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COATING FOR MILK CONTAINERS

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This invention is related to a method of coating milk containers and the product formed thereby. The invention particularly relates to an improved paper coating applied in two steps with a substantial reduction in wax requirement.

A large variety of wax compositions are used commercially for coating paper and paperboard. For example, paraffin wax having a melting point between about 120–150° F. has been used extensively and this has been improved by the addition thereto of various addition agents, such as polyethylene and microcrystalline wax. A large portion of the wax-coated board is used in the milk industry to replace glass bottles, and with the rapid growth of this business the requirement for coating wax is constantly increasing.

The paraffin wax is obtained from selected petroleum waxy distillates which are chilled and mixed with suitable solvents such as liquefied propane, petroleum naphtha, methyl ethyl ketone, toluene, etc., or combinations thereof. The mixture is filtered to remove the wax from the oil and the solvent is removed from the wax. This wax is termed slack wax and still contains about 10–40 percent of oil. In some cases slack wax is also obtained from the cold-pressing of wax distillate per se. The slack wax is then treated to effect further removal of oil, either in conventional sweating operations or by further solvent extraction, producing a crude scale wax having an oil content of about 1–3 percent. By more precise re-sweating operations or crystallization from suitable solvents, the wax is converted to a semi-refined wax having an oil content of 0.5–1 percent or a fully refined wax having an oil content of less than 0.5 percent, usually 0.1–0.3 percent. The presence of any substantial amount of oil in the wax is detrimental to the tensile strength of the wax and hence the fully refined wax is particularly required for coating board used as milk containers.

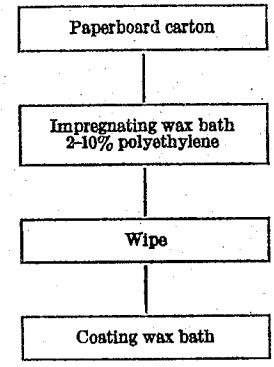
In contrast to paraffin wax, microcrystalline wax is obtained from solvent-extraction operations on the residual portion of crude petroleum. Usually, the residuum is first subjected to a deasphalting treatment to permit proper crystallization of the wax in the subsequent chilling of the mixture with solvent. After filtering, the petroleum obtained is mixed with an additional quantity of solvent; the mixture is chilled and filtered to remove the microcrystalline wax and the solvent is removed from the wax. Microcrystalline waxes have melting points usually in the range of 145–175° F., and their viscosities are about 12–20 centistokes at 210° F., whereas the viscosities of paraffin waxes are about 3–6 centistokes at 210° F.

There are two methods currently used in applying wax to paperboard containers.

A. The preformed carton is dipped in molten wax, drained, cooled and shipped to dairies ready for filling with milk, cream, etc.

B. Many dairies are equipped with machines which continuously perform a series of operations. Flat cartons are introduced into the machine, which forms each carton and glues the bottom flaps. The carton is then dipped in molten wax, drained and cooled. The waxed carton is then filled with milk, cream, etc.

In both cases, the wax is applied in a single-dip operation.



A large potrion of the coating wax is absorbed in the paperboard during the single-dip operation and hence a substantial amount of wax is required to provide an adequate wax coating giving full protection against leakage. I have discovered that this large wax requirement can be substantially reduced by applying wax in two separate operations. The first is one of controlled impregnation of the paperboard with a paraffin wax in which has been blended a substantial amount of an ethylene polymer. Surface coating is eliminated in this operation by wiping the paperboard as it emerges from the liquid wax-polymer blend. The second step is then conducted by either of the two methods previously described for the single-dip operation, using paraffin wax containing little or no ethylene polymer. The combination of these two steps in applying wax results in the aforementioned substantial reduction of wax requirement by virtue of decreasing the amount of impregnation to one-half or less of that from the single-dip operation, while holding the amount of surface coating nearly the same.

