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**METHOD FOR COATING MILK CONTAINERS**  
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This invention relates to containers for liquids which are made from fibrous cellulosic sheet materials and coated with a liquid-resistant petroleum wax. Specifically, the invention is directed to a novel method for wax-coating such containers, whereby the coated containers are rendered especially suitable for filling with liquids, such as milk, which must be packaged and stored under refrigerated conditions.

It is old in the art to employ wax-coated cellulosic containers for packaging liquid products. In accordance with modern packaging techniques, the forming and coating of the containers and their filling with the liquid are closely sequential operations, commonly performed in continuous, automatic machinery. The conventional method comprises machine-forming the containers from pre-cut blanks of cellulosic sheet material; immersing the formed, but open, containers in a bath of molten coating composition; removing the open containers from the wax bath and draining off the excess wax; cooling the coated containers under either refrigerated or normal atmospheric conditions to solidify and harden the protective wax coating; and filling the coated containers with the liquid just prior to closing and sealing.

The conventional coating composition which is employed in accordance with the above method is substantially a petroleum paraffin wax, since such waxes have been found to have properties particularly suited to this use. Petroleum paraffin waxes suitable for container coating applications normally have melting points in the range of from about 125-140° F. (A.S.T.M. D87-57), a penetration at 77° F. of from about 10 to about 30 (A.S.T.M. D1321-57), a viscosity at 210° F. of about 36.0 to about 40.0 Saybolt Universal seconds (A.S.T.M. D446-53), a blocking temperature of about 90° F. and higher (A.S.T.M. D1465-57T) and an oil content of below about 0.5% by weight (A.S.T.M. D721-56T). Mixtures of such petroleum paraffin waxes can also be used. On occasion, small amounts of a high melting petroleum microcrystalline wax can be incorporated in the paraffin wax to improve various properties thereof for container coating applications. For example, from about 1 to about 3 percent by weight of a petroleum microcrystalline wax having a melting point of from about 190 to about 195° F. (A.S.T.M. D127-49), a penetration at 77° F. of from about 3 to about 7 (A.S.T.M. D1321-57), a viscosity at 210° F. of from about 82.0 to about 87.0 Saybolt Universal seconds (A.S.T.M. D446-53) and an oil content of below about 0.5% by weight (A.S.T.M. D721-56T), can be added to the petroleum paraffin wax to reduce serpentine effect in the resulting coating, and to raise the blocking temperature thereof.

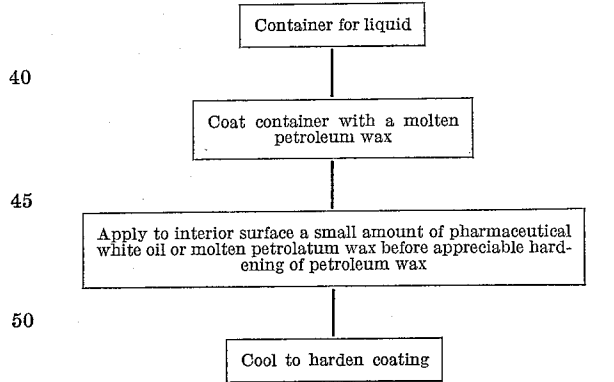
While such waxes and the above technique are generally well suited for packaging liquids which are to be filled and stored at temperatures near room temperatures, particular problems are encountered in packaging liquids, such as milk, which must be filled and stored under refrigerated conditions. In the latter situation, the conventional wax coatings above described have a tendency to crack and flake off when contacted with cold milk during the filling operation. This phenomenon is particularly undesirable since, in many instances, the cracking of the coating causes the container to leak. Furthermore, even though the container may not leak, the presence of free flakes of wax in the liquid contents of

the container creates a negative attitude on the part of the consumer towards the packaging of products in this manner.

