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3,175,986

CARTON BOARD COATING COMPOSITION

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5 Claims. (Cl. 260—28.0)

This invention relates to paraffin wax-containing compositions suitable for coating and laminating paper board, carton board, metal foil, sheet paper, and like sheet materials. More particularly, this invention relates to paraffin wax-containing compositions suitable for coating carton board blanks which are to be formed into containers for liquid products such as milk and fruit juices.

A number of years ago dairies began the commercial use of wax-coated carton board milk containers. The pre-cut, scored, printed blanks were fed into a machine which formed them into containers. These containers, called "cartons," were dipped into a bath of molten wax and after draining and cooling were filled with milk and sealed. This method of producing coated milk cartons had a number of disadvantages. The cartons frequently leaked because of non-uniform coating or because the wax coating cracked and the wax would frequently flake into the milk by reason of its failure to adhere to the carton board properly. It was difficult to maintain the same wax load from carton to carton and thereby control the economics of the coating process. Consequently, there was developed another method of producing coated milk cartons. In this method polyethylene was extruded in the form of a thin film onto a web of carton board and an entire roll of the carton board was coated at one time. From this roll of coated board, blanks were cut, scored and printed. These precoated blanks were utilized to form the milk cartons. This method had numerous advantages over the previous method because the carton-forming and filling machine could be made much simpler and the dairy did not have to become involved with the coating process. The method, however, had disadvantages since frequently an entire roll of paper would be coated with a film having pine holes in it because certain critical coating production variables had not been controlled properly. This imperfect board would not be discovered until the cartons were formed from it and were found to leak. In such cases very large numbers of carton blanks would be useless and wasted. In other instances, the coated blanks would fail to form a proper seal and again would have to be discarded. It also was found that since the printing was applied on top of the polyethylene film, it frequently abraded and the appearance of the finished carton was deleteriously affected. Finally, there was an inherent material loss since in stamping the blanks from the coated sheet, portions of the sheet were irrecoverable waste and had to be discarded.

In accordance with the present invention, a composition has been found having excellent heat sealability properties together with gloss stability which permits paper board blanks to be pre-cut, scored and printed and thereafter coated with such composition to form precoated blanks which can be utilized to form containers such as milk cartons.

It is an object of this invention to provide a heat-sealable, gloss-stable, wax-containing composition for coating and laminating purposes.

It is another object of this invention to provide a wax-containing coating composition suitable for coating carton board.

It is another object of this invention to provide a wax-containing composition suitable for coating pre-cut,

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scored and printed carton board which can be formed into milk containers.

Other objects of this invention will be apparent from the description and claims that follow.

The composition of the instant invention consists essentially of a paraffin wax, an ethylene-vinyl acetate copolymer, a microcrystalline wax and a terpene resin. The critical properties of each component together with the critical amount of such component in the total composition will be described in detail.

The ethylene-vinyl acetate copolymers suitable for producing the compositions of this invention are those copolymers having a polymerized vinyl acetate content in the range from 26 weight percent to 30 weight percent and preferably in the range from 27 weight percent to 29 weight percent of the copolymer. The copolymers suitable for use in producing the compositions of the invention are also characterized by having a melt index in the range from about 1 to 25 and preferably in the range from 2 to 15.

The ethylene-vinyl acetate copolymers can be prepared by copolymerizing a mixture of ethylene and vinyl acetate in a tubular reactor at high pressures, for example at from 15,000 p.s.i.g. to 30,000 p.s.i.g. at temperatures of from about 150° C. to 250° C. in the presence of a peroxidized catalyst such as tert-butyl hydroperoxide. The unreacted monomers are flashed from the copolymer in order to separate the purified copolymer. The quantity of polymerized vinyl acetate in the copolymer is controlled primarily by the vinyl acetate content in the monomer mixture. Copolymers having a melt index within the desired critical range of this invention are obtained by varying the conditions of pressure, temperature, and catalyst concentration in accordance with known commercial methods. Ethylene-vinyl acetate copolymers having the critical properties set forth herein are all available commercially. The melt index of the copolymer is that determined in accordance with ASTM Method D1238—57T.

