United States Patent Office 2,716,614 Patented Aug. 30, 1955

2,716,614

VISCOSE COMPOSITION AND REGENERATED CELLULOSE BANDS

William Grogan O'Connell, Buffalo, N. Y., assignor to E. I. du Pont de Nemours and Company, Wilmington, Del., a corporation of Delaware

No Drawing. Application September 28, 1951, Serial No. 248,885

8 Claims. (Cl. 106—164)

This invention relates to shrinkable secondary container closures for rendering bottles, jars, etc. attractive and tamper-resistant. More particularly, this invention relates to shrinkable secondary container closures having the appearance of metal foil.

Heretofore, it has been customary to enclose the upper part of the neck and the primary closure of bottles of many types of alcoholic liquors, particularly wines, in a thin, decorative metal capsule or a wrapping of metal foil. This practice has also been adopted for a great deal of the bottled non-alcoholic beverages for the primary purpose of sales appeal. However, the metal foil secondary closure has many disadvantages, such as poor fit, which detracts from the appearance, lack of adhesion, which allows it to slip off the bottle neck before the time of willful removal, etc. To overcome these disadvantages, it has been proposed to substitute for the metal foil, shrinkable, non-fibrous bands of regenerated cellulose.

Shrinkable, non-fibrous secondary closures, i. e., bands and caps, of regenerated cellulose, have been known and used extensively for a number of years. They provide an attractive decoration for the container, a deterrent against tampering with the contents thereof, and, when pigmented, an effective background for printing advertising matter.

To seal and/or decorate a bottle, the shrinkable band in gel form is placed over the bottle top (the primary closure, such as a stopper or cap, being in place) and allowed to dry. The evaporation of the water from the band causes it to shrink and fit very firmly around the bottle neck and primary closure to form an attractive, tightly-stretched, tough seal. Although this type of secondary closure may be produced by various procedures, the great bulk of present-day production is manufactured by cutting short sections from continuous lengths of cellulose tubing regenerated from viscose. The manufacture of regenerated cellulose tubing from viscose and its subsequent cutting into short lengths (bands) is described by Webster in U. S. P. 1,647,489, by Vautier and Fays in U. S. P. 2,141,776, and by Dalton in U. S. P. 2,161,561.

An object of this invention is to provide a viscose composition containing wet-ground, heat treated mica and a shrinkable secondary closure of regenerated cellulose which closely resembles metal foil in appearance made therefrom, but which retains all of the advantages inherent in regenerated cellulose shrinkable secondary closures. More specific objects are to provide bands of regenerated cellulose having the appearance of gold, and to provide bands of regenerated cellulose having the appearance of silver. These and other objects will more clearly appear hereinafter.

The objects hereinabove stated are accomplished by the present invention which, briefly stated, comprises incorporating in viscose from which the regenerated cellulose secondary closure is to be formed, from 20 to 65% by weight of finely-divided, heat-treated mica; from 0 to 4% of stainless steel flake; and from 0 to 12% of coloring agent, all based on the weight of the cellulose in the viscose, converting the viscose to gel regenerated cellulose structures of the desired shape, and impregnating the regenerated cellulose shaped structure with an aqueous softener bath containing not more than 10% softener consisting of 3.5 to 8% glycerol and from 0 to 5.0% urea, the ratio of glycerol to urea being not less than 1.6 to 1.

By "heat-treated" mica is meant mica, preferably wet-ground, which has been heated at the temperature at which the pieces or particles adhere together slightly to form a loosely-held, porous cake which can be broken into small chunks by hand.

It is known to heat-treat transparent mica to produce an opaque product of enhanced light reflectivity by heating the natural transparent mica (which contains water tightly bound in the crystal lattice structure thereof) to the temperature at which the mica pieces or particles cake together slightly. The temperature at which this occurs will, of course, be different for each type of mica. Thus, muscovite, which is the most readily available type of mica, cakes together loosely when heated at a temperature of 1710°±10° F. for 12 hours, or at 1875° F. for ten minutes. All types of mica are susceptible to this heat-treatment; and all types of mica with a laminar structure, except those which have a high iron content, are suitable for purposes of this invention. Suitable types include, in addition to muscovite, phlogopite and sericite. A high iron content, such as is found in biotite, causes the mica to turn dark on heating and, in most instances, renders it unsatisfactory for use in bands, caps, etc.

