This invention relates to fatty acid esters and fatty acids and more particularly to a process for refining such materials.

Fatty acid esters and fatty acids have been refined by many methods in order to improve their color. However, such processes generally suffer from one or more of such disadvantages as involving a substantial loss of refined product or of changing desirable properties of the fatty materials treated, or of requiring excessive processing equipment and materials, or of improving the color to only a slight degree. Such disadvantages are encountered for example when treating fatty acid esters and fatty acids by adsorption or extraction methods whereby a considerable loss of yield of refined material is obtained, or by treating with strong reagents such as concentrated sulfuric acid, thereby destroying considerable fatty materials, or by hydrogenation, oxidation or chlorination which may involve not only change in properties, but loss in yield or use of expensive equipment.

It is an object of this invention to provide an improved process for refining fatty acid esters and fatty acids.

It is a further object of this invention to provide an improved process for refining fatty acid esters and fatty acids, which is extremely simple and economical.

It is a further object of this invention to provide an improved process for refining fatty acid esters and fatty acids in which the yield of refined fatty acid esters or fatty acids based on the material treated is substantially quantitative.

A still further object of this invention is to provide a process which will effect a substantial improvement in the color of the treated fatty acid esters or fatty acids without substantially affecting the other properties of these substances.

Other objects of this invention will appear hereinafter.

The objects of the invention are accomplished broadly by treating the unrefined fatty acid esters or fatty acids under ionizing conditions with a metal above hydrogen in the electromotive series and an acid salt. The treatment is carried out under conditions which will provide intimate contact between the fatty acid esters or fatty acids and the acid salt and the metal. As a result, the color of the fatty acid esters or fatty acids is substantially lightened with practically no loss in yield and no perceptible effect on the properties of the materials treated other than color.

In accordance with the process of this invention the treatment may be carried out on the fatty acid esters or fatty acids in the molten condition or on these materials dissolved in a suitable solvent. After the treatment is completed, the fatty acid esters or fatty acids or their solution may be washed to remove any water soluble material, and filtered to remove any suspended matter. If desired, the solution may be filtered prior to the water washing step. In the case of treatment of the fatty acid esters or fatty acids in solution, the fatty acid esters or fatty acids of improved color may then be recovered by evaporation of the solution.

The fatty acid esters and fatty acids which may be refined according to the process of this invention are the fatty acid esters such as vegetable and animal oils, fats and waxes, and the fatty acids which may be derived from them. Examples of such materials are vegetable oils such as tung oil, linseed oil, cottonseed oil, palm oil, etc., or animal oils such as bone oil, whale oil, beef tallow, etc., fish oils, such as menhaden oil, sardine oil, etc., waxes such as sperm oil, beeswax, carnauba wax, montan wax, etc.; and fatty acids such as commercial oleic acid, fatty acid soap stocks, and fatty acids derived from any of the above fats, oils and waxes.

The process of this invention is especially valuable when applied to the unsaturated or drying type fatty acid esters and fatty acids such as, for example tung oil, linseed oil, menhaden oil, oleic acid, linseed oil fatty acids, etc. In the case of treating such drying type fatty acid esters and fatty acids, the advantage of the process of this invention, of having no perceptible effect on the physical properties of the acid esters and fatty acids other than color is especially marked since other processes of refining generally appreciably alter these properties in lightening the color.

As solvents for the fatty acid esters and fatty acids when the treatment is carried out on the fatty acid esters and fatty acids in solution, any solvent for these materials may be employed which is itself inert in the treatment. Among such solvents are, for example, mononyclic aromatic hydrocarbons such as benzene, toluene, xylene, etc.; petroleum hydrocarbons such as gasoline, V. M. and F. naphtha, hexane, heptane, etc.; hydrogenated petroleum hydrocarbons, such as those known in the trade as "Solvesso" solvents; turpentine, terpenes, and solvents such as ethylene dichloride, carbon tetrachloride, cyclohexane, methyl cyclohexane, F menthan, tetrahydro and decahydronaphthalene, isopropyl ether, dichloroethylethyl ether, etc. Where
the treatment is carried out on the fatty acid esters or fatty acids in solution, the concentration of the fatty acid esters or fatty acids in solution is not critical and may be selected to provide adequate solubility of the solution. In general, a concentration within the range of about 10% to 75% by weight may be used, but preferably the concentration is held within the range of 30% to 60% by weight. The acid salts which may be used in the process of this invention are those which contain one or more replaceable hydrogen atoms, such as, for example, sodium acid sulfate, potassium acid sulfate, monoammonium acid phosphate, disodium acid phosphate, monopotassium acid phosphate, dipotassium acid phosphate, calcium acid phosphate, lithium acid phosphate, etc.

