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WAX COMPOSITION

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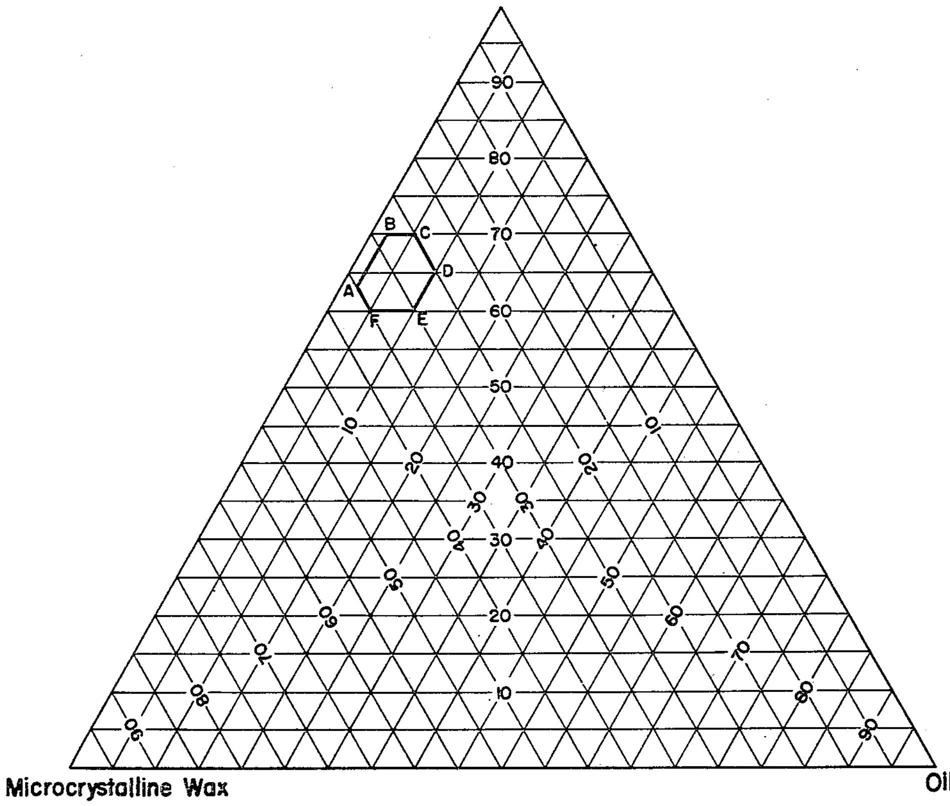

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WAX COMPOSITION

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This invention relates to a novel wax composition and more particularly relates to a solid composition containing paraffin wax, microcrystalline wax and oil, the composition being especially suitable for coating containers for packaging dairy products.

Paraffin waxes have heretofore been employed for a variety of uses, such as water-proofing textiles and papers. In order to obtain certain properties for such waxes, foreign materials such as microcrystalline wax, rubber, beeswax, resins, and the like, have been compounded therewith. Such compositions, however, have not proved entirely satisfactory for certain purposes. For example, waxes and wax compositions heretofore described as suitable for coating containers for dairy products are not suitable for certain applications such as coating containers designed to package cottage cheese. It appears that one or more of the components of cottage cheese, in some manner, passes through the wax barrier so that there after fluids of the cheese composition can pass through the wax coating. The fluids are then absorbed by the fibrous walls of the container, which becomes soggy. The so-spoiled container meets considerable consumer resistance, and in some instances leaks develop in the container. The provision of a suitable wax for coating such compositions is complicated by the requirement that the waxed-coated containers, when stacked in nesting relation, must not stick together at temperatures ordinarily encountered during storage and shipment, i. e., the blocking temperature of the wax must be relatively high.

An object of the present invention is to provide a novel wax composition, made from petroleum hydrocarbons, which is especially suitable for coating containers. Another object is to provide containers for packaging dairy products which will not stick together when stored in contact at temperatures of up to 100° F., and preferably will not stick when stored in contact at temperatures up to 105° F. A further specific object is to provide a container for packaging cottage cheese.

It has now been discovered that by compounding three separate components prepared from petroleum, all of which have specific properties within narrow ranges, the resulting composition is especially suitable for coating fibrous containers, and particularly fibrous containers for packaging cottage cheese.

