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PROCESS FOR THE PRODUCTION OF SUCROSE ESTERS OF FATTY ACIDS

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This invention relates to a process for the production of sucrose esters of fatty acids.

Sucrose esters of fatty acids are known and are commercially used as emulsifiers. More recently, chiefly the sucrose esters of fatty acids having from 12 to 22 carbon atoms and at least one weakly hydrophilic group in the fatty acid chain have gained importance because they are characterized by outstanding detergency characteristics and are readily degraded biologically to absolutely innoxious substances (German patent application Z 10,316).

Sucrose esters of fatty acids are conventionally prepared by transesterification of lower alkyl esters of fatty acids with sucrose in a polar solvent (see British Patents 809,815 and 826,801), the molar ratio of fatty acid alkyl ester to sucrose being about 3:1 and more. Suitable catalysts include above all alkaline compounds, especially potassium carbonate. After completion of the transesterification, the predominant part of the solvent is removed under vacuum. Then a different suitable solvent must be added to prevent decomposition of the sucrose esters by excessively long heating and to precipitate excess sugar. The resultant solution of the sucrose ester must still be washed, and the sucrose ester is left in pure form after evaporation of the extractant.

This process has the disadvantage, above all when carried out in a commercial scale, that it is extremely complicated and requires a great excess of sucrose and large amounts of the polar solvent. Both the excess sucrose and the solvent must be recovered and re-used to make the process economic. This in turn requires additional purification procedures for both substances with the corresponding expense of equipment.

It is an object of the present invention to provide an improved process for the production of sucrose esters of fatty acids having from 12 to 22 carbon atoms, which comprises effecting the transesterification of sucrose with a lower fatty acid alkyl ester in a liquid two-phase system, one phase of which consists at the beginning of the reaction of a solution of the fatty acid alkyl ester in an aliphatic or aromatic hydrocarbon, and the other phase of which comprises sucrose and, if necessary or desired, the catalyst dissolved in a polar solvent.

Suitable polar solvents which may be used for the purposes of the invention are N-methyl morpholine, triethylamine, pyridine, quinoline, pyrazine, N-methyl-pyrazine, N,N'-dimethyl-pyrazine, 2-pyrrolidone, N-methyl-pyrrolidone as well as N,N'-dimethyl formamide and dimethyl sulfoxide which are particularly preferred. Suitable solvents for the lower alkyl esters of fatty acids are non-polar solvents boiling between 100° C. and 200° C., particularly suitable being aromatic and aliphatic hydrocarbons such as toluene, ethyl benzene, gasoline, cyclohexyl benzene, nonyl benzene, ligroin and the like. The ratio by weight of polar solvent to the liquid hydrocarbon is desirably 0.5:1 to 5:1, especially 2:1 to 2.5:1.

The process of the invention is generally applicable to the production of sucrose esters of fatty acids. It has been found to be particularly advantageous for the production of the commercially important sucrose esters of

fatty acids containing at least one weakly hydrophilic group such as a hydroxyl, carbonyl, oxime or alkoxy group.

The principle of the process consists in that the sucrose and the catalyst are initially dissolved at moderately elevated temperature of, for example, 70° to 90° C. in the polar solvent. Then the lower alkyl ester of the fatty acid the sucrose ester of which is desired to be prepared, especially the methyl or ethyl ester, is dissolved in the liquid hydrocarbon and the two phases are reacted at temperatures of between 40° and 180° C., especially about 80° to 90° C., while stirring. Initially, the alkyl ester remains almost completely in the hydrocarbon phase. The lower alcohol liberated during the transesterification distils and, at the same time, the dissolving power of the polar solvent for the hydrocarbon increases because the sucrose ester which is formed is better soluble in the polar solvent than the sucrose. After a reaction period of 5 to 12 hours and in general of about 8 to 9 hours, the transesterification is completed, and the two solvents are distilled off by increasing the temperature in the reaction mixture or the vaccum applied or by both of these measures and the sucrose ester produced remains in the residue.

