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WAX COMPOSITIONS

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The present invention relates to petroleum wax compositions, and more particularly to the production of petroleum wax of high tensile strength from paraffin wax of low tensile strength.

In the refining of lubricating oils from petroleum two general types of waxes are ordinarily removed during the refining procedure. These are known, respectively, as crystalline or paraffin wax and amorphous wax. Crystalline wax, which is characterized by a definite crystalline structure and hard brittle texture, is obtained from the distillate or wax distillate fraction resulting from distillation of the crude, by familiar deoiling methods such as chilling and sweating, and subsequent refining operations, to produce a highly refined non-oily white product. Amorphous wax, as distinguished from the crystalline variety, is characterized by a higher boiling point, usually a higher melting point and less brittleness, and is derived from the residual crude distillation fraction, i. e., the fraction from which heavy lubricating oils are obtained, usually by blending the residual fraction with petroleum naphtha and centrifuging the resulting solution to separate oil. The resulting oily wax, which may contain 40% or more of oil, may be again put in naphtha solution and centrifuged to remove further quantities of oil. The resulting product, sometimes termed petrolatum wax, still contains considerable amounts of oil, generally at least about 5% or 6%.

It has been reported that blends composed of paraffin wax and petrolatum wax derived as above described from certain mixed base crudes exhibit tensile strengths at room temperatures materially greater than that of the paraffin wax alone. However, the addition to paraffin wax of petrolatum wax similarly derived from predominately paraffin base crudes such as Pennsylvania crudes does not result in material increase in tensile strength over that of the unblended paraffin wax.

According to the present invention, it has been found that the tensile strengths of such paraffin waxes can be materially improved by incorporation therein of solvent deoiled amorphous wax. As distinguished from the centrifuge petrolatum wax referred to hereinabove, which contains at least 5 or 6% of oil, the solvent deoiled amorphous waxes of this invention may be considered substantially oil-free in that they contain generally no solvent extractable amount of oil. These solvent deoiled waxes may be prepared by treatment of a suitable residual petroleum fraction or waxy material derived therefrom such as the centrifuge petrolatum wax referred to above, with powerful oil-solvent mixtures under conditions effective to extract or remove the oil from the wax. Such solvent deoiling methods and procedures will be described in more detail hereinbelow.

Stated in other words, the invention resides in wax compositions comprising blends of paraffin and amorphous wax sufficiently low in oil content as to have tensile strengths greater than those of the paraffin wax constituents alone. The effect of small additions of solvent deoiled amorphous wax upon the tensile strength of paraffin wax and also the detrimental effect on tensile strength of the presence of small quantities of oil can be appreciated from the following tabulated comparative data. Both waxes shown were derived from Pennsylvania crude.

Sample No.	Wax composition	Perkins tensile strength @ 70° F.
A	Paraffin wax (M. P. 135° F.)	53
B	Sample A plus 2% solvent deoiled amorphous wax (M. P. 149° F.)	70
C	Sample B plus 0.3% bright stock	42
D	Sample B plus 0.6% bright stock	30

It can be readily seen from the table that the addition of relatively small amounts of solvent deoiled amorphous wax greatly increases the tensile strength of paraffin wax. Likewise the effect of quite small quantities of oil in counteracting the strengthening properties of solvent deoiled wax is clearly evident. Addition of as little as 0.3% oil (sample C) reduced the strength of the blend below that of the paraffin wax.

Another important characteristic of the wax compositions of this invention is their relatively high strengths at low temperatures as, for example, at temperatures from about 50° F. down to 0° F., and particularly after being rapidly chilled to said temperatures. Sudden chilling of paraffin wax results in formation of cracks with consequent weakening of the structure, making such wax objectionable for such uses as wax coatings for paper milk containers which are filled with cold milk at 40 to 50° F. and for waxing paper used in packaging quick-frozen foods at about 0° F. to about 10° F. The wax blends of this invention are particularly suited for such uses inasmuch as they are characterized by reduced cracking and by high strength when subjected to chilling as shown in the following table.

