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DISTILLATION OF FATTY OILS

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5 Claims. (Cl. 202—56)

This invention relates to the treatment of fatty oils, that is, oils of vegetable or animal origin characterized by the presence of fatty acid esters of either monatomic or triatomic alcohols or both. As examples I may instance the vegetable drying oils, such as linseed oil and perilla, and vegetable semi-drying oils, such as corn oil, cottonseed oil, sesame oil, colza oil, castor oil, etc., and animal oils, such as menhaden oil, sardine oil and lard oil.

The object is to provide a treatment whereby from commercial crude oils there may be produced by commercially practicable means a light distillate substantially consisting of completely saturated molecules and a residue comprising highly unsaturated molecules. Both these products may be of commercial value and either one or the other might be considered the primary product and the other as a by-product.

The unsaturated residue may be utilized as a drying oil in the paint and varnish industry, in the manufacture of printing inks and lithographing varnishes, and in the manufacture of putty, linoleum and other products. The light distillate consisting of a mixture of organic acids, esters and alcohols (with or without a small percentage of free fatty acids) is saturated in nature and low in viscosity and specific gravity and, further refined and separated if desired, is useful in the manufacture of pharmaceutical products; as "extreme pressure" or "active oiliness" agent or surface tension reducer for use in petroleum lubricants, and in many chemical industrial applications.

It is possible under laboratory conditions by heating under high vacuum at temperatures below 600° F. to produce from a crude fatty oil a distillate of the general character above referred to, but such process is not of commercial significance.

The process which I am about to describe is characterized by the dilution of the reacting substances in the oil with an inert non-reacting substance which controls the heat reactions with desirable results hereinafter more fully explained. By way of example merely I shall first describe in considerable detail a preferred application of my process as utilized in connection with the treatment of crude castor oil.

The oil is mixed mechanically with about 20% by weight of a petroleum wax of about 155° F. melting point and about 75 seconds Saybolt Universal viscosity at 210° F., this being a common commercial was available on the market as a by-product obtained in the refining of lubricating oils. The mixture is heated slowly in a closed vessel, either directly or indirectly, and when a temperature in the liquid of about 550° F. is reached, a reaction of thermal decomposition begins to take place with the evolution of vapor, the vapors being vented through a condenser and a vacuum pump so as to hold the process at a vacuum of about 660 millimeters (100 millimeters absolute pressure) or higher until about 50% of the total volume of oil is distilled, the temperature of the liquid being gradually increased during the process until a final temperature of about 675° F. is attained. The distillate so produced will be found on redistillation to have a fairly uniform boiling range commencing at about 275° F. and ending at about 675° F. at atmospheric pressure. A slight amount of water is a product of the reaction and this separates from the oily fraction which is immiscible with water although miscible in all proportions with petroleum fractions and the common organic solvents. It is the oily fraction which I herein refer to as the distillate.

It is preferable to carry out the process by which the distillate is produced, as described above, within containers of stainless steel or enameled iron, since at elevated temperatures both the distillate and the residue are darkened in color by contact with iron, and this would be undesirable in some of the applications of the product.

Of the total petroleum wax it will be found that a minor proportion will be in the distillate and the balance in the residue. The wax may be separated by any suitable process, one such process, not new in itself, being to mix the material with a light liquid agent in which the wax is insoluble or nearly so and in which the product is miscible in all proportions and using additional quantities of such agent to give the mixture a viscosity suitable for handling. The agent may be petroleum naphtha of a boiling range of from 150 to 250° F. although, for example, ketones such as acetone or methyl ethyl ketone may be used. In the case of the distillate about an equal volume of petroleum naphtha would be utilized while in the case of the residue a ratio of about three volumes of naphtha to one of residue would provide a suitable viscosity for handling. The mixture is chilled by artificial refrigeration to from 0 to —20° F. and the wax which separates by precipitation is recovered by settling and centrifuging. The naphtha may be recovered by simple distillation and condensation. Both products may be further refined, as, for in-

stance, by filtering through fuller's earth or activated alumina, if it is desired to do so, and the distillate may be separated further into components by physical and chemical means. While, as I have indicated, this method of separation is not in itself new, an important feature of my invention is that it permits such easy and effective separation of the diluent from the products of the oil.

The distillate of fatty oil produced in accordance with my invention, and of which the castor oil distillate above specifically described is an example, may be described as a liquid distillate of a boiling range below 850° F. at atmospheric pressure, of specific gravity 0.80 to 0.950, of viscosity not exceeding 200 seconds S. U. V. at 100° F., and chemically consisting a mixture of esters and alcohols of fatty acids with or without small amounts of free fatty acids and saturated in nature.

The residue may be described as being generally similar chemically and physically to the crude fatty oil from which it was derived, except that the viscosity will be increased considerably, and the specific gravity will be increased slightly. The drying qualities, i. e., the capacity of the product to dry by oxidation and/or polymerization to a hard lustrous, highly resistant film as is desirable in a paint or varnish, are greatly improved in the residual product over the fatty oil which taken as the raw material for the process.

The actual chemical reactions which take place during the process are complex and progressive in their nature. I shall state what I believe to be the general course of the operation although it will be understood that I advance this as a theoretical explanation which is to an extent speculative, and I am not to be understood as binding myself to any particular theory as the results obtained are empirically demonstrated.

