

## UNITED STATES PATENT OFFICE

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## METHOD OF OXIDIZING PARAFFIN WAX AND THE PRODUCT THEREOF

No Drawing.

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This invention, a continuation in part of my copending application filed September 24, 1924, Serial No. 739,527, relates to the oxidation of hydrocarbons and more particularly to the oxidation of paraffin wax.

The primary efforts of prior investigators dealing with the oxidation of paraffin wax have been to produce acids of high molecular weight resembling the natural fatty acids and which might be used to replace the latter in the arts, particularly soap making. By the use of high temperatures and rapid injection of air or oxygen, large amounts of acid may be formed. The purpose of my invention, however, is to produce material having as low an acid value as possible while obtaining a large amount of saponifiable bodies; an additional purpose is to obtain an oxidized wax which is soluble in hydrocarbon oils, especially lubricating oils at temperatures ordinarily encountered in marketing or use.

The saponifiable bodies or esters obtained during the oxidation of paraffin wax are beneficial in increasing the lubricating properties of mineral oils.

Since lubricating oils, particularly motor oils, must have good transparency, it has been my aim to make such oxidized wax completely soluble in lubricating oil so that no clouding would occur even at low temperatures. Paraffin wax by itself is not appreciably soluble in lubricating oils at low temperatures and it is therefore apparent that the oxidized wax used for compounding with oils must not contain any excessive amount of unchanged wax. It has been discovered, however, from extended observation that the presence of oxidized wax increases to a marked degree the solubility of unoxidized wax in the oil; thus it becomes unnecessary in most instances to oxidize the wax excessively and in some cases even as much as 50% of unchanged wax is entirely permissible. On the other hand, it is especially desirable to avoid energetic oxidation at higher temperatures or oxidation at moderate temperatures for an extended period of time. Such treatment results in the formation of a certain amount of material which is partially insoluble in lubricating oils. If a portion of such over-oxidized wax

is compounded with lubricating oil and allowed to stand, a red gummy deposit forms at the bottom of the vessel in which it is stored; this red gum is likely to deposit on moving parts which may be subjected to lubrication with oil containing this gum and may cause trouble in their operation. The present method of oxidation has, therefore, been developed to give an oxidation product which is soluble in lubricating oils at average atmospheric temperatures of this locality, particularly at temperatures above the cloud point or pour point of the original oils, which is free from gum, and which contains a large proportion of saponifiable bodies.

In carrying out the oxidation a charge of paraffin wax, for example crude scale wax having a gravity of between 48° and 50° A. P. I. at 130° F., is placed in an enameled or aluminum vessel which is preferably jacketed or otherwise fitted to permit heating or cooling of the contents. The vessel should also be fitted with an air distributor, preferably located near the bottom, through which air may be introduced in fine streams to promote intimate contact of the air with the oxidizing mass. If the vessel is well insulated, the heat developed by the oxidation will be sufficient to maintain the charge at the proper temperature once it has been brought to that temperature by outside means such as a steam coil. In fact it has been found that it is occasionally desirable to supply external cooling means such as cooled oil to prevent an increase in the temperature beyond that contemplated. The charge of wax may be heated to a temperature of between 260 to 330° F. and air may be introduced in sufficient quantity to maintain the desired temperature.

The mention of this temperature range does not preclude the use of other temperatures in the oxidation, but it is to be noted that excessive temperatures tend to the formation of red gum which is undesirable in the finished product and such temperatures should, therefore, be avoided. In the light of present knowledge the most satisfactory temperatures for the oxidation are about 315 to 320° F.; although satisfactory material has been made commercially at

temperatures as high as 335° F., it is preferred to operate at the lower temperatures.

The oxidation is allowed to proceed until a small sample of the batch indicates a gravity preferably of about 34° A. P. I. at 130° F., although gravities of 33 to 35° A. P. I. are acceptable. By thus observing the gravity of the oxidizing product the quality may be closely controlled, especially as to its freedom from red gum. Observations made over an extended period of time indicate that material falling within this range of gravities is free from gummy products and is entirely soluble in lubricating oils.

A test from which it may be concluded that the oxidized wax is free from gum consists in preparing a 10% solution of the oxidized wax in a light distillate, preferably 86° Bé. gasoline, and allowing the solution to stand until clear. The absence of a precipitate at the bottom of the vessel containing the solution is an indication that the oxidized material is free from gum.

As an illustrative example of the invention, the following operating data may be given: A charge of about 2300 pounds of crude scale wax was placed in an enameled vessel, and after preliminary heating air was introduced to maintain a temperature of about 320° F. The oxidation was continued for a period of about 16 hours or until the batch of oxidizing wax indicated a gravity of about 34° A. P. I. at 130° F. The finished material was found to be of excellent color and showed no traces of gummy material when mixed with 86° Bé. gasoline to form a 10% solution. The oxidized wax had an acid number of about 26 and a total saponification value of about 93, giving a ratio of neutral saponification value to acid value of over 2½ to 1 and preferably around 3½ to 1. Upon compounding 3% of this oxidized wax with 250 pale filtered oil the pour test remained below 0° F. Experience indicates that a 2% solution of oxidized wax in pale filtered lubricating oils is the most desirable proportion for general commercial use and it follows that the above example furnishes an entirely satisfactory product for this purpose.

