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HYDROGENATION OF FATTY OILS

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This invention relates to the treatment of organic compounds of the oleaginous type, and more particularly to the hydrogenation of vegetable oils containing glycerides or esters of saturated and unsaturated fatty acids.

It is common knowledge that the glycerides and other esters of the unsaturated fatty acids are capable of adding on hydrogen at their points of unsaturation or ethylene linkages whereby they become more or less saturated, depending upon the amount of hydrogen added. The amount of hydrogen which the fatty material absorbs is directly proportional to the drop in iodine value of the oil, the iodine value being the per cent by weight of iodine capable of combining with the remaining bonds of unsaturation under well-known standard conditions.

Hydrogenation of fatty bodies, such as vegetable oils, is usually accomplished at elevated temperatures and pressures, and is brought about by contacting the oils with hydrogen gas in the presence of catalytically active metals, mixtures of metals or readily reducible salts of the same. During such hydrogenation process, the unsaturated fatty acids present as glycerides or esters are changed to the corresponding and more completely saturated acids, which causes the consistency of the fatty oils to progressively approach that which is desired.

The most unsaturated fatty acids present in combination as esters in the fatty oils have in general, a "tendency" to become changed to acids of the next degree of saturation before those in turn commence to combine with hydrogen. To illustrate this, we may consider linseed oil as a good example, since it contains in its composition fatty acids of four different degrees of saturation as follows, the analysis shown being representative of an average sample:

	Percent
Linolenic acid with three bonds of unsaturation.....	33.1
Linolic acid with two bonds of unsaturation.....	48.4
Oleic acid with one bond of unsaturation.....	5.0
Saturated acids with no bonds of unsaturation.....	9.1

When an oil of the above composition is catalytically hydrogenated, employing the commonly used means and methods, the linolenic acid "tends" to absorb the hydrogen in preference to the other unsaturated acids, i. e., linolic and oleic, thereby being changed into the acid of the next degree of saturation, which is linolic. When most of the linolenic has changed, the linolic acid so formed and the linolic acid originally pres-

ent in the oil continue to add on hydrogen to become oleic acid which possesses only one unsaturation, which in turn proceeds to add on hydrogen to become fully saturated when most of the linolic acid has changed.

Although there is this "tendency" toward preferential hydrogenation of the more unsaturated bodies, in actual practice the hydrogenation does not occur in the above described ideal stepwise manner. This is believed to be due largely to certain characteristics of the catalyst and hydrogen gas that are commonly used, the effects of which modify and in many cases substantially eliminate this general tendency toward preferential hydrogenation. The desired characteristics of the hydrogen gas are disclosed in my joint application Serial No. 723,838, filed May 4, 1934. The desired type of catalyst forms an important part of the subject matter of the present invention as will be apparent from the description to follow.

One of the usual methods of preparing nickel hydrogenation catalysts is to precipitate a readily reducible salt of nickel, such as the carbonates, hydroxides, etc., from a solution of commercial nickel sulphate. When prepared by this method the catalyst contains not only the substances other than nickel derived from the nickel salt and the precipitant, e. g., commercial soda ash, but contains also varying amounts of absorbed sulphates, depending on the thoroughness with which the precipitate is washed. These sulphates are subsequently reduced to sulphides and it is common to find 1%—2% of sulphide of nickel in ordinary catalysts.

By a number of tests, which I have performed, I have found that the presence of nickel sulphide is undesirable since it accelerates the formation of iso-oleic acid. I have also found that commercial nickel sulphates commonly have present in them such substances as copper, cobalt, iron, sulphur, silicon, carbon, manganese, arsenic, antimony, bismuth, zinc, phosphorus, and traces of impurities not usually found by ordinary chemical analysis. I have found that some of these substances have deleterious effects upon the selective properties of the hydrogenation catalyst and thereby prevent the desired stepwise hydrogenation.

Under the usual hydrogenating conditions employing these ordinary catalysts there is practically always a certain amount of the more completely saturated acids formed before the highly unsaturated acids have been eliminated. However, from the standpoint of the final edible prod-

uct, it is of extreme importance that the conditions of hydrogenation be such that the oil absorbs the hydrogen in as nearly as possible the ideal preferential manner, as will be shown below.