The three components that are combined to produce the composition of the invention are a paraffin wax, a microcrystalline wax and a lubricating oil, each of which has specific properties within narrow ranges, as hereinafter defined. It is essential to the invention that, in parts by weight, from 60 to 70 parts of the paraffin wax, from 20 to 35 parts of the microcrystalline wax, and from 2 to 10 parts of the oil be employed, as will appear hereinafter.

The accompanying drawing is a ternary diagram showing the permissible variations in the concentrations of the three components, as hereinafter described.

The paraffin wax is prepared by distilling slack wax, or its equivalent, and collecting the light distillate fraction boiling within the range of from about 375° F. to 520° F. at 2 mm. of mercury pressure. This fraction is then dissolved in a solvent at an elevated temperature. The resulting solution is cooled to a temperature of from about 30 to 50° F., preferably from about 35° F. to 40° F., when using a mixture of about equal parts by volume of benzene and methyl ethyl ketone as the solvent, or to the temperature that gives the same wax fraction, within the stated limits, when using a different solvent. The wax precipitated at this temperature is separated, preferably by filtering, and after removal separation of solvent forms the paraffin wax of the present composition. The solvent is conveniently removed by distillation.

The resulting paraffin wax has a melting point of about 125–142° F. (ASTM D87–42), a viscosity of about 35–42 SUS at 210° F. (ASTM D446–39), an average molecular weight of about 355–400, and a refractive index at 212° F. of from 1.419 to 1.423.

The microcrystalline wax of the composition of the invention is prepared by distilling slack wax to form a residual wax boiling above about 525° F. at 2 mm. of mercury pressure. This fraction is dissolved in a solvent at an elevated temperature. The resulting solution is chilled to a temperature of from about 40° F. to 60° F. when using a mixture of about equal parts by volume of benzene and methyl ethyl ketone as the solvent, or to a temperature that gives the same wax fraction, within the stated limits, when using a different solvent. The precipitated wax is separated, preferably by filtration, and is again dissolved in a solvent at an elevated temperature. This solution is then chilled to a temperature of from about 100° F. to about 120° F., and preferably about 110° F., when using the benzene-methyl ethyl ketone solvent, or to an equivalent temperature with a different solvent, as above described. The wax precipitated at this temperature is separated from the solution, preferably by filtration. Separation of solvent from the wax, such as by distillation, forms the microcrystalline wax of the present composition.

The resulting microcrystalline wax has a melting point of about 185–200° F. (ASTM D–127–49), a viscosity of about 81–89 SUS at 210° F. (ASTM D446–39), an average molecular weight of about 650, and a refractive index at 212° F. of from 1.435 to 1.438.

In the preparation of both the paraffin and microcrystalline waxes, it is advantageous to include a decolorization step, such as by contacting the molten wax with clay, but decolorization is not necessary to the successful operation of the invention. If desired, the final composition of the process containing the paraffin and microcrystalline waxes and oil can be decolorized in like manner.

In preparing the wax components of the composition, filtration in each instance is advantageously accomplished by means of a rotary vacuum filter having provision for both supplying wash liquid to the filtered wax and subsequent drying by suction. The fresh wash liquid is advantageously of the same composition as the solvent employed for dissolution of the wax, since recovery and recycling problems are thereby simplified.

The solvent employed in preparing the components of the composition should have a preferential solvent power for the oil contained in slack wax, should dissolve the wax at relatively high temperatures, and precipitate wax at lower temperatures. A mixture of methyl ethyl ketone and benzene in approximately equal parts by volume is the preferred solvent, but either of these components may be replaced, in whole or in part, by other ketones such as methyl butyl ketone or acetone, or hydrocarbons and halogenated hydrocarbons such as ethylene dichloride, pentane and hexane, or alcohols such as propyl or the heptyl alcohols.

The oil component of the composition of the invention is preferably prepared by solvent-refining the residual fraction from the vacuum distillation of crude petroleum, such as by extraction with furfural or propane-phenol solvents, dewaxing the oil such as by chilling in the presence of a solvent selected for preferential dissolution of oil, for example, a mixture of methyl ethyl ketone and benzene, and clay-treating the dewaxed oil. An additional distillation step can be included if necessary or desirable to insure the preparation of oil having the properties hereafter described. It will be understood that the oil component can be obtained using different refining steps, or a different sequence of steps, so long as the oil meets the following specifications.