The occurrence of cracking and flaking of the wax coating in filling containers with refrigerated liquids has been reduced, to a degree, by certain changes in the formulation of the wax coating composition. Thus, the incorporation of small amounts of a petroleum distillate oil in the basic petroleum wax coating composition has significantly reduced, but not completely eliminated, the cracking and flaking of the wax coating. There are, however, limits to the extent that such corrective expedients can be carried. Thus, the incorporation of more than a small, even critical, amount of a petroleum-derived oil in the coating composition is deleterious in that it lowers the blocking temperature of slabs composed thereof, i.e., the slabs will stick together during shipment and storage. Furthermore, the presence of excess oil makes the coating on the container tacky, and difficult to handle, either manually or in subsequent machine operations.

In view of the above, a particular object of this invention is to provide a novel method for applying a protective coating of a petroleum wax coating composition to containers made from fibrous, cellulosic sheet materials, whereby the coated containers can be filled with refrigerated liquids without consequent cracking and flaking of the wax coating. Another object of the invention is to provide a novel method for substantially eliminating the cracking and flaking of petroleum wax coatings when fibrous cellulosic containers coated therewith are filled with refrigerated liquids, such as milk, at or even below temperatures in the neighborhood of about 35° F. These and other objects and benefits of the invention will become more readily apparent from a reading of the following detailed description of the invention.

A simplified flow diagram of the method is as follows:



It has now been discovered that the cracking and/or flaking of petroleum wax container coatings on contact with refrigerated liquids can be eliminated or substantially suppressed by applying a small amount of a selected material, hereinafter termed a "plasticizer," to an interior surface of the container, after coating, but prior to appreciable hardening of the coating composition. In accordance with the invention, the plasticizer is selected from the group consisting of a pharmaceutical white oil and a molten petrolatum wax.

It is essential to the successful practice of the invention, that the plasticizer be applied shortly and, in fact, as soon as possible after the molten coating composition has been applied, certainly before any appreciable hardening of the coating film has taken place. This is necessary in order to permit diffusion of the added plasticizer through the coating film on the interior surface of the container to which the plasticizer is applied, so that the plasticizer can become most effectively dispersed in such film.

The invention permits, and contemplates, application of the plasticizer to only an interior surface of the coated container. This is a distinct advantage, since a similar application of plasticizer to exterior coated surfaces is unnecessary. Wax coating on the exterior surfaces of containers are not subject to cracking and flaking from cold shock to the same extent as are similar coatings on interior surfaces, which are directly contacted by the refrigerated liquid during filling. Also, minor imperfections, and the occurrence of some flaking, in the exterior container coating is not as serious as in interior coatings. Furthermore, plasticizers of the nature contemplated herein could normally not be incorporated in the exterior coatings in the amounts hereinafter disclosed, since their application in such amounts would result in the exterior coated surfaces of the containers being too tacky for comfortable manual handling or for trouble-free machine handling.

The white oils which are suitable for use in accordance with the method of this invention are those conventionally known as pharmaceutical grade white oils. These are colorless, odorless, tasteless, and very stable petroleum-derived hydrocarbon oils of relatively low volatility and a wide range of viscosities. Their extremely high purity has rendered them suitable for pharmaceutical and food-stuff applications. In general they are obtained by drastically refining a viscous petroleum fraction. An appropriate fraction of a selected petroleum crude oil, such as a naphthenic crude oil, is first contacted with a concentrated sulfuric acid, or oleum, to react with or selectively dissolve the unstable components of the oil. After removal of the sludge which is consequently formed, residual acidity is neutralized with an alkali, and the inorganic material is removed by washing with a polar solvent such as a lower alcohol or ketone. The oil which has been treated in the above manner is then finished by percolation through clay, or by contact with fuller's earth. Suitable pharmaceutical white oils will have a specific gravity, at 20° C./4° C., of from about 0.825 to about 0.890; a viscosity at 100° F. of from about 50 to about 350 seconds (Saybolt Universal); a closed cup flash point (Pensky-Martens) of from about 310 to about 375° F.; a cloud point of from about 38 to about 52° F.; and a pour point of from about -30° F. to +35° F.

