dragées had to be removed temporarily from the suspension.

suspension. This was done by either the coating operator in a strewing motion, or by the use of complicated spray equipment always being subject to both clogging and breakdown. In contrast, the process of this invention avoids such disadvantages, because according to this process, it is possible to apply the suspension onto the candy cores at room temperature, manually or by machine, without the necessity of either spraying or strewing in powdery materials.

The application of the coating dispersion can be adapted to different types of operation. It is possible to lay on the coating either manually, or semi-automatically or completely automatically. The suspension can be applied by spraying or it can be poured in liquid form directly onto the cores tumbling in an angularly oriented rotating kettle. The suspension is applied until the material to be coated is well moistened. The kettle is then allowed to rotate until the coated cores no longer adhere to one another but instead roll freely. Finally, the coated cores are dried under continuous rotation, preferably with the aid of a warm air stream, or an infrared lamp, or any other equivalent drying means. These steps can be conducted continuously; and it is possible, in this connection, to shorten or even omit the rotating phase (second phase) during the first coating applications. It is also unnecessary to remove the candies from the coating vessel during the course of the entire coating procedure in order to post-dry them, perhaps under vacuum or in drying chambers. A further essential advantage of this invention consists in that all of the process steps can be conducted at ambient temperature. In general, each core is coated with 2 to 500, preferably 50 to 80 separate thin coats, each coat having a thickness of 0.5 to 200  $\mu$ , preferably 5 to 20  $\mu$ .

A particularly economical mode of conducting the process comprises passing a predetermined volume of the suspension through a relay-controlled solenoid valve into the dragee coating vessel to cover the dragees rolling around therein. Suitably, a three-phase time relay is employed, such that in the first phase the suspension is passed through the solenoid valve; in the second phase the rotating operation is conducted, the duration of which can be varied; and in the third phase the heating device is actuated. The suspension can be applied onto cores whose surface is not pretreated. However, it is also possible to coat cores which have been previously provided with a sugarless coating, for instance with a lacquer coating resistant to stomach acids.

The sugar coated candies produced in accordance with this invention cannot be distinguished by their exterior from those which are produced according to the classical method of coating dragees. They likewise taste sweet, can be colored, exhibit a natural silky sheen, and can, if desired, be lacquered. Their elastic and mechanical strengths meet the highest requirements.

The quantity of suspension depends on the weight and the shape of the candy cores. Normally, the quantity is preferably at least about 1%, especially 10 to 100% of the weight of the core, on a dry basis.

With respect to the finished product, the ratio of the components in the coating is the same as that of the coating solution, the exception being, of course, that water and/or other solvents are absent in the final coating.

Without further elaboration, it is believed that one skilled in the art can, using the preceding description, utilize the present invention to its fullest extent. The following preferred specific embodiments are, therefore, to be construed as merely illustrative, and not limitative of the remainder of the specification and claims in any

3,554,767

### 5 EXAMPLE 1

|                       | Percent |
|-----------------------|---------|
| Polyvinyl pyrrolidone | 4       |
| Titanium dioxide      | 4.5     |
| Calcium carbonate     | 4.5     |
| Talc                  | 14.5    |
| Glycerine             | 2       |
| Sucrose               | 38      |
| Water                 | 32.5    |

Polyvinyl pyrrolidone is dissolved in water; then, the remaining components are added under constant stirring; thereafter, the mixture is passed through a fine sieve or a homogenizing device.

### EXAMPLE 2

|                                         | Percent |
|-----------------------------------------|---------|
| Methyl cellulose                        | 0.5     |
| Polyvinyl pyrrolidone                   | 2.0     |
| Aerosil (particulate SiO <sub>2</sub> ) | 4.0     |
| Titanium dioxide                        | 4.5     |
| Calcium carbonate                       | 4.5     |
| Talc                                    | 14.5    |
| Glycerine                               | 2.0     |
| Sucrose                                 | 38.0    |
| Water                                   | 30.0    |

