July 2, 1957 G. D. FRONMULLER ET AL 2,798,085

SYNTHETIC HARD WAX

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Fig. 1.

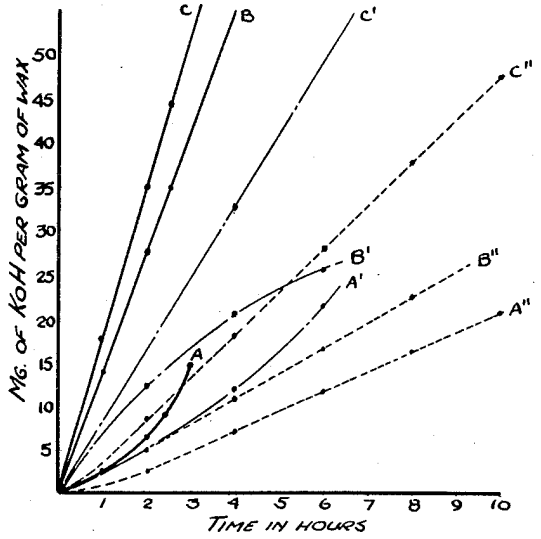


Fig. 2.

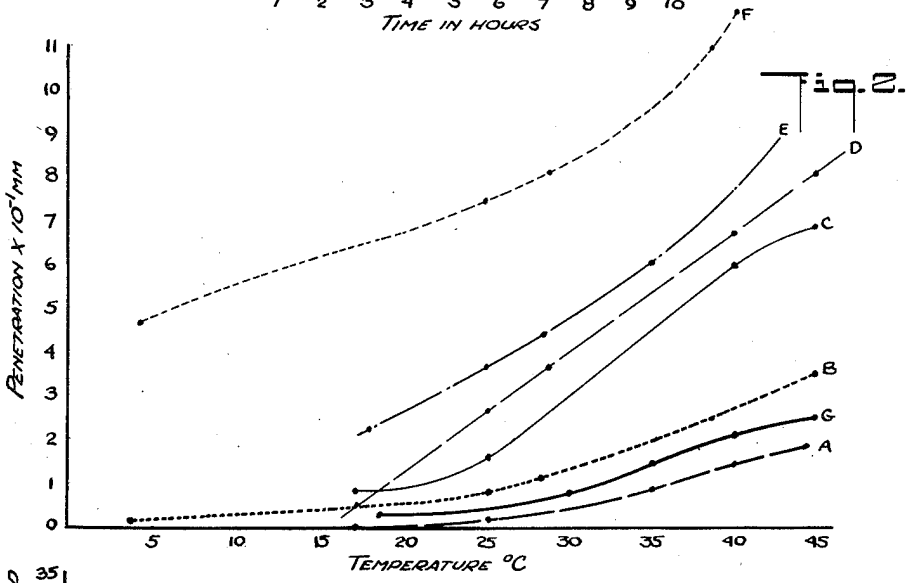
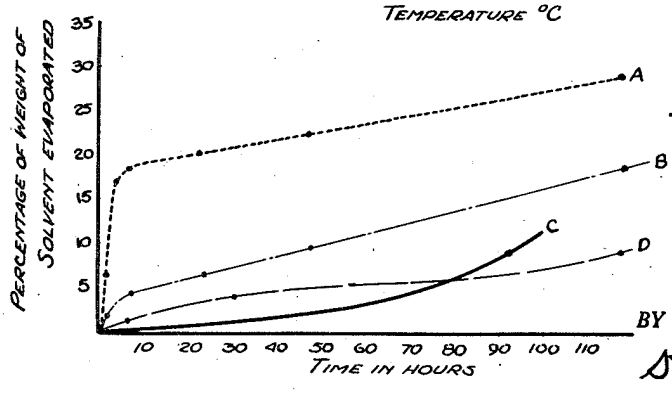


Fig. 3.