Any of the metals above hydrogen in the electromotive series may be employed. It will be preferable, however, to use such metals which form salts having no appreciable discoloring effect on the fatty acid esters or fatty acids. The metals are used preferably, though not necessarily, in a finely divided form. Use of such metals as, for example, cadmium, aluminum, magnesium, tin and zinc is desirable. Zinc in the form of zinc dust, mossy zinc, or zinc powder is particularly well adapted to the process.

In order to produce the necessary refining action, the metal and acid salt should be contacted during the refining process under ionizing conditions. By ionizing conditions are meant conditions under which the acid salt and metal may dissociate, as in the presence of an ionizing solvent such as water. It is, therefore, desirable that at least a small amount of water or some other ionizing solvent be present in order that such ionizing conditions are realized. Thus, the acid salt may be used in the form of a hydrate if it forms one, or in the form of an aqueous solution. Sodium acid sulfate in the form of its monohydrate is particularly desirable. Alternatively a small amount of water or other ionizing solvent may be added. An aqueous solution containing from about 20% of the acid salt to a saturated solution of the salt at the temperature employed may be used.

It will be understood that the acid salt and the metal above hydrogen in the electromotive series are both in contact with the fatty acid esters or fatty acids during the treatment. To provide satisfactory contact of the acid salt and metal with the fatty acid esters or fatty acids, it will be desirable to employ vigorous agitation, since the refining action will be appreciably accelerated thereby.

The speed with which the fatty acid esters or fatty acids become refined is also dependent on the temperature employed in the treatment. While the treatment may be carried out at room temperature, the rate of refining is slow for practical purposes. It is preferable to carry out the treatment at elevated temperatures, and temperatures as high as 200° C. have been found to be satisfactory, although even higher temperatures are possible. A temperature within the range of about 70° C. to about 125° C. is preferable.

When carrying out the treatment of the fatty acid esters or fatty acids in solution it is convenient to operate at the reflux temperature of the solvent. When the treatment is to be carried out above the normal boiling point of the solvent, use of superatmospheric pressure is resorted to. The pressures employed are in no way limiting on the process, and the equipment employed will generally determine the maximum pressure which is practical.

The time of treatment of the fatty acid esters or fatty acids may be varied to produce the results desired. The time necessary to provide satisfactory refining will depend on such factors as the amount of color bodies present in the material refined, the temperature of the treatment, the amount of acid salt employed, and other factors. For this reason it is impossible to state any definite time except in relation to a given set of conditions. Under usual conditions reaction time between about 10 minutes and 5 hours may be satisfactory, although depending on specific conditions, longer or shorter contact times are entirely possible.

The amount of acid salt used may vary over a wide range depending on factors such as the particular acid salt used, the degree of refining desired, the particular conditions of treatment, as well as other factors. Considering the acid salt on an arbitrary basis, for example, ratios of fatty acid esters or fatty acids to acid salt of as high as 40 to 1 on a weight basis are practical. In general, however, ratios varying between 10 of fatty acid to 1 of acid salt, down to 1 of fatty acid ester or fatty acid to 1 or more of acid salt are desirable. A preferred ratio is approximately 3 of fatty acid esters or fatty acids to 1 of acid salt.

The amount of metal above hydrogen in the electromotive series used is not particularly critical. It will be desirable, however, to have an amount of such metal present which will be an excess over the theoretical amount required to react with the acid salt present.

The process of refining fatty acid esters or fatty acids in accordance with this invention may also be carried out as a continuous process if desired. Thus, for example, a solution of the fatty material may be treated with the metal above hydrogen in the electromotive series and an acid salt in a suitable vessel while additional fatty acid esters or fatty acid solution is fed into the vessel continuously and refined fatty acid esters or fatty acid solution is withdrawn from the vessel at substantially the same rate.

