PATENT SPECIFICATION

DRAWINGS ATTACHED

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COMPLETE SPECIFICATION

A Process for the Manufacture of Synthetic Waxes

We, COMMONWEALTH COLOR AND CHEMICAL COMPANY, a Corporation of New York, of 3240 Grace Avenue, Bronx, New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention concerns the production of synthetic waxes and more particularly a method of producing such a product which resembles Carnauba wax in various desirable properties.

Attempts have heretofore been made to produce such a wax by various methods. For instance, tank bottom or microcrystalline waxes have been oxidized to render them emulsifiable, but in the process their hardness suffered. To increase the hardness and toughness of the waxes, resins such as polyethylene were blended in, but then the emulsion forming properties usually suffered.

Also, processes are known whereby high molecular weight hydro - carbons are oxidized in order to form fatty acids for the purpose of preparing scaps and the like. According to one such process, the oxidation was in the presence of a metallic scap having drying properties. However, such a process was unable to produce a wax of a hard type. In another process, normally liquid hydrocarbons were heated in the vapour state in the presence of a catalyst with oxygen - containing

$$n CO + 2n H_2$$
 $\xrightarrow{\text{Cat}}$ $C_n H_2$
 $Heat$
 Cat
 Cat
 Cat
 Cat
 Cat
 Cat
 Cat
 Cat

[Price 3s. 6d.]

gases for the purpose of producing low molecular weight alcohols, aldehydes and acids. It has also been proposed to produce such alcohols having from 8 to 18 carbon atoms by similarly oxidizing the hydrocarbons in the vapour state. However, none of these processes was adapted to produce a wax product.

The present invention is intended and adapted to overcome the difficulties and disadvantages inherent in the prior art, it being among the objects of the invention to produce a wax which is comparable to Carnauba in its hardness, high melting point and its ability to form emulsions.

It is also among the objects of the present invention to provide a process for producing such a product which utilizes a low cost and plentiful raw material and which is conomical in operation.

It is further among the objects of the present invention to provide such a process which is easy to control and which is capable of obtaining the desired characteristics in batch after batch.

In practicing the present invention there is utilized as raw material the hydrocarbon waxes produced synthetically by the Fischer-Tropsch process, which is fully described in the literature. In this process carbon monoxide and hydrogen at atmospheric pressure are passed over a catalyst such as cobalt, iron or nickel at about 200°C. The reactions involved are 65 as follows:

 $C_nH_2n + n H_2O$

 $C_aH_{2n+2}+n\;H_2O$

By varying the proportions of carbon monoxide and hydrogen plus the catalyst, reaction time and temperature, high molecular weight aliphatic hydrocarbons can be produced, as described in the article by Craxford, Trans. Faraday Soc. V. 35. p. 946 (1939). Thus by controlling the process variables a synthetic hydrocarbon wax having the desired molecular weight, melting point, isomer content and hardness can be assured.

If the Fischer-Tropsch hydrocarbon wax is oxidised under the proper conditions and in the presence of specific catalysts in accordance with the present invention, a high melting emulsifiable wax will be produced. Furthermore if this oxidised wax is then treated with certain metal compounds so that the fatty acid salts of this metal are produced, the hardness of the wax is increased without adversely affecting the emulsifiable properties of the wax.

In carrying out the process of the present invention the hydrocarbon wax used is melted and held at a temperature of about 100°C. 25 until an initiator which is a peroxide is added. air or other oxygen containing gas is bubbled through the molten mass continuously during the addition of the peroxide and the subsequent treatment wherein after the addition 30 of the peroxide the temperature of the molten mass is then raised to a temperature of not exceeding 125°C. e.g. 120°C, and a metal catalyst from Group IIb of the Periodic Table is introduced, and after introduction of the 35 metal catalyst the temperature of the molten mass is raised further to a temperature below the decomposition temperature of the wax, e.g. 140 to 155°C. The initiators are either inorganic or organic peroxides, and are capable of acting as agents for promoting the exidation of the hydrocarbon.

