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LOW MELTING PARAFFIN WAX

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1

The present invention relates to improved petroleum paraffin wax products and more particularly to superior low-melting refined paraffin wax products.

Waxes derived from petroleum are classified broadly as paraffin wax and microcrystalline or amorphous wax. Paraffin waxes which are obtained from distillates have relatively low melting points and low tensile strengths, whereas microcrystalline waxes have relatively high melting points and high tensile strengths. The present invention is not concerned with the microcrystalline waxes but rather relates to the paraffin waxes. Of these there are two general types, the normal or straight-chain paraffin waxes and the non-straight-chain paraffin waxes. For the same boiling point or molecular weight range, the non-straight-chain paraffin waxes have lower melting points than the normal paraffin waxes. For example, as compared to an oil-free straight-chain paraffin wax having an average melting point of 140° F. A. M. P., a non-straight-chain paraffin wax of approximately the same molecular weight has an average melting point of about 119° F. A. M. P. In general, the non-straight-chain paraffin waxes having molecular weights about the same as the normal paraffin waxes having average melting points up to about 165° F. A. M. P. will have melting points below about 125° F. A. M. P. (The term "A. M. P." refers to American Melting Point, as defined in ASTM D37-42). Further, the non-straight-chain paraffin waxes are more readily soluble in selective solvents such as are normally used for solvent deoiling slack waxes obtained from dewaxing waxy lubricating oil stock.

The present invention is directed to improved paraffin wax products containing particular ratios of certain of the above-mentioned straight-chain and non-straight-chain paraffin waxes. An object of this invention is to provide a low-melting paraffin wax composition of superior properties. It is a more particular object of the present invention to provide an improved low-melting petroleum paraffin wax for coating paper products such as milk cartons.

Briefly, we have found that a low-melting refined petroleum paraffin wax of superior properties results from the combination of at least 20, but less than 35 volumes of non-straight-chain paraffin waxes having molecular weights predominately in the range of 350-525, preferably 400-500, and 80 to 65 volumes of straight-chain paraffin waxes having an average melting point of about 127-140° F. A. M. P. The resulting prod-

2

uct has an average melting point within the range of 125-135° F. A. M. P., the average melting point of the straight-chain wax portion alone being sufficiently high to yield in combination with the non-straight-chain wax, a wax product with the desired average melting point. Since each of the components individually have low tensile strengths, the high tensile strength of the above wax composition is an unexpectedly advantageous result.

Our new wax product, in addition to having an increased tensile strength, is less brittle and has improved adhesiveness towards paper products. The improved low-melting paraffin wax product is excellent for coating paper cartons such as those used for milk. With these wax products, strong and waterproof films on milk cartons are obtained with a minimum amount of wax, and the resultant coating has less tendency to crack off than wax coatings heretofore obtained. Thin films of these new waxes are flexible and have high resistance to fracture on sudden chilling. In storage of milk cartons coated with the present waxes, less bulging occurs and fewer leakers are found.

As stated above, the present wax compositions have average melting points in the range of 125-135° F. A. M. P., thus including within the scope of the various wax grades of 125/127, 128/130, 133/135 and the broader grade 125/130.

The wax product of the present invention can be obtained in several ways. For example, each of the desired straight-chain paraffin waxes and the desired non-straight-chain paraffin waxes can be obtained separately or isolated (i. e., completely freed from oil by solvent deoiling) and thereafter combined in the proper ratio. Likewise, concentrates of the desired non-straight-chain paraffin wax in admixture with minor amounts of tolerable normal paraffin wax can be produced and combined in the proper ratio with normal paraffin wax concentrates containing small amounts of non-straight-chain waxes. Preferably, selected slack waxes derived from primary and secondary waxy oils are combined prior to solvent-deoiling, whereby, upon deoiling, the superior wax composition of the present invention is produced. This process, as more particularly set forth in the copending application of Charles J. Halamka and Lynn R. LaPorte, Serial No. 204,806, filed January 6, 1951, includes the following steps of solvent-dewaxing a waxy oil to produce a dewaxed oil of low pour point and a primary slack wax. The primary slack wax is then fractionated into primary wax distillates of

relatively narrow boiling point ranges including a low-boiling fraction which, by solvent-deoiling, yields waxes having an average melting point in the range of 127-140° F. and a higher-boiling, intermediate fraction which, by solvent-deoiling, yields waxes having an average melting point of 135-160° F. A. M. P., preferably at least 5° F. higher than the wax resulting from normal solvent-deoiling of the low-boiling primary slack wax distillate selected. Ordinarily, the selected low-boiling and intermediate higher-boiling primary slack wax distillates are adjacent fractions, although in many cases the boiling range of the higher-boiling fraction will overlap that of the lower-boiling fraction and in some cases advantageously overlaps the whole or up to 50% of the lower boiling fraction. The higher-boiling primary slack wax distillate thereafter is solvent-deoiled to produce a wax product and a foots oil. The foots oil, which is a secondary waxy oil, is then solvent-dewaxed to obtain a high quality lubricating oil component and a secondary slack wax. At least a portion of the secondary slack wax is then combined with the selected low-boiling primary slack wax distillate, and the admixture is solvent-deoiled to produce the desired wax product.

