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WAX COMPOSITION COMPRISING A MIXTURE OF SPECIFIC PARAFFIN WAXES AND 1-2% OF A MICROCRYSTALLINE WAX

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1 Claim. (Cl. 208—21)

This invention relates to a new wax composition especially suitable for coating fibrous containers and sheet materials for packaging foods and other articles.

Paraffin waxes have heretofore been employed for coating paper containers and fibrous sheet materials designed to package foods such as cereals, frozen foods, and the like, but such waxes suffer many shortcomings. In order to overcome some of the difficulties, microcrystalline waxes and polymers, such as polyethylene, have been incorporated therewith. Such compositions, however, have also been unsatisfactory in one or more of the following: in presenting an unsightly appearance, especially in exhibiting poor gloss retention, i.e., in losing gloss on storage, in obtaining poor coverage with subsequent softening when filled with liquids, and in blocking at relatively low temperatures, and the like. An especially serious drawback of compositions of paraffin wax and microcrystalline wax is poor gloss retention. For example, although some properties of paraffin wax such as blocking characteristics are improved by incorporating a small amount of microcrystalline wax therein, such incorporation destroys the gloss retention of coatings formed therefrom. Hence, packaged materials coated with such a composition meets with substantial consumer resistance.

An object of the present invention is to provide a wax composition especially suitable for coating fibrous containers for fluids. A particular object is to provide a wax composition effective for coating paper containers for liquids and sheet materials for packaging articles such as foods which imparts a pleasing appearance to the resulting article of manufacture, while obtaining good coverage and good blocking characteristics. A further object is to provide a process for the preparation of such wax compositions. A specific object is to provide a wax composition which, when formed as a coating for a fibrous container or a sheet material, will not lose gloss on storage even for an extended period of time.

It has now been found that by blending two paraffin waxes having specific properties in specific proportions to produce a paraffin wax composition of specific properties, and incorporating therein a specific quantity of a microcrystalline wax having specific properties, a wax composition achieving the above and other objects is obtained.

As used herein, melting points of paraffin waxes are determined by ASTM D87–42 and of microcrystalline waxes by ASTM D127–49, penetrations by ASTM D5–52 and viscosities (SUS-Saybolt Universal Seconds) by ASTM D446–53, unless otherwise stated. By "gloss," as used herein, is meant the light reflecting ability of the coated material, which can be measured by a glossmeter of usual design or rated visually. A "good" rating shows high reflectivity, "poor" shows low reflectivity and an unacceptable product, and "fair" shows medium reflectivity of marginal acceptance. "Aged gloss" is the gloss determined after storage of the coated article for seven days at a temperature of 73±3° F. and a relative humidity of 50±5%. If the "aged gloss" rating is "good," the coated material can be expected to retain its gloss for a relatively long period of storage.

The first paraffin wax must have a melting point of from 131° F. to 136° F., a penetration (at 77° F.) of from 10 to 16 and a viscosity (at 210° F.) of from 35 to 41. On distillation at 2 mm. of mercury pressure, 5% should distill at about 407° F., 50% at about 445° F., and 95% at about 477° F. The paraffin wax meeting these specifications is prepared from slack wax which may be from the dewaxing of lubricating oil or from a topped, high wax content crude petroleum. The slack wax is vaccum distilled and the fraction distilling between about 390° F. and 475° F. at 2 mm. of mercury pressure is collected. This distillate 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 32° F. to 38° F. and the wax which precipitates at this temperature is separated as by filtration. The separated wax is advantageously washed with an additional amount of the solvent used, preferably at the temperature of precipitation, and the wax recovered from the solvent. The recovered wax is the first paraffin wax of the present compositions.

The second paraffin wax of the composition of the invention must have a melting point of from about 148° F. to 156° F., a penetration (at 77° F.) of from 9 to 15 and a viscosity (at 210° F.) of from 40 to 46 SUS. On distillation at 2 mm. of mercury pressure, 5% of the wax should distill at about 469° F., 50% at about 487° F., and 95% at about 556° F.