5 In the manufacture of edible fats from hydrogenized oils, it is essential that the above mentioned more highly unsaturated acids, e. g., linolenic and linolic, be hydrogenated as completely as possible, since it is the glycerides of these acids
10 that are prone to oxidation and accelerate rancidity in the fat. For many years attempts have been made to obtain the ideal hydrogenation, supra, by hydrogenating at comparatively high temperatures, since the latter tend to suppress the
15 formation of fully saturated acids and thereby tend to prevent the product from becoming unduly hard before a sufficient degree of hydrogenation of the highly unsaturated acids is attained. Another attempt to produce this desirable result
20 consists in increasing the amount of catalyst used for effecting hydrogenation. Unfortunately, the use of these relatively high temperatures and excessive amounts of catalysts, although beneficial from the standpoint of reducing saturation
25 of the higher molecular weight acids has an extremely deleterious effect in other respects when the ordinary types of catalyst are employed, particularly in that the formation of hard isomerides of oleic acid is increased. The latter
30 render the product hard and brittle before it is sufficiently hydrogenated to insure its keeping qualities.

The exact composition of these isomerides of oleic acid, collectively called iso-oleic acid, is still
35 uncertain. It is believed, however, that their formation is caused by a shifting of the double bond in the carbon chain of the molecule and also by stereoisomerism of oleic acid to elaidic acid. Hydrogenated vegetable oils containing large
40 amounts of iso-oleic acid make poor shortenings, being brittle and non-plastic, with resultant inferior baking properties. As has been indicated, the amount of iso-oleic acid formed may be limited by employing reduced amounts of catalyst
45 and low hydrogenating temperatures, but these conditions have been shown to give mediocre selectivity of hydrogenation with the consequent formation of undesirably large amounts of the normal saturated acids of high melting point.
50 The shortening resulting from oils so processed cannot be finished satisfactorily to give the most desirable characteristics and also is difficult to digest. The use of high temperatures tends to suppress the formation of large amounts of the
55 saturated fatty substances. Thus, although there may have been some advantage in the use of higher temperatures, the adverse effect of such temperatures in increasing the formation of the isomerides predominates and the final result is a
60 product having very poor characteristics.

Until the present invention, it has been found necessary to effect a compromise with regard to the hydrogenation conditions used. The usual
65 result was the production of a fat containing considerable amounts of overly hard components or else one which was so incompletely saturated with hydrogen that it did not have good keeping qualities.

One object of my invention is to provide a
70 method and means for producing an improved edible product by the hydrogenation of vegetable oils.

Another object of my invention is to provide a
75 method for producing improved edible fats from vegetable oils by selectively hydrogenating the oil

in the presence of a hydrogenation catalyst of such a nature that the highly unsaturated fatty acids and glycerides originally present in the oil are sufficiently saturated to render the final hydrogenated product stable against rancidity and
5 oxidation, and the formation of undesirable solid saturated and unsaturated fatty acids and glycerides is so minimized that the final product will not be excessively hard.

A further object is to provide a selective hydrogenation catalyst free from sulfates and sulfides and other components that normally adversely
10 affect the selective characteristics of the catalyst.

In accordance with my invention I have discovered that the catalyst and particularly some of
15 its commonly associated substances play an important part in the desired selective or step-by-step hydrogenation of fatty oils and in suppressing the formation of iso-oleic acids, a fact which, as far as I know, has not heretofore been realized.
20 I have found that different catalysts, even though they are made of the same metal, for example nickel, have varying intrinsic or specific properties in controlling the course of the hydrogenation reaction, these properties being independent of
25 their activity or amount used. It is not yet fully understood why this occurs and I therefore do not wish to limit my invention to any theory of operation, although the various tests that I have performed indicate that the troublesome side reactions which occur during the hydrogenation process are to a great extent due to certain components normally present in the ordinary catalysts.
30 Some of these components are listed hereinabove.

