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## PURIFICATION OF HARD WAXES BY REMOVAL OF SOFT FATTY COMPONENTS

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This application is made under the act of March 3, 1883, as amended by the act of April 30, 1928, and the invention herein described, if patented, may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment to me of any royalty thereon.

This invention relates to the purification of hard waxes with particular reference to those waxes from which deleterious matter of a fatty nature is removed.

Although most hard waxes of commerce require little if any purification to make them suitable for their many applications, other waxes occur or are obtained in admixture with contaminating substances of a fatty nature which detract from the value of the wax and, in some cases, have to be removed in order to produce an acceptable product. The latter type of wax is exemplified by the product recovered from precipitates resulting from clarifying cane juice in the production of cane sugar.

Natural sugarcane wax of excellent quality exists as a thin coating upon the stalks of sugarcane, but no practical method of recovering this wax directly has ever been evolved. Thus, to obtain this desirable wax, an indirect method of recovery is employed. The method which has been developed for commercial use has consisted of extracting juice clarification precipitates, variously termed clarification muds, cachaza, and press cake, with an organic solvent, but the product so obtained contains such a large proportion of fatty matter that a separation of the waxy matter from the fatty matter must be effected before the wax becomes suitable for commercial use.

For convenience, crude sugarcane wax may be assumed to consist simply of fatty and waxy components, although this classification is not strictly correct. The waxy fraction consists predominantly of the natural wax, but other constituents are present that are foreign to the natural wax. The fatty fraction consists predominantly of the natural fats of sugarcane, but many other constituents frequently associated with fats are also present. In practice, no constant ratio of waxy matter to fatty matter, constituting crude sugarcane wax, has been found to exist. The crude wax may vary in composition from a ratio of about four parts of waxy matter to one part of fatty matter to a ratio of about equal amounts of the two types of matter. It is to be observed that a large degree of purification is required to obtain the desirable hard wax fraction from crude sugarcane wax.

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Even though sugarcane wax was an article of commerce for many years, both in the crude and in the hard wax state, exact information regarding the method used in obtaining the hard product from the crude wax is lacking. It may be assumed, however, that the hard wax was obtained by fractional crystallization, following conventional procedures. It may be concluded that failure to devise a satisfactory method of separating the fatty from the waxy components of the crude wax is partially responsible for the lack of development in the sugarcane wax field.

The object of this invention is to provide a method of fractionating impure waxes into their waxy and fatty components as a means of purification, which is efficient, which is carried out expeditiously, and which enhances the value of the wax.

Although stress will be given to the purification of sugarcane wax in discussing this invention, it is not to be interpreted that this invention is limited in scope to this product. Other waxes may be purified by the application of this invention with similar and beneficial results being obtained. Sugarcane wax was chosen for discussion, as it best exemplifies the type of wax that requires a large degree of purification in order to obtain an acceptable wax.

A study of the processes of crystallization of waxes, and especially of sugarcane wax, revealed a number of facts that have not been previously disclosed. In conventional methods of crystallization, in which crude wax is dissolved in an excess of solvent at an elevated temperature and the solution is cooled to reduce the solubility of the wax, the wax crystallizes in a form having exceedingly poor physical characteristics from the standpoint of separation from the mother solution. Of more importance was the finding that the crystalline structure of the wax could be altered from one which was so very unsatisfactory to one which possesses a high degree of granulation by reducing the ratio of solvent to solute prior to crystallization greatly beyond that used in the ordinary methods of crystallization. Maximum granulation apparently occurs when the ratio by weight of solvent to solute is appreciably less than 1. Furthermore, it was discovered that the desirable granular characteristics of the wax crystals could be retained if the mother solution containing the fatty matter were removed with a solvent that is highly selective toward the fatty constituents, rather than with additional amounts of the solvent from which crystallization occurred. This production and retention of the wax crystals

in granular form makes possible the separation of the hard wax from the fatty components, comprising the crude product, in an efficient and expeditious manner using conventional filtration or centrifugal methods. Moreover, the purified wax is easily obtained in final form as a dry granular powder which enhances the value of a wax, inasmuch as powdered waxes command a premium on the market over the same waxes in customary lump, or cake form.

The type of solvent which may be used in the crystallization operation in the practice of this invention is not critical. Any one of the solvents which are used commercially for the extraction of crude sugarcane wax give similar and satisfactory results. These solvents, however, are generally one of two types—an aromatic hydrocarbon such as toluene or benzene, or a petroleum naphtha such as a close-cut, heptane type—but other solvents may be employed. Practical considerations limit the weight ratio of solvent to solute to be employed generally to within the range of 0.5 to1.0, although other ratios may be used without deviating from the intent of this invention. It is usually desirable to use as low a ratio as is possible and still obtain a mass after crystallization which can be mixed with the fat solvent without too much difficulty. Lower ratios are more generally applicable to impure waxes such as crude sugarcane wax, while higher ratios are used on waxes which have a low content of fatty matter.

