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PROCESS FOR THE PURIFICATION OF DARK-COLORED RAW FATTY ACID AMIDES

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This invention relates to the purification of amides of saturated and unsaturated fatty acids. It more particularly relates to the purification of such compounds with the aid of oxygen yielding agents.

Amides of saturated and unsaturated fatty acids are today in demand to an increasing extent for use as washing and cleaning agents and for other purposes, such as water-proofing agents and wax substitutes.

The manufacture of these amides has been known for a long time and is accomplished, for example, by the method of A. W. V. Hofmann (Ber. 15 [1882], 977) by dry distillation and pyrolysis of the corresponding ammonium salts. In the following decades, this process has been modified in various ways, for example, by using dehydration agents, catalysts or variations in the external conditions, such as pressure and temperature, and these modifications have been published in the literature.

A number of published articles relate to the condensation of the derivatives of ammonium, such as urea, with fatty acids using known reaction procedures which form the corresponding fatty acid amides.

However, the reaction using the classic dehydration method of A. W. V. Hofmann

$$R.COOH+NH_3 \rightleftarrows R.COONH_4 \rightleftarrows R.CONH_2+H_2O$$

which is very simple from the point of view of economy and apparatus requirements, produces a product which is for most purposes not satisfactory.

The color-producing impurities formed by this reaction and the nitriles, isonitriles and other side products which are present in the liquid or solid amides are considerably detrimental to the odor and appearance of the amides and limit their utility.

It is an object of this invention to produce purified amides of saturated and unsaturated fatty acids free from compounds which impart detrimental odor and color characteristics to such amides.

It is a further object of this invention to provide a method for purifying amides of saturated and unsaturated fatty acids with oxygen yielding agents.

These and other objects of my invention will become apparent as the description thereof proceeds.

I have now found that the above objects may be attained and the high-molecular-weight fatty acid amides can be obtained in a satisfactory and marketable form if they are subjected to a treatment with oxygen-yielding agents, especially with hydrogen peroxide, at relatively high temperatures.

It was surprising that an oxidative bleaching and deodorizing effect could be achieved with hydrogen peroxide under the prevailing conditions, because it could not have been expected that high-molecular-weight wax-like fatty acid amides could be admixed with hydrogen peroxide with sufficient dispersion to obtain an effective bleaching effect. I found, however, that by intensive stirring, particularly at temperatures above 100° C., which is above the melting point of the amides or their mixtures, an excellent purification effect could be obtained. In this connection it was observed that the reaction proceeds smoothly and rapidly, especially if small amounts of high-molecular-weight, free fatty acids are still present in the reaction mixture. Under these conditions very pure, slightly odorous fatty acid amides are obtained.

It is most advantageous to operate at temperatures which lie about 20° C. above the melting point of the corresponding fatty acid amides or their mixtures. It is also an advantage of this process that the nitriles or isonitriles which are always present as impurities are completely removed, and probably transformed into the corresponding amides, which is the essential reason for the improvement of the odor.

All types of raw fatty acid amides, such as stearic acid amide, palmitic acid amide, myristic acid amide, lauric acid amide, capric acid amide, and/or caprylic acid amide or also mixtures of carboxylic acid amides as are obtained by cleavage and subsequent amidation of fatty acid mixtures from hardened or unhardened fats or oils, such as coconut fatty acids, soybean fatty acids, sperm oil fatty acids, tallow fatty acids, or hardened tallow fatty acids, linseed oil fatty acids, castor oil fatty acids, and palm oil fatty acids, may be treated according to the present process.

Moreover, the process can be used for the purification of those amides, the nitrogen atom of which is substituted with lower alkyl and/or hydroxy alkyl groups, such as fatty acid monoethanol amides.

In accordance with the invention, primarily hydrogen peroxide is used as the oxygen-yielding agent. The treatment with oxygen may, however, also be performed with peracetic acid or performic acid. If the presence of salts in the end product is not detrimental, persulfates, percarbonates and peroxides of inorganic metal compounds in aqueous solution or suspension may be used.

The quantity of oxygen-yielding agent which is employed should not amount to more than 1 to 5%, based on the amount of amide used. Preferably, a 20 to 60% by volume solution of hydrogen peroxide is used.

The process is best performed in an apparatus in which the particular oxidizing agent is not catalytically decomposed by metallic components of the apparatus, for example, in vessels provided with a stirrer and made of V_2A-steel or aluminum or an enamel-coated apparatus.

The following examples are set forth to enable persons skilled in the art to better understand and practice the invention and are not intended to be limitative.