50	Per cent 125° F. M. P. crystalline wax	100	99	95	90
	Per cent solvent deoiled amorphous wax	0	1	5	10
	Perkins tensile strength, pounds, at—				
	70° F.	47	66	74	83
	50° F.	46	86	89	94
	40° F.	27	66	80	97
55	32° F.	10	36	70	80
	8° F.	7	7	8	60
	0° F.	7	7	8	60

This data indicates that incorporation of solvent deoiled amorphous wax in amounts as low as

1% will impart ample strength to crystalline wax at 40-50° F. while 10% will provide satisfactory strength at 0° F.

It will be understood that proportional amounts of solvent deoiled wax shown in the blends tabulated hereinabove are merely illustrative of the invention but not limitative thereof. Other proportional amounts may be used within the spirit and scope of the invention. For example, blends consisting of about 50% crystalline wax and about 50% amorphous wax have exhibited uniform or even increasing tensile strengths at test temperatures of 0° F. over their strengths at 70° F. A particular advantage resides in the fact that the strengthening effect is particularly marked at very low concentrations of solvent deoiled amorphous wax. For example, the addition of as little as 0.1% of this amorphous wax improves the tensile strength of crystalline wax about 40% at room temperatures in certain instances.

The solvent deoiled amorphous wax employed in the above blends was produced from petrolatum derived by centrifuge deoiling of a naphtha diluted residual lube oil fraction of Pennsylvania crude, by first solvent deoiling said petrolatum at -10° F. with a mixture consisting of 40% methyl ethyl ketone, 35% benzol and 25% toluol which resulted in a wax containing substantially no oily material of 25° F. pour point or lower, and then further solvent deoiling the resulting wax at 47° F. with a similar mixture of solvents. In some instances, depending upon the particular wax source and the thoroughness of the solvent deoiling method used, a single solvent treatment will suffice. Generally, however, stronger blends will obtain when the amorphous wax constituent is produced by repeated solvent deoiling.

Likewise, the amorphous wax constituent may be produced from sources other than centrifuge petrolatum. Thus, for example, amorphous wax resulting from solvent deoiling of cylinder stock is satisfactory.

The tensile strength test used is that described on pages 7 to 9 of U. S. Department of Interior, Bureau of Mines Bulletin No. 388, and consists briefly in applying a lineal pull to a test specimen of ¼ square inch cross-sectional area of the wax to be tested, using as the testing machine the Perkins tester described in said Bulletin, and noting the force in pounds reached at failure of the

test specimen. The procedure set forth in the stated Bulletin was followed exactly except that tensile strengths at temperatures below 70° F. were determined at the temperatures noted in the tables instead of at 70° F. on specimens which had been chilled from room temperature to the test temperatures by immersion in cold liquid immediately prior to testing. All of the wax-blend specimens were prepared by melting together the designated weight proportions of crystalline and solvent deoiled amorphous wax, stirring to a uniform blend and solidifying in the dumbbell-shaped molds shown in the mentioned Bulletin. The test specimens containing only crystalline wax were prepared by melting crystalline wax and then solidifying in the stated molds.

Another disadvantageous characteristic of crystalline wax which is eliminated by incorporation of solvent deoiled amorphous wax is that of "mottling," by which is meant formation of air or gas bubbles in the wax structure. This is particularly undesirable in wax used for candle making. Crystalline wax blends containing about 1% or more by weight of solvent deoiled amorphous wax do not exhibit mottling.

Other properties, desirable in crystalline wax compositions, such as color stability on heating and hardness, are not materially affected by the presence of the solvent deoiled amorphous wax constituent in tensile strength increasing proportions.

I claim:

1. A composition comprising oil-free crystalline paraffinic petroleum wax normally tending to crack upon sudden chilling to sub-atmospheric temperatures and having incorporated therein an amount of solvent-deoiled, substantially oil-free paraffinic amorphous wax effective to inhibit said tendency of the crystalline wax to crack at said low temperatures, said amorphous wax being prepared from conventional centrifuge wax from which the usual 6-10% residual oil has been removed by powerful oil-solvent mixtures.

2. Method for inhibiting cracking of substantially oil-free crystalline petroleum wax upon sudden chilling to sub-atmospheric temperatures which comprises incorporating therein a minor portion of a solvent-deoiled amorphous wax derived from a predominantly paraffinic base crude, said amorphous wax being substantially oil-free.

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