### EXAMPLE 3

|                                                                    | Percent |
|--------------------------------------------------------------------|---------|
| Hydroxyethyl cellulose                                             | 0.5     |
| Polyvinyl pyrrolidone-polyvinyl acetate copolymer (porportion 6:4) | 2.0     |
| Titanium dioxide                                                   | 4.5     |
| Calcium carbonate                                                  | 4.5     |
| Talc                                                               | 18.5    |
| Glycerine                                                          | 2.0     |
| Sucrose                                                            | 38.0    |
| Water                                                              | 30.0    |

### EXAMPLE 4

|                   | Percent |
|-------------------|---------|
| Swelling amylose  | 0.5     |
| Polyvinyl alcohol | 2.0     |
| Titanium dioxide  | 4.5     |
| Calcium carbonate | 4.5     |
| Talc              | 18.5    |
| Glycerine         | 2.0     |
| Sucrose           | 38.0    |
| Water             | 30.0    |

### EXAMPLE 5

|                                               | Percent |
|-----------------------------------------------|---------|
| Carboxy methyl cellulose                      | 0.5     |
| Polyvinyl alcohol-polyvinyl acetate copolymer | 2.0     |
| Titanium dioxide                              | 4.5     |
| Calcium carbonate                             | 4.5     |
| Talc                                          | 18.5    |
| Glycerine                                     | 2.0     |
| Sucrose                                       | 38.0    |
| Water                                         | 30.0    |

### EXAMPLE 6

|                                                          | Percent |
|----------------------------------------------------------|---------|
| Methyl cellulose                                         | 0.5     |
| Polyvinyl pyrrolidone (average molecular weight 650,000) | 0.5     |
| Titanium dioxide                                         | 4.5     |
| Calcium carbonate                                        | 4.5     |

### 6 EXAMPLE 7

|                                                            | Percent |
|------------------------------------------------------------|---------|
| Methyl cellulose                                           | 0.5     |
| Polyvinyl pyrrolidone (average molecular weight 10-15,000) | 10.0    |
| Titanium dioxide                                           | 4.5     |
| Calcium carbonate                                          | 4.5     |
| Silicon dioxide                                            | 4.0     |
| Talc                                                       | 6.5     |
| Glycerine                                                  | 2.0     |
| Sucrose                                                    | 38.0    |
| Water                                                      | 30.0    |

### EXAMPLE 8

|                                         | Percent |
|-----------------------------------------|---------|
| Methyl cellulose                        | 0.5     |
| Polyvinyl pyrrolidone                   | 2.0     |
| Aerosil (particulate SiO <sub>2</sub> ) | 4.0     |
| Titanium dioxide                        | 6.5     |
| Calcium carbonate                       | 6.5     |
| Magnesium stearate                      | 2.5     |
| Glycerine                               | 3.0     |
| Sucrose                                 | 45.0    |
| Water                                   | 30.0    |

The following examples are those for producing the dragées of this invention:

### EXAMPLE 9

26 kg. of cores, each core weighing 65 mg. and consisting of chocolate are mixed manually in a conventional dragée vessel having a diameter at the opening of 90 cm., with 0.5 liter of a suspension (produced in accordance with Example 2), at a rate of rotation of 15 revolutions per minute. The rotation is continued for 10 minutes and then for a further 10 minutes with forced hot air (temperature 30-40° C.; 4.5 m.<sup>3</sup>/minute). This process—coating, rotating, drying—is repeated over again until a weight of the individual coated cores of 100 mg. is reached. Finally, the candies are kept in motion for a while longer with air being blown in, for the purpose of obtaining a silky sheen which is preserved by applying a solution of beeswax in carbon tetrachloride, or another suitable polishing agent, and subsequent rotating for 1 to 2 hours. Total duration of the dragée production process: 16 hours (2 working days).

### EXAMPLE 10

40 kg. of cores, each core weighing 420 mg. and consisting of gum are placed into a dragée coating vessel of a width of 90 cm. at its opening. 25 kg. of a suspension, produced according to Example 5 and mixed with 1% coloring agent (Tartrazine, Hydrazine yellow, FD & C yellow No. 5), are added piecemeal at ambient temperature from a storage vessel with the aid of a solenoid valve controlled by means of a 3-phase timing relay. The three phases of the relay are set as follows: 1st phase (coating application): 5 seconds; 2nd phase (rotating without hot air supply); 10 minutes; 3rd phase (rotating with hot air blown in as in Example 8): 10 minutes. The dragée coating process is continued until the individual weight of the resultant dragée amounts to 600 mg., whereupon a polishing step is conducted as in Example 8. Total duration of dragée manufacturing process: 16 hours (2 working days).