The second paraffin wax of the composition is prepared from a slack wax as above-described, such as by topping a high wax content crude oil and distilling the reduced wax crude under vacuum, and collecting the fraction distilling in the range of from about 450° F. to 565° F. at 2 mm. of mercury pressure. The distillate fraction is dissolved in a solvent which is preferably a mixture of methyl ethyl ketone and benzene in about equal parts by volume, dissolution being at a temperature of from about 165° F. to 190° F. The solution is slowly cooled to a temperature of from about 77° F. to 83° F. and the wax precipitated at this temperature is separated as by filtering. The separated wax is advantageously washed with an additional amount of the solvent used preferably at the temperature of precipitation, and the wax recovered from the solvent. The recovered wax forms the second paraffin wax of the present invention.

The microcrystalline wax of the present invention must have a melting point of from 190° F. to 196° F., (ASTM D127–49), a penetration (at 77° F.) of from 3 to 8, and a viscosity (at 210° F.) of from 80 to 90. This wax is advantageously prepared from a slack wax derived from the dewaxing of lubricating oil. The slack wax 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 composition. The residual fraction is dissolved in a solvent, such as a mixture of equal parts by volume 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 115° F. and the wax precipitated at this temperature separated as by filtering. The separated wax is advantageously washed with an additional quantity of the solvent used preferably at the temperature of precipitation, and the wax recovered from the solvent. The resulting wax is the microscrystalline wax used in preparing the present composition.

On combining the two paraffin waxes of the present invention, a paraffin wax composition is prepared having properties which must be within certain limits. The paraffin wax composition, i.e., the composition prepared by combining the two paraffin waxes in substantially the same quantities, are as follows: The melting point must be within the range of from 139° F. to 146° F. The penetration at 77° F. must be from 9 to 15 and at 100° F. must be from 20 to 26. The viscosity at 210° F. must be from 38 to 43. On distillation at 2 mm. of mercury pressure, 5% of the wax should be distilled at about 415° F., 50% at about 479° F. and 95% at about 542° F. This paraffin wax composition can be prepared only by combining paraffin waxes having the specific properties as above described. Other paraffin waxes having similar properties in some respects, as shown hereinafter by example, do not give equivalent results as obtained with the described wax composition. It is not necessary, however, to first blend the paraffin waxes and then incorporate the microcrystalline therein; the three components can be blended in any order or simultaneously.

As above stated, it is essential that the several components of the wax compositions of the invention be present within narrow ranges. The first paraffin wax, or lower melting paraffin wax, must be present in the range of from 45% to 54%; the second paraffin wax, or higher melting paraffin wax, must be present in a quantity of from 45% to 54%, and the microcrystalline wax must be present in a quantity of from 1% to 2%, the quantities being based on percent by weight of the final composition. It is preferred to employ substantially identical amounts of the two paraffin waxes in order to achieve best results in accordance with the present invention.

In order to demonstate a specific embodiment of the present invention and the advantages thereof, a first paraffin wax was prepared as follows: Slack wax was prepared by topping a high wax content crude petroleum to about 38% bottoms. The bottom fraction was distilled at a pressure of 2 mm. of mercury and the fraction distilling in the range of from about 390° F. to 475° F. was collected. The collected fraction was dissolved in a mixture of about equal parts by volume of methyl ethyl ketone and benzene at an elevated temperaure of about 170° F. The solution was slowly cooled to 35° F. and the precipitated wax was separated by filtering. The separated wax was washed with an additional quantity of the solvent and then was recovered from the solvent. The resulting wax had a melting point of 133° F., a viscosity (at 210° F.) of 37.9 and a penetration (at 77° F.) of 13.

A second paraffin wax component of the present composition was prepared from the same slack wax, namely the slack wax prepared by topping a high wax content crude to about 38% bottoms. The bottom fraction was distilled at 2 mm. of mercury pressure and the fraction distilling between 450° F. and 565° F. was collected. This fraction was dissolved in a solvent consisting of a mixture of about equal parts by volume of methyl ethyl ketone and benzene at a temperature of about 170° F. The resulting solution was slowly cooled to 80° F. and the wax precipitated at this temperature was separated by filtering. The separated wax was washed with an additional quantity of the same solvent at the temperature of precipitation, and then was recovered from the solvent. This second paraffin wax had a melting point of 151° F., and a viscosity (at 210° F.) of 43.6 and a penetration (at 77° F.) of 12.

The two paraffin waxes were blended in equal parts by weight. The resulting paraffin wax composition had a melting point of 142° F., a penetration (at 77° F.) of 14, and a viscosity (at 210° F.) of 40.5. On distillation at 2 mm. of mercury pressure, 5% of the wax distilled at 415° F., 50% at 479° F. and 95% at 542° F.