In the oxidation of the Fischer-Tropsch hydrocarbons, some of the main products are farty acids and their esters. Therefore, the oxidation was controlled by following the formation of acids (acid value) and ester (ester value). If the acid value and ester value (acid value plus ester value = saponification value) are plotted as the oxidation proceeds against time, the effectiveness of the peroxide catalyst can be shown. The curves which are presented are average data from oxidation procedures which will be described later.

The accompanying drawing constituting a part hereof consists of curves illustrating the nature of the invention, and in which Fig. 1 is a series of curves showing the course of exidation of hydrocarbons with and without catalysts;

Fig. 2 shows curves indicating the hardness

of various products and
Fig. 3 illustrates the solvent retention of
various products, including those of the present invention.

Referring to Fig. 1, it illustrates the course of the oxidation of the hydrocarbons under various conditions of operation. The oxidation of the Fischer-Tropsch waxes using benzoyl peroxide as the initiating agent, together with metallic zinc is shown in curves A, B and C, indicated in solid lines. Curve A is the acid value; curve B is the ester value and curve C is the saponification value. Curves A¹, B¹ and C¹ show the course of the oxidation of said wax, using only benzoyl peroxide as the initiating agent, the amount used in both cases being about 2% based upon the weight of the hydrocarbon being treated. Curves A¹¹, B¹¹ and C¹² show the course of the oxidation of the same hydrocarbons where no catalyst at all is used.

The saponification curves, C_1 and C_2 are straight lines passing through the origin. The equation for a straight line passing through the origin can be indicated by Y = mx, where m is the slope of the line. The slope of curves C_2 and C_2 can be viewed as a rate function, e.g. rate of formation of esters and acids with time. Thus if the slopes of the straight lines are known the effectiveness of catalyst or other factors in the rate of oxidation can be evaluated.

TABLE A

| | Curve | Slope (m) |
|-----|---|-----------|
| (1) | Curve C ¹¹ (no catalyst) | 4.9 |
| (2) | Curve C ¹ (0.2% di-benzoyl peroxide) | 8.1 |

Thus it is shown that increase in the rate of oxidation with the use of dibenzoyl peroxide is appreciable as indicated in Table A.

It was also found that if a secondary cata-95 lyst is used in conjunction with the initiating agent (peroxides) the rate of oxidation was further increased. For example, if the oxidation

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of Fischer-Tropsch hydrocarbons is carried out using di - benzuyl peroxide as an initiating agent and zinc as a secondary catalyst the

oxidation rates are increased further as indicated in Fig. 1 at C and Table B.

TABLE B

| | Curve | Slope (m) | Ref. Rates |
|-----|--|-----------|------------|
| (1) | Curve C ¹¹ (no catalyst) | 4.9 | 1 |
| (2) | Curve C ¹ (0.2% di-benzoyl peroxide) | 8.1 | 1.6 |
| (3) | Curve C (0.2% di-benzoyi peroxide plus zinc metal) | 17.8 | 3.6 |

The function of zinc metal as a "catalyst" is not fully known, but the indications are that it may not act as a true catalyst in that the 10 zinc is partially used up during the oxidation. It is believed that the zinc is used up in the production of fatty acid salts. This is indicated by the fact that the oxidised wax has an ash content of 0.3-0.4%. It is also possible 15 that the reaction of the zinc with the oxidation products of the wax may not be the whole cause of the catalytic effect noted. The zinc could also provide the "active" centres upon which the oxygen and hydrocarbon molecules 20 react. Thus, any reaction between the zinc metal and the oxidation products (fatty acids) may be secondary with regard to the catalytic effect shown. Example 1

The hydrocarbon wax is melted and heated to 100°C, in a glass lined or stainless steel vessel. The di - benzoyl peroxide is added whilst an oxidising gas (air) is bubbled through the molten wax. The system is then heated to

120°C. and the zinc (mossy or sheet) is added. The reaction temperature is then maintained at 150°C.—155°C. for the duration of the oxidation.

The air which is passed through the re-35 action vessel is previously dried and filtered. It was found that satisfactory results were obtained if the relative humidity of the air is 24—26% or it has a dew point of 55°F. The air rate used was 10—15 litres per minute per pound of hydrocarbon to be exidized. The oxidation is stopped when the desired acid and ester values are obtained.