Ordinarily if fatty oils are heated alone, a fairly rapid polymerization takes place and a semi-solid or solid mass forms with little or no distillation. With the inert diluent present the molecules are insulated from each other to a large extent and a thermal decomposition takes place beginning at a critical temperature and proceeding with elevation of temperature whereby the moderately unsaturated molecule of the raw material splits into a light, substantially completely saturated molecule and a heavy, unsaturated molecule. The critical temperature at which the thermal decomposition commences is characteristic of the particular fatty oil being treated and will be found to vary among the different oils. The distillate is saturated in chemical nature and much lower in specific gravity than the starting raw material, this being so whether it represents a minor or a major fraction of the raw material. The residue is usually of about the same degree of unsaturation as the raw oil and of about the same specific gravity or slightly higher. It appears that the function of the wax diluent is to make possible the splitting off of small saturated molecules from the larger unsaturated molecules with separation of the former by distillation and condensation, the action being at least in part a true chemical decomposition rather than a mere physical separation. It is the function of the use of vacuum to facilitate the progress of the reaction by rapid removal of the volatile products and by a slight lowering of the above mentioned critical temperature. The same purpose might be served by introducing an inert gas, such as steam, nitrogen or carbon dioxide, into the body of the reacting oil in a manner which will be well understood by those skilled in the art. It may be that a certain amount of polymerization also occurs simultaneously with this decomposition, especially after a high percentage of light saturated material has been distilled off, but if this occurs it is believed to be in small degree only.

It will, of course, be understood that there will be a variation among the various fatty oils in the yields, in the reacting temperatures and the nature of the end products. The inert or diluent material may be petroleum wax of a suitably high melting point, say 120° F. or more. Various suitable waxes are commercially available. The wax may be either the white crystalline or so-called amorphous or microcrystalline variety, provided in either instance that it is relatively free from impurities and miscible in all proportions at the critical reaction temperature with the fatty oil to be treated. 10% or more would be used. As already indicated, heat may be applied either directly or indirectly, and heating under a vacuum, while desirable, is not necessary. The range of reaction temperatures would be between 400° F. and 850° F. with a more usual range of 550° to 675° F. and the heating may be either by a batch process or by continuous distillation. The higher temperatures normally would be attained when a greater yield of distillate was desired. If the primary object is to procure distillate, as much as 80% of the crude oil may be distilled over, whereas if a drying oil residue is primarily desired, as little as 10 or 15% by weight may be distilled. From 40 to 60% by weight of the original oil as distillate might be considered normal operation. The wax occurring in the distillate will ordinarily range from 5 to 40% of the original amount.

I have referred to fatty oils and have instanced a substantial number of crude, vegetable and animal oils to which the process is applicable, but obviously a complete catalog is impossible. In view of the complicated chemical nature of oils and the substantially unlimited range of natural products, it will be clear that it is quite possible that certain crude oils could not successfully be treated, but this will not mislead those skilled in the art since a simple test not of an inventive nature will disclose the fact for any given case. I am not aware of any instances. Moreover, certain crude products might be theoretically subject to the process but for economic or collateral reasons the treatment would be of little utility.

I am aware that it has been proposed to add gums or resins to tung oil when heating the same to prevent it from setting as a solid gel, the purpose apparently being to check or slow up a polymerization process. This is to be contrasted with my process which is rather one of controlled thermal decomposition with a distillation and resultant separation to provide two products.

I am aware that the invention may be embodied in other specific forms without departing from the spirit or essential attributes thereof, and I therefore desire the present embodiment to be considered in all respects as illustrative and not restrictive, as is in fact clear in several matters from the description itself. Reference is to be had to the appended claims to indicate those principles of the invention exemplified by the

particular embodiment described and which I desire to secure by Letters Patent.

I claim:

1. In the treatment of fatty oil the step which comprises mixing therewith about 10% or more of a petroleum wax of a melting point of at least 120° F. and heating the same to a temperature between 400° and 850° F. to produce by destructive distillation a fraction equal to from 10% to 80% of the original oil comprising primarily light, substantially completely saturated molecules with a residue comprising primarily heavy, highly unsaturated molecules.

2. In the treatment of fatty oil the step as defined in claim 1 wherein the reaction temperature is between 550° and 675° F. and the distillate is from about 40% to about 60% of the original oil.

3. In the treatment of fatty oil the step which comprises heating the same at a temperature between 400° and 850° F. under an absolute pressure of not more than about 100 millimeters in the presence of an inert non-reacting hydrocarbon wax to produce therefrom by destructive distillation a relatively light saturated fraction and a relatively heavy unsaturated residual fraction.

4. In the treatment of fatty oil the step which comprises mixing therewith an inert hydrocarbon wax having a melting point of at least 120° F. in amount substantially to inhibit polymerization during the heating hereinafter referred to and heating the mixture to a temperature of 400° F. to 850° F. to cause to be produced and distilled therefrom in weight equal to a substantial fraction of the original fatty oil a distillate saturated in chemical nature and of relatively low specific gravity as compared with the original oil.

5. In the treatment of fatty oil the steps comprising mixing therewith petroleum wax having a melting point of at least 120° F. in amount substantially to inhibit polymerization during the heating hereinafter referred to and then separating a substantial proportion of the oil in the form of a liquid distillate saturated in chemical nature by heating at from 400° F. to 850° F.

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