To obtain satisfactory opacity and reflectivity, the mica must be wet-ground either before or after heat-treatment, preferably in slurry form in a ball or pebble mill. Other wet-grinding procedures such as those used in the mica industry can also be used. Wet-grinding insures that the mica particles will be in the form of platelets which tend toward planar orientation in the extruded band. When wet-grinding is performed before the heat-treatment, the loosely-held, porous cake of heat-treated mica may be broken down into the requisite platelet form and particles size by either wet- or dry-grinding.

Preferably, the size of the heat-treated mica particles (i. e., platelets) should be such that at least 80% of the particels are between 20 and 100 microns measured in their longest dimension. Particle sizes above 100 microns cause low opacity coverage per unit of mica weight, and sizes below 20 microns tend to dull off the band luster. From 20% to 65% mica is used, based on the weight of the cellulose in the viscose. Heat-treated, wet-ground micas are preferred to dry-ground micas.

The absolute opacity of the bands of this invention is improved by the addition of opacifiers such as barium chloride, barium titanate, titanium dioxide, stainless steel flake, etc. A small amount of stainless steel flake adds both to the opacity and to the luster. From 0% to 4% stainless steel flake may be used and/or from 0% to 10% of other opacifiers, based on the cellulose content of the viscose. Bands having a beautiful silver-like appearance may be made from viscose containing 40% heat-treated, wet-ground mica and 0.5 to 2% stainless steel flake. The flake is made from a standard stainless steel alloy containing from 17% to 28% by weight of chromium and from 8% to 16% by weight of nickel, based on the weight of the alloy, with the flake being of such a particle size that substantially all of the flake will pass through a 140 mesh screen and at least 55% of the flake will pass through a 325 mesh screen. A 140 mesh screen has a square screen opening of 105 microns; a 325 mesh screen has a square screen opening of 44 microns.

Bands not containing opacifiers in addition to the heat-treated mica, in some instances, exhibit a pronounced trans-

lucency, especially in the case of bands on brown bottles. The lack of apparent opacity of such bands is caused by a film of softener making an unbroken interface between the band and the bottle. This detrimental translucent effect can be eliminated by using a specific softener combination comprising 3.5% to 8% glycerol (solution concentration) with the various usual preservatives, and from 0% to 5% urea, the total concentration of softener bath not exceeding 10%, and the ratio of glycerol to urea being not less than 1.6 to 1.0.

The examples set forth in the following table further illustrate the practice of this invention. The coloring agents are identified by their official Color Index numbers as given in Rowe's Color Index—1924, or by the numbers of the patents which cover them. Parts are by weight unless otherwise stated:

Ex.	Percent Ingredients in Viscose Based on Cellulose Content			Coloring Agent, Percent	Finished Dried Band		
	H. T. W. G.* Mica, Percent	Stainless Steel Flake, Percent	Other Opacifier		Luster	Color	Absolute Opacity
1	40		2.4% Barium Chloride	3.0 (A)	Fair to Good	Gold	Good.
2	40	1.0	do	2.0 (B)	Excellent	do	Do.
3	40		do	7 (C) + 0.375 (D)	Good	do	Do.
4	35	2.0	do	2.7 (B) + 2.5 (C)	Excellent	do	Do.
5	40	2.0	do		do	Silver	Do.
6	40	1.0	do	5.0 (E)	do	Gold	Do.
7	35	2.4	do	3.5 (B) + 2.5 (C)	do	do	Do.
8	40	1.0	do	3.0 (B) + 1.0 (E)	do	do	Do.
9	40	1.0	do	3.0 (B) + 1.0 (F)	Good	do	Do.
10	40	1.0	3.0% Barium Titanate	1.75 (C) + 1.75 (G)	do	do	Good to Excellent.