It is preferred to take the crude wax from the process of recovery, if by solvent extraction, at a point in the process when it contains the desired ratio of solvent to solute for granular crystallization, but if solvent-free waxes are being purified, the wax is melted with the desired proportion of solvent. Crystallization is induced by cooling the melted wax and wax solvent mixture. Cooling is allowed to proceed at a slow rate with several hours elapsing before the temperature of the mass reaches that of the surrounding air, which should be normal room temperature or only slightly below. Retaining the temperature of the mass at room temperature for another period of a few hours after reaching that point insures establishment of solubility equilibrium. It is desirable to stir the mass slowly during the period of crystallization to prevent it from setting up into a too hard structure. If the crystallization is conducted in a preferred manner, the mass assumes the approximate consistency of a confectioner's coarse sugar fondant with the solid phase consisting of granular wax crystals and the liquid phase consisting essentially of a thick solution of the fatty fraction of crude wax in the wax solvent.

These two phases are separated from one another by an ordinary filtration or centrifugal method after the liquid phase has been diluted to an appropriate extent with a solvent which does not destroy the granular characteristics of the crystalline wax. This latter is very important. Fat solvents which have a low solvent action on hard waxes are required for this service. The limit of solubility of natural sugarcane wax at ordinary room temperature is of the order of 0.4 gm. per 100 ml. of solvent, for if the solubility of wax in the fat solvent exceeds this figure appreciably, the filtering characteristics of the wax are affected, with the result that separation of the wax and fatty phases of the crystallized mass cannot be conducted as efficiently nor as expeditiously. Acetone has been found to be an excellent fat solvent and to have a very low solvent action on the wax; it is preferred over other solvents which may be used for this service. Other suitable fat solvents are those disclosed in Goepfert Patent No. 2,391,893 and Balch Patent No. 2,381,420.

After obtaining the wax by filtration or other means of separation from the solution of fatty matter, the wax is washed until practically free of any residual fatty substances with additional amounts of the fat solvent. The wax is then freed of the solvent which is retained from the washing operation, preferably at a temperature below the softening point of the wax, which is also easily accomplished when acetone is used as the fat solvent, since its boiling point is well below the melting point of all natural hard waxes. If the wax cake is broken up prior to evaporating off the solvent, this operation is expedited. The wax is thus obtained in final form as a dry, granular powder. This is the preferred form for the wax on account of its easier handling and dispensing in comparison to wax in the more usual lump or cake form, and because wax in powdered form brings a premium in price over the same wax in lump or cake form.

Recovery of the fatty fraction of crude wax as a by-product is desirable. It is easily accomplished by distilling off the mixture of solvents used in its separation from the hard wax. The solvents may be fractionated, which conditions them for reuse.

The following examples are cited for the purpose of demonstrating the efficiency and expeditiousness of this invention when conducted in a preferred manner.

*Example I.—Purification of ouricury wax*

One hundred grams of ouricury wax were melted with 100 grams of toluene, and the mixture was allowed to cool to room temperature and to crystallize over a period of five hours. To the crystallized mass, about 200 grams of acetone were added and the mixture was stirred until a smooth slurry was obtained. This slurry was transferred to a Buchner funnel fitted with a filter cloth and suction was applied. The wax cake which was obtained was washed with additional portions of acetone until the mother liquor had been displaced. The washed wax cake was broken up and the residual solvent was allowed to evaporate spontaneously. The resulting product was a hard wax free of soft fatty and waxy matter in the form of a granular powder, light tan in color. The fatty fraction which was recovered from the acetone solution was yellow-green in color and was appreciably softer than beeswax. The filter area was 19.6 sq. in.; the thickness of the wax cake was about ¾ inch; the time required for the filtration and washing cycle was approximately eight minutes; the yield of purified wax was 83 percent of the original wax.

*Example II.—Purification of crude sugarcane wax*

Crude sugarcane wax was extracted with hot toluene from a lot of air-dry juice clarification muds from a factory producing raw sugar. The solution of crude wax was filtered and transferred to a still, where the toluene was distilled off until the residue contained what was believed to be the proper proportion of solvent for crystallizing the wax in granular form. This residue was discharged from the still into a crystallizing vessel, where it was allowed to cool slowly and to stand for a total period of 18 hours. At the end

of this period, the crystallized mass weighing 923 grams was mixed with 1500 ml. of acetone until a smooth slurry was obtained. This slurry was transferred to a Buchner funnel fitted with cloth, and suction was applied. The resulting wax cake was washed with several portions of acetone, totaling about 1500 ml., which effectively removed the fatty matter. The washed wax cake was broken up, and the residual solvent was allowed to evaporate spontaneously. The final product was a hard wax in the form of a dry granular powder, greenish yellow in color. The fatty matter recovered from the acetone solution had a pasty consistency at room temperature and was very dark green in color. Ratio of solvent to solute at time of crystallization was 0.6 by weight; filtering area was 70.9 sq. in.; the thickness of the wax cake was about ¾ inch; the filtration and washing cycle required 20 minutes; the yield of purified wax amounted to 409 grams, or 70.6 percent of the crude wax.

