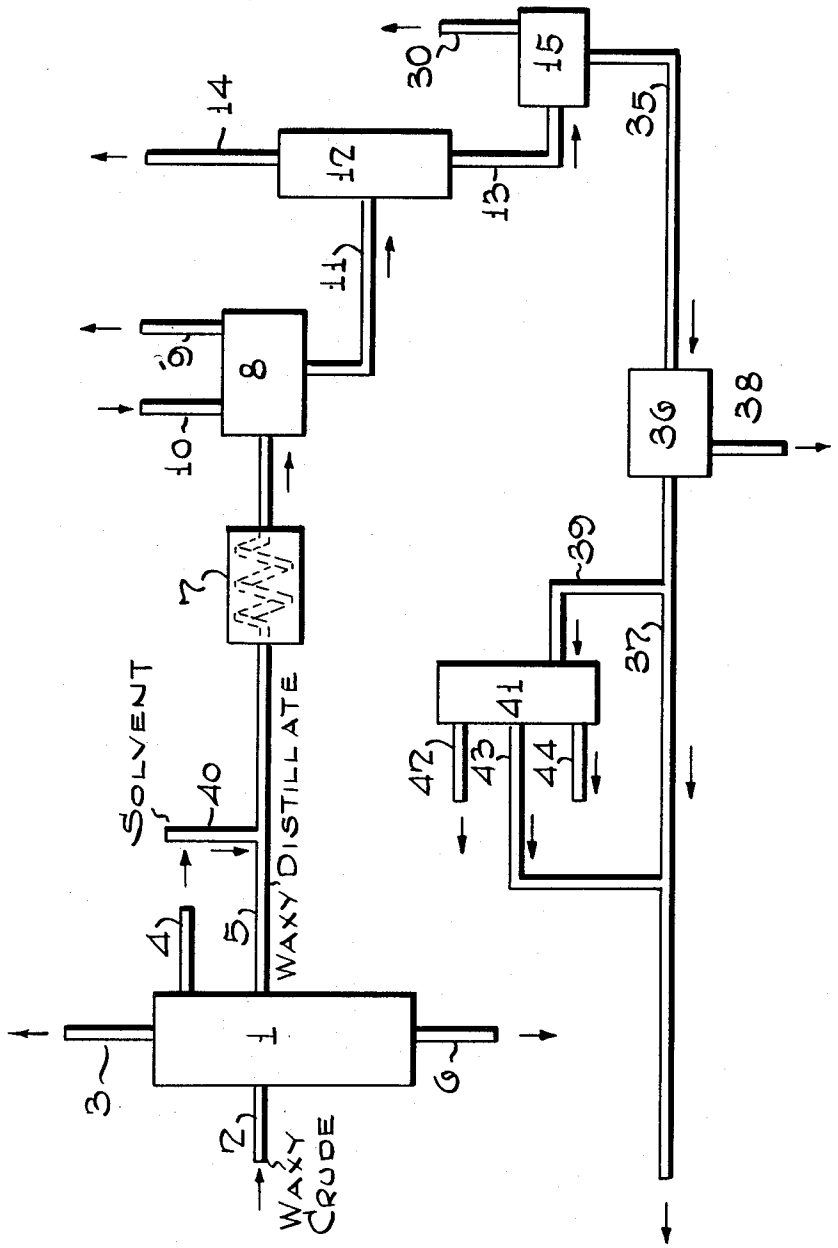
July 24, 1956 S. F. PERRY ET AL 2,756,180

PARAFFIN WAX COMPOSITION FOR PAPER COATING
AND PROCESS OF MAKING SAME
Filed June 12, 1952




Stephen F. Perry Inventor
Herman J. Zoeller
By W. D. Feilmon Attorneys

United States Patent Office

2,756,180

Patented July 24, 1956



2,756,180

# PARAFFIN WAX COMPOSITION FOR PAPER COATING AND PROCESS OF MAKING SAME

Stephen F. Perry, Westfield, and Herman J. Zoeller, Union, N. J., assignors to Esso Research and Engineering Company, a corporation of Delaware

Application June 12, 1952, Serial No. 293,176

4 Claims. (Cl. 196—17)

The present invention is concerned with an improved wax composition which is particularly adapted for the coating of fibrous substances such as paper and the like. In accordance with the present invention, an improved wax composition is secured by the blending of a narrow cut distilled refined wax with a wide cut sweated refined wax. Preferred compositions are secured when the average melting points of the respective fractions are approximately equivalent. The wax composition of the present invention has a high blocking point and a high tensile strength, a combination not secured in either component alone.