It has been found that as the melt index of the copolymer employed in the composition decreases the flexibility of the composition increases; therefore, it is preferred to employ the lower melt index range of copolymers. If the copolymer has a melt index above about 25, it is necessary to employ such large amounts of the copolymer to achieve equivalent flexibility that it would upset the balance of the other components in the composition.

The quantity of ethylene-vinyl acetate copolymer which is utilized in the compositions ranges between 25 weight percent and 40 weight percent based on the total weight of the composition. Preferably the quantity of copolymer ranges between 27 weight percent and 29 weight percent. When quantities less than about 25 weight percent are employed, the composition loses its heat sealability, i.e., its fiber tear properties. When the quantity of copolymer approaches about 40 weight percent in the composition, the viscosity of the composition increases to such a degree that it becomes very difficult to obtain homogeneous blends or to use the composition for coating and laminating. It has been found that a quantity within the range of from 27 weight percent to 29 weight percent not only provides the desired heat sealability properties, but also provides a viscosity in a range which can be utilized conveniently for blending and coating. The copolymer also provides flexibility to the composition so that the composition may be utilized to coat carton blanks which are subsequently formed into cartons by bending along the pre-scored lines.

The microcrystalline wax suitable for producing the compositions of this invention is preferably a petroleum

hydrocarbon wax having a melting point in the range of from 160° F. to 180° F. Microcrystalline waxes are produced commercially by petroleum refiners and such products are also designated as amorphous or petrolatum waxes. They are derived from the less volatile and heavier petroleum fractions as compared with the fractions from which paraffin waxes are obtained. Microcrystalline waxes are generally considerably less brittle as compared with paraffin waxes. They are prepared by conventional distillation and selective solvent extraction processes. Preferably the microcrystalline wax has a melting point in the range of from 170° F. to 180° F., and particularly useful compositions have been produced from microcrystalline wax having a melting point of about 175° F. The melting points set forth are determined in accordance with ASTM Method D127, the so-called Drop Melting Point.

The quantity of microcrystalline wax suitable for producing the instant compositions ranges from 12 weight percent to 30 weight percent, based on the total weight of the composition. Preferably the quantity of microcrystalline wax ranges between 15 weight percent and 25 weight percent, and particularly useful compositions have been produced employing about 20 weight percent of the microcrystalline wax having a melting point of 175° F. When the quantity of microcrystalline wax is below 12 weight percent and in some instances below about 15 weight percent, the flexibility of the composition is deleteriously affected and in addition, the composition loses gloss stability.

A coating composition for use on milk cartons requires a high degree of gloss for a pleasing appearance and sales appeal. Many compositions when first applied to carton materials have the desired gloss. However, upon standing and aging, they lose this gloss, i.e., they do not have gloss stability. It has been found that compositions of this invention should have at least 12 weight percent and preferably 15 weight percent or more microcrystalline wax in order to have the desired gloss stability.

When the quantity of microcrystalline wax employed is in excess of about 30 weight percent, the composition will have a "waxy" feel which is undesirable from a consumer standpoint, and in addition, the sealing properties of the composition may be deleteriously affected. Accordingly, the quantity of microcrystalline wax employed should not exceed about 30 weight percent and preferably should not exceed about 25 weight percent, particularly if the microcrystalline wax is not water white in color, since in such cases the color of the composition also will be deleteriously affected.

The terpene resins which have been found suitable for use in the compositions of this invention are those having melting points (sometimes referred to as softening points) in the range of from 115° C. to 135° C. These terpene resins are hydrocarbon, thermoplastic resins produced by catalytically polymerizing pinenes. The terpene resin produced by the controlled polymerization of pinenes is sold commercially under the trade name "Piccolyte." These resins are available in a variety of melting point grades. Those which have been found to be particularly suitable for use in this invention are "Piccolyte S-115" which has a melting point of about 115° C., and "Piccolyte S-135" which has a melting point of about 135° C. The melting point is determined by the ring and ball method, and in each grade the tolerance is $\pm 3^\circ$ C. Similar terpene resins sold under the trade name "Nirez" have also been found to be suitable, in particular "Nirez 1125" grade, which has a melting point of about 125° C. These terpene resins are very light in color, generally a very pale amber. They have acid numbers and saponification numbers of approximately zero, and they have a specific gravity of about 1.0. The polymers vary in molecular weight with melting point; however, in general, they are in the range of from 860 to 1200.