The petrolatum wax which is suitably employed in this invention is the soft, flexible and adhesive microcrystalline wax derived from the heavy, residual lubricating oil fraction of crude petroleum. Generally, this wax will have a melting point within the range of from about 145° F. to about 170° F. (A.S.T.M. D127-49); a viscosity at 210° F. in the range of from about 65 to about 100 Saybolt Universal seconds (A.S.T.M. D88-44); a penetration at 77° F. of greater than about 18 (A.S.T.M. D5-49); a 5% atmospheric distillation point in the range of from about 425° F. to about 460° F., and a percent distilled at 620° F. under atmospheric pressure in the range of from about 50% to about 70%. For the practice of this invention, the petrolatum wax should not contain in excess of about 5.0 weight percent oil, and preferably should contain much less than 5.0 weight percent oil (modified A.S.T.M. D721-47).

Suitable petrolatum waxes are commercially available, and are conventionally made by partially deoiling a so-called "crude petrolatum stock" which is obtained from the dewaxing of a solvent-refined heavy residual lubricating oil fraction. The latter fraction is commonly recovered from the vacuum distillation of a topped petroleum crude oil having suitable characteristics, such as from those of the Pennsylvania and Mid-Continent types. Ordinarily, this residual lubricating oil fraction is solvent refined, prior to dewaxing, to remove its less stable constituents. Dewaxing is accomplished by dilution with a solvent and subsequent chilling to precipitate the wax, the wax then being separated by filtration or centrifuging. The resulting

crude petrolatum stock contains up to 50 weight percent oil, and is the raw material for the less-oily petrolatum waxes employed in practicing the invention. Deoiling is accomplished by adding a polar solvent, after which the mixture is heated to secure complete solution of the oil and wax in the solvent. The solution is then cooled to cause precipitation of the wax, and the relatively less-oily wax is filtered out and washed with fresh solvent. This process of solvent crystallization and filtration can be repeated until a wax having the desired oil content is obtained. The wax is then freed of solvent and treated with clay or some other adsorbent, by contact or percolation methods, to achieve a satisfactory color and odor.

In the practice of this invention it is only necessary that a small amount of the plasticizer be used, and even very small amounts can accomplish a substantial improvement in the resistance of the wax coating to cracking and flaking when properly applied. The amount of plasticizer used will normally exceed about 0.5 weight percent of the quantity of wax coating composition on the surface to which it is applied. However, in no instance should an amount of plasticizer be used which would lower the melting point of the resulting mixture of coating composition and plasticizer to a value below 100° F. In general, the amount of the plasticizer applied to the coating will depend upon the natures of the particular coating composition and plasticizers employed, as well as upon the degree of improvement desired. For instance, when a pharmaceutical white oil is employed, relatively less need be applied than when a petrolatum wax is used. On the other hand, as much as 20.0 weight percent of a petrolatum wax can, on occasion, be used, such as when the petrolatum wax has a melting point in the upper portion of the range previously defined as suitable, and the above-stated amount will not lower the resultant melting point below 100° F. Suitable amounts of the plasticizer can conveniently be determined, by experimentally measuring the melting point of mixtures of the coating composition and plasticizer in contemplated percentages.

Application of the plasticizer to the coating can be by any convenient means which will achieve a substantially even distribution over the surface contemplated. Where the contemplated surface is small, such as the interior bottoms of relatively small containers, dropwise application of the plasticizer with distribution by diffusion through the yet-fluid coating composition may be satisfactory. However, in most instances, application of the molten plasticizer by spraying through a nozzle of suitable spray pattern is preferred, since a more complete and even coverage of the contemplated surface is thus assured. Where coating of the containers is effected in automatic, continuous machinery, the plasticizer application apparatus can conveniently be coordinated with the operation of such machinery so as to apply the desired, predetermined amount at the proper time to the contemplated surface of each container in sequence.