A typical run which gives satisfactory results is as follows:

500 grams Fischer-Tropsch wax 1 gram di - benzoyl peroxide

60 grams Zinc (mossy or sheet)
The Fischer-Tropsch wax was used in this run and had a melting point (A.S.T.M.) of 220°17, an isomeric content of less than 10%, and 20% to 60% of the wax can be distilled at 670°F. at 2 mm. pressure. To further characterize this wax infra-red absorption data were compiled for the unoxidized and oxidized waxes. These data are given in Table C (below) along with absorption data for carnauba wax. The values given in Table C are the optical density values at the peaks found within the wave length range studied. It may be noted that the peaks found at 3.48, 6.86 and 13.75 and 13.95 were present in all three cases studied. It is believed that these peaks represent different types of carbon hydrogen bonding.

TABLE C

Wave Length (microns)

| | 3.48 | 5.77 | 6.86 | 8.87 | 13.75 | 13.95 |
|--|---|---|---|---|---|--|
| (1) Unovidized Eigeber | Optical Density | | | | | |
| Tropsch wax — 60% distilled over at 670° F. at 2 mm. | .79 | .04 | .26 | .04 | .23 | .24 |
| (2) Oxidized F T Wax Acid value of 23 Sap. value of 58 | .88 | .30 | .37 | .14 | .28 | .28 |
| (3) Flora Carnauba Wax Acid Value — 4—8 Sap. Value — 79—84 | .90 | .38 | .36 | .29 | .293 | .291 |
| | distilled over at 670° F. at 2 mm. (2) Oxidized F T Wax Acid value of 23 Sap. value of 58 (3) Flora Carnauba Wax Acid Value — 4—8 | (1) Unoxidized Fischer— Tropsch wax — 60% .79 distilled over at 670° F. at 2 mm. (2) Oxidized F T Wax Acid value of 23 .88 Sap. value of 58 (3) Flora Carnauba Wax Acid Value — 4—8 .90 | (1) Unoxidized Fischer- Tropsch wax — 60% .79 .04 distilled over at 670° F. at 2 mm. (2) Oxidized F T Wax Acid value of 23 .88 .30 Sap. value of 58 (3) Flora Carnauba Wax Acid Value — 4—8 .90 .38 | (1) Unoxidized Fischer- Tropsch wax — 60% .79 .04 .26 distilled over at 670° F. at 2 mm. (2) Oxidized F T Wax Acid value of 23 .88 .30 .37 Sap. value of 58 (3) Flora Carnauba Wax Acid Value — 4—8 .90 .38 .36 | (1) Unoxidized Fischer— Tropsch wax — 60% .79 .04 .26 .04 distilled over at 670° F. at 2 mm. (2) Oxidized F T Wax Acid value of 23 .88 .30 .37 .14 Sap. value of 58 (3) Flora Carnauba Wax Acid Value — 4—8 .90 .38 .36 .29 | (1) Unoxidized Fischer- Tropsch wax — 60% .79 .04 .26 .04 .23 distilled over at 670° F. at 2 mm. (2) Oxidized F T Wax Acid value of 23 .88 .30 .37 .14 .28 Sap. value of 58 (3) Flora Carnauba Wax Acid Value — 4—8 .90 .38 .36 .29 .293 |

At 5.77 microns, peaks were noted in the oxidized wax and carnauba wax and it was plainly absent in the unoxidized sample. Absorption at this wave length is believed to indicate the existence of carbonyl bonding (e.g. acids, ester, aidehydes, ketones, etc.). Absorption at 8.87 microns also indicates the presence of carbonyl bonding, a peak was noted in the carnauba wax run but was absent in both 10 the oxidized and unoxidized Fischer-Tropsch wax samples. The main indication which these data present is that there was no indication of carbonyl bonds in the Fischer-Tropsch unoxidized wax. It is believed that esters and 15 acids which are the oxidation products cause the peak absorptions, in the oxidized run. The wax samples were melted as they were rested with the infra red rays.