It is accordingly an object of the present invention to provide a method of preparing a suitable impregnant for milk-container paperboard. Another object is to provide a two-step operation for applying wax to milk containers. A still further object is to afford a means whereby the number of cartons treated in accordance with the present invention can be substantially increased over those waxed by the current single-dip operations from the same quantity of wax. These and other objects which will be recognized by those skilled in the art are attained in accordance with the present invention, wherein wax is applied under prescribed conditions in two separate operations.

For this first step, however, the ethylene polymer must be completely in solution with the wax to yield the desired results. It is commonly known that ethylene polymers are difficult to get into solution with wax and for this reason only very limited amounts of ethylene polymers have been used to improve the coating wax. Efforts to bring a substantial amount of polyethylene into solution with a wax have always resulted in the formation of a precipitate of the polymer on the cooling of the solution. I have discovered that this can be avoided by heating the polyethylene and wax, first, to a temperature above the melting point of the polyethylene or at least close to it, second, by cooling the mixture to a temperature below the cloud point of the mixture but above the melting point of the wax, third, reheating to a temperature above the cloud point of the mixture but below a critical maximum temperature found to be a point substantially below the melting temperature of the polyethylene. I have found that if this sequence of steps is followed, the polyethylene-wax structure is homogeneous and, upon cooling below the cloud point of the

mixture, separation into two phases does not occur. In fact, if the mixture is allowed to cool to a temperature well below the cloud point, but above the melting point of the mixture, it forms a gel structure. In this form, the mixture can be reheated to a suitable temperature for application without destroying its effectiveness. This method of mixing polyethylene with wax provides a simple technique for providing a suitable impregnating solution and can be applied to a large variety of waxes and polyethylenes. The method can be used, however, only where a substantial amount of polyethylene is blended with the wax. The lower limit of polyethylene is about 2 percent of the blend, although this cut-off point is not sharply defined. The lowest percent of polyethylene will vary to some extent with different polyethylenes and with different waxes but is in the neighborhood of 2–3 percent by weight of the mixture.

As an illustration of the criticality of the content of polyethylene in the blend in order to practice the teaching of this invention, a polyethylene (Epolene N as sold commercially today by Eastman Chemical Products, Inc. of Kingsport, Tennessee) was blended with a paraffin wax of approximately 128° F. melting point by heating and stirring until a maximum temperature of 225° F. was reached. On cooling to approximately 150° F., the polyethylene precipitated out of solution. This procedure was followed with mixtures of varying polyethylene content and the mixtures were then reheated to a temperature just sufficient to obtain a clear solution, the precise temperature varying with the polyethylene content, and the mixtures were then allowed to cool to 150° F. A gel structure was found to exist at 3 percent or greater polyethylene content, between 2–3 percent the mixture was "soupy" but homogeneous. At 1–1.5 percent polyethylene the blends were fluid and cloudy, but the cloud appeared uniformly throughout each mass. On the other hand, at 0.5 percent, 0.1 percent and 0.05 percent polyethylene, the polyethylene precipitated out of solution. In order to provide the desired intimate blending of polyethylene and wax required for this invention, therefore, the polyethylene content should be at least about 2 percent of the blend. The temperature required for reheating the cooled mixture of polyethylene and wax to effect removal of the cloud is as follows for the various polyethylene contents indicated:

TABLE I

Percent polyethylene[1] in blend:	Temperature, ° F. for clarity (±2° F.)
5	199
4	197½
3	197
2	196
1.5	195
1.0	195
0.5	191
0.1	185
0.05	180

[1] Commercially available, approximately 2500–3000 molecular weight (Staudinger Method).

These tests showed that the blend could not be reheated to a temperature much above the temperatures indicated above without damage to the structure, since when each blend was reheated to 210° F. or higher and then allowed to cool to 150° F. (at least some temperature below its cloud point), the polyethylene precipitated out of solution in all cases.