The quantity of terpene resin suitable for use in the

compositions of the instant invention range from 3 weight percent to 10 weight percent, and in particular about 5 weight percent has been found to be useful. When quantities less than about 3 weight percent are employed, the flexibility of the composition is deleteriously affected, whereas when quantities in excess of about 10 weight percent are utilized, the color and odor of the composition may be deleteriously affected.

In the foregoing discussion concerning the quantities of microcrystalline wax and terpene resin, certain minimum quantities have been specified for each component in order to impart gloss stability and flexibility to the composition. It has been found, however, the neither of these components when used alone in the absence of the other impart the desired gloss stability and flexibility to the composition. Accordingly, therefore, when specifying the minimum quantities of microcrystalline wax and of terpene resin it is understood that these quantities apply only when both components are present in the composition. Thus the composition will have some of the desired gloss stability and flexibility properties when the microcrystalline wax is in the 12 weight percent to 15 weight percent range, provided there is at least 3 weight percent of terpene resin present in the composition, or, it may be stated that the composition will not have its desired gloss stability and flexibility properties when it contains from 3 weight percent to 10 weight percent terpene resin unless there is at least the specified minimum 12 to 15 weight percent of microcrystalline wax present in the composition.

The paraffin wax which constitutes the remainder of the composition in addition to the copolymer, microcrystalline wax and terpene resin, is preferably a refined petroleum hydrocarbon wax having a melting point in the range of from about 145° F. to 155° F. Such paraffin waxes are produced by conventional solvent extraction of waxy distillate oil fractions using, for example, methyl ethyl ketone as the solvent, followed by conventional finishing procedures such as acid treating and clay filtration. A particularly suitable refined petroleum paraffin wax is that having a melting point in the range of about 150° F. to 155° F. It will also be understood, however, that although the paraffin wax constitutes the remainder of the essential components of the composition, the composition may contain minor amounts of other components. For example, certain of the terpene resin grades are produced containing small quantities of antioxidants, and in addition, conventional and well-known wax antioxidants may be utilized in the composition. The amounts of such antioxidants are relatively minor compared with the total composition, generally of the order of several parts per million; for example, 30 parts per million of conventional commercial antioxidants have been found to be suitable.

The compositions of this invention are conveniently prepared by melting the desired amounts of paraffin wax and microcrystalline wax and blending them in the molten condition. A commercial oxidation inhibitor may be added to the molten wax, for example, butyl-hydroxyanisole, in an amount of about 30 parts per million by weight based on the total composition. The temperature of the molten wax mixture is then raised to at least 285° F., although it may be raised as high as 310° F., and the solid pellets of copolymer are added with vigorous agitation at such a rate that the temperature of the mixture does not fall greatly below the 285° F. level, and if it falls as low as 270° F., copolymer addition is discontinued until the temperature is raised to the desired level. After the copolymer has been added, the agitation is continued until a homogeneous mixture is obtained. The terpene resin is next added to the hot mixture. It is added at a temperature above the melting point of the resin, in general at approximately the same temperature at which the copolymer is added. The mixture is stirred until it is completely homogeneous, and thereafter it is packaged

either in bulk or in flakes, according to conventional methods.

The following examples are provided for the purpose of illustrating specific embodiments and features of the invention. In these examples the compositions were prepared in accordance with the method which has been described and were utilized to coat commercial milk carton board.

In determining the gloss stability of the composition, a Hunter Gloss Meter, Type D-16, was utilized. In Examples I and II the carton board was coated by dipping the board into a sample of the composition which was held at a temperature of 220° F. to 225° F. for 3 seconds. The excess wax was metered from the board and the coated board was quenched in water held at 70° F. Gloss meter readings which measure the reflectance of the coating as compared with a standard glass plate were made on the board one-half hour after coating, and thereafter periodically for several days to measure the loss in gloss with time. These gloss meter readings were plotted against time and the slope of the line was used in rating the gloss stability of the composition.