In accordance with my invention, I have prepared my desired selective catalyst, which is also
35 capable of suppressing the formation of iso-oleic acids, from electrolytic nickel. This catalyst is substantially free from sulphates, sulphides, and the other undesirable substances mentioned
40 above, such as sulphur, arsenic, iron, etc. This catalyst is made by precipitating electrolytically nickel hydroxide by passing direct current through a cell using nickel as the anode and using an electrolyte consisting of the dilute solution of
45 an alkali salt of a weak acid such as acetic acid. Other known methods of forming a precipitate electrolytically may be used. It should be noted that I avoid the possible presence and introduction of substances which I have found destroy the
50 actual selective character of the catalyst as pointed out above. The resultant hydroxide is reduced with hydrogen free from all sulphur compounds or other contaminating substances. I preferably use for reduction of my hydroxide the
55 treated hydrogen disclosed in my joint application Serial No. 723,838 filed May 4, 1934. The reducing operation to make the nickel catalytically active is preferably carried out by heating the prepared nickel hydroxide to 300-500° C. in
60 a current of previously treated hydrogen. The activated nickel thus formed is now in suitable form for use as a hydrogenation catalyst for the selective hydrogenation of vegetable oils into the improved edible fats in accordance with this
65 invention.

This type of catalyst is substantially free from sulphides as well as the other deleterious substances which have previously been present. The
70 tendency of these substances previously present in all hydrogenating catalysts to inhibit selective hydrogenation and their tendency to cause the formation of iso-oleic acid was apparently not noticed. I have found, however, that even
75 minute amounts of these substances will sup-

press or destroy the desired selective hydrogenation effects and prevent the formation of the superior hydrogenated fats characteristic of this invention. Apparently previously the effect of these substances was not appreciated because they were present in such relatively small amounts.

One of the practical advantages of electrolytic nickel is that it is a cheap source of very pure nickel. Electrolytic nickel very rarely runs below 99.5% purity, including cobalt, the 0.5% impurity usually being iron and a little copper. Nickel refined by other processes may run as low as 97% pure and is rarely more than 99.5% pure. The method of electrolytic precipitation is a convenient and economic method of converting the nickel into a reducible compound, i. e., nickel hydroxide, but satisfactory results can be obtained also by dissolving the nickel in nitric acid and precipitating the nickel, as nickel carbonate, with soda ash. Another advantage of electrolytic precipitation is that it may be carried out with very dilute electrolytes so that subsequent washing of the precipitate free from soluble salts is comparatively easy.

This special electrolytic nickel catalyst, which is substantially free from all components adversely affecting hydrogenation, may or may not be given a carrier or support of inert material, such as diatomaceous earth or kieselguhr but in the event that such a support is used, extreme care must be taken that it does not contain harmful impurities which would modify the selective action of the catalyst and accelerate the formation of iso-oleic acid. Although this type of catalyst usually possesses good activity and effects a fairly rapid reaction, this property is only incidental to my selective catalyst. In fact, some of the most mildly active kinds of catalysts have been very effective in bringing about the preferred type of hydrogenation reaction, while some ordinary catalysts, although extremely active, possess very poor selective action and accelerate the formation of isomers of oleic acid. It is believed that this is due to the presence of some or all of the commonly associated components mentioned above, even though they are present in minute amounts.

I have also found that a highly desirable selective catalyst may be made by the following method, an example of which is given. 200 parts of acid washed infusorial earth are suspended in a solution of 100 parts C. P. nickel nitrate and 5,000 parts distilled water maintained at a temperature between 60 and 80° C. The solution of 450 parts anhydrous C. P. sodium carbonate and 5,000 parts distilled water are added very slowly with violent agitation. The precipitate is collected on a filter and thoroughly washed with distilled water until free from soluble salts after which it is dried in an oven at 110° C. The dried cake is then pulverized and heated to 400° C. in a furnace through which is passed a rapid stream of dry hydrogen preferably of the treated type disclosed in my joint application referred to above. The reduction with hydrogen is carried out until the evolution of water vapor ceases. The catalyst is used in proportion of about 1/10th of 1% by weight of nickel to the weight of the oil.

It is very important that my new catalysts be protected from harmful impurities during the hydrogenation reaction since even very minute traces of the so-called "poisons", which occur chiefly in the hydrogen gas, have a powerful

modifying action on their exceptional properties and tend to make the hydrogenation reaction non-selective and increases the formation of iso-oleic acid. A very effective method and means of purifying the hydrogen gas is described in my co-pending joint application, supra.