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SYNTHETIC HARD WAX

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17 Claims. (Cl. 260—450)

The present invention is directed to the production of synthetic waxes and more particularly to 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 hydrocarbons are oxidized in order to form fatty acids for the purpose of preparing soaps and the like. According to one such process, the oxidation was in the presence of a metallic soap 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 vapor state in the presence of a catalyst with oxyen-containing 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 vapor 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 economical 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 a raw material source which is plentiful, consistent and economical. A raw material which satisfies these conditions are 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 catalyst such as cobalt, iron or nickel, at about 200° C. The reaction involved is as follows:

$$nCO + 2nH_2 \xrightarrow[\text{Heat}]{\text{Cat.}} C_nH_{2n} + nH_2O$$

$$nCO + (2n+1)H_2 \xrightarrow[\text{Heat}]{\text{Cat.}} C_nH_{(2n+2)} + nH_2O$$

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 a Fischer-Tropsch hydrocarbon wax is oxidized 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 oxidized 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 conducting the process the hydrocarbon used is limited and held at a temperature of about 100° C. until the peroxide initiating agent is added. Air is bubbled through the mass continuously during the addition of the peroxide. The temperature is then raised to 150°–155° C. The temperature may then be reduced to about 120° C. and a suitable metal added thereto such as the metals of group II–*a* and II–*b* of the periodic table. The initiators are generally either organic or inorganic peroxides, and are capable of acting as agents promoting the oxidation of the hydrocarbon.

In the oxidation of the Fischer-Tropsch hydrocarbons, some of the main products are fatty acids and their esters. Therefore, the oxidation was controlled by following the formation of acids (acid value) and esters (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 oxidation 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 A′, B′ and C′ 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 A″, B″ and C″ show the course of the oxidation of the same hydrocarbons where no catalyst at all is used.

The saponification curves C, C′ and C″ 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″ and C′ 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″ (no catalyst)	4.9
(2) Curve C′ (0.2% benzoyl peroxide)	8.1

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

It was also found that if a secondary catalyst is used in conjunction with the initiating agent (peroxides) the rate of oxidation was further increased. For example, if the oxidation of Fischer-Tropsch hydrocarbons is carried out using benzoyl 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)	Rel. Rates
(1) Curve C'' (no catalyst)	4.9	1
(2) Curve C' (0.2% benzoyl peroxide)	8.1	1.6
(3) Curve C (0.2% benzoyl 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 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 oxidized wax has an ash content of 0.3–0.4%. It is also possible 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" centers upon which the oxygen and hydrocarbon molecules 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 benzoyl peroxide is added and an oxidizing 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°–155° C. for the duration of the oxidation.

The air which is passed through the reaction 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 a dew point of 55° F. The air rate used was 10–15 liters per minute per pound of hydrocarbon to be oxidized. 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 benzoyl peroxide
60 grams zinc (mossy or sheet)

The Fischer-Tropsch wax used in this run had a melting point (A. S. T. M.) of 220° F., 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 was compiled in the unoxidized and oxidized waxes. This data is given in Table C (below) along with absorption data in 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.

TABLE C

	Wave Length (microns)					
	3.48	5.77	6.86	8.87	13.75	13.95
	Optical Density					
(1) Unoxidized Fischer-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

It is believed that these peaks represent different types of carbon-hydrogen bonding. 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, esters, aldehydes, 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 the oxidized and unoxidized Fischer-Tropsch wax samples. The main indication which this data presents is that there was no indication of carbonyl bonds in the Fischer-Tropsch unoxidized wax. It is believed that esters and acids which are the oxidation products cause the peak absorptions, in the oxidized run. The wax samples were melted as they were tested with the infra red rays.

This wax was melted at a temperature of 100°–105° C., then the benzoyl peroxide was added. The air was then bubbled through the molten wax at a rate of 12–15 liters a minute. The temperature was then raised to 120° C. and at this point the zinc was added to the reaction mixture. The temperature again was raised to 150°–155° C. for the duration of the reaction. The oxidation was followed by testing 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 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 laboartory runs a three necked round bottom glass flask was used as the reactor. The air was injected by means of a glass tube or a fritted glass gas disperserator 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 condensors and expansion chambers so that any condensable material may be collected. On larger scale operations (50 lbs. of wax) steam jacketed stainless steel or aluminum reactors may be used.

