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PREVENTION OF HYDROGENATION ODOR

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1 Claim. (Cl. 260—409)

The present invention relates to a process for the manufacture of hydrogenated fatty materials and has to do particularly with the preventing of, or the retarding of, the development of the characteristic odors of hydrogenation during the hydrogenation of such materials. The process of the present invention produces hydrogenated fats and oils of improved odor and flavor stability, particularly with respect to that phase of stability known as reversion.

In the hydrogenation of fats and oils there are usually developed characteristic odors which are undesirable and are commonly referred to as hydrogenation odors. These odors are generally developed during hydrogenation regardless of the type of oil treated. The hydrogenation odors may be successfully removed by deodorizing which is a process of steam distillation under high vacuum but the deodorized oils may still have undesirable flavor stability depending upon the type of oil treated. For example, soybean oil although deodorized to an acceptable odor and flavor may in a short time revert or develop undesirable odors and flavors.

An object of the present invention is to provide a process for the treatment of fats and oils whereby during hydrogenation the development of characteristic hydrogenation odors is substantially avoided.

Another object of the invention is to provide a process for the production of hydrogenated oils which are substantially free from hydrogenation odors and which have improved flavor stability.

It has been customary heretofore to subject fats and oils to hydrogenation without using any particular pretreatment to avoid or reduce the development of hydrogenation odors. Although odors developed during hydrogenation may be removed by deodorization, the resulting oils often show undesirable flavor stability and certain oils, such as soybean oil particularly, tend to develop so-called reversion odors and flavors in a relatively short time.

The usual treatment of fatty oils includes such steps as refining with alkali and bleaching with fuller's earth. Fats and oils intended for hydrogenation have also been occasionally deodorized prior to hydrogenation but the method of handling the oil subsequent to the predeodorization has been carried out in such a manner that any beneficial effects on the suppression of the development of hydrogenation odors have not been realized. In accordance with the present invention the aforesaid steps of pretreatment may be used and in addition the fatty material to be hydrogenated is subjected to special handling subsequent to the predeodorization and prior to the hydrogenation whereby the development of hydrogenation odors in the hydrogenation step is substantially avoided.

We have found that if oxidation can be prevented in the interval between predeodorization and hydrogenation, the resultant hydrogenated oil is substantially free of odors. Also an oil treated in such a manner has been found to be substantially nonrevertible and will retain its bland flavor for long periods in storage. The exact reason for this is not known but the theory is that the refined fatty oils contain a mother substance which, upon oxidation, gives a reactive volatile substance which may

or may not be odorless. However, this reactive volatile substance upon hydrogenation produces the typical odors associated with hydrogenation. These odors are considered to be directly related to reversion and if inhibited in accordance with our invention, an oil which is substantially stable and nearly bland in flavor and odor, is obtained.

In order to avoid oxidation it is necessary to adequately protect the oil from air during the deodorization and hydrogenation steps. Several different methods may be utilized alone or in combination to protect the oil from oxidation, namely, maintaining a sufficiently high vacuum on the oil, supplying a blanket of inert gas to the exposed surfacce of the oil, carrying on the deodorization and hydrogenation in the same vessel, and through the use of low temperatures. Theoretically there should be no odors present at all upon hydrogenation of the oil. However, if there is present a certain amount due to shortcomings with respect to technique, it may be deemed advisable to follow the hydrogenation step with a second deodorization step. This second deodorization step does not affect the reversion ability of the oil as the oil has already been made substantially nonrevertible by protection of the oil against oxidation, but this added deodorization will remove any odors which are due to the hydrogenation treatment itself.

More particularly one method of carrying out the invention is to protect the oil from oxidation during the interval between deodorization and hydrogenation. For example, the predeodorized oil may have a blanket of inert gas supplied thereto for the purpose of protecting the exposed surface of the oil from oxidation. In the alternative method a sufficiently high vacuum may be utilized during said interval with the result that no oxidation will take place.