This unoxidized Fischer-Tropsch wax was melted at a temperature of 100-105°C, then the di - benzoyl peroxide was added. The air was then bubbled through the molten wax at a rate of 10-15 litres a minute per pound of hydrocarbon to be oxidized. The temperature was then raised to 120°C, and at this point the zinc was added to the reaction mixture. The temperature again was taised to 150°-155°C. for the duration of the reaction. The oxidation was followed by test-30 ing for the acid and saponification values during the course of the reaction. It was found that by stopping the oxidation when the acid values were between 10-20 and the saponification values were between 30-60, a wax 35 with the desired properties was obtained.

The apparatus which can be used in the oxidation of the Fischer-Tropsch wax was essentially as follows: In laboratory runs a three necked round bottom glass flask was 40 used as the reactor. The air was injected by means of a glass tube or a fritted glass gas disperser and the flow of the air was controlled by means of a Fischer-Porter flowmeter. The wax was heated by an electric mantle and the temperature was controlled by a merc - to - merc thermoregulator. The exhaust gases were passed through a series of condensers and expansion chambers so that any condensable material may be collected. On larger scale operations (50 lbs. of wax) steam jacketed stainless steel or aluminium

reactors may be used. Among the desirable properties of the wax are good emulsifying characteristics so that 55 very fine particle size emulsions may be formed, and a hard wax of a melting point of 850-95°C. with a light colour. These properties are obtained if the conditions of oxidation are followed as described, until a wax with an acid value of 10-20 and a saponification value of 30-60 is obtained. The time (Fig. 1) required to obtain these acid and saponification values is from three to six hours of oxidation under the stated conditions. Other peroxides which may be used are

urea peroxide and lauryl peroxide. It is believed that better results are obtained if the peroxides are soluble in the wax to be oxidized and if the decomposition temperature of the peroxide is higher than the melting point of the wax, which is the case with benzoyl peroxide.

The optimum catalytic effect was found with the use of di - benzoyl peroxide and zinc.

Temperature also has an effect on the rate of the reaction; generally its rate increases with the temperature. A temperature much over 150°C, causes the wax to begin to soften due to some cracking effects. Since a hard wax is desired a compromise reaction temperature of about 150°C. was found to be satisfactory. A prolonged reaction time, especially at high temperatures, also causes the wax to discolour, (e.g. it becomes yellow). Therefore, in order to obtain a desirable oxidized wax it is best to carry out the oxidation at a fast rate at a moderate temperature, e.g. 1400-155°C.

Example 2

In view of the fact that there is a large demand for a hard, high melting wax which can form fine particle size emulsions the emulsion forming properties of the oxidized waxes were studied. It was found possible to form a fine emulsion by one of the standard procedures in the art; the method is as follows:

(1) 40 parts of oxidized Fischer-Tropsch wax with an acid value of 10-15 and a saponification value of 35-60.

(2) 4 parts 2 amino - 2 - methyl - 1 propanel.

(3) 8 parts of oleic acid. (4) 1 part of borax.

(5) 240 parts of water. Melt (1) to 100°C. and add (2) and (3) to the wax. Heat (5) to 95°-100°C, and add (4) to the water. Slowly add the wax emulsifier system to the water - borax system at 95°-100°C. The whole mixture is agitated 110 with a high speed, high shear agitator. After all the wax - emulsifier is added to the water, the emulsion is mixed slowly to room temperature. The emulsion produced in this fashion is a good base for a dry - bright polish.

In studying the hardness of the Fischer-Tropsch waxes it was found that the higher the distillation range of the wax (i.e. a higher average molecular weight) the harder the initial and oxidized wax would be. Therefore, if hardness in waxes is sought after, the starting raw material should be a wax with a high distillation range. The initial hardness of the unoxidized wax will determine to a large extent the hardness of the final oxidized wax; in general the harder the starting raw material is the harder the oxidized wax will be. There is invariably some softening effect in the wax after oxidation as shown in Fig. 2. This softening effect is probably caused by some cracking 130

effects on the hydrocarbons plus the inherent softness of the oxidation products as compared to the hydrocarbons.