*Example III.—Purification of crude sugarcane wax*

A small lot of air-dry juice clarification muds were extracted with hot petroleum naphtha whose average boiling point was approximately 100° C. The solution of crude wax was filtered and concentrated by distilling off the solvent. When the residue was thought to have the desired concentration for granular crystallization, it was discharged from the still into a crystallizing vessel. The mass was allowed to cool slowly and to crystallize over a period of about 18 hours. The crystallized mass was then stirred with 225 ml. of acetone until a smooth slurry was obtained. This slurry was transferred to a Buchner funnel fitted with a cloth, and suction was applied. The resulting wax cake was washed with additional portions of acetone until practically free of fatty matter. The wax cake was broken up and the residual solvent was allowed to evaporate spontaneously. The hard wax was obtained in final form as a dry, granular powder, greenish yellow in color. The fatty matter which was recovered from the acetone solution was very dark green in color and had a pasty consistency at ordinary room temperature. The ratio of solvent to solute at time of crystallization was 0.9; the filter area was 14.2 sq. in.; the wax cake had a thickness of about ½ inch; the time cycle for filtration and washing amounted to 35 min.; the yield of hard wax in the form of a dry, granular powder was 56 grams, which corresponded to 73 percent of the crude wax.

It is to be observed that this invention offers several improvements over the prior art for separating undesirable fatty constituents from impure waxes in order to obtain hard waxes having more desirable properties. The process may be carried out with considerable flexibility without departing from the scope and intent of this invention.

Having thus described my invention, I claim:

1. A method of obtaining granular crystals of sugarcane wax, comprising inducing crystallization by slowly cooling over a period of several hours, to about room temperature, with stirring, a molten crude sugarcane wax and wax-solvent mixture, in which the weight ratio of wax to solvent is between one and two parts of wax to one of the solvent, to obtain a solid phase of granular wax crystals and a liquid phase consisting essentially of a solution of the fatty fraction of crude wax in the wax solvent.

2. A method of obtaining granular crystals of purified hard wax, comprising the steps recited in claim 1 and in which there is added to the liquid phase in contact with the crystals a solvent for the fatty impurities, which solvent dissolves no more than about 0.4 grams of hard wax per 100 milliliters of solvent, and then separating the solid phase of purified granular hard wax.

3. A method of obtaining optimum granulation of crystals of a hard wax in a purified form, comprising the steps recited in claim 1 and in which the liquid phase is diluted with acetone, and the diluted liquid phase then separated from the solid phase of purified granular hard wax.

4. A method of obtaining purified crystals of sugarcane wax from a solution of crude sugarcane wax in a solvent, comprising crystallizing the wax in granular form by cooling the solution over a period of several hours to room temperature, the weight ratio of solvent to wax of which solution is no more than about 1 to 1, to obtain a solid phase of granular wax crystals and a liquid phase comprising a solution of the fatty fraction of crude wax in the wax solvent, mixing both phases with a solvent for fat, which solvent dissolves, at ordinary temperatures, no more than about 0.4 grams of the hard wax per 100 milliliters of solvent, and separating purified granular hard wax from the solvent.

5. The process of claim 4 in which the fat solvent is acetone.

6. A method of purifying sugarcane wax comprising preparing granular wax crystals by slowly cooling a solution of sugarcane wax in a solvent, to about room temperature, with stirring to crystallize the wax, the weight ratio of solvent to wax in the said solution being no more than about 1 to 1, to obtain a solid phase of granular wax crystals and a liquid phase comprising a solution of the fatty fractions of crude wax in the wax solvent, slurrying the liquid and solid phases with a fat solvent which solvent dissolves, at ordinary temperatures, no more than about 0.4 grams of the sugarcane hard wax per 100 milliliters of solvent, and separating the solid phase of purified granular hard wax.

7. The process of claim 6 in which the fat solvent is acetone.

8. A method of purifying natural hard waxes by removing from the impure wax the undesirable fatty fraction while producing the purified wax in the form of a dry granular powder, comprising crystallizing the wax in granular crystal formation from a melted mixture of the impure wax and a wax solvent by stirring and cooling to room temperature over a period of several hours, the weight ratio of wax to solvent during the crystallization being between one and two parts of wax to one of the solvent, diluting the resulting mixture of wax crystals and solvent with a fat solvent, retaining the granular character of the wax crystals, by employing as fat solvent one that is a poor wax solvent, separating the granular wax crystals from the solution of the fatty matter, washing the wax crystals with additional amounts of the fat solvent, and drying the wax crystals at a temperature below the softening point of the wax.

9. The method described in claim 1 in which the wax-solvent is taken from the group consisting of liquid hydrocarbons.

10. The method described in claim 1 in which the wax-solvent is taken from the group consisting of liquid hydrocarbons and in which the liquid phase is diluted with acetone, and the diluted

liquid phase then separated from the solid phase of purified granular hard wax.

11. The process of claim 8 in which the fat solvent is acetone and the wax solvent is a liquid hydrocarbon.

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## REFERENCES CITED

The following references are of record in the file of this patent:



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