United States Patent Office 2,885,341
Patented May 5, 1959

2,885,341

COATING COMPOSITION COMPRISING PARAFFIN WAX, MICROCRYSTALLINE WAX AND PETROLEUM DISTILLATE OIL

John D. Tench, Prospect Park, Pa., and Evan B. Ewan, Swedesboro, N.J., assignors to Sun Oil Company, Philadelphia, Pa., a corporation of New Jersey

No Drawing. Application July 16, 1957
Serial No. 672,120

2 Claims. (Cl. 208—21)

This invention relates to a novel wax composition. More particularly, the invention relates to a novel composition containing a major proportion of a specific paraffin wax and minor proportions of a specific microcrystalline wax and a specific distillate oil, the composition being especially suitable for coating fibrous sheets and containers for packaging.

United States Patent Number 2,624,501 describes and claims a paraffin wax having specific properties which is especially suitable for coating fibrous sheet materials and containers such as milk containers. The claimed paraffin wax has a melting point of from 125° F. to 132° F. and a tensile strength at 40° F. of from 275 p.s.i. to 325 p.s.i. (pounds per square inch), the tensile strength at 40° F. being at least 25 p.s.i. higher than the tensile strength at 70° F. An advantage stated for this wax is that it is unnecessary to compound therewith foreign materials in order to obtain good coatings with the wax. Actual commercial use, however, disclosed certain disadvantages of coatings formed from this wax. Such coatings on fibrous sheet materials may develop minute imperfections which impart a serpentine effect to the coating thereby creating an unsightly appearance, and leakage of packaged liquid materials through the imperfections may develop. Another disadvantage of the wax is that it must be packaged for shipment in small lots to prevent blocking of the slabs of wax, i.e., to prevent fusion of stacked wax slabs into a large, single mass, especially when the stacked wax slabs are exposed to mildly elevated temperatures, such as temperatures from about 80° F. to 90° F.

United States patent application Serial Number 504,366, filed April 27, 1955, by John D. Tench, a coinventor of the present invention is directed to a wax composition which overcomes the serpentine effect, and wax slabs prepared from the described composition do not block on stacking. These advantageous results are obtained by incorporating about 2% by weight of a specific microcrystalline wax in the paraffin wax of said United States Patent Number 2,624,501. While the described wax composition is good for many applications, such as for coating milk containers for packaging milk at 40° F., commercial use has disclosed that it suffers from certain difficulties. The primary difficulty observed with the wax composition is that coatings thereof on packaging materials crack when contacted with relatively cold materials, such as milk at a temperature of 36° F. For example, milk containers coated with this wax composition, when filled with milk at a temperature of 36° F., exhibit cracking of the wax coating. "Cracking" is distinguished from the serpentine effect in that cracking is an actual break in the wax coating which almost always results in leakage of the container.

An object of the present invention is to provide a wax composition especially suitable for coating fibrous sheet materials such as fluid containers. Another object is to provide a wax composition having substantially the properties of the paraffin wax described in United States Patent Number 2,624,501, which when applied as a coating on fibrous materials, does not develop cracks when contacted with a cold, fluid material. A further object is to provide wax compositions which do not develop imperfections such as the serpentine effect. A still further object is to provide a wax composition which, when formed into wax slabs, does not block on stacking.

It has now been found that by incorporating small, critical amounts of a specific microcrystalline wax and of a specific petroleum distillate oil in the paraffin wax of said Patent No. 2,624,501, the properties of the resulting wax composition are substantially the same as those of the paraffin wax, but the resulting wax composition does not crack or develop a serpentine effect when used as a coating on fibrous materials, and wax slabs formed from the wax composition do not block when the slabs are in stacked relationship.

It is essential to the successful preparation of the composition of the invention that the microcrystalline wax have specific properties which are described in said Serial Number 504,366. The microcrystalline wax must have a melting point of from 185° F. to 200° F. (ASTM D127–49), a viscosity of from 81 to 89 SUS at 210° F. (D446–53), a refractive index (n_D^{212}) of 1.435 to 1.438, a penetration of from 30 to 65 at 160° F. (ASTM D5–52), and a penetration of not above 5 at 77° F. (ASTM D5–52). The molecular weight of the microcrystalline wax is approximately 650. A microcrystalline wax which does not have properties within the stated ranges does not give a satisfactory wax composition when incorporated in the paraffin wax of Patent No. 2,624,501.