It was found possible to harden the oxidized wax considerably by forming a small percentage of certain metal salts of the fatty acids in the oxidized wax. Upon forming the metal salts of the fatty acids it was found possible to increase the hardness (decrease the penetration values) by 30% to 60% as indicated in Fig. 2. The polyvalent metals of Groups IIa and IIb in the Periodic Table

show this property of increasing the hardness

of oxidized waxes upon forming their salts

with a small percentage of the fatty acids in the oxidized wax. Of the metals in these groups, strontium and cadmium salts of the fatty acids gave the best results in terms of increasing the hardness of the waxes. Satisfactory results have also been obtained with magnesium, and zinc. These metals upon forming their salts also increased the hardness of the wax but not to the same extent as strontium or cadmium. For comparative purposes the hardness of carnauba wax and a typical oxidized micro crystalline wax are also shown in Fig. 2.

i ili 20 n

15

25

TABLE D (see Fig. 2)

Wax

- A Flora Carnauba Wax
- B Oxidized FT Wax where 40% was distilled at 670° F. at 2 mm. Acid Value of 12. Sap. Value of 47.
- C Strontium salt of oxidized wax E where ash content as SrO was 1% on weight of the wax.
- D Cadmium salt of oxidized wax E where ash content was 1% on weight of wax.
- Dxidized FT Wax where 60% distilled over at 670° F, at 2 mm. pressure,
 Acid Value 13
 Sap. Value 50
- F Typical commercial oxidized microcrystalline wax acid value 20-25 Sap. Value 55-65
- G Strontium salt of oxidized wax indicated in B. Ash content of 1% as SrO.

On forming the metal salts, the hardness is increased considerably. The penetration values decrease from 30% to 70% depending on the temperature and the parent oxidized wax. Curve G (strontium salt of the higher distilled oxidized wax) compares very favourably with carnauba wax in terms of hardness. All the other waxes used as shown are considerably harder than the typical oxidized microcrystalline wax.

EXAMPLE 3

There are a number of methods used to form metal salts of fatty acids. The methods used thus far in our investigation were as follows:

Since the oxidation is carried out in the presence of the metal, some fatty acid salts will be formed. This is the case as described with zinc. Carrying out the oxidation in the presence of zinc metal an ash content of 0.2

to .4% on the weight of the wax is obtained. This ash is in the form of zinc oxide. Thus in the process of oxidation the oxidized waxes will contain a small percentage of zinc salts. The oxidation may be carried out in the presence of another metal of Group IIb e.g. cadmium so that the desired salt formation will take place.

EXAMPLE 4

Another method of forming some acid salts of the fatty acids present in the oxidized wax is to react a salt of the metal with the finished oxidized wax. For example, taking a salt such as strontium acetate or cadmium acetate and mixing it with an oxidized wax at 150°—170°C. for 1 to 3 hours, causes the wax to harden.

(1) 100 gr. of Oxidised F—T Wax with an acid value of 23

(2) 5 gr. of Cadmium Acetate.

50

55

60

Heating (1) and (2) together for 1 to 3 hours causes the acid value to decrease and the hardness of the wax to increase. Similar results were obtained with magnesium acetate and strontium acetate. It is probable that there is some exchange reaction going on between the metal salt and the fatty acids in the wax. The salts used tend to decompose on heating, thus facilitating the salt formation with the 10 fatty acids.

EXAMPLE 5

Still another method used to form a small percentage of the salts is as follows:

(1) 1250 grams of Oxidized FT Wax with an acid value of 16.7 and a zinc ash content of .3%.

12.5 grams of Potassium hydroxide

(3) 91.25 grams of Strontium chloride (SrCl₂.6H₂O)

(4) 2500 grams Water. Melt (1) at 100°C., add (2) to (4) and heat to boil. Then add the potassium hydroxide solution to the wax with agitation. An emulsion is formed due to the formation of the potassium soap of the fatty acids present in the oxidized wax. After the emulsion is formed and the temperature is still at 95° to 100°C., the strontium chloride is added. The emulsion is thereby precipitated. After the emulsion is precipitated it is filtered and washed thoroughly with hot water. Then the wax is dried completely at 110°C. for 4—5 hours. By this method the amount of salt formed can be controlled readily. In the case presented here, the amount of potassium hydroxide used was 223 moles as compared to .342 moles of strontium chloride. The final wax has

of the wax. If all the fatty acids in the wax were to be tied up by the potassium hydroxide in forming the emulsion, 20.8 gr. of KOH should be used (i.e.

a total ash content of 1.25% on the weight

 1250×16.7 1000

In the example cited only 12.5 grams of KOH was used, so that 57.2% of the available fatty acids are tied up. In the exchange reaction between potassium and strontium there resuited a decrease of 5 in the acid value of the wax. This corresponds to complexing 30% of the available fatty acids in the wax or stating this in another fashion the exchange reaction between potassium and strontium is 52.4% efficient. The indications are that only trace amounts of potassium salt remains in the precipitated and dried wax.

