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COATING COMPOSITION COMPRISING PARAFFIN WAX AND MICROCRYSTALLINE WAX

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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 a minor proportion of a specific microcrystalline wax, the composition being especially suitable for coating fibrous sheets and containers for packaging.

U.S. Patent No. 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., 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, has 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.

The development of the serpentine effect can be partially controlled by close regulation of the temperature of the wax at the time of coating the fibrous sheet, the time used for coating this sheet, e.g., the time of immersion of the fibrous sheet material in a molten wax bath, the rate and means of cooling of the resulting coated product, the type of fibrous sheet material coated, and the design of the coating equipment. Close regulation of these variables, however, in commercial useage is not practical. The blocking of wax slabs can be prevented only by packaging the wax slabs in small lots, and by providing a barrier, such as a paper sheet, between the wax slabs. This, however, requires individual handling and hence is inexpensive both in packaging the wax and in its subsequent use.

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 said Patent No. 2,624,501 which, when applied as a coating on fibrous materials, does not develop minute imperfections imparting a serpentine effect to the coating. A further object is to provide a wax composition having substantially the properties of the paraffin wax of said Patent No. 2,624,501 which does not block when slabs formed therefrom are stacked.

It has now been found that by incorporating about 2% by weight of a specific microcrystalline wax, as described hereinafter, in the paraffin wax of said Patent No. 2,624,501, the properties of the resulting wax composition are substantially the same as the properties of the paraffin wax, the resulting wax composition does not develop a serpentine effect when used for coating fibrous materials, and the wax composition does not block when slabs thereof are in stacked relationship.

It is essential to the successful preparation of the composition of the invention that the microcrystalline wax employed have specific properties. 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 de-waxing 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 resulting 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.

As above stated, it is essential that an amount of micro-

crystalline wax of about 2% by weight be incorporated in the paraffin wax. The amount of microcrystalline wax incorporated must always be within the range of from 1.4% to 2.6% by weight. A composition having components outside of this range does not give satisfactory results as shown hereinafter by example.

The incorporation of the microcrystalline wax in the paraffin wax may be by any convenient means such as by admixing the waxes in the molten state to obtain a homogeneous blend. The incorporation of the required quantity of microcrystalline wax in the paraffin wax does not deleteriously affect or even substantially change most of the properties, such as melting point and flexibility, of the paraffin wax which render it especially suitable for coating fibrous materials. However, the tensile strength is somewhat increased thereby enhancing the value of the wax compositions for coating purposes. Generally the tensile strength at 40° F. will be from about 350 to 450 p.s.i., and the tensile strength at 40° F. will be at least 25 p.s.i. higher than the value obtained at 70° F.

The following examples illustrate compositions of the invention:

Example 1

A wax composition according to the invention was prepared by incorporating 2% by weight of a microcrystalline wax in a paraffin wax. The microcrystalline wax 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 No. 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 composition was prepared by adding 2% by weight of the microcrystalline wax, in the molten state, to the paraffin wax, also in the molten state, with agitation to secure a homogeneous blend on cooling.

The properties of the paraffin wax and of the wax composition of the invention were as follows:

	Paraffin wax	Wax composition
Melting point (ASTM D87-42)	127	128
Viscosity at 210° F (ASTM D446-53)	39.0	39.1
Refractive index (n_D^{212})	1.4218	1.4221
Penetration (ASTM D5-52):		
at 77° F	20	15
at 90° F	44	44
Tensile strength (40° F.)	280	430
Tensile strength (70° F.)	250	290
Flash (° F.) (ASTM D92-52)	430	420

As shown by the data, the physical properties of the wax composition of the invention are substantially the same as of the paraffin wax, except that the tensile strength is increased.

Example 2

The paraffin wax component of the composition of the invention and compositions prepared by incorporating various amounts of the microcrystalline wax of Example 1 therein, when used to coat fibrous sheet materials, such as milk container cartons, and when slabs thereof are stacked, give the following results:

Paraffin wax—gives serpentine effect and blocks.
Paraffin wax +½% microcrystalline wax—gives serpentine effect and blocks.
Paraffin wax +1% microcrystalline wax—blocks.
Paraffin wax +2% microcrystalline wax—does not give serpentine effect or block.
Paraffin wax +3% microcrystalline wax—wax coating flakes from the fibrous sheet.

In this example, 175 wax slabs, each about 19 inches by 12 inches by 1⅝ inches, having a total weight of about 1750 pounds, are stacked to form substantially a cube. The slabs formed from the wax composition having 2% by weight microcrystalline wax did not block even when stored for long times at ambient temperature, whereas wax compositions having less than about 2% by weight, and specifically less than about 1.4% by weight of the microcrystalline wax, when so stacked fuse into what is essentially a single wax mass.

Example 3

Wax compositions similar to the wax compositions of the invention were prepared in which microcrystalline waxes other than the required microcrystalline wax of the invention were used.

A wax composition was prepared consisting of the paraffin wax of Example 1 and 2% by weight of a microcrystalline wax having a melting point of 178° F., a penetration of 12 at 77° F., and a viscosity of 73 SUS at 210° F., the test methods being as herein designated for the microcrystalline wax of the invention. Wax slabs prepared from this composition could not be stacked, as described in Example 2, without blocking, i.e., without adhesion of the slabs to each other.

Another wax composition was prepared consisting of the paraffin wax of Example 1 and 2% by weight of a microcrystalline wax having a melting point of 140° F., a penetration of 22 at 77° F., and a viscosity of 87 SUS at 210° F., the test methods being as herein designated for the microcrystalline wax of the invention. Wax slabs prepared from this composition could not be stacked, as described in Example 2, without blocking.

As shown by the foregoing examples, the properties of the microcrystalline wax component of the wax composition of the invention must be within the limits herein described, and the quantity of this wax incorporated in the paraffin wax must be about 2% by weight. When microcrystalline waxes having properties outside of the required limits are used, the resulting wax composition does not meet the requirements for the wax compositon of the invention, and when a quantity of the microcrystalline wax component of the wax composition of the invention is varied from about 2% by weight, as herein described, the resulting wax compositions do not meet the requirements for the wax composition of the invention.

The invention claimed is:

1. A wax composition consisting essentially of a hydrocarbon paraffin wax derived from petroleum and having a melting point of from 125° F. to 132° F., a tensile strength at 40° F. of from 275 p.s.i. 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., and from 1.4% to 2.6% 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 refractive index (n_D^{212}) of from 1.435 to 1.438, a penetration of from 30 to 65 at 160° F., and a penetration of not above 5 at 77° F.

2. A wax composition according to claim 1, said wax composition having a melting point of from 125° F. to 132° F. and a tensile strength at 40° F. of from 350

to 450 p.s.i., the tensile strength at 40° F. being at least 25 p.s.i. higher than the tensile strength at 70° F.

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