In order to determine the flexibility of the compositions, a sample of each composition was injection molded at about 205° F. into a specimen 1¼ inches long, ½ inch wide, and 70±0.5 mils thick. Each specimen was subjected to repeated double flexes (alternating direction), 90° angle bends, at a flex rate of 25 double flexes (25 cycles) per minute at a temperature of 70° F. and a relative humidity of 50 percent. The flex life is the number of cycles to failure which is determined by the specimen being cracked through one half its width.

Example I

In order to demonstrate the effect on the flex life of the composition by the amount of copolymer in the composition and the softening point of the terpene resin used in the composition, four compositions were prepared. In each composition the quantity of each component is given in weight percent.

Composition A:

Ethylene-vinyl acetate copolymer (vinyl acetate content 28 weight percent, melt index 3)	25
Microcrystalline wax, M.P. 175° F.	12
Terpene resin, M.P. 135° C.	5
Paraffin wax, M.P. 145° F. to 155° F.	58

Composition B:

Ethylene-vinyl acetate copolymer (vinyl acetate content 28 weight percent, melt index 3)	25
Microcrystalline wax, M.P. 175° F.	12
Terpene resin, M.P. 125° C.	5
Paraffin wax, M.P. 145° F. to 155° F.	58

Composition C:

Ethylene-vinyl acetate copolymer (vinyl acetate content 28 weight percent, melt index 3)	28.2
Microcrystalline wax, M.P. 175° F.	20
Terpene resin, M.P. 135° C.	5
Paraffin wax, M.P. 150° F. to 155° F.	46.8

Composition D:

Ethylene-vinyl acetate copolymer (vinyl acetate content 28 weight percent, melt index 3)	28.2
Microcrystalline wax, M.P. 175° F.	20
Terpene resin, M.P. 125° C.	5
Paraffin wax, M.P. 150° F. to 155° F.	46.8

When the gloss stabilities of these compositions were determined on the coated specimens of carton board, they were found to be essentially the same. Composition A had a flex life of 76 cycles, and Composition B had a flex life of 114 cycles. This shows that the lower melting point terpene resin improves the flex life of the composition. Composition C had a flex life of 319 cycles, and Composition D had a flex life of 372 cycles. In comparing Composition C with Composition D it will be

apparent that the lower softening point terpene resin improves the flex life as it did in Composition B compared with Composition A. Comparing Composition C with Composition A and Composition D with Composition B, it will be apparent that increasing the amount of copolymer slightly to bring it into the preferred range markedly increases the flex life of the composition.

The pre-cut, scored and coated blanks are sent through a so-called "side-seam sealing" machine in which the opposite edges of the carton are sealed together by forming two 180° bends in the carton so that the opposite edges overlap and can be heat sealed. This forms a carton "flat" which is subsequently used by the dairy in forming a carton for filling and sealing in a single machine operation. The side-seam sealing machine operates at extremely high rates, i.e., 72,000 cartons per hour, so it is apparent that the coating composition must have excellent flexibility properties in order that when the coated carton board is bent rapidly in the sealing machine, the coating over the score lines will not break.

Example II

In order to show the effect on the flex life of the composition by the melt index of the copolymer, four compositions were prepared. In these compositions the terpene resin was omitted in order to show more clearly the effect of the copolymer melt index. In each composition the quantity of each component is given in weight percent.

Composition E:

Ethylene-vinyl acetate copolymer (vinyl acetate content 28 weight percent, melt index 25) ..	30
Microcrystalline wax, M.P. 175° F.	15
Paraffin wax, M.P. about 145° F. to 155° F.	55

Composition F:

Ethylene-vinyl acetate copolymer (vinyl acetate content 28 weight percent, melt index 25) ..	20
Ethylene-vinyl acetate copolymer (vinyl acetate content 28 weight percent, melt index 3) ..	10
Microcrystalline wax, M.P. 175° F.	15
Paraffin wax, M.P. about 145° F. to 155° F.	55

Composition G:

Ethylene-vinyl acetate copolymer (vinyl acetate content 28 weight percent, melt index 25) ..	10
Ethylene-vinyl acetate copolymer (vinyl acetate content 28 weight percent, melt index 3) ..	20
Microcrystalline wax, M.P. 175° F.	15
Paraffin wax, M.P. about 145° F. to 155° F.	55

Composition H:

Ethylene-vinyl acetate copolymer (vinyl acetate content 28 weight percent, melt index 3)	30
Microcrystalline wax, M.P. 175° F.	15
Paraffin wax, M.P. about 145° F. to 155° F.	55

Composition E had a flex life of 52 cycles, Composition F—95 cycles, Composition G—129 cycles, and Composition H—176 cycles. This demonstrates that as the melt index of the copolymer decreases, the flexibility of the composition increases.