*Wet-ground mica (Muscovite) heat-treated at 1710°±10° F. for 12 hours.
(A) Example 10 of U. S. P. 2,559,669 to Schroeder et al.
(B) Color Index Number 1132.
(C) Example 1 of U. S. P. 1,090,621 to Isler.
(D) Color Index Number 1120.
(E) Example 2 of U. S. P. 1,988,205 to Kunz et al.
(F) Color Index Number 1096.
(G) Example 1 of U. S. P. 1,667,848 to Gassner.

The viscoses of this invention illustrated in the foregoing examples may be used in making any of the various known types of bands. Thus, they may be used to make solid opaque bands or bands with opposed transparent stripes such as the window type band of Vautier and Fays in United States Patent No. 2,141,776. They may be used as an outer layer in bands similar to those described in Haff et al., United States Patent No. 2,331,974. They may also be used as an under-layer in making bands similar to those described by Czapek in United States Patent No. 1,918,383.

I claim:

1. An ornamental regenerated cellulose pellicular structure comprised essentially of regenerated cellulose pigmented with from 20% to 65% by weight, based on the weight of cellulose, of particles of wet-ground heat-treated mica, 80% of said particles measuring between 20 and 100 microns, and up to 4% by weight of stainless steel flake.

2. Regenerated cellulose secondary band closures having the appearance of gold, containing 40% by weight, based on the weight of cellulose, of particles of wet-ground heat-treated mica, 80% of said particles measuring between 20 and 100 microns, 2% stainless steel flake, and 3% to 10% of a yellow vat dye.

3. Regenerated cellulose secondary band closures having the appearance of silver, containing 40% by weight, based on the weight of cellulose, of particles of wet-ground, heat-treated mica, 80% of said particles measuring between 20 and 100 microns, and from 0.5% to 2% of stainless steel flake of such particle size that substantially all of the flake will pass through a 140 mesh screen and at least 55% of the flake will pass through a 325 mesh screen.

4. Regenerated cellulose secondary band closures for bottles and the like containing 20% to 65% by weight, based on the weight of cellulose, of particles of wet-ground heat-treated mica, 80% of said particles measuring between 20 and 100 microns, and up to 4% by weight of stainless steel flake, said bands having been softened in a softener bath containing not over 10% softener consisting of from 3.5% to 8% of glycerol and up to 5.0% of urea, the ratio of glycerol to urea being not less than 1.6 to 1.

5. Gel regenerated cellulose shrinkable bands pigmented with from 20% to 65% by weight, based on the weight of cellulose, of silvery-white particles of wet-ground, heat-treated mica, 80% of said particles measuring between 20 and 100 microns, and up to 4% by weight of stainless steel flake and impregnated with a softener bath containing from 3.5% to 10% of glycerol.

6. Viscose containing from 20% to 65% by weight, based on the weight of cellulose in the viscose, of particles of wet-ground, heat-treated mica, 80% of said particles measuring between 20 and 100 microns, and up to 4% by weight of stainless steel flake.

7. Viscose containing 40% by weight, based on the weight of cellulose in the viscose, of particles of wet-ground, heat-treated mica, 80% of said particles measuring between 20 and 100 microns, and from 0.5% to 2% of stainless steel flake of such particle size that substantially all of the flake will pass through a 140 mesh screen and at least 55% of the flake will pass through a 325 mesh screen.

8. Viscose containing 40% by weight, based on the weight of cellulose, of particles of wet-ground, heat-treated mica, 80% of said particles measuring between 20 and 100 microns, 2% stainless steel flake, and 3% to 10% of a yellow vat dye.

References Cited in the file of this patent

UNITED STATES PATENTS

109,416	Johnston et al.	Nov. 22, 1870
1,045,990	Lindsay	Dec. 3, 1912
2,013,739	Wright	Sept. 10, 1935
2,204,063	Atwood	June 11, 1940
2,307,760	Creadlick	Jan. 12, 1943

FOREIGN PATENTS

983,563	France	June 25, 1951
12,543	Australia	of 1933
244,540	Switzerland	Apr. 16, 1947