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3,836,554

PROCESS FOR THE LIGHTENING AND COLOR STABILIZATION OF FATTY-ACID FORERUNNINGS

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No Drawing. Filed July 28, 1971, Ser. No. 167,014

Claims priority, application Germany, Aug. 5, 1970,

P 20 38 804.9

Int. Cl. C09f 5/10

U.S. Cl. 260—419

7 Claims

ABSTRACT OF THE DISCLOSURE

A process for lightening and color-stabilization of fatty-acid forerunnings which comprises heating the fatty acids with technical-grade silicon at a temperature of from about 180° to 250° C. and distilling off the treated fatty-acid forerunnings under vacuum.

This invention relates to a unique process for the lightening and color stabilization of first fraction fatty acids or fatty-acid forerunnings wherein the fatty acids are heated with technical-grade silicon.

According to U.S. Pat. 3,531,506, a substantial lightening of crude fatty-acid forerunnings of 8–12 carbon atoms is achieved, and the darkening thereof is prevented, by heating the crude fatty acids with 0.2–2%, preferably 0.5–1% by weight, of an alkyl ester of silicic acid or polysilicic acid to temperatures of 180–250° C., preferably 200–240° C., and subsequently distilling off the fatty acids.

It has now been found that lightening and color stability are also attained by conducting the heating step in the presence of technical-grade silicon instead of using the alkyl esters of silicic acid or polysilicic acid. The color-stabilizing influence of technical silicon on first-fraction fatty acids or fatty-acid forerunnings has not been described heretofore. The term "fatty-acid forerunnings" is more fully described in U.S. Pat. 3,531,506, the disclosure of which is incorporated by reference in this application.

Therefore, this invention is directed to a process for the lightening and color stabilization of fatty-acid forerunnings having a chain length of C₈–C₁₂ which is characterized in that technical-grade silicon is employed in place of alkyl esters of silicic acid or polysilicic acid, respectively; the fatty acids are heated with about 0.1 to about 0.6%, preferably about 0.2 to about 0.3% by weight, of technical-grade silicon for a period of about 0.5 to about 4 hours, preferably about 1 to about 3 hours, under normal pressure to temperatures of about 180° C. to about 250° C., preferably about 200° C. to about 240° C.; and the acids are then distilled under vacuum. It will be understood that heating may be effected at higher pressures, i.e. about atmospheric, e.g. about 5 atmospheres gauge, but atmospheric pressure is generally preferred.

When operating in accordance with the present invention, the impurities in the fatty acids remain, after the treatment, in the distillation residue bound to the silicon. The process has the advantage that even after the treatment with technical-grade silicon and the subsequent distillation, the present distillate does not have any larger amounts of unsaponifiable compounds than the distillate of the untreated fatty acids. In particular, the distillation

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residue of the treated fatty acids does not contain any newly formed condensation products, as is the case in the conventional processes for preventing discoloration utilizing boric acid or boric acid esters. It will be appreciated that the boron compounds have the effect of Lewis catalysts and promote the formation of unsaponifiable, nondistillable condensation products. When conducting the process of the present invention, hard-to-distill silicon compounds are found in the distillation residue after the distillation process is terminated.

The color stability of the fatty-acid forerunnings treated in accordance with this invention and for comparison purposes, the color stability of the untreated fatty-acid forerunnings were determined by heating samples in a capped glass tube having a diameter of 14 millimeters and a filling level of 115 millimeters to 200° C. for a period of 6 hours. This heating test was conducted in a drying oven equipped with a thermostat which was not opened during the entire duration of the test. The color intensity was measured in iodine color values wherein the iodine color value indicates how many milligrams of free iodine is contained in 100 milliliters of aqueous iodine-potassium iodide solution at the same depth of color when measured at a layer thickness of 25 millimeters. The iodine color value (ICV) and the hazen color value (HCV) were measured in a "Lovibond 1000" comparator and its accessories by Tintometer. It will be appreciated that the process of the present invention can be conducted discontinuously as well as continuously.

EXAMPLE 1

(a) The fatty-acid forerunnings of coconut-palm nut refinery fatty acid employed, i.e. the untreated fatty acid fraction, exhibited the following composition in accordance with gas chromatographic analysis (numerical data in percent by weight): C₈-acid: 0.5; C₈-acid: 54.6; C₁₀-acid: 42.8; C₁₂-acid: 1.8; and C₁₄-acid: 0.3. The acid fraction also contained about 2% of water. These forerunnings were dewatered under agitation and mixed, at a temperature of about 220° C., with 0.2% by weight of technical-grade silicon. Then the reaction mixture was heated to about 240° C., and this temperature was maintained for about 3 hours. Thereafter, the temperature was lowered to about 90° C. by cooling, and the thus-treated fatty-acid fraction was distilled under a vacuum of 1–2 torr [mm. Hg] over a small distillation column; within about 2 hours three fractions were withdrawn.