The new process has the following advantages from the process engineering and economic points of view as compared with known methods: Sucrose and the lower alkyl ester of fatty acid may be fed in a molar ratio of 1:1. Thus, the process is not burdened by excess sugar, and the separation and purification of excess sucrose are eliminated. The total amount of solvent necessary for the transesterification is less than one-half of the quantity previously used. The yield of sucrose ester is increased by about 10% on an average due to the substantially more simple processing and elimination of all additional purification procedures.

EXAMPLE 1

Sucrose in an amount of 51.5 gms. (0.15 mole) was dissolved with 0.5 gm. of potassium carbonate in 120 gms. of dimethyl sulfoxide at 85° C. At the same time, a solution of 0.15 mole of 12-hydroxymethyl stearate in 50 gms. of cyclohexyl benzene was prepared and the two phases were then vigorously stirred for 9 hours at 85° C. under vacuum. Then the two solvents were distilled off under vacuum. The residue consisted of the sucrose ester of 12-hydroxystearic acid. The yield is more than 95%. The product may be used directly as such as the active detergent component of detergent compositions and lowers the surface tension of water of 10 deg. German hardness to 38 dynes/cm.

EXAMPLE 2

A mixture of 90 gms. of dimethyl sulfoxide, 50 gms. of gasoline boiling from 100° to 140° C., 30 gms. of sucrose, 27.6 gms. of 12-hydroxymethyl stearate and 0.5 gm. of potassium carbonate catalyst is vigorously stirred for 9 hours at 85° C. under a vacuum of 100 mm. Hg. After having distilled off the solvents, there remains sucrose-12-hydroxystearate having a surface tension of 38 dynes/cm. The yield is 96%.

Corresponding yields are obtained when transesterifying myristic acid or lauric acid methyl ester with sucrose.

What is claimed is:

1. A process for the production of sucrose mono-esters of fatty acids having from 12 to 22 carbon atoms by transesterification of sucrose with a lower alkyl ester of a fatty acid in inert solvents, which comprises (1) forming a liquid two-phase system, a first phase of this system consisting of a solution of said fatty acid alkyl ester in an aliphatic or aromatic hydrocarbon non-polar solvent having a boiling point above 100° C., and a second

phase of this system containing said sucrose and potassium carbonate catalyst dissolved in a polar solvent at from 70–90° C., said sucrose and said lower alkyl ester of a fatty acid being present in about a 1:1 mole ratio, (2) stirring the said two phases at a temperature of between 40–180° C. while distilling off lower alcohol liberated by the transesterification reaction, and (3) distilling off the remaining solvents.

2. The process of claim 1, wherein the ratio by weight of said polar solvent to said non-polar solvent is from 0.5:1 to 5:1.

3. The process of claim 2, wherein the ratio by weight of said polar solvent to said non-polar solvent is from 2:1 to 2.5:1.

4. The process of claim 1, wherein said fatty acid alkyl ester is the alkyl ester of a fatty acid having at least one weakly hydrophilic group in the fatty acid chain.

5. The process of claim 1, wherein said polar solvent is dimethyl sulfoxide.

6. The process of claim 1, wherein said polar solvent is dimethyl formamide.

7. The process of claim 4, wherein said weakly hydrophilic group in the fatty acid chain is a hydroxyl, a carbonyl, an oxime or an alkoxy group.

8. The process of claim 1, wherein said ester of a fatty acid is 12-hydroxymethyl stearate.

9. The process of claim 1, wherein said non-polar solvent comprises toluene, ethyl benzene, gasoline, cyclohexyl benzene, nonyl benzene, and ligroin.

10. The process of claim 2, wherein said non-polar solvent comprises toluene, ethyl benzene, gasoline, cyclohexyl benzene, nonyl benzene and ligroin.

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