A microcrystalline wax was prepared by distilling a slack wax from the dewaxing of lubricating oil to remove as distillate all materials boiling below 525° F. at 2 mm. of mercury pressure. The bottom fraction was dissolved in a mixture of about equal parts by volume of methyl ethyl ketone and benzene at a temperature of about 190° F. The resulting solution was slowly cooled to 50° F. and the wax precipitated at this temperature was separated by filtration. The separated wax was redissolved in an additional quantity of the same solvent at a temperature of about 190° F. and the resulting solution was slowly cooled to 115° F. The wax precipitated at this temperature was separated by filtering. The separated wax was washed with an additional quantity of the same solvent at 115° F., and the solvent then was removed from the wax. The recovered wax had a melting point of 193° F., a penetration (at 77° F.) of 5 and a viscosity (at 210° F.) of 85. This microcrystalline wax can also be prepared by dissolving the residual fraction from the distillation fo slack wax in a solvent as above described, cooling the solution to 115° F. and recovering the precipitated wax. However, to improve the operation of the filters, it has been found advantageous to perform the separation in two steps as above described.

To the described blend of paraffin waxes was added 2% by weight of the microcrystalline wax prepared as above described. The mixture was heated to convert the wax to molten phase and the molten wax was stirred until a homogeneous composition was obtained. The initial gloss of the wax composition was good and the aged gloss likewise good, no deterioration being observed throughout the test period. In addition to a good appearance, good coverage was obtained and no blocking was observed even at temperatures as high as 110° F. The paraffin wax blend without the addition of the microcrystalline wax had only a fair initial gloss, and a very poor aged gloss.

To demonstrate the critical nature of the microcrystalline wax of the invention, to the same paraffin wax composition was added 2% by weight of a microcrystalline wax having a melting point of 153° F. This microcrystalline wax was prepared as above described except that the temperature of precipitation was lowered to give the lower melting wax. The initial gloss of the coated product was good, but the aged gloss was poor.

A further composition was prepared in which a microcrystalline wax having a melting point of 142° F. was used in a concentration of 2% by weight. Again the initial gloss was good, but the aged gloss was poor.

In order to demonstrate the critical nature of the components of the invention, a paraffin wax was prepared having properties within the limits described for the present blend of two paraffin waxes, but the preparation was by a different process. The paraffin wax was prepared by topping a high wax content crude to 38% bottoms. The bottoms were charged to a vacuum distillation operation and the fraction distilling between about 398° F. and 534° F. at 2 mm. of mercury pressure was collected. This fraction was dissolved in a solvent consisting of a mixture of benzene and methyl ethyl ketone in about equal parts by volume at a temperature of about 180° F., 4 parts by volume of the solvent per part of the wax being used. The resulting solution was slowly cooled to 60° F. and the wax precipitated at this temperature was separated, washed with an additional quantity of the solvent at a temperature of 60° F. and then recovered from the solvent. This paraffin wax had a melting point of 142° F., a viscosity (at 210° F.) of 40 SUS, and a penetration (at 77° F.) of 9. Into this wax was incorporated 2% by weight of the microcrystalline wax, as above described, by heating a mixture thereof and stirring the molten mass to obtain a homogeneous composition. Coatings were prepared from the resulting blend and from the paraffin wax. The coating formed from the paraffin wax ex-

hibited good gloss, both initial and aged, but was not suitable as a coating composition because of blocking even at relatively low temperatures. The coating formed from the composition exhibited poor aged gloss, thereby showing the critical nature of the paraffin waxes operable in the present invention.

The invention claimed is:

A wax composition consisting essentially of from 45% to 54% of a paraffin wax having a melting point of from 131° F. to 136° F., a penetration at 77° F. of from 10 to 16, and a viscosity at 210° F. of from 35 to 41 SUS, and from 45% to 54% of a paraffin wax having a melting point of from 148° F. to 156° F., a penetration at 77° F. of from 9 to 15, and a viscosity at 210° F. of from 40 to 46 SUS, and from 1% to 2% of a microcrystalline wax having a melting point of from 190° F. to 196° F., a penetration at 77° F. of from 3 to 8 and a viscosity at 210° F. of from 80 to 90 SUS.

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