Patent claims

1. A process to prepare a concentrate comprising a tocopherol compound (1), wherein the tocopherol compound (1) is selected from the group consisting of: α-Tocopherol, β-Tocopherol, γ-Tocopherol, δ-Tocopherol, α-Tocotrienol, β-Tocotrienol, γ-Tocotrienol, δ-Tocotrienol, α-Tocomonoenol, β-Tocomonoenol, γ-Tocomonoenol, δ-Tocomonoenol, α-MDT, β-MDT, γ-MDT, δ-MDT and their various mixtures; alpha tocopheryl acetate as a single stereoisomer or as a mixture of any two or more of its eight stereoisomers; the 8-isomer all-rac vitamin E; and the ester derivates of alpha tocopheryl acetate as a single stereoisomer or as a mixture of any two or more of its eight stereoisomers; **the ester derivates of** the 8-isomer all-rac vitamin E, and the process comprises the following steps in the following sequence:

(i) an evaporation step (10) in a first evaporator (20) of a feed stream (30) comprising (a) a fatty acid or its ester and (b) the tocopherol compound (1), wherein the first evaporator (20) is a thin film or short-path evaporator, and the concentration of the tocopherol compound (1) in the feed stream (30) is from 1 to 30% on a weight basis, wherein a first residue stream (50) and a first volatile stream (60) are separated in the evaporation step (10), the first volatile stream (60) comprises the fatty acid or its ester and the tocopherol compound (1), and the content of the tocopherol compound (1) in the first volatile stream (60) is between 5 to 25 wt. %, wherein the residue stream (50) contains 0.5 to 10 wt % of tocopherol compound (1), and the residue stream (50) comprises heavies which comprise fatty acids, monoglycerides, squalenes, sterols, di- and triglycerides, and heavy neutrals;

(ii) a first distillation step (70) in a first distillation apparatus (80) of the first volatile stream (60) to remove a second residue stream (90) and a second volatile stream (100) comprising the fatty acid or its ester, and to form a volatile product concentrate stream (110) comprising the tocopherol compound (1), wherein the first distillation apparatus (80) comprises a first and a second column (81, 82) in fluid communication with each other;

a condensing sub-step in (i) evaporation step (10) for condensing the first volatile stream (60) in the first evaporator (20) equipped with a condensing means, before it is fed to the first distillation step (70);

the (ii) first distillation step in the first distillation apparatus (80) wherein the number of theoretical stages in the first distillation apparatus (80) are between 15 and 30, wherein in the first and second columns (81, 82) the bottom pressure is at most 1 mbar (abs) and the bottom temperature is at most 250°C;

wherein the first and second columns (81, 82) of the first distillation apparatus (80) contain random or structured packings.

2. The process of claim 1, wherein each of the first and second columns (81, 82) is equipped with a second and third evaporator (120, 122) and a first and second condenser (130, 132), wherein the pressure drop in each one of the second and third evaporators (120, 122) and each one of the first and second condensers (130, 132) is less than 0.2 mbar.

3. The process of claim 2, wherein each one of the second and third evaporators (120, 122) is a falling film evaporator or a wiped film evaporator.

4. The process of any one of claims 1 to 3, wherein each of the first and second columns (81, 82) is equipped with a first and second boot (140, 142) having a reduced first and second boot diameter (144, 146) relative to the first and second shell diameters (83, 84) of the first and second columns (81, 82).

5. The process of any one of claims 1 to 4, wherein the flange connections of the first evaporator (20), the first distillation apparatus (80), and the piping in fluid connection with the first evaporator (20) and the first distillation apparatus (80) having a diameter of at least 3 inches are either tongue and groove flanges or lip seal welded flanges or their combinations.

6. The process of any one of claims 1 to 5, wherein the feed stream (30) has been produced in a feed stream (30) production process comprising the steps in the following sequence:

(i) a physical refining step (150) of a vegetable oil (160) to form a first fatty acid distillate stream (170) comprising a free fatty acid and the tocopherol compound (1),

(ii) removing a first portion of the free fatty acid from the first fatty acid distillate stream (170) by means of a second distillation step (190) in a second distillation apparatus (200) to form a second fatty acid distillate stream (210) and a third residue stream (220) comprising a remaining portion of the free fatty acid and the tocopherol compound (1),

(iii) esterifying substantially all of the remaining portion of the free fatty acid in the third residue stream (220) in an esterification step (230) to form an esterified product stream (240) comprising a fatty acid ester, a glyceride, and the tocopherol compound (1),

(iv) transesterifying the glyceride of the esterified product stream (240) in a transesterification step (250) to form a transesterified product stream (260),

(v) removal of a fatty acid ester distillate (264) from the transesterified product stream (260) by means of a third distillation step (270) in a third distillation apparatus (280) to form the feed stream (30).

7. The process of claim 6, wherein a bottom pressure in the second distillation apparatus (200) is less than or equal to 12 mbar (abs).

8. The process of either claim 6 or 7, wherein a bottom pressure in the third distillation apparatus (280) is less than or equal to 12 mbar (abs).

9. The process of any one of claims 6 to 8, wherein the esterification step (230) is carried out in a continuous process using an immobilised heterogeneous catalyst.

10. The process of any one of claims 6 to 8, wherein the esterification step (230) and the transesterication step (250) are carried out using methanol or ethanol as reactants.

11. The process of any one of claims 1 to 5, wherein the feed stream (30) is a residue stream obtained from the distillation of a stream of fatty acid ester.

12. The process of any one of claims 1 to 5, wherein the feed stream (30) is a stream of a fatty acid ester.

13. An apparatus (1000) for the process of any one of claims 6 to 10, wherein the apparatus (1000) comprises the following sequence of units in fluid communication with each other:

(i) a stripping column (290) for a physical refining of a vegetable oil (160) to form a first fatty acid distillate (170) comprising a free fatty acid and a tocopherol compound (1),

(ii) a second distillation apparatus (200) for removing a first portion of a free fatty acid from the fatty acid distillate (170) to form a second fatty acid distillate stream (210) and a third residue stream (220) comprising a remaining portion of the free fatty acid and the tocopherol compound (1),

(iii) an esterification reactor (300) for esterifying substantially all of the free fatty acid in the third residue stream (220) in an esterification step (230) to form an esterified product stream (240),

(iv) a transesterification reactor (310) for transesterifying the glyceride in the esterified product stream (240) to form a transesterified product stream (260),

(v) a third distillation apparatus (280) for removing a fatty acid ester distillate (264) from the transesterified product stream (260) to form a feed stream (30) comprising (a) a fatty acid or its ester and (b) the tocopherol compound (1),

(vi) a first evaporator (20) which is a thin film or short-path evaporator for separating a first residue stream (50) and a first volatile stream (60), wherein the first volatile stream (60) comprises the fatty acid or its ester and the tocopherol compound (1), and

(vii) a first distillation apparatus (80) comprising a first and a second column (81, 82) in fluid communication with each other and for the distillation of a first volatile stream (60) to remove a second residue stream (90) and a second volatile stream (100) comprising the fatty acid or its ester, and to form a volatile product concentrate stream (110) comprising the tocopherol compound (1).