The microcrystalline wax of the invention is prepared from petroleum. Slack wax from the dewaxing of lubricating oil is subjected to vacuum distillation and the material boiling below about 490° F., and preferably below 525° F., at 2 mm. of mercury pressure is separated. The residual fraction is used to prepare the microcrystalline wax of the present wax composition. The residual fraction is dissolved in a solvent, such as a mixture of methyl ethyl ketone and benzene, at an elevated temperature. The wax solution is then cooled to a temperature of from about 95° F. to 110° F. and the wax precipitated at this temperature separated such as by filtering. The resulting wax is the microcrystalline wax used in preparing the present composition. It will be understood that other solvents such as ethylene dichloride, toluene, and the like, may be employed for the dissolution of the wax at an elevated temperature. Substitution of such materials in whole or in part for the benzene and/or the methyl ethyl ketone may require an adjustment of the temperature of precipitation, it being necessary that the final wax product have the properties as herein described. It will also be understood that the microcrystalline wax separated such as by filtering is advantageously washed with an additional quantity of solvent, preferably used at the temperature of the precipitation, and that any excessive solvent is removed from the wax cake by means such as evaporation.

The preparation of the paraffin wax constituent of the present wax compositions is advantageously accomplished as described by Patent No. 2,624,501. Briefly, a slack wax obtained as a by-product from the dewaxing of lubricating oils, and which contains about 30% oil, is subjected to vacuum distillation and a distillate fraction boiling between about 325° F. and 650° F. at 10 mm. of mercury pressure is separated. This fraction is dissolved in a solvent, preferably a mixture of methyl ethyl ketone and benzene in about equal volumes, dissolution being at a temperature of from about 165° F. to 195° F. The solution is slowly cooled to a temperature of from 75° F. to 80° F., and the precipitated wax separated, such as by filtering or centrifuging. The re-

sulting filtrate is further cooled to a temperature of from 25° F. to 30° F., and the precipitated wax filtered. This latter filtered wax, on further treatment, forms the paraffin wax component of the wax composition of the present invention. It is preferred to wash the wax cake, either by washing the filter cake or by repulping and subsequent filtration, using as the washing material the solvent employed in the dissolution of the wax. Also, the washing operation is preferably conducted at a temperature approximating the temperature of the final precipitation.

It is essential to the preparation of the composition of the invention that the petroleum distillate oil employed in the subject composition have specific properties. The oil must have an API gravity at 60° F. of from 27.5 to 33, a viscosity at 100° F. of 100 to 650 SUS (Saybolt Universal seconds) and a pour point of 0° F. maximum. Preferably a solvent refined petroleum distillate oil having the foregoing properties is employed. The zero pour point limitation is necessary in order to prevent the introduction of relatively soft waxes or soft, wax-like materials which, if present, would deleteriously affect the properties of the present composition.

It is essential that the concentration of the three components of the subject wax composition be within certain limits. The paraffin wax component must be present within the range of 95.5% to 96.7%. The microcrystalline wax must be present within the range of from 3% to 4%. The distillate oil must be present within the range of from 0.3% to 0.5%. All quantities are herein expressed as percent by weight unless otherwise designated. It will be noted that the quantity of microcrystalline wax is substantially above that shown to be operable in the compositions of United States Serial Number 504,366.

The incorporation of the microcrystalline wax and paraffin wax can be by any convenient means, such as by blending the waxes in the mloten state, together with the oil, to obtain a homogeneous blend.

The following examples illustrate the compositions of the invention and demonstrate the criticality of the concentration of the several components. In the examples, a "dish test" is used to demonstrate the suitability or nonsuitability of wax compositions for coating containers, such as milk containers. In accordance with this test, a definite amount of the wax composition being tested is introduced into a glass petri dish and is placed in an oven maintained at a specified temperature for a specified minimum time. Thereafter, the dish is cooled at a constant temperature for 30 minutes and then immersed in cold water for 20 seconds. The dish containing the wax coating is then visually inspected and rated. In performing the dish test, the wax composition is heated to about 200° F. and 10±0.1 grams introduced into at least two petri dishes. The petri dishes are of Pyrex and have an outside diameter of 100 mm. The dishes are then placed in an oven maintained at 200° F. to 210° F. for at least 15 minutes. The dishes are then removed from the oven and are cooled at 74±4° F. for 30 minutes. During cooling the bottom of the dish should rest on an insulating material such as asbestos. The dishes and wax are then completely immersed in water at a temperature of 36±0.5° F. for 20 seconds. On removal from the water, the wax coatings are visually examined for cracking. On the rating scales, zero denotes no cracking, and 4 denotes severe cracking. A zero+ rating is used to indicate very slight cracking. A composition which exhibits a rating of 1 or above does not pass the test. The microcrystalline wax employed in the examples was prepared by vacuum distilling slack wax from the dewaxing of lubricating oil to remove the components thereof boiling below about 525° F. at 2 mm. of mercury pressure. The residual fraction was dissolved in a solvent, consisting of a mixture of methyl ethyl ketone in about equal parts, at an elevated temperature. The resulting solution was then cooled to a temperature of about 105° F. and the wax precipitated at this temperature separated by filtration. The wax cake was washed with additional solvent and solvent removed from the washed cake by evaporation.

