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METHOD OF PREPARING OXIDIZED DRYING OIL

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This invention relates to a method of reducing the color formed in an air-blown or oxidized unsaturated vegetable oil when it is subsequently heated to cause it to react, polymerize or harden, and to the product of this method.

It is well known that when pre-oxidized or blown vegetable drying or semi-drying oils are heated above the temperature at which they were oxidized for the purpose of increasing their body or viscosity, or in making a varnish, the color of the oil deepens markedly. In most cases the darkening is far beyond the color permissible in a varnish or paint vehicle, except for colors such as maroon, green or black, where the color of the vehicle is not an important factor. This darkening on heating has therefore largely precluded the use of blown oils in making varnishes where heating is necessary to combine or dissolve resins with the oil. In making a vehicle for a light colored paint, the color of the original blown oil is usually very light, but blown oils without further heat treatment have poor aging qualities when used for this purpose. On the other hand, if the blown oil is heated to 500–600° F., its aging qualities are greatly improved, but the color has been darkened beyond the desirable range.

While the method described hereinafter has usefulness in blown drying oils generally, I have found that it is particularly effective in the blown oils described in my U. S. Patent No. 2,178,604 wherein the oxidation is carried out at a temperature below the usual range of blown oil manufacturing temperatures. Low temperature oxidation, say below 200° F., results mainly in the addition of oxygen to double bonds and to active hydrogens in the fatty acid chains together with a considerable shift of non-conjugated double bonds to conjugated double bonds. I have found that there is a minor amount of splitting of the chains resulting in the formation of short chain oxidized and unoxidized acids, such as pyruvic, glycollic, capric acids, etc. Oxidation at higher temperatures, such as to about 250° F., also probably results in higher breakdown of the oxidized long chains and distillation or breakdown of the acids formed thereby. Present in all vegetable oils are the natural coloring materials: chlorophylls, xanthophylls, carotenes, etc. These coloring materials also probably are susceptible to oxidation of the same type as the fatty acid chains and to the subsequent breakdown with heat.

One characteristic of the oxidized products above mentioned is a greater or less degree of water solubility. The shorter oxidized chains are

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water-soluble, the longer oxidized chains appear to be hydrophilic.

My invention comprises the water extraction of the water-soluble (including hydrophilic) materials present in an oxidized drying oil, to produce a new oil free from these water-soluble or extractable substances.

When this new oil is bodied by heating it evolves much less gas on heating to higher temperatures, up to say 575–600° F., and results in a bodied oil having a color distinctly lighter than that of the same oil handled in the same manner and carried to the same body but not water extracted. I am aware that water extraction has been widely used to remove dirt, dust, mucilage and the like from raw oils, to remove alkali and soap from alkali-treated oils, or excess acid from acid-treated oils, but it should be noted that these operations are confined to refining of substantially unoxidized oils. The herein described method of removing color forming bodies from a highly oxidized light colored oil before bodying so as to obtain the advantage of their absence when the oil is bodied, is new to the best of my knowledge. The results are unexpected in that there is no reason to support that the water-extractable portions of a blown oil include substances which are potential color formers when heated, since the water extract is practically colorless.

The vacuum dehydration of the water extract gives an amber colored residue which is not adequate to produce the difference in color shown by the new extracted oil. The residue darkens on heating in air. However, when the water extract is remixed with the extracted oil and the water carefully evaporated from the oil, and the oil is bodied as ordinarily done, the final color is of the same order as that of unextracted oil.

In this connection, water extraction of the refined oil before oxidation has no appreciable effect towards color reduction of the oxidized and bodied oil derived therefrom.

As an example, an oxidized oil was produced according to the method described in my U. S. Patent No. 2,178,604, using a break free linseed oil which was then blown with air at 150° F. to an increase of .0040 in index of refraction over that of the unblown oil. This blown oil was thoroughly mixed and agitated at room temperature with an equal weight of water. An emulsion was formed which broke slowly. Six to twelve hours were required for a 99% break. Both layers remained somewhat cloudy even after long standing.

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On separation, the water layer showed a pH of 2.6 indicating a strong organic acid content. The loss in weight of the oil was 0.25%, representing water-solubles in the oil. The water-solubles reduce Fehling solution, indicating aldehydic and/or ketonic substances.

The oil layer after water extraction was heated cautiously at first up to 300° F., to eliminate its water content. It became clear at 275–300° F. On continuing the heating to 575° F., the bodying temperature, it darkened less and frothed less than the unextracted oil of the same derivation. It bodied at the same rate as the unextracted oil, and reached a Z–6 body in approximately one hour at 575° F. The color of the unextracted oil was about 15 on the Gardner-Holdt scale after bodying in air to Z–6. The color of the water extracted bodied oil was 12½ after similar treatment. With vacuum bodying under a CO_2 atmosphere the color of unextracted oil was 14, that of the extracted oil 11. The bodying time under vacuum was considerably longer—about 1½ hours to Z–6.

Since the water extract is acid, it would be expected that the acid number of the extracted oil would be lower than that of the unextracted oil. This is found to be the case, the reduction being 25 to 50%. In the above example the acid number dropped from 3.3 for the unextracted oil to 2.3 for the extracted oil.

On bodying in air a more unexpected change occurred, since the acid number of the bodied extracted oil rose only to 5.9 as compared with 13.1 for the bodied unextracted oil. I have no good explanation for this phenomenon. It may be that the water extraction removes a fat-splitting agent which results in the lower acid content noted. Whatever the cause, the low acid number bodied oil is a very desirable product since oils with high acid numbers are less water-resistant and react more with pigments and dry more slowly than those with lower acid numbers. It is well known that low acid bodied oils command higher premiums in the drying oil markets than high acid bodied oils.

Low temperature blown refined soya bean oil handled in the same manner as the above example and vacuum bodied to the same degree gave a color of 15 versus 18 for the comparable unextracted soya bean oil.

Again, refined linseed oil blown to J viscosity at 240° F. according to standard oil blowing practice (as distinguished from low-temperature blowing), after washing and bodying gave a distinctly lighter color than the unwashed oil when bodied.

From the foregoing examples it is observed that the water extraction of the blown oils mentioned which are characteristic of the classes of drying and semi-drying vegetable oils leaves oil residues which behave differently on heat bodying than the unextracted oils in that final color is generally reduced and certain oils show marked reduction in acid content in the bodied state.

These improvements were not predictable on the basis of the prior art since washing unoxidized oils subsequent to standard refining methods shows no comparable effects.

I claim as my invention:

1. The method of inhibiting darkening of vegetable drying oil which has been oxidized by treatment with an oxygen-containing gas at a temperature below 250° F. on subsequent heating above said temperature, which comprises water extracting said oxidized oil prior to said further heating thereof.

2. The process of treating unsaturated vegetable drying oil, which comprises blowing it with an oxygen-containing gas at a temperature below 250° F. and extracting it with water.

3. The process of treating unsaturated vegetable drying oil, which comprises treating the oil with an oxygen-containing gas at a temperature below 250° F., extracting it with water to remove potential color forming materials therefrom, and recovering a treated oil characterized by reduction in acid number and reduction in color darkening and frothing on heating above 250° F., as compared to a like derived, oxidized and heated oil in the absence of said intermediate water extraction treatment step.

4. The process of claim 3 wherein the oil is oxidized at a temperature below 200° F.

5. The process of claim 3 wherein the oxidized and water extracted oil is bodied by heating to a temperature between 500 and 600° F.

6. The process of claim 3 wherein the oxidized and water extracted oil is heated at a varnish cooking temperature to desired varnish oil viscosity.

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The following references are of record in the file of this patent:

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