For the purposes of this invention, a waxy oil is an oil, preferably a lubricating oil stock, derived from crude petroleum containing paraffinic compounds which are normally solid at room temperature. The percentage of such petroleum paraffin wax may vary from 1 to 25% in a virgin crude oil, or from 2 to 50% in topped crude oil or distillate fractions or in waxy raffinates obtained from solvent treating or deasphalting distillate fractions or residua. The slack waxes obtained by dewaxing usually contain about 10-60% of a zero ° F. pour point oil, although the oil content can be as low as 2-3%. The wax in the low-boiling distillates of primary slack wax is substantially all (i. e., more than about 90%) normal or straight-chain paraffinic compounds. In the secondary slack waxes as obtained by solvent-dewaxing a foots oil resulting from deoiling a primary slack wax distillate, the wax is predominately (i. e., more than about 75%) non-straight-chain paraffinic compounds. Especially suitable for the present invention are primary slack waxes derived from petroleum residua, including residua such as viscous lubricating oil stocks which are pretreated such as by deasphalting or refining with aromatic-removing solvents or the like.

As indicated hereinabove, the desired non-straight-chain paraffin waxes are concentrated in the slack wax distillate fractions boiling above those primary slack wax distillate fractions which, on deoiling, yield a wax product with an average melting point of above about 130° F. A. M. P. Hence, foots oils obtained in solvent deoiling to produce such higher melting waxes are suitable sources for the non-straight-chain paraffin waxes. Preferably employed are the foots oils obtained in solvent deoiling primary slack wax distillates to produce waxes having an average melting point above about 140° F. A. M. P. and up to about 165° F. A. M. P. As described hereinabove, such foots oil is subjected to solvent dewaxing to produce a low pour point oil and a secondary slack wax which either can be separately deoiled or combined with the selected low-boiling slack wax distillate before deoiling. Also from deoiling broader boiling range primary wax distillates to yield waxes of low average melting point, foots oils are obtained which contain appreciable

amounts of the desired non-straight-chain paraffin waxes; such foots oils can be dewaxed to produce a secondary slack wax which then can be fractionated to separate the upper boiling 30-60% containing the desired non-straight-chain paraffin waxes.

In preparing the improved refined petroleum paraffin wax composition of the present invention, the dewaxing and deoiling operations are carried out by the various well-known methods involving chilling in the presence of solvents or diluents, including, for example, ketones such as methyl ethyl ketone, aromatic hydrocarbons such as benzene, alcohols such as isopropyl alcohol, petroleum naphthas, or mixtures thereof such as methyl ethyl ketone plus benzene. In solvent dewaxing, ordinarily conducted with a filtering temperature of about 0° F. to -15° F., the primary object is to obtain a substantially dewaxed lubricating oil, i. e., an oil with a zero ° F. or lower pour point; also obtained is the primary slack wax. In deoiling, usually conducted with a filtering temperature of about 35-55° F., the primary slack wax is segregated into oil-free wax and foots oil. The primary slack wax is fractionated, at stated above, into relatively narrow boiling distillates, preferably in a vacuum fractionator. While the fractions are segregated ordinarily on the basis of the average melting point of the waxes produced by deoiling the distillates, it can be generally stated for purposes of illustration that a typical primary slack wax derived from a California waxy crude residuum (25-30% from crude) can be suitably fractionated into three cuts: No. 1 cut, No. 2 cut, and No. 3 cut with average boiling points (at atmospheric pressure) of about 800° F., 900° F. and 1050° F., respectively. For comparative purposes, the deoiling of cut No. 1 alone yields a wax having an A. M. P. of about 135-140° F., the lower-boiling 50-60% of cut No. 1 yielding a wax melting at 125-130° F. A. M. P. Deoiling of No. 1 and No. 2 cuts together yields a wax with a 143-150° F. A. M. P.

