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A METHOD FOR IMPROVING COLOR AND THE COLOR AND ODOR STABILITY OF FATTY AMIDES

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ABSTRACT OF THE DISCLOSURE

Improvement in color and color and odor stability of fatty amides is accomplished by admixing an alkaline-type borohydride with the fatty amide.

This invention broadly relates to fatty amides and more particularly to a method for improving the color and the color and odor stability of fatty amides, and to the composition produced therefrom.

In the synthesis of fatty amides, especially from unsaturated acids, the product obtained has an amber color. Even worse, this undesirable amber color is unstable and during storage or use, it darkens. The odor stability of such fatty amides is also not very good and eventually, a rank odor is given off. These characteristics frequently prevent use of fatty amides, unless drastically purified, in compositions used by consumers or for the manufacture of derivatives which are used for consumer products.

The curse of an unattractively colored product, or a product which darkens or acquires an unpleasant odor with age, has plagued the industry for a long time. Much effort has been expended, but only moderate success has been attained. Since the amides are becoming more useful each day, as antistatic agents, antiblock agents, and as slip agents in plastics, for stabilizers in detergents, and as components in water-resistant coatings, the trade is desperate to find some economical solution to synthesize stable fatty amides.

An object of this invention is to provide a novel purification process for fatty amides.

Another object is to provide an improved purification process for fatty amides which is economical.

Still another object is to provide a purification process for fatty amides which is relatively easy to perform, even by unskilled technicians.

A further object is to provide a purification process which improves the original color of fatty amides.

A still further object is to provide a purification process which imparts color and odor stability to fatty amides.

Another object is to provide a purification process which leaves no undesirable residue in the finished amide so that the amide may be either stored or further processed without subsequent deleterious effect.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

The invention accordingly comprises the several steps and the relation of one or more of such steps with respect to each of the others, and the composition possessing the features, properties, and the relation of constituents, which are exemplified in the following detailed disclosure, and the scope of the invention will be indicated in the claims.

It has now been found that amides may be greatly improved in amide content and reduced color by subjecting the crude amide to washing with a hydrocarbon to remove free fatty acids, nitriles, colored bodies, etc.,

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followed by the addition of a small amount of an alkali metal borohydride or other borohydride derivatives to the resultant wet slurry. The slurry is then dried by stripping solvent therefrom at an elevated temperature.

Upon cooling, the cleaned, borohydride-containing amide is flaked and is ready for use as a component in compositions or as a reactant in further synthesis. The borohydride has no deleterious effect upon such further use, and in fact, imparts a unique color and odor stability to the amide.

The borohydride that may be used may comprise sodium borohydride, potassium borohydride and quaternary ammonium borohydride. The hydrocarbon that is used to wash the amide is one having from 3 to 8 carbon atoms, and preferably, 5 to 8 carbon atoms so that the washing need not be done in a pressurized system to prevent volatilization of the hydrocarbon. Usually the amount of hydrocarbon used is from two to five times that of the amide and the washing is performed at room temperature. However, additional washes and even temperatures above room temperature may be used where the amide is not too soluble in the hydrocarbon, such as with saturated amides.

The invention is more thoroughly described by reference to the following examples:

Example I

A seven foot glass column, 1¼ inches in diameter, is filled with Armid O flakes (oleylamide). Skelly-solve B (a commercial mixture of hydrocarbons, "B" referring to a hexane type with a boiling range of 146° to 156° F.) is then added flowing either up or down through the bed. The amide is allowed to soak in the Skelly-solve B for approximately one hour before additional Skelly-solve B is passed through the bed. After the required weight of Skelly-solve B has been used, the column is drained, and sodium borohydride added (0.05%, on the basis of weight of amide charged). The flakes are then transferred to a distilling flask, and the solvent removed at reduced pressure. Yield, color, and percent free fatty acids are then measured.

It might be noted that when solvent is added to the dry Armid O from the top, the resulting bed is more easily compacted and it takes longer to pass the Skelly-solve B through. Because of this, the runs are preferably made by either filling the column with Armid O flakes and letting the solvent flow upward until the liquid level is above the Armid O level, or adding the required amount of solvent to the column and then adding the Armid O flakes until the solvent is just barely covering the flakes. The data in Table I illustrates the results that are obtainable:

TABLE I.—SOLVENT WASHING WITH TWO PARTS SKELLY-SOLVE B TO ONE PART ARMID O IN A 7 FOOT X 1¼ INCH GLASS COLUMN

Percent NaBH ₄	Initial Gardner Color	Final Gardner Color	Percent F.F.A. ¹	Remarks
0.05	4	1	-----	Filtered and held at 100° C. for 64 hours. Product darkened to Gardner 3-4. ²
0.05	4	1	-----	
0.05	4	1-2	0.11	Held at 100° C. for 48 hours. No change in color.
0.05	4	1-2	0.06	

¹ Free fatty acid as oleic acid.