By means of this process, candies are obtained which can have a coating that is colored in all layers. It is also possible to utilize first a colorless suspension and to apply a colored suspension only at the end of the dragée producing process.

From the preceding examples, it is seen that this invention provides a substantial improvement in the sugar coating of ingestible shaped masses of solid material

Noted

being that on a comparative basis, the coating dispersion of this invention provides improved results over prior art coatings used for the same purpose. For preferred operation, however, the following time cycles are used:

- | Step:                                                                                                                                                             | Time             |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------|
| (a) Coating application                                                                                                                                           | 1 to 60 seconds. |
| (b) Rotating (tumbling) time in a conventional dragée coater having a 90 cm. diameter at the opening and a rotational velocity of 5 to 60 revolutions per minute. | 1 to 60 minutes. |
| (c) Drying time with either forced air having a temperature of 20 to 70° C., or an equivalent heat input.                                                         | 1 to 60 minutes. |

From the foregoing description, one skilled in the art can easily ascertain the essential characteristics of this invention, and without departing from the spirit and scope thereof, can make various changes and modifications of the invention to adapt it to various usages and conditions.

What is claimed is:

1. A sugar coated confectionery consisting essentially of an inner medicament-free dragée, chocolate, chewing gum, raisin, nut or like candy core coated with at least about 1% of the weight of the core, on a dry basis, constituting 2-500 separate thin coats, each coat having a thickness of about 0.5-200 microns, and dried from an aqueous dispersion comprising about 35-60% by weight of a sugar and about 0.5-10% by weight of a vinyl polymer selected from the group consisting of homopolymer of vinyl pyrrolidone, homopolymer of vinyl acetate, homopolymer of vinyl alcohol, copolymer of vinyl pyrrolidone-vinyl acetate and copolymer of vinyl alcohol-vinyl acetate.

2. A coated confectionery as defined by claim 1 wherein said vinyl polymer is polyvinyl pyrrolidone.

3. A coated confectionery as defined by claim 1 wherein said coats further comprise not more than about 30% by weight of another solid material selected from the group consisting of talc, finely divided silica, calcium carbonate, titanium dioxide and mixtures thereof.

4. A coated confectionery as defined by claim 1 wherein said aqueous dispersion further comprises about 0.5% by weight of a member selected from the group consisting of methyl cellulose, ethyl cellulose, carboxymethyl cellulose, hydroxyethyl cellulose, hydroxypropyl cellulose, and starch.

5. A coated confectionery as defined by claim 1 wherein said coats comprise about 10-100% by weight of the core, on a dry basis.

6. A coated confectionery as defined by claim 1 wherein at least 50 separate thin coats are employed.

7. A coated confectionery as defined by claim 2 wherein said coats comprise about 10-100% by weight of the core, on a dry basis.

8. A coated confectionery as defined by claim 1 wherein the aqueous dispersion contains at least about 2% of the vinyl polymer.

9. A coated confectionery as defined by claim 2 wherein the aqueous dispersion contains at least about 2% of the polyvinyl pyrrolidone.

10. A process for the production of a coated confectionery as defined by claim 1 comprising the steps of pouring the aqueous dispersion directly on cores tumbling in an angularly oriented rotating kettle; continuing the rotating until the coated cores roll freely; and then drying the coated cores.

11. A process as defined by claim 10 wherein the process is conducted automatically.

12. A process as defined by claim 10 wherein the coated cores are not removed from the kettle until the entire number of coats are applied.

13. A process as defined by claim 10 wherein all the steps are conducted at ambient temperatures.

14. A process as defined by claim 10 wherein the process is conducted automatically in the same kettle, the time for the coating step being 1-60 seconds, the time for the rotating step being 1-60 minutes, and the time for drying being 1-60 minutes, the entire process taking not more than two days.

15. A sugar coated confectionery as defined by claim 1, wherein at least one of said separate sugar coats contain a coloring agent.