According to another modification of the invention, the oxidation or deterioration of the oil in the interval between deodorization and hydrogenation may be avoided by promptly hydrogenating the oil after deodorization. A convenient method of conducting the hydrogenation immediately after deodorization is to perform both steps in the same apparatus. Thus, the oil may be deodorized in a modified hydrogenation vessel by the usual method of passing steam through the oil at a high temperature and under a high vacuum. When this process has been carried on sufficiently long to obtain a perfectly bland oil, the steam is cut off, a suitable nickel catalyst is introduced, and hydrogen is introduced into the vessel. The vacuum, of course, is broken by the introduction of hydrogen and when the oil is suitably agitated or stirred the hydrogenation process takes place. This process may then be controlled in the usual fashion. With such a process the vessel is modified to the extent that sufficient head space is provided, as in a normal deodorizer, to allow for splashing of the oil without excessive carry-over or entraining. Vacuum equipment is provided such as is common to the operation of conventional deodorizers. A means of agitating the oil is also provided which is a characteristic of hydrogenating vessels. A steam distributor is also a part of this vessel, such as is characteristic of conventional deodorizers.

According to a still further modification, the oil which has been predeodorized may be cooled while continuing the deodorization process consisting of passing steam through the oil under a high vacuum. This cooling process is carried on to a point where dangers from oxidation are averted, for example a temperature between about 150° and 130° F., and preferably below 140° F. The oil may now be removed from the deodorization vessel in the presence of air, pumped quickly to the hydrogenating vessel, and hydrogenated with the use of palla-

dium as a catalyst with temperatures not exceeding 212° F. These lower temperatures are such as to protect the oil so that when it is hydrogenated under these conditions the characteristic odors of hydrogenation are substantially avoided.

A still further modification of this invention is the protection of the oil between the steps of deodorization and hydrogenation with a blanket of inert gas. The use of such a material for protection enables the invention to be carried out in two pieces of equipment, a conventional deodorizer and a conventional hydrogenator, and permits hydrogenation at high temperatures with the use of the conventional nickel catalyst. In this modification the oil is deodorized in a conventional deodorizer by passing steam through the oil at temperatures of 400° F. to 470° F. under a high vacuum until the oil is substantially free from odor and flavor, 3–6 hours usually being necessary. The temperature is then reduced to 380° F., the steam is cut off, the vacuum is broken with nitrogen, and the oil is drawn into the hydrogenator which has been protected with an atmosphere of nitrogen through a line protected in the same fashion. The manner of drawing the oil from one vessel to another is carried out by a difference in pressure obtained by drawing a vacuum on the hydrogenating vessel. As soon as all of the oil has been drawn into the hydrogenating vessel, nickel catalyst dispersed in odor- and flavor-free oil is added, hydrogen is admitted to the vessel, and hydrogenation carried on by agitating the oil in the usual manner.

The following examples are considered illustrative of the process but are not to be construed as limiting the invention thereto:

Example I

A batch of refined bleached soybean oil was steam deodorized at 400–470° F. for 4–5 hours in a modified hydrogenator. Nickel catalyst was introduced into the vessel after cooling the deodorized oil to 350–370° F., and the hydrogenation followed under 0–10 pounds hydrogen pressure to a Zeiss butyro refractive index of 41.0. The oil was then cooled under vacuum to below 140° F. and filtered. Only traces of odor and flavor were detected in the filtered oil, these being easily removed by post deodorization with steam for ½ hour. Intermittent steps in the processing were carried out with the oil in a sealed vessel under a blanket of nitrogen. The same technique described above may be applied to low temperature hydrogenation, using a palladium catalyst.

A sample of the same oil was hydrogenated without predeodorization, as a control. The oil, after filtration, possessed considerable flavor and odor characteristics of hardened soybean oil, but after 5 hours' deodorization at 390–410° F., the oil was odorless and bland in flavor.