In order to illustrate one specific embodiment of this invention, a milk container constructed of fibrous cellulose sheet material is coated in accordance with the following procedure. The coating composition employed consists of a petroleum paraffin wax having a melting point of about 127° F. (A.S.T.M. D87-57), a penetration at 77° F. of about 16 (A.S.T.M. D1321-57), a viscosity at 210° F. of about 38.8 seconds (Saybolt Universal—A.S.T.M. D446-53), and an oil content of about 0.4% by weight (A.S.T.M. D721-56T). The plasticizer which is used in this embodiment is a petrolatum wax having a melting point of about 160° F. (A.S.T.M. D127-49), a penetration at 77° F. of about 25 (A.S.T.M. D5-49), an oil content of about 5.0% by weight (modified A.S.T.M. D721-47), and a viscosity at 210° F. of about 80 seconds (Saybolt Universal—A.S.T.M. D88-44). In accordance with the method of the invention, the open container is first dipped in a bath of molten coating compo-

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sition to acquire a thin adhering film thereof on the interior and exterior surfaces. After removal of the container from the bath, and the draining off of excess coating composition, but before appreciable hardening of the coating has taken place, the petrolatum wax plasticizer, in the molten state, is sprayed onto the interior surfaces of the container consisting of the four sides and bottom. The amount of plasticizer applied is equal in amount to about 10% by weight of the wax coating composition already on such surfaces. In this amount, the melting point of the combined wax coating composition and added plasticizer is not reduced to a value below 100° F. After cooling of the so-coated container to solidify and harden the coating, it is filled with refrigerated milk at a temperature of about 35° F. It is observed that the wax coating applied in the manner disclosed by this invention is markedly superior in resistance to cracking and flaking on contact with cold liquid over that of containers coated with the same coating composition, but without the application of a plasticizer as herein disclosed.

In another embodiment of this invention, a milk container similar to that employed above is coated with a coating composition consisting of 98% of the same petroleum paraffin wax as that used above and containing, in addition, 2% of a petroleum microcrystalline wax having a melting point of about 193° F. (A.S.T.M. D127-49), a penetration at 77° F. of about 5 (A.S.T.M. D1321-57), a viscosity at 210° F. of about 84 seconds (Saybolt Universal—A.S.T.M. D446-53), and an oil content of about 0.4% by weight (A.S.T.M. D721-56T). After the open container has been dipped in a bath of the above-described molten coating composition, it is removed and the excess wax is permitted to drain off. Before appreciable hardening of the wax coating has taken place, a plasticizer consisting of a pharmaceutical grade white oil having a specific gravity, at 20° C./4° C., of about 0.860, and a viscosity at 100° F. of about 200 seconds (Saybolt Universal) is applied, by spraying, to the interior surfaces of the container consisting of the four sides and bottom. The amount of the plasticizer employed is equal to about 1% by weight of the coating composition already on these surfaces. Application of the white oil in this

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amount does not lower the melting point of the combined coating composition and plasticizer to a value below 100° F. After application of the plasticizer, the coated container is cooled to solidify and harden the coating, and the container is then filled with refrigerated milk at 35° F. It is again observed in this example that wax coatings applied in accordance with the invention are markedly superior in their resistance to cracking and flaking upon contact with refrigerated liquids to those applied with the same coating composition, but without the use of a plasticizer.

The invention claimed is:

1. In a process for applying a protective coating of a petroleum wax to containers for liquids, said process comprising coating said containers with a molten petroleum wax coating composition comprising mainly paraffin wax, and cooling the coated containers to solidify and harden the coating, the improvement which comprises applying to an interior surface of the coated container, after coating and before appreciable hardening of the coating composition, a small amount, sufficient to substantially suppress cracking and flaking of the coating upon contact with refrigerated liquids, of a material selected from the group consisting of a pharmaceutical white oil and a molten petrolatum wax.

2. Method according to claim 1 wherein said material selected from the group is a pharmaceutical white oil.

3. Method according to claim 1 wherein said material selected from the group is a molten petrolatum wax.

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