16. A sugar coated confectionery as defined by claim 1, further comprising an outer polishing coat of beeswax.

#### References Cited

##### UNITED STATES PATENTS

|           |        |                   |         |
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##### OTHER REFERENCES

Kohler et al., Deutsche Apotheker-Zeitung 102(1):1:8, Jan. 4, 1962.

SHEP K. ROSE, Primary Examiner

U.S. Cl. X.R.

99-23, 100, 104, 126, 135, 138, 141, 148, 161, 166; 106-162, 210; 117-100, 104, 105.3, 105.4, 161.16; 424-33.35

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156

157

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**SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO  
DIVISION DE NUEVAS CREACIONES**

375.  
2005/07/21

2020  
Bogotá, D.C., Julio 8 de 2005

Doctora  
**EMILIO FERRERO**

|               |                |            |
|---------------|----------------|------------|
| <b>ASUNTO</b> | Radicación No. | 00 051 041 |
|               | Trámite        | 002        |
|               | Evento         | 001        |
|               | Actuación      | 430        |
|               | Oficio No.     |            |
|               | Folios         | 0          |

**05946**

Como resultado del examen de patentabilidad de la solicitud de patente de invención, realizado según lo establece el artículo 45 de la Decisión 486 de la Comisión de la Comunidad Andina, se notifica el siguiente concepto técnico:

**1. Documentos estudiados**

Para el presente estudio se tuvieron en cuenta el capítulo descriptivo comprendido en los folios 2 - 18 y el nuevo capítulo reivindicatorio de los folios 53 - 66 de la solicitud en estudio.

**2. Objeto de la Invención**

El objeto de la presente solicitud de patente de invención consiste en:

Las reivindicaciones 1 – 22 se refieren a una composición de revestimiento de película seca que comprende PVA, plastificante (polietilenglicol ó glicerina), talco y opcionalmente pigmento/colorante/opacificante y lecitina.

Las reivindicaciones 23 – 44 se refieren a una dispersión de revestimiento de película líquida que comprende PVA, plastificante (polietilenglicol ó glicerina), talco, agua y opcionalmente pigmento/colorante/opacificante y lecitina.

Las reivindicaciones 45 – 66 se refieren al método para revestir sustratos que comprende los pasos de (1) mezclar PVA, plastificante (polietilenglicol ó glicerina), talco, agua para formar una dispersión (2) aplicar la dispersión sobre los sustratos (3) secar.

El problema planteado en esta solicitud es la necesidad de proporcionar un revestimiento para tabletas farmacéuticas, suplementos nutricionales, alimentos, confituras, semillas agrícolas, revestimiento que es una película de rápida disolución y tiene una superficie lisa, apariencia brillante, pegajosidad mínima, buena adhesión de película y buena resistencia a la tensión.

Para resolver este problema técnico la solicitud en estudio proporciona una dispersión de revestimiento de película seca que comprende PVA, plastificante

(polietilenglicol ó glicerina), talco, agua y opcionalmente pigmento/colorante/opacificante y lecitina.

El objeto de la invención definido anteriormente se clasificó de acuerdo con la IPC: A 61 K 9/30.

También se reivindica prioridad de la Solicitud de Patente de los Estados Unidos US 09 351 076 de julio 9 de 1999, con respecto a la cual la fecha de presentación de la actual solicitud cumple el plazo máximo permitido.

### 3. Determinación del Estado de la Técnica

El artículo 16 de la decisión 486 de la comisión de la Comunidad Andina que establece que *"El estado de la técnica comprenderá todo lo que haya sido accesible al público por una descripción escrita u oral, utilización, comercialización o cualquier otro medio antes de la fecha de presentación de la solicitud de patente o, en su caso, de la prioridad reconocida."*

*Solo para el efecto de la determinación de la novedad, también se considerará dentro del estado de la técnica, el contenido de una solicitud de patente en trámite ante la oficina nacional competente, cuya fecha de presentación o de prioridad fuese anterior a la fecha de presentación o de prioridad de la solicitud de patente que se estuviese examinando, siempre que dicho contenido esté incluido en la solicitud de fecha anterior cuando ella se publique o hubiese transcurrido el plazo previsto en el artículo 40".*