The polyethylene tested hereinabove and reported in Table I had a molecular weight of about 2500–3000. A straight-chain polyethylene of approximately 750 molecular weight, in the amount of 5 percent in the same paraffin wax of 128° F. melting point, behaved in the same manner as an equal percentage of a branched-chain polymer having a molecular weight of about 2500–3000 (Staudinger method).

In another case, a polyethylene having a molecular weight (Staudinger method) of approximately 20,000 (Alathon 10 sold by E. I. du Pont de Nemours & Co., Inc.) was blended with a paraffin wax in proportion of 2 percent polyethylene. The mixture was heated, with stirring, to a temperature of 335° F. and cooled to 150° F., at which temperature the polyethylene was precipitated out of solution. The mixture was reheated to 205° F., at which temperature the polyethylene went into solution and the mixture remained homogeneous as in a gel structure on being recooled to 150° F.

Thus, the polyethylene used may vary quite considerably and may range in molecular weight from about 500–20,000 (Staudinger method). The lower limit of molecular weight may vary substantially with materials of different structure. The polyethylenes having straight chains demonstrate flow properties somewhat different from those having branched chains, and this fact will determine to a large extent the lower limit of molecular weight of the polyethylenes usable in this invention.

On dipping in a solution formed as indicated hereinabove and possessing an adequate amount of polyethylene as about 2 percent or more, the carton stock absorbs enough of this mixture to provide a good base for the final coating wax and to prevent the impregnation of any substantial amount of the coating wax into the board. The impregnated board carrying the polyethylene-wax blend as impregnant is dipped in the coating wax as the second step of the process to provide the finished coating. Substantially less total wax is applied to the board, but the coating is entirely adequate for milk containers, providing a satisfactory seal. The first dipping operation can be effected at the board plant, whereas the second dipping step can be carried out either at the plant which manufactures preformed cartons or at the dairy plant equipped as described previously.

This technique provides a cheaper coating and materially increases the surface area that can be coated with available wax supplies. Wax is a secondary product in the petroleum business and the amount available fluctuates to a large extent with the production of other petroleum products. Thus, this two-step coating process provides flexibility whereby the number of containers coated with the wax can be increased in spite of low wax supply, in comparison with single-dip operations.

For the various experiments performed, I made test panels 5 inches x 2¾ inches cut from a roll of "half-gallon" carton stock. These panels were air-conditioned in a room held at 70–72° F. and approximately 50 percent relative humidity. The amounts of impregnation and coating were determined from the weights of the panels before and after dipping. In the case of the single-dip operation and in the second step of the two-step process, each panel was dipped in the coating wax, contained in a 600-milliliter, tall-form beaker and held at the desired temperature for 15 seconds, after which the panel was removed by means of a bent paperclip inserted in a hole near one end of the panel. The panel was allowed to drain for 10 seconds and then it was inverted to permit drainage of wax in the opposite direction for 25 seconds. The waxed panel was then dipped for 3–5 seconds in cool water to set the coating. This procedure simulates plant operations. In the case of determining the amount of impregnation, as in the first step of this invention, the panels were dipped in the wax-polyethylene blend for 15 seconds at the desired temperature and were wiped immediately with a cloth to remove the excess of the impregnant.

I found that when the board was dipped for 15 seconds in 100 percent paraffin wax of 128° F. melting point at a temperature of 170° F. and was wiped immediately, the amount of impregnation was 22.8 percent. When the board was dipped for 15 seconds in a blend consisting of 5 percent of a polyethylene having a molecular weight (Staudinger method) of 2500–3000 and 95 percent of

the same paraffin wax and brought into homogeneity as indicated hereinabove and held at 185° F., the amount of impregnation was only 8.0 percent. For further comparison, a similar wax-polyethylene blend was formed in solution at 225° F. and then was cooled to 150° F. The polyethylene was precipitated and in suspension. No attempt was made to re-dissolve the precipitate. The board dipped in this medium at 150° F. absorbed 23.2 percent by weight. This is practically the same amount of impregnation as that obtained when dipping the board in 100 percent paraffin wax at 170° F. It shows that the polymer should be in solution in order to build up the viscosity of the liquid wax and thereby to retard impregnation.