After forming the metal salts of the wax acids, the physical appearance of the wax changes somewhat. It appears as a brittle hard wax whereas before the salt formation it was more amorphous. The crystalline form of the wax appears to have changed after the metal

salts are formed in the wax. In the example cited, strontium chloride was used to form the salt. Other ionic salts of other metals were also tried with similar results, such as cadmium iodide, magnesium chloride or zinc chloride. The relative amounts of metal salts to be used can be varied to a great extent and the amounts used in the cited example are not meant to be restrictive but rather to indicate the general order of magnitude to obtain the desired results.

The emulsion forming properties of the wax do not suffer upon forming these various metal salts. Using the procedure previously described to form the emulsions, very fine particle size emulsions can be formed. The melting points (90° to 95°C.) of the oxidized and the oxidized - salt wax did not change. The colour of the wax changes somewhat depending on what metal salt is being formed. For example, on forming the strontium salt of an oxidized wax the colour changes from pale white to light tan. The hardness increases considerably (Fig. 2) on forming these various salts.

A property which changes considerably is the solvent retention ability of the salt - wax over the oxidized wax. This property is very important in the manufacture of solvent type polishes such as shoe polish and furniture

To illustrate this point, solvent retention studies were performed on:

(a) The unoxidized Fischer-Tropsch wax

Oxidized Fischer-Tropsch wax (c) Oxidized Fischer-Tropsch Wax which contained the cadmium salt of the fatty acids in the wax.

(d) Pl-wa Carnauba wax. The procedure used to study the solvent retention property of the wax was as follows:

Twenty grams of the wax to be studied were dissolved in 57 grams of Stoddard Solvent. The wax-solvent systems were stored in open cups under the same conditions and were weighed periodically.

Fig. 3 shows the results obtained in graphic form. The higher the solvent retention of the product, the more desirable it is. In Fig. 3, A represents the solvent retention of 20 parts of moxidized Fischer-Tropsch wax in solution in 57 parts of Stoddard solvent. Curve B is directed to the solvent retention of 20 parts 115 of oxidized wax in the same amount of solvent, said wax having an acid value of 12 and a saponification value of 44. Curve C represents the solvent retention of a wax oxidized in accordance with the present invention and containing a cadmium salt. Curve D indicates for comparison purposes, the solvent retention of Flora Carnauba wax.

The solvent retention properties of the products of the present invention are greatly improved when the wax is in the form of the metal salt. In this product the solvent evap-

orates more slowly than even Carnauba wax, which is very desirable in the solvent polish field.

The acetyl values, which measure the presence of alcoholic hydroxy groups and the OH groups of hydroxy acids, range from 35 to 53 with an average value of about 43. In the process of treatment of the starting material the peroxide values change, rising to 10 about 18,000 P.P.M. in the second hour and then decreasing to about 11,000 P.P.M. after five hours; in the last hour there is a rapid decrease in the peroxide value down to about 400 at the end of the operation. The range in the products is from 5,000 to 400 p.p.m., and preferably the value should be not over 500 p.p.m.

The distillation range of the products at about 7 mm, mercury pressure is as follows:

| Temperature °C. | Distillate % |
|-----------------|--------------|
| To 170° | 1.85 |
| 200° | 3.90 |
| 250° | 8.00 |
| 300° | 11.80 |
| 350° | 59.70 |

Above this temperature cracking and decomposition takes place,

Other properties of the products include the lactone value which measures the lactones and some of the acid anhydrides present. The lower the value and the harder the wax, which is desirable for buffing and polishing characteristics, better emulsification results. The product has a lactone value by the Leukowitsch method of 3 to 30. The saponification value lies between 30 and 60 and it measures in mg.