Considerando que la fecha de presentación de la primera solicitud de patente de la cual se reivindica prioridad es julio 9 de 1999, se consultaron las bases de datos y los archivos con que cuenta la entidad con fecha de publicación anterior y se encontraron los siguientes documentos que anticipan el objeto de esta solicitud:

| No. | Documento    | Título                                                                                                 | Fecha de Publicación |
|-----|--------------|--------------------------------------------------------------------------------------------------------|----------------------|
| D1  | US 5 885 617 | Composición de cubierta de película que es una barrera contra la humedad, método y forma de recubierta | Mar 23, 1999         |
| D2  | US 4 432 965 | Formulación de dosis de liberación sostenida de Quinidina                                              | Feb 21, 1984         |
| D3  | US 3 935 326 | Proceso para recubrir tabletas con dispersiones acuosas de resina                                      | Ene 27, 1976         |
| D4  | US 4 665 648 | Composiciones formadoras de película para recubrimiento de granos y semillas                           | May 19, 1987         |
| D5  | US 3 554 767 | Dulces recubiertos                                                                                     | Ene 12, 1971         |

### 4. Evaluación de los requisitos de Patentabilidad

El artículo 14 de la Decisión 486 de la Comisión de la Comunidad Andina establece que *"Los Países Miembros otorgarán patentes para las invenciones, sean de producto o de procedimiento, en todos los campos de la tecnología, siempre que sean nuevas, tengan nivel inventivo y sean susceptibles de aplicación industrial"*.

En cumplimiento del artículo 14 de la Decisión 486, se procedió a evaluar el cumplimiento de los requisitos de novedad, nivel inventivo y aplicación

industrial, con el fin de establecer la patentabilidad de la invención reivindicada en la solicitud en estudio.

### **Evaluación de la Novedad**

El Art. 16 de la Decisión 486 de la Comisión de la Comunidad Andina establece: *"Una invención se considerará nueva cuando no está comprendida en el estado de la técnica. El estado de la técnica comprenderá todo lo que haya sido accesible al público por una descripción escrita u oral, utilización comercialización o cualquier otro medio antes de la fecha de presentación de la solicitud de patente, o, en su caso, de la prioridad reconocida."*

*Solo para el efecto de la determinación de la novedad, también se considerará dentro del estado de la técnica, el contenido de una solicitud de patente en trámite ante la oficina nacional competente, cuya fecha de presentación o de prioridad fuese anterior a la fecha de presentación o de prioridad de la solicitud de patente que se estuviese examinando, siempre que dicho contenido esté incluido en la solicitud de fecha anterior cuando ella se publique o hubiese transcurrido el plazo previsto en el Art 40".*

Las reivindicaciones 1 – 22 se refieren a una composición de revestimiento de película seca que comprende PVA, plastificante (polietilenglicol ó glicerina), talco y opcionalmente pigmento/colorante/opacificante y lecitina.

Las reivindicaciones 23 – 44 se refieren a una dispersión de revestimiento de película líquida que comprende PVA, plastificante (polietilenglicol ó glicerina), talco, agua y opcionalmente pigmento/colorante/opacificante y lecitina.

Las reivindicaciones 45 – 66 se refieren al método para revestir sustratos que comprende los pasos de (1) mezclar PVA, plastificante (polietilenglicol ó glicerina), talco, agua para formar una dispersión (2) aplicar la dispersión sobre los sustratos (3) secar.

D1 enseña una composición de revestimiento de película seca que comprende PVA, talco, pigmento/colorante/opacificante y lecitina; enseña una composición de revestimiento de película líquida que comprende PVA, talco, pigmento/colorante/opacificante, lecitina y agua; y enseña el método para revestir sustratos que comprende los pasos de (1) mezclar PVA, talco, agua para formar una dispersión (2) aplicar la dispersión sobre los sustratos (3) secar (ver folios 133 - 139).

D3 enseña una dispersión de revestimiento de película que comprende PVA, plastificante (polietilenglicol), talco, agua y pigmento/colorante/opacificante y lecitina. Enseña el método para revestir sustratos que comprende los pasos de (1) mezclar PVA, talco, agua para formar una dispersión (2) aplicar la dispersión sobre los sustratos (3) secar (ver folios 143 - 151).

D4 enseña una dispersión de revestimiento de película que comprende polímero, plastificante (polietilenglicol ó glicerina), talco, agua y pigmento/colorante/opacificante y lecitina (ver folios 152 - 156).