Among the desirable properties of the wax are good emulsifying characteristics so that very fine particle size emulsions may be formed and a hard wax of a melting point of 85°–95° C. with a light color. 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 initiating agents may be used with satisfactory results. Other peroxides which were used were urea peroxide, lauryl peroxide, potassium persulphate. In general it is believed that those agents which are capable of producing free radicals may act as initiating agents in the oxidation of hydrocarbons. This is thought to be the case with the peroxide class both organic and inorganic. It is further 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.

Metal catalysts which were tried other than zinc were copper and iron. Their catalytic effects were not as great as zinc, plus the fact that both copper and iron cause undesirable color formation in the oxidized wax. Therefore, the optimum catalytic effect was found with the use of 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 to 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 discolor, (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. 140°–155° C.

Example 2

In view of the fact that there is a large demand for a hard, high melting wax which can form a fine particle size emulsion, 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 wax 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-propanol.
(3) 8 parts of oleic acid.
(4) 1 part of borax.
(5) 240 parts of water.

Melt (1) to 100° C. 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 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 (e. g. 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 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. It is believed that most multivalent metals 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. Those metals which appear to give the best results in decreasing the penetration values of the waxes are those elements in group II*a* and group II*b* in the periodic table. 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. Other metals tried thus far were magnesium, zinc, iron and copper. 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.

TABLE D

Wax

A Flora carnauba wax.
B Oxidized F-T 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.
E Oxidized F-T 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 C (strontium salt of the higher distilled oxidized wax) compares very favorably 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:

If the oxidation is carried out in the presence of the metal or one of its salts, 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. It is believed feasible to carry out the oxidation in the presence of another metal, e. g. strontium or 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, on 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 oxidized F-T wax with an acid value of 23
(2) 5 gr. of cadmium acetate

On heating (1) and (2) together for 1 to 3 hours causes the acid value to decrease and the hardness of the wax to also increase. Similar results were obtained with copper acetate, 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 fatty acids.

Example 5

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

(1) 1250 grams of oxidized F-T wax with an acid value of 16.7 and a zinc ash content of .3%.
(2) 12.5 grams of potassium hydroxide
(3) 91.25 grams of strontium chloride ($SrCl_2.6H_2O$)
(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 mole as compared to .342 mole of strontium chloride. The final wax has a total ash content of 1.25% on the weight 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 (e. g. 1250×16.7). 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 resulted 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 remain 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 cadium iodide, copper chloride, magnesium chloride, zinc chloride, etc. 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 color 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 color 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 polish.

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

(*a*) The unoxidized Fischer-Tropsch wax
(*b*) Oxidized Fischer-Tropsch wax
(*c*) Oxidized Fischer-Tropsch wax which contained the cadmium salt of the fatty acids in the wax
(*d*) Flora 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 weighted 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 unoxidized Fischer-Tropsch wax in solution in 57 parts of Stoddard solvent. Curve B is directed to the solvent retention of 20 parts 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 cadimum salt. Curve D indicates for comparison purposes, the solvent retention of Flora carnauba wax.

The solvent retention properties of the product of the present invention are greatly improved when the wax is in the form of the metal salt. In this product the solvent evaporates 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 about 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 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 product is from about 5,000 to about 400, and preferably the value should be not over about 500.

The distillation range of the product at about 7 mm. mercury pressure is as follows:

Temperature, ° C.	Distillate, Percent
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 product include the lactone value which measures the lactones and some of the acid anhydrides present. The lower the value 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. 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.
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.

We claim:

1. A method of treating solid waxy Fischer-Tropsch hydrocarbons which comprises melting said hydrocarbons, introducing into the molten mass an initiator of reaction, said initiator being a peroxide soluble in said hydrocarbon and having a decomposition temperature higher than the melting point of said wax, passing an oxygen containing gas therethrough, introducing a metal taken from group II*b* of the periodic table of elements while introducing said oxygen containing gas and heating

to a substantially higher temperature but below the decomposition temperature of the wax for several hours.

2. A method according to claim 1 wherein, after said heating there is added a compound of a metal having a valence of more than one to form soaps of acids in said product to increase hardness thereof.

3. A method according to claim 1 wherein, after said heating there is added a compound of a metal having a valence of more than one taken from groups IIa and IIb of the periodic table of elements to form soaps of acids in said product to increase hardness thereof.