In those instances where the proportion of little-oxidized or unconverted wax remaining in the oxidized mass may be objectionable, this may be removed from the mass of oxidized material by the well known process of sweating, similar to that commonly practiced in the removal of oil from crude paraffin wax. In this manner the higher melting portions of the oxidized mass may be eliminated from the lower melting portions and the former subjected to further oxidation treatment if desired. If this supplemental treatment is practised, it is possible to discontinue oxidation sooner than otherwise would be possible and yet obtain

oxidized material having a high ratio of neutral saponification number to acid number and also having a complete solubility in pale lubricating oils at ordinary temperatures, such as 20° F.-100° F.

This supplemental sweating treatment may be of considerable importance in the preparation of materials suitable for soap making inasmuch as it enables the production of exceptionally light colored materials of any desired melting point. It has been customary in the prior art, in carrying on the oxidation of the paraffin wax, to effect as complete conversion as possible into acids and other saponifiable matter when seeking the preparation of soap making materials. In following such a procedure, however, as pointed out above, the mass of oxidizing material becomes progressively darker and contains more and more of gummy products and other materials unsuited to saponification.

If, on the other hand, the oxidation is discontinued before these detrimental substances form, the lower melting materials may be segregated from the higher melting portions by sweating and the former being of good color and quality chemically, may be used in soap making and the like, while the higher melting portions may be subjected to further oxidation.

While I have thus specifically described my invention, numerous modifications thereof will suggest themselves to those skilled in the art and it is therefore to be limited only as indicated in the appended claims.

What I claim is:

1. A composition of matter consisting of a paraffin wax which has been oxidized in such a manner as to contain only a minor proportion of unoxidized wax and substantially no gummy products and to be substantially completely soluble to the extent of about 3% in lubricating oils over the range of normal atmospheric temperatures of 20° F.-100° F., said composition having a gravity of about 33° to about 35° A. P. I. at 130° F.

2. A composition of matter consisting of a paraffin wax which has been oxidized in such a manner as to contain only a minor proportion of unoxidized wax and substantially no gummy products and to be substantially completely soluble to the extent of at least 3% in 300 seconds viscosity (Saybolt) lubricating oil at temperatures above the cloud point of said oil, said composition having a gravity of about 33° to about 35° A. P. I. at 130° F.

3. A composition of matter consisting of an oxidized paraffin wax, having an A. P. I. gravity of about 33°-35° at 130° F., formed by oxidizing paraffin wax of about 50° A. P. I. at 130° F., in such a manner as to contain a major proportion of saponifiable constituents, to have a ratio of saponification value

to acid value of about  $3\frac{1}{2}$  to 1, and to be substantially completely soluble to the extent of at least 2% in lubricating oil at temperatures in excess of 20° F.

5 4. The method of oxidizing paraffin wax which consists in blowing the wax with air, while heated to a temperature of about 300° F., for a sufficient period of time to produce an oxidized mass which dissolves to the extent of about 10% in gasoline without deposition of gummy products and completely dissolves to the extent of at least 3% in lubricating oil at temperatures above the pour point of said oil, and then discontinuing  
15 oxidation.

5. The method of oxidizing paraffin wax which consists in blowing the wax with air, while heated to a temperature of about 300° F., for such a period of time that the remaining oxidized mass contains relatively little unchanged wax and completely dissolves in substantial amount in lubricating oils at temperatures in excess of 0° F., and for a period of time less than that required to produce  
25 an over-oxidized mass containing gummy products which precipitate in a 90% gasoline solution, and then discontinuing oxidation.

6. The method of oxidizing paraffin wax  
30 having a gravity of the order of 50° A. P. I. at 130° F. which comprises exposing the wax to the action of an oxidizing gas while under the influence of heat, and discontinuing the oxidation when the gravity of the remaining oxidized mass has attained about 34°  
35 A. P. I. at 130° F., whereby a mass free from gummy products insoluble in hydrocarbon oils is formed.

7. The method of oxidizing paraffin wax  
40 having a gravity of the order of 50° A. P. I. at 130° F. which comprises blowing the wax with air while heated to a temperature of 260° F. to 335° F., until the gravity of the remaining oxidized mass attains about 34°  
45 A. P. I. at 130° F., and then discontinuing the oxidation, whereby a mass free from gummy products insoluble in hydrocarbon oils is formed.

8. The method of oxidizing paraffin wax  
50 having a gravity of the order of 50° A. P. I. at 130° F. which comprises exposing the wax to the action of an oxidizing gas while under the influence of heat, and discontinuing the oxidation when the gravity of the remaining  
55 oxidized mass has attained a range of 33° to 35° A. P. I. at 130° F., whereby a mass free from gummy products insoluble in hydrocarbon oils is formed.

9. The method of oxidizing paraffin wax  
60 having a gravity of the order of 50° A. P. I. at 130° F. which comprises exposing the wax to the action of an oxidizing gas while under the influence of heat for such a period of time that the remaining oxidized mass has  
65 a gravity of about 33° to 35° A. P. I. at 130°

F., then discontinuing oxidation, and subjecting the oxidized mass to a sweating operation to separate the higher melting portions from the lower melting portions.

10. The method of oxidizing paraffin wax  
70 which comprises exposing the wax to the action of an oxidizing gas while under the influence of heat for such a period of time that the remaining oxidized mass shows a ratio of saponification value to acid value of  
75 over  $2\frac{1}{2}$  to 1 and contains only a minor proportion of unoxidized wax and substantially no gummy products and is completely soluble to the extent of at least 2% in lubricating oil at temperatures above the cloud point  
80 of said oil, and then discontinuing oxidation, said oxidized mass having a gravity of about 33° to about 35° A. P. I. at 130° F.

In witness whereof I have hereunto set my hand this 21st day of March 1927.

BENJAMIN GALLSWORTHY.

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