D5 enseña una dispersión de revestimiento de película que comprende PVA, plastificante (glicerina), talco, agua y pigmento/colorante/opacificante; y enseña el método para revestir sustratos que comprende los pasos de (1) mezclar PVA, talco, agua para formar una dispersión (2) aplicar la dispersión sobre los sustratos (3) secar (ver folios 157 - 160).

Se observa que los elementos constitutivos de la composición de las reivindicaciones 1 - 44, es decir: PVA, plastificante (polietilenglicol ó glicerina), talco, agua y opcionalmente pigmento/colorante/opacificante y lecitina, ya estaban contenidos en las composiciones tanto de D3 que enseña una dispersión de revestimiento de película que comprende PVA, plastificante (polietilenglicol), talco, agua y pigmento/colorante/opacificante y lecitina; como de D4 que enseña una dispersión de revestimiento de película que comprende polímero, plastificante (polietilenglicol ó glicerina), talco, agua y pigmento/colorante/opacificante y lecitina y D 5 que enseña una dispersión de revestimiento de película que comprende PVA, plastificante (glicerina), talco, agua y pigmento/colorante/opacificante; por lo cual no puede considerarse nueva la composición de las reivindicaciones 1 - 44.

Se observa también que los pasos que conforman el método para revestir sustratos, de las reivindicaciones 45 a 66, es decir: (1) mezclar PVA, plastificante (polietilenglicol ó glicerina), talco, agua para formar una dispersión (2) aplicar la dispersión sobre los sustratos (3) secar ya se habían mencionado tanto en D1 que enseña los pasos de (1) mezclar PVA, talco, agua para formar una dispersión (2) aplicar la dispersión sobre los sustratos (3) secar; como en D3 que enseña (1) mezclar PVA, plastificante), talco, agua para formar una dispersión (2) aplicar la dispersión sobre los sustratos (3) secar y en D5 que enseña (1) mezclar PVA, plastificante (polietilenglicol ó glicerina), talco, agua para formar una dispersión (2) aplicar la dispersión sobre los sustratos (3) secar; por lo cual no puede considerarse nuevo el método de las reivindicaciones 45 a 66.

### **Evaluación del Nivel Inventivo**

El artículo 18 de la Decisión 486 de la Comisión de la Comunidad Andina establece que *Se considerará que una invención tiene nivel inventivo, si para una persona del oficio normalmente versada en la materia técnica correspondiente, esa invención no hubiese resultado obvia ni se hubiese derivado de manera evidente del estado de la técnica.*

Las reivindicaciones 1 – 22 se refieren a una composición de revestimiento de película seca que comprende PVA, plastificante (polietilenglicol ó glicerina), talco y opcionalmente pigmento/colorante/opacificante y lecitina.

Las reivindicaciones 23 – 44 se refieren a una dispersión de revestimiento de película líquida que comprende PVA, plastificante (polietilenglicol ó glicerina), talco, agua y opcionalmente pigmento/colorante/opacificante y lecitina.

D1 enseña una composición de revestimiento de película seca que comprende PVA, talco, pigmento/colorante/opacificante y lecitina; enseña una composición de revestimiento de película líquida que comprende PVA, talco, pigmento/colorante/opacificante, lecitina y agua; y enseña el método para revestir sustratos que comprende los pasos de (1) mezclar PVA, talco, agua para formar una dispersión (2) aplicar la dispersión sobre los sustratos (3) secar (ver folios 133 - 139).

D2 enseña se refieren a una composición de revestimiento de película que comprende PVA y plastificante (polietilenglicol) (ver folios 140 - 142).

Se observa que los elementos constitutivos de la composición de las reivindicaciones 1 - 44, es decir: PVA, plastificante (polietilenglicol ó glicerina), talco, agua y opcionalmente pigmento/colorante/opacificante y lecitina, ya estaban contenidos en las composiciones tanto de D1 que enseña una composición de revestimiento de película seca que comprende PVA, talco, pigmento/colorante/opacificante y lecitina y enseña también una composición de revestimiento de película líquida que comprende PVA, talco, pigmento/colorante/opacificante, lecitina y agua; como de D2 que enseña una composición de revestimiento de película que comprende PVA y plastificante (polietilenglicol); estas anterioridades entonces sugieren la combinación en una sola composición, de PVA, plastificante (polietilenglicol ó glicerina), talco, agua y opcionalmente pigmento/colorante/opacificante y lecitina, que es objeto de esta solicitud, por lo cual no hay nivel inventivo.