The oil component should boil within the lubricating oil range, and have an API gravity of about 18. Preferably the oil has a viscosity at 100° F. of about 100 SUS, and more preferably above about 1000 SUS. In the preferred embodiment, the oil has a viscosity index of about 90. The oil should, in any event, have an initial boiling point of about 600° F., and preferably above 700° F., at atmospheric pressure.

The three components of the invention are conveniently blended by heating proper portions thereof, as hereinafter defined, to a temperature above the melting point of the microcrystalline wax, and stirring the molten mixture so that on solidification a homogeneous composition is obtained.

The wax composition of the invention exhibits two breaks in the cooling curve (ASTM D87–42), one at from 160 to 175° F. and a second at from 123 to 139° F. The composition has a penetration (ASTM D127–49) at 77° F. of from 12 to 21, and at 100° F. of from 31 to 50, and a viscosity at 210° F. (ASTM D446–53) of from 41 to 51. The composition has a blocking temperature of at least 100° F., and preferably above 105° F.

Example 1

In order to illustrate the compositions of the invention and their process of preparation, there were blended varying quantities of a paraffin wax, a microcrystalline wax and a lubricating oil, the components having properties within the limits given above. The blending was performed at a temperature of about 225° F., which was sufficient to maintain the components in liquid phase.

The paraffin wax was prepared by distilling reduced crude petroleum having a high wax content and separating the fraction boiling between 375 and 480° F. at 2 mm. pressure. This entire fraction was dissolved in a solvent consisting of about equal parts of benzene and methyl ethyl ketone at a temperature of about 180° F. The resulting solution was cooled to 35° F. and the precipitated wax separated by filtration. The wax cake was washed with the same solvent used to dissolve the distillate fraction. The wax was separated from the solvent by distillation and subsequently decolorized by clay contacting. The recovered wax is the paraffin wax employed in the present composition. The paraffin wax had the following properties, as determined by the tests hereinabove designated: melting point =133° F., viscosity at 210° F.=37.9 SUS, oil content=0.4% by weight, color=+27 Saybolt Universal, average molecular weight=365, and refractive index at 212° F.=1.4196.

The microcrystalline wax was prepared by distilling slack wax to remove the fractions boiling above about 525° F. at 2 mm. pressure. The residue was dissolved in a solvent at a temperature of about 210° F. and the resulting solution chilled to a temperature of 50° F. The precipitated wax was separated by filtration. The so-separated wax was again dissolved in solvent at an elevated temperature of about 210° F. The resulting solution was chilled to 115° F. and the precipitated wax separated by filtration. The separated wax was washed with additional quantities of the same solvent used to dissolve the residue. The solvent remaining was then removed from the wax by distillation. The recovered wax had the following properties, using the tests above designated: melting point=193° F., viscosity at 210° F.=85 SUS, oil content=0.4%, average molecular weight=650, and refractive index at 212° F.=1.4363.

The oil component was prepared by distilling crude oil to recover about 15° thereof as residue. The residue was solvent-refined with propane and phenol-cresol, and then dewaxed. The resulting heavy oil was distilled in the presence of clay to yield a heavy residue which was the oil employed in the present composition and had the following properties: A. P. I. gravity=26.3, viscosity at 100° F.=2634 SUS and at 210° F.=153.9 SUS, and viscosity index=95. The initial boiling point of the oil was about 766° F. and it contained about 27.5% aromatic hydrocarbons.

Wax compositions having varying quantities of the paraffin wax, microcrystalline wax and oil, prepared as above described, were compounded and tested as follows: heavy paper stock employed for making cottage cheese containers was cut into strips of about 2 by 6 inches. The strips were dipped into the molten composition of the invention to form a desired weight of wax per unit area of paper stock. The coated paper was then cooled to room temperature. Similar strips were coated with waxes heretofore used for coating such containers. Cottage cheese containing bacteria known to deleteriously affect wax coatings heretofore used was applied to the wax coated paper strips and held in place by glassine paper. The assemblies were then cooled to 40° F. and maintained at that temperature for from 5 to 14 days. After this aging time, the cheese was carefully removed from the strips, which were then rinsed with water and dipped in a dye solution containing a wetting agent. Any passage of cheese components through the wax coating was indicated by passage of the dye solution through the wax barrier to the fibrous paper stock, which was readily observable. The quantities of the wax composition components, and results obtained, were as follows.