In order to illustrate the comparative ability of different types of nickel catalysts and the superiority of my new catalysts in controlling the selectivity of the hydrogenation of unsaturated fatty oils, and their effect on the formation of isomers of oleic acid, several examples are given below. The respective effects of these catalysts are compared by carrying out a carefully standardized reaction whereby 2 kilograms of cottonseed oil are hydrogenated at a temperature of 150° C. and a pressure of 20 pounds per square inch with an amount of catalyst under test equivalent to 0.10%, of the weight of oil, as nickel. In the four following examples I hydrogenated the oil to a predetermined iodine value, which as shown above, measures the degree of saturation. When all the tests are carried out to the same degree of saturation, e. g., 67.5 iodine value, an analysis of the hydrogenated samples will measure to what extent the reaction has been selective and how much iso-oleic acid has been formed. The cottonseed oil used in each of these tests had an original iodine value of 107.7 and was composed of glycerides of fatty acids in the following proportions: 50.9% linolic, 22.7% oleic, and 26.4% saturated acids. Such an oil is liquid at room temperatures, but when hydrogenated to an iodine value of 67.5, it will congeal on cooling to different degrees of hardness, depending largely upon the effectiveness of the catalyst in suppressing the formation of saturated acids and iso-oleic acids, the amount of solid acids formed being indicated by the congealing or solidification point.

Example 1

This first catalyst supported upon kieselguhr was prepared by methods commonly employed in the industry from nickel which had been recovered from old catalyst. The oil hydrogenated to 67.5 iodine value under the standard conditions indicated above had a congealing or solidification point of 31.2° C. and the following fatty acid composition: 14.1% linolic, 50.1% oleic, and 35.8% saturated acids.

Example 2

This second catalyst was prepared from commercial nickel sulphate by well-known methods by reducing nickel carbonate deposited on kieselguhr. This catalyst is representative of what I have referred to as an ordinary type of catalyst. The oil hydrogenated under the same standard conditions, using this catalyst, had a congealing point of 28.8° C. and was composed of fatty acids in the following proportions: 9.9% linolic, 58.4% oleic, and 31.7% saturated acids.

Example 3

This third catalyst was prepared, according to one of my preferred methods mentioned above, from the purest grade of nickel nitrate obtainable from the chemical supply houses. Accordingly the final catalyst was substantially free from sulfates, sulfides, and the other deleterious substances. The hydrogenated oil obtained when using this catalyst had a congealing point of 25.6° C. and the following fatty acid composition: 9.6% linolic, 59.1% oleic, and 31.3% saturated acids.

Example 4

The fourth catalyst was prepared, according to my preferred method, from electrolytically precipitated nickel hydroxide which was made from commercial electrolytic nickel. The hydrogenated oil, produced in the presence of this catalyst, had a congealing point of 24.9° C., the fatty acids being 7.7% linolic, 61.9% oleic, and 30.4% saturated acids.

Examples 1 and 2 illustrate the high congealing points obtained with non-selective catalysts prepared from ordinary materials. Shortenings prepared from such oils will be unduly hard at the degree of saturation required for good keeping properties and oils hydrogenated according to Example 1 in particular have a high melting point which renders them somewhat indigestible as food products, the high melting point being caused by the relatively large amount of saturated acids formed by this non-selective catalyst.

In Example 3 there is obtained, for the same degree of saturation as in Example 2, an oil whose congealing point is only 25.6° C. as against 28.8° C. in Example 2, although the fatty acid analyses are very similar. This is due to the fact that a high percentage of the oleic acid in Example 2 is in reality iso-oleic acid, as shown below. This iso-oleic acid increases the congealing point and hardness of the product. In Example 3, the amount of iso-oleic acid is relatively small, giving a very much softer fat for a fixed degree of saturation. It is evident, therefore, that oils which are hydrogenated with the type of catalyst used in Example 3 may be much more completely saturated with hydrogen to obtain the desirable consistency, thereby reducing to a substantially greater extent the amount of highly unsaturated acid, i. e., linolic acid, so that the hardened product has remarkable keeping properties. Also, it is not embrittled with iso-oleic acid.

Example 4 illustrates a still greater improvement in this respect in that the catalyst not only suppresses the formation of iso-oleic acid to the same or greater extent as in Example 3 but the amount of linolic acid is materially reduced without increasing the amount of saturated acids. In fact the operation is accompanied by a reduction in the amount of the saturated acids. Shortenings prepared from oils hydrogenated with catalysts according to my preferred Example 4 are very plastic and "workable" in addition to possessing high keeping qualities both as fat itself and in the subsequent food products in which the fat is incorporated, such as cakes, doughnuts, biscuits, potato chips, etc. The relatively low amounts of solid acids formed, with the consequent low melting point of the fat, also makes these fats more digestible.