30 of KOH per gram of product, the sum of acids

and esters present. The melting point ranges from 180° to 210°F. The compounds are substantially straight chain substances containing not over about 10% of isomers, and they are practically completely saturated. The consistometer hardness (Abrams) ranges from 35 to 85 at temperatures of 130° to 80°F.

The products have approximately the following properties:

| Acetyl Value | 35 to 53 |
|-------------------------|--------------------|
| Peroxide Value | 400 to 5000 p.p.m. |
| Distillation at 350° C. | 60% |
| Lactone Value | 3 to 30 |
| Saponification Value | 30 to 60 |
| Melting Range | 180° to 210° F. |
| Consistometer Hardness | 35 to 85 |
| Acid Value | 10 to 20 |

British Patent Specification No. 786,654 describes and claims a process for the production of waxes containing oxygen which comprises oxidising a hard wax, which may have been derived from a Fischer-Tropsch synthesis, in a molten state with oxygen or a gas containing free oxygen in the presence of a catalyst active for the oxidation of the parafin wax at

a temperature below 145°C and for so short a time that the acid number of the product is below 40. There is also claimed a process for the provision in the finished product of the presence of fatty acid salts or mixtures thereof. The catalysts described in the specification of Patent No. 786,654 are fatty acid salts of manganese, cobalt, and copper or oxides and

salts such as potassium permanganate, manganese dioxide, iron oxide and silver oxide. The Specification of Patent No. 786,654 contains no reference, however, to an oxidation treatment of waxes in which there is a preliminary stage of adding a peroxide initiator into the molten wax whilst passing an oxygen gas therethrough and not reference to the use of a metal catalyst following the addition of 10 the peroxide initiator or no reference to the use of three different temperature levels for the various stages of the oxidation.

WHAT WE CLAIM IS:-

1. A method of treating solid waxy hydro-15 carbons produced by the Fischer-Tropsch process which comprises melting said hydrocarbons and holding the temperature at about 100°C, until an initiator which is a peroxide is introduced into the molten mass, passing an 20 oxygen containing gas continually therethrough during addition of the peroxide initiator and the subsequent reaction wherein after introduction of the peroxide initiator the molten mass is raised to a temperature not exceeding 125°C whereupon a metal catalyst from Group IIb of the Periodic Table is introduced and the temperature then raised to a temperature below the decomposition temperature of the wax, e.g. 140 to 155°C for several hours, e.g. 3 to 6 hours.

2. A method in accordance with claim 1 wherein the added metal is zinc.

3. A method in accordance with claim 1 or claim 2 wherein the treatment is stopped when the acid value of the wax becomes 10 to 20 and the saponification value 30 to 60.

4. A modification of the treatment claimed in claim 1 wherein after the oxidation treatment a salt of a metal of Group Ha or Group IIb of the Periodic Table is added to the product to form the soaps of acids therein.

5. A method in accordance with claim 4 wherein the metallic salt added is selected from a salt of strontium, cadmium, magnesium or zinc.

6. A method of treating solid waxy hydrocarbon produced by the Fischer-Tropsch process including melting said hydrocarbons and introducing an initiating peroxide and metal catalyst into the molten mass substantially as described.

An oxidised aliphatic Fischer-Tropsch wax when made by any of the methods claimed

in claims 1 to 6. 8. An oxidised aliphatic Fischer-Tropsch wax in accordance with claim 7 melting at a temperature of 180 to 210°F, having a negligible iodine value, an acid value of 10 to 20, an acetyl value of 35 to 53, a peroxide value of 400 to 5,000 p.p.m. a lactone value of 3 to 30, a saponification value of 30 to 60 and a consistometer hardness of 35 to 85 at a

temperature of 130 to 80°F 9. An oxidised aliphatic Fischer-Tropsch wax in accordance with claim 8 wherein the peroxide value does not exceed 500 p.p.m.

10. An oxidised aliphatic Fischer-Tropsch wax in accordance with any of claims 7 to 9, said wax containing in combination a metal taken from Group IIa or IIb of the Periodic 70

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