Example I

10 parts by weight of a raw, dark-colored coconut fatty acid amide mixture (melting point about 95 to 100° C.) consisting of

8% caprylic acid amide
7% capric acid amide
48% lauric acid amide
16% myristic acid amide
9 to 10% palmitic acid amide
3% stearic acid amide
6% oleic acid amide
3 to 2% linoleic acid amide

were heated at 115 to 120° C. in an aluminum vessel, accompanied by stirring. 0.55 part by weight of a 40% by volume hydrogen peroxide solution were gradually added to this mixture in the course of 15 minutes, accompanied by intensive stirring. Oxygen was rapidly evolved and at the same time the amide mixture considerably lightened in color. The mixture was then stirred for an additional 15 minutes. Thereafter, the mass was poured into molds. From the dark brown colored raw amide, a product which was light yellow in color and free from unpleasant odors was obtained.

Example II

In accordance with the procedure of Example I, a raw, dark-colored amide mixture obtained from hardened tallow fatty acid having a melting point of about 100° C. and composed of 1% myristic acid amide, 34% palmitic acid amide and 65% stearic acid amide was treated instead of the coconut fatty acid amide. A light colored product was also obtained which was free from nitriles and had a very faint characteristic odor.

While I have set forth certain specific embodiments and preferred modes of practice of my invention, it will be understood that they are not intended to be limitative and that various changes and modifications may be made in the invention without departing from the spirit of the disclosure or the scope of the appended claims.

I claim:

1. A process for the purification of a raw, dark colored fatty acid amide material, which comprises treating said fatty acid amide material in the molten state with an oxygen yielding agent selected from the group consisting of hydrogen peroxide and adducts thereof, perborates, percarbonates, performic acid, and peracetic acid at a temperature between about 100° C. and a temperature about 20° C. above the melting point of said material, accompanied by stirring of the molten material.

2. A process for the purification of a raw, dark colored fatty acid amide material, said material comprising a mixture of amides of different fatty acids, which comprises treating said fatty acid amide material in the molten state with an oxygen yielding agent selected from the group consisting of hydrogen peroxide and adducts thereof, perborates, percarbonates, performic acid, and peracetic acid at a temperature between about 100° C. and a temperature about 20° C. above the melting point of said material, accompanied by stirring of the molten material.

3. The process of claim 2 wherein the material treated is a coconut fatty acid amide mixture.

4. The process of claim 2 wherein the material treated is an amide mixture obtained from hardened tallow fatty acids.

5. A process for the purification of a raw, dark colored fatty acid amide material, which comprises treating said fatty acid amide material in the molten state with 1 to 5% of an oxygen yielding agent selected from the group consisting of hydrogen peroxide and adducts thereof, perborates, percarbonates, performic acid, and peracetic acid based on the weight of said amide at a temperature between about 100° C. and a temperature about 20° C. above the melting point of said material, accompanied by stirring of the molten material.

6. A process for the purification of a raw, dark colored fatty acid amide material, which comprises treating said fatty acid amide material in the molten state with an oxygen yielding agent selected from the group consisting of hydrogen peroxide and adducts thereof, perborates, percarbonates, performic acid, and peracetic acid at temperatures about 20° C. above the melting point of said material, accompanied by stirring of the molten material.

7. A process for the purification of a raw, dark colored fatty acid amide material, which comprises treating said fatty acid amide material in the molten state with a 20 to 60% solution by volume of hydrogen peroxide at a temperature between about 100° C. and a temperature about 20° C. above the melting point of said material, accompanied by stirring of the molten material.

8. The process of claim 1 wherein the oxygen yielding agent is peracetic acid.

9. The process of claim 1 wherein the oxygen yielding agent is performic acid.

10. A process for the purification of a raw, dark colored fatty acid amide material containing small amounts of free fatty acids, which comprises treating said fatty acid amide material in the molten state with an oxygen yielding agent selected from the group consisting of hydrogen peroxide and adducts thereof, perborates, percarbonates, performic acid, and peracetic acid at a temperature between about 100° C. and a temperature about 20° C. above the melting point of said material, accompanied by stirring of the molten material.

11. A process for the purification of a raw, dark colored fatty acid amide material, comprising a mixture of amides of different fatty acids and containing a small amount of free fatty acids, which comprises treating said fatty acid amide material in the molten state with a 20 to 60% by volume solution of hydrogen peroxide at a temperature about 20° C. above the melting point of said material, accompanied by stirring of the molten material.

References Cited in the file of this patent

UNITED STATES PATENTS

2,340,112	Davis et al.	Jan. 25, 1944
2,745,853	Hamilton et al.	May 15, 1956
2,839,546	Davis	June 17, 1958

FOREIGN PATENTS

466,853	Great Britain	June 4, 1937
1,016,700	Germany	Oct. 3, 1957