In practice it is found unnecessary to actually determine the amount of iso-oleic acid in the final product, since the effect of this acid on the consistency of the product is readily appreciated by correlating the apparent fatty acid composition and the congealing point of the fat. For example, it has been indicated above that although the apparent fatty acid analyses of Examples 2 and 3 are very similar, the congealing or solidification points of the two fats vary by 3.2 degrees centigrade due to the presence in Example 2 of larger amounts of iso-oleic acids, which are included in the amount of oleic acid given under Example 2, namely 58.4%.

To substantiate the above given reasons for the superior characteristics of the edible fats prepared in accordance with my invention, I have

made a determination of the actual amounts of solid unsaturated or iso-oleic acids for two samples of fat hydrogenated in accordance with Examples 2 and 3, supra. I have found that there is 13.5% iso-oleic acid in the fat hydrogenated according to Example 2 and only 7.5% in that hydrogenated in accordance with Example 3. The product of Example 4 would contain even less iso-oleic acid as indicated by the lower congeal point than that of the product of Example 3. The catalyst used in Example 4 was therefore even more effective in the suppressing of the formation of iso-oleic substances. An analysis of the products of Examples 2 and 3 shows the following:

	Linolic acid	Oleic acid	Iso-oleic acid	Solid acids including iso-oleic acids
	Percent	Percent	Percent	Percent
Example 2.....	9.9	44.9	13.5	45.2
Example 3.....	9.6	51.6	7.5	38.8

As shown, there is 6.4% more solid acids in Example 2 than in Example 3 which, as above indicated, causes Example 2 to possess a congealing point 3.2 degrees higher than Example 3. Of course, many commercial hydrogenations using non-selective catalysts give rise to much larger amounts of iso-oleic acid, and it is quite common to find as high as 20-30% of the latter in the hydrogenated fat.

In order to illustrate in a possibly more practical manner the advantages of utilizing my new catalysts in hydrogenated shortening manufacture, I have prepared four shortenings from cottonseed oil separately hydrogenated with the aid of the catalysts described in Examples 1-4, supra. The oil was hydrogenated under the same conditions as used in Examples 1-4 above, except that in the present tests the extent of hydrogenation was controlled by the consistency of the congealed fat instead of by its iodine value, that is, the oil was saturated with hydrogen in each case until hardened products attained the same general consistency or hardness when cooled to normal temperatures, the congealing or solidification point of each fat thus produced being about 29° C.

When these four fats were chilled and textured by well known methods they produced shortenings of almost identical degrees of hardness when compared at room temperature. Determination of the iodine values, which as explained above, measures the degree of saturation, gave 69.0 and 66.9 for the fats hardened with the catalysts in Examples 1 and 2 respectively. These catalysts, as stated hereinbefore, are representative of ordinary catalysts employed in the industry. The fats hydrogenated with the new catalysts, of which Examples 3 and 4 are representative, had iodine values of 59.4 and 57.8.

It is evident from the tests described just above that with the new catalysts a substantially more complete degree of saturation can be obtained than with common catalysts, the improvement being equivalent to 7-11 iodine values, and the product is still of the desirable soft consistency. It is also evident from the tests above that the keeping quality of the shortenings prepared using the new catalysts is greatly enhanced and the shortenings themselves possess improved plasticity over a comparatively wide range of temperature, due to the suppression of solid isomers of oleic acid or iso-oleic acid.

The expressions "unsaturated fatty acids" and "saturated fatty acids", used herein, are intended to include these acids as comprised in fats and oils.

5 It is to be understood that my invention is not limited to the specific materials and methods described hereinbefore by way of example but is subject to various modifications, all of which come within the purview of the following claims.

10 What I claim as new is:

1. A hydrogenation catalyst capable of effecting the selective step-wise hydrogenation of vegetable oils herein disclosed and suppressing the formation of iso-oleic acid, which catalyst
15 consists of electrolytic nickel substantially free from sulphides, said nickel being prepared from electrolytically precipitated nickel hydroxide which is subsequently converted into catalytically active metallic nickel by heat and contact
20 with hydrogen gas.