Se observa que la combinación de las enseñanzas de estas anterioridades: D1 que se refiere a una composición de revestimiento de película seca que comprende PVA, talco, pigmento/colorante/opacificante y lecitina y también a una composición de revestimiento de película líquida que comprende PVA, talco, pigmento/colorante/opacificante, lecitina y agua y D2 que se refiere a una composición de revestimiento de película que comprende PVA y plastificante (polietilenglicol); sugiere la composición de las reivindicaciones 1 - 44 que contiene PVA, plastificante (polietilenglicol ó glicerina), talco, agua y opcionalmente pigmento/colorante/opacificante y lecitina, que es objeto de esta solicitud. Las reivindicaciones no mencionan una característica que conceda un efecto diferente o especial a la composición, así que esta es obvia y evidente para una persona versada en la materia. Entonces debido a que: (a) la conformación de la composición de esta solicitud, a partir de las anterioridades, es evidente para una persona conocedora de la materia; porque siendo bien conocidas las composiciones que contienen PVA, talco, pigmento/colorante/opacificante, lecitina y agua, para una persona conocedora de la materia era evidente el elaborar la composición de esta solicitud de manera similar, teniendo en cuenta estos compuestos; (b) las reivindicaciones de esta solicitud no mencionan una característica que conceda un efecto diferente o especial a las composiciones, que pueda considerarse como un aporte a la técnica, porque las anterioridades ya enseñaban las composiciones que contienen PVA, talco, pigmento/colorante/opacificante, lecitina y agua requeridas en esta solicitud, así que el incluirlas de forma simultánea, era obvio para una persona versada en la materia y (c) el problema planteado en esta solicitud era la necesidad de proporcionar un revestimiento para tabletas farmacéuticas, suplementos nutricionales, alimentos, confituras, semillas agrícolas, revestimiento que es una película de rápida disolución y tiene una superficie lisa, apariencia brillante, pegajosidad mínima, buena adhesión de película y buena resistencia a la tensión; y puesto que para resolver este problema técnico la solicitud proporciona una composición que contiene PVA, talco, pigmento/colorante/opacificante, lecitina y agua gabapentina y pregabalina, es decir de manera similar a como se resolvía en las anterioridades; entonces esta solicitud no tiene **nivel inventivo**.

5. Conclusión

En conclusión, los requisitos de patentabilidad de la presente solicitud están afectados así:

| Item | Documento    | Reivindicaciones afectadas | Requisito que afecta       |
|------|--------------|----------------------------|----------------------------|
| D1   | US 5 885 617 | 1 - 44<br>45 - 66          | Nivel inventivo<br>Novedad |

165 162

|    |              |                   |                    |
|----|--------------|-------------------|--------------------|
| D2 | US 4 432 965 | 1 - 44            | Nivel inventivo    |
| D3 | US 3 935 326 | 1 - 44<br>45 - 66 | Novedad<br>Novedad |
| D4 | US 4 665 648 | 1 - 44            | Novedad            |
| D5 | US 3 554 767 | 1 - 44<br>45 - 66 | Novedad<br>Novedad |

En cumplimiento del artículo 45 de la Decisión 486 de la Comisión de la Comunidad Andina, se le indica que debe dar respuesta dentro del término de sesenta días contados a partir de la fecha de la notificación de la presente comunicación. En caso de no dar respuesta al presente requerimiento o si a pesar de la respuesta subsistieran los impedimentos para la concesión, se denegará la patente.

Notifíquese el presente oficio conforme a lo dispuesto en el numeral 5.2, literal e), del capítulo quinto del título primero de la Circular Unica No. 10 de 2001.

  
**ALIX CARMENZA CÉSPEDES DE VERGEL**  
Jefe de la División de Nuevas Creaciones.

El anterior requerimiento fue notificado por fijación en lista, de fecha  
21 JUL. 2005.

|                             |                                     |
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