Sample	Micro-crystalline Wax	Paraffin Wax	Oil	Protection [1]	Blocking [2]
A	30	70		fail	pass.
B	20	80		fail	pass.
C	30	65	5	pass	pass.
D	15	80	5	pass	fail.
E	30	64	6	pass	pass.

[1] Passage of dye solution through the wax coating, as above described, does not pass the test, and is designated in the table as "fail."

[2] Compositions blocking at below 100° F. were considered to fail the test; the compositions that passed the test had a blocking temperature of at least 105° F.

It will be noted that samples C and E are within the scope of the invention and gave good results. Omitting a component of the composition, or using a component outside the stated limits therefor, defeats the objects of the invention, as shown by the data.

Example 2

Sample C of Example 1 was duplicated except that the paraffin wax employed had a melting point of 140° F. and an average molecular weight of 385. On testing the composition as described in Example 1, good protection was observed, i. e., a "pass" was obtained in the protection test, and a "pass" was also obtained in the blocking test.

Example 3

A composition according to Example 2 was prepared except that a different oil was used. The oil boiled in the lubricating oil range, had a viscosity at 100° F. of about 100 SUS and a viscosity index of about 95. Other properties were within the limitations as described above.

It was found that excellent results were obtained in both the protection and blocking tests as described for Example 1, a "pass" being obtained in both instances.

The proportions of each of the three components to employ are critical in the preparation of the composition of the invention. The quantity of paraffin wax to employ must be within the range of from 60–70% by weight, the quantity of microcrystalline wax to employ must be within the range of from 25–35% by weight, and the quantity of oil to employ must be within the range of from 2–10% by weight. The preferred composition, with which excellent results are obtained, contains 30% by weight of the microcrystalline wax, 65% by weight of the paraffin wax and 5% by weight of the oil. To emphasize the necessity of employing quantities of the wax composition components within the stated ranges, attention is now directed to the accompanying figure. The figure is a ternary diagram showing, by area ABCDEF, the permissible variations in the concentrations of the three components. If a composition defined by any point outside of area ABCDEF, be employed, the objects of the invention are defeated. For example, if a quantity of the microcrystalline wax below the stated range is employed, the blocking temperature of the wax coating becomes prohibitively low, whereas if quantities above the stated range are employed, the wax coating is not sufficiently flexible to provide adequate protection when coated on containers for packaging dairy products. For similar reasons the quantity of paraffin wax used must be within the stated range. The presence of the oil in a quantity within the defined range is necessary so that the wax coating has the proper flexibility, but a quantity of oil above the stated limitation results in a composition that blocks at a temperature below about 100° F.

The invention claimed is:

1. A new composition of matter comprising: (A) from 60 to 70 parts of a paraffin wax having a melting point of from about 125° F. to 142° F., a viscosity of about 35 to 42 SUS at 210° F., a refractive index at 212° F. of from 1.419 to 1.423 and an average molecular weight of from about 355 to about 400; (B) from 25 to 35 parts of a microcrystalline wax boiling above about 525° F. at 2 mm. mercury pressure, and having a melting point of from about 185° F. to about 200° F., a viscosity at 210° F. of from about 81 to 89 SUS, a refractive index at 212° F. of from 1.435 to 1.438; and (C) from 2 to 10 parts of a lubricating oil having an API gravity of above 18 and an initial boiling point of above 600° F.

2. A new article of manufacture comprising a fibrous container for packaging dairy products coated with the composition of claim 1.

3. Process for the preparation of a new composition of matter which comprises admixing in the molten state: (A) from 60 to 70 parts of a paraffin wax having a melting point of from about 125° F. to 142° F., a viscosity of about 35 to 42 SUS at 210° F., a refractive index at 212° F. of from 1.419 to 1.423 and an average molecular weight of from about 355 to about 400; (B) from 25 to 35 parts of a microcrystalline wax boiling above about 525° F. at 2 mm. mercury pressure, and having a melting point of from about 185° F. to about 200° F., a viscosity at 210° F. of from about 81 to 89 SUS, a refractive index at 212° F. of from 1.435 to 1.438; and (C) from 2 to 10 parts of a lubricating oil having an API gravity of above 18 and an initial boiling point of above 600° F., and solidifying the molten composition.

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