2. A hydrogenation catalyst having the property, in a vegetable oil hydrogenating operation, of selectively hydrogenating the more highly unsaturated fatty acids before the less saturated
25 fatty acids are hydrogenated into solid saturated acids, and the property of suppressing the formation of solid unsaturated fatty acids such as iso-oleic acids whereby the hydrogenated vegetable oil product thus produced possesses improved plasticity and keeping properties, said
30 catalyst being made of metallic nickel substantially free from all impurities, said nickel being prepared from electrolytically precipitated nickel hydroxide which is subsequently converted into
35 said metallic nickel by heating the nickel hydroxide in contact with pure hydrogen gas.

3. A hydrogenation catalyst having the property, in a vegetable oil hydrogenating operation, of selectively hydrogenating the more highly unsaturated fatty acids before the less saturated
40 fatty acids are hydrogenated into solid saturated acids, and the property of suppressing the formation of solid unsaturated fatty acids such as iso-oleic acids whereby the hydrogenated vegetable oil product thus produced possesses improved plasticity and keeping properties, said catalyst being made of metallic nickel
45 substantially free from all impurities, said nickel being prepared from electrolytically precipitated nickel hydroxide which is subsequently converted into said metallic nickel by heating the nickel hydroxide to approximately 300° C.-500° C. in
50 contact with pure hydrogen gas.

4. A hydrogenation catalyst having the property, in a vegetable oil hydrogenating operation, of selectively hydrogenating the more highly unsaturated fatty acids before the less saturated
55 fatty acids are hydrogenated into solid saturated acids, and the property of suppressing the formation of solid unsaturated fatty acids such as iso-oleic acids whereby the hydrogenated vegetable oil product thus produced possesses im-

proved plasticity and keeping properties, said catalyst being made of metallic nickel substantially free from all impurities, said nickel being prepared by passing direct current through a cell
5 using nickel as the anode and using as the electrolyte a dilute solution of an alkali salt of a weak acid to electrolytically precipitate nickel hydroxide in said cell and heating said nickel hydroxide to approximately 300° C.-500° C. in the presence of pure hydrogen to produce pure
10 metallic nickel.

5. A hydrogenation catalyst having the property, in a vegetable oil hydrogenating operation, of selectively hydrogenating the more highly unsaturated fatty acids before the less saturated
15 fatty acids are hydrogenated into solid saturated acids, and the property of suppressing the formation of solid unsaturated fatty acids such as iso-oleic acids, whereby the hydrogenated vegetable oil product thus produced possesses improved plasticity and keeping properties, said
20 catalyst being made of metallic nickel substantially free from all impurities, said nickel being prepared by passing direct current through a cell using nickel as the anode and using as the electrolyte a dilute solution of sodium acetate to electrolytically precipitate nickel hydroxide in
25 said cell and heating said nickel hydroxide to approximately 300° C.-500° C. in the presence of pure hydrogen to produce pure metallic nickel.
30

6. In a catalytic oil hydrogenation process for producing edible fats having improved plasticity and keeping properties, the step of contacting the oil with a catalyst prepared from electrolytically precipitated nickel that is substantially
35 free from all impurities, in the presence of hydrogen and at a hydrogenating temperature of not substantially more than 150° C., whereby the formation of solid unsaturated fatty acids such as iso-oleic acids is suppressed and there
40 is obtained a selective hydrogenation of the more highly unsaturated fatty acids before the less saturated fatty acids are hydrogenated.

7. In a catalytic oil hydrogenation process for producing edible fats having improved plasticity and keeping properties, the step of contacting the oil with an electrolytic nickel catalyst that is substantially free from all impurities, in the presence of hydrogen and at a hydrogenating
45 temperature of not substantially more than 150° C., said electrolytic nickel catalyst being prepared from electrolytically precipitated nickel hydroxide that is subsequently converted into catalytically active metallic nickel by heat and contact with pure hydrogen gas, whereby the
50 formation of solid unsaturated fatty acids such as iso-oleic acids is suppressed and there is obtained a selective hydrogenation of the more highly unsaturated fatty acids before the less saturated fatty acids are hydrogenated.
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