In the refining of hydrocarbon oils such as petroleum oils, it is known to segregate paraffin waxes from so-called paraffin distillates, waxy lubes and the like. The segregation of these waxes is secured by a number of processes. For example, it is known to chill the selected wax containing fraction in order to secure crystallization of the wax and to remove the wax crystals from the oil by filtering, centrifuging and the like. It is also known to use various dewaxing solvents such as liquid normally gaseous hydrocarbons, such as propane, as well as other solvents, such as methylethyl ketone and the like. It is also known to utilize in dewaxing operations solvent mixtures wherein one solvent comprises a wax precipitating solvent while the other comprises a solvent having a high solubility for oil. A solvent mixture of this character, for example, comprises 60% by volume of toluene and 40% by volume of methylethyl ketone. In utilizing a mixture of this character, it has been the practice to add the mixture in toto or incrementally to the waxy distillate as it is being chilled. In dewaxing operations, it is also known to use various filter aids and other agents in order to render the dewaxing and filtering operations more efficient.

The wax segregated from the hydrocarbon oil, usually termed "slack wax," contains from about 10% to 40% of oil. The slack wax is refined usually by conventional sweating to produce "crude scale wax" in a manner to reduce the oil content to less than about 5% by weight. The slack wax may be distilled to obtain the desired boiling range wax prior to sweating, if desired. This "crude scale wax" generally has an oil content of about 2% to 3% by weight. In order to remove this oil from the scale wax to produce a refined wax having an oil content below about .5%, usually below about .3%, various procedures have been proposed and employed.

It is also known in the art to prepare wax compositions adapted for various commercial uses by blending various fractions and the like. For example, in the manufacture of wax compositions suitable for the coating of paper, it



is essential that the wax have a high tensile strength and also have a high blocking point.

The tensile strength is determined by a Tinius Olsen machine. The wax to be tested is heated to about 230° F. with constant stirring to remove all traces of moisture. The hot wax is poured into "dumbbell" shape flat molds, filling each mold as full as possible without overflowing. The filled molds are allowed to age for two hours in a constant temperature (70° F.) controlled humidity (50%) room. The wax briquettes which have a cross-sectional area of ¼ sq. in. at the narrow, center section, are removed from the molds without distorting and placed in a water bath at 70° F. before testing. Each briquette is placed in the holder at the base of the balance on the Tinius Olsen machine and sufficient tension applied to the briquette to allow the pointer arm to coincide with the rest point. The tension applied is equal to about 2.5 lbs. The shot control slot is forced open and the shot allowed to flow into a pan resting on a scale. At the instant of rupture the flow of shot will be cut off automatically. The weight of shot is recorded. A total of eight briquettes are ruptured in this manner and those results which check within 10% are averaged. The final average value is added to the correction for balancing the beam (2.5 lbs.). This sum is multiplied by four and the result is expressed as tensile strength—lbs./sq. in.

The wax blocking test is made as follows:

Two-inch wide strips of breadwrap paper (25 lb./ream) are hand coated with wax at about 175° F. and then chilled in a water bath at 40–55° F. to give a wax consumption of about 20 to 30 lbs. of wax/ream. The waxed strip is folded end to end and cut 17½" from fold. A one-inch wide center slice is cut from the folded strip. The waxed paper specimen is inserted between two unwaxed paper strips and placed on the blocking plate (a metal plate across which a temperature gradient is maintained), with the folded end on a prescribed line at the hot end of the plate. Pressure is applied to each strip by covering it with a one-inch square steel bar, cushioned by sponge rubber pads so as to apply the pressure evenly. After exposure on the blocking plate for seven hours, the strips are removed, cooled and carefully pulled apart starting at cool (unfolded) end. The distance to the beginning of the area of complete blocking (fusing together of the wax) is measured from either end of the strip. This distance is converted to the blocking temperature by means of a calibration curve relating temperature to distance along the plate. Average blocking values on a given wax generally agree within 1° F., with a maximum difference of 2° F. This test is conducted in a room held at constant temperature (70–73° F.) and constant humidity (50%).

A high blocking point is desirable since this is a measure of the minimum temperature at which the wax coated paper sticks when two sheets are held together under pressure and then pulled apart. Wax surface is marred at the blocking point or higher temperature. The narrow the boiling point and melting point range of the distilled product, the higher the blocking point of the wax coated paper stock.

Associated with increased blocking point, attributed to the narrow cut paraffin wax, there is also a considerable decrease in the tensile strength of the wax. A high tensile strength is desirable from a sales appeal and con-

sumer's point of view since tensile strength appears to be related to the durability of the wax surface on the paper. That is, the higher the tensile strength of the wax and more scuff resistant the wax surface and the higher the sealing strength of the paper, with no "flaking off" on the cut edge. A wide cut paraffin wax possesses good tensile strength but has a low blocking point.