Tests were also made for comparative purposes to demonstrate the saving in wax by using this two-step process. The results are shown in Table II, as follows:

TABLE II

Wax consumption data [1]

Single-dip operation: 100 percent paraffin wax of 128° F. M.P.—@ 170° F	40.4 percent.
Two-step process:	
(1) Absorption from blend of—95 percent 128° F. M.P. paraffin 5 percent polyethylene of 2500–3000 mol. wt.—@ 185° F	8.0 percent in first step.
(2) Coating from 100 percent paraffin wax of 128° F. M.P.—@170° F	18.6 percent in second step.
Total consumption	26.6 percent.

[1] By weight of test panels and using test procedures outlined hereinabove.

It is seen from the above indicated example that the number of cartons coated could be increased

$$\frac{40.4}{26.6}$$

or 1.5 times the present output when two-step operations are substituted for the present single-dip operation.

The first dipping operation is intended to effect a limited penetration of the mixture into the paperboard and provide a base for the attachment of the second coating operation. It is desirable to limit the amount of the first blend on the paperboard, therefore, by wiping the board after the dipping operation. This can be done at the board plant by continuously drawing the impregnated paperboard into contact with wiping blades mounted to remove the excess, liquid wax-polyethylene blend from the exterior of the paperboard and to permit drainage back to the source of application. The paperboard may be dipped into the liquid at various temperatures, such as, for example, 150–200° F. It is by fixing the temperature, the composition of the wax-polyethylene blend, and the period of time during which the board is submerged in the liquid blend, that the amount of impregnation is controlled. As stated previously, the second step is then conducted by either of the two methods described for the single-dip operation.

For the initial dipping material, the polyethylene content may vary from about 2 percent to about 10 percent by weight of the finished blend, depending upon the grade of polyethylene. A more preferred range of polyethylene content is between about 3–7 percent by weight of the blend, when using a polyethylene having a molecular weight (Staudinger method) of about 1500–4000.

I claim:

1. A two-step coating operation for milk containers comprising first dipping the container material in an impregnating solution of petroleum wax blended with 2–10 percent of polyethylene, wiping the container material to remove surface wax from the container material, dipping the container material in a coating bath at least substantially free of polyethylene and formed of more than 98 percent petroleum wax to apply a finished coating to the container material, draining the container material and cooling to set the finished coating, thereby providing a satisfactory coating for the container with a reduced requirement for wax.

2. A two-step coating operation for milk containers comprising first dipping the container material in an impregnating solution of petroleum wax blended with 3–7 percent of polyethylene, having a molecular weight (Staudinger method) of about 1500–4000, removing surface wax from the container material, dipping the impregnated container material in a coating bath formed of at least 98 percent petroleum wax to apply a finished coating to the container material, draining the container material and cooling to set the finished coating, thereby providing a satisfactory coating for the container with a reduced requirement for wax.

3. A milk container formed of paperboard impregnated with a mixture of wax and at least 2 percent polyethylene by weight of the mixture and a surface coating wax containing substantially no ethylene polymer and more than 98 percent petroleum wax located on the exterior of the impregnated paperboard.

References Cited in the file of this patent

UNITED STATES PATENTS

1,824,885	Hammond	Sept. 29, 1931
2,391,620	Quarles et al.	Dec. 25, 1945
2,392,972	Cheyney	Jan. 15, 1946
2,758,100	Bailly et al.	Aug. 7, 1956
2,857,352	Etherington et al.	Oct. 21, 1958
2,859,190	Cubberley et al.	Nov. 4, 1958
2,882,246	Leatherman et al.	Apr. 14, 1959

FOREIGN PATENTS

642,442	Great Britain	Sept. 6, 1950