4. A method according to claim 1 wherein, after said heating there is added a compound of iron to form soaps of acids in said product to increase hardness thereof.

5. A method according to claim 1 wherein, after said heating there is added a compound of copper to form soaps of acids in said product to increase hardness thereof.

6. A method of treating solid waxy Fischer-Tropsch hydrocarbons which comprises melting said hydrocarbons, introducing into the molten mass an initiator of reaction, said initiator being a peroxide soluble in said hydrocarbon and having a decomposition temperature higher than the melting point of said wax, passing an oxygen containing gas therethrough, introducing a metal taken from group IIb of the periodic table of elements while introducing said oxygen conaining gas and heating to a substantially higher temperature but below the decomposition temperature of the wax for 6 to 7 hours.

7. A method of treating solid waxy Fischer-Tropsch hydrocarbons which comprises melting said hydrocarbons, introducing into the molten mass an initiator of reaction, said initiator being a peroxide soluble in said hydrocarbon and having a decomposition temperature higher than the melting point of said wax, passing an oxygen containing gas therethrough, introducing a metal taken from the group consisting of zinc, copper and iron while introducing said oxygen containing gas and heating to a substantially higher temperature but below the decomposition temperature of the wax for several hours.

8. A method of treating solid waxy Fischer-Tropsch hydrocarbons which comprises melting said hydrocarbons, introducing into the molten mass an initiator of reaction, said initiator being a peroxide soluble in said hydrocarbon and having a decomposition temperature higher than the melting point of said wax, maintaining a temperature of about 100°–125° C. and passing an oxygen containing gas therethrough, introducing a metal taken from group IIb of the periodic table of elements while introducing said oxygen containing gas and heating to a substantially higher temperature but below the decomposition temperature of the wax for several hours.

9. A method according to claim 8 wherein the temperature is raised to about 140°–175° C. after the addition of said metal.

10. A method according to claim 9 in which said metal is zinc.

11. An oxidized aliphatic wax melting at temperatures of 180°–210° F., the infra red absorption at wave length 8.87 microns showing the absence of a peak and showing a peak at 5.7, having a negligible iodine value, an acid value of 10–20, and an acetyl value of about 35–53, the saponification value being about 30–60.

12. An oxidized aliphatic wax melting at temperatures of 180°–210° F., the infra red absorption at wave length 8.87 microns showing the absence of a peak and showing a peak at 5.7, having a negligible iodine value, an acid value of 10–20, and an actyl value of about 43, the saponification value being about 30–60.

13. An oxidized aliphatic wax melting at temperatures of 180°–210° F., the infra red absorption at wave length 8.87 microns showing the absence of a peak and showing a peak of 5.7, having a negligible iodine value, an acid value of 10–20, and an acetyl value of about 35–53, the saponification value being about 30–60, the peroxide value being about 400–500 P. P. M.

14. An oxidized aliphatic wax melting at temperatures of 180°–210° F., the infra red absorption at wave length 8.87 microns showing the absence of a peak and showing a peak at 5.7, having a negligible iodine value, an acid value of 10–20, and an acetyl value of about 35–53, the saponification value being about 30–60, said wax containing soaps of metals having a valence of at least 2.

15. An oxidized aliphatic wax melting at temperatures of 180°–210° F., the infra red absorption at wave length 8.87 microns showing the absence of a peak and showing a peak at 5.7, having a negligible iodine value, an acid value of 10–20, and an acetyl value of about 35–53, the saponification value being about 30–60, said wax containing soaps of metals taken from groups IIa and IIb of the periodic table of elements.

16. An oxidized aliphatic wax melting at temperatures of 180°–210° F., the infra red absorption at wave length 8.87 microns showing the absence of a peak and showing a peak at 5.7 having a negligible iodine value, an acid value of 10–20, and an acetyl value of about 35–53, the saponification value being about 30–60, said wax containing soaps of iron.

17. An oxidized aliphatic wax melting at temperatures of 180°–210° F., the infra red absorption at wave length 8.87 microns showing the absence of a peak and showing a peak at 5.7, having a negligible iodine value, an acid value of 10–20, and an acetyl value of about 35–53, the saponification value being about 30–60, said wax containing soaps of copper.

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