Both desirable properties can be attained to a considerably greater degree in a new product by a process which involves the blending of the narrow cut distilled wax with a wide cut sweated wax preferably of the same average melting point.

The present invention will be more fully appreciated by reference to the drawing illustrating one embodiment of the same.

Referring specifically to the drawing, a waxy crude oil is introduced into distillation zone **1** by means of feed line **2**. Temperature and pressure conditions in zone **1** are adapted to remove overhead by means of lines **3** and **4** low boiling hydrocarbon fractions. A waxy distillate fraction is removed as a side stream by means of line **5**, while a residual oil is removed by means of line **6**.

The waxy distillate is mixed with solvent introduced by means of line **40** and flows through a chilling zone **7** which may comprise a plurality of chilling stages wherein the temperature of the waxy distillate is progressively reduced. A typical operation is to introduce the feed oil into an initial chilling stage at a temperature of about 130° F.; to introduce the feed oil into the second chilling stage at a temperature of about 90° F.; to introduce the feed oil to the third chilling stage at a temperature of 60° F.; to introduce the feed oil to the fourth chilling stage at a temperature of about 25° F. and to chill the same in the fourth chilling stage to a temperature in the range from about −10 to +10° F.

The operation of the respective chilling stages may be varied appreciably and either direct or indirect chilling means utilized. For purposes of illustration, it is assumed that a solvent mixture is used and that the wax-precipitant comprises methylethyl ketone and that the aromatic solvent having a high solubility for oil comprises toluene. It is also assumed that 3 to 4 volumes of total solvent mixture is utilized per volume of waxy oil being dewaxed. The solvent mixture comprises 75% by volume of methylethyl ketone and 25% by volume of toluene. The wax crystals in a slurry of solvent and oil are passed to filtering zone **8** wherein the solid wax particles are segregated from the oily constituents and solvent by any suitable filtering or separation means. The filtering zone may comprise drum filters, plate-and-frame presses, centrifuges or suitable equivalent equipment for the separation of the precipitated waxy constituents from the oily constituents. The oil and a portion of the solvent is removed from zone **8** by means of line **9** and the wax cake is preferably washed with a wash solvent introduced into filtering zone **8** by means of line **10**. Slack wax and solvent are removed from zone **8** by means of line **11** and passed to a distillation zone **12** wherein a separation is made between the wax and the solvent. It is to be understood that other means of separating the solvent from the wax may be utilized if desirable.

The wax substantially free of solvent is removed from distillation zone **12** by means of line **13**. The solvent mixture comprising methylethyl ketone and toluene is removed overhead from zone **12** by means of line **14** and preferably recycled to the system. The oil-solvent mixture removed from filtering zone **8** by means of line **9** is introduced into a distillation zone wherein a separation is made between the oily constituents and the solvent mixture.

While the drawing illustrates a solvent dewaxing operation with respect to the production of the slack wax, it is to be understood that the waxy constituents may also be separated in a conventional plate-and-frame pressing operation. The slack wax may be further refined to crude scale wax in zone **15**, which comprises a conventional sweating operation, or solvent deoiling operation, or the like. The oil removed from zone **15** by means of line **30** may preferably be recycled to zone **7**. The scale wax is removed from zone **15** by means of line **35** and may be further refined by treatment with sulfuric acid or fuller's earth, bauxite or other absorbent materials, or by hydrogenation under mild treating conditions.

The scale wax removed from sweating zone **15** containing from about 2% to 3% of oil by weight is passed by means of line **35** to a sweating or deoiling zone **36** wherein the oil content is reduced below about .5%, usually to below about .3%. The finished refined wax of this oil content is removed by means of line **37** while the segregated oil is removed by means of line **38**. This refined wax has a relatively wide boiling range as for example in the range from about 320° F. to 585° F. (All boiling ranges herein are at 10 mm. mercury absolute pressure.) In accordance with the present invention, a portion of this wax is segregated by means of line **39** and passed to a wax distillation zone **41**. Temperature and pressure conditions are adjusted in zone **41** to segregate relatively narrow cut refined wax fractions, as for example a low boiling wax fraction boiling in the range from about 320° to 480° F., an intermediate boiling wax fraction boiling in the range from about 480° to 520° F., and a high boiling wax fraction boiling in the range from about 520° to 585° F. The low boiling wax fraction is removed by means of line **42**, the intermediate boiling wax fraction is removed by means of line **43**, while the high boiling wax fraction is removed by means of line **44**. In accordance with the present invention the narrow cut wax fraction, preferably the intermediate boiling range narrow cut wax fraction, is blended with the wide boiling refined wax removed by means of line **37**. It is preferred that the amount of narrow cut wax utilized be in the range from about 40–70%, preferably in the range from about 40–60% by weight based upon the total wax.

The present invention is broadly concerned with an improved wax composition comprising a refined wax of a relatively wide boiling range such as 320° F. to 585° F., having an oil content below about 1%, preferably below about 0.5%, in combination with a narrow cut refined distilled wax fraction such as 480° F. to 520° F. which preferably has an average melting point substantially equivalent to the melting point of the wide cut fraction. The narrow cut distilled fraction has an oil content below about 1%, preferably below about 0.5%. In general, the spread between the initial and final boiling points of the wide boiling wax fraction is from about 200° F. to 300° F., while the spread of the narrow cut fraction is from about 30° F. to 100° F. A wax composition of this type is particularly adapted for the coating of paper or other fibrous materials since it has a high blocking value and a high tensile strength. While the invention has been described with respect to securing the narrow cut fraction by distillation, it is within the concept to produce the narrow cut fraction by other segregation means, such as by fractional crystallization or by the use of selective precipitating solvents to separate narrow cut wax fractions.

The process of the present invention will be more fully understood by the following example illustrating the same:

## EXAMPLE

A sweated wax having an oil content below about .5% was tested and its properties compared with the desired characteristics of a paper coating wax. A portion of this wax was distilled to produce a narrow cut wax fraction. The waxes were then blended and the properties determined. The wax compositions produced met the required

properties of a paper coating wax. The results of these operations are as follows:

*Paraffin wax composition for paper coating*

IMPROVEMENT IN PHYSICAL PROPERTIES OF PARAFFIN WAX BY BLENDING NARROW AND WIDE BOILING POINT WAX FRACTIONS

| Inspections | Desirable Physical Properties | Feed to Distillation [1] | Narrow Cut Distilled Wax | Blends of Narrow Cut and Feed | |
|---|---|---|---|---|---|
| | | | | [3] 65 [4] 35 | [3] 50 [4] 50 |
| Melting Point, °F. | 130–132 | 132.0 | 131.0 | 131.0 | 131.5 |
| Tensile Strength, p. s. i. @ 70° F. | 300 (min.) | 390 | 70 | 270 | 300 |
| Blocking Point, °F [2] | 100 (min.) | 95 | 109 | 102 | 100 |
| Boiling Point Range, °F. (5–95%) @ 10 mm. Mercury Absolute Pressure | | 320–585 | 480–520 | | |

[1] Sweated paraffin refined wax.
[2] Minimum temperature at which two strips of wax coated paper stick together when pressed under standard test conditions and then pulled apart. A marred wax surface is evident at blocking point.
[3] Wt. percent N. C.
[4] Wt. percent feed.

What is claimed is:

1. Process for the manufacture of a wax composition suitable as a fibrous coating material which comprises removing waxy constituents from a waxy petroleum oil, treating said waxy constituents to reduce the oil content to below about 0.5% and to produce a wax fraction consisting essentially of constituents having a boiling range of about 320° F. to about 585° F., at 10 mm. mercury absolute pressure, segregating said wax fraction into an initial and into a secondary stream, distilling said initial stream to segregate an intermediate narrow wax cut consisting essentially of constituents having a boiling range of about 480° F. to 520° F. at 10 mm. mercury absolute pressure, blending said narrow wax cut with said secondary stream boiling in the range from about 320° F. to 585° F. in an amount of about 40% to 60% by weight based on the total of the first and second waxes.



2. An improved wax composition of an oil content of less than about 1% and which is suitable for the coating of fibrous materials which comprises a first refined paraffin wax consisting essentially of constituents having a boiling range of about 320° F. to about 585° F. at 10 mm. of mercury absolute pressure and a second refined paraffin wax consisting essentially of constituents having a boiling range of about 480° F. to about 520° F. at 10 mm. of mercury absolute pressure, the average melting points of said wax constituents being substantially equal.

3. The composition of claim 2 wherein the average melting points of said wax constituents are about 130°–132° F.

4. The composition of claim 2 wherein the amount of said first wax constituent is in the range of from about 40% to 60% by weight based on the total of the first and second waxes.

**References Cited** in the file of this patent

UNITED STATES PATENTS

| | | |
|---|---|---|
| 2,467,959 | Bowman et al. | Apr. 19, 1946 |
| 2,603,589 | Schaerer | July 15, 1952 |
| 2,611,734 | Ridenour | Sept. 23, 1952 |
| 2,624,501 | Ferris | Jan. 6, 1953 |
| 2,670,318 | Halamka et al. | Feb. 23, 1954 |
| 2,670,323 | Hunter et al. | Feb. 23, 1954 |