The acid salt and metal may be replenished as necessary and the refined solution continuously filtered, water washed, and passed to a continuous evaporator for removal of the solvent.

The use of an inert atmosphere such as provided by nitrogen, hydrogen, carbon dioxide, etc., during the various refining steps is often of assistance in obtaining pale colored products. As illustrative of the improved process of refining fatty acid esters or fatty acids in accordance with this invention, the examples below are cited as typical of the various embodiments.

The colors shown are the colors corresponding to grades of the Standard U. S. Rosin type.

Example 1

Seventy-five parts by weight of a commercial "Red OIl" or oleic acid was dissolved in 310 parts by weight of benzene. This solution was refluxed for 1 hour with 15 parts by weight of zinc powder and 23 grams of sodium acid sulfate monohydrate. The treated solution was filtered to remove suspended matter, water washed, and the solvent removed from the refined oil by vacuum distillation. The color of the oil was improved from an 1- to a WG grade.
The iodine number of the oil was substantially unchanged.

**Example 2**

The experiment of Example 1 was duplicated with tung oil of color grade N+. The oil was refined to a WW grade.

**Example 3**

Sixty parts by weight of menhaden oil was dissolved in 120 parts by weight of benzene and refluxed for one hour with 25 parts by weight of zinc powder and 40 parts by weight of sodium acid sulfate monohydrate. The benzene solution was decanted, water washed and the solvent removed from the refined oil by vacuum distillation. The color of the oil was improved from a G+ to an M grade.

**Example 4**

Sixty parts by weight of menhaden oil was heated to 95° C. with 20 parts by weight of zinc and 60 parts by weight of 50% aqueous monosodium acid phosphate. The mixture was maintained at this temperature for 45 minutes under constant agitation. The oil was separated from the treating solution, filtered and water washed. The color of the oil was improved from a G+ to a K grade.

**Example 5**

25 g. zinc powder and 40 g. NaHSO4 · H2O was added to a solution of 100 g. carnauba wax in 400 g. ethylene dichloride, and the mixture refluxed for 1 hour with agitation. The solution was decanted from the bleaching agent while hot, and water washed. Evaporation of solvent by vacuo distillation left a substantially quantitative yield of bleached wax which was substantially lighter in color than the original (from dark brown to light brown).

**Example 6**

A refining treatment was carried out exactly as in Example 5, except the wax refined was montan wax. The refined material was substantially lighter than the original (from black gray to gray). Because of the opacity of the original and bleached waxes, an accurate color could not be obtained.

It will also be understood that the details and examples hereinafter set forth are illustrative only and that the invention as broadly described and claimed is in no way limited thereby.

What I claim and desire to protect by Letters Patent is:

1. The process of refining a material selected from the group consisting of fatty acid esters and fatty acids which consists of treating said material under ionizing conditions with a metal above hydrogen in the electromotive series and an acid salt.

2. The process of refining a material selected from the group consisting of drying type fatty acid esters and drying type fatty acids which consists of treating said material under ionizing conditions with a metal above hydrogen in the electromotive series and an acid salt.

3. The process of refining a material selected from the group consisting of fatty acid esters and fatty acids which consists of treating said material under ionizing conditions with a metal above hydrogen in the electromotive series and sodium acid sulfate.

4. The process of refining a material selected from the group consisting of drying type fatty acid esters and drying type fatty acids which consists of treating said material under ionizing conditions with zinc and sodium acid sulfate monohydrate.

5. The process of refining a material selected from the group consisting of drying type fatty acid esters and drying type fatty acids which consists of treating said material under ionizing conditions with sodium acid sulfate monohydrate and zinc at a temperature within the range of about 70° to about 125° C.

6. The process of refining linseed oil which consists of treating said linseed oil under ionizing conditions with sodium acid sulfate monohydrate and zinc at a temperature within the range of about 70° to about 125° C.

7. The process of refining tung oil which consists of treating said tung oil under ionizing conditions with sodium acid sulfate monohydrate and zinc at a temperature within the range of about 70° to about 125° C.

8. The process of refining menhaden oil which consists of treating said menhaden oil under ionizing conditions with sodium acid sulfate monohydrate and zinc at a temperature within the range of about 70° to about 125° C.

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