To illustrate the present invention, a deoiled low-melting straight-chain paraffin wax obtained by solvent deoiling the lower-boiling 50-60% of the above-described No. 1 cut was taken for comparison. This wax had an average melting point of about 129° F. A. M. P. and a tensile strength of about 80 p. s. i. To this was added 30% of a predominately non-straight-chain paraffin wax (i. e., about 80% non-straight-chain wax) obtained by deoiling the secondary slack wax from dewaxing the foots oil produced during deoiling the above-described No. 1 and No. 2 cuts. The resultant blend had an average melting point of about 127° F. A. M. P. and a tensile strength of 263 p. s. i. In another instance, 30 volumes of the above secondary slack wax was added to 70 volumes of the heretofore-described whole No. 1 cut of the primary wax distillate, and the admixture solvent deoiled. The product had an average melting point of about 129° F. and a tensile strength of 298 p. s. i. In another instance, a product was produced by deoiling a blend of 27 volumes of the above secondary slack wax, 63 volumes of the hereinabove-described whole No. 1 cut, and 10 volumes of a third slack wax (containing a 50-50 mixture of non-straight-chain and normal paraffin waxes) obtained by dewaxing the foots oil resulting from deoiling another portion of said whole No. 1 cut. The product had an average melting point of about 129° F. A. M. P. and a tensile strength of about 325 p. s. i.

5

Various ways of producing the improved low-melting wax compositions of the present invention can be employed other than the specific preferred embodiments given above for purposes of illustration. While the present wax compositions consist essentially of petroleum paraffin waxes of the designated types and in the specified ratios, small amounts of additives for various purposes can be incorporated into the final composition. Thus, the term "consisting essentially of" as used in the claims means that the composition is made up almost entirely of the ingredients recited and these ingredients are the main and characterizing ones, but this expression does not exclude the presence of minor amounts of other ingredients which are commonly in wax compositions or which do not change the essential character of the composition. Thus, small amounts (from 0.01 or less up to 1 or 2%) of antioxidants such as various phenolic antioxidants, e. g., 2,6-di-tertiary-butyl 4-methyl phenol, tensile strength improvers such as microcrystalline wax, and the like can be incorporated in the final wax composition.

We claim:

1. A substantially oil-free paraffin wax composition having an average melting point within the range of 125-135° F. A. M. P. which consists essentially of at least 20% but less than 35% of non-straight-chain distillate paraffin waxes having molecular weights predominately in the range of 350-525 and the remainder being straight-chain distillate paraffin wax having an average melting point within the range of 127-140° F. A. M. P. and an average molecular weight substantially below that of said non-straight-chain wax.

2. A substantially oil-free paraffin wax composition having an average melting point within the range of 125-135° F. A. M. P. which consists essentially of at least 65% of a straight-chain distillate paraffin wax having an average melting point of 127-140° F. A. M. P. and at least 20% of a non-straight-chain distillate paraffin wax having an average melting point below about 125° F. A. M. P. and an average molecular weight of about 400-500 said straight-chain wax having an average molecular weight substantially below that of said non-straight-chain wax.

3. A substantially oil-free paraffin wax composition having an average melting point within the range of 125-135° F. A. M. P. and having a high tensile strength which consists of at least 20% but less than 35% of a paraffin wax selected from non-straight-chain distillate paraffin waxes having molecular weights predominately in the range of 350-525, and the remainder consisting essentially of straight-chain distillate

6

paraffin wax having an average melting point within the range of 127-140° F. A. M. P. and an average molecular weight substantially below that of said non-straight-chain wax, the average melting point of said straight-chain wax being sufficiently high to yield an improved tensile strength wax product with the desired average melting point within the range of 125-135° F. A. M. P.

4. A substantially oil-free paraffin wax composition having an average melting point within the range of 125-135° F. A. M. P. and having a high tensile strength which consists essentially of 65-80% of a straight-chain distillate paraffin wax having an average melting point of about 127-140° F. A. M. P. and at least 20% but less than 35% of a non-straight-chain distillate paraffin wax having an average molecular weight in the range of 400-500 said straight-chain wax having an average molecular weight substantially below that of said non-straight-chain wax.

5. The paraffin wax composition of claim 4, wherein the non-straight-chain paraffin wax is obtained from a secondary slack wax produced upon solvent dewaxing a foots oil resulting from solvent deoiling a primary slack wax distillate which by deoiling yields a wax product having an average melting point above about 140° F. A. M. P.

6. A substantially oil-free distillate paraffin wax composition of high tensile strength and having an average melting point within the range of 125-135° F. A. M. P. which consists essentially of 20 to 35% of non-straight-chain distillate paraffin wax having molecular weights predominantly in the range of 400-450 and the remainder being straight-chain distillate paraffin wax having an average melting point within the range of 127-140° F. A. M. P. and an average molecular weight substantially below that of said non-straight-chain wax, said straight-chain wax alone having a tensile strength substantially lower than the final composition.

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