Example III

The following compositions were prepared and utilized to coat a large number of milk carton blanks. The quantity of each component is given in weight percent.

Composition J:

Ethylene-vinyl acetate copolymer (vinyl acetate content 28 weight percent melt index 3)	28.2
Microcrystalline wax, M.P. 175° F.	20
Terpene resin; M.P. 135° C.	5
Paraffin wax, M.P. 152° F.	46.8

Composition K:

Ethylene-vinyl acetate copolymer (vinyl acetate content 28 weight percent, melt index 3)	28.2
Microcrystalline wax, M.P. 175° F.	20
Terpene resin, M.P. 125° C.	5
Paraffin wax, M.P. 152° F.	46.8

Each composition also contained 30 parts per million of butylhydroxyanisole.

The coated blanks were formed into cartons, filled with milk, and sealed. They were found to be superior to wax coated cartons in flake-resistance and appearance, i.e., in gloss and gloss stability. They were equal to extruded polyethylene coated cartons in these properties. They were superior to polyethylene coated cartons since the printed surface was protected and could not be abraded from the carton surface.

We claim:

1. A composition for coating and laminating consisting essentially of a paraffin wax having a melting point in the range from about 145° F. to 155° F. and the following components in weight percent based on the weight of the total composition:

Ethylene-vinyl acetate copolymer	25-40
Microcrystalline wax, M.P. 160° F. to 180° F.	12-30
Terpene resin, M.P. 115° C. to 135° C.	3-10

wherein the vinyl acetate content of the copolymer ranges from 26 weight percent to 30 weight percent and the melt index of the copolymer ranges from 2 to 25.

2. A composition for coating and laminating consisting essentially of a paraffin wax having a melting point in the range from about 145° F. to 155° F. and the following components in weight percent based on the weight of the total composition:

Ethylene-vinyl acetate copolymer	27-29
Microcrystalline wax, M.P. 160° F. to 180° F.	15-25
Terpene resin, M.P. 115° C. to 135° C.	3-10

wherein the vinyl acetate content of the copolymer ranges for 27 weight percent to 29 weight percent and the melt index of the copolymer ranges from 2 to 15.

3. A composition for coating and laminating consisting essentially of a paraffin wax having a melting point in the range from about 150° F. to 155° F. and the following components in weight percent based on the weight of the total composition:

Ethylene-vinyl acetate copolymer	27-29
Microcrystalline wax, M.P. 170° F. to 180° F.	15-25
Terpene resin, M.P. 125° C. to 135° C.	3-10

wherein the vinyl acetate content of the polymer ranges from 27 weight percent to 29 weight percent and the melt index of the copolymer ranges from 2 to 15.

4. A composition for coating and laminating consisting essentially of the following components in weight percent based on the weight of total composition:

Ethylene-vinyl acetate copolymer	28
Paraffin wax, M.P. 150° F. to 155° F.	47
Microcrystalline wax, M.P. about 175° F.	20
Terpene resin, M.P. about 135° C.	5

5. A composition for coating and laminating consisting essentially of the following components in weight percent based on the weight of the total composition:

Ethylene-vinyl acetate copolymer	28
Paraffin wax, M.P. 150° F. to 155° F.	47
Microcrystalline wax, M.P. about 175° F.	20
Terpene resin, M.P. about 125° C.	5

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MORRIS LIEBMAN, *Primary Examiner*.

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,175,986

March 30, 196

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It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 1, line 44, for "pine" read -- pin --; column 2, line 26, for "perodixed" read -- peroxide --; column 4, line 15, for "flebility" read -- flexibility --; column 5, line 21 for "ratnig" read -- rating --; column 8, line 13, for "polymer" read -- copolymer --.

Signed and sealed this 24th day of August 1965.

(SEAL)

Attest:

ERNEST W. SWIDER
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents