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METHOD OF TREATING REFINED SUGAR CANE WAX AND PRODUCT RESULTING THEREFROM

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This invention relates to a method of treating refined cane wax and the product resulting therefrom. More particularly the invention relates to a method of decolorizing, deodorizing, increasing the gel strength and improving other physical qualities of refined cane wax and the new and improved wax produced by such method.

When the sugar juices are expressed from cane and cleaned by sedimentation and filtration, a residue, known as cachaza, is produced. It is from this cachaza that crude cane wax is extracted, as by means of a solvent extraction process taught by F. H. Rhodes et al. in U. S. Patent No. 2,428,813.

The crude cane wax has essentially three components; a hard wax fraction, a soft or fatty fraction and a resinous fraction. Whereas uses for the hard wax fraction have been appreciated commercially, it must first be separated from the soft, fatty fraction. This is known as "defatting." Balch, in U. S. Patent No. 2,381,420, accomplishes this step through the diffusion of the soft fraction in acetone. A more expedient process was set forth by Swenson, U. S. Patent No. 2,456,656. After the soft fraction has been eliminated, it has been found expedient for most intended uses, that the dark pitch-like resinous material be extracted from the hard wax fraction and such methods are referred to as "deresination processes." McLoud, in U. S. Patent No. 2,456,641, teaches elimination of the resinous component by means of a lower alcohol. Deresination, prior to the separation of the hard from the soft fraction, is set forth by Wilder in U. S. Patent No. 2,456,661.

Whereas the wax on the surface of the cane is hard and pale in color, upon being extracted from the cachaza and defatted, the resulting hard fraction wax is brown-black in color. Even after deresination, it is a dark green-brown. The hard fraction wax, having been in contact with all of the constituents of the plant and the products of bacterial and mold attack, retains a most pungent and moldy odor. Then, too, in this condition, the wax cannot be used conventionally as, for example, in a polish, since it has substantially no gel-forming power, but after admixture with a hot solvent, precipitates upon subsequent cooling and much of the solvent separates as a clear supernatant liquid. Furthermore, when not properly deresinated, I have found that conventionally-refined hard fraction wax, when admixed with paraffin hydrocarbons, precipitates a sludge. This is a decisive impediment to the use of the wax in commercial applications which demand total or nearly-total miscibility.

In order to overcome the above-described limitations, treatment or purification of the wax through the use of conventional organic bleach materials and bleaching processes has been investigated. Where the wax so treated has not been first properly deresinated, the black gummy resin impairs color change. In addition, the resultant product has little or no gel-forming potency. Even in the case where the wax has been first properly deresinated, we have found that treatment with the conventionally-advocated chemical bleaches and bleach processes causes a change in color to a dull reddish hue. Furthermore, these bleaches have demonstrated a deteriorating effect on the wax, reducing the molecular structure so that the waxes are soft and tacky and should they be incorporated in a conventional type wax-polishing composition, upon buffing the applied film, substantial smear and tackiness is evident upon the treated surface.

In an effort to eliminate the undesirable color and odor of the hard fraction wax and derive a modified wax which has strong gel-forming power, we have discovered a method of treating refined surgar cane wax having a soluble content of at least 97 per cent by weight in boiling isopropanol, which comprises melting the wax, maintaining the wax at a temperature of from 80° C. to 150° C. while introducing oxygen-containing gas into the wax until the acid number of the wax is increased to at least 20, but not more than 35, and saponification number is increased to at least 45, but not more than 75. Agitation of the molten wax is simultaneously conducted while the oxygen-containing gas is contacted therewith.

We have also discovered, as a result of the employment of the above process, a new and improved cane wax being at least 97 per cent soluble by weight in boiling ispropanol, which wax is substantially odorless, a pale beige in color, has an acid number of from 20 to 35, a saponification number of from 45 to 75 and strong gel-forming characteristics.

Now, having indicated in a general way, the nature and purpose of this invention, the following examples will illustrate the invention. It is to be understood, however, that such examples are presented merely as illustrations of the invention, and are not construed as limiting the same.

Example 1

Employed in this example was 100 pounds of hard fraction sugar cane wax which had been deresinated to the extent that at least 97 per cent by weight of the wax was soluble in boiling isopropanol and which bore the following characteristics:

1. Color—dark green brown
2. Odor—pungent, moldy
3. Acid No. 9
4. Saponification No. 31
5. Gel strength.—10 per cent by weight of hard fraction cane wax and 10 per cent carnauba wax blended with 80 per cent hot naphtha, cooled, resulted in a mixture which was thoroughly fluid.

The 100 pound charge was melted in a suitable vessel having a diameter of 22 inches by heating to a temperature of approximately 92° C. While the molten wax was maintained at that elevated temperature, air was introduced into the wax in the bottom of the vessel by means of a gas dispersion tube at a rate of approximately 450 cubic feet per hour. Simultaneous with the entrance of air into the vessel, mechanical agitation was conducted immediately above the air entrance. The exothermic reaction was continued for approximately one and one-half hours after which the wax having attained an acid number of 20 was cooled. This wax bore the following characteristics:

1. Color—pale tan yellow
2. Odor—substantially deodorized
3. Acid No. 20
4. Saponification No. 47
5. Gel strength.—A paste wax composition comprising 10 per cent by weight of the treated cane wax, 10 per cent carnauba and 80 per cent naphtha was produced. The paste indicated a cone penetrometer value of 192.4.

Example 2

100 pounds of hard fraction sugar cane wax which had been deresinated to the extent that it was 95 per cent soluble by weight in boiling isopropanol, and otherwise having substantially the same original characteristics of the wax treated in Example 1, was processed exactly as was the wax in Example 1. The resultant wax indicated the following characteristics:

1. Color—red-brown
2. Odor—substantially deodorized
3. Acid No. 20.4
4. Saponification No. 31
5. Gel strength.—The paste wax composition was produced with 10 per cent by weight of this wax, 10 per

cent carnauba and 80 per cent naphtha. The paste wax had a cone penetrometer reading of 216.4.

Example 3

100 pounds of hard fraction sugar cane wax which had been deresinated to the extent that at least 97 per cent by weight of the wax was soluble in boiling isopropanol, and which bore identical characteristics to that treated in Example 1, was treated with the process set forth in Example 1. However, the exothermic reaction was allowed to continue for three and one-half hours at which time the wax attained an acid number of 36. After the treated wax was cooled, it was examined and found to have the following characteristics:

1. Color—pale beige
2. Odor—substantially deodorized
3. Acid No. 36
4. Saponification No. 69
5. Gel strength.—A paste wax composition comprising 10 per cent by weight of the treated cane wax, 10 per cent carnauba and 80 per cent naphtha was produced. The paste indicated a cone penetrometer value of 101.0.

Example 4

Twenty grams of hard fraction sugar cane wax which had been deresinated to the extent that it was at least 97 per cent soluble in boiling isopropanol was melted in a suitable vessel with twenty grams of paraffin oil. The mixture was thoroughly stirred and poured into an aluminum evaporating dish, filling it to the brim. After the mixture had cooled to room temperature, it was allowed to stand for approximately four hours at 25° C. Using a needle carrying a 300 gram weight, five needle penetrometer readings were taken of this mixture and the resultant average reading was 59.

Twenty grams of hard fraction sugar cane wax, deresinated to the extent that at least 97 per cent by weight of the wax was soluble in boiling isopropanol was processed in the manner set forth in Example 1. The treated wax was melted in twenty grams of paraffin oil, cooled, and allowed to stand at a temperature of approximately 25° C. for approximately four hours. Five needle penetrometer readings were taken, a 300 gram weight being carried by the needle and the mean penetrometer value was 45.

Example 5

Seven grams of hard fraction sugar cane wax, deresinated to the extent that the wax was at least 97 per cent soluble in boiling isopropanol, processed exactly as described in Example 1, was dissolved by heating at 100° C. in 79 parts petroleum oil having a Saybolt viscosity of 100 seconds at 100° F. 14 parts of powdered carbon black was stirred into the hot liquid. Agitation was continued until the resultant ink product appeared to be homogeneous, and then one drop was placed on the surface of the slide on a microscope stage and then covered with a glass. A shearing action was exerted on the sample drop by causing a rotary pressure on the cover glass with the points of tweezers. This rotary shearing action is continued. The wax under test (observed through a microscope using a magnifying power of 200X) demonstrated good flow-producing properties since the aggregates of carbon black rapidly broke down under the shearing action. The background of the field and the smaller carbon particles themselves appeared a dark brown in color. The particles did not become aligned into chains or attached to one another as aggregates when the shearing action was stopped, but continued to move about in the liquid medium independent of one another.

The carbon dispersion test, as outlined above, was repeated, the same materials and quantities thereof being used, with the exception that the hard fraction wax employed, although deresinated, was not treated in the manner described in Example 1. When tested under a microscope, it was noted that the carbon particles flowed past one another only as long as the shearing action was exerted, but when the action was stopped, the particles immediately became aligned into rows so that the field might be said to have the appearance of a sand beach on which the sand has been washed into shallow rills by the action of the waves.

It is mandatory to the successful operation of the process described herein that not only must the sugar cane wax to be treated by exclusively hard fraction and deresinated, but specifically, it must be deresinated to the extent that it does not contain more than 3 per cent by weight of resin or, conversely, as stated above, that the wax be at least 97 per cent soluble in boiling isopropanol. This critical limitation is exemplified in Example 2, where the hard fraction wax to be treated had been deresinated to the extent that it was 95 per cent soluble by weight in boiling isopropanol. The substantial deficiencies in color, penetration value and gel strength of the resultant treated wax are obvious by comparison with these characteristics of the wax resulting from the treatment set forth in Examples 1 and 3.

It is necessary in the prescribed process that the oxidation reaction be continued until the acid number is increased to 20, but not more than 35. This observance of the acid number is a gauge by which the processor is able to appreciate at what stage the wax being processed has been sufficiently altered. If the action is stopped before the acid number of the wax is increased to 20, the improvements in the qualities thereof are not as marked. If the process is allowed to continue until the acid number is extended above 35, the chemical changes in the wax continue and there is excessive breakdown in the molecular structure. However, when the acid number is increased to a point approaching 35, slight improvements are evidenced in the treated wax over that which is the result of a reaction stopped at 20. This is evident by comparison of the physical characteristics of the wax derived in Example 3 (where an acid number of 35 was reached) and that of Example 1.

It is preferred that agitation of the molten wax be conducted while the oxygen-containing gas is being admitted to the vessel. The fine bubbles of gas, upon entrance into the vessel have the tendency to coalesce too rapidly to present an effective contact area. When the agitation is conducted adjacently above the gas entrance, it promotes a shearing action and redistributes ascending gas, not only maintaining a minute bubble size, but also by folding action delaying the escape of the air from the surface of the molten wax.

The reaction may be conducted within a fairly wide temperature range, from 80° C. (the hard fraction wax has a melting point of 79° C.) to 150° C. This upper limit must be observed since at this temperature, pyrolytic effects develop a red coloration in the wax which restricts the amount of bleaching which can be attained. For maximum decolorizing effect, it is preferable that reaction be conducted at a temperature of less than 130° C.

It will be noted that in Example 1 where a 100 pound charge of hard wax fraction was being treated, the oxygen-containing gas was introduced into the molten wax at a rate of approximately 450 cubic feet per hour or approximately 10 cubic feet per hour per kilogram of wax. It should be appreciated that this flow rate may be varied depending upon several factors, as for example, the size and shape of the vessel, type of agitation employed, and the amount of time to be allowed the reaction. However, we have found that in almost all applications, when the gas employed is air, containing the usual 20 per cent oxygen, the flow rate is not less than three nor more than fifteen cubic feet per hour per kilogram of wax.

In comparing the five qualifying characteristics of the hard fraction waxes treated in the prescribed manner (Examples 1 and 3) and that resulting from treatment in other than the prescribed manner (Example 2), it will be noted that the hard fraction treated in the prescribed manner possesses substantially improved characteristics. These are featured especially in a comparison of color and gel strength. The gel strength shown in the examples as indicated by the cone penetrometer reading of the wax-solvent and paste wax mixtures was arrived at by the accepted American Society for Testing Materials method, Designation: D217–48. The needle penetrometer readings conducted in Example 4 were concluded as a result of the conventional needle penetrometer test as specified by the American Society for Testing Materials, Designation: D5–49.

This new and improved wax, as described in Example 1, is not only capable of finding new application in the polishes and coating industry, but also in specific industrial applications as, for example, the carbon paper industry. The two major deficiencies of hard fraction sugar cane wax, and even that which has been subsequently deresinated to the extent that it is at least 97 per cent

soluble by weight in boiling isopropanol, are oil retention penetration and carbon dispersion.

"Oil retention penetration" is defined as the penetration in .1 mm. at 25° C. of a needle penetrometer, the needle carrying a 300 gram load into a mixture of equal weight of the wax to be tested in paraffin oil. This type of pentrometer reading indicates the oil retention potential of the wax; the greater the penetrometer reading, the less the oil retention power. It will be noted that the wax treated in the process described herein, when tested in this manner in Example 4, bore a substantially smaller pentrometer reading and therefore is recognized as being substantially greater in oil retention capacity.

The technique for estimating the dispersing properties of a wax in carbon black inks as set forth in Example 5 strongly indicated superior carbon dispersing characteristics of the treated hard-fraction deresinated sugar cane wax over the untreated hard fraction deresinated wax.

Thus, through the practice in our prescribed process, as stated in the claims hereafter, a new and improved wax is produced which bears functional and physical improvements over the unprocessed wax or the hard-fraction deresinated wax which may be processed in any other manner known to the art.

Other modes of applying the principle in the invention may be employed instead of those explained, change being made as regards the process herein described and/or its new and improved wax product, providing the step or steps stated or the new and improved wax product described in any of the following claims or the equivalent of such stated step or steps or product be employed.

We claim:

1. A method of treating refined sugar cane wax having a soluble content of at least 97 per cent by weight in boiling isopropanol, which comprises melting the wax, and maintaining said molten wax at a temperature of from 80° to 150° C. while introducing and dispersing oxidizing gas into said molten wax until the acid number of said wax is increased to at least 20, but not more than 35, and the saponification number is increased to at least 45, but not more than 75.

2. A method of treating refined sugar cane wax having a soluble content of at least 97 per cent by weight in boiling isopropanol, which comprises melting the wax, and maintaining said molten wax at a temperature of from 80° to 150° C. while introducing and mechanically agitating oxidizing gas into said molten wax until the acid number of said wax is increased to at least 20, but not more than 35, and the saponification number is increased to at least 45, but not more than 75.

3. A method of treating refined sugar cane wax having a soluble content of at least 97 per cent by weight in boiling isopropanol, which comprises melting the wax, and maintaining said molten wax at a temperature of from 80° to 150° C. while introducing and dispersing oxidizing gas into said molten wax until the acid number of said wax is increased to at least 20, but not more than 35, and the saponification number is increased to at least 45, but not more than 75, said oxidizing gas being introduced into said molten wax at a rate of from 3 to 15 cubic feet of gas per kilogram of wax per hour.

4. A method of treating refined sugar cane wax having a soluble content of at least 97 per cent by weight in boiling isopropanol, which comprises melting the wax, and maintaining said molten wax at a temperature of from 80° to 150° C. while introducing and mechanically agitating oxidizing gas into said molten wax until the acid number of said wax is increased to at least 20, but not more than 35, and the saponification number is increased to at least 45, but not more than 75, said oxidizing gas being introduced into said molten wax at a rate of from 3 to 15 cubic feet of gas per kilogram of wax per hour.

5. A method of treating refined sugar cane wax having a soluble content of at least 97 per cent by weight in boiling isopropanol, which comprises melting the wax, and maintaining said molten wax at a temperature of from 80° to 150° C. while introducing and mechanically agitating oxidizing gas into said molten wax until the acid number of said wax is increased to at least 20, but not more than 35, and the saponification number is increased to at least 45, but not more than 75, said oxidizing gas being introduced into said molten wax at a rate of approximately 10 cubic feet of gas per kilogram of said wax.

6. A light-colored, substantially odorless sugar cane wax being at least 97 per cent soluble by weight in boiling isopropanol, and having an acid number of from 20 to 35, a saponification number of from 45 to 75 and strong gel-promoting characteristics.

7. A light-colored, substantially odorless sugar cane wax being at least 97 per cent soluble by weight in boiling isopropanol, and having an acid number of from 20 to 35, a saponification number of from 45 to 75 and having strong gel-promoting and paste forming characteristics.

8. A light-colored, substantially odorless sugar cane wax being at least 97 per cent soluble by weight in boiling isopropanol, and having an acid number of from 20 to 35, a saponification number of from 45 to 75, and strong gel-promoting characteristics, said wax, when incorporated in a paste product, being capable of forming a firm crystalline structure.

9. A light-colored, substantially odorless sugar cane wax being at least 97 per cent soluble by weight in boiling isopropanol, and having an acid number of from 20 to 35, a saponification number of from 45 to 75, and strong gel-promoting characteristics, said wax forming a viscous gel and evidencing substantially no precipitation upon cooling a petroleum distillate solvent having said wax dissolved therein.

References Cited in the file of this patent

UNITED STATES PATENTS

2,640,841 Hatt et al. ---------- June 2, 1953

FOREIGN PATENTS

5,066 Great Britain ---------- 1915