The flavor stability of the control was then compared to that of the above described predeodorized oil by incubating the samples in an oven at 140° F. After three days incubation, the control had deteriorated noticeably, now having the typical so-called reverted flavor, while the sample representing the predeodorized oil received the same flavor rating that it had received before incubation, indicating no flavor deterioration.

Example II

A batch of refined bleached soybean oil was steam deodorized in glass equipment for 5 hours at 445° F. After deodorization, the oil was hydrogenated immediately to a Zeiss butyro refractive index of 41.2 in glass apparatus at atmospheric pressure and 190–210° F. with a palladium on charcoal catalyst. After cooling to below 140° F. under hydrogen and removal of the catalyst, the oil was odorless and almost bland. Complete blandness was obtained by post deodorization at 390–410° F. for ½ hour.

A control hardened in the same manner without predeodorization and protection against oxygen possessed the strong flavor and odor characteristic of hydrogenated soybean oil. The control was deodorized 5 hours at 390–410° F.

After incubation of the oils for 1 week at 120° F., the control had reverted and was unacceptable for use in edible products, while the sample representing the predeodorized oil was almost bland, and possessed no odor or flavor characteristic of reverted soybean oil.

Example III

A sample of refined bleached cottonseed oil was steam deodorized at 445° F. for 4 hours. Immediately after removal from the deodorizer, the oil was hydrogenated in an iron converter to a Zeiss butyro refractive index of 40.3 using palladium catalyst at 200–210° F. and 5 lb. hydrogen pressure. The hardened oil had no odor or flavor characteristic of hydrogenation and, in fact, was almost bland. After post deodorization for ½ hour at 390–410° F. the oil was stable in flavor for 6 days when stored in an oven at 120° F.

Example IV

A batch of refined bleached linseed oil was predeodorized at 450–470° F. for 4–5 hours. Immediately after removal from the deodorizer, the oil was hydrogenated to a Zeiss butyro refractive index of 43.2 in stainless steel apparatus with a nickel catalyst, at 360–390° F. and 25 lb. hydrogen pressure. The resulting oil was practically devoid of hydrogenation odor. After filtration, the oil was post deodorized for 3 hours at 390° F. The bland product was stable in flavor for 1–2 days at 140° F.

Another batch of this linseed oil, hardened in the same manner without predeodorization and protection possessed a very strong hydrogenation odor, almost identical to that of hardened soybean oil. After filtration, bleaching, and deodorization for 5 hours at 390–410° F., the oil was odorless, but reverted rapidly in the mouth leaving a bitter metallic after taste. Only several hours at 140° F. were required to completely revert this oil.

Example V

Refined bleached whale oil was steam deodorized for 3 hours at 445° F. to blandness. After hardening to a Zeiss butyro refractive index of 42.0 with nickel catalyst at 370–390° F. and 20–30 lb. hydrogen pressure, the oil possessed only slight odor and flavor. Removal of the catalyst followed by post deodorization for 1 hour at 445° F. resulted in a colorless bland oil that was stable for approximately 3 days at 140° F.

A control batch hydrogenated similarly possessed an intense hydrogenation odor and flavor. After deodorization of this oil for 5 hours at 445° F., it was stable for only 1–2 days at 140° F. When stored at room temperature, the control reverted in two weeks, but the predeodorized sample was almost bland after a month.

Obviously, many modifications and variations of the invention as hereinbefore set forth may be made without departing from the spirit and scope thereof, and therefore only such limitations should be imposed as are indicated in the appended claim.

We claim:

A method for retarding the development of characteristic odors of hydrogenation in hydrogenated fatty materials, which comprises steam: deodorizing said fatty material prior to hydrogenation thereof; supplying a blanket of inert gas thereto subsequent to deodorization thereof, whereby non-oxidizing conditions exist; and thereafter, subjecting said material under such conditions to hydrogenation.

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