² This darkening occurred because the borohydride was removed by the filtration.

Yields are generally in the range of 88.5-94%.

General comments.—There is no significant difference in the various hydrocarbons used as solvents. Results are always better when freshly prepared, lighter-colored flakes are used. When regular Armid O flakes are allowed to darken in storage, the color is much more difficult to remove.

Example II

200 gal. heptane is added to a 500 gal. Dopp kettle. Temperature of the heptane is 56° F. This kettle has twelve counter-rotating arms for mixing. However, no agitation is used during the washing cycle because the soft wet flakes would be destroyed and the arms help to stabilize the bed. 1500 lbs. of Armid O flakes (2.85% F.F.A. and Gardner 4 color) is then added and the bed is leveled. At this stage about 3–4 inches of heptane is above the level of the Armid O. The Armid O is allowed to soak for one hour. The heptane is then drained from the bottom of the kettle and fresh heptane is simultaneously added to the top, maintaining a 4–5 inch level of heptane above the amide. After a total of 400 gal. has been added, the heptane is drained as completely as possible. ¾ lb. sodium borohydride is then added. The batch is melted and the remaining heptane is distilled off. After all the heptane has been removed, the batch is drained, cooled, and packed in 50 lb. fiber drums. Yield is 1409 lbs. with a Gardner color of 1 and free fatty acid content of 0.1% as oleic acid.

Example III

Armid O (not solvent washed) having an initial Gardner color of 4 is treated with 0.05% sodium borohydride and subjected to a heat stability test. The test consists of holding Armid O in a capped 2 oz. bottle at an elevated temperature for at least 18 hours. A sample is considered to have passed the test if the final color is not darker than Gardner 10. A control is also run concurrently without the borohydride. It will be found that the final color of the control will be about Gardner 11–12, whereas that containing borohydride will be about Gardner 7–8 or even better.

Example IV

In this example, the batch size comprises a 2000 lbs. lot of Armid O. The amide is solvent-washed, treated with 0.05% sodium borohydride, stripped of solvent, and drummed. The drums are then shipped to a distant loca-

Table II illustrates the type of results that are obtainable when the amide of the above example is subjected to heat stability test such as described in Example III:

TABLE II.—HEAT STABILITY TESTS

5	Percent NaBH ₄	Initial Color	Gardner Final	Temp., °C.	Hours	Remarks
	0.05	2+	2-3	95-100	66	Solvent washed, no air.
	0.05	2+	2-3	95-100	66	Do.
	None	2+	8-9	95-100	66	Do.
	0.05	2-3	6-7	110	24	Solvent washed, open to air.
10	None	5-6	11	110	24	No wash, open to air. Vacuum distilled material.
	None	10-11	14	110	24	No wash.
	0.05	1	3	110	24	Solvent washed.
	None	5	9	110	24	No solvent wash.

Example V

A 500 gallon Dopp kettle is charged with 265 gallons of heptane and cooled to 65–75° F. Then 2000–2200 lbs. of Armid O flakes are added and the bed is leveled so that a few inches of solvent will be above the amide. The batch is soaked for 45 min., and then the solvent is drained from the bottom at a rate of about 3 gallons per minute. Simultaneously, fresh heptane is added at 65–75° F. to the top of the batch, maintaining a 2–3 inch level of heptane above the amide. After a total of 600 gallons of heptane have been used, the flow of heptane is stopped to the top of the batch, but draining is continued until as much solvent as possible has been drained. Atmospheric steam is then supplied to the jacket of the Dopp kettle until the amide has melted. One lb. of sodium borohydride is then added and the batch transferred to another vessel for stripping of solvent.

All slop heptane is preferably recovered and reused to effect further economies in the purification process just described.

Examples VI and VII

Armid E (Erucic amide) and Armid HT (saturated amide derived from hydrogenated tallow acids) can be processed similarly as in Example I with the following variations in procedure:

(a) The ratio of solvent to amide is preferably 3 parts to 1.

(b) After an initial soak of one hour in the solvent, the first solvent is drained and fresh solvent added at the top simultaneously until all the initial solvent has been replaced by fresh solvent. The batch is then allowed to soak for another hour. The washing is then continued as with the Armid O.