The resulting microcrystalline wax had a melting point of about 192° F., a viscosity of about 86 SUS at 210° F., a penetration of about 47 at 160° F. and about 3 at 77° F., a refractive index (n_D^{212}) of about 1.4363, and a molecular weight of about 650.

The paraffin wax was prepared as described in Patent Number 2,624,501, and had a melting point of about 127° F., a tensile strength of about 250 at 70° F. and of about 280 at 40° F.

The oil employed was a distillate solvent refined oil having an API gravity (60° F.) of from 31 to 33, a viscosity at 100° F. of about 110 SUS and a pour point of 0° F. maximum. The compositions of the examples were prepared by melting the paraffin wax and blending therewith a microcrystalline wax and/or the distillate oil.

In preparing the compositions of the examples, the described components are blended at a temperature above the melting points of the waxes employed so that, on cooling, a homogeneous blend is obtained.

Example 1

In order to demonstrate the necessity of maintaining the concentration of the components of the present composition within the described limits, various compositions were prepared and tested by the dish test. The compositions used and the ratings thereof are shown in the following table:

Composition	Paraffin (percent)	Microcrystalline (percent)	Oil (percent)	Rating (Dish Test)
1	100			2
2	98	2		4
3	97	2	1	3
4	97	3		1
5	97.5	2	0.5	3
6	96.9	3	0.1	2
7	96.8	3	0.2	1
8	96.7	3	0.3	0
9	96.5	3	0.5	0
10	97.5	2	0.5	1

Only compositions 8 and 9, which illustrate compositions according to the invention, passed the dish test.

Example 2

A composition containing 9% paraffin wax, 3.5% microcrystalline wax and 0.5% oil, each of said materials having the properties as above described, was prepared and used to coat a cardboard milk carton. The coated milk carton was filled with milk at 36° F. No cracking of the wax coating or leaking of the container was observed.

Another composition was prepared by blending 98% of the paraffin wax with 2% of the microcrystalline wax. The resulting composition was used to coat a cardboard milk container. On filling the coated container with milk at 36° F., cracks developed in the wax coating covering the bottom of the container and severe leakage of the milk through the carton was observed.

When compositions containing more than 4% of the microcrystalline wax are used, the wax coating is uneven and furrowed, and hence such compositions are unsuitable for coating milk containers in that customer resistance is experienced and leakage, especially at the furrows, may develop. Also, when the compositions containing more than 0.5% oil are used, blocking is observed even at relatively low temperatures, e.g., at about 70° F., and hence such compositions are unsuitable in accordance with the present invention.

Example 3

The properties of a composition containing 96.7% paraffin wax, 3% microcrystalline wax and 0.3% distillate oil is compared to the properties of the paraffin wax alone in the following table:

	Paraffin Wax	Wax-Composition
Melting point (ASTM D87-42)	127	128
Viscosity at 210 F. (ASTM D446-53)	39.0	39.2
Refractive Index (n_D^{212})	1.4218	1.4222
Penetration (ASTM D 5-52):		
at 77° F	20	15
at 100° F	105	85
Flash (°F.) (ASTM D92-52)	430	420

As shown by the table, the physical properties of the wax composition of the invention are substantially the same as of the paraffin wax.

On storing wax slabs prepared from the compositions of the present invention for long periods of time under ambient temperature conditions, which includes a temperature of about 90° F., no blocking of the slabs is observed.

The invention claimed is:

1. A wax composition consisting essentially of a hydrocarbon paraffin wax derived from petroleum and having a melting point of 125° F. to 132° F., a tensile strength at 40° F. of from 275 to 325 p.s.i., the tensile strength at 40° F. being at least 25 p.s.i. higher than the tensile strength at 70° F., from about 3% to 4% by weight of a microcrystalline wax derived from petroleum and having a melting point of from 185° F. to 200° F., a viscosity of from 81 to 89 SUS at 210° F., a penetration of from 30 to 65 at 160° F., and a penetration of not above 5 at 77° F., and from about 0.3 to 0.5% by weight of a petroleum distillate oil having an API gravity at 60° F. of from 27.5 to 33, a viscosity at 100° F. of from 100 to 650 SUS and a pour point of 0° F. maximum.

2. Wax composition according to claim 1 wherein said distillate oil is a solvent refined distillate oil.

References Cited in the file of this patent

UNITED STATES PATENTS

2,361,582	Adams et al.	Oct. 31, 1944
2,624,501	Ferris	Jan. 6, 1953
2,744,031	Mumma	May 1, 1956
2,773,812	Tench	Dec. 11, 1956
2,783,161	Padgett	Feb. 26, 1957
2,816,845	Capell et al.	Dec. 7, 1957