As fraction I, 7.0% by weight was obtained; as fraction II, 82.6% by weight, and as fraction III, 6.3% by weight, based on the anhydrous fatty-acid forerunnings employed. The amount of residue was 4.1% by weight.

(b) For comparison purposes, the untreated first-fraction fatty acid was distilled under the same conditions. Additional data is compiled in the table following Example 2.

EXAMPLE 2

(a) Fatty-acid forerunnings from the pressure cleaving, i.e. cracking, of coconut oil and palm nut oil were dewatered at 450 torr and mixed at a temperature of 220° C., with 0.2% by weight of technical-grade silicon. During a period of 3 hours a temperature of 240° C. was then maintained. Subsequently, the temperature was reduced by cooling to 90° C. and the thus-treated fatty-acid forerunnings were distilled under a vacuum of 1–2

torr over a small distillation column; within about 2 hours three fractions were taken off.

As fraction I, 7.5% was obtained; as fraction II, 82.2% by weight, and as fraction III, 6.5% by weight. The residue was 3.8% by weight.

(b) For comparison purposes the untreated fatty-acid forerunnings were distilled under the same conditions.

The results of these two examples are compiled in the following table:

TABLE

Example	Fraction I			Fraction II			Fraction III			Residue	
	Percent by wt.	HCV	ICV after 6 hrs. at 200° C.	Percent by wt.	HCV	ICV after 6 hrs. at 200° C.	Percent by wt.	HCV	ICV after 6 hrs. at 200° C.	Percent by wt.	Unsapo-nifiable com-pounds, percent
1(a)-----	7.0	30	15	82.6	15-20	7	6.3	60	20	4.1	1.9
1(b)-----	7.1	150	50	82.2	100	50	6.2	85	30	4.5	1.8
2(a)-----	7.5	20	8	82.2	10-15	4-5	6.5	30-40	10-15	3.8	2.0
2(b)-----	7.3	85	50	82.6	60	30-40	6.0	60	30	4.1	2.1

NOTE.—HCV=hazen color value; ICV=iodine color value.

From the data in the table, it will be seen that the process of this invention greatly reduces the discoloration found in the fatty-acid forerunnings of acids containing from 8 to 12 carbon atoms.

Furthermore, it will be recognized that although the above examples were conducted by using technical-grade silicon, silicon of greater or less purity may also be effectively employed, the technical-grade being preferred because of its availability and cost.

As technical-grade silicon known products may be used, which are silicon in mixture with certain impurities or silicon produced by melting and refining processes, which products are silicides of some metals, partly the metals being present as impurities, partly metals being present as a desired metal content, i.e. in ferro silicon alloys. Such metals are e.g. alkali metals and/or alkaline earth metals as Na, K, Mg or Ca; Al, B or Ti; heavy metals as Ni, Fe, Mn as caused by raw materials of said melting processes, while other elements as C, P, O, N, S are present as impurities of only small amount. Some of these products having only silicon contents of 15 to 45 wt. percent, most of 45 to 98 wt. percent Si.

While the novel principles of the invention have been described, it will be understood that various omissions, modifications and changes in these principles may be made by one skilled in the art without departing from the spirit and scope of the invention.

What is claimed is:

1. A process for the lightening and color stabilization of fatty-acid forerunnings of 8 to 12 carbon atoms which

comprises heating the fatty acids with about 0.1 to about 0.6% by weight of technical-grade silicon to temperatures of about 180° C. to about 250° C., and thereafter distilling off the treated fatty acids under vacuum.

2. The process of claim 1 in which said process is conducted in a discontinuous as well as in a continuous manner.

3. The process of claim 1 in which about 0.2 to about 0.3% by weight of the silicon is heated with the fatty-

acid forerunnings.

4. The process of claim 1 in which the fatty-acid forerunnings and the silicon are heated for a period of about 0.5 to about 4 hours.

5. The process of claim 1 in which the distillation forerunnings and the silicon were heated under normal pressure.

6. A process for the lightening and color-stabilization of fatty acid forerunnings containing from 8 to 12 carbon atoms which comprises heating the fatty acids with about 0.1 to 0.6% by weight of technical-grade silicon for about 0.5 to about 4 hours under normal pressure at a temperature of about 180° C. to about 250° C. and thereafter recovering the treated fatty acids by distillation.

7. The process of claim 6 in which the fatty-acid forerunnings and the silicon are heated for a period of from about 1 to about 3 hours.

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