Table III summarizes the data:

TABLE III.—SOLVENT WASHING OF AMIDES¹ OTHER THAN ARMID O

Solvent	Ratio of Solvent to Amide	Percent NaBH ₄	Initial Gardner Color	Final Gardner Color	Percent Yield	Percent F.F.A.
Skelly-solve B	2-1	-----	5	3	96	2.1
Do	2-1	-----	5	1-2	89.5	0.7
Do	2-1	-----	5	1	97.3	1.5
Do	3-1	-----	9	5	-----	Nil
Do	3.2-1	0.2	15	7	85.5	Nil
Skelly solve B+5% Acetone	3.2-1	-----	15	6-7	86	Nil
Skelly-solve B+10% Acetone	3.8-1	0.2	16	9	76	Nil
Skelly-solve B	2.7-1	0.2	10-11	5-6	87	Nil
Do	4-1	0.2	10	7	84.5	Nil
Do	3.8-1	0.2	9	5	86	0.34
Skelly-solve B+10% Acetone	3.8-1	0.2	9	3	83	Nil
Skelly-solve B	4-1	0.2	6	3-4	86	Nil

¹The first three lines indicate data run on amide prepared from hydrogenated tallow acids while the remaining data is on amides prepared from erucic acid.

tion to simulate trade conditions. They are then opened and the amide re-melted, filtered, and flaked. The final product has the following analysis:

I.V.	84.2
F.F.A., percent	nil
M.P., ° C.	74
Odor	Bland
Color	Gardner 1

The preferred wash liquid is a hydrocarbon having from three to eight carbon atoms, the lower ones (3 to 4 C's) being useful only where a pressure system is utilized to combat volatility. Usually, hydrocarbons such as pentane, hexane, heptane, or octane and isomers thereof and mixtures thereof, such as Skelly-solve, are useful. The usual range of wash liquid to amide being washed is 2 to 1 to 75 5 to 1.

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The washing is accomplished usually at room temperature, although with the higher amides, temperatures above room temperature to facilitate the washing may be used.

The amides to which the technique of this invention has particular application are the fatty amides such as the C₈ to C₂₄ saturated and unsaturated amides, specifically, octyl amide, nonyl amide, decyl amide, undecyl amide, dodecyl amide, tridecyl amide, tetradecyl amide, pentadecyl amide, hexadecyl amide, heptadecyl amide, octadecyl amide, nonadecyl amide, eicosyl amide, hencicosyl amide, docosyl amide, octenyl amide, nonenyl amide, decenyl amide, undecenyl amide, dodecenyl amide, tridecenyl amide, tetradecenyl amide, pentadecenyl amide, hexadecenyl amide, heptadecenyl amide, octadecenyl amide, nonadecenyl amide, eicosenyl amide, hencicosenyl amide, docososenyl amide, octadecadienyl, amide, octadecatrienyl amide, eicosatetraenyl amide, and docosapentenyl amide, or mixtures of amides such as would be derived if the natural fats and oils were amidated and particularly the amides derived by amidating tallow, coconut oil and soybean oil.

The borohydrides which are operable in the technique of this invention are sodium borohydride, potassium borohydride and quaternary ammonium borohydride. The concentration ranges vary between 0.05 to 0.2% by weight of the amide.

It will thus be seen that the objects set forth above, among those made apparent from the preceding description, are efficiently attained and, since certain changes may be made in carrying out the above method and in the composition set forth without departing from the scope of the invention, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

It is also to be understood that the following claims are intended to cover all of the generic and specific features of the invention herein described, and all statements of

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the scope of the invention, which, as a matter of language, might be said to fall therebetween.

Now that the invention has been described, what is claimed as new and desired to be secured by Letters Patent is:

What is claimed is:

1. A process for improving the color and odor stability of fatty amides comprising admixing 0.05 to 0.2% by weight of an alkaline-type borohydride with said fatty amides.

2. The process of claim 1 wherein said borohydride comprises sodium borohydride.

3. A process for improving the color and the color and odor stability of fatty amides comprising washing said fatty amides with hydrocarbons, and then subsequently admixing 0.05 to 0.2% by weight of an alkaline-type borohydride with said fatty amides.

4. The process of claim 3 wherein said borohydride comprises sodium borohydride.

5. A color and odor stable fatty amide composition comprising at least one fatty amide admixed with 0.05 to 0.2% by weight of an alkaline-type borohydride.

6. The composition of claim 5 wherein said borohydride comprises sodium borohydride.

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